ECSS-E-HB-32-20 Part 4A
20 March 2011

EUROPEAN COOPERATION

ECSS

FOR SPACE STANDARDIZATION

Space engineering

Structural materials handbook - Part 4: Integrity control, verification guidelines and manufacturing

NOTE:
This pdf-file does not contain automatic cross-references. To make use of the cross-references please use the MS Word version of this document.

ECSS Secretariat
ESA-ESTEC
Requirements & Standards Division
Noordwijk, The Netherlands

Foreword

This Handbook is one document of the series of ECSS Documents intended to be used as supporting material for ECSS Standards in space projects and applications. ECSS is a cooperative effort of the European Space Agency, national space agencies and European industry associations for the purpose of developing and maintaining common standards.

This handbook has been prepared by the ECSS-E-HB-32-30 Working Group, reviewed by the ECSS Executive Secretariat and approved by the ECSS Technical Authority.

Published by: ESA Requirements and Standards Division
ESTEC, P.O. Box 299,
2200 AG Noordwijk
The Netherlands

Change log

ECSS-E-HB-32-20 Part 4A 20 March 2011	First issue

Table of contents

Change log 3

Introduction 28

33 Aspects of damage tolerance 29

33.1 Introduction 29

33.1.1 Damage tolerance 29

33.1.2 Damage events 29

33.1.3 Damage tolerance criteria 29

33.2 General guidelines 30

33.2.1 Fracture control 30

33.2.2 Durability 30

33.2.3 Damage 30

33.2.4 Defects in composites 30

33.2.5 Impact damage 33

33.3 Damage tolerance criteria 34

33.3.1 General 34

33.3.2 Load paths 35

33.3.3 Environment 35

33.3.4 Damage 35

33.3.5 Residual strength 36

33.3.6 Safe-life 36

33.3.7 Fail-safe 36

33.3.8 Stiffness 36

33.3.9 Loads 36

33.3.10 Environmental factors 36

33.3.11 Inspection 36

33.3.12 Summary 37

33.4 Defect standardisation 37

33.4.1 General 37

33.4.2 Types of defects 38

33.5 Effect of macroscopic defects on static load 40
33.5.1 Composite failure 40
33.5.2 Use of fracture mechanics 40
33.5.3 Multi-angle laminates 40
33.6 Test and analysis 41
33.6.1 General 41
33.6.2 Constituent tests 43
33.6.3 Lamina tests 44
33.6.4 Laminate tests 44
33.6.5 Structural element and component tests 44
33.6.6 Structural sub-component or full-scale tests 44
33.6.7 Delamination 44
33.7 Other features of damage-tolerance design 46
33.7.1 Acoustic fatigue 46
33.7.2 Proof testing 46
33.7.3 Metallic parts 46
33.7.4 Thermal and meteoroid protection system 46
33.7.5 Impact dynamics – crashworthiness 46
33.7.6 Flammability, toxicity and off-gassing 46
33.7.7 Lightning protection 47
33.8 Improving damage tolerance with higher toughness laminates 47
33.8.1 General 47
33.8.2 Material-related aspects 47
33.8.3 Lay-up aspects 48
33.9 References 48
33.9.1 General 48
33.9.2 ECSS documents 49
33.9.3 MIL standards 49

34 Inspection and quality assurance 50
34.1 Introduction 50
34.2 Fabrication and quality assurance 50
34.2.1 Quality assurance 50
34.3 Inspection, maintenance and repair 51
34.3.1 Inspection and maintenance 51
34.3.2 Repairs 52
34.4 Inspection 52
34.4.1 Basic considerations 52

34.5 Definition of inspection procedures 53
34.5.1 Procedures 53
34.5.2 Thin composite laminates 54
34.5.3 Thick composite laminates 54
34.6 Non-destructive inspection techniques 55
34.6.1 Introduction 55
34.6.2 Damage tolerant designs 55
34.6.3 Advances in NDT 55
34.6.4 Techniques 56
34.6.5 Smart technologies for condition monitoring 57
34.7 Defects and anomalies for detection 57
34.7.1 General 57
34.7.2 Emphasis of NDT development 58
34.8 Overview of NDT techniques 58
34.8.1 Selection of NDT technique 58
34.8.2 Detection of defects 59
34.9 Traditional contact NDT techniques 65
34.9.1 General 65
34.9.2 Visual inspection 65
34.9.3 Dye penetrant 65
34.9.4 Magnetic particle 65
34.9.5 Mechanical resonance or impedance 65
34.10 Ultrasonic techniques 66
34.10.1 Introduction 66
34.10.2 Modes of examination 67
34.10.3 Ultrasonic signal presentation 67
34.10.4 Data and image processing 67
34.10.5 Equipment calibration 68
34.10.6 Large area scanning 69
34.10.7 Acousto-ultrasonics 69
34.10.8 Lamb wave (guided wave) 70
34.10.9 Rayleigh wave techniques 71
34.10.10 Backscattering techniques 71
34.10.11 Transducer technology 72
34.10.12 Air-coupled techniques (non-contact) 75
34.11 Acoustic emission 78
34.12 Laser ultrasonics 79

34.12.1 General ... 79
34.12.2 Basic principles ... 80
34.13 Holography ... 81
34.13.1 Introduction ... 81
34.13.2 Laser interferometric techniques ... 82
34.13.3 Vibration, pressure and thermal loading ... 83
34.13.4 Electronic imaging ... 83
34.13.5 Examination of space structures ... 84
34.14 Laser shearography ... 85
34.14.1 Introduction ... 85
34.14.2 Basic technique ... 85
34.14.3 Characteristics of shearography ... 86
34.15 Thermography ... 87
34.15.1 Introduction ... 87
34.15.2 Techniques ... 88
34.15.3 Heat sources and pulse duration ... 92
34.15.4 Detection capabilities ... 92
34.15.5 Limitations of thermography ... 93
34.16 X-ray radiography ... 94
34.16.1 Introduction ... 94
34.16.2 X-ray techniques ... 95
34.16.3 Digital radiographic system developments ... 96
34.16.4 Defect resolution ... 99
34.17 Computer tomography ... 99
34.17.1 Introduction ... 99
34.17.2 Basic technique ... 99
34.17.3 Attributes of CT scanning ... 100
34.17.4 Micro-tomography ... 101
34.18 Eddy currents ... 101
34.18.1 Introduction ... 101
34.18.2 Developments in eddy current techniques ... 102
34.19 Other NDT techniques ... 103
34.19.1 General ... 103
34.19.2 Microwave ... 104
34.19.3 Heat and photosensitive agents ... 104
34.19.4 Electrical impedance ... 104
34.19.5 Acoustic microscopy ... 104

34.19.6 Nuclear magnetic resonance (NMR) 105
34.19.7 Neutron radiography 105
34.19.8 Gamma radiography 105
34.19.9 D sight - Diffracto sight 105
34.20 NDT for adhesive bond defects 106
34.20.1 General 106
34.20.2 Disbonds, voids and porosity 106
34.20.3 Cohesion and adhesion 107
34.21 NDT for space applications 107
34.21.1 Introduction 107
34.21.2 Developments in inspection 108
34.21.3 Summary of NDT techniques 108
34.22 Rupture tests 110
34.22.1 Application 110
34.23 Test related items 111
34.23.1 General 111
34.23.2 Major items 111
34.23.3 Qualification tests 112
34.24 Structural test reports 123
34.25 Analysis and test documents 124
34.26 References 126
34.26.1 General 126
34.26.2 ECSS documents 138

35 Objective of verification 139
35.1 Introduction 139
35.1.1 General 139
35.1.2 Composite structures 139
35.1.3 Composite materials 139

36 Procurement specifications for raw materials 141
36.1 Introduction 141
36.1.1 Guidelines 141
36.2 Specification methods 141
36.2.1 Fiche sheet principle 141
36.2.2 Specific material specification 142
36.3 Guidelines on resin procurement 142
36.3.1 Scope 142
36.3.2 Basic information to be provided by the supplier 143

36.4 Guidelines on carbon and other fibre prepreg procurement 147
36.4.1 Scope 147
36.4.2 Description and classification of prepreg 147
36.4.3 Prepreg charachteristics for qualification and batch testing 148
36.4.4 Laminate Characteristics 151
36.4.5 Test methods 152
36.5 References 153
36.5.1 ECSS documents 153
36.5.2 ESA publications 153

37 Philosophy of assessment of design allowables 154
37.1 Introduction 154
37.2 Carbon fibre reinforced plastics 154
37.2.1 Design values 154
37.3 Definition of design allowables 155
37.3.1 'A' basis allowable 155
37.3.2 'B' basis allowable 155
37.4 Development procedure 155
37.5 Test data 155
37.5.1 Material 155
37.5.2 Data 156
37.6 References 160
37.6.1 General 160

38 Manufacturing techniques 161
38.1 Introduction 161
38.1.1 Processing techniques 161
38.1.2 Process selection 161
38.2 Process selection criteria 162
38.3 Hand lay-up 162
38.3.1 General 162
38.3.2 Wet lamination 163
38.3.3 Prepreg lay-up 166
38.4 Autoclave moulding 170
38.4.1 Use 170
38.4.2 Basic process 170
38.4.3 Bag moulding methods 171
38.4.4 Features of bag moulding 174
38.4.5 Bagging techniques 175

38.4.6 Tool materials 176
38.4.7 Cure schedules 178
38.5 Filament winding 179
38.5.1 Basic process 179
38.5.2 Applications 179
38.5.3 Winding process 180
38.5.4 Mandrels 181
38.5.5 Sandwich constructions 181
38.5.6 Tape winding 182
38.6 Filament-winding machines 182
38.6.1 General 182
38.6.2 Polar winding 182
38.6.3 Helical winding 184
38.7 Resin transfer moulding 185
38.7.1 General 185
38.7.2 Basic process 185
38.7.3 Applications 186
38.7.4 Fibre preforms 186
38.7.5 Resin injection 187
38.7.6 Process variables 188
38.7.7 Process advantages 188
38.8 Pultrusion 189
38.8.1 Use 189
38.8.2 Basic process 189
38.8.3 Pull-forming 190
38.9 Table rolling 191
38.9.1 Tubular sections 191
38.10 Pressure and mechanical forming 191
38.10.1 General 191
38.10.2 Fibre metal laminates 191
38.10.3 Thermoplastic composites 192
38.11 Injection moulding 192
38.11.1 General 192
38.11.2 Short fibre composites 192
38.12 Radiation curing 193
38.12.1 Process developments 193
38.12.2 EB electron beam curing 194

38.12.3 Microwave 198
38.12.4 UV curing 199
38.13 Preform technology 199
38.13.1 General 199
38.13.2 Braids 200
38.13.3 Commingled fibre performs 200
38.14 Fibre placement 201
38.14.1 Introduction 201
38.14.2 Advantages 204
38.14.3 Repeatability in lay-up and properties 205
38.14.4 Mixed materials in the same lay-up 206
38.14.5 Major advantages of fibre placement compared with other technologies 207
38.14.6 Examples of European launcher and satellite structures made by fibre placement 208
38.15 Out of autoclave 209
38.15.1 Introduction 209
38.15.2 Aerospace structural parts 209
38.15.3 Composite materials 210
38.15.4 Tooling 210
38.16 References 211
38.16.1 General 211
38.16.2 ECSS standards 217

39 Machining techniques 218
39.1 Introduction 218
39.2 Basic rules 218
39.2.1 General 218
39.2.2 Equipment 218
39.3 Routing 219
39.3.1 Carbon/epoxy composites 219
39.3.2 Aramid/epoxy composites 219
39.3.3 Trimming and bevelling 220
39.4 Sanding 221
39.4.1 General 221
39.4.2 Fit or trim 221
39.4.3 Bonding 222
39.4.4 Aramid/epoxy composites 223
39.5 Sawing 223

39.5.1 Basic rules 223
39.5.2 Bandsawing 223
39.5.3 Circular sawing 224
39.5.4 Sabre sawing 225
39.6 Countersinking 226
39.6.1 General 226
39.6.2 Carbon and glass reinforced epoxy 226
39.6.3 Aramid/epoxy composites 226
39.7 Counterboring 227
39.8 Milling 228
39.8.1 Basic Rules 228
39.8.2 Plunge-cut milling 228
39.9 Drilling 229
39.9.1 Basic problems 229
39.9.2 Drill bits for composites 230
39.10 Orbital drilling 232
39.10.1 Introduction 232
39.10.2 Advantages 233
39.10.3 Drilling parameters 233
39.10.4 Equipment 233
39.10.5 Application examples 234
39.11 References 235
39.11.1 General 235
39.11.2 ECSS standards 235

40 Manufacturing costs 236

40.1 Introduction 236
40.2 Cost drivers 237
40.2.1 General 237
40.2.2 Project cost drivers 237
40.2.3 Mass for cost budgets 237
40.2.4 Materials selection 237
40.2.5 Design for manufacturing 238
40.2.6 Manufacturing expertise 238
40.2.7 Tooling and consumables 238
40.2.8 Processing times 238
40.2.9 Labour costs 239
40.2.10 Part count 239

40.2.11 Unit numbers 239
40.2.12 Product life and sourcing 240
40.3 Cost optimisation 240
40.3.1 Load-carrying structures 240
40.4 Cost advantages of composites 245
40.4.1 Material costs 245
40.4.2 Manufacturing costs 245
40.5 Satellite thrust cylinders 245
40.6 Launcher fairing cylinders 246
40.6.1 General 246
40.6.2 Potential fairing designs 246
40.6.3 Selection of designs 250
40.6.4 Process and material trade-off 251
40.7 Thermoplastic versus thermoset 253
40.7.1 General 253
40.7.2 Recurring spacecraft components 254
40.7.3 Thermoplastic composites in aircraft 259
40.7.4 Conclusions 261
40.8 Automation 261
40.9 Cost analysis methods 262
40.10 References 262
40.10.1 General 262

41 Repair of composites 264

41.1 Introduction 264
41.2 Damage classification 264
41.2.1 Life-histories of composite structures 264
41.2.2 Inspection and criticality of damage 264
41.2.3 Repair zones 265
41.3 Damage categories 265
41.3.1 Impact energy levels 265
41.3.2 Damage types 266
41.4 Basic repair concepts 269
41.4.1 General 269
41.4.2 Basic types of repairs 269
41.4.3 Repair selection 269
41.5 Flush repair concepts 271
41.5.1 Applications 271

41.5.2 Post-repair properties 271
41.5.3 Design 271
41.5.4 Manufacture 271
41.6 Bonded external repair concepts 272
41.6.1 Applications 272
41.6.2 Post-repair properties 272
41.6.3 Design 272
41.6.4 Manufacture 273
41.7 Bolted external repair concepts 273
41.7.1 Applications 273
41.7.2 Post-repair properties 274
41.7.3 Design 274
41.7.4 Manufacture 274
41.8 Sandwich structure repair concepts 275
41.8.1 No core damage 275
41.8.2 Core damage 275
41.8.3 Manufacture 275
41.9 References 276
41.9.1 General 276

42 Basic characteristics of new advanced materials 278

42.1 Introduction 278
42.2 Material groups 278
42.2.1 Metallic materials 278
42.2.2 Ceramic materials 279
42.2.3 Material classification 279
42.3 Features of alloys and metal matrix composites 281
42.3.1 Structural alloys 281
42.3.2 Properties 282
42.3.3 Material developments 282
42.3.4 Effects of reinforcements 283
42.3.5 Processing 283
42.4 Features of ceramic and inorganic composites 284
42.4.1 Features 284
42.4.2 Properties 285
42.4.3 High-temperature use 285
42.5 High temperature and interface phenomena 286
42.5.1 Effects of processing temperature 286

42.5.2 High-temperature applications 287
42.6 Refractory and ceramic fibres 288
42.6.1 Introduction 288
42.6.2 Types of refractory and ceramic fibre 288
42.6.3 Use in composites 289
42.6.4 Thermal stability 290
42.7 References 303
42.7.1 General 303

43 Technical ceramics 305
43.1 Introduction 305
43.1.1 General 305
43.1.2 Technical ceramics for space structures 306
43.2 Material groups 307
43.2.1 Composition 307
43.2.2 Oxides 307
43.2.3 Carbides 309
43.2.4 Nitrides 309
43.2.5 Elemental 311
43.2.6 Glass-ceramics 313
43.2.7 Sources 313
43.3 Design aspects 315
43.3.1 Guidelines 315
43.3.2 Property and performance evaluation 316
43.4 Processing aspects 319
43.4.1 Manufacturing processes 319
43.4.2 Health and safety 320
43.4.3 Green shapes 320
43.4.4 Densification 323
43.4.5 Combined shaping and densification 323
43.4.6 Vapour-phase techniques 324
43.4.7 Machining 324
43.4.8 Joining 324
43.5 Applications 325
43.5.1 Industrial 325
43.5.2 Aerospace 326
43.5.3 Materials 327
43.6 Aluminium oxide: Alumina 331

43.6.1 Typical characteristics ... 331
43.6.2 Typical properties ... 332
43.7 Aluminium nitride ... 335
43.7.1 Typical characteristics ... 335
43.7.2 Typical properties ... 336
43.8 Boron carbide ... 338
43.8.1 Typical characteristics ... 338
43.8.2 Typical properties ... 338
43.9 Boron nitride ... 340
43.9.1 Typical characteristics ... 340
43.9.2 Typical properties ... 341
43.9.3 Space application examples ... 342
43.10 Carbon ... 344
43.10.1 Typical characteristics ... 344
43.10.2 Typical properties ... 347
43.11 Silicon oxide: Silica ... 351
43.11.1 Typical characteristics ... 351
43.11.2 Typical properties ... 351
43.12 Silicon carbide ... 353
43.12.1 Typical characteristics ... 353
43.12.2 Typical properties ... 354
43.12.3 Materials for space applications ... 356
43.12.4 Cesic® ... 357
43.12.5 SiC 54S® ... 363
43.12.6 SiC-100® ... 366
43.13 Silicon nitride ... 378
43.13.1 Typical characteristics ... 378
43.13.2 Typical properties ... 379
43.14 Zirconium oxide: Zirconia ... 380
43.14.1 Typical characteristics ... 380
43.14.2 Typical properties ... 380
43.15 Structural glass: Glass ceramics ... 382
43.15.1 Introduction ... 382
43.15.2 Commercially-available glass and glass ceramics ... 382
43.15.3 Glass ceramics for space applications ... 387
43.15.4 ZERODUR® ... 388
43.15.5 ZERODUR®: Properties ... 390

43.15.6 ZERODUR®: Design 394
43.15.7 ZERODUR®: Application examples 401
43.15.8 ULE® Ultra low expansion titanium-silicate glass 401
43.15.9 ULE®: Properties 402
43.15.10 ULE®: Design 404
43.15.11 ULE®: Application examples 404
43.16 Speciality materials 405
43.16.1 Introduction 405
43.16.2 Beryllia 405
43.16.3 Sapphire 406
43.16.4 Synthetic diamond 406
43.17 CVD diamond 407
43.17.1 Introduction 407
43.17.2 Typical characteristics 408
43.17.3 Typical properties 408
43.17.4 Application examples 410
43.18 Cesic®: NIRSpec optical bench 414
43.18.1 Introduction 414
43.18.2 Material characteristics 414
43.18.3 Development of optical bench 415
43.18.4 Conclusions 422
43.19 Zerodur®: Applications case study 422
43.19.1 Introduction 422
43.19.2 Terrestrial mirrors 423
43.19.3 Optical bench 423
43.20 References 424
43.20.1 General 424
43.20.2 Sources 432
43.20.3 ECSS documents 432
43.20.4 Other standards 432
44 Magnesium alloys and their composites 433
44.1 Introduction 433
44.1.1 General 433
44.1.2 Features 433
44.1.3 Alloy classification 433
44.1.4 Environmental aspects 435
44.1.5 Machining 435

44.1.6 MMC - metal matrix composites 436
44.1.7 Material availability 436
44.1.8 Future developments 437
44.2 Magnesium alloys 437
44.2.1 Chemical composition 437
44.2.2 Mechanical properties 440
44.3 Discontinuously reinforced magnesium composites 446
44.3.1 Types of discontinuous reinforcement 446
44.3.2 Effect of particulate additions 447
44.3.3 Processing 447
44.3.4 Properties 447
44.4 Continuously reinforced magnesium composites 448
44.4.1 Types of composite 448
44.4.2 Features 449
44.4.3 Processing 449
44.4.4 Properties 449
44.4.5 Environmental resistance 450
44.5 Potential applications 451
44.5.1 Alloys 451
44.5.2 Composites 451
44.5.3 Space 451
44.6 References 453
44.6.1 General 453
44.6.2 Sources 455
44.6.3 Organisations 455
44.6.4 ECSS standards 456
44.6.5 ASTM standards 456

45 Copper alloys and their composites 457

45.1 Introduction 457
45.2 Copper alloys 457
45.2.1 Precipitation hardening 457
45.3 Copper ODS alloys 458
45.3.1 Types and effects of dispersions 458
45.3.2 Properties 458
45.4 Discontinuously reinforced copper composites 460
45.4.1 General 460
45.4.2 Problems 460

45.5 Continuously reinforced copper composites 461
45.5.1 General 461
45.5.2 Types and effects of reinforcements 461
45.5.3 Properties 461
45.6 Potential applications 462
45.7 References 462
45.7.1 General 462

Figures

Figure 33.2-1 – Summary: Defects in composite materials 32
Figure 33.4-1 - Metal structures: Idealisation of a volumetric defect 38
Figure 33.4-2 - Composite structures: Idealisation of defects by standard circular defect 39
Figure 33.6-1 - Building block approach: Test programme development 42
Figure 33.6-2 - Building block approach: Composite materials 43
Figure 34.10-1 - Leaky-Lamb-wave technique for composites 71
Figure 34.10-2 - Detection of fibre orientation by backscattering measurement 72
Figure 34.10-3 – Phased-array transducers: Element configurations 73
Figure 34.10-4 – Phased-array transducers: Functionality 74
Figure 34.10-5 - Non-contact ultrasonics: Air-coupled sensor configurations 76
Figure 34.10-6 - Non-contact ultrasonics: Capacitive sensors in plate wave configuration set-up 77
Figure 34.10-7 – Airscan™ (air-coupled ultrasonics) system: Non-contact NDI of Fokker solar panel substrates 78
Figure 34.12-1 - Laser ultrasonic test system: Diagrammatic representation 80
Figure 34.13-1 - Holography by electronic imaging with random vibration excitation 84
Figure 34.14-1 - Electronic shearographic system: Schematic diagram 85
Figure 34.15-1 - Basic thermographic technique 88
Figure 34.15-2 - Thermal-wave imaging: Photo-optical detection 90
Figure 34.15-3 - Thermal-wave imaging: Photothermal and piezoelectric detection 91
Figure 34.15-4 - Thermal-wave imaging: Photoacoustic gas cell 91
Figure 34.16-1 - Typical digital radiography systems 97
Figure 34.17-1 - Computer tomography facility 100
Figure 34.19-1 - D sight - Diffracto sight schematic 106
Figure 37.5-1 - Probability of exceedance: Tensile strength 0° 157
Figure 37.5-2 - Probability of exceedance: Tensile strength 90° 158
Figure 37.5-3 - Probability of exceedance: Compressive strength 90° 159
Figure 37.5-4 - Probability of exceedance: Interlaminar shear strength 160

Figure 38.3-1 - Hand lay-up process for mat or cloth 163
Figure 38.3-2 - Prepreg: Direct lay-up 167
Figure 38.3-3 - Prepreg: Indirect lay-up 167
Figure 38.4-1 - Vacuum bag moulding method, with vertical bleeder 172
Figure 38.4-2 - Pressure bag moulding 172
Figure 38.4-3 - Pressure bag moulding: Press modifications 173
Figure 38.4-4 - Autoclave moulding method, with vertical bleeder 173
Figure 38.4-5 - Bag moulding: Edge bleed out system 176
Figure 38.4-6 - Typical cure schedule for autoclaved prepregs 178
Figure 38.5-1 - Filament winding: Impregnation of fibres 180
Figure 38.6-1 - Filament winding: Polar vertical 183
Figure 38.6-2 - Filament winding: Polar horizontal 183
Figure 38.6-3 - Filament winding: Polar tumbling 184
Figure 38.6-4 - Filament winding: Helical 184
Figure 38.7-1 - Resin transfer moulding (RTM): Process 185
Figure 38.8-1 - Pultrusion: Basic process 190
Figure 38.12-1 – EB curing: Examples of parts and structures 197
Figure 38.13-1 - Preform technology: Architecture of woven commingled AS4/PEEK 150g 0°/90° 201
Figure 38.14-1 – Fibre placement: Schematic view of the system 202
Figure 38.14-2 – Fibre placement: Head 203
Figure 38.14-3 – Fibre placement: General view of working area 203
Figure 38.14-4 – Fibre placement: Complex curvature 204
Figure 38.14-5 – Fibre placement: Specific orientations 205
Figure 38.14-6 – Fibre placement: Complex trajectories 206
Figure 38.14-7 – Fibre placement: Variation of band width 206
Figure 38.14-8 – Fibre placement: Mixed-material lay-up 207
Figure 39.3-1 - Router: Opposed helix for aramid/epoxy 220
Figure 39.3-2 - Router for trimming aramid/epoxy composites 221
Figure 39.4-1 - Sanding of composites 222
Figure 39.5-1 - Reverse bandsawing aramid/epoxy laminates 224
Figure 39.5-2 - Circular sawing: Feed rate versus thickness of composite materials 225
Figure 39.5-3 - Sabre sawing: Alternating tooth blade for aramid/epoxy 226
Figure 39.6-1 - Countersink for aramid/epoxy composite 227
Figure 39.8-1 - Proper milling technique 228
Figure 39.9-1 - Drill bits for composites 230
Figure 39.9-2 - Daggar drill in hand feed units 232
Figure 39.10-1 – Orbital drilling principle 232

Figure 39.10-2 – Orbital drilling: Equipment mounted on a jig 233
Figure 39.10-3 – Orbital drilling: Application examples 234
Figure 40.3-1 - Mass/cost sensitivity for central thrust structure 242
Figure 40.3-2 - Mass/cost sensitivity for complete structure 243
Figure 40.3-3 - Mass to cost envelope for material improvements 244
Figure 40.6-1 - Candidate fairing and intertank constructions....................247
Figure 40.6-2 - Fairing cylinder costs for different designs (life cycle costs per unit)....................248
Figure 40.6-3 - Launcher fairing cylinder: Kepner-Tregoe evaluation scores for the section designs 250
Figure 40.6-4 - Launcher fairing: Unit life cycle costs for sandwich fairing cylinder designs for different fabrication processes 252
Figure 40.6-5 - Launcher fairing: Kepner-Tregoe evaluation scores for processes to fabricate the monocoque and sandwich cylinder....................253
Figure 40.7-1 - Thermoplastic components: Manufacturing unit costs as a function of production volumes....................257
Figure 40.7-2 - Relative part costs for thermoplastic aircraft components: Dornier 328 landing flap rib 260
Figure 41.3-1 - Damage categories: Scratch 266
Figure 41.3-2 - Damage categories: Notch 266
Figure 41.3-3 - Damage categories: Chipping 267
Figure 41.3-4 - Damage categories: Debonding 267
Figure 41.3-5 - Damage categories: Delamination....................268
Figure 41.3-6 - Damage categories: Perforation 268
Figure 41.4-1 - Basic repair concepts 270
Figure 41.5-1 - Typical flush repair....................271
Figure 41.5-2 - Flush repair, used by Airbus Industrie 272
Figure 41.5-3 - Flush repair with adhesive film and cover plies 272
Figure 41.6-1 - External repair: Pre-cured patches 273
Figure 41.6-2 - External repair: Cured in place 273
Figure 41.7-1 - Bolted and bonded repair using a composite patch....................274
Figure 41.8-1 - Sandwich repair 276
Figure 42.2-1 - Classification of advanced metallic and ceramic materials with temperature 280
Figure 42.2-2 - Specific strength against temperature for advanced materials....................281
Figure 42.6-1 - SiC Nicalon fibre tensile strength at elevated temperature....................293
Figure 42.6-2 - SiC Nicalon standard grade fibre tensile strength at elevated temperature 294
Figure 42.6-3 - SiC Nicalon ceramic grade fibre: tensile strength at elevated temperature 295

Figure 42.6-4 - SiC Nicalon fibre: tensile strength after high temperature exposure in air 296

Figure 42.6-5 - SiC Nicalon fibre: tensile strength after high temperature exposure in argon 296

Figure 42.6-6 - SiC fibres Nicalon and Tyranno: tensile strength at elevated temperature 297

Figure 42.6-7 - SiC fibres Nicalon and Tyranno: modulus at elevated temperature 298

Figure 42.6-8 - Alumina fibres: tensile strength at elevated temperature 298

Figure 42.6-9 - Alumina fibres: modulus at elevated temperature 299

Figure 42.6-10 - Alumina fibres: tensile strength against exposure to elevated temperature 299

Figure 42.6-11 - Alumina fibres: tensile strength against exposure time 300

Figure 42.6-12 - Alumina-boria-silica single filament: tensile strength at elevated temperature in air 300

Figure 42.6-13 - Alumina-boria-silica single filament: tensile modulus at elevated temperature in air 301

Figure 42.6-14 - Modulus for various fibres and matrix materials 302

Figure 42.6-15 - Coefficients of thermal expansion for various fibres and matrix materials 303

Figure 43.4-1 – Technical ceramics: Manufacturing processes 320

Figure 43.9-1 – Technical ceramics: pyrolitic boron nitride – structure 341

Figure 43.12-1 – Cesic®: Basic processing sequence 358

Figure 43.12-2 – Cesic®: Silicon-infiltration processing equipment 359

Figure 43.12-3 – Cesic®: Coefficient of thermal expansion 361

Figure 43.12-4 – Cesic® ceramic: Example of integrally-stiffened panel 362

Figure 43.12-5 – SiC 54S® ceramic: Process sequence 363

Figure 43.12-6 – SiC 54S®: NIRSpec prototype mirror 366

Figure 43.12-7 – SiC-100®: Production process - schematic for Herschel primary mirror 368

Figure 43.12-8 – SiC-100®: 1m diameter ‘Demonstration Mirror’ – ready to polish 373

Figure 43.12-9 – SiC-100®: 1m diameter ‘Demonstration Mirror’ – green part joining by ceramic bonding 374

Figure 43.12-10 – SiC-100®: 1.35 m diameter HERSCHEL Demonstrator Breadboard – before and after brazing 374

Figure 43.12-11 – SiC-100®: 3.5 m diameter HERSCHEL Primary Mirror – manufactured by brazing 12 SiC segments 375

Figure 43.12-12 – SiC-100®: 3.5 m diameter HERSCHEL Primary Mirror – after assembly 376

Figure 43.12-13 – SiC-100®: NIRSpec Optical Bench Breadboard 377

Figure 43.12-14 – SiC-100®: Optical Assembly of NIRSpec for James Webb Space Telescope 377

Figure 43.15-1 - Glass ceramics: Coefficient of thermal expansion with temperature - materials for optical instruments in space 387

Figure 43.15-2 - Zerodur®: Production process schematic........ 388

Figure 43.15-3 - Zerodur®: Annealing process 389

Figure 43.15-4 - Zerodur®: Summary of engineering data 390

Figure 43.15-5 - Zerodur®: Optical transmission curve 391

Figure 43.15-6 - Zerodur®: Thermal expansion – cryogenic behaviour........ 392

Figure 43.15-7 - Zerodur®: Coefficient of thermal expansion – different glass ceramics 392

Figure 43.15-8 - Zerodur®: Thermal expansion of different glass ceramics 393

Figure 43.15-9 - Zerodur®: Thermal conductivity characteristics of different glasses 393

Figure 43.15-10 - Zerodur®: Failure probability for test surfaces processed with bonded grit of different sizes........ 398

Figure 43.15-11 - Zerodur®: Failure probability of Zerodur for test surfaces processed with loose grit of different sizes........ 398

Figure 43.15-12 - Zerodur®: Failure probability, F, as a function of test surface area, SL 399

Figure 43.15-13 - Zerodur®: Bending strength under constant load........ 399

Figure 43.15-14 - ULE®: Transmission curve 403

Figure 43.15-15 - ULE®: Thermal expansion characteristics........ 404

Figure 43.17-1 – Technical ceramics: CVD diamond – sand erosion 412

Figure 43.18-1 – NIRSpec optical bench development: Cesic ceramic demonstrator structure........ 416

Figure 43.18-2 – NIRSpec optical bench development: Test set-up and I-beam sample with joint line........ 417

Figure 43.18-3 – NIRSpec optical bench development: Test set-up for instrumented bench cells........ 417

Figure 43.18-4 – NIRSpec optical bench development: Test set-up for ‘shaker’ vibration tests........ 418

Figure 43.18-5 – NIRSpec optical bench development: Test set-up for cryogenic temperature test........ 418

Figure 43.18-6 – NIRSpec optical bench development: Thermal contraction under cryogenic test........ 420

Figure 43.18-7 – NIRSpec optical bench development: Linear displacement under cryogenic test........ 421

Figure 43.18-8 – NIRSpec optical bench development: Out-of-plane displacement under cryogenic test 421

Figure 43.19-1 – Zerodur®: Optical bench for LISA pathfinder mission........ 423

Figure 44.5-1 - Magnesium alloys: Momentum wheel bracket for satellite - Example 452

Figure 44.5-2 - Carbon fibre-reinforced magnesium alloy: Space mirrors (experimental) - Example........ 453

Figure 45.3-1 - Copper ODS alloys: Elevated temperature stress-rupture properties of Glidcop ODS alloys compared with some high conductivity copper alloys........458

Tables

Table 34.8-1 - Detection of manufacturing defects by contact NDT techniques 60
Table 34.8-2 - Detection of manufacturing defects by non-contact NDT techniques 61
Table 34.8-3 - Detection of in-service damage by contact NDT techniques 62
Table 34.8-4 - Detection of in-service damage by non-contact NDT techniques 63
Table 34.14-1 - Defect detection by shearography 87
Table 34.15-1 - Thermography: Summary of diameter/depth ratios from different studies 93
Table 34.15-2 - Thermography: Detection levels established for flat bottomed holes (FBH) from ESTEC study 93
Table 34.18-1 - Detection of damage by eddy current and ultrasonics 103
Table 34.21-1 - Summary of NDT techniques for polymer composite space structures 109
Table 34.23-1 - Interrelationship of items influencing the test 112
Table 34.23-2 - Effects of local failure modes on global behaviour of the structure.............113
Table 34.23-3 - Detection of techniques to various failures occurring during test...............114
Table 34.23-4 - Interrelation between failure modes and their possible locations 115
Table 34.23-5 - Interrelation between type of loading and possible failure modes 116
Table 34.23-6 - Failure modes which have to be covered by the specific tests 117
Table 34.23-7 - Appropriate instrumentation of particular areas of the structure 118
Table 34.23-8 - Interrelation between particular areas of the structures and possible dangerous loads 119
Table 34.23-9 - Testing of particular areas of the structure 120
Table 34.23-10 - Load combinations for the relevant cases 121
Table 34.23-11 - Testing different load cases 122
Table 36.3-1 - Resin procurement: General Information 143
Table 36.3-2 - Resin procurement: Base resin...144
Table 36.3-3 - Resin procurement: Hardener...144
Table 36.3-4 - Resin procurement: Catalyst...145
Table 36.3-5 - Resin procurement: Modifier 145
Table 36.3-6 - Resin procurement: Solvent..145
Table 36.3-7 - Resin procurement: Blend 146
Table 37.5-1 - Test data for HM carbon/epoxy specimen 156
Table 38.3-1 - Wet lay-up and laminating: Troubleshooting guide......................................165
Table 38.3-2 - Hand wet lay-up: Selection factors 166
Table 38.3-3 - Process steps for prepreg lay-up 168

Table 38.4-1 - Tooling for composites: Relative merits of various types for CFRP 177

Table 38.12-1 – EB curing: Advantages and drawbacks of epoxy and acrylate resins........ 195

Table 38.12-2 – EB curing: Mechanical properties of some acrylate and epoxy resins....... 196

Table 38.12-3 – EB curing: Typical mechanical properties of unidirectional IM carbon fibre/acrylate resin 196

Table 40.3-1 - Cost optimisation: Eurostar Bus/Inmarsat 2 study 241

Table 40.6-1 - Launcher fairing constructions: Mass and cost of various designs 247

Table 40.6-2 - Launcher fairings: Kepner-Tregoe trade study evaluation criteria 249

Table 40.7-1 - Selected thermoplastic component: Cost/producibility trade-off analysis 256

Table 40.7-2 - Selected thermoplastic components: Relative manufacturing costs and major contributors to cost 258

Table 42.6-1 - Reinforcing fibres available for use in inorganic composites 289

Table 42.6-2 - Continuous fibre and filament reinforcements used in MMCs and CMCs 291

Table 43.2-1 – Technical ceramics: Oxides - general features 308

Table 43.2-2 – Technical ceramics: Carbides - general features 309

Table 43.2-3 – Technical ceramics: Nitrides - general features 310

Table 43.2-4 – Technical ceramics: Carbons - general features 312

Table 43.2-5 – Technical ceramics: Sources - examples 314

Table 43.3-1 – Technical ceramics: ISO standards - summary 318

Table 43.3-2 – Technical ceramics: ISO standards - drafts 319

Table 43.4-1 – Technical ceramics: ‘green’ shape processes 321

Table 43.5-1 – Technical ceramics: Materials - mechanical components 328

Table 43.5-2 – Technical ceramics: Materials - thermomechanical components 329

Table 43.5-3 – Technical ceramics: Materials - electrical 330

Table 43.5-4 – Technical ceramics: Materials – optical and dimensionally-stable structures 331

Table 43.6-1 – Technical ceramics: alumina – typical properties 334

Table 43.6-2 – Technical ceramics: ZTA zirconia toughened alumina – typical properties 335

Table 43.7-1 – Technical ceramics: Aluminium nitride – typical properties 337

Table 43.8-1 – Technical ceramics: boron carbide – typical properties 339

Table 43.9-1 – Technical ceramics: boron nitride – typical properties 343

Table 43.10-1 – Technical ceramics: carbon types – typical properties 347

Table 43.10-2 – Technical ceramics: carbon-graphite grades – typical properties 348

Table 43.10-3 – Technical ceramics: carbon foams – typical properties 350

Table 43.11-1 – Technical ceramics: silica – typical properties 352

Table 43.12-1 – Technical ceramics: silicon carbides – comparison of materials properties produced by different process routes 355

Table 43.12-2 – Cesic®: Mechanical properties 360

Table 43.12-3 – Cesic®: Thermal properties 361

Table 43.12-4 – SiC 54S®: Mechanical properties 364

Table 43.12-5 – SiC 54S®: Thermal properties 365

Table 43.12-6 – SiC 100®: Manufacturing size constraints - practical size limitations for finished part 370

Table 43.12-7 – SiC 100®: Typical characteristics 371

Table 43.12-8 – SiC 100®: Micro-VCM test E544 - outgassing values 371

Table 43.13-1 – Technical ceramics: silicon nitrides – comparison of materials properties produced by different process routes 379

Table 43.14-1 – Technical ceramics: partially stabilised zirconia – typical properties 381

Table 43.15-1 – Technical ceramics: Zerodur® glass – typical properties 384

Table 43.15-2 – Technical ceramics: ULE® glass – typical properties 385

Table 43.15-3 - Zerodur®: Characteristic strength, Weibull factor and stress corrosion factor 397

Table 43.15-4 - Zerodur®: Fracture toughness of some glasses 400

Table 43.15-5 - ULE®: Summary of engineering data 402

Table 43.17-1 – Technical ceramics: CVD diamond – typical properties 409

Table 43.17-2 – Technical ceramics: CVD diamond – Weibull modulus 409

Table 43.17-3 – Technical ceramics: CVD diamond – Young's modulus 410

Table 43.17-4 – Technical ceramics: CVD diamond – typical optical properties for high-power laser systems 413

Table 43.17-5 – Technical ceramics: CVD diamond – typical dielectric properties for high-power microwave systems 414

Table 43.19-1 – Zerodur®: Overview of terrestrial astronomical telescope mirrors 423

Table 44.1-1 - Magnesium alloys: ASTM designation system - alloys and tempers 434

Table 44.1-2 - Magnesium alloys: Sources - examples 436

Table 44.2-1 - Magnesium alloys: Nominal chemical compositions 438

Table 44.2-2 - Magnesium alloys: Typical mechanical properties, at room temperature 440

Table 44.2-3 - Magnesium alloys: Typical low temperature tensile properties at -196°C 442

Table 44.2-4 - Magnesium alloys: Typical tensile properties at elevated temperatures 443

Table 44.2-5 - Magnesium alloys: Typical elevated temperature creep properties 444

Table 44.2-6 - Magnesium alloys: Typical fatigue and fracture toughness properties at room temperature 445

Table 44.2-7 - Magnesium alloys: Damping capacity of selected alloys and other metals 446

Table 44.3-1 - SiC particulate reinforced AZ61 magnesium alloy: Typical mechanical properties 447

Table 44.3-2 - SiC particulate reinforced magnesium alloy: Typical mechanical properties 448

Table 44.4-1 - Continuous fibre reinforced magnesium MMC materials: Examples 448

Table 44.4-2 - Magnesium continuous reinforced MMCs: Typical mechanical properties 450

Table 45.3-1 - Copper ODS alloys: Ambient temperature tensile properties 459

Table 45.3-2 - Copper ODS alloys: Short term properties at 538°C 460

Table 45.3-3 - Copper ODS alloys: Creep strength at 538°C 460

Table 45.5-1 - Continuous fibre reinforced copper: Thermal conductivity of carbon/Cu and carbon/Al composites 462

Introduction

The Structural materials handbook, ECSS-E-HB-32-20, is published in 8 Parts.

A glossary of terms, definitions and abbreviated terms for these handbooks is contained in Part 8.

The parts are as follows:

Part 1	Overview and material properties and applications	Clauses 1 - 9
Part 2	Design calculation methods and general design aspects	Clauses 10 - 22
Part 3	Load transfer and design of joints and design of structures	Clauses 23 - 32
Part 4	Integrity control, verification guidelines and manufacturing	Clauses 33 - 45
Part 5	New advanced materials, advanced metallic materials, general design aspects and load transfer and design of joints	Clauses 46 - 63
Part 6	Fracture and material modelling, case studies and design and integrity control and inspection	Clauses 64 - 81
Part 7	Thermal and environmental integrity, manufacturing aspects, in-orbit and health monitoring, soft materials, hybrid materials and nanotechnoligies	Clauses 82 - 107
Part 8	Glossary	

33
Aspects of damage tolerance

33.1 Introduction

33.1.1 Damage tolerance

Damage tolerance is the ability of a composite material structure to resist the onset of damage and perform to the stipulated design parameters, with damage present, throughout its remaining life time.

The guidelines presented are to be read in conjunction with normative ECSS standards ECSS-Q-ST-20; ECSS-Q-ST-40; ECSS-Q-ST-70; ECSS-E-ST-30-series and ECSS-E-ST-32-01.

33.1.2 Damage events

Composite structures are usually exposed to a variety of events during their life. This can include 'normal' in-service loading, in-service events that result in damage initiation and structural degradation, along with environment-related events.

Damage can also occur during manufacture or handling before the structure enters service.

The ability of a composite material to resist certain potential damage events is known as 'damage resistance'. It differs from durability because durability addresses the prevention of damage under normal operating conditions.

33.1.3 Damage tolerance criteria

Many agencies impose damage tolerance criteria. The details of such criteria are tailored to the structure of interest, although there are some common themes, [See: 33.3]:

- Defects are often standardised for analysis purposes, [See: 33.4], i.e.
  - defects that can arise during manufacture, [See: 19.2],
  - damage that can occur whilst in-service, [See: 19.3].
- The effect of macroscopic defects on static load; failure modes and the use of fracture mechanics, [See: 33.5].
- Testing and analysis: cyclic loading, no-growth criterion, residual strength and inspection, [See: 33.6].
- Environment-related aspects, e.g. flammability, lightning and other protection systems, acoustic fatigue, impact dynamics, [See: 33.7].

33.2 General guidelines

33.2.1 Fracture control

The damage tolerance evaluation of laminated composite structures is based on the fracture control requirements of normative standard ECSS-E-ST-32-01, [See also: 34.10 for analysis and test documentation]

33.2.2 Durability

Durability considerations are typically combined with damage tolerance to meet economic and functionality objectives. Specifically, durability is the ability of a structural application to retain adequate properties, e.g. strength, stiffness, and environmental resistance, throughout its life to the extent that any deterioration can be controlled and repaired, if there is a need, by economically acceptable maintenance practices, Ref. [33-2].

Durability largely addresses economic issues, while damage tolerance is focussed on safety. For example, durability often addresses the onset of damage from the operational environment. Under the principles of damage tolerance design, the small damages associated with initiation can be difficult to detect, but do not threaten structural integrity, Ref. [33-2].

33.2.3 Damage

The location of damage initiation sites are often difficult to identify and thorough inspections are needed using traditional techniques and NDT non-destructive testing techniques.

In many metals, failure involves the growth of a small crack perpendicular to the applied stress.

By contrast, in laminated composite materials stress relaxation and fracture results from combinations of:

- Splitting parallel to the fibres,
- Matrix microcracking ,
- Fibre failure,
- Delamination ,
- Pulling out of fibres,
- Pulling out of complete plies.

33.2.4 Defects in composites

33.2.4.1 General

Defects in composite elements can be described as either:

- Globaldeviations, or
- Local imperfections.

Figure 33.2.1 summarises the various types of defects that can occur in composite materials, Ref. [33-4].

33.2.4.2 Global

Global deviations are related to:

- A complete element, or
- A large area of an item.

The effect of a global deviation is to reduce the capability of an element. The level of reduced performance is dependent on the particular deviation, e.g. that occurring within a cure cycle. Adequate control can be achieved by, [See also: Chapter 19]:

- A process companion sample; also known as a ‘witness’, or
- Non-destructive checks of key points.

As the whole item is affected, control is related more to the quality, i.e. coverage of all potential sources of degradation, than to quantity.

Macroscopic ply defects

- Wrinkles, waviness, misalignment
- Prepreg variability exceeds pre-set values
- Local resin enrichment
- Fibre starved areas
- Porosity, voids
- Contamination
- Variable cure, oven temperature inhomogeneity
- Non-uniform agglomerations of hardener agents

Laminate defects

- Interlaminar defects by delamination:
  - at edges and corners (splintering)
  - inside (blister)
- Resin enrichment:
  - interlaminar
  - on outer surface
- Surface defects:
  - scratches with fibre breakage
  - dents without fibre breakage
  - fibre break away from impact
- Foreign particles
- Ply incorrect:
  - missed
  - wrong material
  - missed orientation
  - overlap
  - underlap gap
- Cracks at edges or corners

Component defects

- Holes:
  - oversized holes
  - breakout or broken fibres on hole exit
  - mislocated and repaired holes (resin)
  - out-of-round hole
  - resin-starved bearing surfaces
- Fasteners
  - tear-out or pull-through in countersink
  - over-torqued or undersized fasteners
  - improper seating of fasteners
  - tool impressions
- Thickness deviation on:
  - manufactured hardware
  - process control coupons
- Non-uniform bond thickness
- Warpage on:
  - detailed parts
  - assembled parts

Figure 33.2-1 – Summary: Defects in composite materials

33.2.4.3 Local

Local imperfections, identified by microscopic inspection of composite materials, identifies local irregularities, such as:

- Non-uniform distribution of resin and fibres.
- Interrupted or broken fibres.
- Disbonds at fibre-to-matrix interfaces.
- Voids.
- Inclusions.

As these local defects are statistically distributed, they are covered by the lower bound of material properties.

Significantly larger imperfections can cause progressive failure. Only local macroscopic deviations are considered here, which although macroscopic, the volume of a critical local defect can be rather small. Adequate control procedures prevent such potential defects occurring in the whole item.

The screening of the structure needs to be performed with sufficiently high-resolution, sensitivity and reliability. Therefore the control problems relate to both quality and quantity.

33.2.4.4 Proof test

The use of proof testing for flight articles, as a means of damage tolerance demonstration, can be considered on a case-by-case basis. In considering any such demonstration, it is necessary to review the test level and ensure that:

- The test does not introduce any unwarranted damage, and
- The worst limit load cases are covered by such a test.

33.2.5 Impact damage

33.2.5.1 Laminates

Impact damage is contained within the laminate itself.

33.2.5.2 Sandwich structures

In contrast to laminates, in sandwich constructions there are two load paths separated by a core that is responsible for shear load transfer.

Impact damage is typically unsymmetrical, so this needs a better understanding of the progression of the damage and the residual strength. Damage tolerance of sandwich structures is more complex than laminated structures as damage can include:

- Penetration or delamination of the facings; sometimes both.
- Core crushing
- Facing-to-core debonding

Cores tend to absorb and retain moisture which can reduce mechanical properties as well as increasing the structural weight.

Typically impact damage is not uniform through the thickness of the sandwich structure, since an impactor or projectile can penetrate or damage the outward facing skin whilst the inner skin remains undamaged.

Composite facings or skins on sandwich structures typically fail as a result of:

- Matrix cracks
- Fibre fracture
- Fibre buckling
- Delamination.

If only one facing is damaged significantly, this can cause a redistribution of stresses in the damaged facesheet.

Visual inspections for damage then become more difficult because the core can mask the damage or hinder the effectiveness of a non-destructive evaluation technique.

33.3 Damage tolerance criteria

33.3.1 General

33.3.1.1 Types of flaw

For composite structures the damage tolerance philosophy is primarily concerned with the types of flaw, i.e.:

- A large undetected manufacturing defect, which can be assumed to be an inclusion or other defect that causes a delamination.
- A surface scratch, where the length and depth of the scratch is defined and the orientation and location is assumed to be in the most critical location for a given structure.
- Undetected impact damage, where the damage area is assumed to be consistent with BVID barely visible impact damage and in a critical location, [See also: 19.5].

An undetected impact damage is the most serious of the types of flaw for many structures.

33.3.1.2 Effect of flaw

In composite structures, assumptions are made about the flaws, i.e.:

- The growth of a flaw is unstable.
- The initial flaw does significantly affect the static strength.
- The main aim is to reduce maintenance by the use of analysis and testing to demonstrate that the damage does not amount to a safety risk over the design life of the structure.

33.3.1.3 Threats

To construct a damage tolerance assessment for composite structures, the threats are defined for each structure, .i.e.:

- Damage from manufacturing processes.
- Accidental damage from assembly and handling.
- Accidental damage from in-service operation.

33.3.1.4 Modes of damage

The next step is to define the modes of damage for each threat, e.g. porosity, delamination, puncture, crushed core.

For each mode of damage, characteristics such as initial flaw size, threshold of detectability, critical size, and whether growth during loading are defined.

In many composite structures subjected to spectrum or block cycle loading, the growth of impact damage is characterised by periods of little or no growth at a low load level followed by relatively large growth at relatively high loads. A significant effect of impact damage is the significant reduction in static strength once the impact has occurred and often the most critical damage is induced by a low velocity impact event, [See also: 19.5].

The most critical mode of loading for metallic structure is tension, which causes crack opening. The critical modes of loading for composite structure is identified for purposes of selecting principal structural elements and for developing appropriate testing.

33.3.2 Load paths

In damage-tolerance assessments, a no-growth criterion is applied in demonstrating the integrity of individual load paths or the independence of load paths.

33.3.3 Environment

Structural details, elements, and subcomponents of critical structural areas, designated FCIs fracture critical items, are tested under environmental loads representative of operational usage. This testing can form the basis for validating a no-growth approach to the damage-tolerance requirements. The testing assesses the effect of the environment on the flaw growth characteristics and the no-growth validation.

33.3.4 Damage

Damage levels, including low-level impact damage, typical of those that can occur during fabrication, assembly and operation are introduced. The extent of such initially detectable damage is established and is consistent with the inspection techniques employed during manufacture and in operation.

Flaw or damage growth data is obtained from operation-related load cycling of such intrinsic flaws or delaminations or mechanically introduced damage.

The number of cycles needed to validate a no-growth concept is statistically significant and can be determined by such load or life considerations as reflect the operational usage. The growth or no-growth evaluation is established by tests supported by analysis or by tests at the coupon, element or subcomponent level.

A no-growth evaluation is applied, unless there is sufficient evidence for growth evaluation. This is related to the operational use inspection programme being acceptable for the particular FCI.

33.3.5 Residual strength

The extent of damage for the residual-strength assessments is established. Residual-strength evaluation by component or sub-component testing or by analysis supported by test evidence is performed considering the particular damage.

33.3.6 Safe-life

In the case of a safe-life demonstration, the evaluation demonstrates compliance with the fracture control requirements of ECSS-E-ST-32-01.

33.3.7 Fail-safe

In the case of fail-safe structures, the residual strength is sufficient to permit the safe operation at limit loads throughout the time interval between schedule inspections.

33.3.8 Stiffness

It is also demonstrated that the stiffness properties have not changed beyond acceptable levels in relation to aero-elastic and any other stiffness requirements as a result of the damage identified for the residual-strength assessments.

33.3.9 Loads

The structure is able to withstand static loads, considered as limit loads, that are expected during the completion of the service life on which damage resulting from obvious discrete sources occur, e.g. dropped tools, meteoroid impact. The extent of damage is based on each discrete source. This damage definition and any verification programme is the subject of review.

33.3.10 Environmental factors

The effects of temperature, humidity and other environmental factors which can result in material property degradation are addressed in the damage-tolerance evaluation.

33.3.11 Inspection

An inspection programme is developed consisting of frequency, extent and methods of inspection for inclusion in the maintenance plan.

The inspection intervals are established such that the extent of the damage is carefully checked with respect to the residual-strength capability including the no-growth design concept. Any areas deemed non-inspectable are subject of a special design and test programme.

33.3.12 Summary

The main stages of a damage tolerance evaluation are, Ref. [33-1]:

- Select the principal structural elements to be evaluated.
- Define or develop for each location:
  - stress spectrum.
  - environment.
  - crack growth rate data.
  - fracture toughness data.
  - structural category.
  - critical damage size under limit load and in-service load.
  - crack growth curves from spectrum loading.
- Establish initial damage size.
- Establish damage threshold of detectability for each location.
- Validate residual strength and crack growth analysis methods with testing.

33.4 Defect standardisation

33.4.1 General

33.4.1.1 Metal structures

The assessment of defects in homogeneous and isotropic metallic structures by fracture mechanics still involves simplifications. The real volumetric defect, e.g. a pore, is conservatively represented by a mathematical cut of the size of its projection; as shown in Figure 33.4.1. The sharp edges of the assumed crack are more severe than those of real defects.

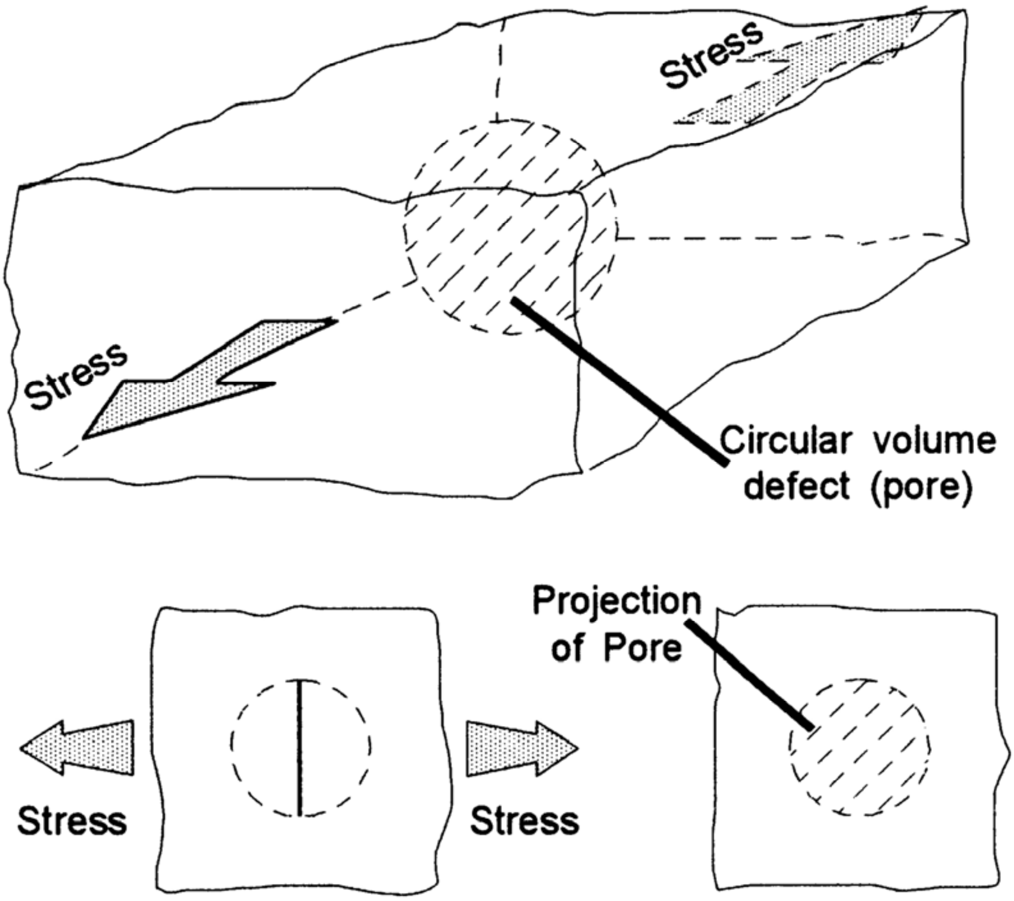


Figure 33.4-1 - Metal structures: Idealisation of a volumetric defect

33.4.1.2 Composite structures

The assessment of the effect of defects needs a simplified representation of the real defect in order to reduce the complexity added by the material heterogeneity and anisotropy.

Like fracture mechanics, a few standard types of defects cover most local deviations. This applies to:

- Assessment and verification, and
- Degree of resolution in non-destructive inspection

33.4.2 Types of defects

For the simplification of real defects, [See: Chapter 19], the factors to consider include:

- Local defects: For the thin laminates often used for light-weight space structures, most local defects can be represented by a through-the-thickness defect. It is difficult to justify that a surface defect, e.g. a severe scratch in a laminate having a few plies, relates to a part of the plies only, so this approach is not considered to be too conservative.
- Delaminations: The separation of plies can be a source of primary failure in a composite structure under compressive loads. Very local delaminations in the vicinity of notches are known to reduce the effect of the stress concentrations.

- Circular defects: Experience shows that circular defects, through a laminate, usually have a slightly higher influence on residual strength than slots having a length equivalent to the diameter.

Standardised defects that enable an assessment of most types of manufacturing- and operationally-induced defects are:

- Circular holes, and
- Circular delaminations.

Figure 33.4.2 shows the idealisation of real defects in composite structures using a standard circular defect.

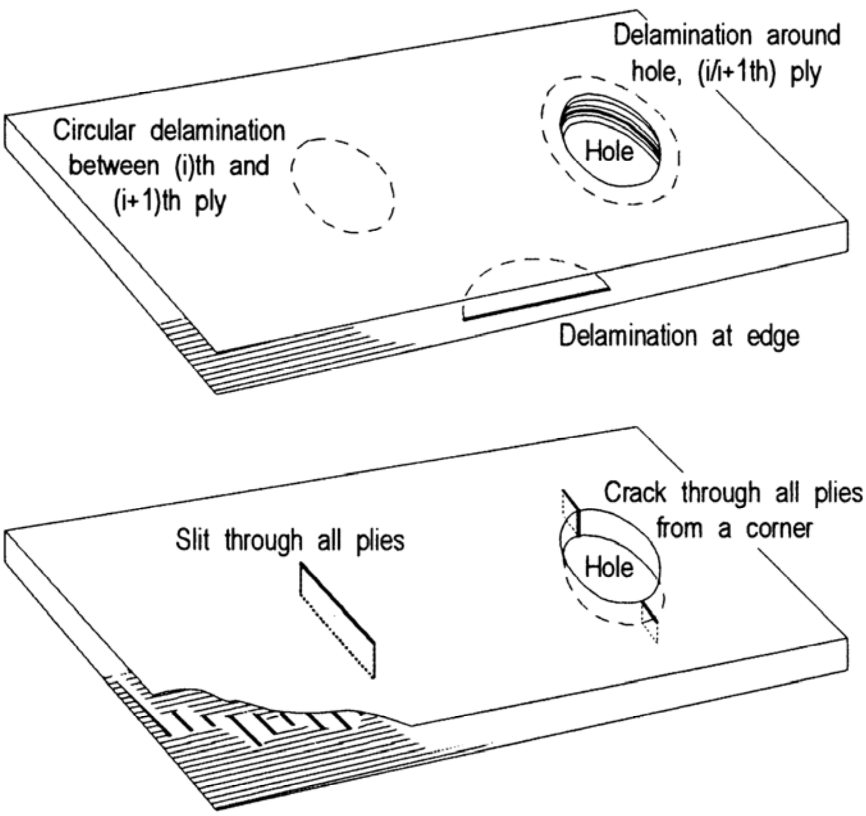


Figure 33.4-2 - Composite structures: Idealisation of defects by standard circular defect

33.5 Effect of macroscopic defects on static load

33.5.1 Composite failure

In composite materials, mainly CFRP, fracture behaviour is primarily elastic, i.e. there is no plastic deformation compared with that of ductile metals.

Materials with such brittle failures are sensitive to notches and cracks under static and cycling loading, especially with respect to residual strength under quasi-static loads.

Careful observation of the failure process during testing of notched specimens shows that under steadily increasing loads, the failure initiates within individual plies, either along or transverse to fibres, but also between certain plies long before the maximum load capability is reached. The partial damage within or between certain plies produces a redistribution of the local stresses that in turn results in a complicated, interactive failure process.

Any attempt to relate the final failure to the distribution of initial stresses is questionable.

[See also: Chapter 14]

33.5.2 Use of fracture mechanics

33.5.2.1 Isotropic materials

The wide application of LEFM linear elastic fracture mechanics to homogeneous isotropic material is supported by its relative simplicity. One parameter, the toughness, can describe the failure load of a pre-cracked element.

33.5.2.2 Anisotropic plates

The conditions under which the techniques of isotropic fracture mechanics can be directly applied to anisotropic plates are described.

33.5.3 Multi-angle laminates

The conditions cannot be maintained correctly for multi-angle laminates. The inhomogeneity is created by the:

- Fibre/matrix (micro-heterogeneity), and
- Stacking (macro-heterogeneity).

With the intent to generate a failure criterion phenomenologically, one can consider the laminate to be sufficiently homogeneous for the stresses resulting from external loading to be related to the total thickness of the laminate (gross stresses).

Implicitly, with the assumption of homogeneity, the considerations are limited to defects passing through the thickness, i.e. holes.

Furthermore, as the toughness of unidirectional laminates is different under longitudinal and transverse loading, the toughness of an arbitrary multi-angle laminate cannot be expected to be a material constant.

In phenomenological fracture models, the initiation and propagation of damage, occurring within or between the different plies, long before the critical loading is reached are recognised by assuming an effective size of defect which is larger than the geometric dimensions of the notch.

33.6 Test and analysis

33.6.1 General

33.6.1.1 Scope

The nature and extent of analysis or tests on complete structures, or portions of the primary structure, depend upon applicable previous damage-tolerant designs, construction, tests and operational experience on similar structures. In the absence of experience with similar designs, approved structural development tests of components, subcomponents and elements are performed.

33.6.1.2 Location

Since there are a large number of locations on each composite structure to be evaluated, it is impractical to evaluate the damage tolerance at each location by testing. Analysis is therefore needed. The analysis procedures associated with damage tolerance evaluation are based on fracture mechanics, which include stress intensity, interlaminar fracture toughness and propagation growth rates of cracks or delamination.

33.6.1.3 Strength

Analytical calculations are used for residual strength calculations and fatigue analyses under spectrum loading, which are two major ingredients in damage tolerance evaluation. Sufficient testing is needed to validate the analysis methods. Full-scale structural testing occurs too late to be useful during the design phase, but it is used later to enable initial damage tolerance evaluations to be updated.

33.6.1.4 Environment

The effects of environment can be accounted for in both the analysis stage of the assessment and during the materials characterisation stage.

The effects of moisture diffusion and temperature can be included as an additional loading condition in the analyses. Additionally, the effects of moisture and temperature on the delamination characterisation properties can be determined by conducting tests under hostile conditions or after ageing (long-term or accelerated) of the materials consistent with their application.

33.6.1.5 Tests

As part of the damage tolerance approach, tests are clearly defined and performed, Ref. [33-2]:

- static testing of structural elements with various modes of damage and various modes of loading - to provide residual strength information
- cyclic testing of structural elements with various modes of damage and various modes of loading - to provide damage growth (or no growth) information

- Cyclic testing of important full-scale components - to verify that damage would not grow to a critical size in the full-scale article during the life of the structure
- Static testing of important full-scale components - to verify that they would carry required loads with any damage growth inflicted from cyclic loading
- Appropriate testing to validate any analyses methods used

33.6.1.6 Test programme

The approach taken for compiling a test programme from materials selection to structural validation is known as the 'building block approach'; as shown in Figure 33.06.1, [See also: 33.2].

The advantage of the approach is that it enables:

- Identification by experimentation of the multiplicity of failure modes that composite structures can experience.
- Identification of 'knock-down' or 'enhancement' factors for environmental conditions, when testing of full-scale structures in a hostile environment is not feasible.

The application of realistic service loads and environments to the test coupon or sub-structure are emphasised within this approach.

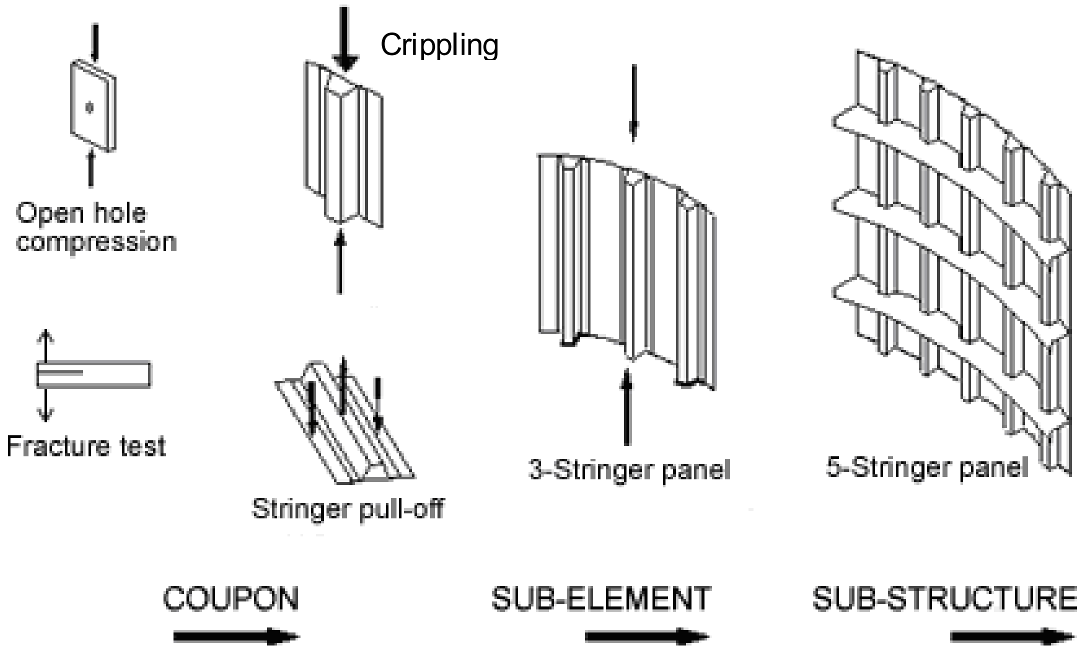


Figure 33.6-1 - Building block approach: Test programme development

33.6.1.7 Composites

Five levels of tests are necessary for composites; as shown in Figure 33.6.2:

- Constituent,
- Lamina,
- Laminate,
- Structural element,
- Structural sub-component.

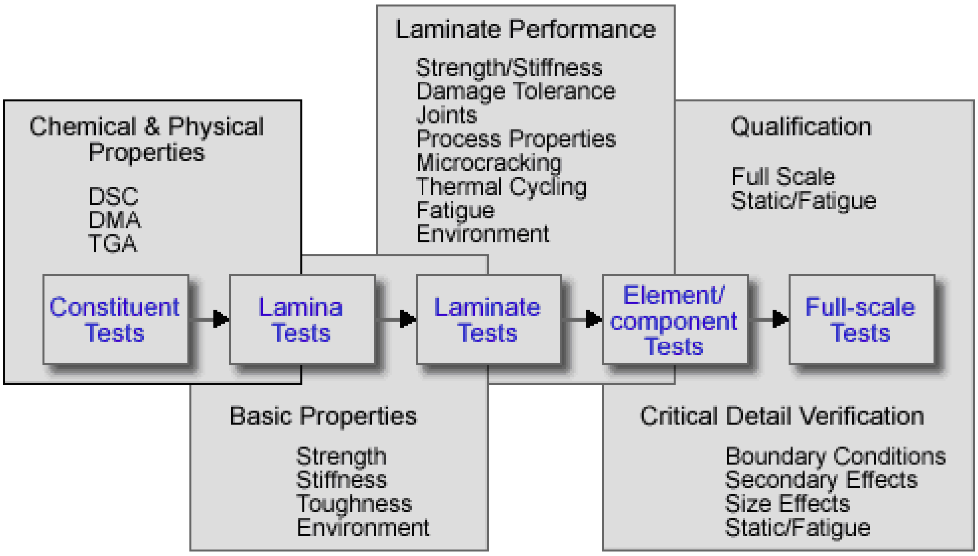


Figure 33.6-2 - Building block approach: Composite materials

33.6.2 Constituent tests

The individual properties of fibres, fibre forms, matrix materials, and fibre-matrix pre-forms are evaluated. The key properties include:

- Matrix density,
- Fibre density,
- Fibre tensile strength,
- Fibre tensile modulus.

33.6.3 Lamina tests

The properties of the fibre and matrix together in the composite material form are evaluated. The key properties include:

- Fibre areal weight,
- Matrix content,
- Void content,
- Cured ply thickness,
- Lamina tensile strengths and moduli,
- Lamina compressive strengths and moduli,
- Lamina shear strengths and moduli.

33.6.4 Laminate tests

Laminate testing characterises the response of the composite material in a particular laminate design, such as quasi-isotropic. The key properties include:

- Tensile strengths and moduli,
- Compressive strengths and moduli,
- Shear strengths and moduli,
- Interlaminar fracture toughness,
- Fatigue resistance.

33.6.5 Structural element and component tests

The ability of the material to tolerate common laminate discontinuities is evaluated. The key properties include:

- Open and filled hole tensile strengths,
- Open and filled hole compressive strengths,
- Compression after impact strength,
- Joint bearing and bearing bypass strengths.

33.6.6 Structural sub-component or full-scale tests

The behaviour and failure mode of increasingly more complex structural assemblies are evaluated. The selection of which is structure and application dependent.

33.6.7 Delamination

33.6.7.1 In-service

The threat of delamination arising from in-service loading has been one of the factors in limiting the adoption of laminated composite materials in greater volume for primary structure.

While other damage modes such as matrix cracks can occur first, delaminations result in larger stiffness drops and reduction in load-bearing capabilities.

Delaminations can occur from interlaminar stresses arising from geometric or material discontinuities from design features, e.g. an edge, a hole, a dropped ply. However, they can also occur from matrix cracks or from interlaminar stresses caused by structural loading, such as in a curved laminate, or by foreign body impacts.

A delamination, once initiated, grows under fatigue loads. During delamination growth, the structural loads can be redistributed such that another delamination occurs in another location.

The delaminations can continue to grow and accumulate until a structural failure occurs, such as buckling or fibre failure.

Alternatively, the delamination can be arrested and the structure maintains some level of integrity.

Although delamination does not always cause a total loss of the load-bearing properties of the component, it is usually a precursor to such an event. Therefore, knowledge of the resistance of a composite to interlaminar fracture is useful not only for product development and material screening, but a generic measurement of the interlaminar fracture toughness of the composite is useful for establishing design allowables for damage tolerance analyses of composite structures.

33.6.7.2 Role of interlaminar fracture tests

Interlaminar fracture tests are performed by manufacturing test samples with precracks. The type of test is defined based on the mode of loading of the test sample, i.e.

- Mode I – tests such as DCB
- Mode II – tests such as 4ENF, ELS, ENF
- Mode III ECT, SCB (modified)
- Mixed Mode I/II – tests such as MMB, FPS/ADCB, CLS, stabilised MMB

33.6.7.3 Dynamic loading

In most cases the components experience spectrum fatigue loads rather than constant amplitude fatigue loads. For Mode I testing, the load needs to remain in tension. Therefore, the parts of the spectrum where the load causes the delamination to close are represented by zero load in the DCB tests.

With appropriate fixture design, the mode II test specimen can be loaded through zero and thus fully represent a delamination in a structure where the spectrum contains tension and compressive loads.

When the delamination has compressive loads closing the delamination (equivalent to a negative G_I), the influence of friction and subsequent heating can be an issue.

For a full characterisation for spectrum loads, the delamination onset curves and the delamination growth curves are generated for different R-ratios and blocks of loading to establish the synergistic effects.

33.7 Other features of damage-tolerance design

33.7.1 Acoustic fatigue

The resistance of the composite structure to noise damage is demonstrated by analysis or test. In particular, it is demonstrated that no-growth damage areas, as established in relation to quasi-static loading criteria, do not propagate.

33.7.2 Proof testing

Within the fracture control requirements, all composite structures and components are classified as potentially fracture critical items, PFCI, [See: ECSS-E-ST-32-01].

33.7.3 Metallic parts

Composite components can contain metallic parts, e.g. attachment fittings or larger substructures in areas of high load-transfer.

Special attention is paid to fracture critical items, FCIs. to investigate the treatment of areas of high load-transfer in order to ensure there is no unacceptable local yielding when design ultimate loads are sustained, [See: ECSS-E-ST-32-01].

33.7.4 Thermal and meteoroid protection system

The interface with the composite structure and any thermal protection system is taken into account in the selection of FCIs, and related inspection and maintenance.

Any interactive effects in relation to local loadings, such as the response to impact loads and acoustic excitation, are also considered.

33.7.5 Impact dynamics – crashworthiness

Crashworthiness is demonstrated by test or by analysis supported by tests that, under realistic and survivable conditions, the survival characteristics are commensurate with those achievable with a conventional metallic aircraft structure.

33.7.6 Flammability, toxicity and off-gassing

The properties of materials and processes used in space systems are established according to normative ECSS standards.

[See: ECSS-Q-ST-70; ECSS-Q-70-71]

[See also: ESMAT website for outgassing and flammability data from the ESTEC Materials and Processes databases]

33.7.7 Lightning protection

It is demonstrated by appropriate analysis and test that the structure can dissipate static electrical charges, provide necessary electromagnetic protection and provide an acceptable means of diverting electrical current arising from the lightning strike so that the vehicle is not endangered.

A test and inspection procedure is established to ensure satisfactory maintenance of the lightning protection system.

33.8 Improving damage tolerance with higher toughness laminates

33.8.1 General

Changes made to various composite material parameters can cause the notch sensitivity to range from insensitive to very sensitive. Some guidelines for creating tough laminates are presented, Ref. [33-2].

33.8.2 Material-related aspects

33.8.2.1 Fibre-to-matrix bond

To obtain a tough laminate, the strength of the interfacial bond between the fibre and matrix has to be optimised. This is achieved by correct surface treatment of the fibre. If the bond is too strong, the material always fails in a brittle manner irrespective of lay-up.

33.8.2.2 Types of fibres

Materials which can store large amounts of strain energy are expected to be tough. Consequently fibres with high strength, intermediate modulus and high failure strain are expected to be good candidates for a tough laminate. Hybridisation provides much scope for developing composites with increased toughness.

33.8.2.3 Plies

Although the interlaminar fracture toughness of a laminate does not appear to vary with specimen thickness, it has been shown to be sensitive to the thickness of individual plies.

In general, thick plies delaminate more easily and therefore decrease the interactions between adjacent cracked and uncracked plies, so increasing the toughness. However, it is recognised that a compromise can be made for other mechanical properties, such as fatigue and environmental response. It is generally widely recognised that thicker plies increase the susceptibility to transverse cracking by an increase in volume of material under stress.

33.8.3 Lay-up aspects

33.8.3.1 Stacking sequence

While the effects of stacking sequence on the notched strength are unclear, it has been shown that arranging plies to encourage delaminations between shear cracks in 45° plies and adjacent load bearing 0° fibres, e.g. [±45°, 0°, 0°]s, results in a higher notched strength.

If, however, the delamination is suppressed, e.g. [0°, ±45°, 0°]s, then the shear cracks that develop in the 45° plies at relatively low stresses have a detrimental effect on the 0° fibres. This results in a relatively brittle behaviour with the 0° fibres failing in a step-wise manner along the 45° line.

The [±45°]s lay-ups, although they are relatively weak, are notch insensitive. Large damage zones develop across the entire width of a test specimen resulting in a controlled, non-catastrophic failure.

Owing to their relative weakness in the longitudinal directional, [±45°]s lay-ups are unlikely to be used to resist tensile loads alone. However, they can be used as 'crack-arrest' strips in a laminate containing fibres in the load-bearing direction.

33.8.3.2 Reinforcement style

The style of reinforcement has an important effect on the notched strength. In cases where the formation of a damage zone is largely suppressed, a relatively brittle behaviour occurs. Changes in ply orientation are also known to affect the notched strength.

33.9 References

33.9.1 General

[33-1] J. Tomblin et al
'Review of damage tolerance for composite sandwich airframe structures'
August 1999

[33-2] MIL-HDBK-17: Polymer matrix composites

[33-3] ESA PSS-03-207 - Guidelines for carbon and other advanced material prepreg procurement specifications; not transferred to the ECSS document system

[33-4] MBB/ERNO
'Integrity control of carbon fibre reinforced plastics (CFRP) structural elements - Executive summary'
ESA Contract 4442/80/NL/AK (SC)

33.9.2 ECSS documents

[See: ECSS website]

ECSS-E-ST-30-series:	Mechanical
ECSS-E-ST-32-08	Materials
ECSS-E-ST-32-01	Fracture control; previously ESA PSS-01-401.
ECSS-Q-ST-20	Quality assurance
ECSS-Q-ST-40	Safety
ECSS-Q-ST-70	Materials, mechanical parts and processes; previously ESA PSS-01-70
ECSS-Q-70-71	Data for the selection of space materials and processes; previously ESA PSS-01-701

33.9.3 MIL standards

MIL-HDBK-17	Polymer matrix composites

34 Inspection and quality assurance

34.1 Introduction

An inspection programme is undertaken on components and assembled structures, carried out in conjunction with defined quality assurance procedures. Detailed and explanatory documents are kept on all aspects of integrity control.

Inspection using more than one non-destructive testing technique can be necessary. In addition, NDT can be carried out in conjunction with the mechanical testing or proof loading of components and assembled structures. For reusable and long-life structures, in-service inspection is also defined.

Points to be considered with respect to the philosophy of inspection and the application of techniques are:

- During the design of structures, consideration is given as to how the assemblies are inspected in order to detect and characterise any significant defects or anomalies which can occur.
- It is preferable to enhance production quality procedures to minimise the occurrence of manufacturing defects, rather than rely later on inspection to cover all eventualities.

Provided that sufficient forethought is given to damage tolerant design, an example of an approach applied is ultrasonic testing at the pre-assembly component level, which is then followed by visual in-service inspection; as part of post-qualification or acceptance testing.

34.2 Fabrication and quality assurance

34.2.1 Quality assurance

ECSS-Q-ST-20 and the associated standards give details of the quality assurance system. These requirements aim to guarantee the quality and reproducibility of the basic composite materials.

34.2.1.1 Material procurement specification

In establishing a procurement specification for carbon and other advanced fibre prepreg, guidelines are provided in Ref. [34-39].

As a minimum, a procurement specification contains:

- Basic fibre properties, including type, number and frequency of test.
- Basic matrix properties and chemical characterisation, including type, number and frequency of test.

- Basic composite properties, including type, number and frequency of test.
- Condition of fabrication, including the process method, lay-up, tooling, workshop environment.
- Significant parameters of the cure cycle, e.g. time, temperature, pressure.
- Cured component properties, including type, number and frequency of test.
- Inspection criteria, at every stage.
- Storage and handling conditions, throughout the processes.

The strict application and control of such specifications guarantee the quality and reproducibility of the product. No modification of the specification is made before all of the subsequent effects on the quality of the structure have been verified.

34.2.1.2 Composites quality control plan

A specific quality control plan for the composite that ensures the necessary liaison between design, engineering, manufacturing and quality disciplines is established. This plan defines the production route and the importance of any defects likely to occur during the fabrication process. It is adaptable to any special engineering requirements that arise with individual composites parts or areas as a result of:

- Potential failure modes,
- Damage tolerance and flaw growth requirements,
- Loadings,
- Inspectability, and
- Local sensitivities to manufacture and assembly.

34.3 Inspection, maintenance and repair

34.3.1 Inspection and maintenance

34.3.1.1 General

A non-destructive inspection (NDI) programme is developed that defines the frequency, extent and methods of inspection for incorporation into requisite design development and maintenance plans. The programme includes:

- Material procurement,
- Manufacture, [See also: Chapter 8].
- Assembly,
- Operation,
- Test programmes.

[See also: ECSS-E-ST-30 series; ECSS-E-ST-32-01 for fracture control]

34.3.1.2 Programme approval

The contents of the programme are subject to approval, which covers:

- The nature, size and distribution of defects covered by particular inspection techniques, [See: 33.2 for typical defects].
- The ability to apply intended methods for every fracture critical item, FCI, configuration.
- Initial inspection of all finished items by the NDI method relevant to the assumed initial defect or damage. NDI is performed for the total item, even though only one location is analysed.
- Inspection can be necessary for limited-life items.
- Verification of the structural redundancy for fail-safe items before each flight.
- Post test NDI for all proof-tested items.

34.3.2 Repairs

Repair procedures also need analysis and test to demonstrate that they are able to restore the structure to a flightworthy condition, [See also: Chapter 41].

Repair procedures are also accompanied by necessary inspection for subsequent operational use.

34.4 Inspection

34.4.1 Basic considerations

34.4.1.1 Manufacturing

To limit the number of scrap items, some inspection and testing can be performed at an early stage of manufacture. However, some inspection or testing can be performed only on completely assembled elements, e.g. proof testing.

34.4.1.2 Damage

The possibility of human introduced damage during later activities, e.g. integration and acceptance testing, also needs to be recognised. The fact that damage can occur during check-out and operation is a good reason for not reducing, below a certain level, the size of defects that are detected.

34.4.1.3 Inspection techniques

A combination of different methods is likely to be needed in order to detect all of the defects that are identified as intolerable, e.g.:

- Ultrasonic C-scans to detect delaminations, and
- X ray to detect gaps, foreign particles and serious levels of fibre breakage.

34.4.1.4 Damage tolerance

In view of the difficulty of detecting defects, it is often more economic to design a CFRP item to a relatively higher damage tolerance than stated initially. This does not necessarily result in higher masses if the notch sensitivity of the material and laminate configuration is evaluated, selected and controlled accordingly.

34.4.1.5 Design strategy

The design strategy balances the differing requirements arising from the inevitable conflicts between, e.g.:

- Structural performance,
- Low masses,
- Sensitivity of elements,
- Economic manufacturing,
- Low scrap, and
- Repair rates.

Non-destructive inspection (NDI) is, in this respect, a synonym for a set of control activities tailored to achieve the necessary quality.

The key elements and the level of control are determined by the designer.

34.5 Definition of inspection procedures

34.5.1 Procedures

34.5.1.1 General

The inspection procedures are generated and their sensitivity verified, Ref. [34-1].

General inspection procedures are reviewed with respect to:

- Their applicability,
- The known constraints,
- The provisions necessary,
- Whether or not they need to be adapted to a particular element,

34.5.1.2 Sensitivity

The expected or required sensitivity is established considering:

- Limited access, e.g. to cavities inside structures.
- Disturbances of transducers, e.g. at free edges.

34.5.1.3 Calibration

All NDI techniques are calibrated to defined standards at regular intervals, during the:

- Verification programme, and
- Use of equipment, e.g. test coupons with known positions and sizes of representative defect-types for the items under test.

34.5.2 Thin composite laminates

A basic procedure is proposed, Ref. [34-1], to simplify the situation for the thin laminates typically used in spacecraft, e.g. less than 2 mm thick, such as shells or facings of sandwich panels. This comprises of:

a. Define adequate manufacturing process controls, including:

    1. material sensitivity to notches,

    2. accurate laminate configuration,

    3. adequate curing processing.

Preferably this is accomplished by non-destructive methods or by those techniques that record results automatically.

b. Design the structure with an allowable stress or strain reflecting a circular hole through the thickness of, say, 6 mm diameter and a circular delamination of, say, 12 mm diameter.

c. Select NDI methods, procedures and criteria capable of reliably detecting defects of the selected sizes, even if surface defects consist of scratches through one ply only with a length of the assumed hole.

d. Consider the needs for the inspection of joints separately, e.g. holes and adhesive bonds.

e. Accept all hardware inspected and found without indications under the defined condition.

f. Where deviations are present, assess all occurrences which exceed the set criteria case-by-case by applying established nonconformance procedures.

34.5.3 Thick composite laminates

Thicker laminates, e.g. greater than 2 mm thick, need special consideration, as they usually do not comply with the assumption of a through-the-thickness hole enveloping the actual defect.

Thick composites imply the presence of high loads and multiple lamina. Inspection techniques capable of detecting defects at all depths within the composite are used.

34.6 Non-destructive inspection techniques

34.6.1 Introduction

Non-destructive testing, NDT, has an important role in the application of composites and advanced materials to space structures, Ref. [34-2], [34-3]. Initially, the development of NDT techniques was driven by the need to detect defects and anomalies in new materials, such as CFRP. This was influenced by uncertainty as to the significance of defects in structures and a wish to detect all identifiable features. With a better understanding of material capabilities, further development of NDT techniques is aimed at those defects known to be significant in structural integrity.

NDT technologies are closely associated with the demands of aircraft inspection, Ref. [34-4]. The space industry benefits from this by modifying techniques to suit the needs of space programmes, be it for launchers, satellites, space stations or spaceplanes, Ref. [34-5].

There is one rule which applies to all aerospace projects:

All structures should be designed and manufactured in such a way that they can be inspected, both during production and once assembled.

34.6.2 Damage tolerant designs

In a damage-tolerant approach, flaws are assumed to exist in fracture-critical components. As part of this approach, factors to be evaluated include:

- The critical flaw size that results in failure of a component when subjected to known service stress and temperature conditions.
- The growth rate of subcritical cracks and consequently the time that a component containing a subcritical flaw can operate safely in service.
- The inspection performed to detect defects before catastrophic failure of the component occurs.

The assumed initial flaw size is based on the intrinsic material flaw size distribution and the manufacturing inspection capability. For manufacturing, an inspection reliability of 90% probability of detection (POD) at the lower-bound 95% confidence level is generally accepted for the assumed flaw sizes. These flaw sizes are intended to represent the maximum size of damage that can be present in a critical location after manufacture and inspection, Ref. [34-2].

34.6.3 Advances in NDT

It is widely accepted that more than one NDT technique is needed to detect all possible defects, anomalies and damage present within an assembled structure.

To control inspection costs, it is prudent to use the smallest number of NDT techniques necessary to detect critical defects. Greater emphasis is placed on using rapid, automated inspection systems. The more traditional ultrasonic, radiographic and holographic techniques are now being complemented by newer technologies, such as laser-based ultrasonics or shearography and computer tomography.

Development in NDT is driven by:

- Availability of cheap, powerful computers,
- Digital signal processing,
- Image enhancement techniques,
- Real-time image processing and presentation,
- Need for large area scanning systems, e.g. for ageing aircraft,
- Rapid inspection of aircraft to save costs.

The advantages gained from the new developments include:

- System automation and image processing.
- Non-contact and non-invasive inspection, [See also: 34.10].
- Inspection of thick-section composites.
- High-resolution techniques for polymer, metal and ceramic matrix composites.
- Increased availability of powerful, portable inspection systems.

Automated systems can reduce inspection costs by reducing inspection time and operator intervention. This applies when the test requirements and sequence has been established and verified for each item or structure, Ref. [34-91].

34.6.4 Techniques

34.6.4.1 General

There is now a wide range of inspection systems based on a variety of principles; each performs a different function. In broad terms, the techniques can be grouped as being applicable to (some NDT methods can be applied in both groups):

- laboratory (L) and production (P) environments for the examination of components and assembled structures.
- in-service (S) environments for the examination of fully assembled structures.

34.6.4.2 Laboratory and production based NDT

For component and assembled structures, the techniques available include:

- C-scan ultrasonics, both conventional and air-coupled, [See: 34.10].
- Holography, [See: 34.13].
- Radiography, [See: 34.16].
- Computer tomography, [See: 34.17].

- Laser shearography, [See: 34.14].
- Thermography, [See: 34.15].

34.6.4.3 In-service based NDT

The techniques for fully assembled structures include:

- Portable conventional and air-coupled ultrasonics, [See: 34.10].
- Eddy currents, [See: 34.18].
- Laser shearography, [See: 34.14].
- Thermography, [See: 34.15].

34.6.4.4 Other techniques

Techniques that provide a supporting role include:

- Visual inspection, [See: 34.9].
- Coin tapping, [See: 34.9].
- Dye penetrants, [See: 34.9].
- Resonance bond-testers, [See: 34.9].
- Acoustic flaw detectors, [See also: 34.20]
- Acoustic emission, [See: 34.11].

34.6.5 Smart technologies for condition monitoring

Easy access to computing power initiated the development of inspection systems that are integrated into structures to provide continual monitoring of the thermal and strain responses. Such systems are equally applicable to in-service damage detection.

[See also: Chapter 92 for potential space applications of smart technologies]

34.7 Defects and anomalies for detection

34.7.1 General

NDI can be applied at any time to assess the condition of a component, assembly or structure, Ref. [34-6], [34-7]. Factors to consider include:

- New composite mouldings are assessed against quality standards that aim to establish acceptance levels for anomalies, such as voidage, resin content, fibre misalignment and rucking, presence of foreign bodies, microcracking and missing plies.
- Subsequent fabrication stages can introduce the possibility of machining damage, delaminations, poor hole quality, adhesive bond integrity, missing components, poor honeycomb splicing, component misalignment and continuity of potting compounds.

- Sandwich panel constructions and co-curing give rise to greater complexity from a single moulding cycle with the possibility for a wide range of defects to occur, including core-to-skin disbonds.
- In-service inspection is mainly related to establishing the presence of significant defects, such as impact damage, delaminations, fibre breakage, disbonds, moisture uptake, water retention in honeycombs.

In-service inspection is only appropriate to some space structures. Developments in NDT are mainly associated with inspecting aircraft.

34.7.2 Emphasis of NDT development

As composites have become more widely applied to aerospace structures, the emphasis placed on NDT techniques has changed. The extensive use of sandwich honeycomb constructions, co-curing and adhesive bonding enables more complex constructions to be manufactured. With these the factors of greatest concern include:

- Missing or misplaced materials and subcomponents,
- Disbonds and delaminations,
- Integrity of fixing points around fasteners and inserts,
- Damage from low energy impacts, between 0.5 J and 10.0 J typically, which can cause ‘barely visible impact damage’ (BVID), Ref. [34-8].

Where feasible, a preference is given to the use of non-contact NDT techniques instead of the more traditional methods, Ref. [34-9].

34.8 Overview of NDT techniques

34.8.1 Selection of NDT technique

Numerous techniques are used for the inspection of materials, components and structures, Ref. [34-3], [34-4], [34-6], [34-7], [34-9].

Factors involved in the selection of an appropriate NDT technique include:

- Application: Laboratory, production or in-service.
- Materials to be inspected.
- Component or structure dimensions and geometry.
- Accessibility to area to be inspected.
- Ability to detect a certain type of defect.
- Sensitivity to defect size.
- Availability of reliable equipment and resources.
- Recording and understanding of results.

- Equipment cost.
- Inspection speed, hence cost.

Owing to a lack of standardisation of NDT techniques, it is customary for composite manufacturers to select a technique and prove its acceptability for each component or structure produced; provided that the design has stipulated the inspection requirements.

NDI methods which have been applied to aerospace materials and structures are described. The basic techniques, e.g. ultrasonics, holography and X-ray, have been used successfully for many years. The physical recording media, e.g. film, has evolved to electronic data storage and manipulation.

34.8.2 Detection of defects

34.8.2.1 General

Basic guidelines on which NDT techniques can detect which specific type of anomaly or defect are given for:

- Manufacturing, assembly and payload integration, using:
  - contact methods in Table 34.8.1
  - non-contact methods in Table 34.8.2
- In-service for long-life structures, using:
  - contact methods in Table 34.8.3
  - non-contact methods in Table 34.8.4

Table 34.8-1 - Detection of manufacturing defects by contact NDT techniques

Defect	Visual Inspection	Tap Test	Ultrasonic C-Scan	Acoustic Flaw Detector	Fokker Bond Tester	Eddy Currents	Acoustic Emission
Porosity	Only possible on outer surface	NS	Successful	NS	NS	NS	NS
Prepreg gaps	Successful on outer surface	NS	Successful	NS	NS	NS	NS
Contamination (solid)	Successful on outer surface	NS	Success is particle size dependent	NS	NS	Only if metallic	NS
Contamination (backing sheet)	Detection only possible in thin laminates	Detection only possible in thin laminates	Successful	Successful	Successful	NS	NS
Fibre alignment lay-up order	Detection on outer surface	NS	Detection of lay-up order possible using pulse echo	NS	NS	Detection Possible	NS
Fibre/resin ratio variations	NS	NS	Detection possible	NS	NS	Detection Possible	NS
Prepreg joints	Successful on outer surface	NS	Detection in thin laminates	NS	NS	Detection Possible	NS
Interply delaminations	Possible on thin laminates	Possible on thin laminates	Successful	Good detection	Good detection	NS	Detection Possible
Microcracking	NS	NS	Detection Possible	NS	NS	NS	Detection Possible
Skin to core debonding	Outer surface disturbance can be visible	Successful for thin skins	Successful	Good detection	Good detection	NS	Detection Possible

Table 34.8-2 - Detection of manufacturing defects by non-contact NDT techniques

Defect	X-ray Radiography	Holography	Thermography	Laser Ultrasonics	Air-coupled Ultrasonics (1) (2)	Computer Tomography	Laser Shearography
Porosity	Successful	NS	NS	NS	Successful(1)	Successful	NS
Prepreg gaps	Successful	Successful, mainly on thin laminates	NS	NS	Detection Possible(1)	Successful	Detection possible on thin laminates
Contamination (solid)	Successful	NS	NS	NS	? (1)	Successful	NS
Contamination (backing sheet)	Not suitable	Successful	Possible detection in thin laminates	Detection Possible	? (1)	Detection Probable	Detection Possible
Fibre alignment lay-up order	Successful	Detection possible	NS	NS	NS(1)	Successful	Detection Possible
Fibre/resin ratio variations	Detection possible	NS	NS	NS	? (1)	Detection Possible	NS
Prepreg joints	Successful	Detection in thin laminates	Detection Possible	NS	? (1)	Successful	Detection possible on thin laminates
Interply delaminations	NS	Successful	Detection Probable	Detection Possible	Successful(1)	Detection Probable	Detection Probable
Microcracking	On edges with penetrant enhancement	NS	NS	NS	Detection Possible(1)	NS	NS
Skin to core debonding	NS	Successful	Detection Probable	Detection Possible	Successful(1)	Detection Probable	Detection Probable

Table 34.8-3 - Detection of in-service damage by contact NDT techniques

Defect	Visual Inspection	Tap Test	Ultrasonic C-Scan	Acoustic Flaw Detector	Fokker Bond Tester	Eddy Currents	Acoustic Emission
Matrix Microcracking	NS	NS	Detection Possible	NS	NS	NS	Detection Possible
Delaminations	Only on surface	Possible if severe	Successful	Successful	Successful	Only if Severe	Detection Possible
Broken Fibres	Only on surface	NS	Detection Possible	NS	NS	Detection Possible	Detection Possible
Crushed Core	Possible if severe	NS	Detection Possible	NS	NS	NS	NS
Skin:Core Debonding	Possible if severe	Possible if severe	Successful	Successful	Successful	NS	NS
Skin Debonding	Possible if severe	Possible if severe	Successful	Successful	Successful	NS	Detection Possible
Water in sandwich panels	NS	NS	Detection Possible	NS	NS	NS	NS

Key: NS - Not Successful.

Table 34.8-4 - Detection of in-service damage by non-contact NDT techniques

Defect	X-ray Radiography	Holography	Thermography	Laser Ultrasonics	Air-coupled Ultrasonics(1)	Computer Tomography (if available)	Laser Shearography
Matrix Microcracking	Possible on edges with penetrant	NS	NS	NS	Detection Possible	NS	NS
Delaminations	Detection Possible	Successful	Detection Probable	Detection Possible	Detection Possible	Successful	Detection Probable
Broken Fibres	Detection Probable	Detection Possible	Detection Possible	Detection Possible	?	Detection Probable	Detection Possible
Crushed Core	Successful	Detection Possible if severe	Detection Possible if severe	NS	Detection Possible	Successful	Detection Possible if severe
Skin:Core Debonding	Detection Possible	Successful	Detection Probable	Detection Possible	Successful	Detection Probable	Detection Probable
Skin Debonding	Detection Possible	Successful	Detection Probable	Detection Possible	Successful	Detection Probable	Detection Probable
Water in Sandwich Panels	Successful	Detection Possible	Successful	NS	Detection Possible	Successful	Detection Possible

Key: NS - Not Successful; ? - To be determined; (1) AIRSCAN™, [See also: 34.10]

The size and shape of defects detectable by a particular technique depend on the nature of the materials under test, the structural configurations and the equipment used, e.g. probe type, test frequency, resolution of the scanning mechanism and recording media. It is therefore impossible to state definite detection limits for a particular NDI technique because each is application-dependant.

34.8.2.2 Contact versus non-contact methods

Contact methods are those techniques where there is a physical interaction with the component or structure being inspected. This includes:

- Human contact with the structure during inspection.
- Manual operation of probes.
- Use of water or gel couplants, including water immersion.
- Roller probes.
- Direct mechanical loading, as in acoustic emission.
- Application of surface coatings or penetrants to highlight defects.

Any contact with a material introduces the possibility of contamination, which is undesirable for space payloads. Non-contact techniques tend to limit contamination sources.

Those techniques reliant on manual operation can introduce uncertainties regarding operator consistency and possible error. If human judgement is used to interpret the significance of a signal reading, this can differ between individuals. To limit any such inconsistencies, operators are provided with training to particular industry standards.

Automated systems aim to overcome any human operator-related inconsistencies. They tend to need extensive set-up and fine tuning by qualified engineers in order to provide sufficient confidence in the results produced, especially when compared with manual techniques. It is also not uncommon for new automatic systems to produce large numbers of false indications during the set-up phase.

Electronic storage of the information obtained during inspection enables signal data and images to be compared or enhanced when specific features are being addressed. Historical comparisons are also feasible between components made at different times. Such requirements tend to favour non-contact techniques, where the object can be scanned and rotated during examination to show different orientations.

[See also: 34.9 – Contact techniques; 34.10 – Ultrasonic ‘air-coupled’ techniques]

34.9 Traditional contact NDT techniques

34.9.1 General

Each technique is coded as suitable for application in the laboratory [L], during production [P] or in-service [S] during the periodic re-examination of a component or structure.

34.9.2 Visual inspection

Visual inspection is the oldest and most economical form of NDT. It is widely used for locating defects and damage on, or near, the surfaces of materials or components. Some level of contact is necessary, e.g. by the inspector touching the item, although no additional chemical substances are used.

It enables a rapid survey of large areas, or a detailed examination of small areas using optical aids such as magnifying lenses, borescopes and endoscopes. Visual inspection can be followed by another type of NDT inspection to quantify the size of a sub-surface defect.

Application: [L, P and S] – Contact or non-contact.

34.9.3 Dye penetrant

In this technique a liquid dye is applied to the component surface, and is trapped by any surface breaking defects. The component is then cleaned, but some of the dye is retained in the defects present. This is then 'absorbed' by a developing agent to produce a contrasting 'stain' which indicates the position of a defect. Dyes can be coloured or fluorescent under UV light.

Application: [L, P and S] – Contact.

34.9.4 Magnetic particle

A magnetic field is generated in the component. Any surface defects or near-surface cause field leakage and preferentially attract the applied magnetic particles. The particles are normally in the form of a suspension of magnetic iron oxide powder in a hydrocarbon carrier fluid. The magnetic field is induced by magnets or applied flux or current. In general, defects are oriented across the magnetic field to be detectable, with sub-surface defects detectable if their size and position is sufficient to disturb the field.

Application: [L, P and S] – Contact.

34.9.5 Mechanical resonance or impedance

The types of techniques that can be classed as mechanical resonance or impedance include:

- Tap testing is often conducted along with visual examinations. Components are tapped with a coin or special hammer. Variations in sound (resonance) between good and bad areas are usually detected by the operator hearing the differences. Tap tests have a rather limited sensitivity, but can detect fairly large delaminations under the right conditions. It is also difficult to assess which areas have or have not been tested, given a particular area to be screened.

- Bond testers measure mechanical impedance under high frequency oscillation. It compares good with bad areas, providing data on the cohesive, but not the adhesive, nature of the bond; except in the extreme case of no adhesion, [See: 34.20].
- Acoustic flaw detectors work on a similar principle to bond testers.

Application: [L, P and S] – Contact.

34.10 Ultrasonic techniques

34.10.1 Introduction

34.10.1.1 General

There are a large number of techniques known generally as 'ultrasonic' which can be applied in the laboratory, in production and in-service. Each has their strengths and weaknesses, so for any given circumstances one can be more appropriate than the others.

Most techniques involve some contact with the component or structure, but air-coupled systems aim to avoid contamination of composite, space-destined structures by water or gel-based couplants. This non-contact technique was evaluated by Alenia for CFRP, Ref. [34-94], and was selected by Fokker to inspect solar panel substrates, Ref. [34-91].

Ultrasonic testing uses high-frequency sound waves; often in the kHz to MHz range. The sound energy is reflected or scattered by defects lying within the sound path. The principal relies on transducers, or probes, to emit and receive sound. The orientation of transducers in relation to both the structure and the defects determines which features are detected and resolved. The presentation of signal responses in C-scan form represents the baseline means of examining materials. It has become the accepted means of inspecting composites owing to its versatility in detecting defects, Ref. [34-3], [34-10].

Conventional C-scan ultrasonics is by no means an ideal method as it needs a contact path between the transducer and the component under test, usually achieved by use of liquid couplant. To inspect large areas, the probes are generally moved using a mechanical scanning device. This can be large and complex in order to inspect contoured sections of aircraft made from composites, Ref. [34-95].

As the name suggests, air-coupled ultrasonics do not use couplants, but rely on air transmission between the probes and the component under test to both send and receive the ultrasound.

34.10.1.2 Sound waves

Composites are anisotropic (fibre and resin), orthotropic (variable fibre orientation) and laminar in construction. The location, alignment and orientation of potential defects varies accordingly. It is therefore necessary to utilise different sound-wave behaviours in composites to enable defects to be detected in different constructions.

A wave is a transient time and position phenomenon which conveys energy through space. The speed of a sound wave (time of flight) and the strength (energy) are the parameters used for locating defects and quantifying their size. The wave behaviours which are utilised in NDT are:

- bulk waves (plane or spherical), e.g. longitudinal waves
- transverse bulk waves, e.g. shear or distortional waves

- surface effects near a boundary, e.g. Rayleigh waves.
- plate effects (in a medium with two parallel boundaries, i.e. a laminate), e.g. Lamb waves, Ref. [34-11], [34-12].

34.10.2 Modes of examination

Ultrasonic equipment is usually configured to operate in one of three main modes, i.e.:

- Through-transmission (T-T); using two transducers placed on opposite sides of the component. One transducer sends the ultrasonic pulse, the other receives it.
- Pulse-echo (P-E); using one transducer placed normally (longitudinal waves) or at a selected angle (shear waves) on the component surface. This single transducer both sends and then receives the ultrasonic pulse. Some techniques also use a reflector plate placed on the back face of the component.
- Resonance or resonant frequency; by creating a continuous ultrasonic wave in the material. Sensing the received signal phase, amplitude or resonant frequency shift indicates the material impedance or stiffness.

These modes are the basis for the well-established and proven forms of ultrasonic NDT.

A couplant medium is normally used to ensure a good acoustic path between the transducers and the component. Alternatively the component and transducers are completely immersed in a suitable coupling liquid, e.g. water.

34.10.3 Ultrasonic signal presentation

When a component is examined, the ultrasonic signal is presented in various forms for interpretation. These are:

- A-scan: A single point signal describing the ultrasonic response of material immediately beneath the transducer.
- B-scan: A scanned line showing features at identifiable depths.
- C-scan: An area scan showing volumetric defects in the material.

34.10.4 Data and image processing

Image enhancement techniques are invaluable tools when applied to the interpretation of C-scans. The use of image enhancement enables the display of features which cannot be viewed when an ultrasonic C-scan is initially formed, Ref. [34-10], [34-89]. Post processing of data can include, Ref. [34-89]:

- Image presentation:
  - Look-up tables and colour coding.
  - Image reversal.
  - Grey scale ramping or saw-tooth scaling.
  - Isometric projection.
  - 3-D imaging.
  - Binary display imaging.

- Image histogram evaluation:
  - Image enhancement.
  - Equalisation enhancement.
  - Logarithmic forcing functions.
  - Windowing and level slicing.
  - Sub-area evaluation.
- Kernel multiplications:
  - Low- or high-pass filtering and smoothing.
  - Selective orientation filtering and shadowing.
- Two-dimensional Fourier analysis:
  - Fibre orientation distribution.
  - Effects of pixel sizes.
  - Image enlargement.
  - Mask multiplication filtering.
- Full volume imaging:
  - 3-D databases.
  - Peak amplitude images.
  - Integrated amplitude images.
  - Statistical imaging.
  - Frequency domain imaging.
  - 3-D presentation.

Data processing is not only useful for ultrasonics but is equally applicable to other NDT techniques producing digital data.

34.10.5 Equipment calibration

Ultrasonic equipment needs calibration to ensure consistent and reliable detection of defects. This can be achieved using reference specimens containing defects of known dimensions and locations.

With ultrasonics there are many possible sound-wave modes, transducer types and frequency ranges. A systematic means of describing and comparing techniques and devices can be useful. The receiver operating characteristic, ROC, is one such means, Ref. [34-13]. The ROC method assesses the performance of a system and shows the probabilistic character of defect detection. It is based on the general theory of signal detection by counting the input and output from a diagnostic system which includes the task of separating a signal from background noise, Ref. [34-13].

34.10.6 Large area scanning

There are several means of scanning components and mouldings by passing a transducer over the surface of interest, Ref. [34-11], [34-14], [34-95]:

- X-Y-Z scanning frames for:
  - small individual components
  - large aircraft sections, Ref. [34-95].
- X-Y scanning frames for flat panels.
- Programmable robotics for repetitive testing.

The types of transducers used include:

- Roller probes for mainly flat constructions.
- Water-jet probes for shaped mouldings with cut-outs, Ref. [34-95].
- Air-coupled probes for CFRP composites and solar panel substrates, Ref. [34-91], [34-92], [34-94].

To reduce the time to inspect large areas, such as aircraft, portable scanning systems are now being introduced, Ref. [34-15], [34-16]. Examples include LACIS 'large area composite inspection system' and MAUS 'mobile automated ultrasonic scanner'. The main features of these are:

- Use in pulse-echo (depth and amplitude), ultrasonic-resonance and eddy-current modes, i.e. interchangeable.
- Multiple transducers, giving strip coverage via simple oscillatory motion of each transducer.
- Portable, wheeled scanner with automatic transducer alignment for contour following.
- Manual operation of scanner.
- Water drop coupling.
- Automated data collection and enhanced C-scan imaging through a lap-top computer.
- Scan rates of up to 9 m^2 per hour.

The LACIS or MAUS derivatives are used principally for detecting impact damage and bondline defects.

34.10.7 Acousto-ultrasonics

Also known as the stress-wave factor method (SWF), acousto-ultrasonics uses stochastic wave propagation to detect and quantify defect states, damage conditions and variations of mechanical properties within composites, Ref. [34-10].

It is not necessarily intended to resolve each individual flaw, but instead, to assess the collective effect of diffuse flaw populations. Accordingly, the SWF approach was devised to evaluate the integrated defect state due to porosity, matrix crazing, fatigue damage, fibre bunching, fibre breaks, resin richness, poor curing or poor fibre-to-matrix bonding.

SWF can be described as a generalised approach to ultrasonic testing using two probes (transmitter and receiver). The received signal is a result of multiple interactions within the material microstructure, i.e. multiple, reflected, scattered and mode-converted waves.

Signal interpretation is all important. With sufficient correlation with measured properties from test coupons, it is indicated that SWF is capable of quantifying residual ultimate tensile strength of fatigue-damaged composites, interlaminar shear stress and adhesive bond strength.

34.10.8 Lamb wave (guided wave)

34.10.8.1 General

Lamb waves are capable of propagating relatively long distances in thin plate geometries and laminated structures. Lamb waves offer a potentially powerful NDE technique for assessing the health of such structures. Lamb wave propagation properties strongly depend on the thickness and the mechanical properties of the material. Changes in the effective thickness and material properties caused by structural flaws, such as disbonds, Ref. [34-104], [34-105], [34-106], corrosion, and fatigue cracks, can be detected efficiently by measurements of variations in Lamb wave propagation. Owing to the strong dispersion of the Lamb wave modes, the propagation velocity is a characteristic function of the product of the imposed acoustic frequency and the effective thickness of the material.

Lamb wave techniques are envisaged for health monitoring to detect damage in composite material (diagnosis based on Lamb wave propagation analysis) using a permanently attached guided wave array, Ref. [34-107], [34-108]. By taking measurements at different stages in the life cycle of the structure, using a permanently attached device on the structure, comparative measurements can be performed to detect emerging defects.

Several researchers have shown that Lamb waves are viable in principle, but have limitations, including:

- strong sensitivity to the material properties,
- thicknesses of the adherends,
- relative insensitivity to defects at the bondline layer.

Embedded modes, which propagate along an embedded layer, are largely insensitive to the adherends. The dispersion curves show a major improvement in sensitivity to the properties of the layer and to the boundary conditions between layer and the adherends. The difficulty is to generate these specific modes, so current applications are limited to homogeneous and isotropic materials, e.g. adhesive bonding with thin metallic plates or glass. Although attempts have been made with composite materials, the technique remains a laboratory research tool. A significant effort is needed to model the phenomena to account for variations in material properties in order to meet industry needs.

Conventional methods of exciting and detecting guided waves include the angle incidence technique, inter-digital transducers (IDTs) and EMATs.

34.10.8.2 Leaky-Lamb-wave techniques

Lamb waves occur in plates as a result of reflections between the adjacent surfaces found in laminates. In some instances, these waves can be used when more conventional techniques, such as a single probe pulse-echo mode fails to locate defects. Leaky-Lamb waves are also termed obliquely insonified ultrasonic waves, Ref. [34-17].

In this technique, access to the composite is only needed from one face. For thick composites, e.g. 30-ply CFRP, it can be difficult to resolve delaminations in the first few plies. By using a transmitter and receiver, 10mm apart, it is feasible to detect near-field, subsurface defects, Ref. [34-11]. The basic equipment used can be the same as that used in normal contact pulse-echo mode. This can be a commercial broadband probe with a digital ultrasonic flaw detector.

The utilisation of Lamb waves is seen as a low-cost means of inspecting large structures for delaminations, Ref. [34-18]. By placing a transmitter and receiver adjacent to each other on the surface of a composite, it is feasible to detect defects by comparing outgoing and reflected signals, as shown in Figure 34.10.1, Ref. [34-12]. The transmitter and receiver are located at a point, but have the ability to inspect the surrounding composite when rotated or line scanned. It is possible to detect 20mm delaminations at a distance of up to 500mm.

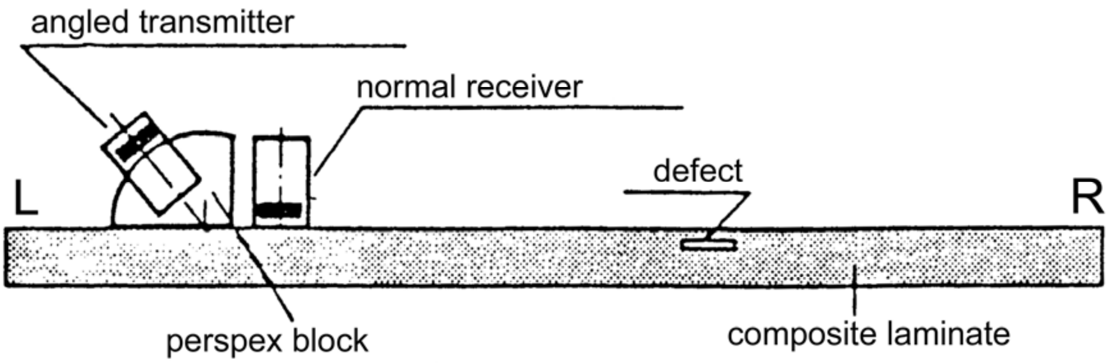


Figure 34.10-1 - Leaky-Lamb-wave technique for composites

34.10.9 Rayleigh wave techniques

Rayleigh waves are not applied to polymer composite examination. They are more appropriate for ceramic composites and coatings, Ref. [34-9].

34.10.10 Backscattering techniques

These are capable of detecting matrix cracking, porosity, fibre orientation and fibre misalignment in individual plies, Ref. [34-3], [34-20]. The technique, as shown in Figure 34.10.2, enables a transducer to be rotated about a normal to the surface axis at various angles of incidence, Ref. [34-3]. The maximum back-scattered energy is received by the transmitting/receiving transducer when the transducer axis is normal to the defect. Thus, the shape, location and orientation of the defect can be approximated by measuring the pulse signal amplitude as a function of rotation (polar) angle.

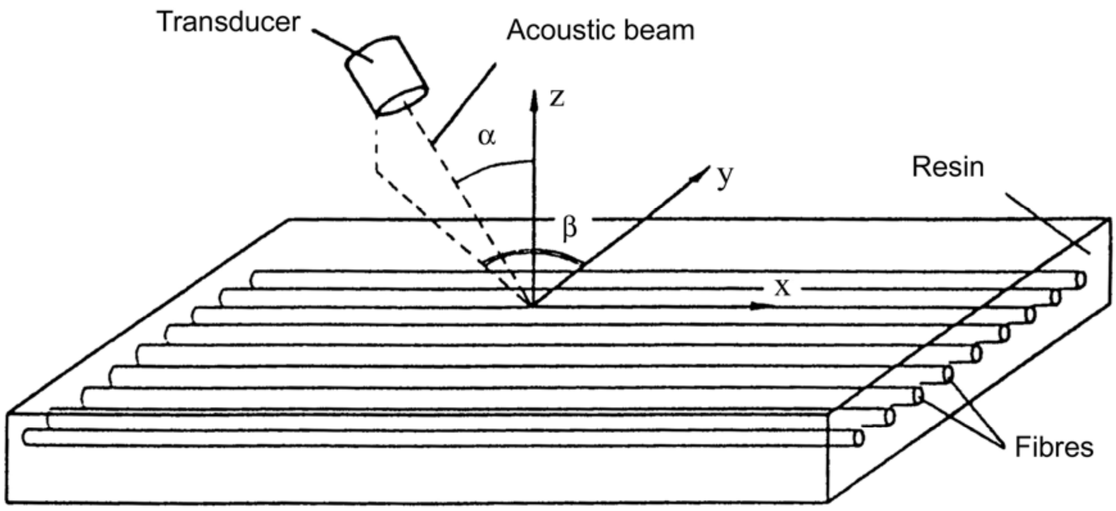


Figure 34.10-2 - Detection of fibre orientation by backscattering measurement

34.10.11 Transducer technology

34.10.11.1 General

Transducers, or probes, emit the ultrasonic waves into the subject material or receive the resulting signal. In some systems the probes can perform both functions. The active element within a probe is usually made of a piezoelectric ceramic or polymer (PVdF). The dimensions of the piezoelectric element largely define the effective frequency range of the transducer, although the actual operating frequency is determined by the signal from the driving electronics. Transducers generally have a stated frequency value, giving their optimum sensitivity and the centre of their effective frequency range.

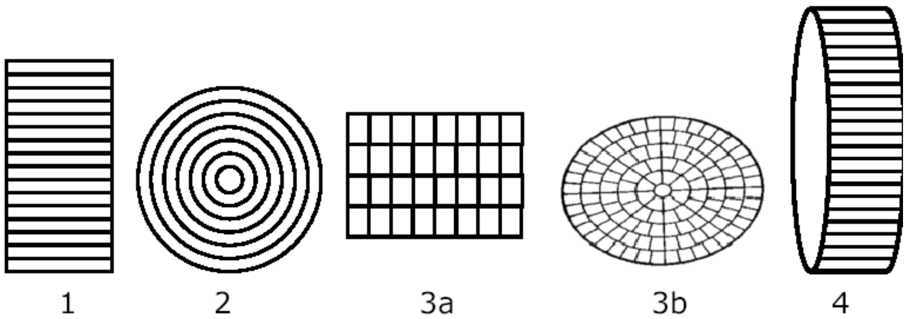
Some of the transducer and applied signal variables which differ between testing techniques include:

- Focused or unfocused probes.
- Ultrasound frequency:
  - selected with respect to material, defect size and defect location.
  - high frequencies for better defect resolution.
  - low frequencies for low signal attenuation.
- Probe alignment to surface:
  - normal,
  - angled, or
  - oblique.
- Signal pulse profile:
  - wave train (pulse) duration,
  - signal energy.
- Mode of operation, e.g. pulse-echo, through-transmission.

34.10.11.2 Phased-array transducers

Piezo-composite materials have provided a new ultrasound probe technology for the non-destructive testing of materials; known as 'Phased-array transducers'. The probes comprise a large number of individual elements each of which can be driven independently. The transducer elements are usually organised in linear, annular, circular or matrix arrays, as shown in Figure 34.10.3.

Phased-array transducers can be used with conventional coupling agents (gel, water) either as a contact or an immersion configuration. In theory, all the applications using ultrasound techniques can use phased-array probes.



1. Linear array
2. Annular array with uniform pitch (non constant surfaces)
3. Matrix arrays (checkerboard and sectored rings)
4. Circular array

Figure 34.10-3 – Phased-array transducers: Element configurations

The probes are connected to specially-adapted drive units enabling independent, simultaneous emission and reception on each channel. The units also enable different electronic time delays for each channel during both emission and reception. Electronic scanning, focusing and deflection can then be carried out; as shown schematically in Figure 34.10.4, Ref. [34-101].

Theoretically, all the applications using ultrasound techniques can use phased-array probes.

In comparison to classical ultrasonic techniques, the main advantages of phased-array probes are the improvement of inspection performances, the reduction of inspection delays, and the possibility of a complete inspection of an item in one scan. The conventional mechanical scanning is replaced by the much faster electronic scanning (advantage for large structures). Electronic scanning in depth can become interesting for structures of large thickness. Moreover, electronic focusing enables the use of a single probe for working at different depths. The electronic focussing can be advantageous for structures including heterogeneities of small dimensions too. Recent investigations have revealed the capability of such sensors to detect fibre waving.

Electronic deflection enables the angle of incidence to be varied with a single probe giving access to particular, inaccessible areas and facilitating inspection of structures with complex shape.

Electronic scanning

Groups of elements are sequentially activated to move the beam along the transducer (with electronic delays to focus the group).

Electronic focusing

Electronic delays are applied to each element to focus the beam.

Electronic deflection

Electronic delays are applied to each element to deflect the beam

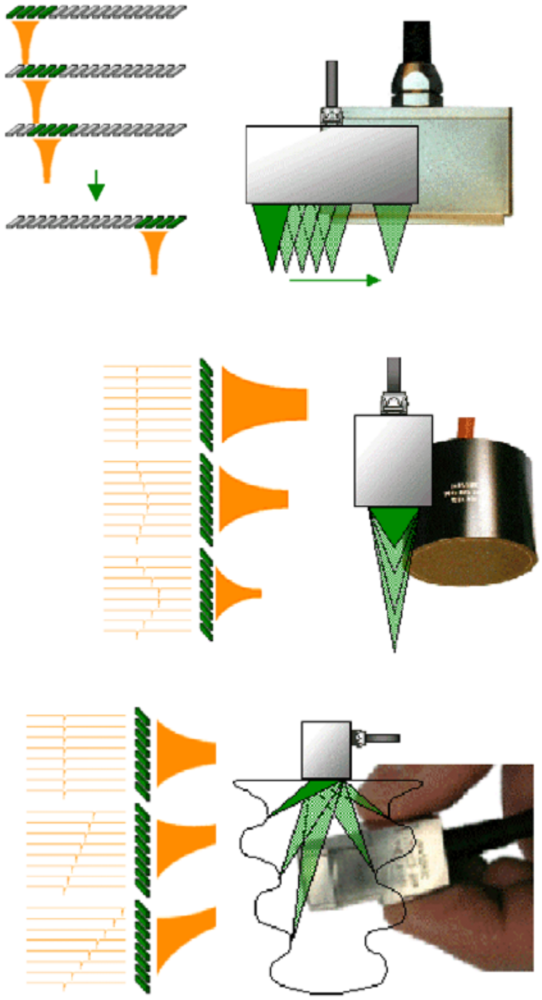


Figure 34.10-4 – Phased-array transducers: Functionality

Advanced functions of ultrasonic phased-arrays are emerging for the non-destructive testing of aeronautic parts, increasing further the potential benefits of the technology, e.g. for emission: multi-beam generation; for reception: differentiated sum of elementary signals, for emission-reception: iterative process.

Currently available phased-array transducers are expensive, mainly because MHz-order frequencies imply tight tolerances and high manufacturing precision. On-going work aims to develop cheaper, low frequency phased-array systems for damage detection in aircraft structures.

The initial applications of ultrasonic phased-array probes were for complex-shaped structures (areas with difficult access) and in the metallurgic field (to reduce control delay by electronic scanning). NASA, McDonnell Douglas and Boeing Space have used phased-array technology for the inspection of welded space structures, e.g. friction stir welds (Delta IV tank) and laser welds. The technology has been applied successfully to large aeronautic structures made of composite materials and honeycomb parts (industrialisation of the phased array system).

Some examples of the successful applications of phased-array technology within the Europe include NDT of laser-beam welding of skin stiffeners for the fuselage of Airbus A 318 (St-Nazaire) and for the inspection of composite parts of Airbus A 380 (Nantes).

34.10.12 Air-coupled techniques (non-contact)

34.10.12.1 General

The use of liquid couplants has traditionally been necessary because of the sound signal attenuation caused by air at high frequencies, e.g. MHz-range. Strong reflections at the interfaces between transducer-to-air and air-to-material under test produce very weak transmitted signals. Advances in acoustic transducer matching-to-air technology in combination with tone burst excitation offer the opportunity for air-coupled ultrasonics, Ref. [34-21], [34-92].

Air-coupled ultrasonic techniques have become reliable and useful methods for non-contact non-destructive testing.

34.10.12.2 Applications

Numerous cases exist where coupling agents, such as water, gels, grease, glycerine, are either prohibited or not desirable, e.g. green powders, uncured polymers, solar panel sandwich supports, some thermal protection systems sensitive to water ingress, rough surfaces, non-flat surfaces and porous materials, such as ceramics and carbon/carbon composites.

Non-contact transducers have been used for quantitative and qualitative ultrasonic evaluation of a wide range of materials, including, Ref. [34-96], particulate and fibrous multi-layer composites, including prepregs; composite honeycomb structures; ceramics (green and sintered); metal (powder-based); rubbers and plastics.

34.10.12.3 Transducers and configuration

Techniques used in non-contact modes include operation of transducers in through-transmission, separate transmitter and receiver placed on the same side ('pitch-catch' configuration) and recently a single transducer in direct reflection mode; as shown in Figure 34.10.5.

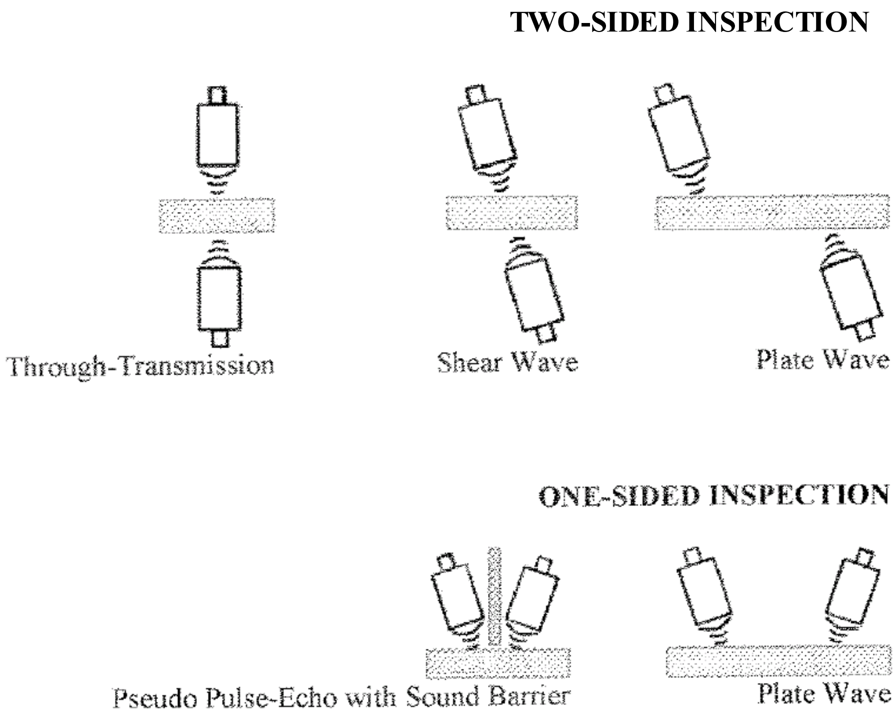


Figure 34.10-5 - Non-contact ultrasonics: Air-coupled sensor configurations

A broad range of frequencies, typically from 50 kHz to 5 MHz, are possible using different sensor technologies: piezo-ceramic, piezo-composite or capacitive transducers, Ref. [34-97]. Some of them provide improved focussing which gives better resolution.

Capacitive-type transducers, characterised by high bandwidths, have been used to evaluate composites and other materials. They have been successfully used for the generation and detection of guided waves in anisotropic viscoelastic materials in a non-contact mode; as shown in Figure 34.10.6, Ref. [34-98].

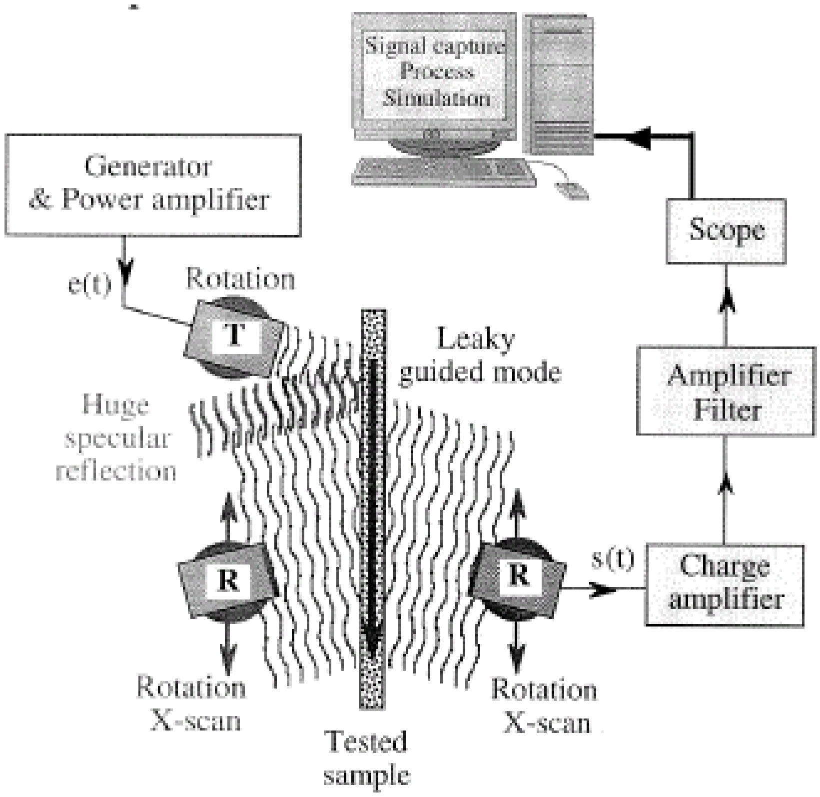


Figure 34.10-6 - Non-contact ultrasonics: Capacitive sensors in plate wave configuration set-up

Although the applications for non-contact transducers at frequencies above 3MHz in ambient air are limited, transducers between 200kHz to 3MHz have been used for several applications, including space, aeronautical and medical industries.

The availability of various frequencies and digital filters enable an optimisation between high resolution for composites and honeycombs, e.g. 0.5MHz to 1MHz, and high penetration for materials such as foam, rubber and wood (20kHz to 200 kHz).

34.10.12.4 Airscan™ - solar panel substrates

Airscan™, a commercial system from QMI (USA), operates in through-transmission or single-sided inspection (via Lamb waves). It has been evaluated for CFRP structures, some of which contain highly-attenuative materials, such as honeycombs and foams, Ref. [34-94].

Some of the features of the system include:

- General operating frequency of 400kHz or 50kHz, Ref. [34-92], [34-94], compared with 1MHz or higher for contact ultrasonic techniques.
- The signal frequency can be altered for the thickness of the composite.
- A wave train-like signal consisting of up to 15 sequential pulses.
- Optimised incident angle for the transmitting transducer.
- Super-low-noise preamplification of received signals.

The Airscan™ system was validated by Fokker as a means of reducing the cost and lead time for NDI of solar panel substrates. Figure 34.10.7 shows a schematic diagram of the Fokker facility, Ref. [34-91].

Qualification tests have been performed for:

- ARA (advanced rigid array) for commercial geostationary solar arrays, and
- Flatpack - developed for Envisat-1.

Each of the sandwich panel constructions is different, notably in their aluminium core cell-size and thickness. Each has a further complication in the form of a co-cured Kapton film on one of the CFRP skins providing:

- Electrical insulation (between solar cell and sandwich),
- Protection against atomic oxygen (Flatpack panels), and
- Thermal control.

Skin-to-core disbonds were detected in both types of solar panel substrate, [See also: Table 34.8.2 and Table 34.8.4]. The system also detected delaminations in CFRP panels. The results are presented in C-scan form.

Further studies aim to assess the suitability of the technique for the inspection of other flat and curved items, e.g. aircraft floor panels, antennas, central cylinders and electronic circuit boards, Ref. [34-91].

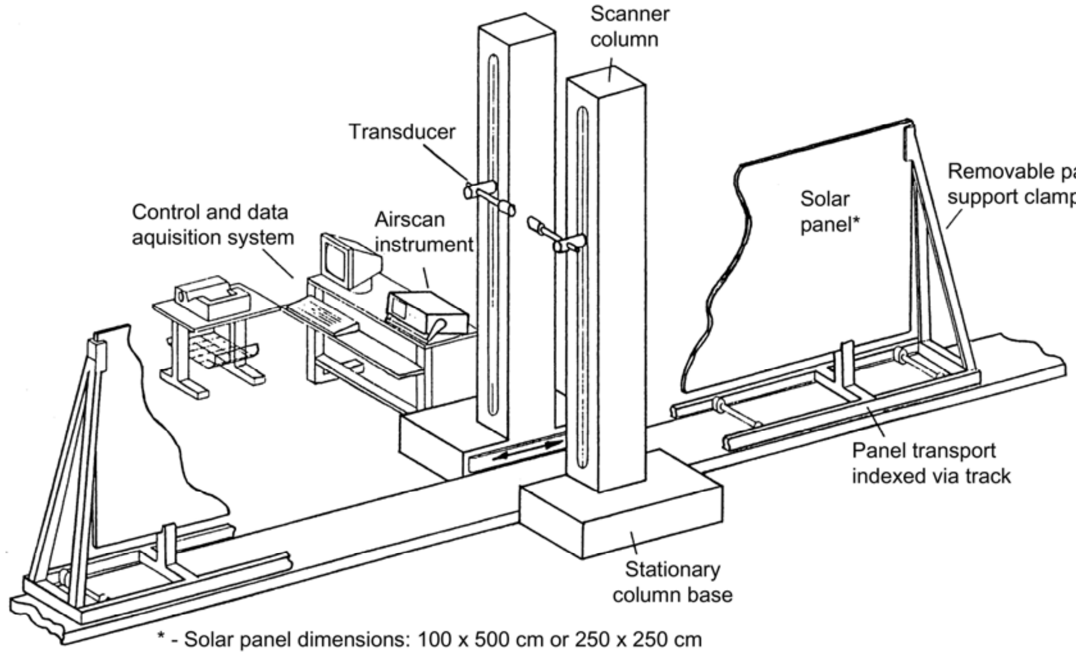


Figure 34.10-7 – Airscan™ (air-coupled ultrasonics) system: Non-contact NDI of Fokker solar panel substrates

34.11 Acoustic emission

AE, acoustic emission, is a contact technique that relies on mechanical straining of the component or structure under test. AE can be applied in the laboratory, in production and in-service.

AE techniques detect the sounds produced by a structure as a result of mechanical loading, Ref. [34-3], [34-22]. These are then identified and related to critical events, such as matrix cracking, delaminations and fibre failure.

Developments of AE have mainly concentrated on the interpretation of signals and the significance of noise emanating from stressed components and structures. For composites, some broad comments include:

- Composites under progressively increasing load produce acoustic events at strain levels well below the elastic limit.
- The point at which acoustic events begin in relation to previous loadings can be described as the Felicity ratio.
- Approaching the elastic limit, acoustic events occur increasingly in the sequence of:
  - random local matrix cracking,
  - transverse ply failure,
  - interlaminar shear failure, and lastly
  - fibre breakage.
- To enable identification of damage events during the testing or inspection of a structure, the acoustic signature of each type needs to be known. Such signatures are often material and structure dependent. Computer pattern recognition is important.

AE can be applied to proof-loaded structures, such as pressure vessels, where the proof test is obligatory and historical data is available from testing numerous items.

When structures are tested to ultimate failure load, AE can be used to identify local damage occurring prior to final failure. Testing is generally easier with components, where the load application is usually simpler and the AE response easier to interpret.

The technique has been evaluated for measuring density and porosity in carbon-carbon components, Ref. [34-23].

The improved availability of cheap computing power has enabled AE to gain greater acceptance for the inspection of structures. Triangulation methods, by comparison of the arrival time of the same sound event at several different transducers, locate the positions of acoustic emission sources in assembled structures, such as aircraft. The frequency and amplitude description of the event is compared with reference data to determine the type of damage.

AE has been evaluated for condition monitoring systems for reusable space vehicle structures, such as tanks, [See: 92.3].

34.12 Laser ultrasonics

34.12.1 General

Laser ultrasonics are non-contact techniques that can be applied in the laboratory, in production and in-service.

The development of laser ultrasonic systems was largely in response to a need for non-contact NDI methods for the examination of assembled structures, Ref. [34-20], [34-24], [34-25], [34-26], [34-27]. An example of this type of technique is LUIS ‘laser ultrasonic inspection system’ from General Dynamics, Ref. [34-27] and Lockheed, Ref. [34-28].

34.12.2 Basic principles

34.12.2.1 General

Figure 34.12.1 shows an example of a laser ultrasonic system, Ref. [34-28]. The sequence of events in the emission and reception of an ultrasound pulse is:

a. A high-power laser pulse is directed at the surface to be inspected, typically from a Nd-YAG or TEA CO2 laser.

b. An ultrasound pulse is generated at the surface, usually by thermoelastic induction (rapid localised expansion by transient heating) for polymer matrix composites.

c. The ultrasound response is detected by a second laser operating as part of an interferometer system.

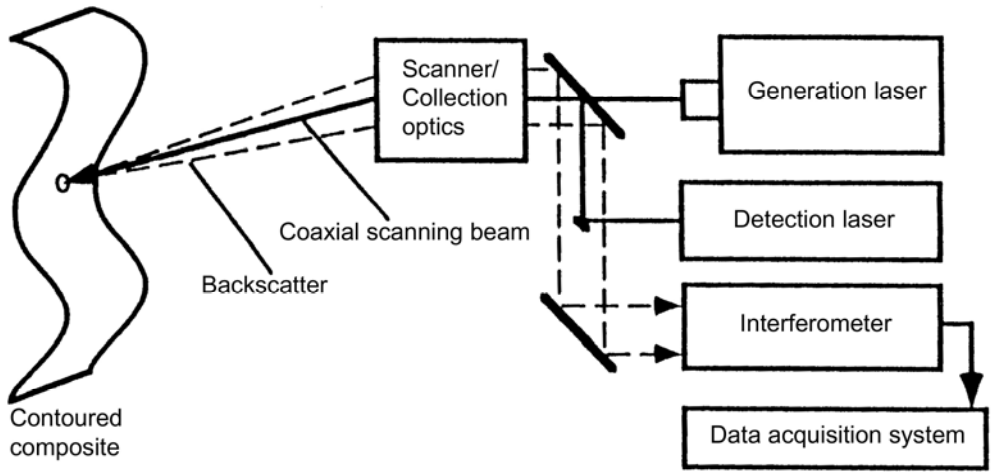


Figure 34.12-1 - Laser ultrasonic test system: Diagrammatic representation

The basic modes of detection are, Ref. [34-24]:

- Optical heterodyning or simple interferometric detection: The wave scattered by the surface interferes with a reference wave derived directly from the laser. This technique is sensitive to optical speckle and the best sensitivity is obtained when one speckle is effectively detected. This needs a focused beam, enabling the measurement of the ultrasonic displacement over a very small spot.

- Velocity or time-delay interferometric detection: Based on the Doppler frequency shift produced by the surface motion and its demodulation by an interferometer having a filter-like response. This technique is sensitive primarily to the velocity of the surface and is therefore very insensitive to low frequencies. The filter-like response is obtained by giving a path delay between the interfering waves within the interferometer. Two wave interferometers (Michelson, Mach-Zehnder) or multiple-wave interferometers (Fabry-Pérot) can be used. This technique enables many speckles to be received and provides a large detecting spot.

The ultrasound produced can be of the usual types, i.e. longitudinal, shear, plate and Rayleigh, Ref. [34-29]. The nature of the ultrasound generated is principally a function of the laser and material properties, such as the:

- Laser pulse duration, wavelength and pulse energy,

- Laser spot size,

- Thermal properties of the composite,
- Laser absorption properties of the composite.

Any inspection system relies on a means of fast optical scanning of the surface to produce a C-scan image. The laser need not be orientated perpendicular to the surface and oblique angles can be accommodated.

Features in composites that have been detected using laser ultrasonics are, Ref. [34-25], [34-27]:

- Surface cracks,
- Delaminations,
- Additional plies,
- Skin-to-core disbonds,
- Solid contaminants (backing film).

It is also possible to detect cracks in aluminium alloy sheet, Ref. [34-29].

34.12.2.2 Disadvantages

Laser-ultrasonic techniques generally have a poorer signal-to-noise ratio than conventional ultrasonics, making defect detection more uncertain. Recent improvements in digital data processing have aided the necessary signal processing and image enhancement of results. Prototype facilities are under development, Ref. [34-28].

34.12.2.3 Applications

In addition to the evaluation of composite structure, possible applications for laser ultrasonics include:

- Remote examination for high-temperature applications.
- Complex shapes and confined recesses.
- Remote analysis in vacuum and corrosive environments.

34.13 Holography

34.13.1 Introduction

Holography is a non-contact technique, except for direct vacuum loading that can be applied in the laboratory and in production.

It is a process whereby a holographic image of a component is superimposed on the same part while it is stressed slightly. Stresses are induced by heating, loading or vacuum which cause disproportionate out-of-plane displacements in defective areas. Discontinuities in the resulting interference fringe patterns can indicate the presence of defects.

The technique has been applied to space structures, notably satellites, from an early stage because of its largely non-contact mode of operation. There are now various different versions, which have benefited from the introduction of digital technology, Ref. [34-30]. Holography can be used to measure accurately small out-of-plane displacements, to micron-level resolution, which can be necessary for antennas undergoing thermal distortion.

34.13.2 Laser interferometric techniques

34.13.2.1 General

There basic techniques which can be used for interrogating a structure are, Ref. [34-3]:

- Interferometry,
- Moiré, and
- Speckle photography.

34.13.2.2 Hologram interferometry

The original holographic technique relied on a split beam of coherent light (laser), one part reflecting off of an object and interfering with the second part on a photosensitive plate. The hologram produced preserved all of the information regarding both the amplitude and the phase of the recorded light wavefront. A complete, three-dimensional reconstruction of the original image is then possible.

When viewing the stressed object and superimposed unstressed hologram, distortion fringes are produced which differ with the applied load. In such cases, the hologram behaves in the same way as an interferometer. Controlling the distortion yields a tool that can be used for interferometric measurements.

The interference fringe patterns can be observed in 'real time' or as a 'double exposure', where the features of each can be summarised as:

- Real time: Precise alignment of an unstressed holographic image with the stressed object. These interfere, and displacements can be detected of orders of magnitude of the wavelength of the employed light. Any change in the state of the object's surface causes a simultaneous alteration of the fringe system, hence the term 'real-time', Ref. [34-32]. This technique does however need a high degree of precision and vibration cannot be tolerated.
- Double exposure: This is simpler, as a single holographic plate is used to make two successive exposures of the object, before the plate is developed. The interference fringes are then 'frozen' at a single stress state in the combined hologram. The resolution is excellent and can be down to 0.2 μm, Ref. [34-33].

34.13.2.3 Moiré techniques

The Moiré effect is an optical phenomenon obtained when two grids consisting of parallel lines with a sufficiently small pitch are superimposed and observed in either transmission or reflection. Any relative strain disturbance between the two grids produces a diagnostic banding pattern. The formation of Moiré fringes is used to evaluate relative displacements on object surfaces. This approach is more appropriate to strain measurement than defect detection.

34.13.2.4 Speckle photography

A laser beam illuminating a rough surface gives rise to a scattered intensity distribution which takes the form of alternately bright and dark spots. This is an interference pattern, known as a field of speckles. The measurement of the displacements that an object undergoes between two recorded states involves optical filtering of the double-recorded images to reveal a Moiré fringe pattern. This in turn depicts the in-plane displacement component. In effect, the natural surface irregularities are used in place of Moiré grid lines.

All speckle methods have the advantage over Moiré techniques of not needing a grid printed onto the object surface. Electronic speckle pattern interferometry, ESPI, is based on this principle.

34.13.3 Vibration, pressure and thermal loading

34.13.3.1 General

For holography to work, physical displacements are induced in the object being tested. Although the displacements, in effect strains, need only be very small, the object can be large or awkwardly shaped making direct physical loading inappropriate or difficult. Various solutions have been devised to produce the desired effects.

34.13.3.2 Vibration loading

Vibrational loading can be achieved with a piezoelectric transducer attached to the subject or test fixture. If vibrational stressing is used, a time average hologram is taken while the structure is excited. A random excitation signal is generated, filtered to the desired frequency range and amplified to drive the transducer, Ref. [34-34].

34.13.3.3 Pressure loading

The use of differential pressures is particularly appropriate for sandwich structures where the internal cavities can be used to create a temporary straining mechanism, Ref. [34-35], [34-36]. The pressure differential can be established by placing the component or structure in a vacuum chamber, or inside a transparent vacuum bag, Ref. [34-37]. In that the pressure eventually equalises, real time images of the fringe patterns are preferable.

Direct vacuum loading can also be achieved by clamping a vacuum shell onto an area of a larger surface, Ref. [34-35]. However, this then becomes a contact NDT technique.

34.13.3.4 Thermal loading

The use of transient thermal loading to induce surface displacements can be achieved by:

- Hot air blowers, Ref. [34-38].
- Infrared heaters, Ref. [34-32], [34-33].

It is usual to heat the object and then to monitor the cooling stage. This method can highlight defects as well as offering information on the thermal distortion of structures under simulated space conditions, Ref. [34-32], [34-33].

34.13.4 Electronic imaging

The double exposure of a photographic plate is an accepted means of achieving high accuracy of recorded displacements. It is however time consuming, costly and operator dependent. This is not always a problem for the examination of space payloads, such as reflectors, but it is less attractive in other industrial sectors demanding rapid inspection.

The speed of the process can be increased by using a video recording. Holographic images can be recorded (30 frames/second) and analysed without the need for film.

Image-plane holograms are produced by a speckle interferometer using a charge coupled device CCDTV camera, Ref. [34-34]. The information can be stored digitally for automated processing and colour enhancement. Figure 34.13.1 shows the basic configuration of an electronic system, Ref. [34-34].

Electronic imaging has been criticised for not offering the same image quality as conventional double-exposure plate photography. This is likely to change because of rapid progress of computer enhancement and the improved resolution of imaging devices. Near-surface delaminations of 2.5 mm have been detected.

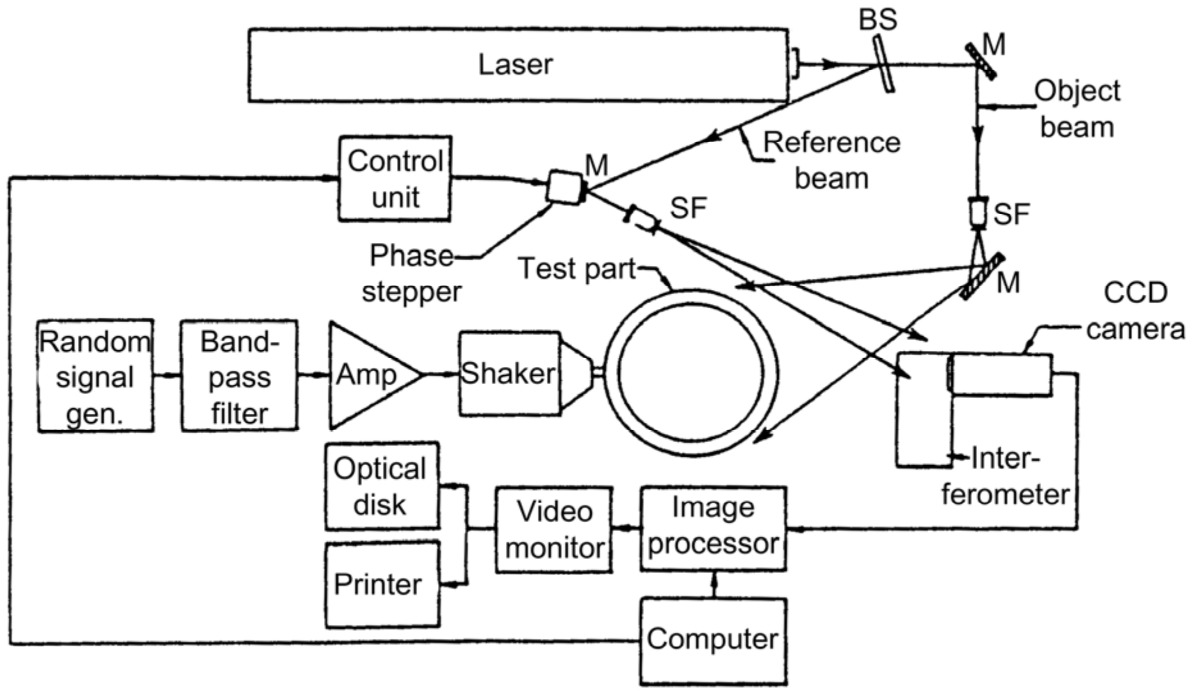


Figure 34.13-1 - Holography by electronic imaging with random vibration excitation

34.13.5 Examination of space structures

34.13.5.1 General

Holography is not just limited to defect and damage detection, but can also be applied to measuring displacements and distortions. Some areas where this has been applied include:

- Shear and compression tests of panels using electronic speckle pattern interferometry, ESPI.
- Thermal distortions of antennas, Ref. [34-32], [34-33].
- Vibration response of panels and structures, Ref. [34-39].

34.13.5.2 Limitations

Holographic techniques can be disturbed by mechanical vibration which alters the respective positions of the object and hologram. Consequently the object needs sufficient isolatation mechanically to inhibit such movement.

Although early holography techniques tended to be time consuming (film technology) and costly, image capture, digital signal processing and handling have improved greatly.

34.14 Laser shearography

34.14.1 Introduction

Laser shearography is a non-contact technique, except for direct vacuum loading, that can be applied in the laboratory, in production and in-service.

It is a relatively new technique, which is similar to holography and aims to provide a means of rapid, real-time, large-area inspection. The advent of electronic digital image processing has motivated its development, Ref. [34-40].

Image-shearing speckle pattern interferometry, commonly known as 'shearography', is a full-field laser-based interferometric technique. Shearography measures strain fields on the surface of the subject, i.e. measurement of out-of-plane surface displacement (dy/dx) of a body under load. Measurement sensitivity as low as 0.1 μstrain can be achieved, Ref. [34-41]. Shearography began as an electronic technique and so avoided the use of photographic plates, Ref. [34-9], [34-30], [34-40], [34-41], [34-42].

34.14.2 Basic technique

34.14.2.1 General

Shearography is a 'self-referencing', common-path, interferometric method, as shown in Figure 34.14.1, Ref. [34-40].

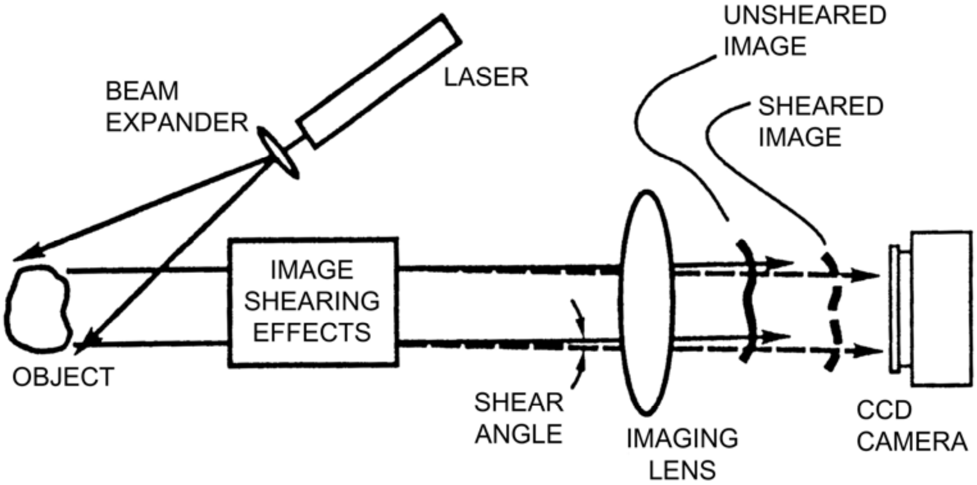


Figure 34.14-1 - Electronic shearographic system: Schematic diagram

Beamsplitting is performed by the shearing optics in such a way that a double image of the object is formed by the imaging lens (usually a commercial lens) on the CCD sensor. The direction and magnitude of the image separation, or 'shear', is determined by the orientation of the shearing optics.

The two wavefronts, labelled as 'sheared' and 'unsheared', are coherently added in the image plane to produce a speckle interference pattern that represents a unique spatial signature of the topography of the surface of the object. Loading techniques, such as thermal, vibration and vacuum pressure, can then be used to induce surface displacement, Ref. [34-9].

34.14.2.2 Portable shearography systems

Portable systems use a vacuum shell applied to the flat or curved structure (e.g. aircraft) to induce a small pressure differential. The operator holds a video monitor and control box during the test, Ref. [34-42].

34.14.3 Characteristics of shearography

The features of shearography include:

- It can operate in C-scan mode.
- Both portable and fixed scanning systems are possible.
- Alignment to surface is not critical.
- It is influenced by surface reflectance.
- The detection resolution becomes poorer as the field of view expands.
- It is more versatile than holography, but less accurate.

Shearography has been used on assembled structures, such as aircraft, Space Shuttle and Atlas Centaur.

Table 34.14.1 gives the types of defects detected, Ref. [34-42].

Table 34.14-1 - Defect detection by shearography

Stressing Technique	Material	Defect Type Detected
Thermal	CFRP sheet	Delaminations Impact damage Some foreign material
	Honeycomb sandwich	Skin to core debonds Crushed core
	All materials	[Thermal expansion]
Vacuum	CFRP sheet	Impact damage Delaminations Porosity
	Honeycomb sandwich	Skin to core and far side debonds Sheared core Crushed core
	Rubber bonded to metal or composites	Debonds
	Foam core panels	Near surface and deep debonds
	Cork to metal or composites	Debonds
Vibration	Honeycomb sandwich	Skin to core debonds Crushed core
	Foam to composite or metal panels	Debonds
Microwave	Materials with high dielectric constant	Entrapped moisture

34.15 Thermography

34.15.1 Introduction

Thermography is a non-contact technique that is suitable for use in the laboratory, in production and in-service.

All thermography techniques rely on the presence of a defect disturbing the heat flow in a component. The surface thermal distribution is monitored by infrared cameras. The different rate at which radiant energy is transmitted or diffused to the monitored surface produces temperature variations which can indicate the presence of defects. All techniques can now benefit from digital signal processing and real-time image display. Thermography for detection of defects and discontinuities is principally a surface or near-surface technique.

Thermography is a versatile concept because it is applicable to a wide range of materials and structures, including composites and metals. It has also been applied to ceramic matrix composites to determine the integrity of oxidation-resistant coatings, Ref. [34-51]. Wherever heat flow, conduction or radiation can be induced, thermography is a possible evaluation tool.

34.15.2 Techniques

34.15.2.1 General

All thermographic techniques aim to establish heat flow patterns on the surface which can be recorded and examined, Ref. [34-49]; by observation and measurement of the thermal diffusivity and effusivity, Ref. [34-43].

Some systems can show the presence of significant impact delaminations over large areas, whilst others offer better resolution over more limited areas. Figure 34.15.1 shows the basic technique, Ref. [34-44].

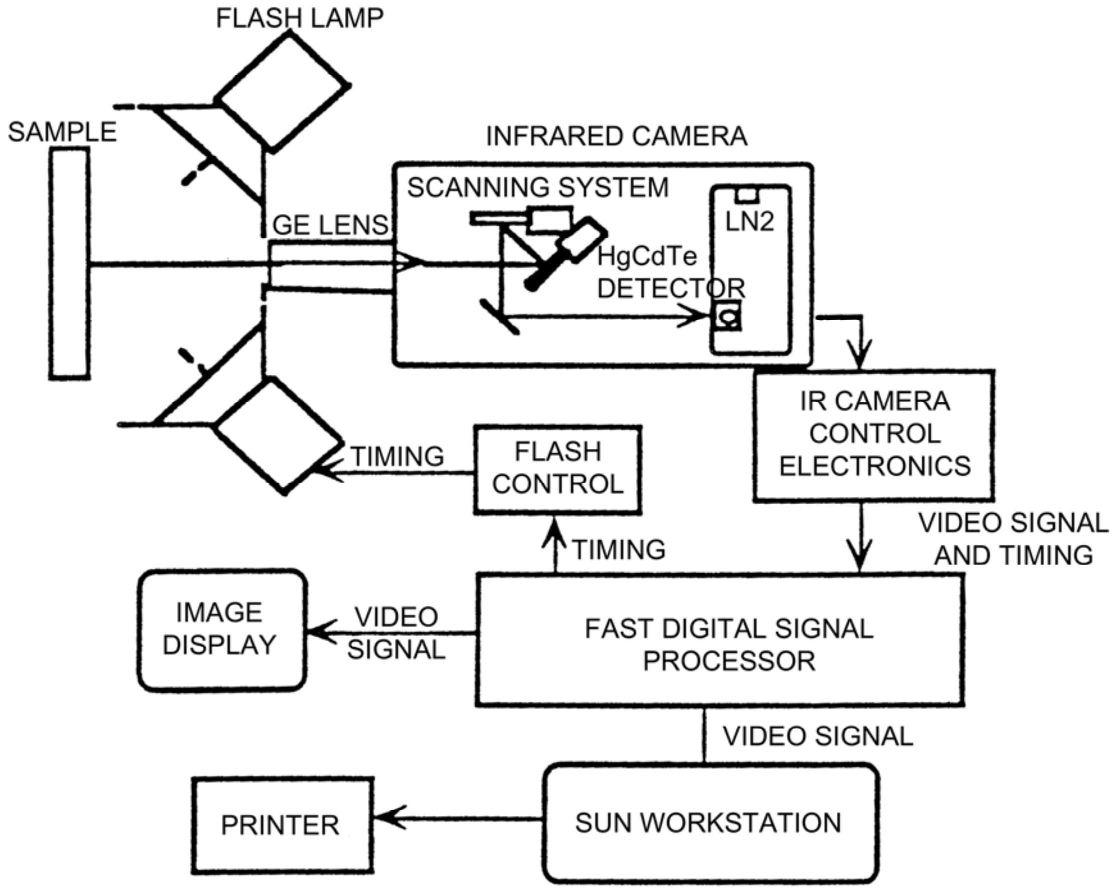


Figure 34.15-1 - Basic thermographic technique

34.15.2.2 Real-time video thermography

This is a general classification for all techniques in which contours of equal temperature (isotherms) are mapped over a surface. The assumption is that defects, inhomogeneities, or other undesirable conditions of the object under test appear as local 'hot' or 'cold' spots. Inspection can be undertaken from one side, where delamination-type defects appear as hot spots, or through-transmission by viewing the back face where cold spots occur. In practice, through-thickness inspection is rare.

Surface thermal distributions can be detected by contact or non-contact methods. Contact methods include thermocouples, temperature sensitive paint or liquid crystals, but these are less attractive than non-contact methods which rely upon infrared radiation to determine the temperature distribution of the radiating source.

Photon-effect devices, sensitive to the infrared wavelengths generated, are used in real-time video thermographic camera systems that scan the entire surface very rapidly. The wavelength of the emitted infrared radiation is related to the temperature of the emitting surface through Planck's radiation law. Different materials have different surface emissivities.

34.15.2.3 Pulsed video or transient thermography

This is the most common form of thermography applied to composite structures, Ref. [34-43], [34-45], [34-46], [34-47], [34-48], [34-88]. The object is heated by a high-intensity pulse of infrared light usually from a xenon or quartz lamp. This typically produces an increase in surface temperature of about 10°C. The heat flow away from this surface is then monitored for about 30 seconds, i.e. in single-sided mode.

Some of the typical heating regimes used are:

- Two 600 mm xenon flash tubes, each delivering 4 kJ over a discharge period of 6 ms giving typically 0.8 J/cm², Ref. [34-48].
- Four xenon flash tubes (rated to 20 kJ total) in a square illuminating 300 mm x 300 mm of surface (up to 2.5 J/cm²), with pulse of 5 ms, scanning at 5 m/min, Ref. [34-46], [34-47].
- Eight xenon lamps, each 6 kJ, with 2 ms pulse duration, Ref. [34-44].

Thermogram images can be taken at intervals from 40 ms to 125 ms, i.e. from 8 per second to 25 per second.

The technology has several distinct advantages as an up-to-date, non-contact technique. These include:

- Portable and compact, with its own data acquisition and image processing.
- Capable of detecting a wide range of defects in thin-section composite constructions, when optimised.
- Appropriate for different materials (polymers, metals and all composites) for detection of delaminations and disbonds.
- Capable of detecting corrosion of alloys and within bonded metal joints, Ref. [34-43].

34.15.2.4 Thermal-wave imaging

Thermal-wave imaging techniques use intensity-modulated heat sources with a variety of detectors to monitor differences in the response of the material to the generated thermal wave. Imaging contrast is provided by variations in the response of the material to the thermal-wave generation. The intensity-modulated heat sources are generally either laser beams or particle beams, the latter in vacuum only. In addition to modulating the intensity of the heat source, e.g. by mechanical chopping of the laser beam, the laser is scanned over the surface.

A point-by-point image of the surface can be built up by electronic processing of the monitored signal. These thermal waves are highly damped and quickly dissipate within a thermal-wave wavelength or two.

Depending on the type of detecting system used to monitor the response, such techniques are usually described as either:

- Photo-optical, which normally have a second laser beam for the detection of thermal-wave effects; as shown in Figure 34.15.2, Ref. [34-49].

- Photothermal, which are generally used for large area inspection; as shown in Figure 34.15.3, Ref. [34-49].
- Photoacoustic, which monitor pressure pulses established in closed acoustic cells by the conduction of heat from the surface of the examined object in air; as shown in Figure 34.15.4, Ref. [34-49].

Thermal-wave techniques can be used to assess the quality of adhesive bonds and the integrity of coatings on metal substrates. The high resolution possible using thermal-wave imaging has been demonstrated by the detection of 10μm near-surface defects in ceramics. It has also been used in a 'microscope' mode for imaging microstructures.

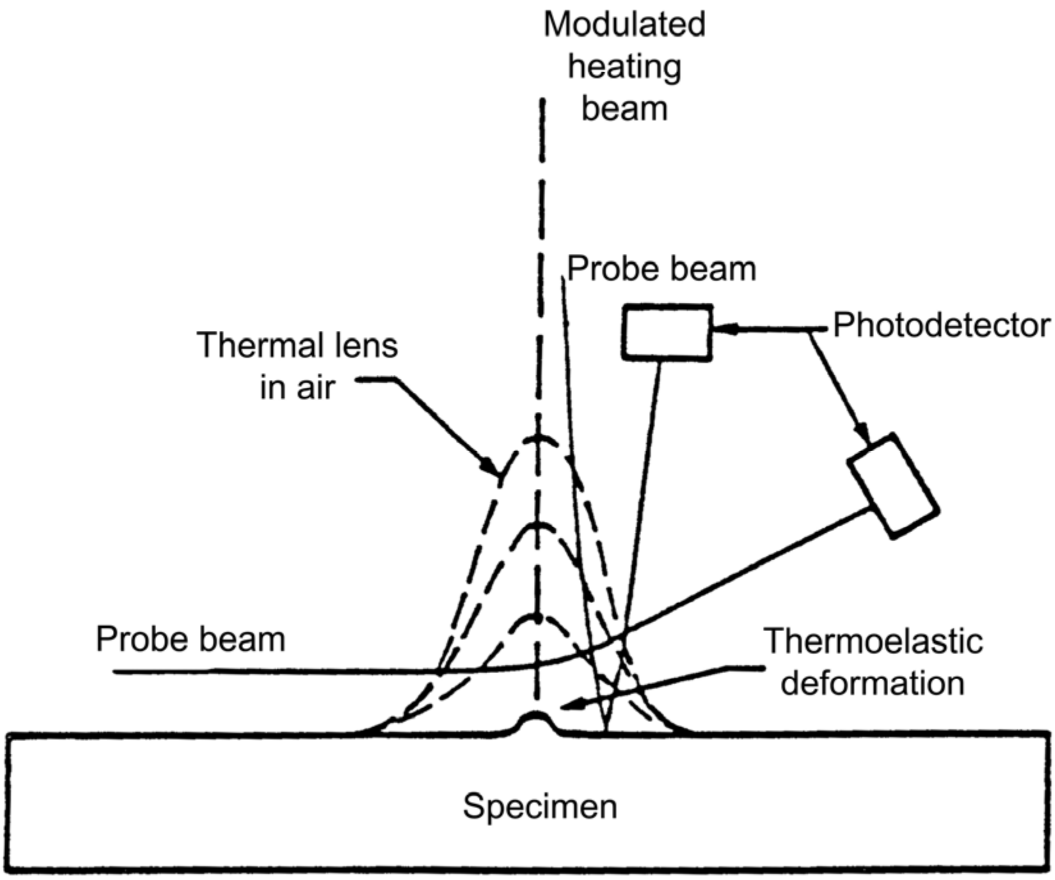


Figure 34.15-2 - Thermal-wave imaging: Photo-optical detection

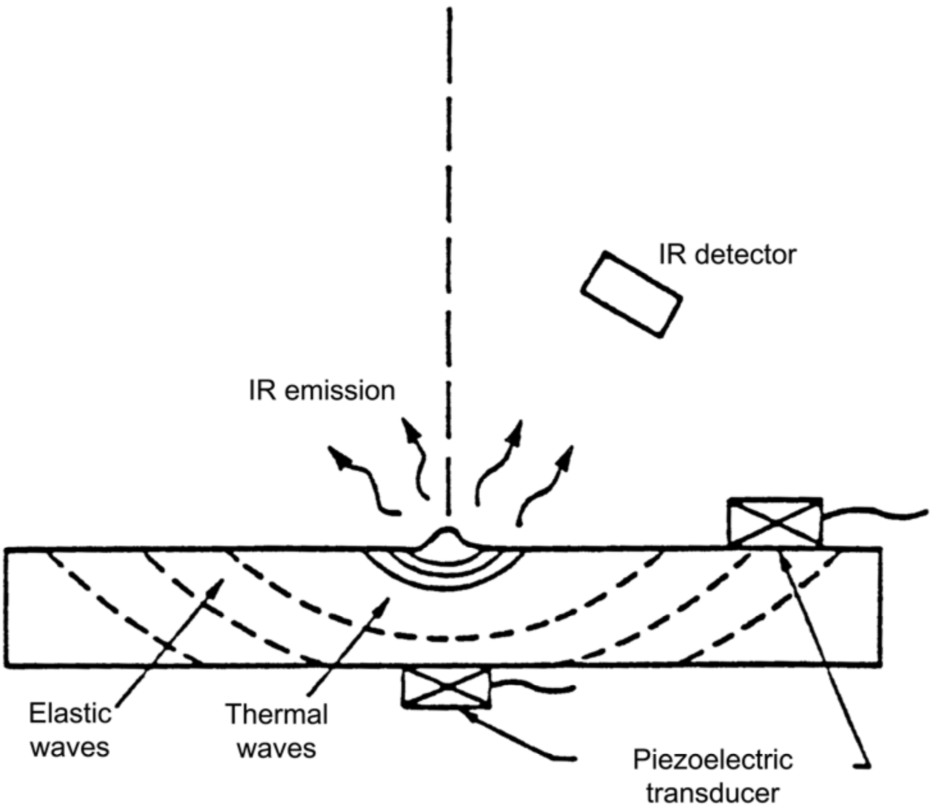


Figure 34.15-3 - Thermal-wave imaging: Photothermal and piezoelectric detection

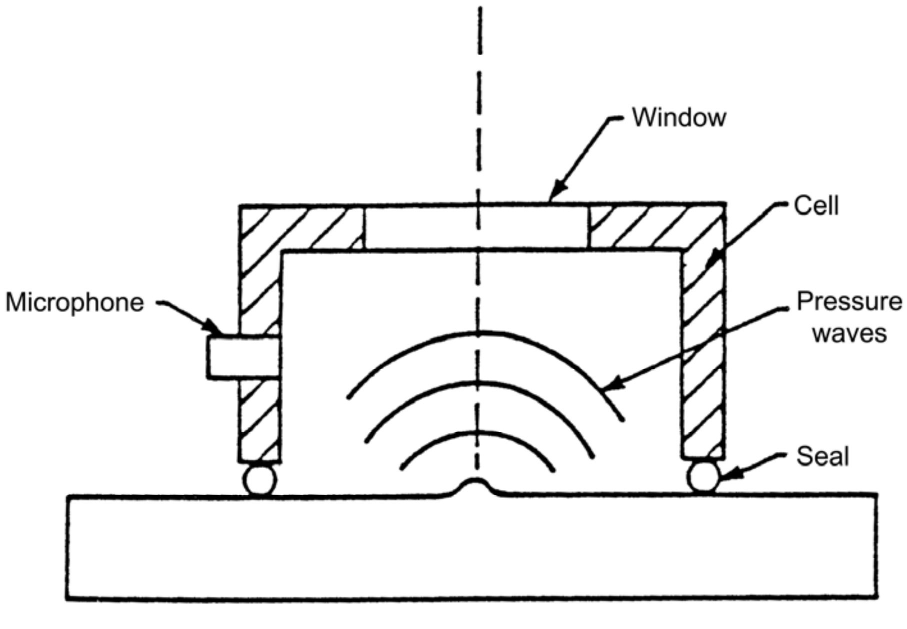


Figure 34.15-4 - Thermal-wave imaging: Photoacoustic gas cell

34.15.2.5 Thermoelastic stress measurement technique

SPATE 'stress pattern analysis by thermoelastic emission' uses heat generated from cyclic loading as a means of observing defects, Ref. [34-49], [34-50]. An infrared camera monitors the radiation emitted at the maximum and minimum levels of mechanical loading during each cycle. Since the local surface temperature is proportional to the instantaneous volumetric strain, the temperature measured by the infrared emission is related to the local principal strains. A raster scan is made from point to point along the surface of the object to develop an image of the principal strain pattern on the surface of the object under test.

The SPATE 9000 system is capable of detecting stress changes of 0.4MPa and temperature changes of 0.001°C, Ref. [34-50].

It can be a slow inspection method and, because the object is mechanically loaded, is in effect a 'contact' NDT technique.

34.15.2.6 Vibrothermography

This uses mechanical vibrations to set-up resonance in damaged areas, leading to locally increased dissipation of heat. Damage in surface plies is more likely to be detected than subsurface or back surface damage because of the strong attenuation of heat conducted through the thickness of the composite.

34.15.3 Heat sources and pulse duration

Early thermographic techniques used hot-air guns and quartz halogen lamps. These gave relatively long heat pulses and proved that it was possible to detect defects such as surface delaminations, typically 10 mm in size. The steepness of the temperature gradient provides the 'contrast' between defective and nondefective areas. The use of more rapid heat pulses, such as those provided by xenon tubes, improves the technology by enabling smaller defects to be detected, e.g. about 1 mm to 5 mm. This also improves the scanning rate.

The progression to laser pulses with narrowly focused points improves the resolution even further, so that sub millimetre-sized defects can be detected. This is perhaps more appropriate for metals (small cracks) and metal or ceramic composites (microcracking, porosity and fine delaminations) than the larger delaminations usually found in polymer composite constructions.

The thermal conductivity and surface emissivity of the test materials largely define the desired pulse energy level and duration.

34.15.4 Detection capabilities

An exercise conducted by NNDTC, AEA Technology, UK in conjunction with ESA/ESTEC established the detection capabilities of the Talytherm 8-90 transient-thermography system, Ref. [34-48].

A summary of the results of the work with flat bottom holes, FBH, control defects for a variety of materials is shown in Table 34.15.1, Ref. [34-48]. Table 34.15.2 gives the detection limits for FBHs in mild steel, aluminium and carbon-carbon materials, Ref. [34-48].

Table 34.15-1 - Thermography: Summary of diameter/depth ratios from different studies

Material	Ratio (Best Experimental)	Ratio (Best Quoted)	Number of Data Points	Range of Depth of Data Points
Aluminium	0.4 for d = 5.0 mm	0.5	6	1.0 - 5.0 mm
Mild Steel	2.0 for d = 2.0 mm	1.5	5	1.0 - 2.0 mm
C-C	2.0 for d = 3.2 mm	4.0	3	1.0 - 3.2 mm
CFRP	2.1 for d = 3.0 mm	1.8	10	1.0 - 3.0 mm
GRP	1.6 for d = 7.0 mm	-	2	5.0 - 7.0 mm
Delrin	9.1 for d = 2.2 mm	-	4	1.0 - 2.2 mm

Key: d = depth

Table 34.15-2 - Thermography: Detection levels established for flat bottomed holes (FBH) from ESTEC study

Depth of FBH (mm)	Min. Diameter Detectable (mm)	Diameter/ Depth Ratio	Pulse Required (kJ)	Temperature Window (°C)
Mild Steel				
0.5	2.0	4.0	1, 2 or 4	4, 6, 15
1.0	2.0	2.0	4	4, 6
2.0	4.0	2.0	4	4
Aluminium				
0.5	4.0	8.0	1, 2 or 4	4, 6, 15
1.0	4.0	4.0	1, 2 or 4	4, 6, 15
2.0	8.0	4.0	2 or 4	4, 6
Carbon - Carbon				
1.0	4.0	4.0	4	4, 6
2.0	4.0	2.0	4	4
3.0	6.0	2.0	4	4
4.0	6.0	1.5	4	4

34.15.5 Limitations of thermography

The limitations of thermography usually show themselves when the defects do not lie near the inspection surface. Through-thickness inspection is rarely feasible, and this is creates particular difficulties when sandwich constructions are inspected for defects adjacent to the back-face skin, e.g. skin-to-core disbonds.

The ‘manufacture’ of representative defects in composite panels poses problems when thermography systems are optimised or calibrated. For ultrasonics, defects such as interlaminar debonding can be simulated by including areas of plastic film. Thermographic techniques detect the large differences in the local thermal conductivity between the composite and its defects. Such defects are generally thin air gaps (delaminations) which have very poor thermal conductivity, whereas a solid 'inclusion', such as plastic film, does not respond in an acceptable manner because the local thermal conductivity remains fairly high. For this reason, flat bottomed holes, FBH, drilled from the back-face are used to establish the detection capabilities of thermography for different materials. Detection levels are then defined by a diameter to depth ratio; where 'diameter' is the hole diameter and 'depth' is the maximum subsurface distance from the inspection face at which the bottom of the hole can be detected, Ref. [34-43].

When a comparison is made of the results from different studies over a period of 10 years, a considerable degree of scatter is evident in the claims for detection levels, ratios of diameter/depth, in different materials, Ref. [34-48]. Inconsistencies can be attributed to variations in materials, heat-pulse energy or duration and the efficiency of detection systems.

Thermography is continually improving, but a point has been reached where the best techniques available need to be evaluated under representative conditions to define their true capabilities.

34.16 X-ray radiography

34.16.1 Introduction

34.16.1.1 General

X-ray radiography is non-contact, except when using contrast-enhancing media, and can be applied in the laboratory, in production and in-service. It is a long-established means of inspecting components and structures. Traditionally, the image was recorded on photographic film, giving high resolution and good contrast, but was rather time consuming. Film is being replaced by electronic imaging, enabling real-time viewing and image manipulation or processing.

34.16.1.2 Advantages of radiography

The advantages of film X-ray radiography include:

- The ability to image the internal structure and defects within a subject, be it a laminate, sandwich panel or assembled structure.
- Differentiation of material phases with differing densities or thicknesses.
- Clear detection of the presence and orientation of individual components, e.g. honeycomb core, potting compound, fasteners and solid contaminants or foreign bodies.
- Reasonable detection of resin enrichment, porosity, prepreg gaps, fibre alignment and lay-up order in composites.

For CFRP composites, low X-ray tube voltages (eV) need to be used, due to the low atomic numbers of carbon and hydrocarbon matrices, i.e. they have a low X-ray stopping ability.

34.16.1.3 Limitations

The limitations of film X-radiography on polymer composites include:

- Film X-rays provide a two-dimensional image (shadowgraph) through the subject, therefore the presence of an anomaly can be detected but the location with respect to depth is unknown.
- X-rays cannot detect disbonds, delaminations and air gaps when these defects are normal to the irradiation direction, e.g. when inspecting most laminates and sandwich panels. The amount of contrast produced by any near-two-dimensional defect is highly dependant upon its orientation with respect to the X-ray beam.
- Film X-rays are time consuming, both in exposure and film processing, and cannot provide real-time imaging. Therefore they are deemed costly for many applications.

The advent of digital radiography and computer tomography go some way to overcoming these limitations, [See: 34.17].

The use of contrast-enhancing media, such as tetrabromoethane, TBE, for the detection of near-surface defects, e.g. cracks and delaminations, is considered relevant for test purposes only. Penetration of the laminate is needed, albeit small, to enable entry of TBE. This contaminates the composite and the inspection becomes a contact process.

34.16.2 X-ray techniques

34.16.2.1 General

There are various ways in which X-radiography can be undertaken, including:

- Basic radiography,
- Microfocus,
- Backscattering,
- Scattering And Refraction,
- β- backscattering.

34.16.2.2 Basic radiography

This is the traditional technique where variations in radiation absorption by different constituents produced a shadow image through the subject material or component.

Excellent contrast can be achieved between the features within the object, provided that the diverging X-ray beam is controlled correctly. The image characteristics are determined by the:

- Material density,
- Material thickness,
- Applied electron voltage (X-ray tube eV), and
- Exposure time.

It is a powerful tool for non-destructive testing of metal, polymer composite and multi-material structures.

34.16.2.3 X-ray microfocus

This is similar to conventional X-ray inspection, but a very narrow beam of X-rays is used to examine the local microstructure of materials. The technique can resolve very small defects. Some applications are the examination of carbon-carbon and CMC.

34.16.2.4 Backscattering

This relies on the Compton effect. A finely collimated beam of X-rays is directed through a material at an oblique angle to the surface. Some of the photons are scattered back through the surface. A finely collimated detector can then be used to measure the radiation which is coming from a small volume of material at the intersection of transmitted and scattered radiation paths, Ref. [34-52]. This is in effect a single-sided method, with access to the backface not required.

The backscattering technique has been used for thickness and density measurement of carbon-carbon materials, Ref. [34-52]. C-scan images can also be constructed.

34.16.2.5 X-ray scattering and refraction

Various modifications to traditional X-ray inspection are appearing, based on scattering and refraction behaviours, including:

- Scanning diffractometry, Ref. [34-53].
- Scanning topometry, Ref. [34-53].
- Diffraction scanning microscopy, Ref. [34-53].
- X-ray refractometry, Ref. [34-53], [34-54].

34.16.2.6 β- backscattering

This is a similar technique to X-ray scattering, but using electrons.

34.16.3 Digital radiographic system developments

34.16.3.1 General

The advent of low-cost computing power and charge-coupled device CCD cameras has enabled film photography to be superseded by digital systems, Ref. [34-55]. The quality of the viewable X-ray image depends on the radiographic equipment and digital data system.

The major X-ray systems in current use are shown in Figure 34.16.1, Ref. [34-55]. Each system provides different imaging characteristics.

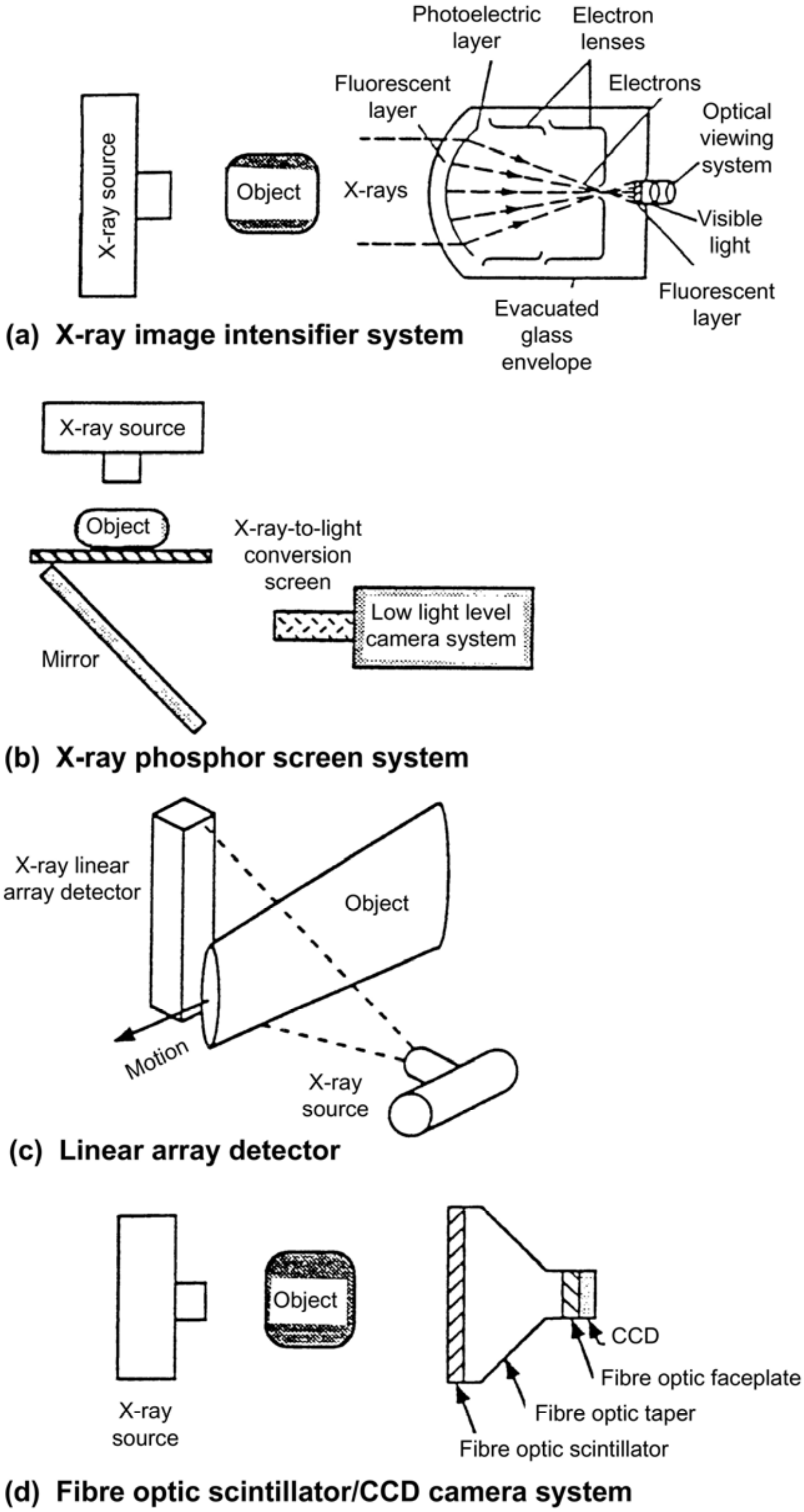


Figure 34.16-1 - Typical digital radiography systems

34.16.3.2 X-ray image intensifier

This is widely found in medical and industrial applications. Typical vacuum tube systems obtain their gain value (x 30 to x 10000) by the use of an input X-ray-to-light phosphor screen that converts X-ray imaging photons into light photons. The light photons are captured by a photocathode and converted to electrons. These are accelerated to many thousand eV and are electrostatically focused onto an output phosphor. Here the electrons are converted back into light photons to provide an optical image corresponding to the impinging X-rays. A lens is then attached to this output phosphor, and a television camera is used to collect the optical image. The intensification is not solely electronic, but can include a reduction in area; whereby the electrons from a large-area input screen are focused on a small output screen.

34.16.3.3 X-ray phosphor/camera system

In some cases image intensification is not necessary, which provides more flexibility. The X-ray phosphor can be removed from the vacuum tube and used directly on or shaped over the object. The glass window that absorbs much of the low X-ray energy imaging photons is no longer needed, so higher contrast images can be obtained. An image 'isocon' camera is typically used in this system.

34.16.3.4 Linear array scanner

A fanned X-ray beam is scanned across the object, and the emerging X-rays are collected by a linear array detector. The collected data is built up into an image by a computer. The disadvantages of this system include the:

- Long image-acquisition times,
- Low X-ray beam utilisation efficiency, resulting in high X-ray tube loading.

In most systems the detector consists of an X-ray phosphor in direct contact with a semiconductor photodiode array. The crystals are diced into thick, highly X-ray-absorbing pieces and become discrete pixels in the system.

An advantage of linear array systems is that a collimated fan beam and detection system has significantly reduced X-ray scatter compared with non-collimated beams, so producing very accurate images; hence its use in CT computerised tomography systems.

34.16.3.5 Fibre optic CCD system

Further developments of X-ray phosphor technology and CCD quality have provided an opportunity to custom-design an X-ray inspection system; such as the FOS CCD camera, Ref. [34-56].

A fibre-optic scintillator is in many ways equivalent to a fibre-optic faceplate window except that luminescent glass fibres are used as the core material. Core diameters in these faceplates are usually between 10 µm and 20 µm. When exposed to X-rays, scintillation within each core is channelled to the face of the plate. This provides a very sharp image plane that can be directly bonded to a CCD array. The FOS/CCD system offers high X-ray absorption, improved image acquisition time and X-ray beam utilisation over the linear array because the FOS can be made thick for high X-ray absorption efficiency. As the CCD is a solid-state device, it is not prone to geometric distortions and shading problems that are inherent in tube systems. As CCD chips become larger and contain higher pixel densities, field-of-view limitations of the FOS/CCD system become less important. A FOS/CCD system is lightweight and rugged and can easily be built into a scanning system for inspection of large aerospace assemblies.

34.16.4 Defect resolution

Like ultrasonics, X-ray techniques are evolving to make them more appropriate to inspection activities. The main developments centre on:

- Single-sided inspection using backscattering techniques, as needed for assembled structures.
- High-resolution methods for ceramic matrix and carbon-carbon composites, and for individual components.

34.17 Computer tomography

34.17.1 Introduction

Computer tomography is a non-contact method that can be used in the laboratory and in production.

Three-dimensional X-ray imaging is possible using computer-aided tomography, CAT, which builds up successive cross-sections of the component, Ref. [34-10], [34-57]. This method is also described as computer tomography, CT.

Initially a medical technique, it has been used to examine high-value or critical items with complex materials and geometries. The equipment is large, non-portable and expensive, but can produce excellent results with components otherwise difficult to inspect reliably. It is also applicable to structures containing a variety of material types, e.g.:

- Metal matrix composites, Ref. [34-56],
- Ceramic-based materials, Ref. [34-58],
- Rocket motor or missile parts,
- Turbine blades, and
- Highly-contoured carbon fibre based composite components.

34.17.2 Basic technique

A thin slice of radiation is passed through an object and the transmitted intensity is measured by a detector array, Ref. [34-59]. The radiation levels are typically from 10 keV to 40 keV for low-energy radiation or from 80 keV to 120 keV for high-energy, Ref. [34-57].

The object is rotated in order to provide views from many directions. The data is then processed by computer to derive a two-dimensional X-ray attenuation map of the slice, corresponding to volume elements within the interior of the object. If multiple slices are taken over the entire part, a complete three-dimensional reconstruction can be made. The computer enables this three-dimensional map to be examined in a number of ways (spatial analysis). A CT facility is shown in Figure 34.17.1, Ref. [34-60].

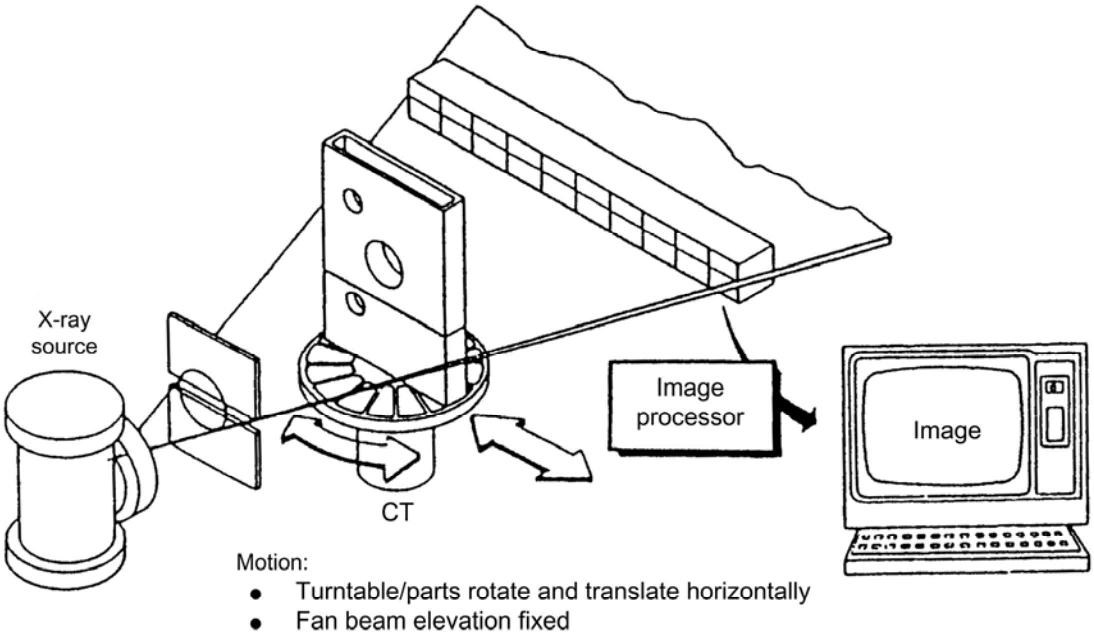


Figure 34.17-1 - Computer tomography facility

The 2D- and 3D-image reconstruction overcome two of the main deficiencies of conventional radiography by defining the location (depth) of a defect and visualising defects near-normal to the X-rays, as in planar delaminations, Ref. [34-61].

CT can be applied to quality acceptance inspection, product development and failure analysis. It provides accurate quantitative data on material density distribution, presence of constituent parts and dimensions.

34.17.3 Attributes of CT scanning

34.17.3.1 General

The principal attribute of CT is the detailed internal examination of discrete, complex assemblies and subcomponents. Some practical examples of this include:

- Composite sandwich wing leading edges and airfoil sections, Ref. [34-56], [34-60], [34-62].
- Potted transformers, Ref. [34-59].
- CFRP 'J' and 'Z'-stiffeners, Ref. [34-60].
- Bonded 'T'-spars, Ref. [34-62].
- Metal castings, Ref. [34-59].
- Titanium components made by superplastic forming and diffusion bonding (SPF-DB), Ref. [34-63].
- Helicopter rotor blades, Ref. [34-64].

An indication of the quantitative accuracy of CT scanning can be stated as:

- Measurement of material density variations in the range 0.1% to 1.0%, Ref. [34-63].
- Typical measurement accuracy in the range 0.05 mm to 0.25 mm (50 μm to 250 μm), for components 0.05 m to 0.5 m in size.

The availability of a technique which can conduct non-destructive 'sectioning' of complex components, as opposed to the alternative destructive examination has important implications in product development, Ref. [34-59].

Reduced development time can result in significant initial cost savings and a reduced risk of poor designs being manufactured. This can result in large savings over the lifetime of a programme.

The ability to detect 'volumetric defects' by a non-invasive inspection technique enables a range of defects to be detected, including those of primary concern in composites, i.e.:

- Delaminations,
- Core-to-skin disbonds,
- Misplaced materials, and
- Foreign bodies

34.17.3.2 Limitations

The fundamental limitations of CT are:

- Expensive equipment.
- Not portable.
- Limitation of component size; usually less than 1 m, but typically 500 mm.

Facilities for inspecting large structures, up to 1.8 m and eventually 3.8 m in diameter have been reported. These are intended to be used for the inspection of solid rocket motors, Ref. [34-65].

34.17.4 Micro-tomography

With the right equipment, CT can be applied to the examination of material microstructures with spatial resolution down to 20μm; termed non-invasive microscopy, Ref. [34-63], [34-66]. The pixel size set by the detector determines the level of resolution which can be achieved.

This technique can be useful for examining complex materials such as ceramic matrix composites, Ref. [34-58], or assemblies for reusable space vehicles, such as C-SiC air intake ramps.

34.18 Eddy currents

34.18.1 Introduction

Eddy current techniques can be classed as either contact (by hand) or non-contact, when automated scanning is used. It can be applied in the laboratory, in production and in-service.

In this technique current loop paths, eddy currents, are induced in a conductive material, by passing an alternating current through a coil-containing probe, Ref. [34-67]. The probe is placed on or near the conductive surface of the component. Defects are detected by monitoring disturbances in the induced magnetic fields.

It is primarily used for conductivity, crack and corrosion detection in metals, and is widely used for manual or automated aircraft inspection, Ref. [34-69]. Eddy current techniques for metals work in the 10 kHz to 1 MHz range. It is particularly appropriate for examining bolt and rivet holes to locate cracks.

Sufficient electrical conductivity for eddy currents to be induced can also be found in CFRP (> 40% volume fibre), carbon-carbon, Ref. [34-70], C-SiC and metal matrix composites. Practical inspection systems are therefore feasible, Ref. [34-10]. Automated inspection has been carried out of solid rocket motor casings, forgings and segments, Ref. [34-68], in conjunction with electromagnetic acoustic transducers (EMAT).

34.18.2 Developments in eddy current techniques

34.18.2.1 General

Computerised eddy current scanning systems are suitable for the inspection of composite constructions. Some observations concerning eddy currents as applied to CFRP are, Ref. [34-71]:

- High frequencies are needed, Ref. [34-72], e.g.:
  - 10 MHz to 500 MHz for UD CFRP, and
  - 1 MHz to 30 MHz for woven CFRP laminates.
- Eddy currents are affected by laminate edges, so peripheral defects can be difficult to resolve.
- Coil probe design is in important in relation to the laminate construction. The number of coils, their shape (horseshoe, pancake elliptical) and width should be optimised in relation to the laminate thickness, Ref. [34-70], [34-72].
- Eddy current probes can be operated to produce C-scan images.
- Induced eddy currents tend to be concentrated near the surface of the material adjacent to the coil, although some control of the penetration depth can be achieved by varying the characteristics of the applied signal.

Eddy current techniques look promising for the locating of:

- Broken carbon fibres.
- Changes in carbon fibre volume fraction.
- Changes in carbon fibre lay-up and orientation, Ref. [34-10].
- Detecting disbonds between metal sheet and composite laminate, Ref. [34-73].

34.18.2.2 Limitations

Delaminations are difficult to detect unless the interlaminar damage is fairly severe (ply separation). The detection of delaminations is improved if:

- More than 20% of interfacial ply contact is lost, or
- Fibres have been broken, to at least 8% of the thickness, Ref. [34-72].

Table 34.18.1 gives the results of a test programme to compare the detection capability of an eddy current technique with that of ultrasonics, Ref. [34-72].

Table 34.18-1 - Detection of damage by eddy current and ultrasonics

Construction	Impact Energy (J)	Eddy Current Image	Ultrasonic Image Spot Diameter (mm)	Visible Damage
Sandwich: 0.5 mm skins 6.8 mm core	0.69 1.33 2.39	Not visible Halo Halo	11 - 15 16 - 20 20 - 26	Not visible Dent, BF Hole
Sandwich: 2.0 mm skins 6.8 mm core	2.83 5.59 9.12 17.5	BV halo Halo Halo Halo	18 - 22 37 33 41	Not visible Dent Dent Hole
Laminate: 7.2 mm CFRP	5.72 10.7 27.3 37.5 41.7	Not visible BV dark spot Dark spot, halo Halo Light spot	15 22 29 33 41	BV dent Dent, CM Dent, CM Dent, CM Dent, CM

Key: Skins - woven CFRP; Core - aluminium.
BV - Barely visible; BF - Broken fibres; CM - Cracked matrix.

34.18.2.3 Future developments

Some of the developments that increase the relevance of eddy current testing to both metals and composites include:

- Scanning systems for assembled structures with multiple materials.
- Improved defect detection for CFRP and carbon-carbon.
- Condition monitoring of alloy structures and fasteners.

34.19 Other NDT techniques

34.19.1 General

There are other techniques which can be used for inspection, but they are not common and tend to have specialised roles. The techniques described are:

- Microwave
- Heat and photosensitive agents
- Electrical impedance

- Acoustic microscopy
- Nuclear magnetic resonance (NMR)
- Neutron radiography
- Gamma radiography
- D sight - Diffracto sight

34.19.2 Microwave

Microwave techniques are non-contact and can be applied in the laboratory and in production.

Any disruption in microwave through-transmission in composites indicates the presence of discontinuities, or variations in dielectric material properties, Ref. [34-10]. Changes in amplitude or phase can be observed, enabling the detection of defects, thickness and degree of resin cure.

The technique has been applied to filament-wound structures, and also for single-sided examination of sandwich panels, Ref. [34-74]. Typical defects, such as delaminations and skin-to-core disbonds, can be detected when the equipment is scanned over the surface and operated in the X-band range with frequencies between 8.2 GHz and 12.4 Hz and at 24 GHz.

34.19.3 Heat and photosensitive agents

Techniques using thermochromic and photochromic substances are contact methods and suitable for use in the laboratory and in-service.

Thermochromic and photochromic substances show changes in colour or light characteristics depending on their exposure time and temperature history. They can be applied to surfaces of components as paints, part of a crack detection system or combined with the resin matrix.

34.19.4 Electrical impedance

Impedance techniques are contact methods suitable for use in the laboratory.

For CFRP materials, electrical impedance measurements can provide a means of indicating the presence of delaminations, microcracks and moisture absorption, Ref. [34-76]. Initial work involved the immersion of the composite in an electrolyte, so it has not yet been demonstrated as a practical inspection method.

34.19.5 Acoustic microscopy

These techniques are contact methods, suitable for use in the laboratory or in production. They are principally used for achieving high-resolution imaging of small areas. The various techniques are, Ref. [34-76]:

- Scanning laser acoustic microscope (SLAM), which provides real-time images in transmission mode throughout the thickness of the object.
- Scanning acoustic microscope (SAM), which provides high-resolution images of surface and near-surface when raster scanned and operated in reflection mode.
- C-SAM, a variant of SAM, which provides good or moderate penetration in pulse-echo mode and images at a specific depth.

These techniques are only appropriate to polymer composite structures under exceptional circumstances, but are more likely to be of value for ceramic matrix composites.

34.19.6 Nuclear magnetic resonance (NMR)

NMR is a non-contact method that can be applied in the laboratory and in production.

It is an analytical technique commonly used to assess the chemistry of materials. However, in some specific circumstances NMR can be applied to materials problems, such as:

- Detection of moisture in composites.
- Curing and thickness of adhesive bonds.
- Assessment of the adhesive liner between propellant and insulator in solid rocket motors, Ref. [34-77].

34.19.7 Neutron radiography

Neutron radiography is a non-contact method suitable for use in the laboratory and in production. It is similar to the X-ray technique.

Images are formed on film by neutrons from a radiation source. Certain elements show high neutron absorption, such as boron, hydrogen, lithium and cadmium, whilst others are virtually transparent. Application examples include:

- Composites:
  - fibre alignment,
  - moisture,
  - resin or adhesive build-up.
- Bonded joints:
  - bond-line corrosion.

Neutron radiation computer tomography can also be undertaken on gas turbine engine blades and aluminium forgings, Ref. [34-78].

34.19.8 Gamma radiography

Using a radioactive source, the gamma rays pass through the sample and are detected on film or by meters. Changes in the absorption characteristics because of defects, density or thickness variations in composites are detectable.

34.19.9 D sight - Diffracto sight

This is a non-contact method suitable for use in the laboratory, in production and in-service.

D sight is an optical technology in the visible spectrum, Ref. [34-79], [34-80]. It uses a CCD camera, a white light source mounted slightly below the camera lens, and a retro-reflective screen. The surface to be inspected is positioned between the camera-light and the retro-reflective screen, as shown in Figure 34.19.1, Ref. [34-79].

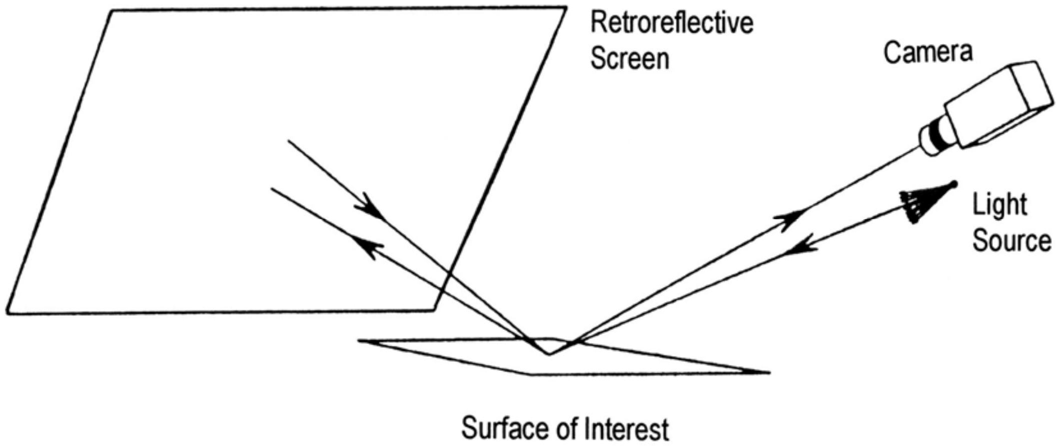


Figure 34.19-1 - D sight - Diffracto sight schematic

The retro-reflective screen is the critical optical element in D sight. It returns light falling on its surface efficiently back along the incident direction; unlike a mirror or a projection screen. The light returned by the retro-reflector is slightly dispersed owing to the physical and optical characteristics of the micro-beads making up the screen surface, but most of the light returns within a 1° cone.

When the surface to be inspected is illuminated by the light source at a low grazing angle, any local curvature variations on this surface act as a focus or disperse the light onto the retro-reflective screen. A distinct pattern of light intensities then describes the viewed surface and highlights any anomalies, such as those associated with impact damage and near-surface delaminations.

The technique has been developed for inspecting aircraft and is limited to detecting surface and near-surface damage.

34.20 NDT for adhesive bond defects

34.20.1 General

The main classes of defect that occur in adhesive joints are, Ref. [34-81]:

- Complete disbonds, voids or porosity in the adhesive layer.
- Poor adhesion, i.e. a weak interface between the adhesive layer and one or both adherends.
- Poor cohesion, i.e. a weak adhesive layer.

34.20.2 Disbonds, voids and porosity

Some disbonds are essentially large, flat voids that can be caused by a complete lack of adhesive or by the adhesive being applied unevenly to one adherend only. Disbonds can also be caused by the presence of grease or other contaminants on an adherend, e.g. release agent. In these cases the surfaces of the defect are generally in close proximity or touching, which makes their detection difficult. Disbonds can also occur as a result of impact or environmental degradation after manufacture.

Porosity is caused by volatiles or air trapped in the adhesive. Inadequate drying of composites can cause absorbed moisture to vaporise during the adhesive cure cycle and produce bubbles in the adhesive.

Such defects can seriously impair bond performance but can be detected by:

- Conventional water-coupled ultrasonics: Sensitive to disbonds, delaminations and porosity.
- Ultrasonic bond testers: For disbonds and delaminations, but not porosity:
  - 100 kHz to 1 MHz.
  - Bondascope, which measures magnitude and phase of the ultrasonic impedance.
  - Fokker bond tester II, using a spectroscopic approach monitoring frequency and amplitude changes in the first two modes of through-thickness vibration.
- Sonic vibration and mechanical impedance: For large defects.
- Thermography, Ref. [34-15], [34-45], [34-84], holography and shearography: For the larger near-surface, air-gap defects.
- Eddy currents: Possible for disbonds between composite and metal, Ref. [34-85].

34.20.3 Cohesion and adhesion

A weak adhesive layer, giving poor cohesive properties, can result from incorrect mixing or formulation of two-part adhesives, or inadequate curing. Since one-part film adhesives also suffer from these problems, miscuring seems the most likely cause. There is some evidence to suggest that the Fokker bond tester Mk II can detect poor cohesion under some circumstances.

Poor adhesion is likely to be caused by surface contamination or incorrect surface preparation prior to bonding. A further possibility is contamination by release agent on peel plies. High resolution is needed within a thin layer of about 1 µm thick, which implies the use of ultrasonics at high frequencies (20 MHz to 200 MHz). This leads to severe signal attenuation problems.

The inspection of adhesive bonds for poor cohesion or adhesion remains an area of weakness within NDT techniques, and no reliable method exists currently. As the desire to detect these anomalies continues, it remains a motivation for new developments, Ref. [34-82], [34-83]. Until a technique is proven, adhesion and cohesion problems are best eliminated by precise process monitoring, with control test samples made alongside production items.

34.21 NDT for space applications

34.21.1 Introduction

The number of NDT techniques available has increased considerably within 20 years. They are still evolving in the areas of:

- Non-contact inspection.
- Digital signal manipulation and image presentation.
- Automation for rapid inspection and real-time imaging.

It is widely accepted that more than one technique is usually necessary to detect all possible defects or anomalies that can occur in composite constructions. However, critical defect types are usually identified for a particular design and method of construction and this enables NDT techniques to be selected by their detection capabilities.

The detection of gross defects remains a main requirement for damage tolerant designs. Such defects can be classified as missing or misaligned material, delaminations, disbonds and impact damage.

Conventional C-scan ultrasonics and X-ray examination remain the most powerful techniques for comprehensive defect and anomaly detection.

34.21.2 Developments in inspection

The development of inspection techniques has gone beyond the stage of providing a means of detecting defects in specific materials, such as CFRP. Techniques should be adaptable in order to inspect a range of materials, complex subassemblies and integrated structures. Some examples of developments that are aimed at assisting inspection within space programmes include:

- Sandwich constructions by non-contact techniques, Ref. [34-21], [34-27], [34-30], [34-32], [34-33], [34-74].
- Assembled structures by non-contact techniques, Ref. [34-9], [34-12], [34-15], [34-16], [34-37], [34-41], [34-43], [34-47], [34-64], [34-69], [34-79], [34-80], [34-88].
- Detection of impact delaminations in polymer composites, Ref. [34-8], [34-38], [34-46], [34-61], [34-75], [34-76].
- Integrity of adhesive bonds, Ref. [34-45], [34-81], [34-82], [34-83], [34-84], [34-85].
- Rocket motor casings, Ref. [34-65], [34-68], [34-77].
- Integrity control of high-temperature structures, Ref. [34-90].
- Air-coupled ultrasonic inspection, Ref. [34-21].
- Microstructural examination of ceramic composites, Ref. [34-19], [34-51], [34-59], [34-90].
- Coated carbon-carbon components by eddy currents, ultrasonics, thermography and β-backscattering, Ref. [34-52], [34-86].
- NDE of titanium MMC rings by ultrasonics and computer tomography, Ref. [34-87].

[See: Chapter 76 for integrity control aspects of high-temperature structures]

34.21.3 Summary of NDT techniques

Table 34.21.1 gives a summary of the relevance of the main NDT technologies to the inspection of polymer composite structures.

[See: Chapter 80 for NDT of metal- and ceramic-based materials and assemblies]

Table 34.21-1 - Summary of NDT techniques for polymer composite space structures

Technique	Comments
Conventional C-scan ultrasonics [C]	Good all-round capabilities. Good for flat or curved composite panels and sandwich constructions.
X-ray techniques [NC]	Good all round capabilities. Good for components, sandwich constructions and assembled structures.
Holography [NC]	Good capabilities for inspecting assembled structures. Applicable to sandwich constructions, e.g. antennas.
Acoustic Emission [C]	Of very restricted use, e.g. to pressure vessels during proof loadings.
Laser Ultrasonics [NC]	Requires further development. Possible detection of surface or near-surface defects, e.g. delaminations and disbands. Applicable to rapid inspection of assembled structures.
Laser Shearography [NC]	Probable detection of surface or near-surface defects, e.g. delaminations and disbonds. Applicable to rapid inspection of assembled structures.
Thermography [NC]	Probable detection of surface or near-surface defects, e.g. delaminations and disbonds. Applicable to rapid inspection of assembled structures.
Computer Tomography [NC]	Good capabilities for defect detection in discrete components or subassemblies, e.g. CMC and C-C.
Eddy Currents [C or NC]	Requires further development for application to CFRP and C-C. Established technique for inspecting metal constructions. Applicable to inspection of assembled structures.

Key: [C] - contact method; [NC] - noncontact method.

34.22 Rupture tests

34.22.1 Application

34.22.1.1 General

Since many load cases have low margins of safety, it is not possible to perform rupture tests under all circumstances.

34.22.1.2 Envelope load case

An envelope load case is defined and the test is conducted up to rupture, where:

- The envelope should be defined at an early stage of the project
- This envelope load becomes a dimensioning load case, which can lead to mass penalties
- The interaction between loads is not well known near rupture

34.22.1.3 Single load cases

A single load case (the one which has either lowest margin of safety or highest degree of uncertainty of analysis) is defined for rupture, where:

- It does not give the real margins of safety of the structure.
- The test is done at the end of the test sequence but the structure becomes unavailable for further eventual tests.

34.22.1.4 No rupture test

When a no rupture test is done, it should be selected at project level, since the selection takes into account:

- Mass budgets,
- Degree of safety,
- Degree of ability to carry higher loads,
- Cost effects (number of qualification specimens).

For composite structures, rupture often occurs suddenly because no plasticity exists. Hence, it is difficult to know from a test how far the structure is from rupture. To improve the situation, the structure is instrument with:

- Strain gauges (many) to correlate models. The output enables reference to development rupture tests on smaller parts.
- Acoustic emission devices which can indicate of the internal state of damage.

34.22.1.5 Displacement measurement

To measure displacements under load, techniques such as holography, Ref. [34-33], shearography and EPSI electronic speckle pattern interferometry are applicable.

34.23 Test related items

34.23.1 General

An overview of test related items is presented, which describes the influence of each item of the test and their relationship to each other.

34.23.2 Major items

The main items that can be identified are:

- Failure modes of the structure or any part of the structure, e.g.:
    - matrix cracking,
    - rupture of fibres,
    - buckling.
- Controls at any phase of the test of the structure or any part of the structure, e.g.:
    - deformation measurement,
    - visual inspection,
    - acoustic emission.
- Relevant areas of the structure, e.g.:
    - locally loaded areas,
    - stiffener edges,
    - holes.
- Type of loads the structure can possibly experience during its life or during tests, e.g.:
    - static,
    - dynamic,
    - acoustic,
    - impact loads.
- Type of tests applied during all phases of the product, e.g.:
- development,
    - qualification test.

The interrelationship between these items is shown in Table 34.23.1.

Table 34.23-1 - Interrelationship of items influencing the test

	Failure Modes	Controls	Areas	Loads	Tests
Failure Modes	X	X	X	X	X
Controls	X	O	X	O	O
Areas	X	X	O	X	X
Loads	X	O	X	X	X
Tests	X	O	X	X	O

0: Not applicable

34.23.3 Qualification tests

34.23.3.1 General guidelines

Some general guidelines for qualified testing are given. The interrelationship of the effects is indicated in the tables cited.

- Effects of local mode of failure on the global behaviour of the structure are assessed; as given in Table 34.23.2.
- Test instrumentation enables the detection of any mode of failure during tests; as given in Table 34.23.3.
- Local analysis lists the possible modes of failure for each area of the structure. Critical points are studied during tests; as given in Table 34.23.4.
- Effects of each type of loads as well as combinations are assessed; associated modes of failure are listed; as given in Table 34.23.5.
- Effect of each mode of failure are analysed through the appropriate stages during the life of the project; as given in Table 34.23.6.
- Each particular area of the structure is instrumented with respect to its probable mode of failure; as given in Table 34.23.7.
- Each particular area is analysed and, if necessary, tested with respect to load presumed to be dangerous; as given in Table 34.23.8.
- Particular areas are tested at appropriate stages of the project; as given in Table 34.23.9.
- In relevant cases, load combinations are used for tests; as given in Table 34.23.10.
- Relevant types of load cases are tested at relevant steps of the project; as given in Table 34.23.11.

Table 34.23-2 - Effects of local failure modes on global behaviour of the structure

Local †	**Global**								
	Matrix Cracking	Fibre Rupture	Cracks	Delamination	Rigidity Loss	Melting	Debonding	Buckling	Rupture
Matrix Cracking	X	X			X				
Fibre Rupture		DX	X		X			X	X
Cracks	X	X	PX						X
Delamination				PX	X		X	X	X
Rigidity Loss					X			X	
Melting									
Debonding							PX	X	X
Buckling	X	X		X	X				X
Rupture									

Key: P Propagation D Dispersion

† If local phenomena arise at given load, effects should be analysed and taken into account. The analysis should be based on development tests and verified during qualification tests.

Table 34.23-3 - Detection of techniques to various failures occurring during test

Failure	Test Means †									
	Stress Meas.	Def. Meas.	Visual Inspec.	Endoscopy	X-ray	C-Scan	Thermo-graphy	AE.	Noise	Residual Stress
Matrix Cracking	✓m	☑			Destr.			✓		Destr. ✓
Fibre Rupture	✓m	☑			Destr.			✓		Destr. ✓
Cracks			✓	✓		✓	✓			
Delamination			☑	☑		✓	✓		✓	
Rigidity Loss	✓m	✓								
Melting			✓	✓						
Debonding	☑	☑				✓	✓			
Buckling	✓								✓	
Burst	✓	✓	✓	✓				✓		
Rupture	✓	✓	✓	✓				✓		

Key: ☑ Only if nominal or damage is external

✓ Detected — Matrix cracking, fibre rupture, buckling, rigidity

m Loss of Modulus — Loss detected if station gauges are well located

† The relevant instrumentation has to be used in order to detect such phenomena.

Table 34.23-4 - Interrelation between failure modes and their possible locations

Failure	**Location †**							
	Running Section	Local Load	Sandwich Edge	Stiffener Edge	Frame	Hole	Reinforced Door	Connection Parts
Matrix Cracking	✓	✓				✓	✓	✓
Fibre Rupture	✓	✓				✓	✓	✓
Cracks	✓	✓		✓		✓	✓	✓
Delamination		✓	✓	✓		✓		✓
Rigidity Loss	✓					✓	✓	✓
Melting								
Debonding		✓	✓	✓	✓			
Buckling	✓				✓	✓	✓	
Burst								
Rupture								

†: Possible locations of failure should be specifically instrumented to allow the study of the phenomena.

Table 34.23-5 - Interrelation between type of loading and possible failure modes

Failure	**Load †**							
	Residual Stresses	Acoustic	Static	Thermal	Dynamic	Fatigue	Impacts	Load History
Matrix Cracking	✓		✓	✓	✓	✓	✓	✓
Fibre Rupture			✓				✓	✓
Cracks	✓	✓	✓					
Delamination	✓	✓	✓					
Rigidity Loss			✓	✓	✓	✓	✓	
Melting				✓				
Debonding		✓	✓	✓	✓	✓	✓	✓
Buckling			✓	✓	✓		✓	✓
Burst			✓	✓			✓	
Rupture			✓	✓			✓	✓

†: Effects of these loads should be assessed and verified during tests.

Table 34.23-6 - Failure modes which have to be covered by the specific tests

Failure	**Test**					
	DVPT Tests	Qualif. Tests	Rupture Tests	Procurement Tests	Fabrication Tests	Proof* Tests
Matrix Cracking	☑	✓				✓■
Fibre Rupture	☑	✓		✓	✓	
Cracks	☑					✓
Delamination	☑	✓			✓	
Rigidity Loss		✓				Useless
Melting	☑					
Debonding	☑	✓				✓
Buckling	☑	✓				
Burst						
Rupture		✓				✓

Key: ■ Proof tests should not develop matrix cracking and if it is the case, the effect should be taken into account in the dimensioning of the structure and in the damage tolerance analysis.

☑ Should be studied during development.

* Proof test necessity should be discussed.

Table 34.23-7 - Appropriate instrumentation of particular areas of the structure

Instrumentation	**Location**						
	Pressurised Section	Local Load	Stiffener Edge	Frames	Holes	Reinforced Doors	Connect Parts
Stress Measurement	✓	✓	✓	✓	✓	✓	
Definition Measurement	✓					✓	
Visual Impact		✓	✓		✓		✓
Endoscopy		✓	✓				✓
X-ray	✓		✓	✓	✓	✓	
C-Scan	✓		✓	✓	✓	✓	
Thermography	✓						✓
Acoustic Emission	✓				✓		
Moiré Method	✓						
Residual Stresses	✓						

Table 34.23-8 - Interrelation between particular areas of the structures and possible dangerous loads

Location	Load							
	Residual Stresses	AE	Static	Thermal	Dynamic	Fatigue	Impact	Load History
Running Section		✓	✓	✓	✓		✓	✓
Local Load	✓		✓	✓	✓	✓		✓
Sandwich Edge		✓	✓	✓				
Stiffener Edge	✓	✓	✓	✓		✓	✓	
Frames			✓	✓	✓			
Holes	✓	✓	✓	✓		✓		
Reinforced Doors			✓	✓	✓			
Connection Parts		✓	✓	✓	✓	✓		✓

Table 34.23-9 - Testing of particular areas of the structure

Location	Test					
	DVPT Tests	Qualif. Tests	Rupture Tests	Procurement Tests	Fabrication Tests	Proof Tests
Running Section		✓			✓	
Local Load	✓	If possible				
Sandwich Edge	✓	✓			✓	
Stiffener Edge	✓	✓			✓	
Frames		✓				
Holes	✓	✓		✓		
Reinforced Doors		✓				
Connection Parts	✓	✓				

Table 34.23-10 - Load combinations for the relevant cases

Load	**Load**						
	Residual Stresses	Acoustic	Static	Thermal	Dynamic	Fatigue	Impact
Residual Stresses			✓	✓		✓	
Acoustic	✓		✓				
Static	✓	✓		✓			✓
Thermal			✓				
Dynamic							
Fatigue	✓						
Impact			✓				

Table 34.23-11 - Testing different load cases

Load	**Test**					
	DVPT Tests	Qualif. Tests	Rupture Tests	Procurement Tests	Fabrication Tests	Proof Tests
Residual Stresses	✓				✓	
Acoustic	✓	If MS low				
Static	✓	✓	If any	✓	✓	If any
Thermal	✓	If MS low				
Dynamic		✓				If any
Fatigue	✓					
Impact	✓					
Load History	✓	In some sense				

Key: MS: Margin of safety

34.24 Structural test reports

The guidelines indicate what can reasonably be expected to appear in a structural report.

The items included are:

- Title sheet:
  - distribution list,
  - issue,
  - date.
- Alteration sheet:
  - to include a summary of changes
- Contents list:
  - section number,
  - title,
  - issue number.
- Summary page
- References:
  - applicable papers.
- Introduction:
  - structure,
  - purpose,
  - scope,
  - project.
- Structure description
- Design requirements:
  - general,
  - specific,
  - unique to project.
- Materials list:
  - property values,
  - sources,
  - qualities.
- Analysis methods, for those used in the report.
- FEM model description: This should be clear and unambiguous. If necessary, break the model into major subassemblies. Use FEM grids and structure or component drawings together as superimposed or exploded views.

- Assumptions: Those made affecting the idealisations of the structure, e.g. load application, modelling, static and dynamic analysis.
- Results: Where computer results (e.g. FEM) are included, present only those that have actually been used, or appear in subsequent calculations.
- Analyses:
  - static,
  - dynamic.
- Reserve factors (R.F.) or margin of safety (M.S.): A summary includes:
  - drawing, part number or title,
  - material, type, specification number or grade,
  - loading case,
  - load, moment and stress with type, applied and allowable plus units,
  - actual R.F or M.S value,
  - any appropriate remarks,
  - location within report of associated calculation, i.e. section or page number.
- Strength and dynamic response: compliance statement and conclusion.
- Loading cases:
  - list,
  - descriptions.
  - numbering sequence
- Units: State those used in report; preferably the same throughout.
- Co-ordinate systems, e.g.:
  - orientation,
  - location of origins,
- Appendices or annexes.

34.25 Analysis and test documents

34.25.1.1 Damage tolerance

34.25.1.2 General

All potential fracture critical items, PFCI, are analysed, and the analysis documentation, as a minimum, contains the items detailed.

When testing is used in addition to analysis, the test method and results are also included.

34.25.1.3 Safe life items

A description of the item with identification of the material used with full details of the method of fabrication including lay-up, e.g. number of plies, their sequence and orientation; and a clear sketch of all assumed defects and damages.

- A description of the analysis and test performed. A reference to the stress report.
- The loading spectrum and how it has been derived.
- Material data and how they have been derived.
- Environmental conditions and their effects.
- In the demonstration of no growth behaviour, any low strain criterion used are fully described and justified with appropriate supporting test evidence.
- Demonstration that the interval between the time any damage becomes detectable to the time at which the extent of damage reaches the stated limits for the required residual strength capability is at least four times the scheduled inspection interval in the approved maintenance plan.
- Summary of the significant results.

34.25.1.4 Fail safe items

This includes all items under that definition and also includes items in which special design features have been incurporated and demonstrated to be satisfactory, in that they ensure crack arrest and are capable of sustaining limit loads in the damaged condition between regularly scheduled inspections. For all fail safe items:

- A description of the item; as per safe-life items,
- Failure modes assumed,
- Stress analysis with new loading distribution,
- Fatigue analysis of the most critical item,
- Summary of the significant results.

34.25.1.5 Contained items

A description of the containment analysis including:

- Velocity or energy of the item as it strikes the containing item (shell), with worst case sharpness assumption,
- Elastic or plastic shell deformation,
- Stresses in the shell, attachments, brackets, or other similar items during impact.

34.26 References

34.26.1 General

[34-1] C.N.E.S.
H SG 1 10 CNES, 10.06.1988

[34-2] J.D. Achenbach
'Introduction: The science and technology of NDE'
Chapter 1, Flight-vehicle materials, structures, and dynamics - assessment and future directions
Vol. 4 Tribological Materials and NDE
ASME Publication, 1992, ISBN 0-7918-0662-6

[34-3] I.M. Daniel
'NDE of composite materials'
Chapter 11, Flight-vehicle materials, structures, and dynamics - assessment and future directions
Vol. 4 Tribological Materials and NDE
ASME Publication, 1992, ISBN 0-7918-0662-6

[34-4] A.L. Siedl
'Inspection of composite structures - Parts 1 & 2'
Part 1, SAMPE Jnl, Vol.30, No. 4, July/August 1994, p38-44
Part 2, SAMPE Jnl, Vol.31, No. 1, January/February 1995, p42-48

[34-5] 0. Forli
'In-orbit in-service inspection'
Proceedings of the International Symposium on Space Applications of Advanced Structural Materials. March 1990
ESA SP-303, p157-162

[34-6] S.K. Burke et al
'Non-destructive characterisation of advanced composite materials'
Materials Forum (1994) 18, p85-109

[34-7] R. Smith & C. Hobbs
'The need for NDT of advanced materials'
14th International European SAMPE Conference, October 1993
ISBN 3-9520477-0-8, p25-34

[34-8] X.E. Gros
'Review of NDT techniques for detection of low energy impacts in carbon reinforcements'
SAMPE Journal, Vol.31, No. 2, March/April 1995, p29-34

[34-9] H. Tretout et al
'Review of advanced NDT methods for composites aerospace structures'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures '94, March 1994
ESA WPP-070, p629-634

[34-10] J. Summerscales, Editor
'Non-destructive testing of fibre-reinforced plastics composites - Volume 2'
Elsevier Applied Science, ISBN 1-85166-468-8, 1990

[34-11] B.S. Wong et al
'Ultrasonic testing of solid fiber-reinforced composite plates'
SAMPE Journal, Vol. 30, No. 6, November/December 1994
p36-40

[34-12] P. Cawley
'The rapid non-destructive inspection of large composite structures'
Composites, Vol. 25, No. 5, 1994, p351-357

[34-13] G.R. Tillack et al
'Inspection performance in ultrasonic testing'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures '94, March 1994
ESA WPP-070, p639-644

[34-14] G. Bennett et al
'Development of real time low cost high performance scanning systems for advanced ultrasonic imaging'
39th International SAMPE Symposium, April 11-14, 1994
p1344-1355

[34-15] N. Wood
'Large area composite inspection system (LACIS) - automation of traditional inspection techniques'
39th International SAMPE Symposium, April 11-14, 1994
p1375-1390

[34-16] 'Mighty MAUS'
Aerospace America, November 1991, p46-47

[34-17] Y. Bar-Cohen et al
'Composite material property non-destructive characterisation using obliquely insonified ultrasonic waves'
39th International SAMPE Symposium, April 11-14, 1994
p1316-1329

[34-18] N. Guo & P. Cawley
'Lamb waves for the NDE of composite laminates'
Review of Progress in Quantitative Non-destructive Evaluation
Volume 11B, 1992, Plenum Press, ISBN 0-306-44206-X
p1443-1450

[34-19] M. Bashyam & J.L. Rose
'Surface acoustic wave techniques for ceramic matrix composite materials characterization'
Review of Progress in Quantitative Non-destructive Evaluation
Volume 11B, 1992, Plenum Press, ISBN 0-306-44206-X

p1483-1490

[34-20] B.R. Tittmann
'Experimental techniques in ultrasonics for NDE and material characterization'
Chapter 3, Flight-Vehicle Materials, Structures, and Dynamics - Assessment and Future Directions
Vol. 4 Tribological Materials and NDE
ASME Publication, 1992, ISBN 0-7918-0662-6

[34-21] W.A. Grandia
'Advances in non-destructive testing non-contact ultrasonic inspection of composites'
39th International SAMPE Symposium, April 11-14, 1994
p1308-1315

[34-22] W. Sachse & M.R. Gorman
'Acoustic emission measurements of aerospace materials and structures'
Chapter 8, Flight-Vehicle Materials, Structures, and Dynamics - Assessment and Future Directions
Vol. 4 Tribological Materials and NDE
ASME Publication, 1992, ISBN 0-7918-0662-6

[34-23] G.M. Butyrin & A.V. Demin
'Non-destructive evaluation of density and porosity in porous materials'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures '94, March 1994
ESA WPP-070, p673-676

[34-24] J-P. Monchalin
'Laser-ultrasonics'
Chapter 4, Flight-Vehicle Materials, Structures, and Dynamics - Assessment and Future Directions
Vol. 4 Tribological Materials and NDE
ASME Publication, 1992, ISBN 0-7918-0662-6

[34-25] J-P. Monchalin
'Progress towards the application of laser-ultrasonics in industry'
Review of Progress in Quantitative Non-destructive Evaluation
Vol. 12A, p495-506, 1993, Plenum Press, ISBN 0-306-44483-6

[34-26] A.D.W. McKie & R.C. Addison
'Rapid inspection of composites using laser-based ultrasound'
Review of Progress in Quantitative Non-destructive Evaluation
Vol. 12A, p507-516, 1993, Plenum Press, ISBN 0-306-44483-6

[34-27] F.H. Chang et al
'Laser ultrasonic inspection of honeycomb aircraft structures'
Review of Progress in Quantitative Non-destructive Evaluation
Vol. 12A, p611-616, 1993, Plenum Press, ISBN 0-306-44483-6

[34-28] T.E. Drake
'Laser ultrasonic testing of aerospace materials'
39th International SAMPE Symposium, April 11-14, 1994
p725-739

[34-29] C.M. Scala et al
'Laser ultrasonics: emerging technology for aircraft NDE'
Non-Destructive Testing - Australia
Vol. 31, No. 4, July/August 1994, p90-92

[34-30] J.F. Clarady & M. Summers
'Electronic holography and shearography NDE for inspection of modern materials and structures'
Review of Progress in Quantitative Non-destructive Evaluation
Vol. 12A, p381-386, 1993, Plenum Press, ISBN 0-306-44483-6

[34-31] G. Di Chirico
'Optical methods for non-destructive inspection of structural components'
Testing of Metals for Structures, Naples, Italy, 29-31 May, 1990
p337-343

[34-32] I. Cabeza et al
'Thermal distortion measurement on antenna-reflectors by real-time holographic interferometry in ambient pressure'
Proceedings of the International Symposium on Environmental Testing for Space Programmes - Test Facilities & Methods
ESTEC, 26-29 June 1990, ESA SP-304, p403-407

[34-33] H.U. Frey
'Holography - A successful tool for distortion measurements on antennas under space simulation conditions'
Proceedings of the International Symposium on Environmental Testing for Space Programmes - Test Facilities & Methods
ESTEC, 26-29 June 1990, ESA SP-304, p395-402

[34-34] J.F. Clarady
'Electronic holographic NDE'
Review of Progress in Quantitative Non-destructive Evaluation
Volume 9, 1990, Plenum Press, p1031-1038

[34-35] M.V. Rao et al
'Dual vacuum stressing technique for holographic non-destructive testing of honeycomb sandwich panels'
NDT International, Vol. 23, No. 5, October 1990, p267-270

[34-36] T.E. Tay et al
'Application of holographic interferometry in the detection of flaws in composite plates'
Advanced Composites '93, International Conference on Advanced Composite Materials
Wollongong, Australia, 15-19 Feb, 1993, p683-686

[34-37] P. Ferraro et al
'Holographic interferometry as inspection technique on aerospace composite structure'
Proceedings of the International Symposium on Space Applications of Advanced Structural Materials. March 1990
ESA SP-303, p151-156

[34-38] G. Cavaccini et al
'Detection of impact damage in composites by holographic interferometry'
Proceedings of the International Symposium on Space Applications of Advanced Structural Materials. March 1990
ESA SP-303, p145-150

[34-39] G.B. Chai et al
'Vibration analysis of laminated composite plates: TV-holography and finite element method'
Composite Structures 23 (1993), p273-283

[34-40] J.B. Deaton & R.S. Rogowski
'Electronic shearography: current capabilities, potential limitations, and future possibilities for industrial non-destructive inspection'
Review of Progress in Quantitative Non-destructive Evaluation
Vol. 12A, p395-402, 1993, Plenum Press, ISBN 0-306-44483-6

[34-41] J.W. Newman
'Production and field inspection of composite aerospace structures with advanced shearography'
Review of Progress in Quantitative Non-destructive Evaluation
Vol. 10B, p2129-2133 1991, Plenum Press
ISBN 0-306-43903-4

[34-42] J.W. Newman
'Test composites with lasers'
Materials Engineering, July 1992, p10-11

[34-43] C. Hobbs
'The inspection of aeronautical structures using transient thermography'
Proceedings of the NDT for Corrosion in Aerospace Structures Conference, 12 Feb 1992, 6,1 to 6.10, ISBN 0903409992

[34-44] T. Ahmed at al
'Infrared thermal wave studies of composites'
Review of Progress in Quantitative Non-destructive Evaluation
Vol. 10B, p2173-2179 1991, Plenum Press

ISBN 0-306-43903-4

[34-45] W.P. Winfree
'Thermal QNDE detection of airframe disbonds'
NASA-CP-3160, 1991, p249-260

[34-46] T. Jones & H. Berger
'Thermographic detection of impact damage in graphite-epoxy composites'
Materials Evaluation, December 1992, p1446-1453

[34-47] R. Danjoux et al
'MECIR : A new Apparatus for the control of materials by infrared thermography'
SPIE Vol 1320 Infrared Technology and Applications, 1990
p282-291

[34-48] C.P. Hobbs et al
'The application of transient thermography for the non-destructive testing and evaluation of aerospace materials'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures '94, March 1994
ESA WPP-070, p645-654

[34-49] E.G. Henneke & B.S. Tang
'Thermal methods of NDE and quality control'
Chapter 6, Flight-Vehicle Materials, Structures, and Dynamics - Assessment and Future Directions
Vol. 4 Tribological Materials and NDE
ASME Publication, 1992, ISBN 0-7918-0662-6

[34-50] J. Webber
'NDT of CFRP structures: evaluation of a new method'
Proceedings of the International Symposium on Space Applications of Advanced Structural Materials. March 1990
ESA SP-303, p163-170

[34-51] S. Abbe et al
'Suivi de l'effet de viellissement en milieu oxydant de matériaux composites C/SiC par des techniques non destructives'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures '94, March 1994
ESA WPP-070, p687-692

[34-52] J. Bouteyre et al
'Thickness and density measurement of carbon-carbon materials by using backscattering of X-rays'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures '94, March 1994
ESA WPP-070, p655-660

[34-53] M.P. Hentschel et al
'New X-ray scanning topographic approaches to non-destructive evaluation'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures, March 1992
ESA SP-336, p229-232

[34-54] M.P. Hentschel et al
'New X-ray refractography for non-destructive investigations'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures '94, March 1994
ESA WPP-070, p661-664

[34-55] R.A. Buchanan et al
'Digital radiography in the aerospace industry'
Chapter 7, Flight-Vehicle Materials, Structures, and Dynamics - Assessment and Future Directions
Vol. 4 Tribological Materials and NDE
ASME Publication, 1992, ISBN 0-7918-0662-6

[34-56] C. Bueno et al
'High resolution digital radiography and 3D computed tomography of composite materials'
39th International SAMPE Symposium, April 11-14, 1994
p766-778

[34-57] L. Castaldello et al
'Advanced materials non destructive testing using high resolution dual energy tomography'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures '94, March 1994
ESA WPP-070, p635-638

[34-58] D. Ekenhorst et al
'Non-destructive inspection of ceramic matrix composites components by computerised tomography'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures '94, March 1994
ESA WPP-070, p665-672

[34-59] R.H. Bossi et al
'X-ray computed tomography for the aircraft/aerospace industry'
Review of Progress in Quantitative Non-destructive Evaluation
Vol. 10B, 1991, Plenum Press, ISBN 0-306-43903-4, p2121-2127

[34-60] R.H. Bossi et al
'X-ray computed tomography of composites'
26th International SAMPE Symposium, April 15-18, 1991
p224-238

[34-61] E.A. Birt et al
'Imaging of impact damage in graphite epoxy composites'
Review of Progress in Quantitative Non-destructive Evaluation
Vol. 12A, p897-904, 1993, Plenum Press, ISBN 0-306-44483-6

[34-62] R.H. Bossi & G.E. Georgeson
'Applied computed tomography for materials & processes development'
39th International SAMPE Symposium, April 11-14, 1994
p740-753

[34-63] G. Georgeson and R. Bossi
'X-ray computed tomography for advanced materials and processes'
In Developments in Ceramic and Metal-Matrix Composites
The Minerals, Metals, & Materials Society, 1991, p143-155

[34-64] R. Oster
'Computed tomography (CT) as a non-destructive test method used for composite helicopter components'
Proceedings of the 17th European Rotorcraft Forum
24-27 Sept 1991, Paper ERF91-87

[34-65] P.D. Tonner & J.H. Stanley
'Supervoltage computed tomography for large aerospace structures'
Materials Evaluation, December 1992, p1434-1445

[34-66] C. Bathias et al
'Application of X-ray tomography to the non-destructive testing of high performance polymer composite.'
ECCM 6, Sept 1993
Woodhead Publishing, ISBN 1-85573-142-8, p617-622

[34-67] B.A. Auld
'Electromagnetic methods in NDE'
Chapter 5, Flight-Vehicle Materials, Structures, and Dynamics - Assessment and Future Directions
Vol. 4 Tribological Materials and NDE
ASME Publication, 1992, ISBN 0-7918-0662-6

[34-68] G.E. McNeelege & C. Sarantos
'Robotic NDE Inspection of advanced solid rocket motor casings'
AIAA-94-1222-CP, p354-366

[34-69] M.W. Siegel
'Automation for non-destructive inspection of aircraft'
AIAA-94-1223-CP, p367-377

[34-70] R. Frankle & R. Menich
'Eddy current inspection of composite materials'
39th International SAMPE Symposium, April 11-14, 1994
p2451-2462

[34-71] X.E. Gros
'Eddy current testing: inspecting composite materials'
Materials World, April 1995, p180-181

[34-72] M.P. De Goeje & K.E.D. Wapenaar
'Non-destructive ispection of carbon fibre-reinforced plastics using eddy current methods'
Composites, Volume 23, No. 3, May 1992, p147-157

[34-73] D.W. Lowden et al
'Visualising defect geometry in composite materials'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures '94, March 1994
ESA WPP-070, p683-686

[34-74] S.I. Ganchev et al
'Microwave non-destructive evaluation of thick sandwich composites'
Materials Evaluation, April 1995, p463-467

[34-75] C. Wölfinger et al
'Damage detection in composite materials by monitoring electrical impedance'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures '94, March 1994
ESA WPP-070, p677-682

[34-76] A.C. Wey & L.W. Kessler
'Quantitative measurement of delamination area in low-velocity impacted composites using acoustic microscopy'
Review of Progress in Quantitative Non-destructive Evaluation
Volume 11B, 1992,
Plenum Press, ISBN 0-306-44206-X, p1563-1568

[34-77] L.J. Burnett et al
'Solid rocket motor NDE using nuclear magnetic resonance'
Review of Progress in Quantitative Non-destructive Evaluation
Vol. 12A, 1993, Plenum Press, ISBN 0-306-44483-6, p663-670

[34-78] G. Pfister et al
'Non-destructive testing of materials and components by computerized tomography with fast and thermal reactor neutrons'
Nuclear Science and Engineering: 110, p303-315 (1992)

[34-79] J.P. Komorowski et al
'Inspection of aircraft structures using D sight'
39th International SAMPE Symposium
April 11-14, 1994, p754-765

[34-80] J.P. Komorowski et al
'Application of Diffracto sight to the non-destructive inspection of aircraft structures'
Review of Progress in Quantitative Non-destructive Evaluation
Vol. 12A, 1993, Plenum Press, ISBN 0-306-44483-6, p449-455

[34-81] P. Cawley
'NDT of adhesive bonds'
Chapter 13, Flight-Vehicle Materials, Structures, and Dynamics - Assessment and Future Directions
Vol. 4 Tribological Materials and NDE
ASME Publication, 1992, ISBN 0-7918-0662-6

[34-82] T. Pialucha & P. Cawley
'The detection of a weak adhesive/adherend interface in bonded joints by ultrasonic reflection measurements'
Review of Progress in Quantitative Non-destructive Evaluation
Volume 11B, 1992
Plenum Press, ISBN 0-306-44206-X, p1261-1266

[34-83] P.N. Dewen & P. Cawley
'An ultrasonic scanning technique for the quantitative determination of the cohesive properties of adhesive joints'
Review of Progress in Quantitative Non-destructive Evaluation
Volume 11B, 1992
Plenum Press, ISBN 0-306-44206-X, p1253-1260

[34-84] D.R. Prabhu & W.P. Winfree
'Automation of disbond detection in aircraft fuselage through thermal image processing'
Review of Progress in Quantitative Non-destructive Evaluation
Volume 11B, 1992
Plenum Press, ISBN 0-306-44206-X, p1323-1330

[34-85] D.W. Lowden
'Quantifying disbond area'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures, March 1992
ESA SP-336, p223-228

[34-86] P. Plotard & C. Le Floch
'Non-destructive inspection of carbon-carbon with adapted coating for oxidation'
Proceedings of the International Symposium on Space Applications of Advanced Structural Materials. March 1990
ESA SP-303, p171-180

[34-87] G.Y. Baaklini et al
'NDE of titanium alloy MMC rings for gas turbine engines'
DE-Vol. 55, Reliability, Stress Analysis, and Failure Prevention
ASME 1993, ISBN 079181172250, p239-250

[34-88] H. Trétout et al
'Infrared thermography development for material evaluation of hermes space plane components'
Proceedings of the International Symposium on Space Applications of Advanced Structural Materials. March 1990
ESA SP-303, p181-186

[34-89] G. Sala et al
'Special problems of 3-D ultrasonic QNDI techniques and applications to composite aerospace structures'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures, March 1992
ESA SP-336, p209-216

[34-90] 'Integrity control for high temperature structural applications'
Daimler-Benz Aerospace - Dornier Reports
Phase I Final Report, November 1993
Phase II Final Report, February 1995
ESTEC Contract 9907/90/NL/PP(SC)

[34-91] R. W. A van der Ven & H. Bolks: Fokker, NL.
'Air coupled ultrasonic C-scan (AIRSCAN) for non-destructive inspection'
ESA Conference on Spacecraft Structures, Materials & Mechanical Testing, March 1996. Abstracts, p94

[34-92] W.A. Grandia & C.M. Fortunko: QMI/NIST, USA
'NDE applications of air-coupled ultrasonic transducers'
IEEE International Ultrasonics Symposium, Seattle, 7-10 November, 1995

[34-93] ESA PSS-03-207 - Guidelines for carbon and other advanced material prepreg procurement specifications.

[34-94] J. O. Strycek & H. Loertscher: QMI Inc., USA.
'Ultrasonic air-coupled inspection of advanced material'
NDT Net. Vol. 4 No. 12, December 1999.

[34-95] Ultrasonic Sciences Ltd., UK
'Multi-axis automatic ultrasonic inspection systems for advanced composite aircraft structures'.
Private communication – 2003.

[34-96] M.C. Bhardwaj & G F Stead
'Phenomenal advancements in non-contact ultrasound for composites characterisation'
46th International SAMPE Symposium May 6-10, 2001

[34-97] J.S. McIntosh et al.,
'The characterisation of capacitive micromachined ultrasonic transducers in air'
Ultrasonics 40 (2002) p477-483.

[34-98] M. Castaings & B. Hosten
'The use of Electrostatic, Ultrasonic, Air-coupled Transducers to Generate and Receive Lamb Waves in Anisotropic, Viscoelastic Plates'
Ultrasonics, v. 36, p361-365 (1998).

[34-99] E. Bloome et al.
'Recent observations with air-coupled NDE in the frequency range of 650 kHz to 1.2 MHz'
Ultrasonics 40 (2002) p153-157.

[34-100] R. Stoessel et al.
'Air-coupled ultrasound inspection of various materials'
Ultrasonics 40 (2002) p159-163

[34-101] J. Poguet et al.
'Phased array technology: Concepts, probes and applications', 8th European Congress on Nondestructive testing, June 17-21, Barcelona, Spain.2002

[34-102] Mahaut et al.
'Development of phased array techniques to improve characterisation of defect located in a component of complex geometry'
Ultrasonics 40 (2002) p165-169

[34-103] J. Pena et al.
'Low-cost, low frequency phased array system for damage detection in panels'
Proceedings of the 2nd European workshop on structural health monitoring, 2004

[34-104] M. J. S. Lowe and P. Cawley
'The Applicability of Plate Wave Techniques for the Inspection of Adhesive and Diffusion Bonded Joints'
Journal of Destructive Evaluation, Vol 13, No 4, 1994

[34-105] R. Seifried, L. J. Jacobs & J. Qu
'Characterization of adhesive bond properties with Lamb Waves'
Review of Progress in Quantitative Non destructive Evaluation Vol. 20, Edited by D. O. Thompson and D E Chimenti

[34-106] P. B. Nagy & L. Adler
'Nondestructive evaluation of adhesive joints by guided waves'
J Appl Phys. 66 (10), 15 november 1989

[34-107] M. Lemistre & D. Balageas
'Structural health monitoring system based on diffracted Lamb wave analysis by multiresolution processing'

Smart Materials and Structures 10 (2001) p504-511

[34-108] P. Fromme et al.
‘A guided wave array for structural health monitoring’
Proceedings of the 2nd European workshop on structural health monitoring 2004

34.26.2 ECSS documents

[See: ECSS website]

ECSS-Q-ST-20	Space product assurance - Quality assurance; previously ESA PSS-01-20
ECSS-E-ST-30 series	Space engineering - Mechanical
ECSS-E-ST-32-01	Fracture control; previously ESA PSS-01-401

35
Objective of verification

35.1 Introduction

35.1.1 General

The objective of verification is stated along with those additional factors which are to be considered for structures manufactured from fibre-reinforced composite materials compared with metal structures.

35.1.2 Composite structures

35.1.2.1 Key factors

To verify a composite structure, items which are considered are:

- Adequacy of design,
- Manufacturing, and
- Testing.

35.1.3 Composite materials

35.1.3.1 Additional factors

In addition to the requirements of a metallic design, the factors specific to composites are:

- Procurement specification: A detailed specification for all the raw material(s), e.g.
  - prepregs,
  - resins.
- Incoming control: Procedure for all raw materials
- Design allowables: Philosophy of assessment.
- Tests: To support the composite design.

- Manufacturing:
  - Procedure for all parts.
  - Curing process procedure.
  - Assembly: Procedure for the assembly of the structure.

35.1.3.2 Verification procedure

The verification procedure comprises of:

- Hardware, and
- Software, e.g. assessment of design allowables.

35.1.3.3 Verification guidelines

A general verification procedure for composite structures can be derived from the guidelines given for:

- Raw materials, [See: Chapter 36].
- Design allowables, [See: Chapter 37].

The guidelines provide the fundamentals for a given procedure.

36 Procurement specifications for raw materials

36.1 Introduction

36.1.1 Guidelines

The guidelines presented contain information to assist in the preparation of specifications for the procurement of advanced composite constituents and prepregs. Also described are the parameters that it is necessary to control or monitor for qualification and batch control.

The guidelines are not intended to be definitive specifications. In many cases, it is not necessary to specify all the parameters outlined. It remains the judgement of those preparing a specification to include only what is really necessary, thereby avoiding an excessively complex specification and the unduly high cost of complying with them.

36.2 Specification methods

36.2.1 Fiche sheet principle

36.2.1.1 General

There is a tendency for specifications to be written on what is tautologically described as the 'fiche-sheet' principle.

The structure has obvious benefits for a hardware manufacturer using a number of prepreg materials. Unfortunately, the system is fraught with practical difficulties, which become apparent when a particular material needs a special test or when various manufacturers are persuaded to supply a material to the same specification procedure.

However, the trend towards the 'fiche-sheet' specification structure is growing, owing to the reduction in paperwork and administrative effort that it affords.

36.2.1.2 Master document

The fiche-sheet structure needs the creation of a master document in which items are defined, including:

- Ordering,
- Delivery,
- Qualification,
- Acceptance,
- Batch release, and
- Test method procedures.

It does not include specific numerical property data requirements.

36.2.1.3 Fiche sheet

A separate document - the 'fiche-sheet' - is issued as a supplement to the master document and lists the required property data for a specific material.

36.2.2 Specific material specification

Owing to problems identified with the fiche-sheet principle, most materials are still ordered to a specific material specification, which is established by the manufacturer and customer in collaboration.

36.3 Guidelines on resin procurement

36.3.1 Scope

The guideline gives information on the preparation of a specification for the procurement of a particular resin system.

The main items which are needed for the identification of requirements for batch control and the qualification of a resin system are listed.

36.3.2 Basic information to be provided by the supplier

36.3.2.1 Introduction

Where constituent items are ordered separately, the associated information is supplied for each item, i.e.:

- General; as shown in Table 36.3.1.
- Base resin; as shown in Table 36.3.2.
- Hardener; as shown in Table 36.3.3.
- Catalyst; as shown in Table 36.3.4.
- Modifier; as shown in Table 36.3.5.
- Solvent; as shown in Table 36.3.6.
- Blend; as shown in Table 36.3.7.

The supplier also confirms that the material is free from contamination.

All the information requirements described are for the approval of the material. All those marked with an asterisk are needed for batch control. If one or more of the mentioned parameters is out of specification, certain re-testing can be required.

36.3.2.2 General

All the information described in Table 36.3.1 are for the approval of the material. All those marked with an asterisk are needed for batch control.

Table 36.3-1 - Resin procurement: General Information

	Information	Batch
1.	Manufacturer's name and product name	*
2.	Procurement specification number	*
3.	Batch number and the identification numbers as required	*
4.	Release date	*
5.	Packaging requirements	*
6.	Storage and transportation constraints	*
7.	Allowable shelf life	*
* Necessary as part of a batch test		

36.3.2.3 Base resin

All the information described in Table 36.3.2 are for the approval of the material. All those marked with an asterisk are needed for batch control.

Table 36.3-2 - Resin procurement: Base resin

	Information	Batch
1.	Chemical compliance: supply of infrared (I.R.) spectrum analysis	
2.	Density	*
3.	Solids content or volatile content	*
4.	Viscosity or melting point †	*
5.	Epoxy content or equivalent †	*
6.	Refractive index (only for liquids)	*
Key:	† if applicable * Necessary as part of a batch test	

36.3.2.4 Hardener

All the information described in Table 36.3.3 are for the approval of the material. All those marked with an asterisk are needed for batch control.

Table 36.3-3 - Resin procurement: Hardener

	Information	Batch
1.	Chemical compliance: supply of I.R. spectrum analysis	
2.	Density	*
3.	Volatile or solids content	*
4.	Amine number or anhydride value or nitrogen content	*
5.	Viscosity or melting point	*
6.	Refractive index (only for liquids)	*
* Necessary as part of a batch test		

36.3.2.5 Catalyst

All the information described in Table 36.3.4 are for the approval of the material. All those marked with an asterisk are needed for batch control.

Table 36.3-4 - Resin procurement: Catalyst

	Information	Batch
1.	Chemical compliance: supply of I.R. spectrum analysis	
2.	Density	
3.	Solids content or volatile content	*
4.	Viscosity or melting point †	*
5.	Epoxy content or equivalent †	*
6.	Refractive index (only for liquids)	*

Key: † if applicable
* Necessary as part of a batch test

36.3.2.6 Modifier

All the information described in Table 36.3.5 are for the approval of the material. All those marked with an asterisk are needed for batch control.

Table 36.3-5 - Resin procurement: Modifier

	Information	Batch
1.	I.R. Spectrum	
2.	Density	*
3.	Viscosity or melting point	*
4.	Refractive index (only for liquids)	*

* Necessary as part of a batch test

36.3.2.7 Solvent

All the information described in Table 36.3.6 are for the approval of the material. All those marked with an asterisk are needed for batch control.

Table 36.3-6 - Resin procurement: Solvent

	Information	Batch
1.	Density	*
2.	I.R. Spectrum	
3.	Refractive index	*

* Necessary as part of a batch test

36.3.2.8 Blend

All the information described in Table 36.3.7 are for the approval of the material. All those marked with an asterisk are needed for batch control.

Table 36.3-7 - Resin procurement: Blend

	Information	Batch
	Un-cured (prior to release for production use)	
1.	Gel time at defined temperature	*
2.	Viscosity versus temperature, or	*
3.	Viscosity versus time at defined temperature	*
4.	Density	
5.	Heat of polymerisation	
	Cured	
1.	Glass transition temperature	*
2.	Density	
3.	Shrinkage due to curing	
4.	Water absorption	
5.	Outgassing	
6.	Radiation effects	
* Necessary as part of a batch test		

36.3.2.9 Thermo mechanical properties

The determined properties are:

- Compression strength.
- Compression modulus.
- Tensile strength.
- Tensile modulus.
- Elongation.
- Impact strength.
- Poisson's ratio.
- Hardness.
- Density.
- Coefficient of thermal expansion, CTE.
- Shear modulus versus temperature.

The supplier indicates:

- the types of testing used to establish the properties, and
- their typical values, and
- the likely property variation that can be expected.

36.4 Guidelines on carbon and other fibre prepreg procurement

36.4.1 Scope

36.4.1.1 General

The guidelines describe the parameters which are necessary to control or monitor prepreg in order for validation and batch control..

For detailed information, refer to ESA PSS-03-207.

36.4.1.2 Thermosetting resin matrix

The guidelines are intended as a general guide for organisations preparing specifications for the procurement of particular thermosetting resin impregnated reinforcing fibre systems (prepreg).

36.4.1.3 Thermoplastic matrix

Whilst the guidelines concentrate on the procurement of thermosetting resin prepreg they are, in many respects, also applicable to thermoplastic matrix prepreg.

36.4.2 Description and classification of prepreg

36.4.2.1 Fibre type

The guidelines are aimed primarily at purchasers of prepreg containing carbon fibres. However, many of the parameters described are applicable to prepregs of other continuous reinforcing fibres, such as glass, boron, aramids, silicon carbide and alumina.

Hybrid prepregs, containing a mixture of reinforcing fibres, such as glass and carbon, also need similar specification control.

36.4.2.2 Prepreg type

The most common type of prepreg is the unidirectional (UD) variety. A unidirectional prepreg contains a parallel planar array of fibres, usually several fibres thick, suitably pre-impregnated with either a partially cured or an un-cured specified thermosetting resin.

Prepregs incorporating woven arrays of reinforcing fibres, i.e. fabrics, are an important group of materials. The properties of the fibre and resin constituents of the prepreg can be defined by reference to appropriate specifications for these constituents.

36.4.2.3 Resin type

[See: 36.3 for guidelines on producing a resin procurement specification]

36.4.2.4 Prepreg classification

Lamination of prepreg plies and curing under defined conditions of temperature, pressure and time in a moulding press or autoclave produce components with required strength or stiffness characteristics.

The classification of a prepreg can be in terms of:

- The required mechanical properties of a cured laminate, frequently specified to be those at the operating temperature and humidity of a component, or
- The type of resin or fibre can be used as a basis for prepreg classifications.

In either case, the material supplier provides guidelines on the range of applicability of a particular prepreg and its constituents.

36.4.2.5 Thermoplastic matrix prepregs

These are supplied in unidirectional and fabric forms but do not possess the same intrinsic physical characteristics of thermoset prepregs. Thermoplastic prepregs tend to have:

- Poor drape, and
- No tack.

A procurement specification of thermoplastic matrix prepregs contains fewer requirements than those for thermoset prepregs. Factors relating to material content and distribution are important but those relating to matrix chemistry are less critical.

36.4.3 Prepreg charachteristics for qualification and batch testing

36.4.3.1 Specification

It is envisaged that all the characteristics described satisfy the approval of a prepreg to a specification. Many of the requirements are necessary for batch release to a specification. It is usually the responsibility of the user to define a schedule of acceptance tests to ensure that released material complies with the specification to which it is released.

It is further envisaged that the constituent resin and fibre system in the prepreg have been validated, batch released and accepted according to appropriate specifications.

A procurement specification defines all the items.

36.4.3.2 Identification and marking

A specification usually defines the way in which a consignment of prepreg is identified by the manufacturer. The requirements for reeled prepreg tape and cut unidirectional prepreg sheet are often slightly different.

36.4.3.3 Release documentation

It is usual practice for information additional to that set out in 'Identification and Marking' to be supplied as release documentation accompanying each batch or shipment (whichever is the smaller) of material released to a specification.

36.4.3.4 Packaging

Both faces of a prepreg are protected by non migratory, non-contaminating backing materials.

36.4.3.5 Prepreg construction

The construction parameters of a prepreg that are commonly specified are:

- Thickness
- Cohesion
- Gaps and slits
- Splicing
- Dimensions:
  - width,
  - length.
- Resin content
- Fibre content
- Prepreg mass
- Volatile content
- Tack
- Edges
- Flammability, toxicity and safety
- Shelf life and shop life
- Fabric parameters:
  - weave type
  - areal weight
  - warp and weft characteristics
- Characteristics as provided by:
  - HPLC
  - DSC
  - DMA

36.4.3.6 Prepreg quality and defects

Prepreg material as supplied inevitably exhibits some defects.

The definition of defective material is a significant area derived by agreement between manufacturer and customer, and can include:

- Discontinuous tows (excluding allowable tow splices),
- Prepreg splices,
- Cured or hardened resin particles,
- Fibre starved areas,
- Resin variations:
  - excess resin, or
  - resin starved areas,
- Gaps or slits (in excess of those defined),
- Whorls,
- Fuzz balls,
- Any imperfection in fibre alignment due to tow slackness:
  - kinks,
  - waves, or
  - twisted fibres.
- Foreign material,
- Contamination, and
- Any other features that adversely affect its performance.

36.4.3.7 Batch acceptance testing by the supplier

Items to be defined are:

- Test parameters,
- Batch acceptance and rejection criteria, and
- Retest conditions.

36.4.3.8 Incoming inspection by the user

It is common practice for a user to check incoming material by re-testing for some or all the batch acceptance requirements.

Re-testing is at the user's discretion and is not normally incorporated into a procurement specification.

36.4.4 Laminate Characteristics

36.4.4.1 Minimum properties

It is usual practice to specify minimum physical and mechanical properties for a laminate produced from the prepreg by means of a defined cure schedule.

36.4.4.2 Cure schedule

The defined cure schedule is for specification purposes and need not correspond to the component production cure; it is, however, within acceptable temperature limits. A number of curing cycle repetitions having no adverse influence on the laminate mechanical properties can be incorporated, if necessary.

Parameters and their interrelationships to be defined are:

- Cure time (with allowable range);
- Cure temperature (with allowable range);
- Permissible range of heat-up and cool-down rates;
- Cure pressure range or cycling;
- Pre- and post-cure dwell phases and their purpose.

36.4.4.3 Manufacturing factors

If so desired, items for the preparation of test laminates can all be specified, e.g.:

- Tooling,
- Prepreg stacking sequence,
- Release and bleed cloth layers, and
- Stack assembly for preparing test laminates.

The sample-cutting pattern from the laminate can also be specified. It is the conventional practice to manufacture unidirectionally reinforced laminates for specification testing purposes. Special tests can be necessary in the case of woven fibre prepreg.

36.4.4.4 Qualification testing

Various mechanical and physical properties are determined at ambient temperature and humidity against defined test standards agreed between the supplier and purchaser. These include:

- Longitudinal tensile strength
- Longitudinal tensile modulus
- Longitudinal compressive strength
- Longitudinal compressive modulus
- Transverse tensile strength and modulus
- Transverse compressive strength
- Longitudinal flexural strength and modulus

- Transverse flexural strength and modulus
- Short beam shear strength (interlaminar shear strength)
- Fibre volume fraction and void content
- Laminate density
- Laminate outgassing and offgassing performance
- Flammability
- Toxicity

If necessary, additional elevated temperature or humidity conditions can be prescribed.

36.4.4.5 Batch and incoming inspection testing

Guidelines are suggested as a practical minimum for realistic quality control testing of a prepreg, for approval, batch and incoming acceptance purposes.

Other laminate tests can be defined to reflect the service conditions, e.g. compression or elevated temperature tests.

Usually, a minimum of five individual tests for each property are carried out, the individual results are recorded as part of the release documentation.

- Fibre volume fraction: To be within a specified tolerance, usually about ± 3 % of a defined figure in the range 60 to 65%.
- Longitudinal flexural strength: To be greater than a defined minimum, with a specified maximum coefficient of variation of results, usually about 6%.
- Longitudinal flexural modulus: To be within a defined range, with a specified maximum coefficient of variation of results of usually about 6%.
- Interlaminar shear strength: To be within a defined range at ambient temperature and to be greater than a specified minimum at a defined elevated temperature, with a specified maximum coefficient of variation of results, usually less than 10%.

The values quoted are typical for carbon fibre prepreg.

36.4.5 Test methods

Unfortunately, it is not feasible to give fully explicit guidance on applicable test methods for incorporation into specifications. Practices vary considerably from country to country and from one composite hardware constructor to the next.

Probably the most comprehensive range of applicable test methods is that published by ASTM and ANSI, but even this is not exhaustive.

For some properties, e.g. prepreg tack, no national standard test methods have been published: the non-standard ones that do exist have often been written especially for specification purposes.

[See also: Chapter 7 for test methods; ESA PSS-03-207 Appendix 2]

36.5 References

36.5.1 ECSS documents

[See: ECSS website]

ECSS-E-ST-32-08	Materials
ECSS-Q-ST-70	Materials, mechanical parts and processes
ECSS-Q-70-71	Data for the selection of space materials and processes

36.5.2 ESA publications

The documents cited have not yet been transferred to the ECSS documentation system.

ESA PSS-03-207	Guideline for carbon and other advanced fibre prepreg procurement specification

37 Philosophy of assessment of design allowables

37.1 Introduction

Design allowables are defined, [See: 37.3], and a procedure for the generation of allowable data for development purposes is provided, [See: 37.4].

Test data for a carbon fibre/epoxy composite is included, [See: 37.5].

37.2 Carbon fibre reinforced plastics

37.2.1 Design values

37.2.1.1 General

Strength is verified by analysis under usage of material data which is achieved with a probability of 99% on a confidence level of 95%, i.e. 'A' values.

MIL HDBK-5, Ref. [37-2], gives information on how those design values can be generated with batch-to-batch variation and deliveries from different suppliers. The necessary information in this respect is not available and consequently the approach of MIL HDBK cannot be formally applied. Furthermore, the extension to different suppliers is considered as not justified if a material is used only from one source.

37.2.1.2 Procedure

To overcome the problems highlighted with respect to MIL-HDBK-5, a procedure is outlined on how to generate design allowables for unidirectional CFRP elements, manufactured from 914C/HM, 0.1 mm thick prepregs.

The procedure intentionally covers the variability of CFRP material and defines design values which can be achieved also in future manufacture, provided that the defined quality assurance provisions are taken.

37.3 Definition of design allowables

37.3.1 'A' basis allowable

At least 99% of the population of values is expected to equal or exceed the 'A' basis mechanical property allowable, with a confidence of 95%.

37.3.2 'B' basis allowable

At least 90% of the population of values is expected to equal or exceed the 'B' basis mechanical property allowable, with a confidence of 95%.

37.4 Development procedure

Mechanical properties, relevant for an actual application, are often not tested during incoming inspection. If they are, the values obtained from a few samples do not enable the generation of 'A' basis design allowables, [See also: 37.3].

In order to obtain data which can be used during the development, the policy to be implemented is:

- The minimum acceptance value of a small sample is considered as the lowest average value of any batch accepted.
- Based upon present knowledge, the minimum values (99% probability of exceedance) are estimated under individual consideration of their typical scatter with one batch.
- The data of a medium size sample are considered as normally having a Gaussian distribution. A slight reserve factor covering uncertain effects on scatter of approximately 5% has been applied additionally, but which can be removed if enough statistical data become available in time.
- The ‘design allowables’ are not justified by the supplier. This results in high cost and possibly delays in delivery. Testing, where necessary, is the responsibility of the user. Therefore:
    - Receiving inspection is based on low sample numbers against acceptance values which need to be exceeded by all the specimens in small sample size; ≤ 6 specimens.
    - The customer performs additional material testing to establish the statistical distribution. This can enable acceptance of a batch which does not meet the acceptance requirement, but can show lower scatter within the batch.

37.5 Test data

37.5.1 Material

The test data presented are for the high modulus, HM, carbon fibre/epoxy system:

- Prepreg 914C MS-4-40: batch 75/51487
- Fibre charge E2M 352.

37.5.2 Data

37.5.2.1 General

Table 37.5.1 gives the properties of the material obtained from tests. The statistical-based 'probability of exceedance' data for the same batch of material are given for:

- Tensile strength UD 0° in Figure 37.5.1.
- Tensile strength UD 90° in Figure 37.5.2.
- Compression strength UD 0° in Figure 37.5.3.
- Interlaminar shear strength UD ±45° in Figure 37.5.4.

Table 37.5-1 - Test data for HM carbon/epoxy specimen

		Specimen Cross-section (mm x mm)	$\bar{\sigma}$ **(N/mm²)**	**SD (N/mm²)**	**99%/95% Values (N/mm²)**
Tensile: UD 0°	R_1^{tu}	1 x 7	1121	122	738
Tensile: UD 90°	R_2^{tu}	Cross-section at Failure	45.6	5.8	27.3
Compression: UD 0° †	R_1^{cu}	Facing 0.2 x 25	898	45	756
Interlaminar shear strength: 0°/±45°	R_{13}^{su} (0/±45	2.3 x 10	64.5	7.8	40.0

Key: † Four point sandwich bending.
Values, excluding ILS, relate to 60% by volume fibre content.

37.5.2.2 Tensile strength UD 0°, batch 352

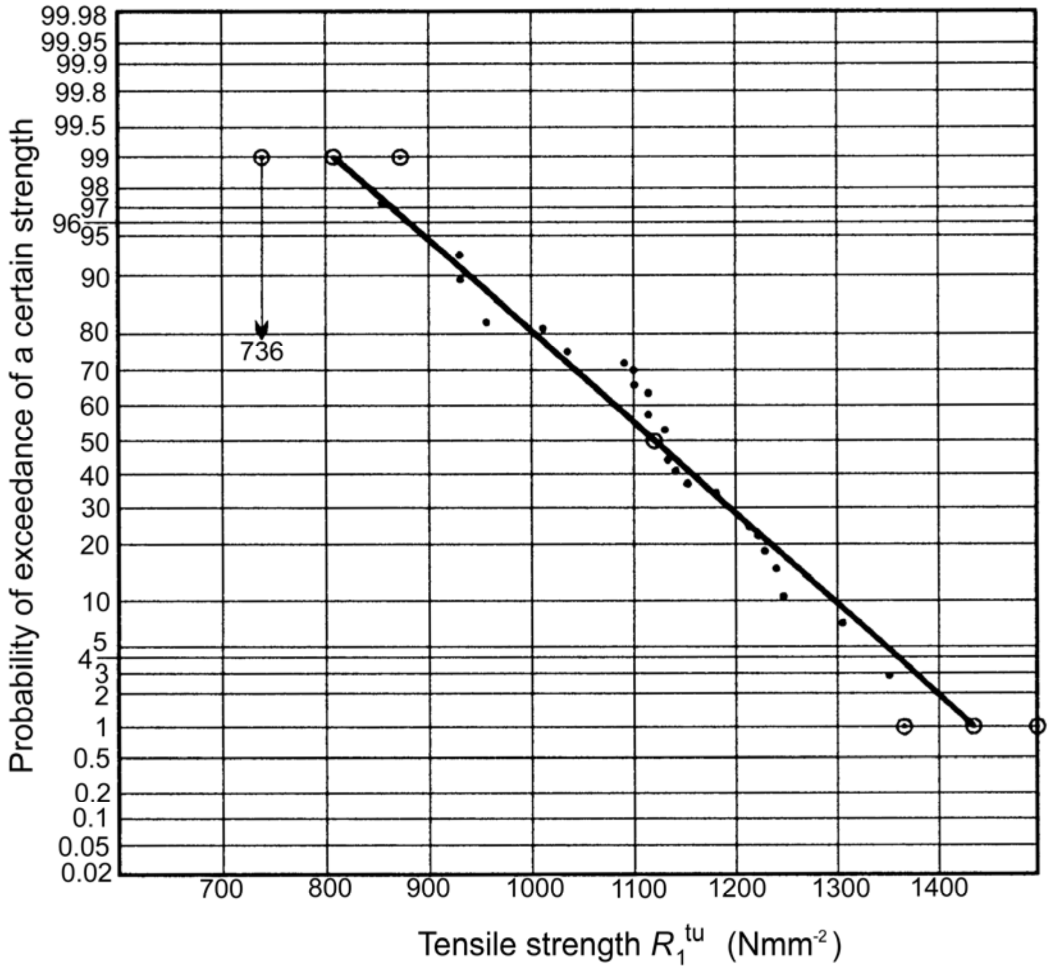


Figure 37.5-1 - Probability of exceedance: Tensile strength 0°

37.5.2.3 Tensile strength UD 90°, batch 352

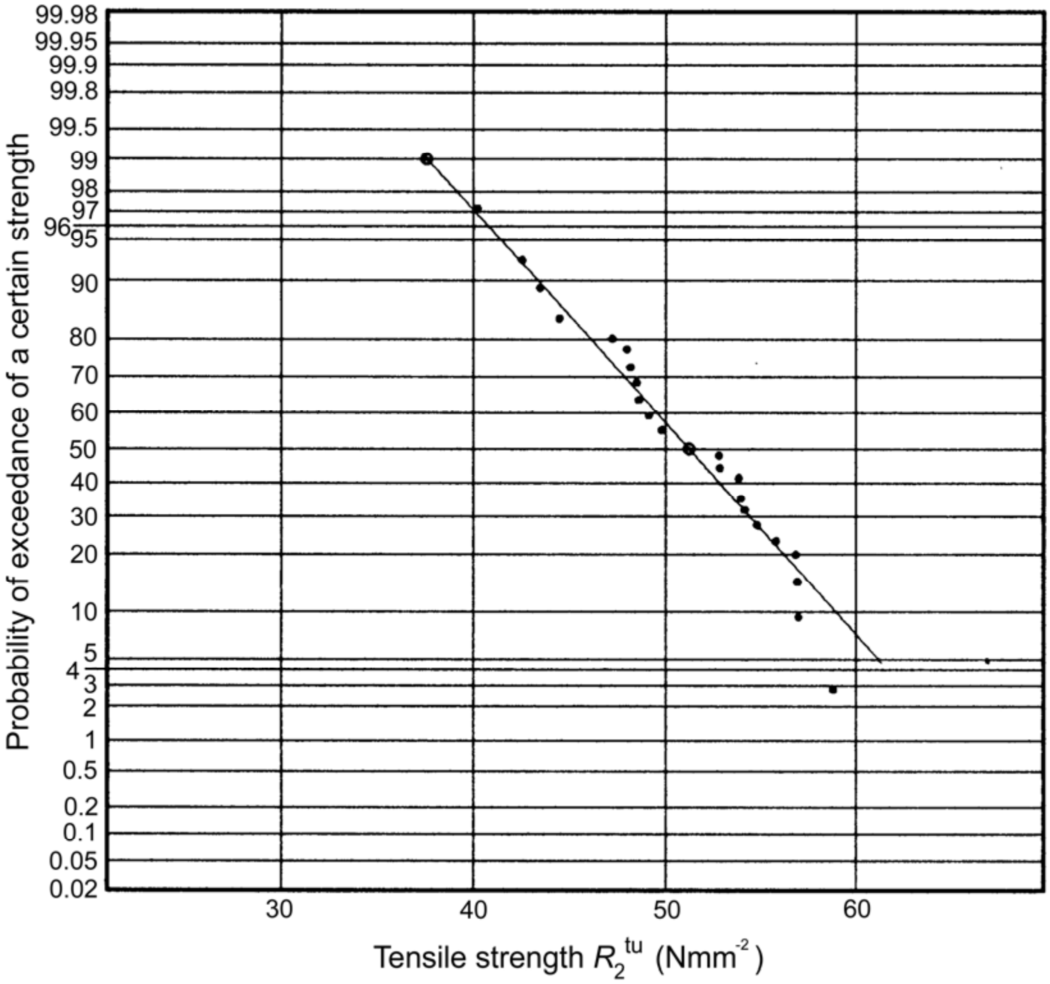


Figure 37.5-2 - Probability of exceedance: Tensile strength 90°

37.5.2.4 Compression strength UD 0°, batch 352

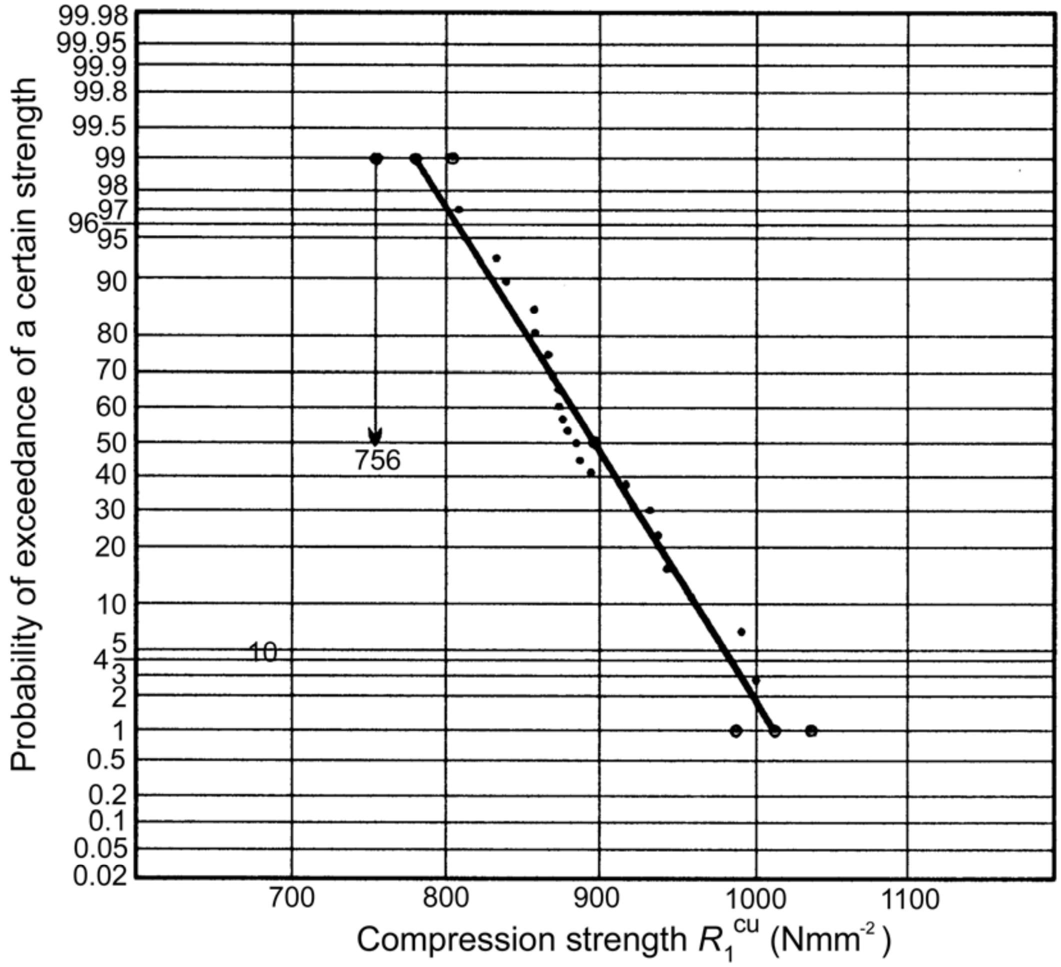


Figure 37.5-3 - Probability of exceedance: Compressive strength 90°

37.5.2.5 Interlaminar shear strength UD ±45°, batch 352

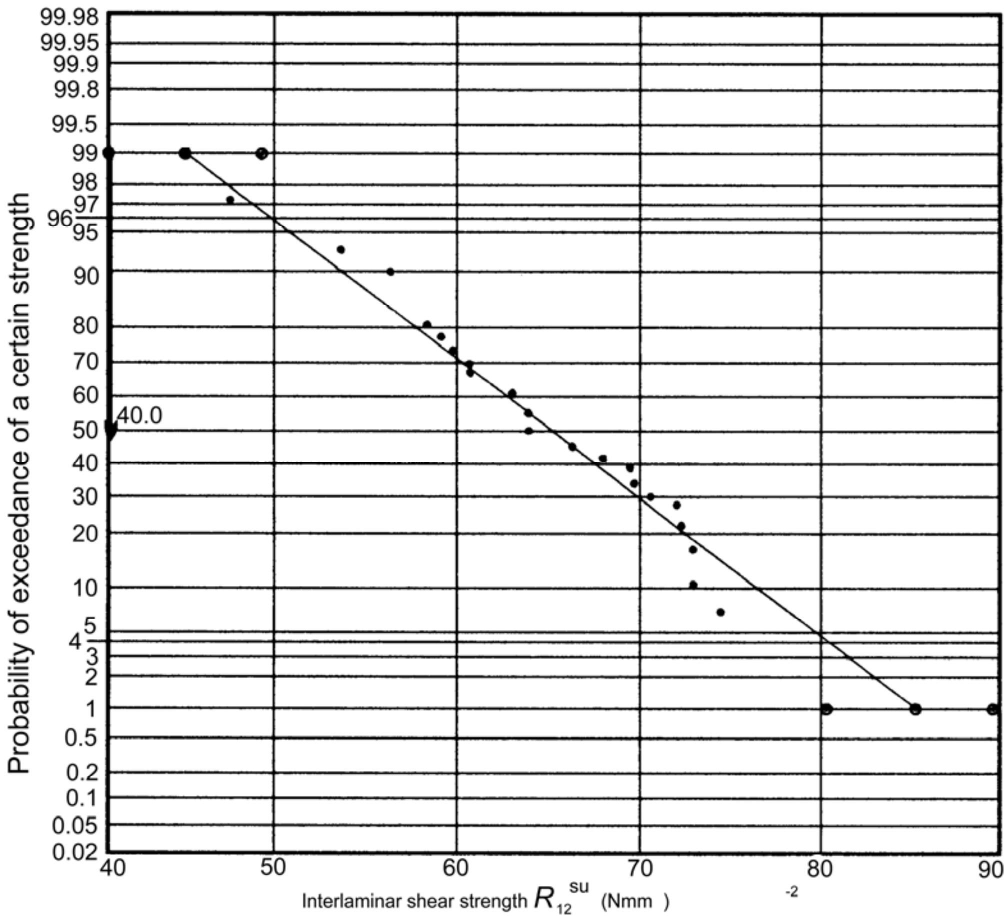


Figure 37.5-4 - Probability of exceedance: Interlaminar shear strength

37.6 References

37.6.1 General

[37-1] MBB/ERNO
Unpublished work

[37-2] MIL HDBK-5F: Metallic materials and elements for aerospace vehicle structures
November, 1990

38 Manufacturing techniques

38.1 Introduction

38.1.1 Processing techniques

Fibre-reinforced plastics can be processed by a number of methods, Ref. [38-1], [38-2].

The majority of composite space structures are assembled from prepreg configurations, which are laid-up by hand and then moulded in an autoclave, [See: 38.4].

Filament winding, [See: 38.5], is also used, mainly for cylindrical configurations, [See also: Chapter 29]. Variants of these widely accepted process methods are described.

Supporting process technologies are also described. These include:

- Resin transfer moulding, [See: 38.7].
- Pultrusion, [See: 38.8].
- Pressure forming, [See: 38.10].

Information is also provided on developing technologies which have a possible future use, such as radiation curing, [See: 38.12].

38.1.2 Process selection

The selection of a suitable process is influenced by a various factors, including:

- Design of structure,
- Materials selection,
- Equipment availability,
- Component configuration,
- Unit numbers, and
- Cost-to-mass trade-offs.

The selection of a manufacturing route is often the result of a trade-off between a minimum mass design and acceptable cost.

[See also: ECSS-Q-ST-70; ECSS-Q-70-71]

38.2 Process selection criteria

The selection of a process is, in theory, determined by identifying the most cost-effective solution for the desired structural configuration. This is an idealised assumption which is based on the expectation that there is also an optimum technical design solution for the application.

In reality, a number of designs are usually able to fulfil the performance specification, Ref. [38-3].

For example, acceptable design configurations for thrust cylinders have been produced by:

- Sandwich constructions with composite face skins and light alloy honeycomb cores,
- Rib-stiffened monolithic CFRP designs, and
- Filament wound CFRP arrangements, Ref. [38-4], [38-5].

The selection of a process is driven by a number of considerations, of which the main ones are:

- Can previous designs and manufacturing experience be used to find a solution?
- For a new design concept, is there access to appropriate manufacturing facilities?
- Does the process enable an optimum fibre orientation and fibre volume fraction to be achieved?
- Does the process achieve the desired component shape and dimensional tolerances?
- What is the part count for the identified process route?
- What are the subsequent machining and finishing operations after component processing?
- Can the process accommodate load introduction points in components?
- For design and quality control purposes, what material property data from the process route needs to be obtained?
- What are the cost implications with respect to man-hours, tooling, consumable materials and process times? [See also: Chapter 40]

The selection of a manufacturing route is often based on trade-offs between competing designs to minimise mass and acceptable costs.

38.3 Hand lay-up

38.3.1 General

Fibre-reinforced plastics processing has traditionally involved the manual lay-up of materials. This is often the only route possible when low numbers of items are made to any one design. When the scale of manufacturing operations increases, the amount of manual involvement can be reduced by automation. The aim is to reduce costs and improve product consistency. Automated and programmable prepreg cutting systems were introduced on this basis.

Manual lay-up is used in:

- wet laminating, and
- prepreg lay-up, prior to autoclaving.

38.3.2 Wet lamination

38.3.2.1 General

Wet lay-up by hand is a method of moulding components at room temperature using thermosetting polyester and epoxy resins, usually with mat or woven roving reinforcements. A chemical reaction, initiated in the resin by a catalytic agent, produces hardening of the resin in the finished part.

38.3.2.2 Process

The mould and the gel covering are properly prepared. Chopped strand mat, cloth or woven rovings are cut to shape from reels using a utility razor knife, large scissors or an electric cloth-cutting machine.

Measured amounts of resin and catalyst are then thoroughly mixed together. The resin mixture can be applied to the reinforcement either outside the mould or inside it. To ensure complete removal of air and wet-out of the fibres, the resin is applied first and the reinforcement is placed on top. Brushes, squeezers and rollers are used to compact the material against the mould surface and to remove any entrapped air, as shown in Figure 38.3.1.

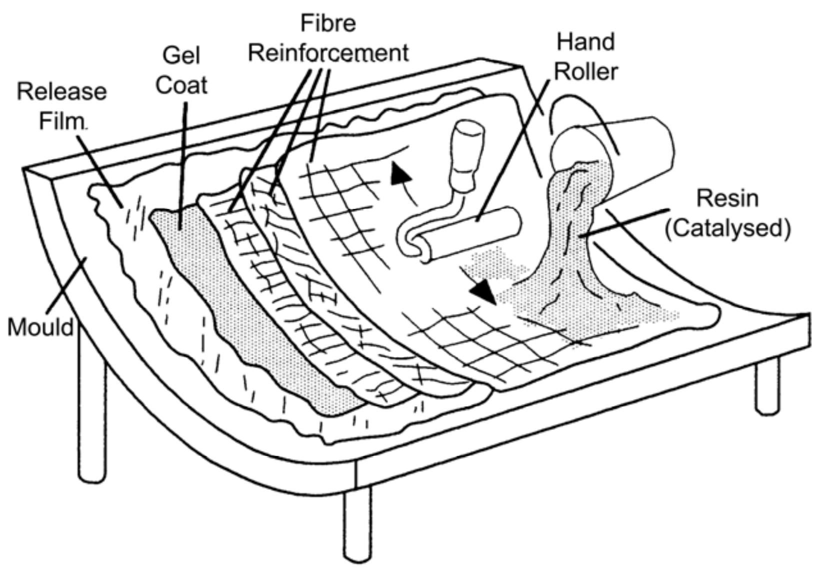


Figure 38.3-1 - Hand lay-up process for mat or cloth

Additional layers of mat or woven roving can then be applied until the total thickness has been achieved. Layers of mat and woven roving are usually alternated to ensure good interlaminar bonding, to avoid air entrapment and to ensure the highest strength.

Fibre volume contents vary with the reinforcement type used, typical values are:

- All-mat laminate: 25% to 35%.
- Mat and woven-roving laminate: 35% to 45%.
- All-cloth moulding: 50%.

Table 38.3.1, Ref. [38-2], describes factors to be considered when selecting the wet lay-up process. It details what is possible with a hand lay-up technique. Some of the selection factors associated with the wet lay-up route are given in Table 38.3.2, Ref. [38-1].

Wet laminations are rarely used in space structure configurations. Some launcher components can be appropriate, if low cost takes precedence over mass-efficient designs. All-cloth mouldings are preferred because they offer acceptable mass-efficiency. Low numbers of complex shapes, such as ducting, can be made by this process.

Table 38.3-1 - Wet lay-up and laminating: Troubleshooting guide

Problem	Cause	Solution
Cure in thickened rods or strings.	Pre-gelling.	Keep mixing containers clean and free of previously catalysed gel coat. Use throw-away mixing containers.
Cracking and fissuring.	Larger cracks caused by too thick areas of gel coat or excessive exotherm or thin point in lay-up. Fissuring because of front or reverse impact blow.	More uniform application of gel coat and better mixing with catalyst. Prevent accidental or injuring blows.
Fibre pattern: random fibres from mat or cross-hatch from woven roving weave.	High exotherm, coarse weave material too close to gel coat.	Cure laminate in steps: use lower exotherm resin. Put more mat in front of woven roving. Best solution is application of an intermediate layer of more rigid resin-containing Vitro-Strand fibres.
Lay-up draining on vertical surfaces.	Resin too low in viscosity, resin with insufficient thixotropic agent; mould or room too warm.	Most probable correction is to increase thixotropic agent content of resin.
Bubbles.	Air entrained in reinforcement after combination with resin.	Add 0.2% green pigment to lay-up resin to see voids. Work lay-up more freely with brushes, squeegees, or serrated rollers. If possible, apply a liberal quantity of resin onto work before applying reinforcement, so that the resin forces air out from the bottom.
Bridging over small radius curves such as lap-strokes, etc.	Reinforcement too stiff: curves below design-allowables.	Select more highly wettable or soluble mat or woven roving. Use loose-mixed putty to caulk small radii curvatures prior to lay-up. Redesign mould.
Thin areas.	Gaps in lapping reinforcement caused by improper placement or short-cutting, etc.	Correct placement and cutting errors. Lay in patches to correct thin spots prior to removal from mould. Try pre-wetting of reinforcement by resin prior to placement in mould.
Fibres protruding from inner lay-up surface.	Usually unavoidable if mat is sole reinforcement.	For finish layer, apply woven fabric, woven roving, or veil mat on inside. After cure, sand and apply splatter paint.
Cracked or resin-rich areas usually at bottom or well-point.	Drainage of resin in large lay-up to a low point and, because of high exotherm, results in cracking: possibly too high a resin to glass ratio.	Introduce more thixotropic agent into resin. Continue to squeegee excess resin out of collection points until gelling occurs. Add additional reinforcement.
Warpage of part.	Unbalanced laminate. Flat surface.	Use symmetric lay-up. Design slight radius in surface,
Distortion of part.	Under-cured in mould.	Allow full cure in mould.
Cracking next to stiffening members.	Hard spot.	Use fillet in corner where stiffener meets laminate.
Low impact strength.	Insufficient glass: too much flexing.	Use bag moulding, more woven roving, roving, use stiffener or sandwich construction.
Slow curing laminating resin.	Environment changes.	Adjust catalyst to weather changes.
Roller picks up fibres when working on mat.	Too close to gel time: styrene evaporation; rolling too fast.	Adjust gel time; adjust fans; dip roller in styrene or fresh resin; more deliberate rolling.

Table 38.3-2 - Hand wet lay-up: Selection factors

Characteristic	Typical Limits
Minimum inside radius	6.4 mm
Moulded-in holes	Large
Trimmed in mould	Yes
Undercuts (split mould)	Yes
Minimum draft recommended	2° (0.035 rad)
Minimum practical thickness	0.76 mm
Maximum practical thickness	Virtually unlimited
Normal thickness variation	± 0. 50 mm
Maximum thickness build-up	As desired
Corrugated sections	Yes
Metal inserts	Yes
Limiting size factor	Mould size
Metal edge stiffeners	Yes
Bosses	Yes
Fins	Difficult
Hat sections	Yes
Moulded-in labels	Yes
Raised numbers	Yes
Translucency	Yes
Strength orientation	Random or directional
Typical reinforcement content	25 to 65 wt. %

38.3.3 Prepreg lay-up

38.3.3.1 General

Fabrication of advanced composite structures needs the accurate placement of the reinforcing fibres, within a matrix, in an orientation that provides the engineering properties specified in the design.

Prepreg materials are supplied in a B-stage condition, in which the thermosetting resin has a level of tack. Thermoplastic prepregs do not have this inherent feature, [See also: 6.36 for processing methods for thermoplastic-based composites].

38.3.3.2 Basic processes

Layer after layer of UD prepreg tapes, or prepreg fabrics, are placed in a prescribed orientation, by:

- Direct lay-up, where plies are laid up directly on ply, without separate templates, as shown in Figure 38.3.2.
- Indirect lay-up, where separate ply templates with subsequent stacking of the plies, as shown in Figure 38.3.3.

It is common practice to pre-assemble plies on special plastic templates. This procedure is performed in a controlled environment.

Table 38.3.3 shows the detailed process steps for prepreg lay-up.

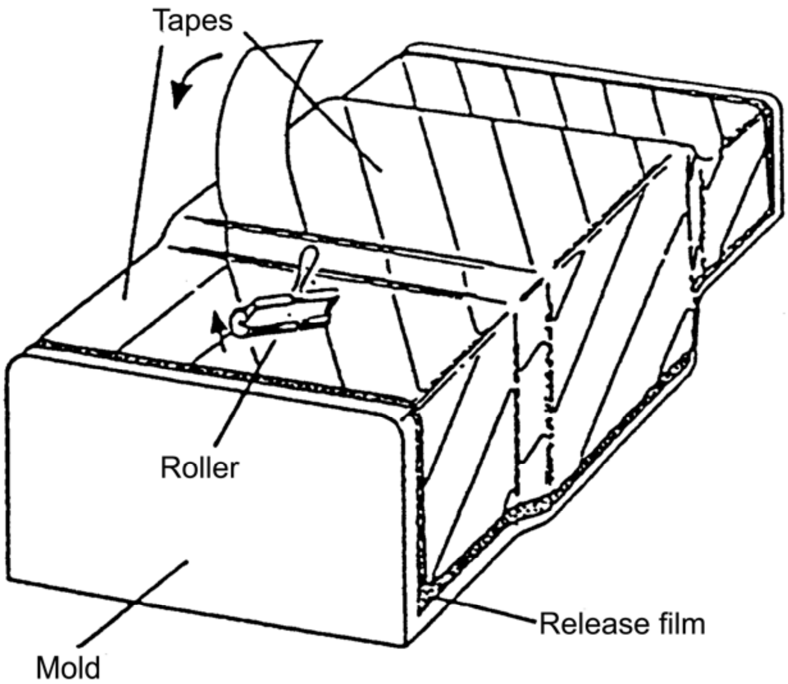


Figure 38.3-2 - Prepreg: Direct lay-up

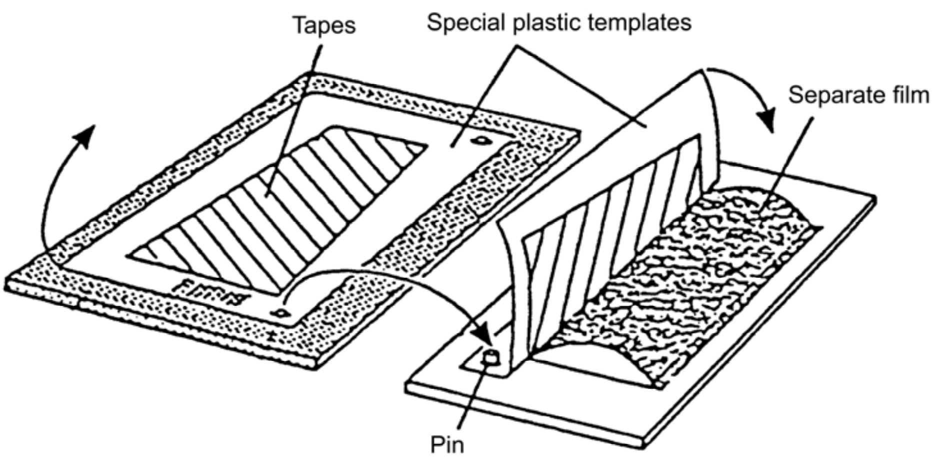


Figure 38.3-3 - Prepreg: Indirect lay-up

Table 38.3-3 - Process steps for prepreg lay-up

Stage	Process Steps
Preparation of individual plies	(1) Allow sealed prepreg package to warm to RT before opening; to avoid moisture condensation. (2) Record reel/sheet/batch numbers as it is used. (3) Wipe exposed templates with solvent on clean lint-free cloth, air dry. (4) Unroll sufficient prepreg for one strip. (5) Cut to required length using clean scissors or sharp knife. (6) Place prepreg on template. (7) Repeat (4) to (6) ensuring no gaps occur between adjacent strips within the ply (layer). (8) Work prepreg against template to insure good contact. (9) Remove separator strip (backing film). (10) Inspect each ply for: - fibre damage, - overlaps or gaps, - foreign matter contamination. (11) Repeat (4) to (10) for all plies. (12) Store finished lay-up flat in clean, moisture proof film. **Caution**: Minimise the time prepregs are kept at RT.
Preparation of mould	(1) Clean, to remove all foreign material. (2) Polish, to create smooth surface. (3) Solvent-wipe and air dry. (4) Apply release (parting) agent. (5) Apply non-porous coated fabric, if part is not to be cured in contact with mould.

Stage	Process Steps
Transfer lay-up to mould	(1) Apply peel ply, if required. (For adhesive bonding or painting). (2) Align template on tool, noting fibre orientation. (3) Remove air trapped between plies, with roller or squeegee. (4) Remove template, taking care if prepregs are very tacky. (5) Inspect for damage and contamination. (6) Repeat (1) to (5) until all plies are assembled. **Caution**: Document each ply set.
Preparation for cure	(1) Cover lay-up with perforated release material. Do not extend over edges. (2) Assemble and seal boundary supports (dams) around edges.

38.4 Autoclave moulding

38.4.1 Use

Consolidation of stacked thermosetting prepregs by autoclave curing is the most widely used means of preparing composites with a high fibre volume fraction. With autoclave chambers available up to 4 m diameter, there are few size limitations on manufacturing composite sections for satellites and launchers.

The capital investment in an autoclave facility is high and processing times are long. Therefore, access to an optimum-sized autoclave (from a cost perspective) can be a major criterion in a successful project.

38.4.2 Basic process

B-stage prepregs are heated gradually to induce a controlled resin flow and assist in the elimination of entrapped air and volatiles. Pressure is applied over a bag membrane to induce full consolidation before resin gelling occurs. Full curing is achieved by maintaining the bag under pressure at elevated temperature.

There are a number of variations to the basic process. These are often represented by plots of time against applied pressure and temperature.

The possible variables within the process include:

- Debulking,
- Heating (ramp) rates,
- Type of mould material,
- Application of positive or vacuum pressure to the bagged moulding,
- Dwell periods,
- Controlled bleed or zero-bleed prepreg systems,
- Peel Plies,
- Co-Curing,
- Cooling rates and pressure release.

Post-curing of the laminated component can be undertaken after the part is removed from the mould.

38.4.3 Bag moulding methods

38.4.3.1 General

Bag-moulded fibre reinforced resin-matrix composites and bonded structures can comply with the different standards of quality for different design criteria. The bag moulding process can be adapted and optimised to produce composites which perform adequately, and compete with alternative types of construction.

Innovative adaptations of bag moulding processes are often made to create single, complex, composite structures, rather than several separate parts which then need joining together, i.e. reduced part-count.

38.4.3.2 Autoclave process

A prepreg stack, with all of the necessary release layers, is draped over a mould. A polymer membrane is then sealed to the mould surface, away from the prepreg, to form the bag.

In the autoclave chamber, the bag is placed under vacuum, so that the bag and prepreg are pulled-down against the mould surface. Positive external pressure is applied to the moulding by pressurising the autoclave chamber. The advantage of an autoclave is the ability to apply a 0.69 MPa or about 6 bar (100 psi) positive pressure to augment the 0.1 MPa (15 psi) from the vacuum. This enables low voidage composites to be made from high fibre volume fraction prepregs, without the need for matched die, pressure tooling.

38.4.3.3 Modified processes

Bag moulding can be modified to enable a positive pressure to be applied during consolidation without use of an autoclave. Some of the process modifications include:

- Moulding without an additional external pressure, i.e. with only the 1 bar (15 psi) atmospheric pressure from the vacuum as the consolidating force. This relies on good resin flow and bled prepregs to ensure the elimination of voidage. Fibre volume fractions can be lower than by full pressure consolidation.
- Using the mould as part of the pressure containment, or
- Modifying a platen press to enable gas pressurisation.

The various processes illustrated are:

- Vacuum bag in Figure 38.4.1, Ref. [38-2].
- Pressure bag in Figure 38.4.2 and Figure 38.4.3 for press modifications, Ref. [38-2].
- Autoclave moulding in Figure 38.4.4, Ref. [38-2].

In Figure 38.4.1 and Figure 38.4.4, the arrangements shown are for preparing flat laminates.

For complex shapes produced by these methods, the use of rigid caul plates is often not feasible.

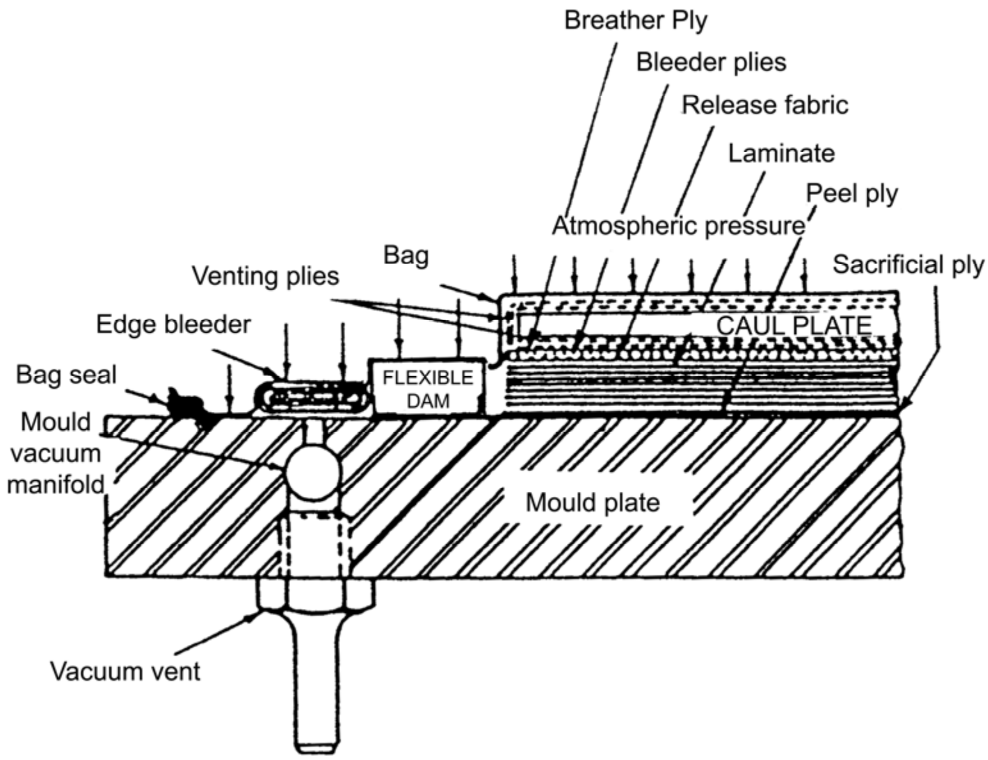


Figure 38.4-1 - Vacuum bag moulding method, with vertical bleeder

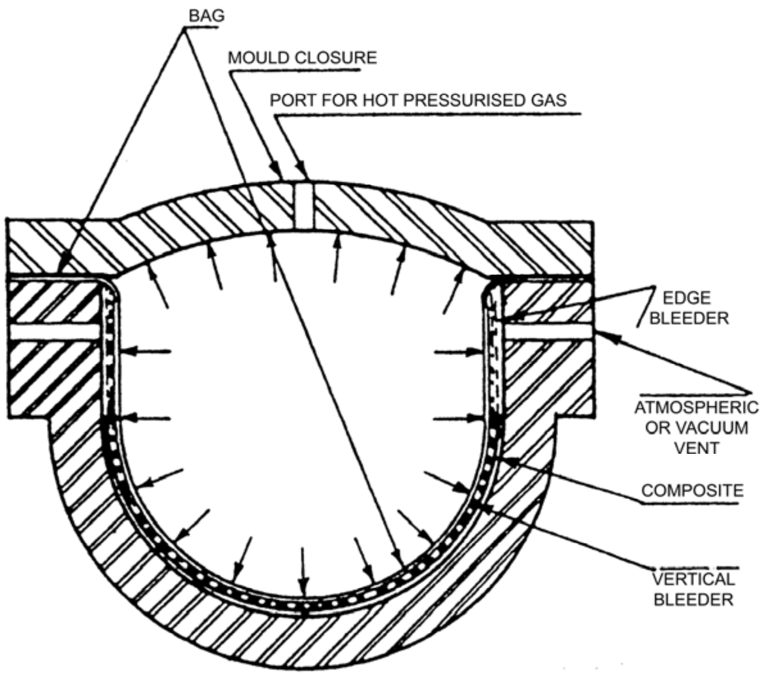


Figure 38.4-2 - Pressure bag moulding

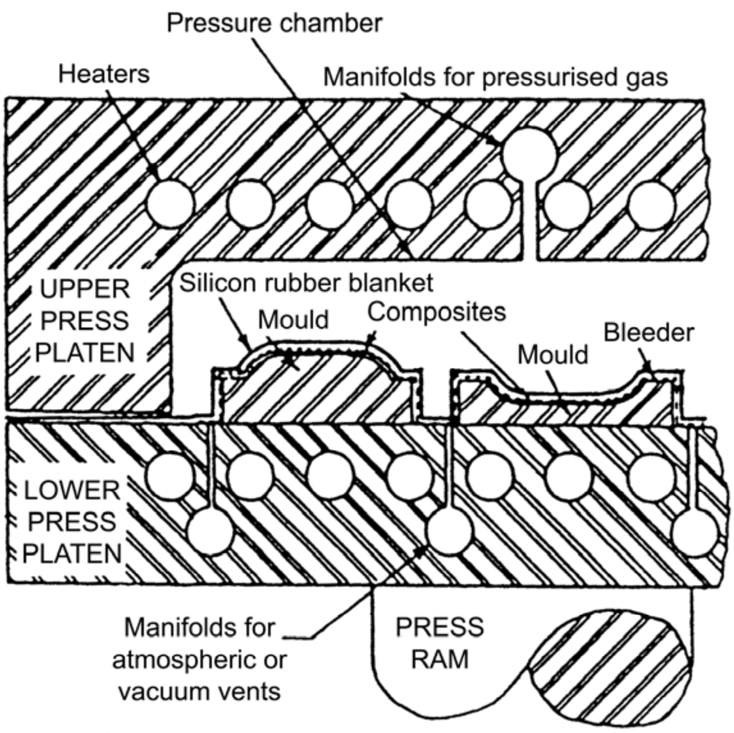


Figure 38.4-3 - Pressure bag moulding: Press modifications

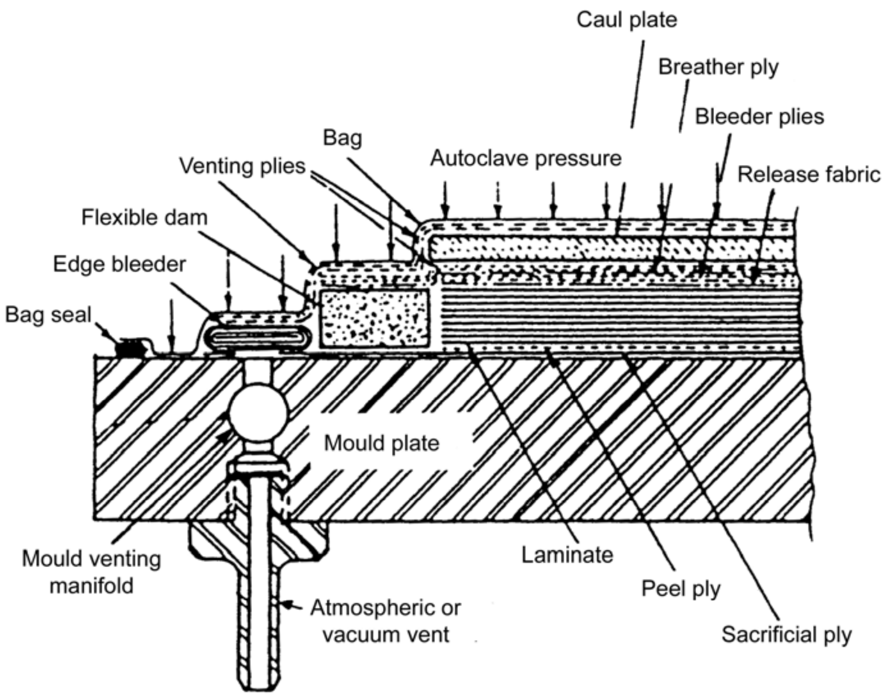


Figure 38.4-4 - Autoclave moulding method, with vertical bleeder

38.4.4 Features of bag moulding

38.4.4.1 Bags

Bags are thin, flexible membranes or silicone rubber shapes, which separate the lay up from the pressurising gases during the composite cure. Consolidation and densification of the lay-up is achieved by the resulting pressure differentials across the bag contents. The various types are:

- Vacuum bags: Consolidation and densification of vacuum bag mouldings can be achieved by atmospheric pressure alone as the bagged lay ups are evacuated during the cure cycles. Vacuum bag moulding is the least limited as to the size which can be processed.
- Pressure bags: Pressure bagged and autoclave cured composites are pressurised by hot gases. Vents to the atmosphere, or vacuum, provide for the escape of volatile reaction by products and the entrapped air from the curing composites.
- Autoclave bags: The bagged lay ups in autoclaves are usually vented to a pressure lower than that applied to the bag.

  NOTE These are similar to pressure bags.

38.4.4.2 Consolidation

Consolidation is achieved when the separate prepreg plies within the lay ups, together with other adherends (if present), are bonded together.

38.4.4.3 Densification

Densification results in diminution of voids, and removal of excess resin, if appropriate.

38.4.4.4 Cure

Although vacuum bag moulded composites can be cured at room temperature, most are cured at elevated temperatures to improve the properties. The types of cure can be grouped as:

- Thermal cures: These are best attained in circulating air ovens, but can be achieved using infrared-heated or passive convection ovens.
- Alternative curing methods, [See also: 38.12]:
  - Induction,
  - Dielectric,
  - Microwave,
  - Xenon flash,
  - Ultraviolet,
  - Electron beam, and
  - Gamma radiation.

38.4.4.5 Other features

During cure, the various bag moulding methods:

- Prevent blistering in the composites,

- Control pressure and heat application, and
- Control the ratio of fibre-to-resin.

38.4.5 Bagging techniques

38.4.5.1 Debulking

Debulking reduces the possibility of porosity in critical areas. It can be necessary when preparing prepreg stacks with a large number of individual plies, or when complex shapes are involved. The process involves placing some of the uncured plies in the mould and forming them to the mould surface using a vacuum bag. The bag is then removed and further plies added to the mould stack. If necessary, it is repeated several times especially for thick laminates.

38.4.5.2 Vacuum bag moulding

The bags form a membrane enabling the air to be evacuated from the laminate and to generate the atmospheric pressure necessary for com-paction against the mould.

38.4.5.3 Autoclave bag moulding

The bags serve to apply the compacting gas pressure to the construction during the cure. The atmosphere within the bag is vented to a lower pressure to enable removal of trapped air and reaction products.

38.4.5.4 Zero-bleed and bleed systems

The need for a bleed system depends on the prepreg resin formulation, where:

- Zero-bleed prepreg systems have gained wider acceptance as a means of producing composites with predetermined fibre volume fractions. These prepregs have resins with optimised viscosity and gelation characteristics to avoid excessive resin flow to eliminate voidage.
- Controlled bleed prepregs remain and are used as a means of assisting consolidation.

Bleed out systems are designed to maintain the reduced pressures within the bag. If this is not done, gas pressure within the bag prevents consolidation.

The correct application of pressure achieves:

- Consolidation of the successive plies,
- Completion of the resin impregnation of the fibre,
- Elimination of:
  - void-creating volatiles,
  - reaction by products, and
  - entrapped air.
- reduction of the excess resin in the lay-up.

The bagged lay-up includes the bleed-off system designed for the composite part. Bagged lay ups can be vertically or edge bled.

The classical differences between the two are illustrated for:

- Vertical bleed, [See: Figure 38.4.4].
- Edge bleed in Figure 38.4.5, Ref. [38-2].

Vertical bleed is usually more efficient than edge bleed.

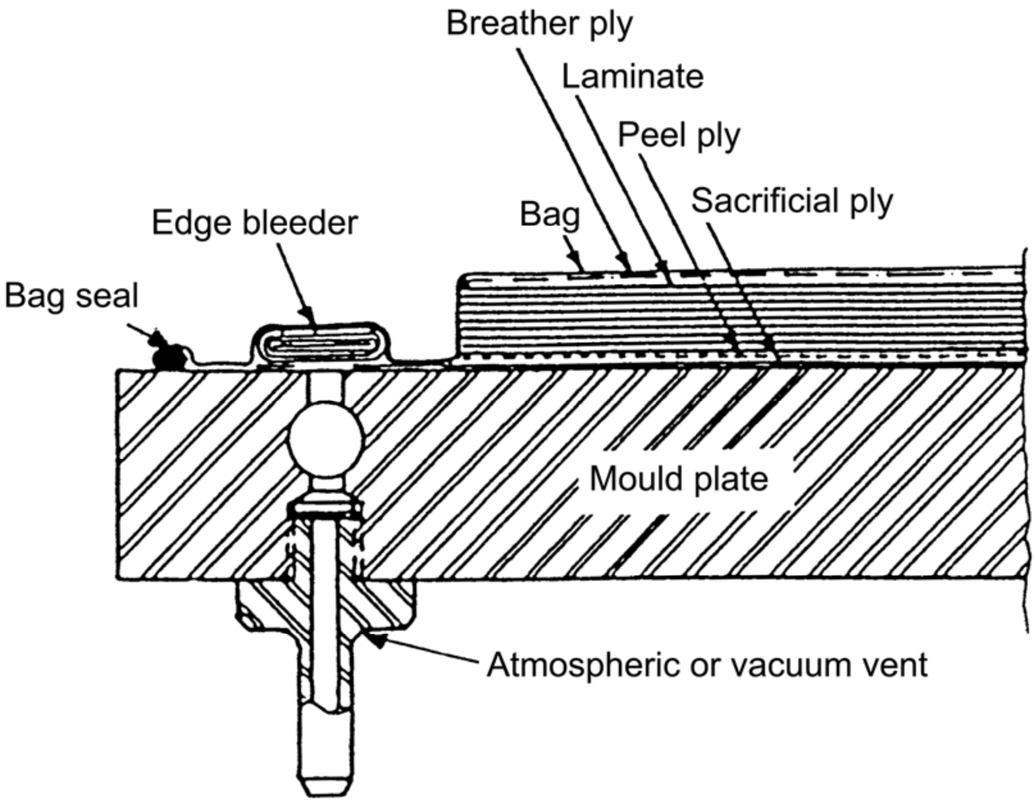


Figure 38.4-5 - Bag moulding: Edge bleed out system

38.4.6 Tool materials

38.4.6.1 General

Various mould or tooling materials are used, notably steel, aluminium, composites and Invar.

Each has its own merits and the choice is usually influenced by a combination of the:

- Mould cost, e.g. material, shape, precision.
- Thermal conductivity,
- Thermal expansion: matching CTE (tool material) with CTE (moulding material),
- Durability (reuse).

The merits of various tool materials are shown in Table 38.4.1. The comments apply to CFRP mouldings.

Table 38.4-1 - Tooling for composites: Relative merits of various types for CFRP

Factor	Steel	Aluminium	CFRP	Invar
Cost	Low	Medium	Medium/High	High
Thermal Conductivity	Medium	High	Medium/Low	Medium
Thermal Expansivity	Medium	High	Low	Low
Mould mass	High	Medium	Low	High
Durability	Good	Reasonable	Reasonable	Reasonable

38.4.6.2 Steel

Steel moulds are used because it is a low-cost material, easily machined and durable. For manufacturing accurately dimensioned CFRP components, the CTE difference between steel ($+15 \times 10^{-6}$ /°C) and CFRP (from -1 to $+3 \times 10^{-6}$ /°C) can be too great. Steel expands appreciably during heating to the cure temperature and then contract to a greater extent than the moulding on cooling.

38.4.6.3 Aluminium

Aluminium has good thermal conductivity, which assists in obtaining an even temperature distribution. However, its thermal expansion is very high ($+23 \times 10^{-6}$ /°C). It is also more easily damaged than steel and can suffer thermal distortion with repeated heating and cooling up to 180°C; a typical processing temperature for thermosetting CFRP.

38.4.6.4 Composite (CFRP)

By using CFRP for the mould tool, a close CTE match can be achieved with the moulding. Consequently, the dimensions of the mould change little during the processing cycle. As CFRP tools cannot be machined from stock material, they are moulded from a master. This in turn is achieved by machining a replica of the final component surface and preparing a mould from it. The replica is usually steel or machinable ceramic. The tool can be made using low temperature curing epoxy prepreg, which is post-cured to give a tooling CFRP with a high glass transition temperature. CFRP tooling should be carefully stored and handled to prevent distortion or damage. There is often a limit on the number of elevated-temperature mouldings which can be taken from a single CFRP tool.

38.4.6.5 Invar

The CTE of Invar is 0.9×10^{-6} /°C, which is similar to that of CFRP components. The high material cost and low thermal conductivity detract from its wide-scale usage.

38.4.6.6 Other tool materials

Other materials are occasionally used, including:

- Graphite
- Cast ceramic

These can be easily shaped but are not particularly durable. They can be used for prototype moulds.

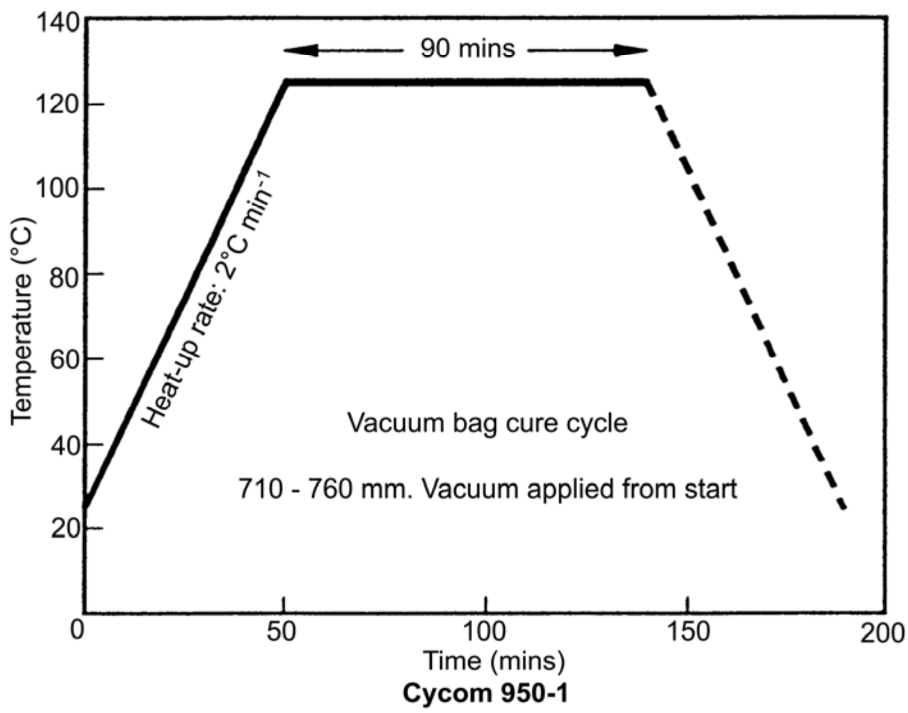
38.4.7 Cure schedules

A typical cure schedule for the moulding of thermosetting CFRP is given in Figure 38.4.6.

Cure schedules are usually recommended by prepreg manufacturers, based on the preparation of simple, flat laminates.

For complex-shaped mouldings, it can be necessary to modify the recommended cure schedule to produce acceptable quality laminates. This can be achieved through experience of a particular prepreg or by evaluation studies for a new prepreg system.

The critical aspect of any cure schedule is the total time. This is determined by establishing the optimum conditions for consolidating the prepreg to produce void-free composites of the correct fibre volume fraction. The total cure cycle cannot be too long or too short.



Comments: For pressured cure cycle apply pressure from start.

For vacuum cured thick sections (>3.2 mm), insertion of a 60 minute dwell at 100°C is recommended.

Figure 38.4-6 - Typical cure schedule for autoclaved prepregs

Features of the cure schedule are:

- Temperature ramp-rate, usually varies between 1 and 6 °C/min. It is determined by the:
  - gel characteristics of the resin,
  - resin viscosity (zero-bleed or controlled-bleed systems),
  - thermal inertia of the autoclave, assembled tooling and moulding.
- Dwell periods, which are used to eliminate voidage by enabling resin flow prior to gelation. There is an incentive to reduce or avoid dwell periods in order to reduce the total elapsed time.

- Time at maximum temperature, which is determined by the extent of curing (crosslinking); typically from 2 hours to 4 hours.
- Cool-down rate, which is largely dictated by how quickly the autoclave and tooling assembly can be cooled to the point where handling the moulding is possible, taking into account that:
  — uneven cooling of a moulding is can cause distortion.
  — vacuum is maintained on the moulding during cooling and possibly some positive overpressure on the bag.

Although reducing the maximum temperature in the autoclave can reduce residual stresses in the cured moulding, it often results in an extended cure cycle in order to obtain the same crosslink density in the cured resin.

38.5 Filament winding

38.5.1 Basic process

Filament winding was one of the earliest techniques used to produce composite material structures. This process uses continuous strands of glass, carbon or aramid fibres which are continuously impregnated with a low flow resin. The strands are then wound onto a mandrel, which provides the final geometry.

38.5.2 Applications

Various structures can be fabricated using this technique, including:

- Cylindrical tubes; the most common application, Ref. [38-4].
- Containers for fluids, gases or solids. These can have openings at their poles.
- Flat panels, by first winding on a mandrel, then cutting the finished lay-up along the length of the mandrel, flattening and curing under pressure.
- Geodesic lattice structures.

Filament winding provides an efficient means of obtaining optimised pressure vessel structures, [See also: Chapter 29].

Depending on the applied loads, weight savings can be obtained by changes to:

- fibre angle, or
- thickness of lay-up.

Modern winding machines, known as winding robots, can produce very complex geometry parts by using non linear winding programs.

38.5.3 Winding process

The main process steps consist of:

- Fibre preparation: After the choice of the appropriate fibre, given the defined requirements for the structure, fibre batches are:
  - dried, in an oven at about 130°C for several hours.
  - stored, at 60°C for some days.
- Fibre impregnation, by continuously pulling fibres through a bath of resin, as shown in Figure 38.5.1. To control the resin content, the impregnated fibres are squeezed through a pair of rollers.
- Tensioning: The impregnated strand passes through rollers to provide a certain pre-tension in the fibre.
- Strand guidance: The strands are passed through a guide, and then to the mandrel.
- Winding, of the structure.
- Pre-cure: The final filament-wound structure is then heated by infrared light to about 80°C in order to pre-cure the resin.
- Final cure: After pre-cure, the structure is placed in an autoclave to perform the final curing at normally 130°C for several hours. The cure temperature depends on the particular resin system.
- Remove mandrel: Depending on the type, [See also: Mandrels]:
  - Re-usable are extracted from the structure, or
  - Lost are broken and removed.
- Post-cure, if necessary.

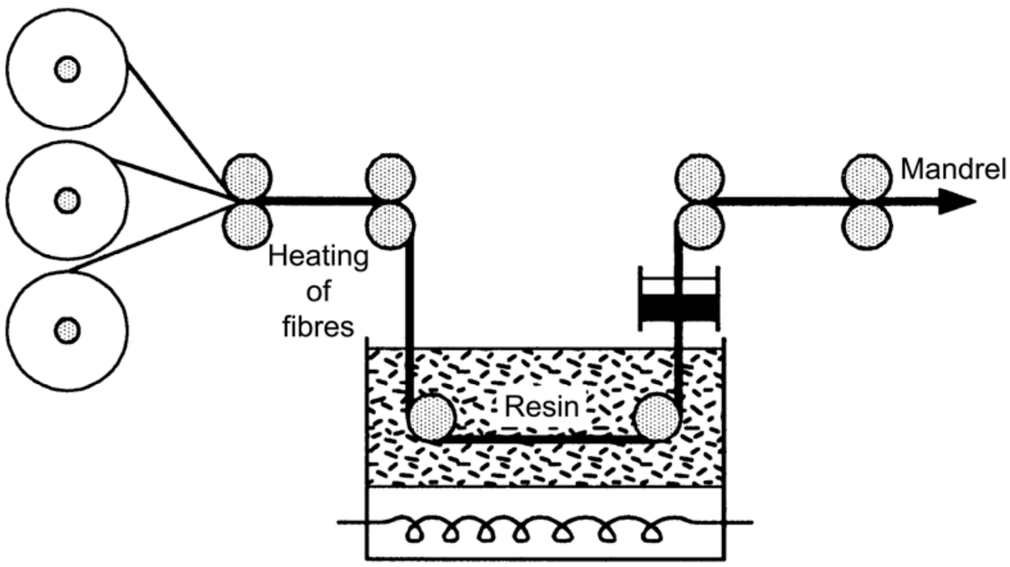


Figure 38.5-1 - Filament winding: Impregnation of fibres

38.5.4 Mandrels

38.5.4.1 General

Geometry and quality of the inner surface of the filament-wound structure depend on the material and on the surface of the mandrel used. Factors to be considered include:

- extraction,
- material,
- surface preparation, and
- component shrinkage.

38.5.4.2 Reusable

For wound tubes, a mandrel of polished or chrome-plated steel is commonly used. Other materials, e.g. aluminium, need more effort concerning release agents. A collapsing mandrel is another possibility to enable its release from the structure.

38.5.4.3 Lost

For spheres, ellipsoid or cylindrical containers having a wound end, normally lost mandrels are used. These can be made from:

- Sand in a soluble binder,
- Gypsum,
- Foam, or
- Low melting point materials.

These mandrels need a final surface treatment to provide good release.

38.5.4.4 Integral

For applications that need an inner liner to prevent leakage or damage to the composite structure, e.g. pressure vessels. The mandrel can form part of the finished structure. This is also called over-wrapping, i.e. where the liner is used as the mandrel.

38.5.4.5 Component shrinkage

Most resin systems, and therefore the reinforced structures, tend to shrink after curing. This can make the mandrel difficult to remove. A mandrel with a higher coefficient of thermal expansion, relative to the composite structure, can be heated during the winding so that on cooling down it contracts away from the component, so aiding its removal.

38.5.5 Sandwich constructions

It is feasible to filament wind sandwich constructions. Cylindrical constructions have been manufactured by directly winding onto honeycomb material, [See also: 29.12].

For cylinders, the sequence used can be:

- Winding of inner skin onto mandrel,
- Bonding of honeycomb core onto inner skin,
- Over-winding outer skin to complete the cylinder.

38.5.6 Tape winding

Narrow, continuous widths of B-stage prepreg can be used instead of fibres wetted with uncured resin. The width of prepreg tape is limited when winding complex shapes to ensure that the fibres conform to the mould in an acceptable manner. This route is suited to winding cylinders, but not necessarily for direct winding onto honeycomb core material.

Tape winding is also applicable to thermoplastics, Ref. [38-6].

[See also: 6.26]

38.6 Filament-winding machines

38.6.1 General

Machines, designed to perform winding processes, are commonly either:

- polar - planar, or
- helical.

All machines are available in several variants, which add versatility and compensates for some of their weaknesses.

The development of computer-controlled, filament-winding robots has made complex winding patterns for large structures feasible, [See: 29.2].

38.6.2 Polar winding

Polar machines are generally used for the production of containers with diameter-to-length ratios greater than 0.5.

- Vertical mandrel: Polar winding machines normally have the mandrel mounted in a vertical position, which enables a simpler construction of the rotating arm because deflection of the mandrel is eliminated; as shown in Figure 38.6.1. This arrange-ment is limited to structures of moderate size.
- Horizontal mandrel: For large windings, the mandrel is supported in a horizontal position and the rotating thread guide places the fibres on the mandrel, as shown in Figure 38.6.2.
- Tumbler: A variation of the normal polar winding system is the tumbling process in which the thread guide remains stationary while the mandrel rotates or tumbles in a near horizontal plane, as shown in Figure 38.6-3. The inclination of the mandrel to the horizontal determines the winding angle.

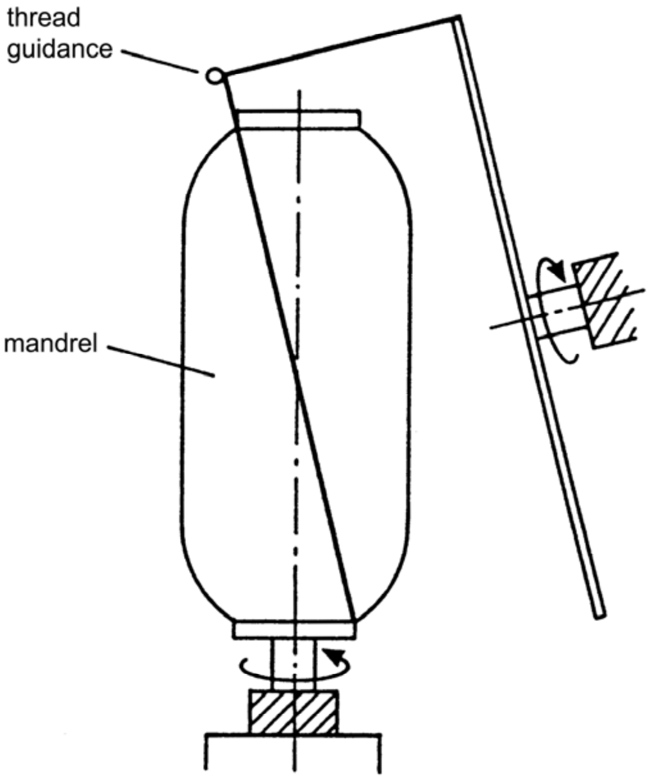


Figure 38.6-1 - Filament winding: Polar vertical

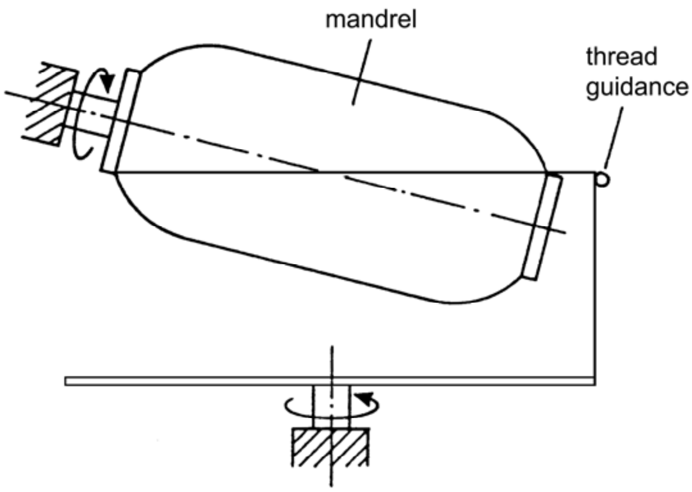


Figure 38.6-2 - Filament winding: Polar horizontal

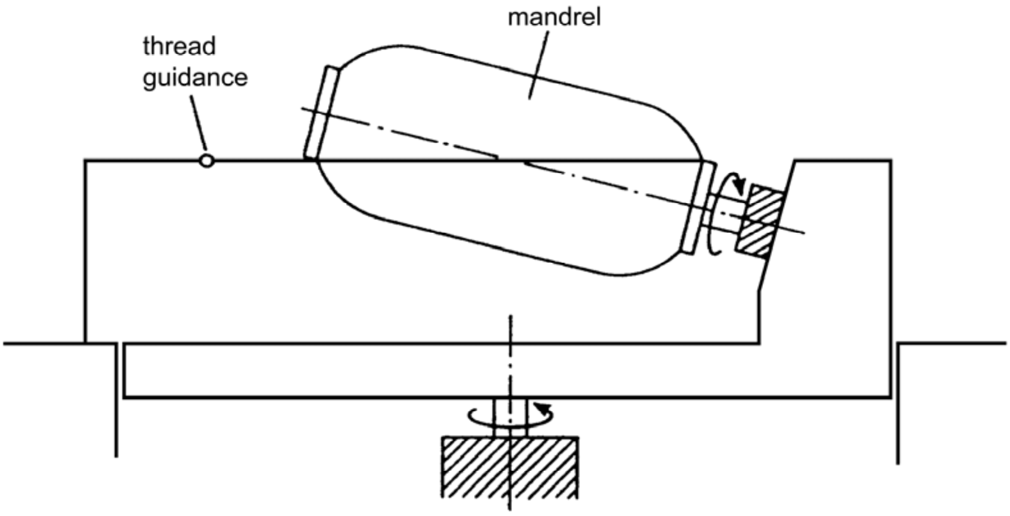


Figure 38.6-3 - Filament winding: Polar tumbling

38.6.3 Helical winding

Helical winding machines consist of a rotating mandrel and a reciprocating thread guidance carrier, as shown in Figure 38.6.4.

In addition a third motion perpendicular to the mandrel axis is applied to improve the fibre placement over the end domes.

To achieve optimum placement of the fibres on a geodesic path, sophisticated control of the thread guidance is necessary. The winding angle is determined by the ratio of mandrel rotation to the speed of the thread carrier.

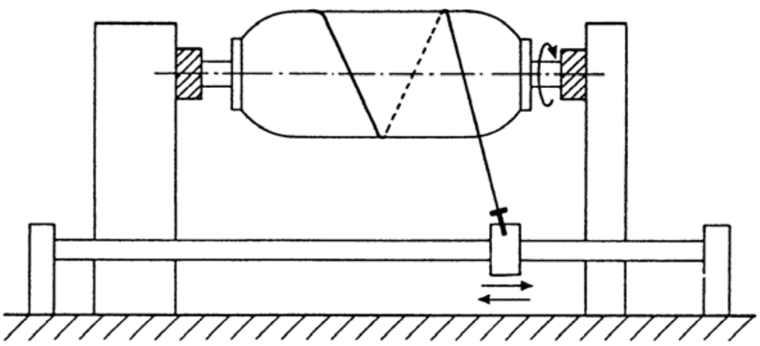


Figure 38.6-4 - Filament winding: Helical

38.7 Resin transfer moulding

38.7.1 General

Resin transfer moulding, RTM, is a lower cost component production process than prepreg lay-up and autoclave curing. Certain complex shapes can be more readily made by RTM than by other moulding routes, Ref. [38-7], [38-8].

38.7.2 Basic process

A fibre preform, formed to the intended component shape, is placed in a matched mould, which is then closed. A low viscosity resin is injected into the mould and all the fibres wetted through until the mould is filled. The resin is injected under pressures of about 0.69MPa (100 psi), with or without vacuum assistance in the mould cavity. A heated curing cycle is applied. The cure conditions are dependent on the resin used.

The similarities between RTM and resin injection moulding, RIM, can make it difficult to distinguish between them.

The RTM process is a simple concept, as shown in Figure 38.7.1. However, the processing parameters should be carefully controlled to ensure high quality net-shape components.

Proprietary technology packages are becoming available which match the resin and fibre preform technology. An example is the 'Injectex' system from Brochier-Ciba Geigy.

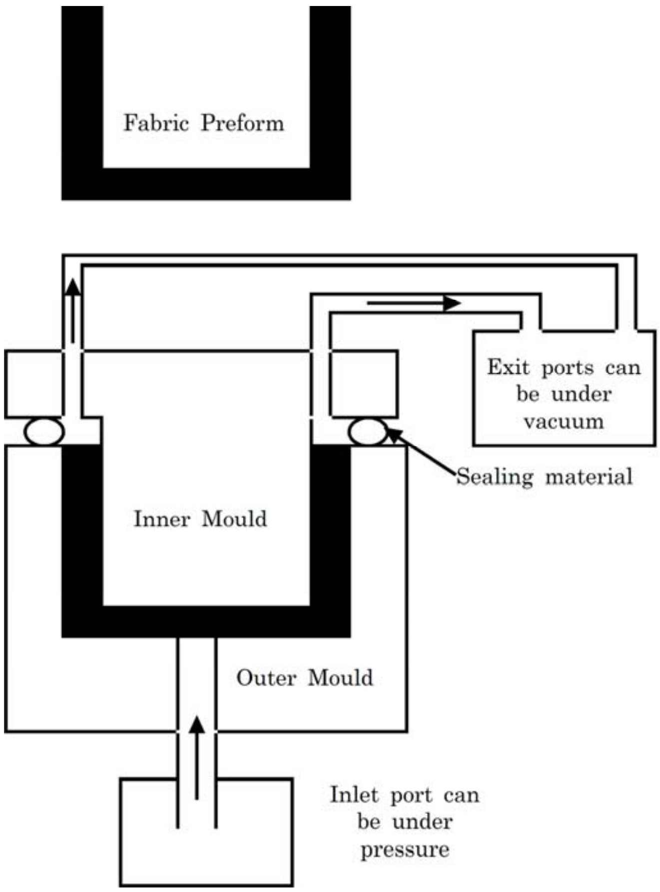


Figure 38.7-1 - Resin transfer moulding (RTM): Process

38.7.3 Applications

Within industry generally, RTM is applied where the component production numbers are high; typically measured in hundreds or thousands per year. This is not normally the case with space structure manufacturing which rarely exceeds ten units in total, except for launchers, e.g. Ariane series, or for elements in lattice structures.

The attraction of the process for space applications is closely linked with the need for complicated component shapes, i.e. those which cannot be readily produced by other composite moulding routes. Some examples of such components include:

- nose cones,
- radomes,
- integrally-stiffened panels,
- complex shaped ducting,
- CFRP adapter rings; as for Ariane launchers, Ref. [38-9],
- 'J', 'T', 'C, and 'I' structural shapes, Ref. [38-10],
- braided rings and tubes, Ref. [38-11],
- nozzle components, Ref. [38-12].

RTM is most practical for thicknesses in the range of 2 mm to 12 mm and not for very thin sections. Tolerances on wall thickness depend on the accuracy and stiffness of the mould tooling. Typically wall thickness can have a tolerance of ±0.25 mm. Fibre volume fractions are usually slightly lower than those achieved with pressure consolidated prepregs.

Polymer composite mouldings made by RTM are used for preparing high-temperature composites. Using pyrolysis, the moulding (green part) can be converted to carbon-carbon or SiC-based constructions. These preforms are subsequently densified by further infiltration methods, e.g. CVD, CVI or further resin additions.

[See: Chapter 88 for manufacturing techniques used for high-temperature composite components]

38.7.4 Fibre preforms

38.7.4.1 General

The development of versatile and cost-effective fibre placement technology is central to RTM manufacturing as achieve high-productivity.

The use of machinery to create a net-shape preform with all the fibres in the desired orientations and quantities is essential to avoid costly manual labour. Preforms can be produced from:

- Woven fabrics,
- Tubular or flat braiding,
- Stitched layers,
- Multidirectional weavings,
- Knitted sections.

Fibre volume fractions can be varied between 30% and 70% by volume, but 50 volume % is typical. Both carbon and glass fibres are used.

38.7.4.2 Binders

A polymeric binder is often used to hold the preform together. Such binders have to be compatible with the matrix resin and assist resin impregnation.

38.7.4.3 Cores

Foam cores can be incorporated with the fibre preform to provide support for thin wall constructions.

38.7.5 Resin injection

38.7.5.1 General

A new generation of thermosetting resins are formulated specifically with optimum viscosity and cure characteristics for RTM processing. These include:

- Epoxies,
- Bismaleimides,
- Others, including: cyanate esters, phenolics and silicones.

38.7.5.2 Epoxies

Examples of epoxy resins formulated for RTM processing include:

- Shell RSL-1895/W, Ref. [38-13], [38-14] and DPL 862/RSC-763 (two-part liquid epoxies),
- Shell Epon 9400/9450 and 9405/9470, Ref. [38-11],
- Hercules HBRF-311 and HBRF-314,
- 3M PR-500 (hot-melt),
- Dow Plastic DER 300 (hot-melt) and Tactix 123,
- BP E905L, Ref. [38-14],
- Ciba Geigy XU MY722/RD 91-103, Ref. [38-15].

38.7.5.3 Bismaleimides

Examples of bismaleimide resins formulated for RTM processing include:

- X206-44 (Dow-United Technologies) based on BASF 5250-4 prepreg resin (hot-melt),
- Modified Matrimid 5292 (Ciba Geigy), Ref. [38-16],
- Compimide 65 FWR (Technochemie/Shell), Ref. [38-17],
- XNF-650 (Hexcel).

38.7.5.4 Others

Other chemical groups of resins for RTM processing include:

- Cyanate esters, e.g. Dow XU71787, Ref. [38-18], [See also: 6.35].
- Phenolics, Ref. [38-12],
- Silicones, Ref. [38-12].

38.7.6 Process variables

38.7.6.1 General

The process variables can be summarised as, Ref. [38-19]:

- Resin viscosity (initially 25 cps to 50 cps) depending on:
  - temperature,
  - pot life.
- Dry fibre preform and binder have to be wettable by liquid resin, e.g. 'Injectex' system.
- Application of pressure and vacuum to control resin infiltration.
- Position and number of inlet and outlet ports.
- Initial degassing of resin.
- Evacuation of air from preform.
- Tooling material for matched male and female mould parts.
- Mould clamping and sealing.
- Mould temperature and post-cure.

38.7.6.2 Resins

The resin can be either a one-part system (hot-melt, similar to prepreg formulation) or two-part (liquids mixed at the point of use). One-part systems are normally used for aerospace components, Ref. [38-20], because this enables:

- greater confidence in chemical composition from the resin system supplier,
- similar cured-resin performance to that achievable by prepregs,
- room temperature storage for up to 6 months.

38.7.6.3 Resin flow modelling

For successful mouldings, the flow of resin into the mould cavity and preform should be optimised, Ref. [38-21], [38-22]. Problems to avoid are:

- Gelling of the resin before the preform is completely filled.
- Incomplete resin flow, leaving trapped air and hence voidage.

Such defects are limited, largely by trial and error, if a large production run is anticipated. For very limited unit production numbers, the process needs to be optimised at the planning stage to avoid unsuccessful prototypes. Consequently, computer-aided design tools are often used to model the process, Ref. [38-21].

38.7.7 Process advantages

The individual process advantages of RTM are not necessarily unique. But taken as a whole, RTM can be appropriate for some component designs. The characteristics of RTM include:

- Preforms enable multi-directional reinforcement placement to improve damage tolerance.
- Good surface detail and accuracy.

- Complex shapes, with integral stiffening, can be made in a single moulding.
- Near net-shape parts needing minimum machining.
- Reinforcement lay-up is dry instead of tacky prepreg.
- Refrigerated shipping and storage is not always necessary.
- Close process control can reduce the need for non-destructive inspection.
- Sections having variable thickness are possible.
- Total process times are reduced compared with autoclaving.

38.8 Pultrusion

38.8.1 Use

Pultrusion is a well-established, industrial manufacturing process for both thermoset and thermoplastic composites. It is similar to the extrusion of metal alloys and plastics. The process is ideally suited to the continuous manufacturing of constant cross-section profiles, in hundreds or thousands of metres (total). There are, however, few space applications which consume such large quantities of pultrusion in a single, standard size. The process can be useful for applications, such as:

- Tubes for large lattice structures,
- Profiles for constant section rib stiffeners,
- Ducting, where off-the-shelf pultruded sections are suitable.

Continuous sections up to 1m wide can be made by pultrusion, including integral stiffeners and double-skin constructions.

In the early 1980s, NASA examined the possibility of using an in-orbit pultrusion facility to produce thermoplastic composite sections in space rather than transporting formed lengths from Earth. The sections were then to have been assembled to form the main structural elements of a space station.

38.8.2 Basic process

38.8.2.1 Thermosetting resins

The basic process is shown diagrammatically in Figure 38.8.1. The process steps are:

- Spools of fibres (sufficient for the production run) are purchased and arranged to make up the necessary fibre fraction in the final composite section.
- Fibre tows are gathered and drawn through a resin bath and impregnated.
- Impregnated fibres are continuously drawn (maximum 1m/min) through a heated die of the desired cross-section to shape the composite.
- A long die is used, which incorporates appropriate heating elements.
- The total elapsed time within the heated die region is sufficient to fully cure the resin.

NOTE Selection of the resin is important in determining the production rate.

- Haul-off equipment takes the finished product away from the die, and the pultrusion is cut to specified or manageable lengths.

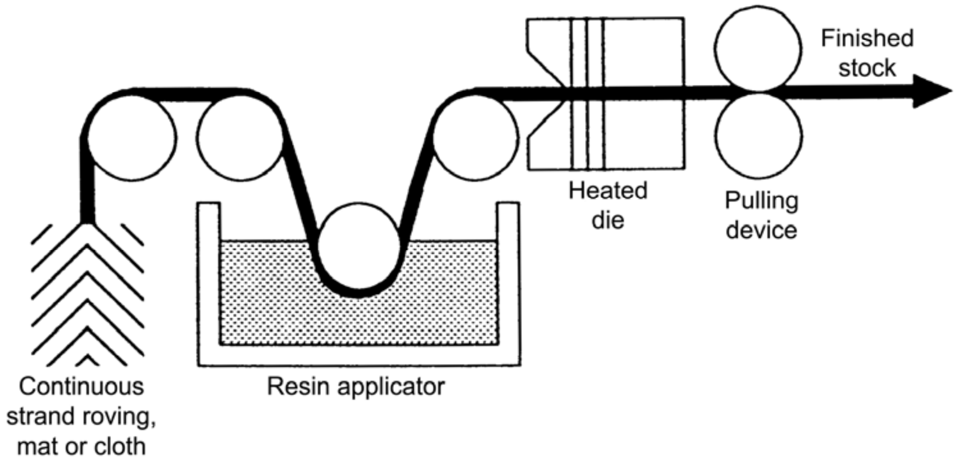


Figure 38.8-1 - Pultrusion: Basic process

The start-up costs can be high, because of the:

- Complex heated, shaped die, and
- Procuring sufficient spools of fibre for the cross-sectional area of pultrusion to be made; cost depends on the type of carbon fibres specified.

38.8.3 Pull-forming

38.8.3.1 General

This is a modified pultrusion process incorporating direct shape moulding.

38.8.3.2 Process

Fibre and resin are combined by drawing them through a shaped die to the desired length. That length is then enclosed in a mould and formed to the final shape by application of direct heat and pressure. The fibres are cut as the mould closes. This method enables changes in section to be made in the moulding. A high degree of automation is retained because the continuous drawing process can be operated using a number of moulds.

38.9 Table rolling

38.9.1 Tubular sections

Table rolling can be used for producing finite lengths of cylindrical or fine tapered tubular sections.

The process can be applied to:

- unidirectional thermosetting fibre prepregs for small diameter sections,
- fabric prepregs for larger diameters.

Prepreg is rolled onto a steel mandrel former and bagged for autoclave moulding. The process needs careful control to prevent ply rucking on small diameter sections. This can occur during the consolidation as the rolled prepreg compacts to the final diameter.

The wall thickness of the sections produced can vary because the prepreg ends are overlapped.

38.10 Pressure and mechanical forming

38.10.1 General

Consolidated laminates of some types of composite materials can be shaped by plastic deformation, i.e. pressure or mechanical forming. The materials this can be applied to are:

- FML 'fibre metal laminates', [See: 46.1.5 for materials]
- Thermoplastic matrix composites, [See: 6.26].

Variations of the basic pressure forming technique are used for these main types of materials.

The amount of plastic deformation is restricted by the:

- Limited retention of laminate integrity.
- Control of reduction in thickness.
- Shape complexity, i.e. multiple curvatures and deep recesses.

38.10.2 Fibre metal laminates

ARALL™, GLARE™ and CARE™ are FML materials, [See: 46.17]. These are hybrid laminates containing metal and thermoset composite. FML can be formed as if they were ductile metal sheets. Appropriate process techniques include:

- Roll forming,
- Brake press bending,
- Rubber-backed bending,
- Stretch forming, and
- Rubber pad forming.

38.10.3 Thermoplastic composites

38.10.3.1 Continuous fibre-reinforced composites

The processing options include:

- Press claving,
- Diaphragm moulding,
- Hot or cold forming, and
- Deep drawing.

[See: 6.26 for process descriptions]

Diaphragm moulding, in conjunction with a modified autoclave, has produced parts with complex curvatures, Ref. [38-23]. For example: PEEK APC2 aircraft spoiler sections, up to 2.2 m long, have been made within 20 minutes.

[See also: 38.11 for moulding of short-fibre reinforced thermoplastic composites]

38.11 Injection moulding

38.11.1 General

Injection moulded is usually associated with the high-volume manufacture of industrial and consumer products, i.e. large number of identical parts. Occasionally, injection-moulded, thermoplastic components are used in space applications, but the high mould costs need to be justified. It can be feasible for small attachments, where other composite processing routes are not practical.

38.11.2 Short fibre composites

38.11.2.1 General

For mass-efficient designs, glass or carbon fibre-reinforced polymers are preferred. These contain about 30% of reinforcement phase in order to provide adequate stiffness. The matrix is normally a high-performance engineering thermoplastic, such as PEEK, PEI or liquid crystal polymers, LCP, Ref. [38-24].

[See: 6.17 for further information on thermoplastic matrices]

38.11.2.2 Complex shapes

Glass fibre-reinforced LCP liquid crystal polymer components have been used in a space programme where good thermal insulation properties were needed, Ref. [38-8].

The items targeted for injection moulding were:

- High flow latch bracket,
- Cantilever pipe support,
- Pyro valve,
- Pressure transducer,
- Fill and drain valve,
- Pipe support, and
- Liquid filter.

38.12 Radiation curing

38.12.1 Process developments

38.12.1.1 General

Ionising radiation, such as EB electron beam, X-ray or Gamma-ray are mostly used for:

- Sterilisation of food or medical devices,
- Crosslinking of thermoplastic-based material,
- Drying of coatings and inks.

38.12.1.2 Composites

The use of heat in elevated-temperature processes is the accepted method for cross-linking (curing) of thermosetting resin formulations.

With the overall aim of improving process efficiency, there is an increasing interest in using different forms of electromagnetic energy in order to obtain crosslinking, Ref. [38-25] to [38-34].

Resins are formulated to be receptive to radiation energy and so achieve efficient crosslinking.

Using energy sources, other than heat, within industrial processes for composites is a fairly recent innovation, so equipment with a proven capability is uncommon.

The broad categories of radiation applied to composite resin curing are:

- Electron beam, EB.
- Microwave.
- Ultraviolet light, UV.

38.12.1.3 Potential benefits

The possible benefits of using electromagnetic radiation curing compared with conventional thermal processing include:

- Reduced process times, i.e. rapid cure,
- Reduced energy consumption,

- Lower process temperatures,
- Reduced internal stresses in the cured composite,
- Improved polymerisation,
- Reduced retention of volatiles.

38.12.2 EB electron beam curing

38.12.2.1 Thermosetting resins

Use of EB-curing for composite materials is still limited. However, the technology offers numerous significant advantages over conventional heat curing processes, including:

- EB techniques offer the capability of curing very large structures 'out-of-autoclave', using low energy beams coupled with winding machines.
- Complex shapes and assemblies can be processed easily
- Thermal gradients in thick composite parts are more manageable.
- Tooling is less expensive due to the lower temperatures that they experience.
- Shelf-life of most curable formulations is very advantageous.
- VOC emissions are reduced.
- Processing times are shorter.
- Energy costs are reduced.

38.12.2.2 EB-curing resin formulations

The interaction of ionising radiation with the precursor materials creates initiation sites for the subsequent chemical reactions. The nature of these reactions depend on the precise formulation of the resin. In general, three types predominate, i.e.:

- Polymerisation,
- Crosslinking,
- Material degradation by chain scission.

In the case of formulations with radiation-sensitive components (monomers or prepolymers), the interactions can lead to rapid polymerisation. The reactive resins can be broadly grouped by their polymerisation mode:

- Radical: mainly (meth)acrylate-based formulations,
- Cationic: epoxy resins.

Typical advantages and drawbacks of both systems are summarised in Table 38.12.1.

Table 38.12-1 – EB curing: Advantages and drawbacks of epoxy and acrylate resins

Process stage	Characteristics of EB curing formulations, by polymerisation mode			
	Free-radical (Acrylates)		Cationic (Epoxy)	
	Advantages	Drawbacks	Advantages	Drawbacks
Pre-cure properties	Low cost chemicals Shelf stability	-	Compatibility with thermoplastic additives	Poor initiator compatibility Low stability
During cure	No initiator needed Easier control of the reaction	Sensitivity to oxygen High volume contraction	Insensitivity to oxygen Low volume contraction	Sensitivity to bases and nucleophiles (water)
Post-cure properties	-	Brittleness Current carbon sizing not adapted	No termination	-

The curing mechanisms, the influence of formulation, dose, dose rate, dose application sequence and temperature on the curing process, reaction kinetics and network properties are well known, Ref. [38-40].

For some acrylate-based systems, the polymerisation rate is proportional to the square root of the dose rate at the very beginning of the polymerisation. The termination reaction is bimolecular, i.e. reaction of two radicals, at polymerisation from the liquid to gel state. Then, the reaction is diffusion-controlled with the reaction rate proportional to the dose rate. Temperature thus influences both the reaction rate and the ultimate curing level.

The dose and dose rate application in large composite structures can be modelled, as well as the reaction kinetics. Hence, the properties of the composite materials in terms of extent of curing can be accurately predicted with regard to the processing conditions. Consequently, the process is predictable, controllable and reliable for industrial applications, Ref. [38-41], [38-42].

Ageing of EB cured composite structures has also been studied and no particular sensitivity of the EB-cured systems was observed. The toughness of EB-cured formulations has been improved and is similar to that of heat-curing resin systems. The mechanical performance of the materials has increased and is suitable for very high-performance applications, Ref. [38-43], [38-44], [38-45]. When aged in water, EB-cured resins have the same behaviour as heat-cured resin systems.

Some typical matrices being used for EB-curing are presented in Table 38.12.2.

Table 38.12-2 – EB curing: Mechanical properties of some acrylate and epoxy resins

Resin system	Fracture toughness [1] K_{1C} (MPa.m$^{1/2}$)	Glass transition temperature [2] Tg (tan δ) °C
Acrylate:		
EP-AC	0.6	170
EAR2	0.6	210
EB600-BPaPx	1.5	140
Epoxy:		
CR2 (epoxy)	0.9	205

Key: (1) Fracture toughness measured according to standard IGC 04 26 680;

(2) Glass transition temperatures measured by DMA, resonant mode, 5°C/min

38.12.2.3 Properties

Properties of EB-cured unidirectional composite are given in Table 38.12.3.

Table 38.12-3 – EB curing: Typical mechanical properties of unidirectional IM carbon fibre/acrylate resin

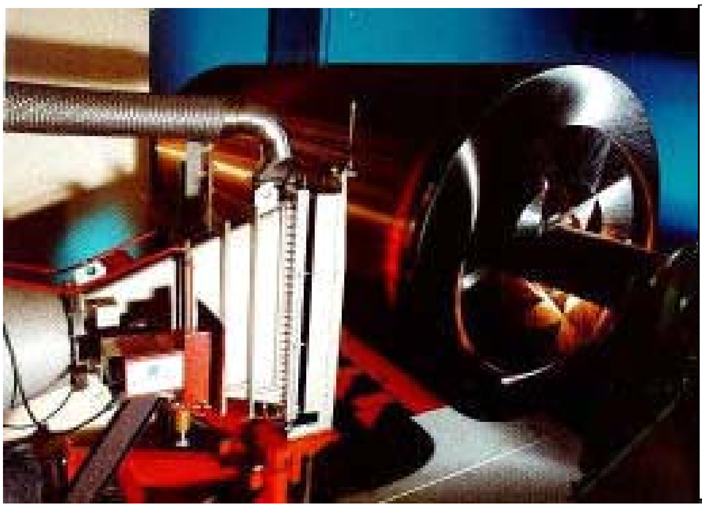
UD properties, at 23°C	IM carbon fibre/EP-AC acrylate resin (65% fibre volume fraction)
Tg (°C)	170
Longitudinal tensile properties:	
Strength (MPa)	2600
Young's modulus (GPa)	174
Longitudinal compression properties:	
Strength (MPa)	1000
Young's modulus (GPa)	141
Longitudinal bending properties:	
Strength (MPa)	1600
Young's modulus (GPa)	150
Interlaminar shear properties:	
Strength (MPa)	80

The first generation of EB-cured composites showed that the generic mechanical properties are very similar to the first-generation of heat-cured epoxy resins used for aeronautic applications.

The major weaknesses compared with newer composites with tough resins, such as IM7/977-2, are the lower interlaminar and transverse tensile strengths.

38.12.2.4 Demonstration parts and structures

Several large, demonstration parts and structures have been manufactured using EB technologies; as illustrated in Figure 38.12.1. The items shown are full-scale components cured by electron beam processing using a 10 MeV electron beam.



Large wound carbon fibre composite vessel

Resin characteristics:

- Acrylate resin (UCB)
- Pot life >12 months, at room temperature
- Viscosity: 0.72 106 mPa.s at room temperature
- Tg: 170°C

Helicopter swash-plate in BMI-based composite

Resin characteristics:

- BMI resin
- Pot life > 1 day at the injection temperature (60°C)
- Viscosity:< 100 mPa.s at T<65°C
- Tg: 268°C

Demonstration boat hull

Resin characteristics:

- Thixiotropic acrylate polyester (UCB)
- Pot life >12 months at room temperature no styrene content
- Viscosity: 2000 mPa.s at room temperature
- Tg: 107°C

Figure 38.12-1 – EB curing: Examples of parts and structures

More recently Boeing designed and built an 'Electron Beam Cure-on-the-Fly' automated tape placement machine. This technique uses a low energy electron beam, i.e. <500 keV. It is intended for the fabrication of large structures without incurring the large capital and tooling expenditures inherent in autoclave curing, Ref. [38-46], [38-47]. The technique is very attractive for producing large composites structures envisaged for advanced aerospace structures.

In Europe, Laben Proel Tecnologie Division, part of the Finmeccanica Group, is involved in composite EB curing using a 'layer-by layer' low energy electron beam technology, Ref. [38-48].

38.12.2.5 Thermoplastic composites

The crosslinking of continuous fibre-reinforced thermoplastics has also been studied, Ref. [38-28].

38.12.2.6 Facilities

EB processing facilities are large and expensive. Industrial EB units are used for processes involving crosslinking of electrical insulation cables, thermoplastic products, rubbers, coatings and adhesives. They are rarely applied forthe curing of composites.

Organisations with facilities known to be capable of composite curing are:

- EADS – Space Transportation, St Médard en Jalles, France.
- LABEN-Proel Technologie S.p.A, Firenze, Italy.
- Acsion Industry, Canada.
- Boeing with NASA LaRC, USA

38.12.3 Microwave

38.12.3.1 General

The use of microwaves for curing both thermosetting composites and adhesives has been studied, Ref. [38-25], [38-30], [38-33]. The curing process relies on the generation of temperatures equivalent to conventional thermal processing. However, the heating effect is localised within the composite and avoids the thermal inertia and heating of the surrounding equipment.

38.12.3.2 Composites

In terms of energy consumption, the temperature of the composite can be raised quickly and efficiently. The consolidation of prepregs relies on pressure applied using vacuum bagging and autoclaving, [See: 38.4].

Curing occurs in a tuneable microwave cavity or a resonant microwave chamber; typically at 2.45 GHz. The conclusions made in early work were that:

- The process had to be optimised to avoid areas of:
  - under-curing, or
  - thermal degradation (over-heating).
- Rapid gelling resulted in unacceptable levels of porosity.
- Combining thermal heating and microwave heating reduced some of the porosity problems.
- Conventional off-the-shelf prepregs were not ideally suited to microwave processing, due to their resin viscosity and gelling characteristics.

The attraction of the technique is that the cure processing time is reduced. It does however suffer from the possible need for:

- Specialised prepregs and adhesives.
- Consumables materials and moulds should avoid interfering with the microwave coupling process.

38.12.3.3 Adhesives

Results of the microwave curing of conventional AF163-2K, EA9689 and EA9391 adhesives showed that, Ref. [38-25]:

- Lap shear strengths were between 52% and 90% of those achieved by conventional thermal cure cycles.
- The lower values were attributed to the presence of voidage from trapped volatiles.
- Total process times were reduced by two-thirds compared with conventional thermal curing.

38.12.4 UV curing

The UV curing process relies on the uncured resin being in an exposed position on the surface being irradiated. It has been demonstrated for wet lay-up configurations containing polyester resins receptive to UV.

There is no published work describing the application of UV curing to high-performance thermosets, such as epoxies, so the process appears to be of limited interest for space structures.

38.13 Preform technology

38.13.1 General

A preform is an assembly of fibres in the desired net-shape of a final moulding, which can be used in both the manufacture of:

- polymer-based composites, [See: 6.31], and
- non-polymer based composite materials, [See: Chapter 88].

The development of various preform technologies has been driven by factors such as:

- A wish to reduce or eliminate manual labour costs in assembling a lay-up of material prior to moulding.
- A need to introduce 3-D reinforcement to avoid the interlaminar, through-thickness weaknesses of conventional laminated plies.
- The use of automated equipment to create complex shapes and changes in section not feasible by using ply stacks.

The use of performs is therefore considered necessary in some applications, because of inadequacies in conventional laminate constructions, Ref. [38-35].

Developments in RTM, resin transfer moulding, [See: 38.7], are providing the greatest incentive to develop efficient means of preparing fibre preforms. This is primarily aimed at reducing processing costs.

38.13.2 Braids

Braids are particularly suitable for producing tubular sections, [See: 6.31]. Their use in 4 m struts has been proposed for large truss structures, Ref. [38-11].

The manufacture of tubes by RTM is claimed to improve tolerance to both low- and hyper-velocity impact damage and also resistance to microcracking, compared with UD prepreg or filament wound constructions.

38.13.3 Commingled fibre performs

Commingled yarns contain a mixture of continuous thermoplastic fibres and reinforcing fibres, [See: 6.18].

The technique was developed as a means of combining the matrix and reinforcement in a dry preform. The preform is then heated to melt the thermoplastic, and consolidated using pressure. The use of PEEK thermoplastic fibres has been studied, Ref. [38-36].

Some examples of the possible applications include:

- Aircraft structures, examples of items evaluated are, Ref. [38-36]:
  - Y-spars: AS4 (6K)/PEEK 150g.
  - Commingled angle interlock 0°/90° woven preforms with stitched ±45° plies; as shown in Figure 38.13.1, Ref. [38-36].
  - Autoclave consolidated, with graphite tooling.
  - 50% higher strain capacity than 2-D laminates.
  - Commingled AS4/PPS 0°/90° I-beams.
- Potential space applications, Ref. [38-37]:
  - commingled thermoplastic fibres and UHM carbon fibres.

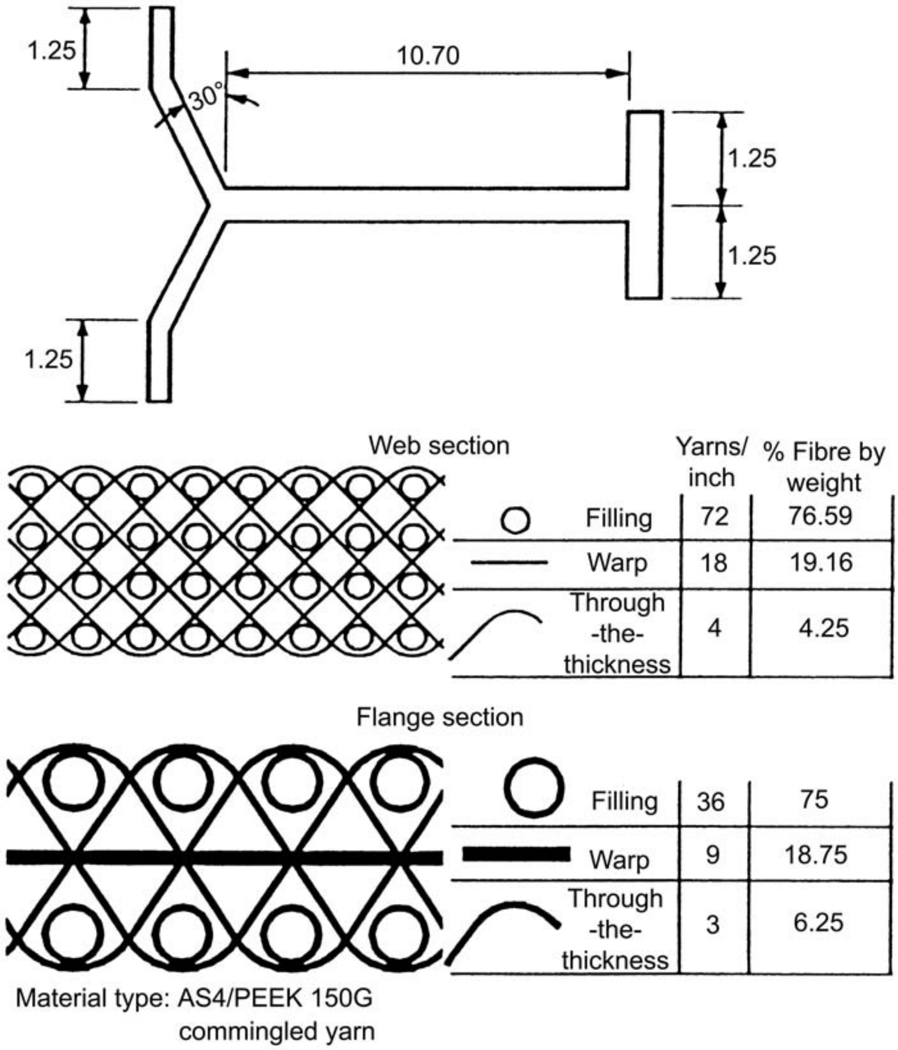


Figure 38.13-1 - Preform technology: Architecture of woven commingled AS4/PEEK 150g 0°/90°

38.14 Fibre placement

38.14.1 Introduction

FP 'Fibre placement' is an advanced computer-automated production technology for creating CFRP carbon-fibre aerospace structures. The computer-controlled system places carbon-fibre layers onto a mandrel using a direct interface to a CAD-CAM design system, such as CATIA. This enables direct information transfer from the drawing to the part.

The system has two main elements:

- Head, which has six-degree-of-freedom feed and roller system to lay CFRP bands onto the mandrel. It is capable of producing a very wide variety of complex curvature shapes.
- Mandrel, which provides a surface on which to build up the final shaped part.

The system, shown schematically in Figure 38.14.1, is very adaptable and imposes few restrictions on the components that can be made. In particular, parts do not need any symmetry of revolution.

The FP system installed at EADS-CASA Espacio can handle thirty-two 3 mm wide CFRP tows simultaneously, combining them through a collimator and onto the mandrel. The head can place a CFRP band up to 100 mm width at the same time.

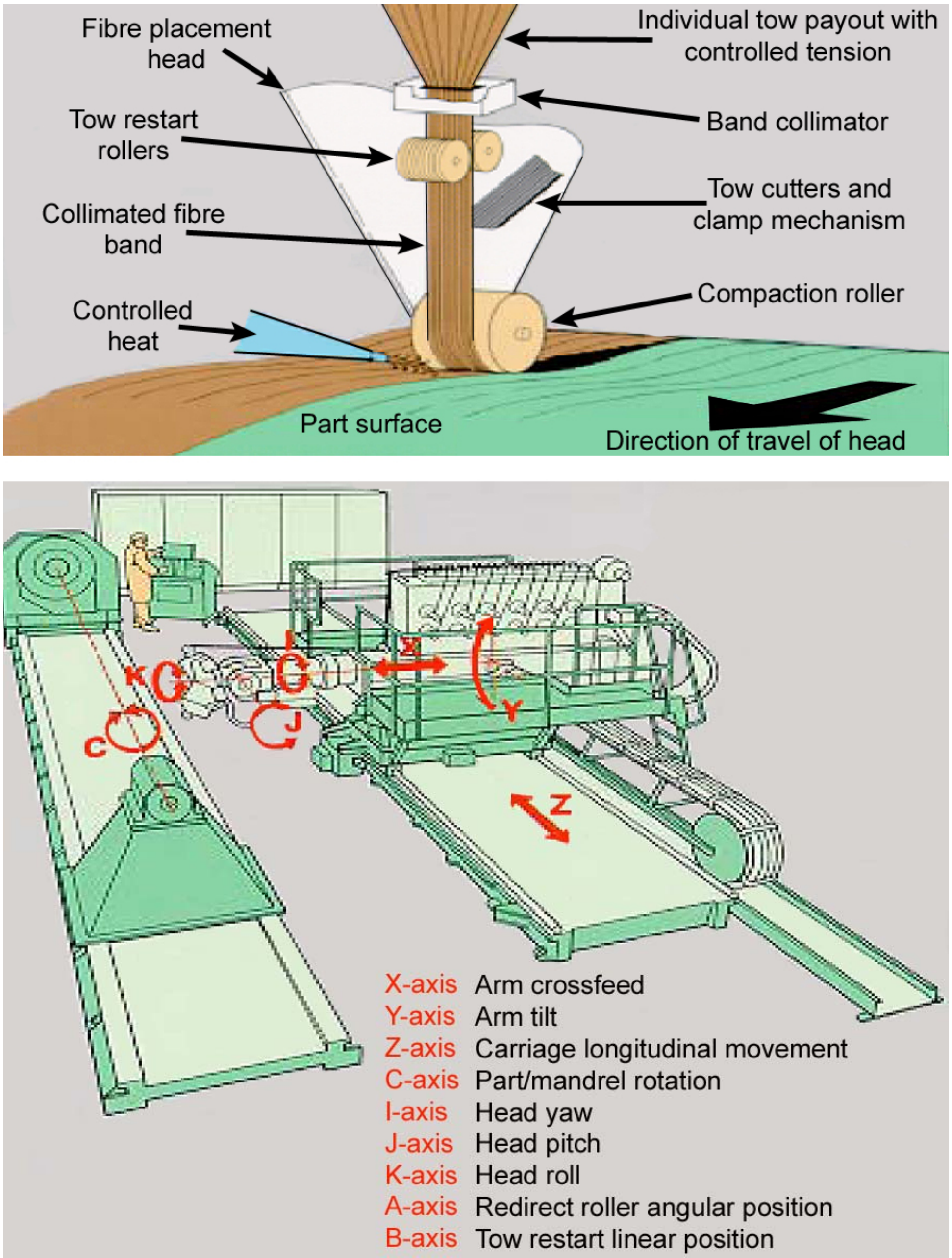


Figure 38.14-1 – Fibre placement: Schematic view of the system

It is possible to stop and restart placement of any of the individual tows and so change the total width of the applied band. This provides a great deal of flexibility in a continuous lay-up operation, e.g. when implementing holes or adding local reinforcements.

The collimated band is applied to the mandrel by means of a compaction roller. The compaction pressure is controlled automatically to enable placement of even layers over light honeycomb cores.

The fibre stock material is kept in a controlled environment at -18ºC to prevent the resin from starting to cure before lay-up. As the band is placed on the mandrel, the fibres are heated to achieve the necessary resin viscosity for compaction by the rollers. Figure 38.14.2 shows the main arm and placement head, Figure 38.14.3 shows a general view of the working area.

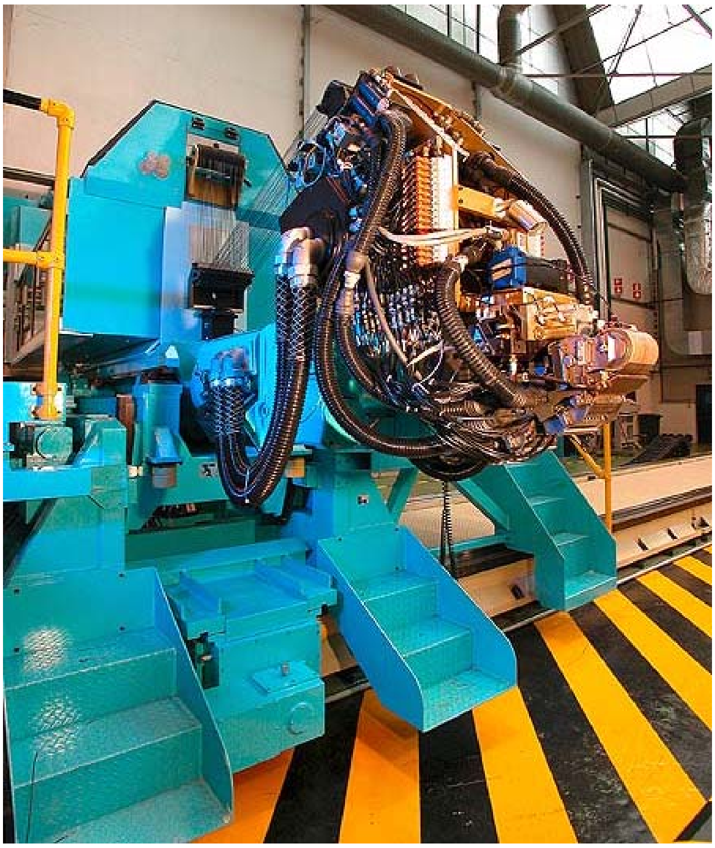


Figure 38.14-2 – Fibre placement: Head

Figure 38.14-3 – Fibre placement: General view of working area

38.14.2 Advantages

38.14.2.1 Productivity

Increases in productivity result from:

- Reduction in scrap material from 30% to 50% in conventional processing compared with 2% to 7% with fibre placement.
- Increased lay-up speed.
- Reduction in the number of pre-curing operations, e.g. intermediate debulking, manual cutting, positioning of layers.

38.14.2.2 Complex-shaped components

The fibre placement head can follow almost any complex curvatures with pressure from the compaction roller always remaining normal to the mandrel surface, as shown in Figure 38.14.4. This enables a very wide variety of complex 3D shapes, with mixtures of concave and convex surfaces to be formed. Consequently many components, not feasible with filament winding technology, can be produced.

FP technology can, for instance, produce smooth cone-to-cylinder transitions in a single lay-up without additional metallic rings.

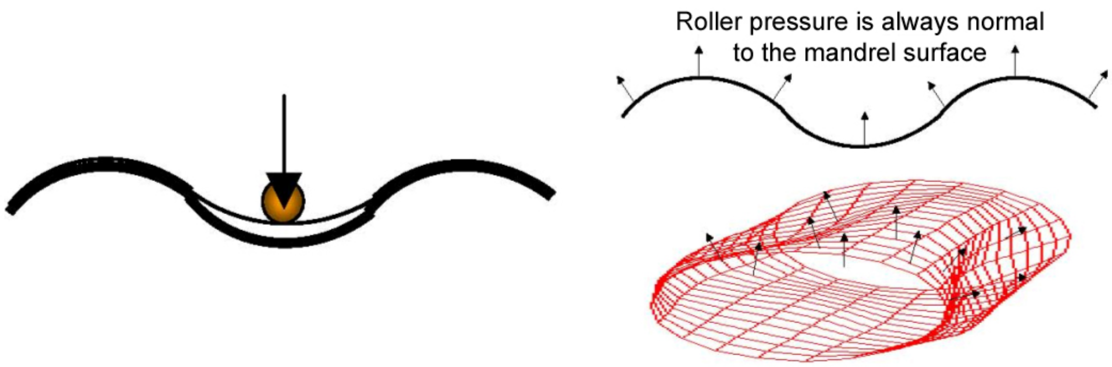


Figure 38.14-4 – Fibre placement: Complex curvature

38.14.2.3 Single shot lay-up of large structures

If manual lay-up is used for large structures, the time taken for material placement can result in partial curing before completion and hence reduction in properties. The higher speed of FP technology avoids this problem.

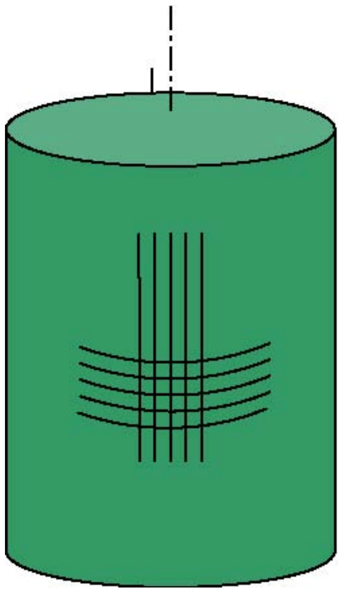
38.14.2.4 Holes and reinforcements in components

Most composite aerospace structures include local reinforcements, holes and other discontinuities. In many cases, the manufacturing operations needed to produce these are very time consuming and can need special tools.

In a single production operation, fibre placement can implement hole and reinforcement patterns simultaneously within the general lay-up. This can provide significant reductions in time and cost.

38.14.2.5 Fibre placement along specific orientations

In the case of satellite-launcher primary structures, ply orientation is a very important design parameter in order to achieve optimum performance with minimum mass. Production technologies such as filament winding do not always enable optimum fibre orientation because a continuous fibre trajectory is needed. Fibre placement has none of these restrictions, fibres can be placed at any orientation (including 0º and 90º in cones or cylinders) to maximise performance-to-mass ratio.



Fibre placement enables the incorporation of purely axial plies

Figure 38.14-5 – Fibre placement: Specific orientations

38.14.3 Repeatability in lay-up and properties

38.14.3.1 General

Based on some preliminary estimations, FP technology gives a ply-positioning accuracy four times better than manual placement, and six times better for angular orientation. Improved lay-up tolerances provide reduced manufacturing deviations that lead to a higher quality final product.

38.14.3.2 Complex trajectory placement

When laying up structures such as cones it is not possible to have a constant ply angle unless the fibre follows a complex trajectory, as shown in Figure 38.14.5. A deviation from this curve can lead to non-optimum thermo-mechanical properties.

Fibre placement can follow any kind of complex path to get the necessary material configuration in the final part.

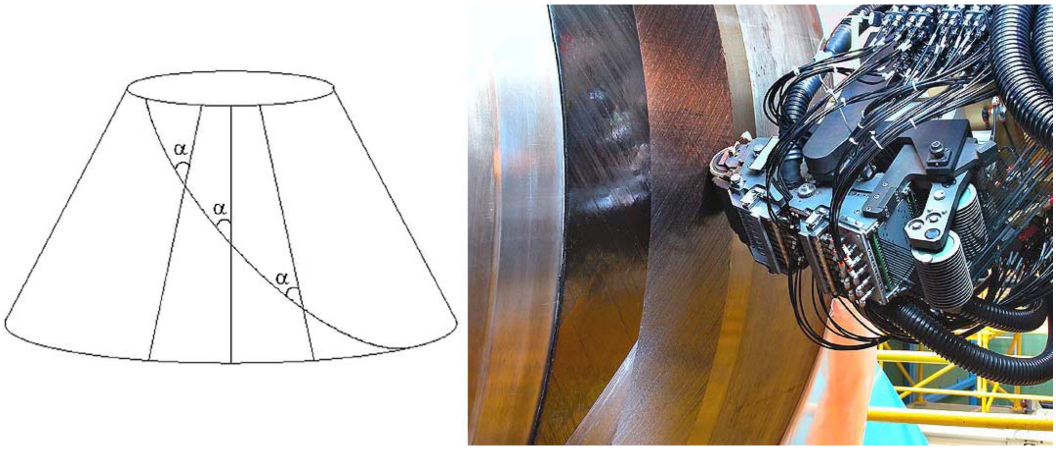


Figure 38.14-6 – Fibre placement: Complex trajectories

38.14.3.3 Variable width of band

Variations in laminate thickness can occur where the overall dimensions of a component vary along its main axis. For example, in the case of a simple conical shape, if the width of the applied band remains constant it produces an increasing final laminate thickness along the cone (due to increased overlapping at the smaller end of the cone).

To prevent this effect, the FP system can alter the width of the applied band, hence keeping the overall thickness constant along the component. This is achieved by increasing and decreasing the number of individual tows making up the band, as shown in Figure 38.14.7.

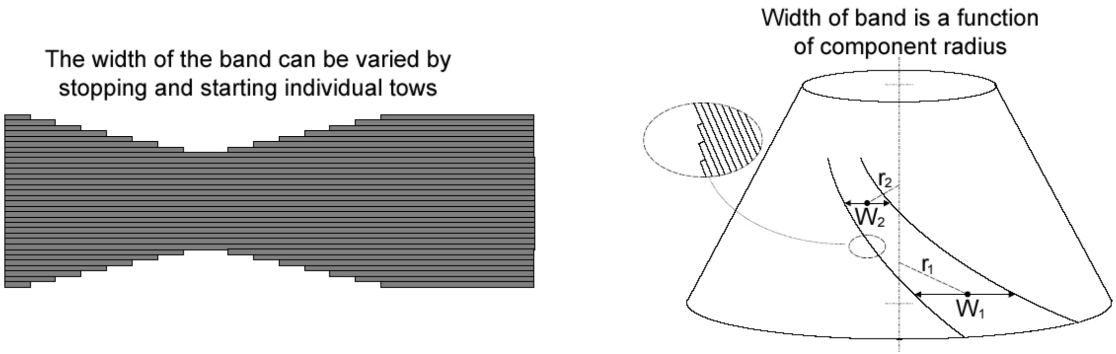


Figure 38.14-7 – Fibre placement: Variation of band width

38.14.4 Mixed materials in the same lay-up

Different materials can be used simultaneously in the same layup band by varying the tows entering the collimator. For example, in space applications, it is sometimes desirable to reinforce an area by substituting a high-strength fibre for the high-modulus one used in the general lay-up. This enables the reinforced area to withstand higher loads with less mass.

Fibre placement makes it possible to introduce conductive fibres or other kind of material for specific design needs.

Figure 38.14-8 – Fibre placement: Mixed-material lay-up

38.14.5 Major advantages of fibre placement compared with other technologies

38.14.5.1 Filament winding

- Lower cost:
  - higher lay-up speed (filament-by-filament compared with band-by-band).
  - less manufacturing operations (holes and reinforcements made as part of the lay-up process)
- Better performance:
  - optimum fibre orientation (0º and 90º).
  - complex shapes (cone-to-cylinder transition without rings).
  - optimum reinforcement layers (materials and orientations).
- Better quality:
  - more accurate tolerances.
  - less maufacturing deviations.
- Flexible design:
  - no shape limitations.
  - no symmetry of revolution required.

38.14.5.2 Manual placement

- Lower cost:
  - higher lay-up speed (manual vs. automatic).
  - less manufacturing operations (holes and reinforcements made as part of the lay-up process).
  - direct data transfer from drawing to part.
  - less scrap material.
  - no intermediate compaction.
- Better performance, improved tolerances (position and orientation).

- Better quality:
  - more accurate tolerances (less maufacturing deviations).
  - better repeatability.
- Flexible design:
  - large structures possible in a single process.
  - no shape limitations.

38.14.6 Examples of European launcher and satellite structures made by fibre placement

38.14.6.1 Sandwich structures

- Ariane 5-ISS
  - Cylinder: 5.5 m diameter, 2.9 m height.
  - Core: aluminium alloy honeycomb with several core densities.
  - Skins: different thicknesses and transitions from sandwich to monolithic.
- Ariane 5-Adapter-1194FP
  - Cone: 2.6 m to 1.2 m diameter, 0.9 m height.
  - Core: aluminium alloy honeycomb with several core densities
  - Skins: different thicknesses.
- Ariane 5-VEB-ATV Outer cylinder
  - Cylinder: 5.5 m diameter, 1.2 m height.
  - Core: aluminium alloy honeycomb with several core densities.
  - Skins: different thicknesses.
- Ariane 5-VEB-ATV Inner cone
  - Cone: 5.4 m to 3.9m diameter, 0.8 m height.
  - Core: aluminium alloy honeycomb with several core densities.
  - Skins: different thicknesses.

38.14.6.2 Monolithic

- Ariane 5-Adapter-1194H
  - Cone: 2.6 m to 1.2 m diameter, 0.9 m height.
  - Skins: different skin thicknesses, with integrated CFRP interface lower ring.
- Ariane 5-Cone 3936
  - Cone: 3.9 m to 2.6 m diameter, 0.8 m height.
  - Skins: different thicknesses, with integrated CFRP interface upper and lower rings.
- Eurostar 3000LX Central Tube, Ref. [38-39].

— Cone and cylinder in one composite part: 1.2 m to 0.8 m cone diameter, 0.6 m cone height, 0.8 m cylinder diameter, 2.1 m cylinder height, 2.7 m total height,

— Skins: different thicknesses.

38.15 Out of autoclave

38.15.1 Introduction

The term OOA 'out of autoclave' is used to describe a number of composite manufacturing processes that do not need an autoclave to achieve proper consolidation and cure. These processes can be broadly grouped as:

- oven-curing of vacuum-bagged prepreg lay-ups.
- liquid resin infiltration of fibre preforms, such as RTM, [See: 38.7]; vacuum-assisted, e.g. VARTM; other derivative proprietary processes, e.g. SQRTM infiltration of prepreg lay-up, Ref. [38-49].
- resin film infusion (RFI), a GKN Aerospace process, which involves laying-up of dry multi-axial fabric interleaved with resin film that is then cured in an oven or on heated tools, Ref. [38-50].
- non-thermal cure processes, e.g. EB – electron beam, Ref. [38-51], microwave, UV cure, [See: 38.12].
- other proprietary processes, e.g. Quickstep® moulding technology, which uses a fluid as a heat transfer media rather than a gas, [38-52], [38-53].

As the size of composite components increase, so does the investment necessary for an autoclave of the appropriate size and its associated facilities. The advantages claimed for OOA processing compared with autoclaving, include:

- reduced processing times.
- reduced processing costs.

Savings can result because resins have lower cure temperatures. Reduced tooling costs are also possible because of the lower pressures compared with autoclaving.

For RTM-type processes, matched-mould tools are necessary. Some derivative processes for large thin parts use tooling that incorporates the resin injection and venting system. In these cases tooling costs are significant and usually need to be amortised over a series of identical parts.

38.15.2 Aerospace structural parts

A number of aerospace projects have identified the possible need for OOA composite manufacture, e.g. European TANGO project (Technology application to the near term business goals and objectives of the aerospace industry) considering composites for large commercial aircraft structure; European FAST 20XX project (Future High-Altitude High-Speed Transport, comprising a suborbital and a hypersonic point-to-point transport system); NASA's Constellation programme; US Ares V launch vehicle, civilian aircraft fairings, Ref. [38-49].

However, there are some concerns regarding OOA compared with traditional autoclaved parts, including:

- Overall quality of OOA parts, e.g. higher void content, lower fibre volume fractions, reduced mechanical properties, because of the lower consolidation pressures. Some OOA processes claim fibre volume fractions only about 2% lower than autoclaved parts, with void contents stated from 'near-zero' to <1%. However, the precise values are both process and component shape dependent.
- Geometry of parts: Components with deep draught, abrupt changes in section or sharp corners can suffer local porosity from poor consolidation and poor surface finish. This usually applies to prepreg lay-ups rather than RTM-type processes.
- Qualification is more complicated for RTM-type processes that can be adapted for a particular component. As a result, the final component properties vary with the process parameters. Considerable effort is being placed on generating databases of material properties, Ref. [38-58].
- RTM-processing of large, thin parts (such as aerodynamic surfaces) need additional vents to ensure escape of volatiles and air.

38.15.3 Composite materials

38.15.3.1 Prepregs

In general, the rheological characteristics of resins in prepregs are modified to enable proper consolidation under OOA conditions, i.e. lower temperature, lower pressure.

Examples of commercially-available epoxy resins for prepregs destined for OOA include:

- MTM45-1 and MTM46 ACG – Advanced Composites Group
- CYCOM 5320 – Cytec Engineered Materials
- HexPly M34, EF01, M9.1F/M9.6F, M10 – Hexcel.

38.15.3.2 Resins

The temperature–viscosity characteristics of the resin are important to ensure flow and impregnation of the preform under the injection pressure (often from 0.7 MPa to 1.4 MPa).

Epoxies, bismaleimides and cyanate ester resins are commercially-available for RTM-type processes. They are often one-part systems that become less viscous when heated, e.g. HexFlow from Hexcel, MVR from ACG, CYCOM-RTM range from Cytec.

38.15.4 Tooling

38.15.4.1 Types

A variety of tooling solutions are being proposed for OOA, including:

- ACTs - advanced composite tools, usually carbon fibre-based composites, Ref. [38-57].
- carbon foams, Ref. [38-54], [38-57].
- ceramics, Ref. [38-55]

The characteristics for OOA tooling are effectively the same as for autoclave tools, i.e. low-no CTE, low mass, ease of machining, ease of incorporating systems (heating, venting), resistance to thermal cycling over processing temperatures, long life.

Carbon foams tools can be used in ovens, Ref. [38-57], or made to be 'self-heating' by passing an electric current through the tool body, thereby heating only the tool and part thus avoiding the need for an oven, Ref. [38-54].

Heated ceramic tooling, developed under ESA-funding by Éire Composites, successfully demonstrated moulding flat carbon fibre/PEEK thermoplastic laminates, Ref. [38-55].

The tool comprised a Pyromeral ceramic, Ref. [38-56], reinforced with carbon fibres to provide thermal degradation resistance, strength and low thermal expansion.

The properties of laminates made using the Pyromeral tool were determined by mechanical testing (tensile, compression, in-plane shear); bolt bearing; NDT (C-scan), fractography and void analysis, outgassing, degree of crystallinity (by DSC) and compared favourably with those produced by autoclave, Ref. [38-55].

38.16 References

38.16.1 General

[38-1] M.M. Schwartz
'Composite materials handbook'
McGraw Hill, 1984, ISBN 0-07-055743-8

[38-2] G. Lubin
'Handbook of composites'
Van Nostrand Reinhold, 1982, ISBN 0-442-24897-0

[38-3] M.J. Robinson et al
'Advanced composite structures for launch vehicles'
SAMPE Quarterly, January 1991, p26-37

[38-4] G.M. Teare
'Filament winding of satellite structures'
International Symposium: Space Applications of Advanced Structural Materials, ESTEC
21-23 March 1990, ESA SP-303, p257-260

[38-5] C.A.L. Kemper et al
'Design and manufacturing of filament wound thrust cylinder'
International Symposium: Advanced Materials for Lightweight Structures, ESTEC, 25-27 March 1992
ESA SP-336, p51-56

[38-6] K. Lähteenkorva et al
'Tape winding of thermoplastic composites'

International Symposium: Advanced Materials for Lightweight Structures, ESTEC, 25-27 March 1992
ESA SP-336, p203-206

[38-7] M. Wadsworth
'Resin transfer molding of composite aircraft structures'
SAE Technical Paper 891042, April 1989

[38-8] I. Marchbank
'Automated R.T.M. for an airframe component'
Materials and Processing - Move into the 90's
Edited by S. Benson et al. European SAMPE Conference 1989
Elsevier Science Publishers. p75-86

[38-9] A. Jiminez et al
'Design and manufacturing of CFRP rings by RTM'
International Symposium: Advanced Materials for Lightweight Structures, ESTEC, 25-27 March 1992
ESA SP-336, p143-148

[38-10] G.C. Sharpless
'Advancement of braiding/resin transfer molding from commercial to aerospace applications'
SME Technical Paper EM91-217, 1991

[38-11] A.K. Munjal et al
'Design and fabrication of high quality graphite/epoxy braided composite tubes for space structures'
35th International SAMPE Symposium
April 2-5 1990, p1954-1967

[38-12] C.W. Dietrich & D.C. Giedt
'Resin transfer molding and compression molding processing of nozzle components as a low-cost alternative to tape wrapping'
AIAA Paper 93-2011

[38-13] E.B. Stark et al
'A new high performance resin system for resin transfer molding of aerospace structures'
37th International SAMPE Symposium
March 9-12 1992, p1421-1431

[38-14] A. Falcone et al
'Resin transfer molding of textile composites'
Boeing Defence and Space Group, NASA-CR-191505
July 1993, 139 pages

[38-15] A. Wang et al
'A new high temperature epoxy system for RTM application'
37th International SAMPE Symposium
March 9-12 1992, p482-492

[38-16] K.A. Barrett et al
'Injectable bismaleimide systems'
35th International SAMPE Symposium
April 2-5 1990, p1007-1020

[38-17] E.B. Stark et al
'Resin transfer molding (RTM) of high performance resins'
35th International SAMPE Symposium, April 2-5, 1990, p782-795

[38-18] J.E. Stockton
'Structural resin transfer molding of high temperature composites'
34th International SAMPE Symposium
May 8-11, 1989, p1032-1040

[38-19] M.C-Y. Niu
'Composite airframe structures'
Published by Commlit Press Ltd, January 1992
ISBN 962 7128 06-6

[38-20] G.J. Sundsrud
'Advantages of a one-part resin system for processing aerospace parts by resin transfer moulding (RTM)'
Proceedings of the 14th International European Chapter Conference of SAMPE: 'Broadening Horizons with Advanced Materials & Processes', Birmingham, October 1993, p.251-258, ISBN 3-9520477-0-8

[38-21] F.N. Scott & A. Koorevaar
'Computer aided engineering of the resin transfer moulding (RTM) process'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures, ESTEC
March 1994, ESA-WPP-070, p335-340

[38-22] V.M. Karbhari et al
'Effect of material, process, and equipment variables on the performance of resin transfer moulded parts'
Composites Manufacturing, Vol. 3, No. 3, 1992, p143-152

[38-23] W. Werner & G. Ziegmann
'Development of techniques for polymeric diaphragm forming of continuous fibre reinforced thermoplastics'
Proceedings of the International Symposium on Advanced Materials for Lightweight Structures, ESTEC
March 1994, ESA-WPP-070, p367-371

[38-24] G.H.F. Nayler
'Vectra insulative support structures within spacecraft'
International Symposium: Advanced Materials for Lightweight Structures
ESTEC, 25-27 March 1992, ESA SP-336, p291-298

[38-25] G.B. Gaskin et al
'Electromagnetic curing of epoxy adhesive systems'
38th International SAMPE Symposium, May 10-13 1993
p380-390

[38-26] C.B. Saunders et al
'Electron curing of fiber-reinforced composites; recent developments'
38th International SAMPE Symposium, May 10-13 1993
p1681-1691

[38-27] D. Beziers et al
'Electron beam curing of composites'
ECCM 4, 1990, p73-78

[38-28] A.B. Strong
'Crosslinking of thermoplastic composites using electron beam radiation'
SAMPE Quarterly, July 1991, p45-54

[38-29] C.B. Saunders et al
'Recent developments in the electron-beam curing of fiber-reinforced composites'
37th International SAMPE Symposium, March 9-12 1992
p944-954

[38-30] F.Y.C. Boey
'Techniques in the microwave processing of thermoset composite using a high pressure autoclave'
23rd International SAMPE Technical Conference
October 21-24, 1991, p15-24

[38-31] C.B. Saunders et al
'Radiation-curable carbon fiber prepreg composites'
Polymer Composites, December 1988, Vol. 9, No. 6, p389-394

[38-32] C.B. Saunders et al
'Radiation-curable prepreg composites'
Atomic Energy of Canada Limited Report AECL-9560, 1988

[38-33] J. Wei et al
'Microwave processing of crossply continuous graphite fiber/epoxy composites'
SAMPE Journal, Vol. 27, No. 1, Jan./Feb. 1991, p33-39

[38-34] J.P. Fouassier & J.F. Rabek
'Radiation curing in polymer science and technology, 1993'
Vol. I: Fundamentals and Methods, ISBN 1-85166-929-9
Vol. II: Photoinitiating Systems, ISBN 1-85166-933-7
Vol. III: Polymerization Mechanisms, ISBN 1-85166-934-5
Vol. IV: Practical Aspects and Applications, ISBN 1-85166-938-8
Published by Chapman & Hall, London

[38-35] J. Brandt et al
'The application of three-dimensional fibre preforms for aerospace composite structures'
International Symposium: Space Applications of Advanced Structural Materials, ESTEC, 21-23 March 1990, ESA SP-303 p71-77

[38-36] J. Suarez & J. Mahon
'Consolidation of graphite/thermoplastic textile preforms for primary aircraft structure'
The First NASA Advanced Composites Technology Conference, Part 1, p293-338 (N93-30439)

[38-37] C. Blair & G.A. Jensen
'Process development and characterisation of ultra high modulus, drapable graphite/thermoplastic composites for space applications'
37th International SAMPE Symposium, March 9-12, 1992
p115-127

[38-38] J. Pascual & J. Trigo: EADS-CASA Espacio (E)
'Application of New Technologies for Eurostar Central Tube'
Paper 161: ESA SP 581: Proceedings of European Conference on Spacecraft Structures, Materials and Mechanical Testing, ESTEC, Noordwijk (NL) 10-12 May 2005

[38-39] J. Pascual & J. Trigo: EADS-CASA Espacio (E)
'Application of New Technologies for Eurostar Central Tube'
ESA SP 581: Proceedings of European Conference on Spacecraft Structures, Materials and Mechanical Testing, ESTEC, Noordwijk (NL) 10-12 May 2005

[38-40] B. Defoort et al.: EADS-ST (Bordeaux, F)
'Electron-beam polymerization of acrylate compositions 6: Influence of processing parameters on the curing kinetics of an epoxy acrylate blend'
Macromol. Chem. Phys., 202,16, pp 3149-3156, (2001)

[38-41] B. Defoort et al : EADS-ST (Bordeaux, F)
'Modelling of the EB curing of fibre-reinforced composite materials for aerospace applications'
Presentation 23rd Meeting of the Adhesion Society, February 2000, Myrtle Beach-SC, USA, pp392-394

[38-42] B. Defoort et a:. EADS-ST (Bordeaux, F)
'Electron Beam curing of acrylate resins for composites: Modelling reaction kinetics'
45th International SAMPE Symposium, May 21-25, 2000, Long Beach-CA, USA, pp2223-2234

[38-43] B. Defoort et al: EADS-ST (Bordeaux, F)
"Investigations to improve the properties of EB cured composites: A status report"
47th International SAMPE Symposium, May 2002

[38-44] B. Defoort & L.T. Drzal: EADS-ST (Bordeaux, F)
'Adhesion between Carbon fibres and Cationic matrices in Electron-Beam Curing Processed Composites'
46th International SAMPE Symposium, 6-10 May, 2001, Long Beach-CA, USA, pp 2063-2075

[38-45] B. Defoort & L.T. Drzal: EADS-ST (Bordeaux, F)
'Influence of Thermal Postcuring on Electron Beam Cured Composites'
46th International SAMPE Symposium, 6-10 Mai, 2001, Long Beach-CA, USA, pp 2550 -2562

[38-46] D L Goodman et al.
'Automated Tape Placement with in-situ Electron Beam Cure'
International SAMPE Symposium and Exhibition, May 23-27, 1999, Long Beach, CA

[38-47] J. W. Burgess et al: Boeing Company/ NASA Langley (USA)
'Development of a 'Cure-on-the-fly' Automated Tape Placement Machine for Electron Beam Curable Prepregs'
46th International SAMPE Symposium and Exhibition, May 6-10, 1999, Long Beach, CA; SAMPE 2001 - Long Beach, CA May 6 - 10, 2001

[38-48] F.Guasti: LABEN-Proel Technologie (I)
'Pressure Vessels: A Possible Application of low Energy E-Beam curing'
44th International SAMPE Symposium and Exhibition, May 23-27, 1999, Long Beach, CA

[38-49] K. Mason
'Autoclave Quality Outside the Autoclave?'
'High Performance Composites' (2006)
http://www.compositesworld.com/articles/autoclave-quality-outside-the-autoclave.aspx

[38-50] 'GKN Aerospace Develops Manufacturing Processes for Complex Composite Structures'
http://www.azom.com/news.asp?newsID=6054

[38-51] C. A. Byrne: Science Research Lab Inc, Ma, (USA)
'Non Autoclave Materials for Large Composite Structures'
Report No. A897483 (November 2000)
Abstract: http://www.stormingmedia.us/89/8974/A897483.html

[38-52] M. Kaiser et al: Inst. für Flugzeugbau, Uni. Stuttgart / Eurocopter GmbH (D)
'Out Of Autoclave Manufacture of Structural Aerospace Composite Materials'
http://www.quickstep.com.au/files/document/165_Out_of_Autoclave_Processing_via_Quickstep.pdf

[38-53] Quickstep Technologies Pty Ltd (Australia)
http://www.quickstep.com.au/

[38-54] TRL - Touchstone Research Laboratory Ltd (USA)
'Carbon Foam Self-heated Tooling for Out-of-Autoclave Composites Manufacturing'
Proposal No. 08-1 X4.06-9473, NASA SBIR 2008 Solicitation
http://sbir.nasa.gov/SBIR/abstracts/08/sbir/phase1/SBIR-08-1-X4.06-9473.html?solicitationId=SBIR_08_P1

[38-55] A. Murtagh: ÉireComposites Teo (Éire)
'Application of Thermoplastic Composite Out of Autoclave Vacuum Processing Technology to Non-Space Structures'
EC4706-020-RPT-A Final Report (October 2008),
ESTEC Contract No.19729/06/NL/PA

[38-56] Pyromeral http://www.pyromeral.com/index.htm

[38-57] ACG – Advanced Composites Group (UK)
'Technical Report – New Tooling Developments', (March 2008)
http://www.advanced-composites.co.uk/PSG_Electronic_Files/Tooling_PSG_Files/PDFs/Technical%20Report%20New%20Tooling%20Developments%20A1-%2016th%20March%202009.pdf

[38-58] ACG – Advanced Composites Group (UK)
'Out of Autoclave Processable Prepregs and Resin Films: An Overview of Recent Developments and Shared Databases' (2006), Abstract
http://www.sae.org/technical/papers/2006-01-3164

38.16.2 ECSS standards

[See: ECSS website]

ECSS-Q-ST-70	Materials, mechanical parts and processes
ECSS-Q-70-71	Data for the selection of space materials and processes

39 Machining techniques

39.1 Introduction

The various techniques used to machine composites are described and the precautions necessary to prevent damaging the components.

The techniques used can vary with the particular composite material. Aramid composites often need different tools from those used for carbon- and glass-reinforced materials.

Special drill bits are available for composites to overcome the problems of using drills designed for metals, [See: 39.9].

[See also: ECSS-E-HB-32-21 for machining operations related to adhesive bonding; ECSS-E-HB-32-22 for machining practices for inserts in sandwich panels; ECSS-E-HB-32-23 for machining practices for bolted joints]

39.2 Basic rules

39.2.1 General

The machining of composite materials should meet the basic criteria of:

- No fraying.
- No delamination of the cured composite edge.

39.2.2 Equipment

Standard machining equipment with small modifications is often used. The cutting speeds and feeds used depend upon:

- the thickness of the composite,
- the type of cutting method.

Various cutting tools are used for the machining operations, including:

 — countersink,
 — cut off wheels,

— router bits,
— bandsaw blades,
— high speed steel drills,
— reamers.

It is particularly important to keep all the tools sharp, in order to provide good quality cuts and to minimise the possibility of delamination.

Sufficient back-up support of the part to be machined is essential to reduce chipping and delamination, e.g. by using aluminium.

In some cases machining of polymer matrix composites can cause local overheating and destruction of properties. In these situations cooling methods should be used.

39.3 Routing

39.3.1 Carbon/epoxy composites

Generally, routing with a higher torque but low speed router works best. Diamond cut carbide bits are preferable because of higher feed rates at less operator effort than with a six flute configuration. To extend the tool life and to increase the cutting force, a suitable coolant is used (no effect on cut edge quality).

39.3.2 Aramid/epoxy composites

The cutter shown in Figure 39.3.1, Ref. [39-1], achieves satisfactory results.

Routing with a conventional aircraft tool steel router can be difficult, whereas the opposed helix router can be used for:

- Cutting laminates into sections.
- Cutting slots and notches.
- Trimming honeycomb sandwich panels.

An opposed helix cutter enables laminates up to 6.4 mm thick to be trimmed without problems. A conventional router needs much more effort for composite material thicker than 3.2 mm.

Figure 39.3-1 - Router: Opposed helix for aramid/epoxy

39.3.3 Trimming and bevelling

39.3.3.1 Carbon and glass reinforced epoxy

Trimming and bevelling with a conventional router against a guide can be used successfully.

39.3.3.2 Aramid/epoxy

The quality of aramid/epoxy trimmed edges can be improved by using a router, as shown in Figure 39.3.2, Ref. [39-1].

Trimmed edges of aramid/epoxy can also be improved by sanding.

With diamond cut carbide type router bits, good cuts can be made up to a composite thickness of about 6.9 mm.

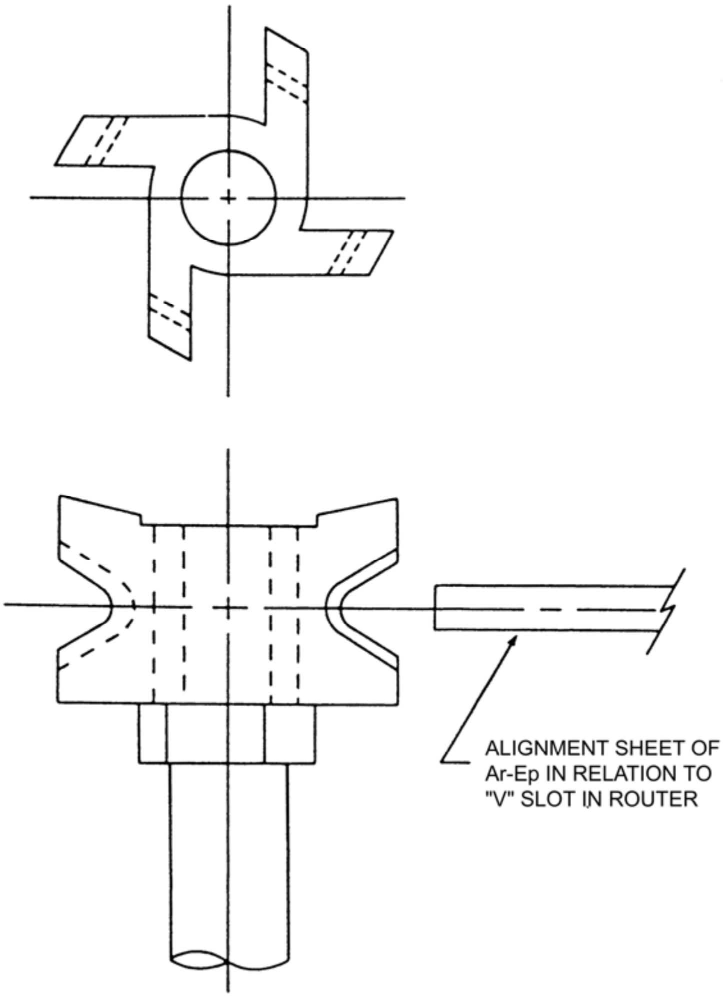


Figure 39.3-2 - Router for trimming aramid/epoxy composites

39.4 Sanding

39.4.1 General

There are several important points to be noted when sanding composite laminates. These points relate to sanding processes for:

- Fit or trim, and
- Adhesive bonding.

39.4.2 Fit or trim

Guidelines for sanding composites correctly include:

- Use tools correctly; as shown in Figure 39.4.1, Ref. [39-1].
- Use a right angle sander of 20,000 rpm or greater.
- Bulk removal (dry): Use 50 mm aluminium oxide disks.

- Edge deburring (water): Use 50 mm 240-grit to 320-grit, silicon carbide.
- Address edges at proper angle.

During sanding operations:

- Do not try to remove any defects on surfaces of parts by sanding.
- Do not over-sand beyond trim areas.
- Do not deburr holes; countersink holes with sanders.

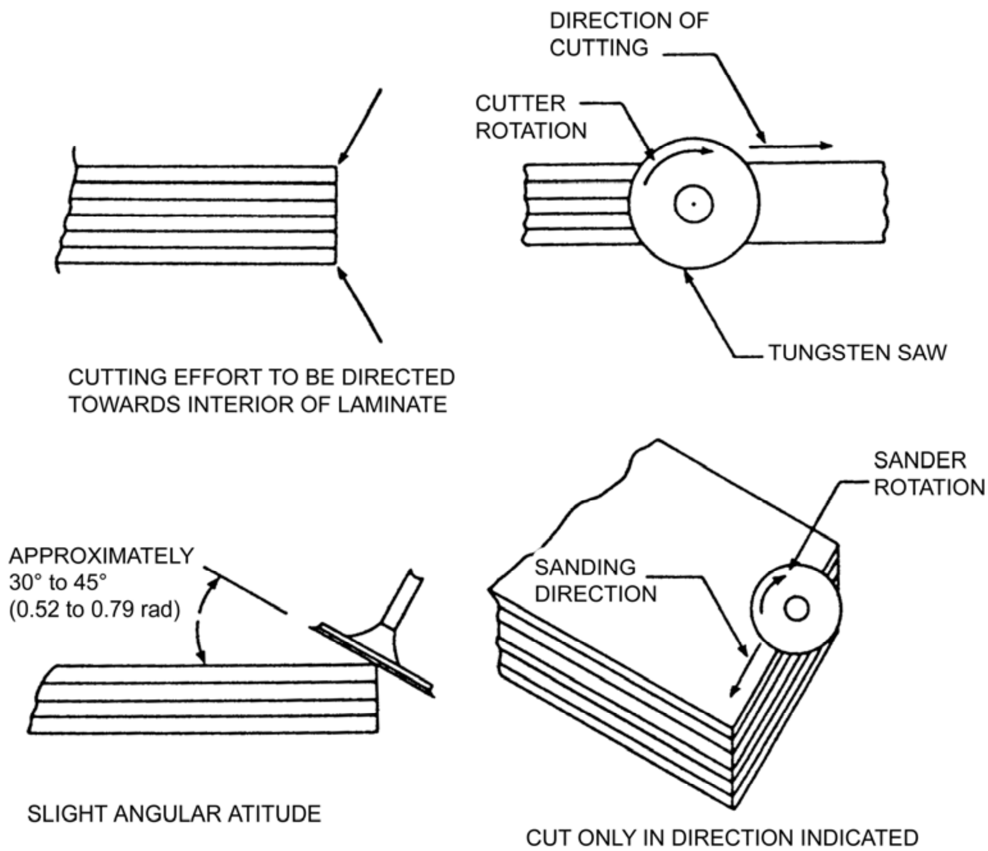


Figure 39.4-1 - Sanding of composites

39.4.3 Bonding

Guidelines for sanding composites correctly prior to adhesive bonding include:

- Lay out the area to be sanded.
- Use 240-grit.
- Use tools of 20,000 rpm or greater.

[See: ECSS-E-HB-32-23 for further guidance on the surface preparation for bonded joints]

39.4.4 Aramid/epoxy composites

Wet paper is used for sanding of aramid/epoxy laminates to provide cooling and also to prevent build-up of waste between abrasive particles. The preferred grit sizes are 120 and 240.

39.5 Sawing

39.5.1 Basic rules

Sawing of cured laminates can be successfully performed with:

- Band saws,
- Circular saws, or
- Sabre saws.

The composite is clamped to eliminate vibration that can cause delamination. Saws need frequent checking to maintain sharpness.

39.5.2 Bandsawing

39.5.2.1 General

Normally a fine, offset, high-strength-steel-stagger-tooth blade is used. As a general rule, the saw blade pitch is selected by the laminate thickness to be cut corresponding to the height of three saw-teeth. This cutting method uses the heel rather than the hook of the cutting tooth blade for cleaner cuts. The cutting blade is always sharpened before cutting.

Surface speed in the range of 22.9 m/s to 33 m/s are used; with 30.5 m/s preferred. The best feed rates are from 3 mm/s to 5 mm/s.

39.5.2.2 Aramid/epoxy composites

Bandsaw cutting of aramid/epoxy is usually performed with a fine tooth blade, preferably using water as a coolant. The bandsaw is run in reverse, so that the heel of the tooth enters the composite first; as shown in Figure 39.5.1, Ref. [39-1].

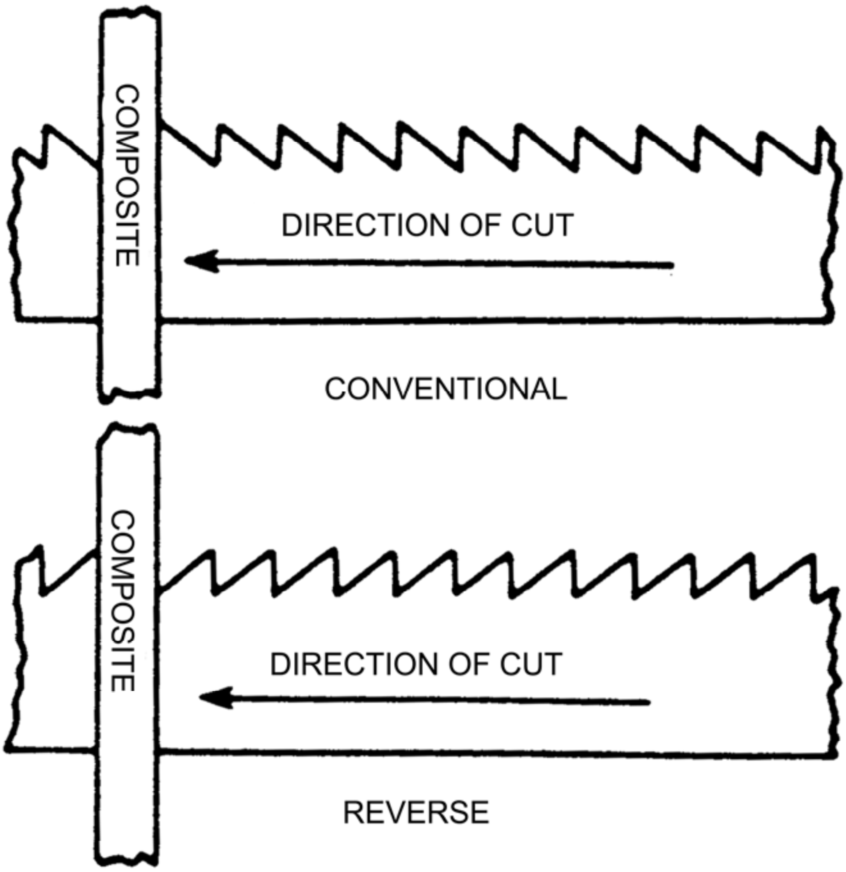


Figure 39.5-1 - Reverse bandsawing aramid/epoxy laminates

39.5.3 Circular sawing

39.5.3.1 General

Diamond blades are preferred for most composite materials. Cutting speeds for composites normally vary between about 10 m/s to 50 m/s.

39.5.3.2 Carbon/epoxy composites

A 6.4 mm carbon/epoxy laminate is best cut with a carbide-tipped blade and speed between 10 m/s and 20 m/s.

Figure 39.5.2 shows that 1.3 mm/s maximum feed rate is ideal for all parts up to 2.5 mm thick, Ref. [39-1]. Thicker parts are cut at proportionately lower feed rates.

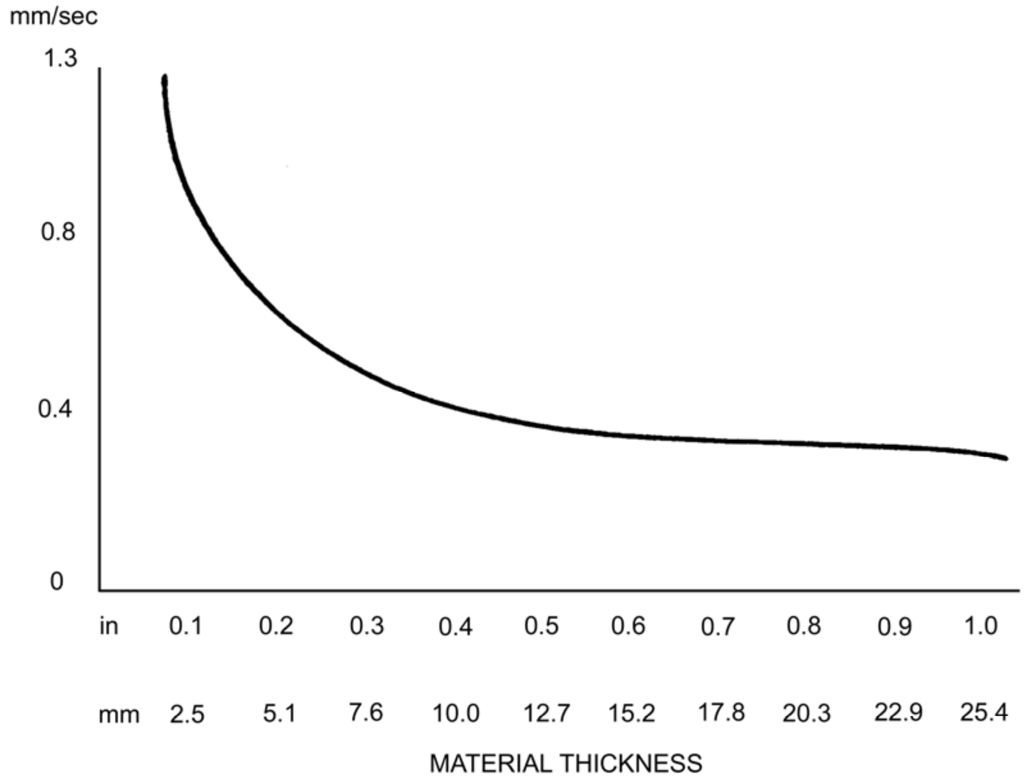


Figure 39.5-2 - Circular sawing: Feed rate versus thickness of composite materials

39.5.3.3 Aramid/epoxy composites

Aramid/epoxy can be cut successfully using a circular metal slitting saw.

39.5.4 Sabre sawing

39.5.4.1 Aramid/epoxy composite

Sabre saws normally use the blade shown in Figure 39.5.3, Ref. [39-1], to cut aramid/epoxy, which cuts the outermost fibres on both sides of the laminate toward the interior. This type of blade has alternating sets of five teeth in opposed directions. Blade speeds of 2500 strokes per minute are advisable.

Blade speed and feed rates vary with material thickness.

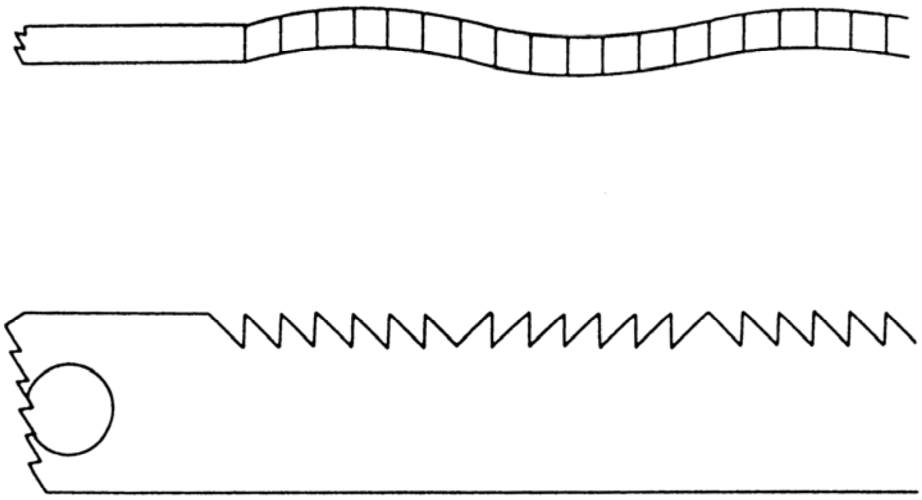


Figure 39.5-3 - Sabre sawing: Alternating tooth blade for aramid/epoxy

39.6 Countersinking

39.6.1 General

Carbide, high-strength steel, diamond-plate countersinks can be used depending on the composite.

39.6.2 Carbon and glass reinforced epoxy

Carbide countersinks have shown good results with optimum combinations of speed and relief angles. Conventional countersinking tools from 1750 rpm to 6000 rpm have been used successfully.

39.6.3 Aramid/epoxy composites

Figure 39.6.1 shows a countersink used for aramid/epoxy composite, Ref. [39-1].

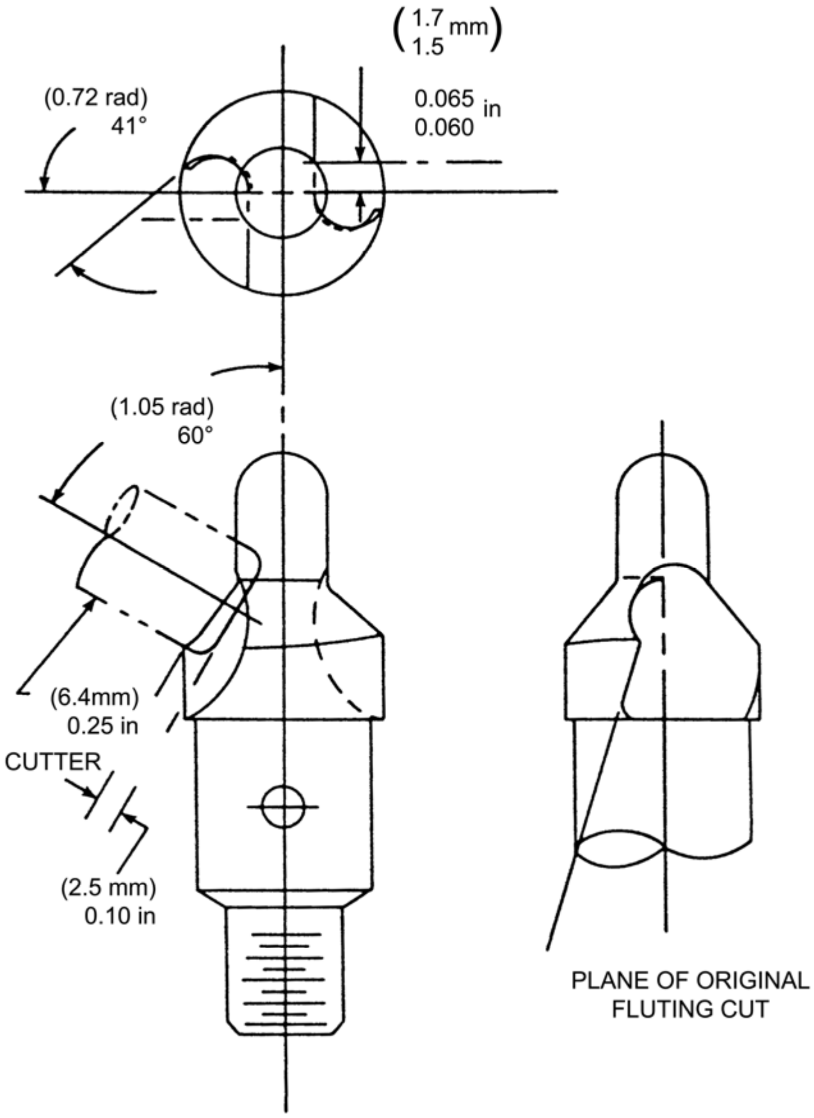


Figure 39.6-1 - Countersink for aramid/epoxy composite

39.7 Counterboring

Carbon, aramid and glass reinforced epoxy composites are normally counterbored using carbide cutting tools.

In general, only a few counterbores can be made per tool, because the entire cutting edge bears on the workpiece material during the operation.

Compared with countersinking of carbon/epoxy, [See also: 39.6], counterboring torque and thrust forces are generally 3 times higher.

39.8 Milling

39.8.1 Basic Rules

39.8.1.1 General

To produce a surface cut that extends past the edge of a composite laminate, the edge of the laminate is milled first to prevent delamination, as shown in Figure 39.8.1.

The use of fluorocarbon coolant is advisable because of its cooling efficiency. The coolant is applied as a spray mist during machining; the distance between the spray applicator and cutter is adjusted so that frost forms on the cutter.

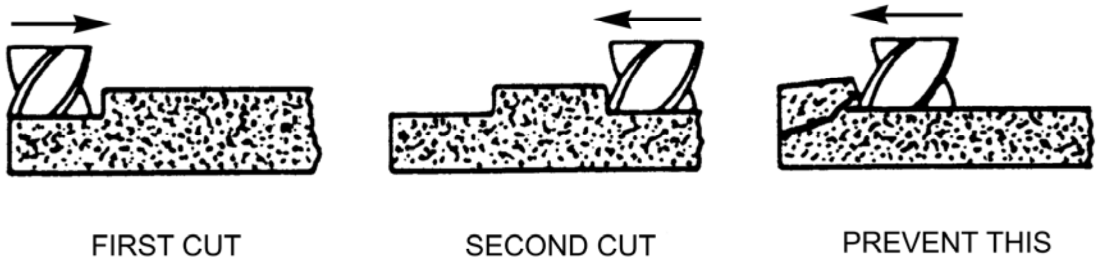


Figure 39.8-1 - Proper milling technique

39.8.1.2 Cutter types

Guidelines for the selection and use of cutters are:

- Ensure that cutters are sharp; dull ones cause delamination.
- Always use high-speed-steel cutters of the four-flute, positive rake type.
- Use carbide milling cutters of a positive-rake type, with chip loads of 0.10 mm to 0.15 mm per tooth.
- Radiused-end mills last longer than square-cornered ones.

39.8.1.3 Carbon/epoxy composites

High-speed-steel end mills or carbide cutters can be used provided that they are multi-fluted. Four-flute end mills are advisable for efficiency and to reduce the cutting forces to a point where there is less chance of delamination.

39.8.1.4 Aramid/epoxy composites

Conventional fluted cutters are used satisfactorily at speeds of 0.4 m/s and a feed rate of 5 mm/s.

39.8.2 Plunge-cut milling

Plunge-cut milling is not recommended, unless there is sufficient back-up support to prevent delamination.

39.9 Drilling

39.9.1 Basic problems

39.9.1.1 General

Metal working drill bits were not designed for cutting composites and their use can provoke significant damage to a composite arising from:

- Abrasivity: Glass/epoxy and carbon/epoxy are so abrasive that only tungsten carbide tooling can drill through them.
- Heating: For metal-working, the tip heats the metal to provide the plastic flow needed for efficient cutting. Since composites cannot tolerate this heat, the cutting speed is slowed down to ensure that the heat is as low as possible.
- Rake: Drill designers had to abandon cutting tips with neutral and negative rakes and wide chisel points because a drill with a neutral rake scrapes the material and causes it to resist penetration by the drill tip. The operator exerts pressure to drill the hole, and this pressure causes the heat build-up.
- Pressure: Neutral rake also tends to push the reinforcing fibres out in front, needing a great deal of pressure to penetrate the piece. This pressure causes the fibres to bend, resulting in furry, undersized holes.
- The pressure also produces excessive heat, which causes galling and chip dogging in the resin.
- Feed: The release of pressure as the tool bit breaks through the part causes a sudden and momentary increase in feed rate. As the tool plunges through the last few fibres, the cutter shaft, not the cutting edge, removes the remaining material. The result is chipping and cracking.
- Chip formation: The best way to analyse a drilling operation is to examine the chips. The ideal chip form for a composite is a dry, easily moved chip that looks like confectioner’s sugar.

If the speed of the cutting tool is too high, heat makes the resin sticky and produces a lumpy chip; if the cutting edge is scraping and not cutting the plastic, the chips are large and flaky. Either type eventually clogs an evacuation system.

39.9.1.2 Tool design for composites

Factors in the design of tools for composites include:

- Positive rake: The reinforcing fibres are pulled into the workpiece where they are sheared or broken between the cutting edge and the uncut material. Positive rake on the cutting edge removes more material per unit of time and per unit of pressure than negative rake, but the more positive the rake, the more sensitive and fragile the cutting edge becomes.
- Chisel edge: A small chisel edge improves the penetration rate, which increases the number of machining operations per hour. The optimum chisel edge for composites is as close to a point as possible.
- Chip handling: The chips are produced and then removed immediately above the entrance of the hole. From this point a properly designed vacuum system can dispose of the chips in conformance with safety and environmental standards.

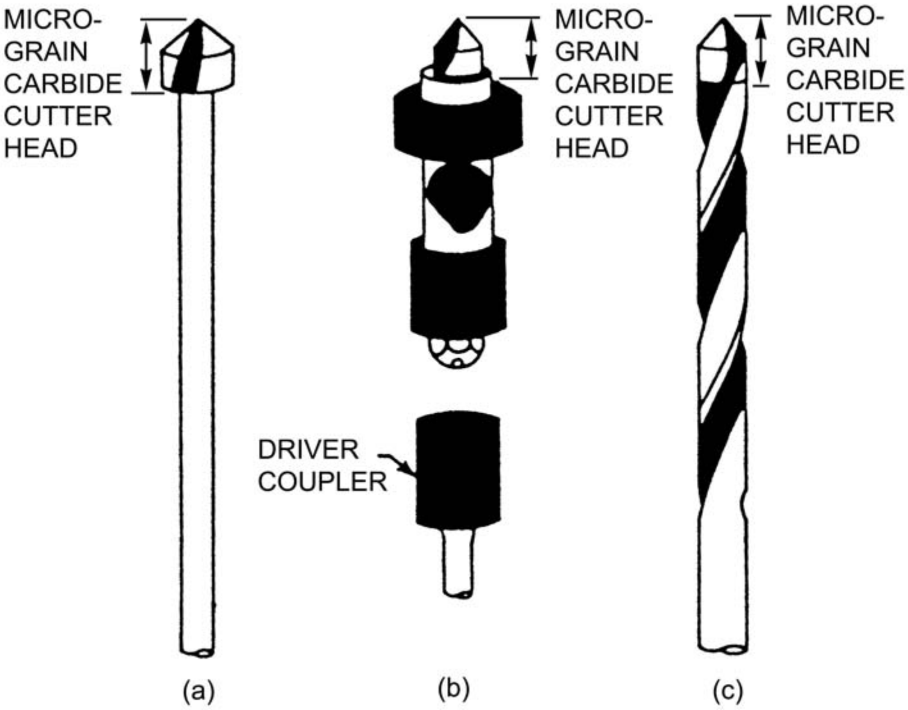
39.9.2 Drill bits for composites

39.9.2.1 General

Tooling has developed that improves greatly the drilling of glass-, carbon- and aramid-epoxy composites and their hybrids. Some tool bits are made of tungsten carbide particles of less than 1 μm.

Figure 39.9.1 shows examples of drill bits for composites, Ref. [39-1], where:

- Solid-shank drills are used in automatic drilling equipment.
- Twist drills are used in automatic drilling equipment.
- Drill-guide systems are designed for use with an air or electric drilling motor.



(a) Solid shank drill, (b) Drill guide system, (c) Fluted twist drill

Figure 39.9-1 - Drill bits for composites

Fitted with a socket adapter, drill sizes from 3 mm to 25.4 mm can be used with the drill-guide system. The internal compression control spring regulator withdraws the tool after drilling. Its pressure control compensates at 'breakthrough', where a sudden increase in feed rate occurs as the tool bit breaks through the last few fibres.

Partially drilled holes are avoided because the operator depresses the unit completely each time; no change in pressure is felt as the hole nears completion. The drills speeds are between 1.5 m/s to 3 m/s; as recommended.

39.9.2.2 Aramid/epoxy composites

Guidelines for drill bits suitable for aramid composite include:

- Spade drills are best for drilling aramid/epoxy leaving very little fuzz and fraying at the edges of holes. Like carbide drills, these drills have a tendency to burn under prolonged uses.

- Conventional drills can also be used if a firm sacrificial backing is used on the exit surface.
- Twist and flat-ended high-speed-steel drills perform quite well, especially with a firm backup of the composite to eliminate fuzzing and delamination at the exit hole.
- Backup can be provided by leaving the glass peel ply on both sides of the composite during drilling. A 0.08 mm layer of fibre glass on the surfaces of the composite produces clean entrance and exit holes.
- Drill speeds range from 25,000 rpm to 35,000 rpm. The high speed steel drill resembles a twist drill fluted on the end.

39.9.2.3 Glass/epoxy composites

Drills with titanium diboride coating improve the tool life in the order of 9 times.

39.9.2.4 Carbon/epoxy composites

Guidelines for drill bits suitable for carbon composite include:

- Tungsten carbide drills, shaped like a standard twist drill, can be used successfully; with a backing plate. They can be used for 50 to 60 holes before it needs re-sharpening. Standard drills need resharpening after for 5 to 6 holes, typically.
- Various types of coolants have been evaluated to prevent drills from breaking, dulling or burning. A lubricant called 'Boelube' has performed well, but water is the best.
- To reduce surface delamination during drilling, it is advisable to have a layer of woven glass of 0.08 mm on both sides of the laminate.

39.9.2.5 Solid carbide daggar drills

The performance of this type of drill bit can be summarised as:

- Quite successful, producing a clean hole with little or no break-out.
- Drilling has been achieved without using backup material.
- Since the daggar drill maintains the tolerance of the hole, no reaming is necessary.
- Dagger drills are suitable for use in both:
  - hand held units, as shown in Figure 39.9.2.
  - a fixed machine using air feed.
- Good results are achieved for
  - hand fed units at 900 rpm.
  - air feed units at 2100 rpm with a 1.3 mm/s feed rate.
- Drilling carbon/epoxy and metal simultaneously has produced high quality holes and countersinks.

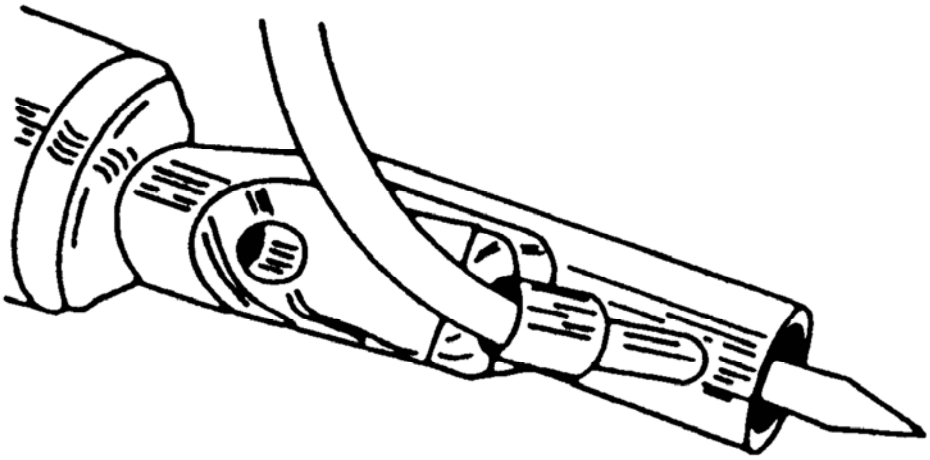


Figure 39.9-2 - Daggar drill in hand feed units

39.10 Orbital drilling

39.10.1 Introduction

Orbital drilling is in effect the milling of circular holes. The cutting tool rotates in the usual way but, at the same time, the tool axis is moved around a circular path about the desired hole centre and the tool is fed into the material, as shown in Figure 39.10.1.

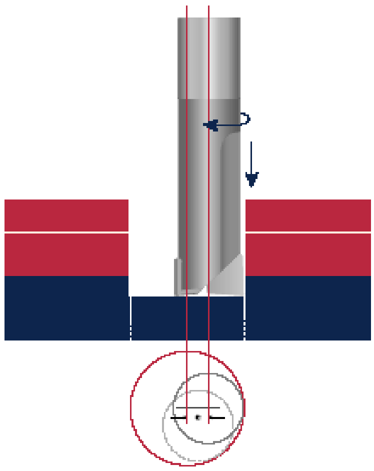


Figure 39.10-1 – Orbital drilling principle

Different hole sizes can be machined using the same tool by altering the diameter of the orbital movement. The final hole diameter is equivalent to the cutting tool diameter plus the orbital diameter.

39.10.2 Advantages

Orbital drilling offers a number of advantages compared with conventional machining techniques, including:

- Tight tolerance holes.
- Burrless holes in metals.
- Delamination free holes in composites.
- Various hole sizes using a single tool.
- Single operation, using one tool in one set-up.
- Good surface finish.

39.10.3 Drilling parameters

In order to obtain good results, three parameters should be adjusted, i.e.:

- Tool rotation speed,
- Orbital rotation speed,
- Feed speed.

The correct setting of each of these parameters is essential to optimise the machining and to avoid unwanted effects. Each setting is established by testing the process on representative samples.

39.10.4 Equipment

Figure 39.10.2 shows a Twinspin orbital drilling head from Novator AB (Sweden) mounted to a jig. In this case the equipment is being used for composite machining by Saab Ericsson Space.

Figure 39.10-2 – Orbital drilling: Equipment mounted on a jig

39.10.5 Application examples

Figure 39.10.3 illustrates some of the machining operations possible using orbital drilling.

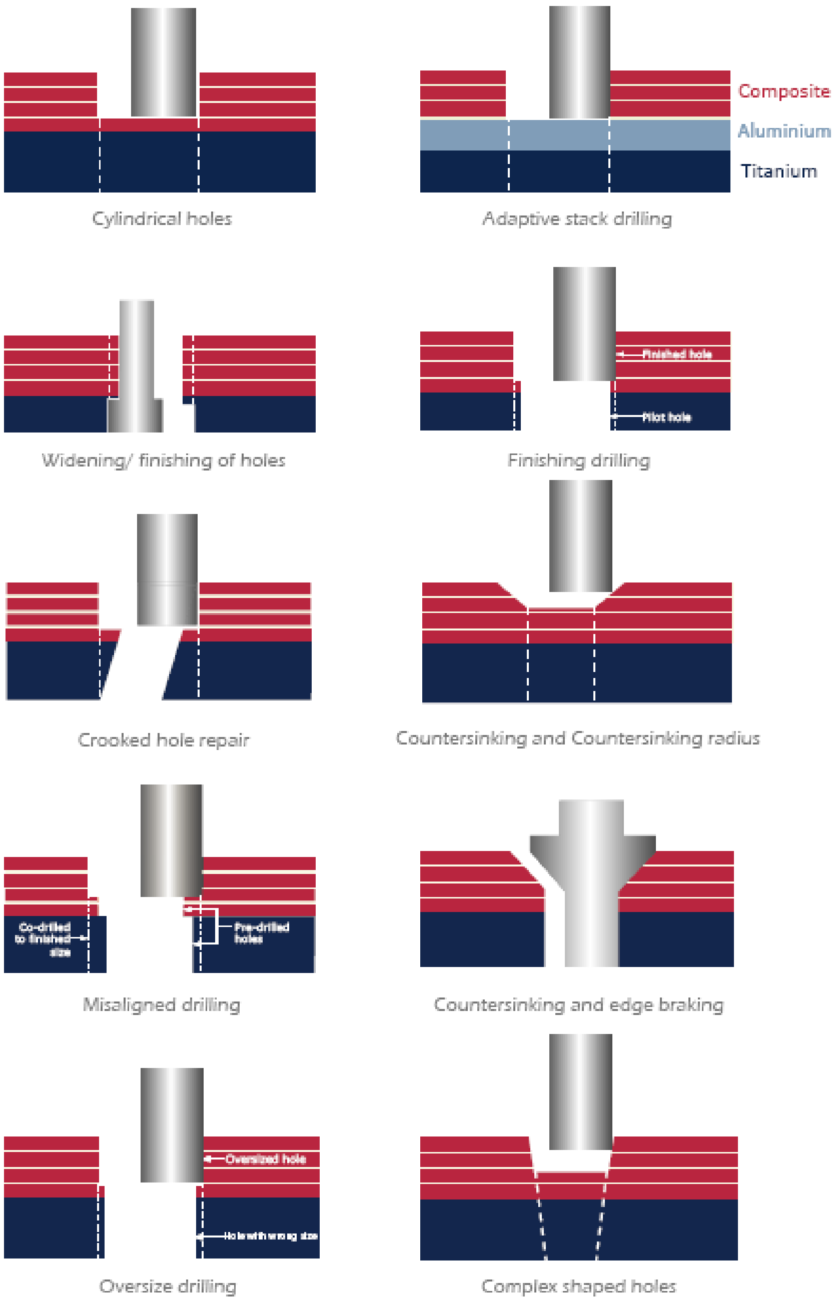


Figure 39.10-3 – Orbital drilling: Application examples

39.11 References

39.11.1 General

[39-1] M.M. Schwartz
'Composite materials handbook'
McGraw Hill, 1984

39.11.2 ECSS standards

[See: ECSS website].

ECSS-E-HB-32-21	Adhesive bonding handbook; previously ESA PSS-03-210
ECSS-E-HB-32-22	Insert design handbook; previously ESA PSS-03-1202.
ECSS-E-HB-32-23	Threaded fastenershandbook; previously ESA PSS-03-208

40 Manufacturing costs

40.1 Introduction

The selection of materials and appropriate manufacturing processes has important implications concerning the costs within projects. Composites are used because they offer a range of benefits, of which mass-saving is the most prominent, Ref. [40-1], [40-2], [40-3]. There are often a number of designs and configurations that fulfil adequately a particular structural configuration. Some of the competing designs can involve a choice between light-alloys and composites. A part of all design selection approaches is to establish an acceptable balance between the:

- total cost of manufacture, and
- efficiency of the structure to meet its functional role.

The factors contributing to the manufacturing and fabrication costs of structures are described, [See: 40.2]. The items considered contribute to the direct accumulated manufacturing cost, i.e.:

- materials,
- consumables,
- process times,
- capital equipment,
- labour.

Quantifying and allocating manufacturing costs to individual space composite structures and components is difficult because:

- design and process development costs are very high,
- numbers of units produced is often very low, e.g. no more than five for satellites, typically,
- structural verification and qualification costs are high.

Consequently, within a project, it is not easy to establish the true manufacturing costs for individual components and assemblies.

There is always a strong incentive to retain proven materials and associated manufacturing technologies because development and verification activities often represent a large part of the costs.

40.2 Cost drivers

40.2.1 General

Compared with aircraft construction, the space industry uses fairly small total quantities of composite materials. It is advisable to use materials with a clear commercial future, i.e. those procured by more than one industrial sector. Where specialist, low-volume prepregs are concerned, e.g. UHM CFRP, the material cost is very high and the choice limited. The same applies to other materials, e.g. aluminium-lithium alloys, metal-matrix and ceramic-matrix composites.

The issues that contribute significantly to the accumulated manufactured cost are:

- Project costs,
- Mass for cost budget,
- Materials selection,
- Design for manufacturing,
- Manufacturing,
- Tooling and consumables,
- Processing times,
- Labour costs,
- Part count,
- Unit numbers,
- Product life and sourcing.

40.2.2 Project cost drivers

By general consensus, these factors are, Ref. [40-4]:

- Specification envelope:
  - mass budget, and
  - materials selection.
- Qualification philosophy.
- Standardisation: conflicts between mass and cost.
- Methodology: to reduce time for individual tasks or processes.

40.2.3 Mass for cost budgets

Achieving the mass targets or budgets is fundamental to the success of space structures. If the mass budget is too restrictive, then more expensive material technologies can be needed.

40.2.4 Materials selection

This is influenced by the key factors:

- Existing knowledge base, and
- Mass budget: degree of difficulty.

The selection process reflects the naturally conservative philosophy of obtaining the maximum capability from an existing technology before applying a new one.

40.2.5 Design for manufacturing

A classical error made in the early years of composite development was to apply an existing metallic design to the composite equivalent. This did not take account of the advantages offered by composites and the means of manufacture. Components were not optimised for mass and were invariably expensive.

40.2.6 Manufacturing expertise

The existing availability of manufacturing facilities and production expertise is a strong incentive to use existing manufacturing processes. It enables costs to be more accurately predicted before making commitments to a new component design.

Where a new manufacturing route is necessary, it is appropriate to consider the:

- Use of a sub-contractor who already has the facilities and skills for that manufacturing route.
- Development costs of using a new process, and whether these can be amortised over future production or other projects.

40.2.7 Tooling and consumables

Tooling is generally a one-off, non-recurring cost. For low production runs, cost of tooling is a very significant contributory factor to unit costs.

Some processes, such as autoclaving, use large quantities of consumables, such as:

- Bagging materials,
- Breather plies,
- Peel Plies,
- Release films.

Consumables are recurring costs because the materials cannot be reused. Other manufacturing routes, such as filament winding, use much less consumable material.

40.2.8 Processing times

Whilst a short processing time is desirable, it is not always essential and largely depends on the component or assembly, i.e.:

- Large complex assemblies: A long cure schedule need not be a significant cost factor if only a few units are being made.
- Smaller components (produced in larger numbers): Reduction of process times is a major cost driver.

40.2.9 Labour costs

40.2.9.1 General

This remains one of the most difficult items to quantify, because organisations calculating it on a different basis.

40.2.9.2 Direct costs

The direct costs associated with the actual manufacture of components, include:

- Material and tool preparation,
- Lay-Up,
- Equipment operation,
- Demoulding,
- Machining and trimming, and
- Inspection.

The cost of design and testing is added to these to give an overall figure.

40.2.10 Part count

40.2.10.1 General

It is important that composite designs offer a reduction in the part count compared with metallic solutions.

40.2.10.2 Light-alloy

Complex constructions in light-alloys are often made up of tens of individual components. These are then fastened or bonded together. The material cost can be low but the fabrication costs high.

40.2.10.3 Composites

The aim is to produce a small number of composite components for subsequent assembly. In some cases, a single part can be feasible, e.g. by co-curing. Whilst the material and consumable costs can therefore be high, subsequent fabrication costs can be reduced.

40.2.11 Unit numbers

As more components of the same design are made, their unit cost is lower. This is because non-recurring costs can be amortised over a greater numbers of units. The main costs are:

- design, and
- tooling.

Additional savings can be made within:

- Inspection and testing.
- Improved consistent quality.

Some manufacturing routes offer greater cost-savings than others.

40.2.12 Product life and sourcing

It is always advisable to double-source materials, such as prepregs. If it becomes necessary to change from the first to second choice, the processing will be amenable to the change without incurring further significant development or modification costs.

Materials, such as prepregs and adhesives, tend to have a finite commercial life before being superseded by an improved product. It is advisable to establish the future availability with the supplier before selecting that material for a new component or structure.

40.3 Cost optimisation

40.3.1 Load-carrying structures

40.3.1.1 Eurostar Bus: Inmarsat 2 derivative

A summary of the points arising from a BAe study for ESTEC of the Eurostar generic satellite bus are described, which considered its first commercial derivative to meet the Inmarsat 2 mission, Ref. [40-4]. It demonstrates the use of composites within the context of an overall project and the cost optimisation of a communications satellite.

The common features which enable one satellite to be assessed against another are:

- First cost,
- Direct operating cost,
- Operating life, and
- Operational capability.

Whilst first cost savings can be made at the expense of other features, cost optimisation through improvements in efficiency can be achieved which do not adversely impact on the other key factors. The fundamental trade-off throughout any optimisation is that of mass for cost.

40.3.1.2 Mass- cost impact of material selection

The main components contributing to the mass and cost of the satellite bus were:

- Central thrust structure,
- Payload and equipment panels,
- Struts,
- Brackets and cleats.

These groups were chosen to simplify the comparative analysis by combining elements with common design drivers.

Table 40.3.1 lists the range of candidate materials chosen for analysis, Ref. [40-4].

Table 40.3-1 - Cost optimisation: Eurostar Bus/Inmarsat 2 study

Material	Specific Modulus	CTE	Thermal Conductivity	Cost per kg †
Al Alloy	Low	High	High	£3-5
AL-Li Alloy	Low	High	Medium	£15-20
Titanium	Low	Medium	Medium	£15-20
Beryllium	High	Medium	High	>£800
HM CFRP	High	Low	Low	>£125
APC 2	Medium	Low	Low	>£125
MMC_p	Low/medium	Medium	Medium	>£150
MMC_f	High	Low	Low/medium	>£5000

Key: † - Procured materials costs in 1989.

For each candidate material, at least two configurations for each of the components were designed; representing a minimum mass and a minimum cost philosophy. In some cases a mid-point configuration was available where the existing Eurostar baseline was not considered to be minimum mass.

Figure 40.3.1 shows the relationship between mass and cost for the central thrust structure, Ref. [40-4]. Each line has three points; these are (from left to right):

- Minimum mass,
- Mid-point mass,
- Minimum cost.

For mass reduction, curves having shallow slopes result in a greater mass reduction per unit increase in cost.

For cost reduction, curves with the steepest slopes offer greater cost reductions for unit increase in mass.

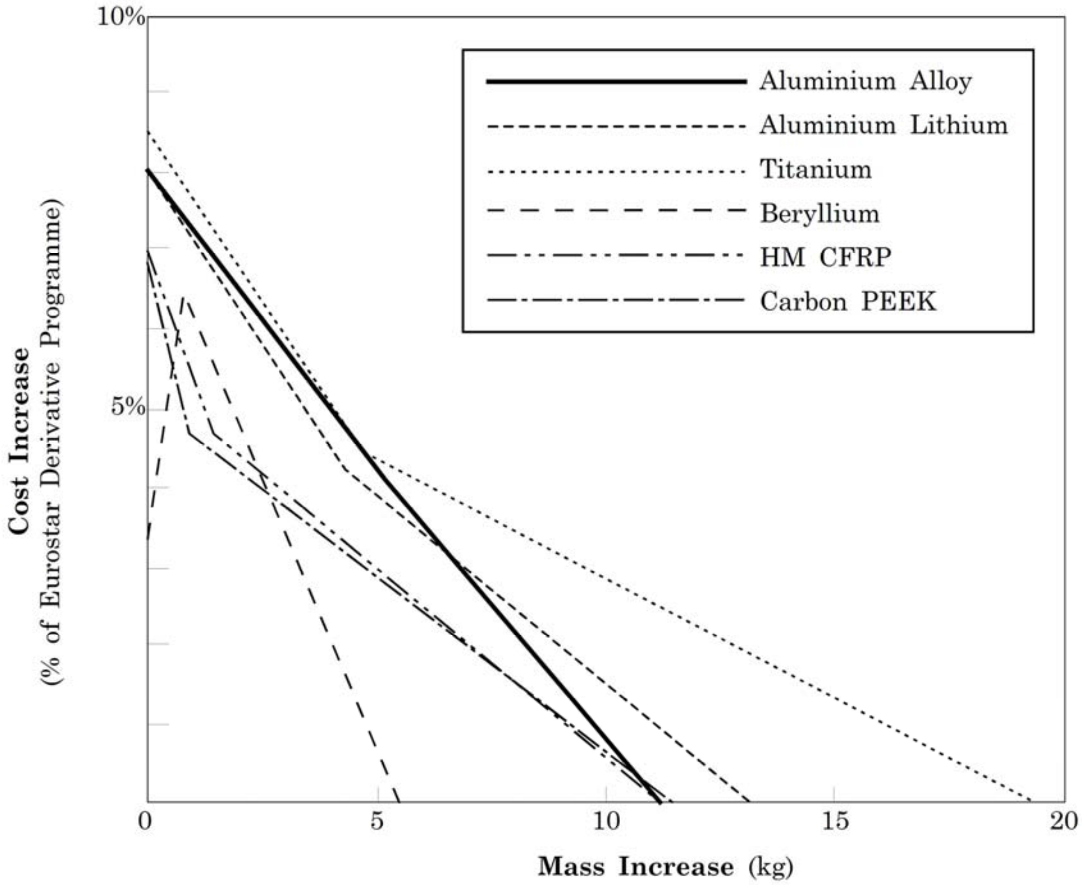


Figure 40.3-1 - Mass/cost sensitivity for central thrust structure

Figure 40.3.2 shows the next step of the entire structure with accumulated mass-cost profiles, Ref. [40-4]. This illustrates that, overall, there are no significant winners and losers. Indeed, the differences are generally insignificant compared with the basic material capabilities in terms of mass and cost.

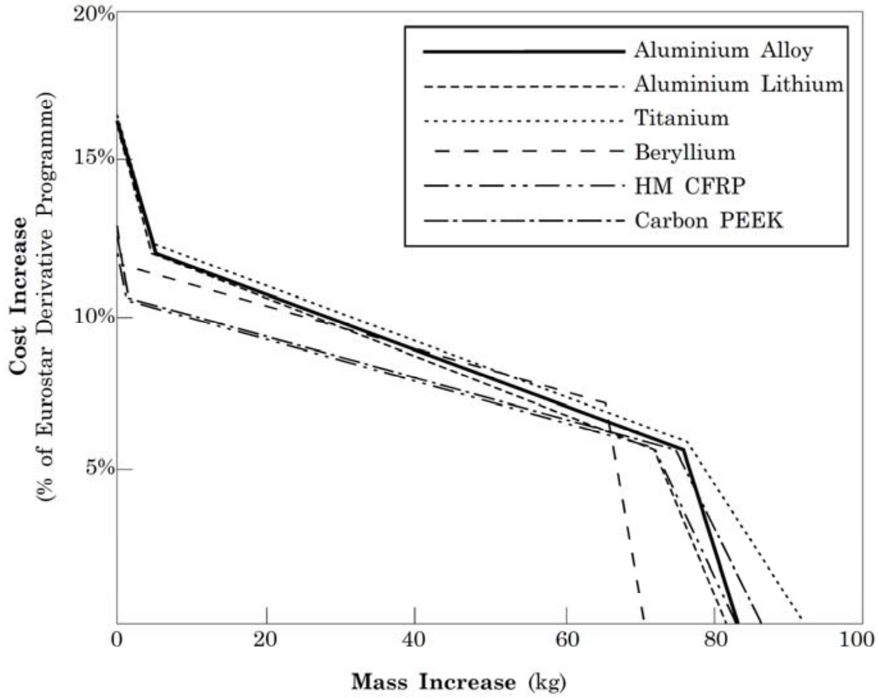


Figure 40.3-2 - Mass/cost sensitivity for complete structure

The material selection philosophies considered were, Ref. [40-4]:

- 'minimum mass scenario', or
- 'minimum cost scenario'.

As seen in Figure 40.3.2, Ref. [40-4]:

- 'Minimum mass scenario' reflects a typical design policy to use existing materials up to their minimum mass limit prior to selecting new more efficient materials.
- 'Minimum cost scenario' is an approach where the most efficient materials are used with a minimum cost design philosophy.

The current design philosophy, or the 'Min. mass scenario' has provided a generally optimised mass to cost trade-off by showing that the cost impact of materials with a higher structural efficiency cannot be supported in a minimum cost design.

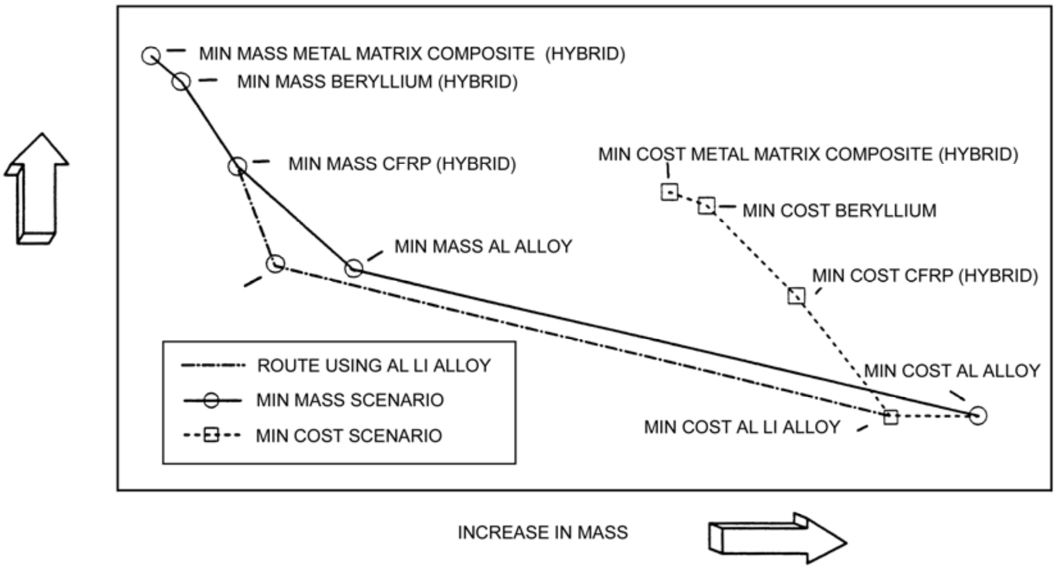


Figure 40.3-3 - Mass to cost envelope for material improvements

40.3.1.3 General guidelines

These can be summarised as:

- Maximise available mass budget.
- Maximise qualification by similarity.
- Maximise standardisation of:
  - parts,
  - materials,
  - methods, and
  - sources.
- Standard features or items are likely to be those which:
  - do not impact on external interfaces,
  - have a small mass impact,
  - apply to at least one third of programmes.
- Use methods that enable automation and integration of processes and tasks.
- Use existing materials up to their minimum mass capability before selecting more structurally efficient options.
- The use of common geometry is essential.

40.4 Cost advantages of composites

40.4.1 Material costs

Historically, composites have been considered to be expensive in comparison with established light-alloys, such as aluminium. The direct material purchase costs per kilogram are very different, in some cases, by orders of magnitude.

40.4.2 Manufacturing costs

The material costs are typically in the range of 10 % to 25 % of the total manufacturing cost. Composites offer opportunities to reduce costs on other aspects of manufacturing and fabrication.

In order to control the 'total manufactured cost' of a component or assembly, the advantages offered by composites that need to be properly exploited are:

- A lower part count compared with metallic designs.
- Fewer fasteners compared with metallic designs.
- Lower material wastage compared with machined metal designs.
- Reduced machining costs by net-shape forming.
- Co-curing of parts.
- Use of preforms and 'broadgoods' instead of UD prepregs.

The reduction and control of labour costs during the fabrication stages also should be addressed.

40.5 Satellite thrust cylinders

Historically, composite satellite thrust cylinders for European space programmes, [See also: 29.12], have been made by a variety of construction methods, including:

- Autoclaved, light-alloy honeycomb sandwich.
- Rib-stiffened CFRP designs.
- Filament-wound CFRP.

The cylinders are manufactured by different contractors in small numbers for each programme. There appears to be no general consensus as to which is the most cost-effective route for producing a common structural configuration. It is assumed that the direct cost of manufacturing each cylinder is a small proportion of the total programme cost. The dominant factors are the identity of the prime contractor and the preferred technology within that organisation. The design selection process is therefore not driven by immediate technical or economic considerations because there is not an open choice of method of construction. The effective use of existing in-house technology dominates the control of development and verification costs for a particular programme.

40.6 Launcher fairing cylinders

40.6.1 General

A study by MDSSC 'McDonnell Douglas Space Systems Co.' investigated the construction options for a very large launcher fairing, with forebody (ogive) and intertank (ALS), Ref. [40-5].

The aim of the design trade-off study was to establish the total system cost sensitivity to structural mass, and if heavy, but lower cost, monocoque designs were feasible.

The key factors considered in the trade-off were:

- cost,
- structural loads,
- minimum gauge considerations,
- separation systems,
- acoustic and thermal protection.
- damage tolerance to:
  - hail, and
  - lightning strikes.

All of these factors contributed to the overall mass of the constructions.

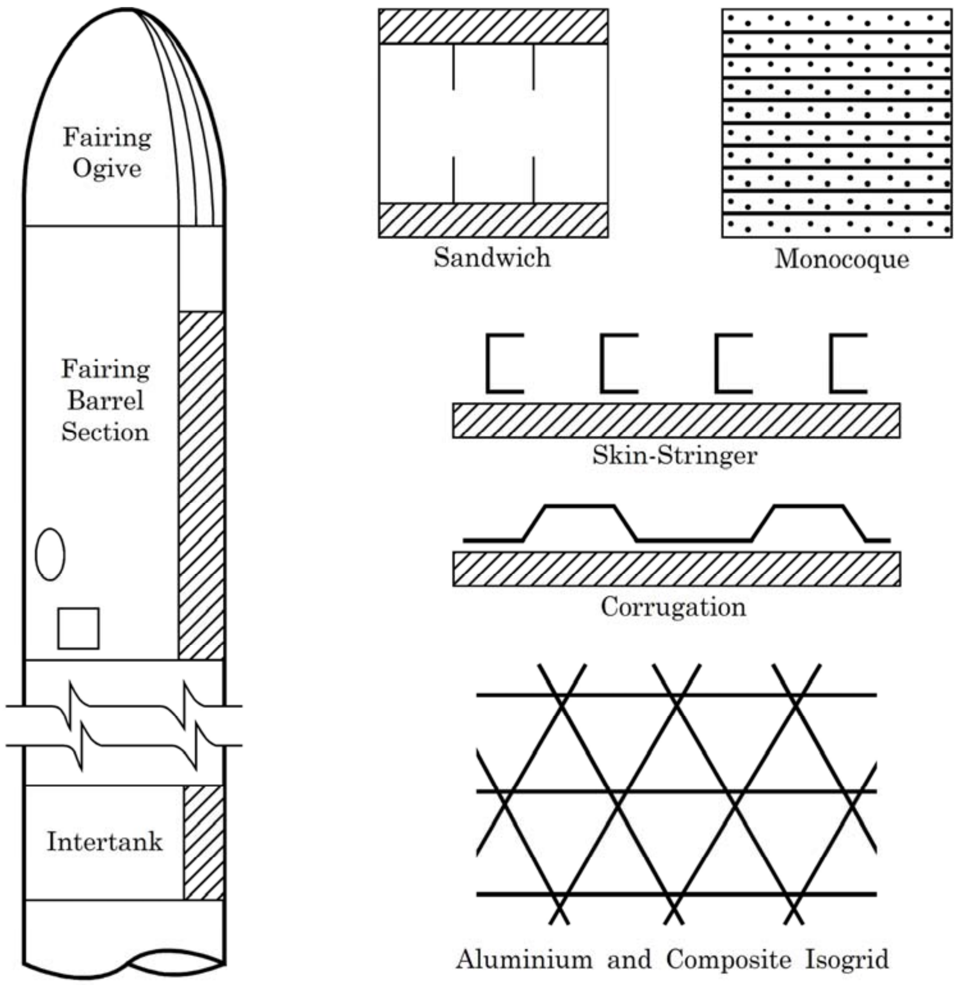
The general system requirements were:

- Fairing plus adapter mass allocation: 15000 kg.
- Intertank mass allocation: 4500 kg.
- Cylinder geometry: 10.8 m diameter.
- Cost amortised over 120 units over 10 years.

40.6.2 Potential fairing designs

40.6.2.1 General

The dimensions and prospective constructions are shown in Figure 40.6.1, Ref. [40-5]. Information is presented for the fairing cylinder and covers the direct findings only.



Information is presented for the fairing cylinder and covers the direct findings only.

Figure 40.6-1 - Candidate fairing and intertank constructions

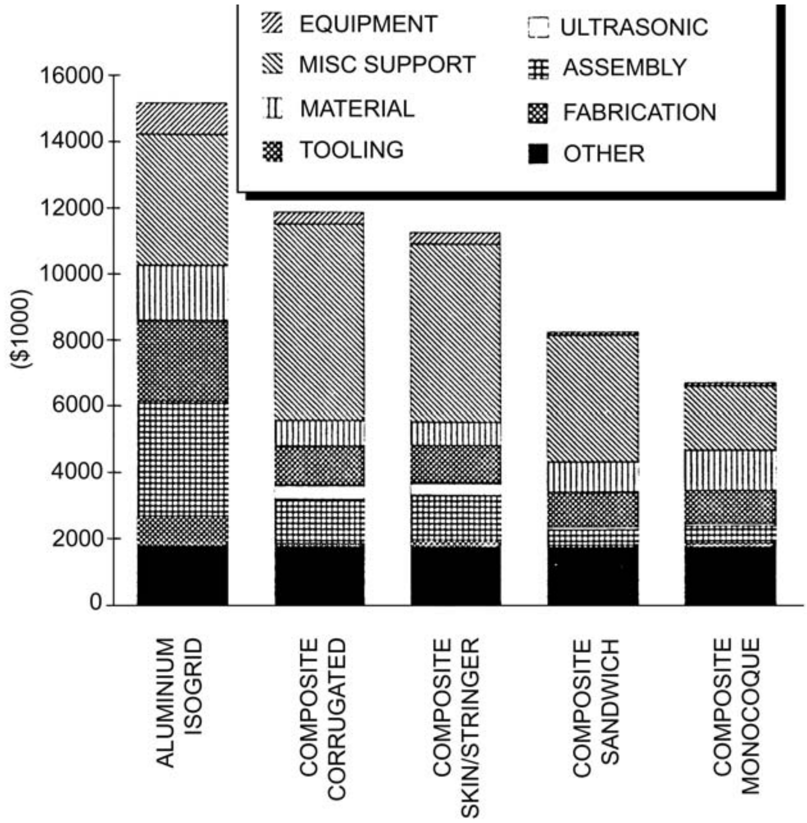
40.6.2.2 Mass-cost analysis

The mass and cost of respective fairing constructions are shown in Table 40.6.1, Ref. [40-5]. A breakdown of the contributory costs for the different designs is shown in Figure 40.6.2, Ref. [40-5].

Table 40.6-1 - Launcher fairing constructions: Mass and cost of various designs

Constructions	Unit Mass (kg)	Unit Cost ($ x1000)
GRP Monocoque	26360	-
CFRP Monocoque †	15225	6800
Al-Li Isogrid	7725	15100
GRP Sandwich	6910	-
CFRP Isogrid	5090	-
CFRP Sandwich †	5020	8300
CFRP Corrugated	4865	11800
CFRP Skin/Stringer	4455	11300

Key: † - designs for detailed examination.



Comments:

Miscellaneous Support, includes:
- design engineering,
- planning,
- operations support,
- QA,
- business management,
- shipping.

Other, includes common items, e.g.:
- utilities,
- separation hardware,
- access doors,
- nose cap.

Figure 40.6-2 - Fairing cylinder costs for different designs (life cycle costs per unit)

Based on lower production costs (from cost breakdowns), the options selected for detailed evaluation were:

- CFRP monocoque, and
- CFRP sandwich.

A key to producing low-cost structures is to minimise the part count. This reduces design analysis, development and tooling costs. Failing to achieve it accounts for the high 'miscellaneous support costs' seen with the isogrid, corrugated and skin/stringer concepts.

40.6.2.3 Design-construction trade-off

In addition to cost analysis, a decision analysis process was used to screen options against several weighted criteria; shown in Table 40.6.2, Ref. [40-5]. These criteria define the total evaluation scores.

Table 40.6-2 - Launcher fairings: Kepner-Tregoe trade study evaluation criteria

Criteria	Definition	Weighting Factor
Non-recurring cost	One time DDT and E	4
Recurring cost	Production, launch, flight and operations costs	9
Life cycle cost	Total life costs of the option, sum of non-recurring and recurring costs	10
Producibility	The capability of the option to be manufactured and assembled with relative ease and simplicity. The relative degree of the design complexity of the option	8
Mass	The physical mass of the option	7
Maintainability	The ease with which the option can be retained in or restored to an operational condition	4
Inspectability	The capability to readily inspect the part as processed or following field or transportation use	5
Repairability	The capability to repair a part damaged or found to be discrepant during fabrication and assembly	4
Flexibility	The ability to modify the design and construction for number and size of openings or alter geometry (i.e. diameter and length)	4

The fairing cylinder section scores are shown in Figure 40.6.3, Ref. [40-5], where a low score indicates a high mass or a high cost.

The criteria weights and scores were reached by consensus between the MDSSC and Hercules concurrent engineering teams.

The CFRP monocoque and sandwich constructions achieved the highest scores and underwent evaluation in the process trade and cost analysis refinements.

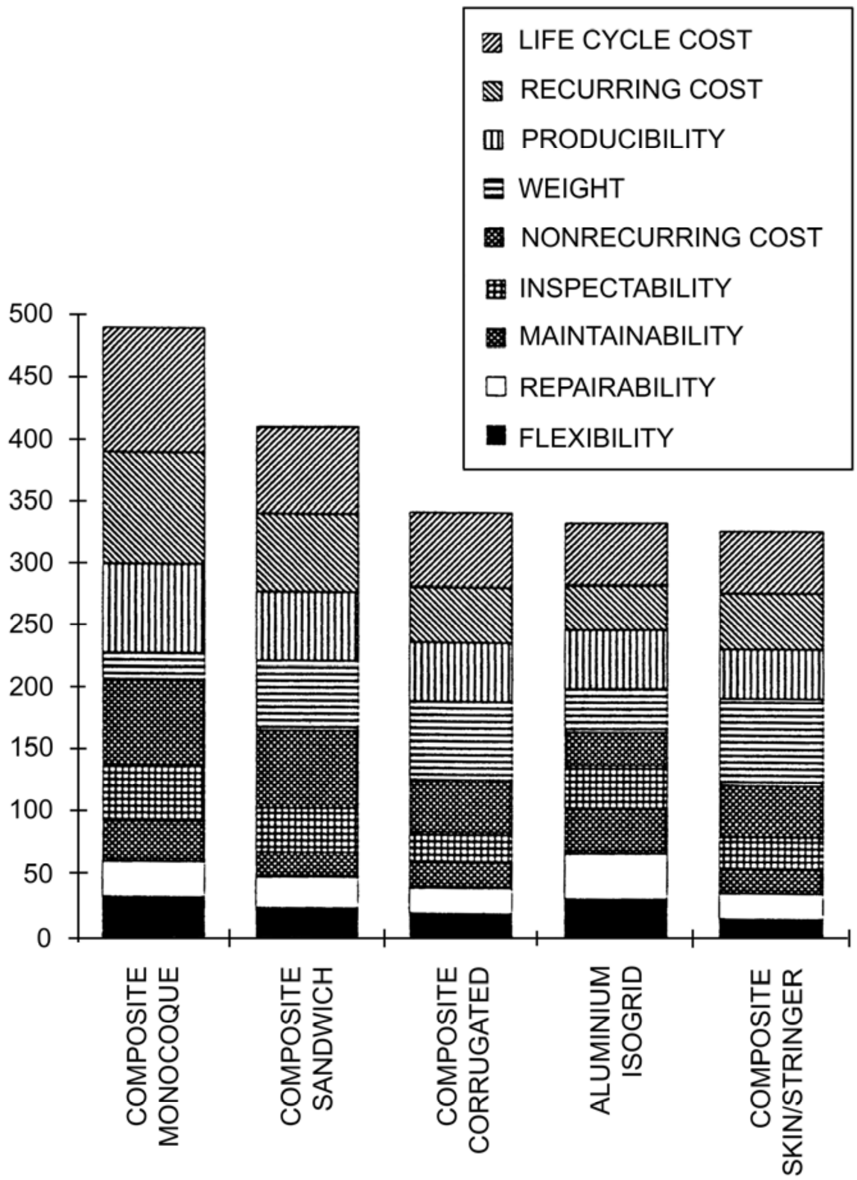


Figure 40.6-3 - Launcher fairing cylinder: Kepner-Tregoe evaluation scores for the section designs

40.6.3 Selection of designs

40.6.3.1 Fairing cylinder

The CFRP sandwich was considered the best candidate for the fairing cylinder, with CFRP monocoque as the first alternative, because:

- The system cost sensitivity to mass for the monocoque was unknown and possibly high.
- The mass margin applied, if necessary, added durability to the lightweight sandwich design.
- The monocoque development offered little benefit for improvement of the existing vehicle.

40.6.3.2 Intertank

The data were even more conclusively in favour of a sandwich construction.

40.6.3.3 Forebody

The thermal, lightning strike, erosion resistance made the semi-monocoque design with ring stiffeners more appropriate.

40.6.4 Process and material trade-off

40.6.4.1 General

Various options for fabricating large composite sections were studied. Automation had a high priority in order to avoid the high costs of hand lay-up. Also included were:

- Ancillary structures:
  - stiffeners: longitudinal and circumferential,
  - separation rails.
- Materials:
  - fibres,
  - resins,
  - adhesives, and
  - core materials.
- NDT techniques.

40.6.4.2 Sandwich fairing

The accumulated costs for the differing processes applied to a sandwich fairing are shown in Figure 40.6.4, Ref. [40-5]. It illustrates that the choice of process has a small effect on final cost. The highest and lowest cost totals differ by only 10%. This is mainly because the composite lay-up process simply generates a large panel and most of the cost of the fairing is incurred later during assembly and general support activities.

An accurate assessment of process reliability, producibility, risk minimisation and other criteria from the Kepner-Tregoe evaluation are important; as shown in Figure 40.6.5, Ref. [40-5].

Based on the processing assessment as a whole, the best options can be summarised as:

- Fairing cylinder and intertank: Broadgoods dispensing and filament winding.
- Forebody: Filament winding and fibre placement.

40.6.4.3 Materials selection

For the sandwich constructions, an intermediate modulus, IM, carbon fibre CFRP with an aluminium honeycomb core was preferred.

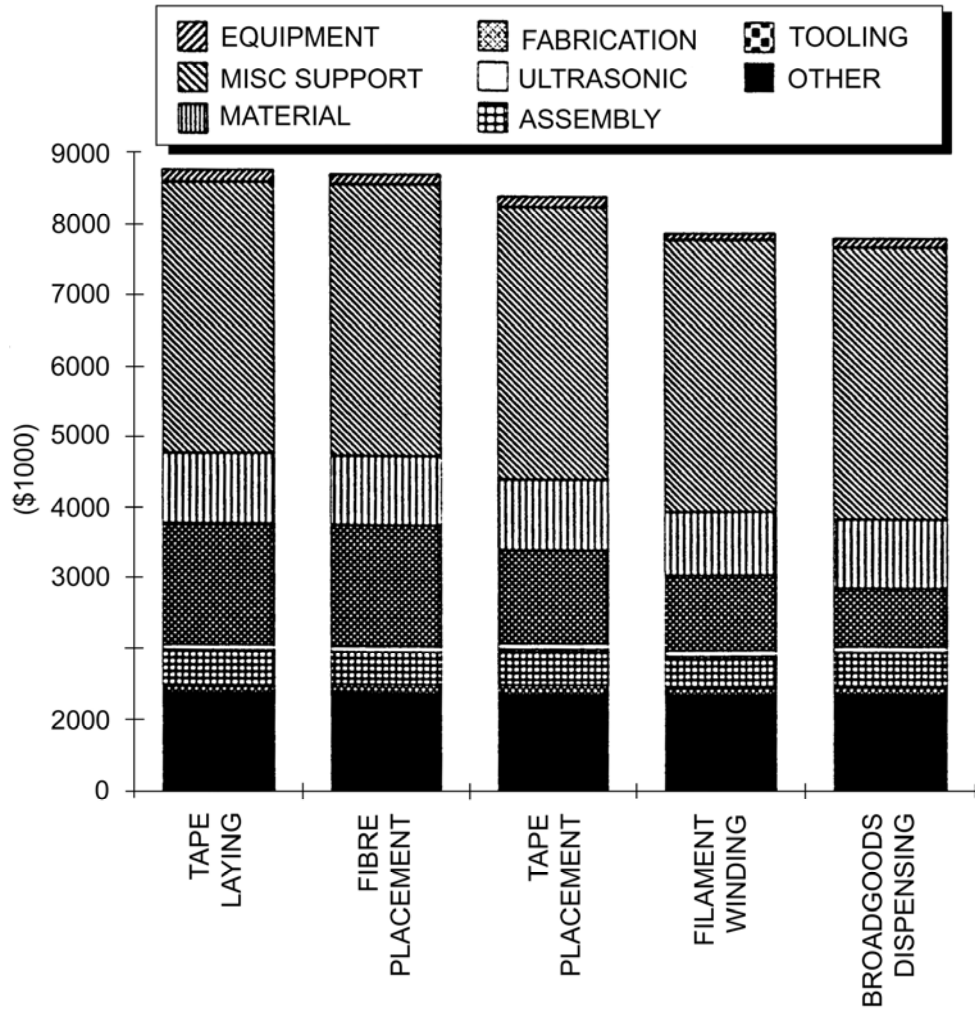


Figure 40.6-4 - Launcher fairing: Unit life cycle costs for sandwich fairing cylinder designs for different fabrication processes

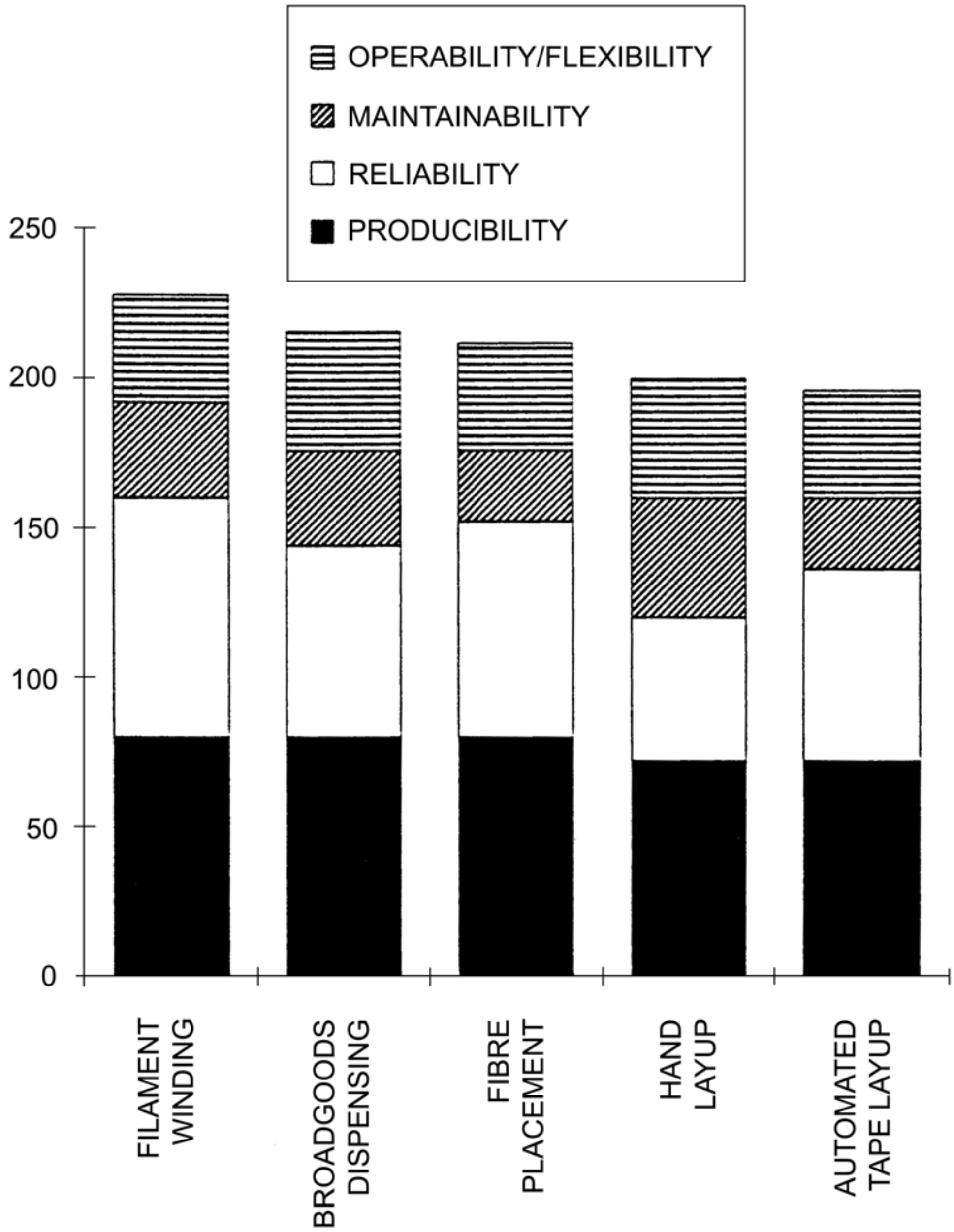


Figure 40.6-5 - Launcher fairing: Kepner-Tregoe evaluation scores for processes to fabricate the monocoque and sandwich cylinder

40.7 Thermoplastic versus thermoset

40.7.1 General

Within the composites industry, claims are made that the manufacturing costs for continuous fibre-reinforced thermoplastic composites components are lower than the equivalent thermoset versions. The potential savings are associated with the shorter processing times for thermoplastics compared with thermosets. This invites questions as to why thermosets still dominate aerospace applications, and why thermoplastic composites are virtually absent from space structures and programmes, particularly in Europe.

The position of European space contractors can be summarised as:

- The production runs for the majority of satellite constructions are small and do not warrant the use of thermoplastics.
- The range of commercial thermoplastic materials available, especially with higher modulus fibres, is small in comparison with thermoset prepregs.
- Thermoplastic prepregs are more expensive to purchase than their thermoset equivalents.
- There is little or no incentive to invest in thermoplastic forming technology when comprehensive thermoset moulding facilities exist.

Opportunities are identified where thermoplastics can be appropriate, solely on the grounds of lower total manufactured costs compared with thermosets. Other technical benefits associated with thermoplastics are not considered here.

[See also: 6.16 for technical aspects; 6.27 for economic-related factors]

40.7.2 Recurring spacecraft components

40.7.2.1 General

A cost study by TRW Space and Technology Group, USA, identified some opportunities for thermoplastics, Ref. [40-6]. The study premise was a commercial demand for small, lightweight, low-cost satellites in the future, which needs large-scale production of components (exceeding 1000 units) with a low cost per unit.

40.7.2.2 Processes

Processes were compared with autoclaving of a thermoset design; those evaluated were:

- Superplastic diaphragm forming.
- Hot-transfer press moulding.
- Filament winding.

40.7.2.3 Components

The common baseline configurations studied were:

- C-channel (diagonal brace):
  - Dimensions: 380 mm x 25 mm.
  - Mass: 36 g.
- Angle tab (to prevent buckling in beams):
  - Dimensions: 190 mm x 19 mm.
  - Mass: 10 g.
- Antenna mast support strut tube:
  - Dimensions: 2.4 m long, 57 mm internal diameter, 1.27 mm. wall thickness
  - Mass: 1150 g.

40.7.2.4 Unit costs

Unit costs were determined for production numbers of 1, 10 and 100 units.

The economic-based assumptions included:

- Labour costs included both direct and indirect costs,
- An 85% learning curve for fabrication processes,
- Vendor-supplied composite material costs at production volumes,
- Developmental start-up costs for thermoplastic processes not included in the manufacturing cost per part,
- Labour costs based on:
  - part cycle time,
  - learning curve, and
  - degree of skill required,
- Typical fabrication operations included in labour costs:
  - ply cutting,
  - lay-up,
  - bagging,
  - consolidation,
  - trim, and
  - inspection.
- Cost estimates for components consisted of:
  - nonrecurring tooling costs, i.e. design, fabrication and materials,
  - diaphragm-forming cage assembly depreciation,
  - recurring material costs and recurring fabrication labour costs derived from the cycle times.
- Items not included:
  - autoclave depreciation,
  - equipment purchase costs for the thermoplastic processing (small compared with the cost of operating an autoclave).

The relative costs for the selected components were calculated and showed that cost reductions are possible for thermoplastics when unit numbers exceed 10; as given in Table 40.7.1, Ref. [40-6].

For 1 unit or for 100 units, the cost implications are very different for each component, as shown in Figure 40.7.1, Ref. [40-6]. It illustrates that thermoplastics are not attractive until a certain number of units are made, e.g. at least 10 for C-channel and angle tabs. Whereas filament wound struts made of thermoplastics are favourable for any number of units compared with autoclaving. This is also true for filament wound thermoset when compared with autoclaving (not included in the study).

Table 40.7-1 - Selected thermoplastic component: Cost/producibility trade-off analysis

T300/934 Epoxy		IM7/PEEK Thermoplastic		Manufacturing Cost Reduction per Part (1)	
Fabrication Method	**Total Fabrication (hrs/part) (2)**	**Fabrication Method**	**Total Fabrication (hrs/part) (2)**	**10 off**	**100 off**
C-channel					
Autoclave	16.5	Superplastic diaphragm forming	6	32%	49%
Angle Tabs(4)					
Autoclave	1.1	Hot transfer press moulding	0.35	20%	43%
Antenna Mast					
Autoclave	34	Filament winding	20(3)	40%	43%

Key:

(1) Includes non-recurring tooling and recurring fabrication costs.

(2) Actual fabrication time (does not include trim/inspection time)

(3) Includes computer programming time for winding pattern

(4) Based on ship-sets of six parts

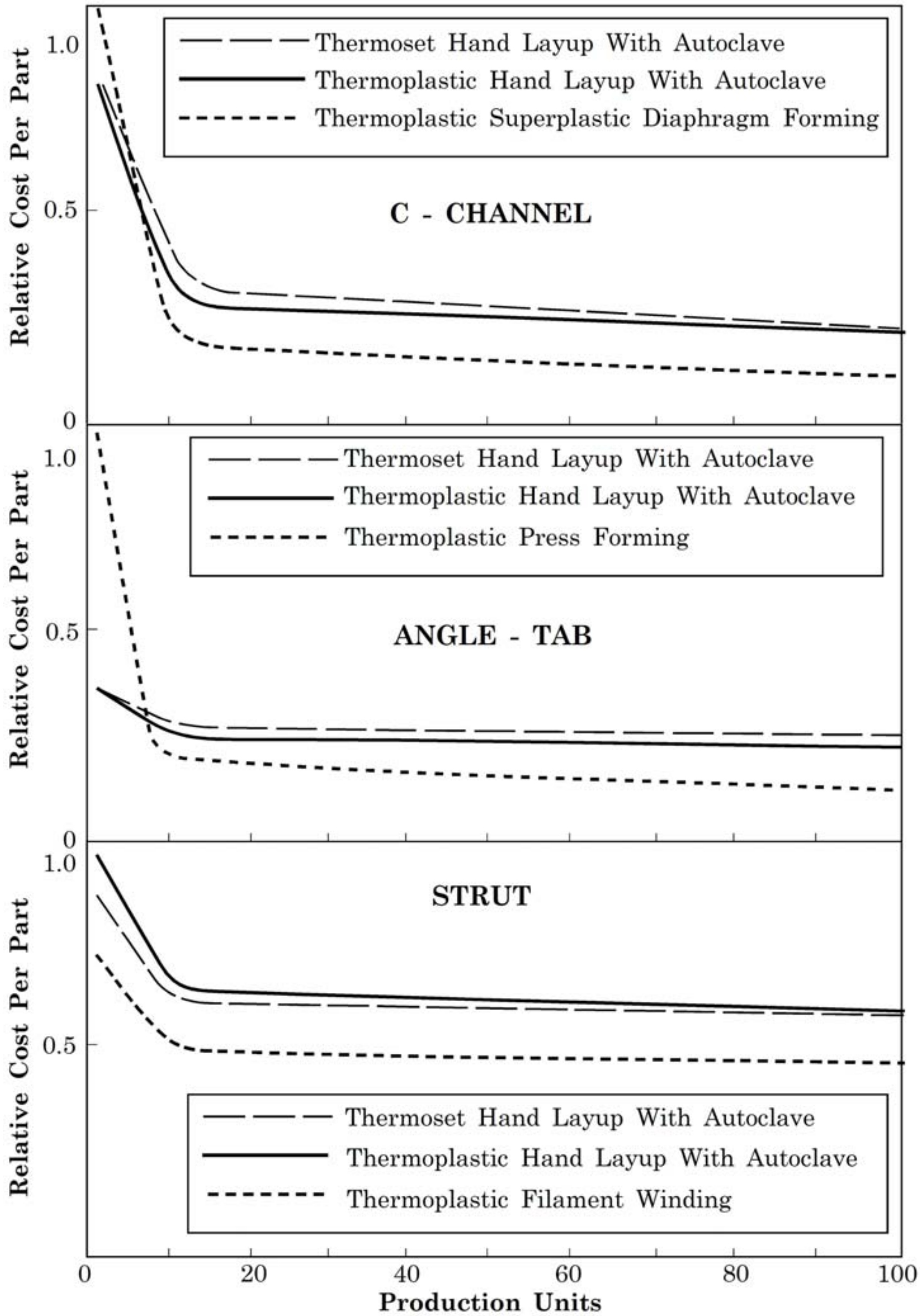


Figure 40.7-1 - Thermoplastic components: Manufacturing unit costs as a function of production volumes

Table 40.7.2 lists the major cost contributors for producing low numbers of units, i.e. from 1 to 5, Ref. [40-6]. This shows costs to be dominated by tooling.

When higher unit numbers are made, labour costs predominate. This is where thermoplastics offer the highest benefits.

Table 40.7-2 - Selected thermoplastic components: Relative manufacturing costs and major contributors to cost

Method of Manufacture	Relative Cost of 1 Unit	Major Cost Items †	Relative Cost of 100 Units	Major Cost Items †
C-Channel				
Autoclave thermoset	100	Tooling:71% Labour:26%	100	Labour:95%
Autoclave thermoplastic	99	Tooling:71% Labour:26%	96	Labour:93%
SDF thermoplastic	114	Tooling:64% Equipment:23% Labour:11%	51	Labour:84%
Autoclave thermoset	100	Labour:70% Tooling:25%	100	Labour:93%
Angle tab				
Autoclave thermoplastic	98	Labour:69% Tooling:25%	92	Labour:90%
H-TPF thermoplastic	250	Tooling:85% Labour:13%	57	Labour:74% Materials:16%
Autoclave thermoset	100	Labour:52% Tooling:37% Materials:11%	100	Labour:81% Materials:16%
Strut				
Autoclave thermoplastic	110	Labour:45% Tooling:41% Materials:14%	102	Labour:75% Materials:22%
FW thermoplastic	87	Labour:41% Tooling:36% Materials:23%	58	Labour:48% Materials:46%

Key: † : largest contributor first

40.7.3 Thermoplastic composites in aircraft

Both Fokker (NL) and Dornier Luftfahrt (D) use thermoplastic parts in production components, e.g.:

- Fokker 50 ice protection plates:
  - Cetex PEI.
- Dornier 328 ice protection plates and landing flap ribs:
  - Cetex PEI/T300 (from Ten Cate).

Ref. [40-7] gives information of the cost of the landing flap ribs.

The components are relatively simple shapes, with typical dimensions of 375 mm x 140 mm. The preferred manufacturing route is by hot pressing.

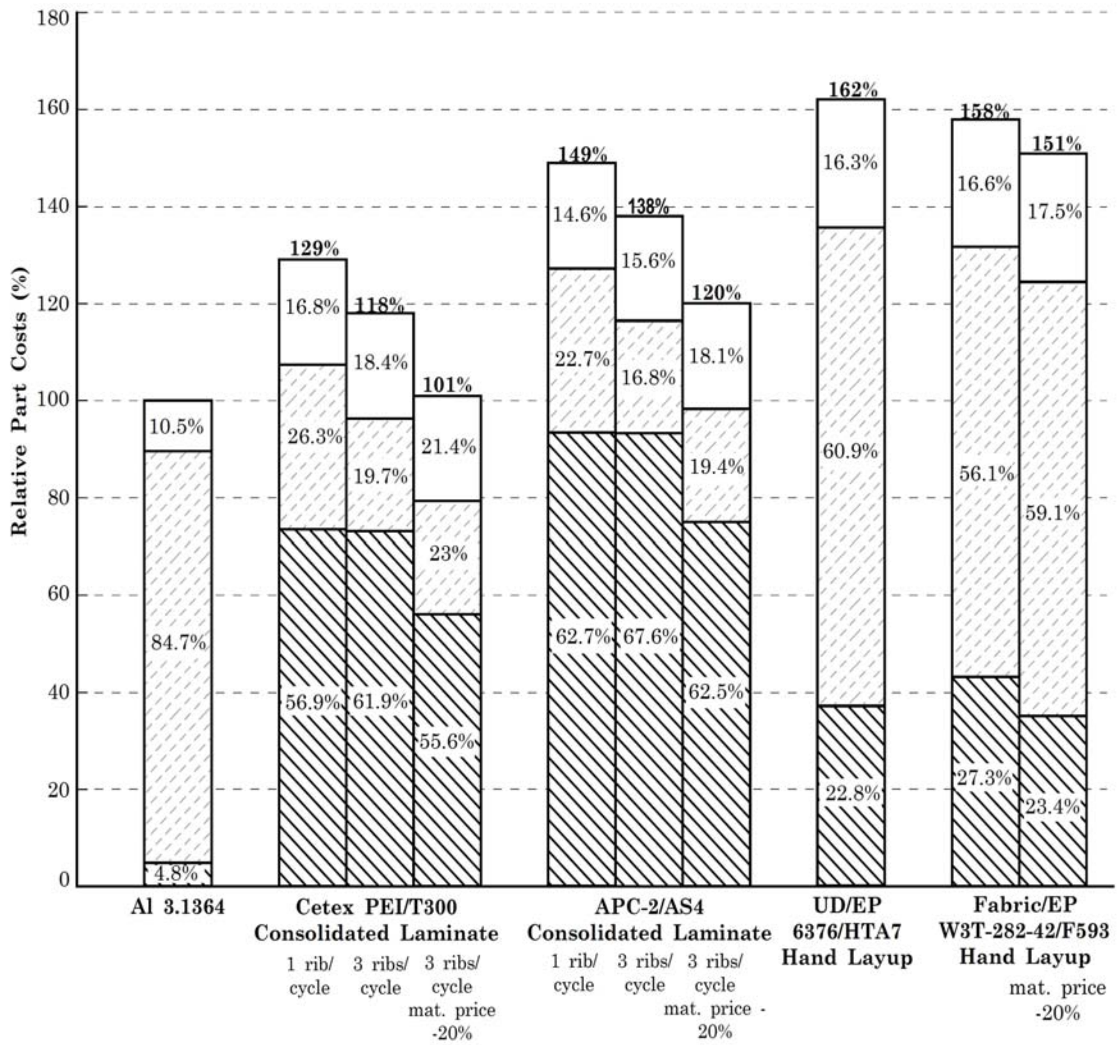
The possible constructions costed were:

- Aluminium (Al 3.1364),
- Thermoplastic Cetex PEI/T300,
- Thermoplastic ICI PEEK APC-2/AS4,
- UD Epoxy CFRP (6376/HTA7), and
- Fabric Epoxy CFRP (W3T-282-42/F593).

The thermoplastic variants were costed for both 1 and 3 ribs per cycle and a 20% reduction in material prices. Figure 40.7.2 shows a breakdown of the relative part costs, Ref. [40-7].

It was concluded that the thermoplastic variant was cost-effective compared with thermosets, but not with aluminium unless three parts were made simultaneously in each cycle.

The high material cost of PEEK APC2 is a disadvantage compared with the cheaper Cetex PEI.



Key: Calculation basis: Landing flap rib thickness: 1.25 mm in CFRP, 1.0 mm in Al. Area: 0.11 m². Mass: 220g (CFRP), 300g (Al). For 400a/c, all costs in 1991, numbers include quality costs and material surcharges.

Figure 40.7-2 - Relative part costs for thermoplastic aircraft components: Dornier 328 landing flap rib

40.7.4 Conclusions

Some conclusions on the cost viability of thermoplastic composites include:

- Autoclave moulded thermoplastic composites do not offer any cost-savings over autoclaved moulded thermosets.
- Small to medium sized, simple shapes can be made at lower cost in thermoplastics compared with thermoplastics, providing that rapid forming techniques are used.
- Unit production needs to exceed a certain number before it is attractive to use thermoplastics.
- For complex shapes, the cost difference between thermoset and thermoplastics reduces considerably.
- The use of thermoplastic composites in space structures can be considered where unit production numbers are relatively high, such as for:
  - launchers
  - common satellite structures, e.g. within series of communication and micro-satellites.

40.8 Automation

Automated composite processing is only of value if it achieves cost-savings, compared with labour-intensive activities, which exceed the capital equipment cost.

The basic areas where this can be the case are:

- Cutting and stacking of prepregs, i.e. lay-up.
- High numbers of units produced.

Given the limited consumption of materials for satellite constructions, automation can be difficult to justify. The market for launchers is different because unit numbers are higher and a larger amount of material is handled and processed.

For designers of automated systems, the cost-effectiveness is an important design parameter from the original design concept stage. A larger percentage of the final cost of a part is determined in the early phases of the life cycle of a product, i.e. at the design phase as opposed to production phase.

A study on the opportunities for automation concluded that, Ref. [40-8]:

- The cost effectiveness of a manufacturing process is a trade-off between raw material costs and downstream manufacturing costs determined by that material.
- High cost, value-added materials are most cost-effective since they need the least amount of labour during processing. i.e. 3D constructions, broadgoods, preforms, braids and fabrics as opposed to UD tape.
- Automated techniques which reduce labour can use low cost materials effectively, e.g.:
  - automated tape lay-up, and
  - filament winding.

40.9 Cost analysis methods

Several attempts have been made to devise methods for quantifying the cost of producing composite components by different manufacturing routes, Ref. [40-6], [40-8], [40-9], [40-10], [40-11].

Generally, such exercises are only of value where the number of component units produced is reasonably high.

The analysis methods attempt to compare different processes that often produce subtly different components. The methods often make assumptions and can only produce valid results if there is agreement on:

- Costing of labour and overhead rates.
- Equipment costs.
- Allocation of development costs.

40.10 References

40.10.1 General

[40-1] University of Delaware
'Composite design guide'
Issue 9, 1983

[40-2] G. Lubin
'Handbook of composites'
Van Nostrand Reinhold, 1982

[40-3] V.M. Karbhari & D.J. Wilkins
'Selecting materials, processes & shapes: getting it right the first time'
37th International SAMPE Symposium
March 9-12, 1992, p1379-1391

[40-4] B.A. Reid & A. Pradier
'Cost optimisation of load-carrying structures'
Proceedings of the International Symposium 'Space Applications of Advanced Structural Materials'
ESTEC, The Netherlands, 21-23 March 1990, ESA SP-303, p397-402

- BAe TP8598 - Cost optimisation of load carrying structures, Phase 1 Final Report, ESTEC Contract No. 7529/88/NL/PH
- BAe TP8673 - Cost optimisation of load carrying structures, Phase 2 Final Report, ESTEC Contract No. 7529/88/NL/PH
- BAe TP8683 - Cost optimisation of load carrying structures guidelines, Phase 1 Final Report, ESTEC Contract No. 7529/88/NL/PH

[40-5] M.J. Robinson et al
'Advanced composite structures for launch vehicles'
SAMPE Quarterly, January 1991, p26-37

[40-6] E.M. Silverman & W.C. Forbes
'Cost analysis of thermoplastic composites processing methods for spacecraft structures'
SAMPE Journal, Vol. 26, No. 6, November/December 1990, p9-15

[40-7] W. Werner
'Cost effectiveness of structural applications of fibre reinforced thermoplastics'
Proceedings of the International Symposium 'Advanced Materials for Lightweight Structures', ESTEC, The Netherlands
25-27 March 1992, ESA SP-336, p185-190

[40-8] M.F. Foley & E. Bernardon
'Thermoplastic composite manufacturing cost analysis for the design of cost effective automated systems'
SAMPE Journal, Vol. 26, No. 4, July/August 1990

[40-9] M.F. Foley
'Techno-economic automated composite manufacturing techniques'
SAMPE Quarterly, January 1991, p62-68

[40-10] E. Wang & T. Gutowski
'Cost comparison between thermoplastic & thermoset composites'
SAMPE Journal Vol.26 No.6, Nov/Dec 1990, p19-26

[40-11] T. Gutowski et al
'Development of a theoretical cost model for advanced composite fabrication'
Composites Manufacturing, Vol. 5, No.4, 1994, p231-239

41
Repair of composites

41.1 Introduction

Damage to composite structures has been studied, and the types of damage categorised, [See: 41.2]. Repair concepts have been developed for laminates and sandwich structures.

The means of categorising types of damage are described. The various repair concepts developed to restore the properties of the structure are presented, [See: 41.4].

41.2 Damage classification

41.2.1 Life-histories of composite structures

NASA and prime aircraft contractors have compiled life histories of composite structures. This showed that nearly all types of damage to structures occur from:

- ground handling during assembly, or
- maintenance, and
- impact of foreign object.

[See: 41.3 for all the types of damage that need to be repaired]

41.2.2 Inspection and criticality of damage

Each of the accidental damage types are assessed using various inspection techniques and then classified regarding their criticality. The inspection techniques commonly used, either singularly or combined, are, [See also: Chapter 34]:

- Visual examination.
- Tapping test.
- Ultrasonic examination.
- X ray examination.

41.2.3 Repair zones

41.2.3.1 General

The aircraft industry usually divides a structure into repair zones, and determines the 'allowable damage' for each zone, Ref. [41-3]. The structural components can be classified as:

- Structural components (safe-life design)
- Primary structural components (fail-safe design)
- Secondary structural components

41.2.3.2 Structural components (safe-life design)

Such parts are classified as those whose failure can result in a significant loss of structural performance, i.e. launcher, satellite or space structure.

The repair concept should meet all the aspects of a 'safe-life' design. If it cannot, the whole part is scrapped and replaced.

41.2.3.3 Primary structural components (fail-safe design)

Failure of a primary component can have serious consequences on the operation of a structure, e.g. reducing the performance of a 'safe-life' component under cyclic loading. All such components are subject to further inspection.

A repair to a damaged primary structure parts has to guarantee the original strength and stiffness throughout the inspection interval.

41.2.3.4 Secondary structural components

Failure has no direct or immediate effects on the safe operation of secondary structural parts.

Repairs are normally for 'cosmetic' reasons and reconstitute the component profile.

41.3 Damage categories

41.3.1 Impact energy levels

The types of damage which lead to the failures, [See: 41.2], are usually classified by their impact energy level, i.e.:

- Low energy impact at moderate speed, e.g.:
  - handling incidents,
  - dropping a tool during assembly or maintenance.
- High energy impact at high speed, e.g.:
  - hail,
  - small stones from the runway.
- High energy impact at very high speed, e.g.:
  - In-orbit debris,
  - meteoroids.

41.3.2 Damage types

41.3.2.1 General

The damage produced by each type of impact is categorised and a suitable repair procedure considered, [41-1], [41-2], [41-3].

41.3.2.2 Scratches

The damage has no effect on the structural strength, but is repaired in order to prevent a subsequent attack of the part by humidity, UV, particle radiation or atomic oxygen.

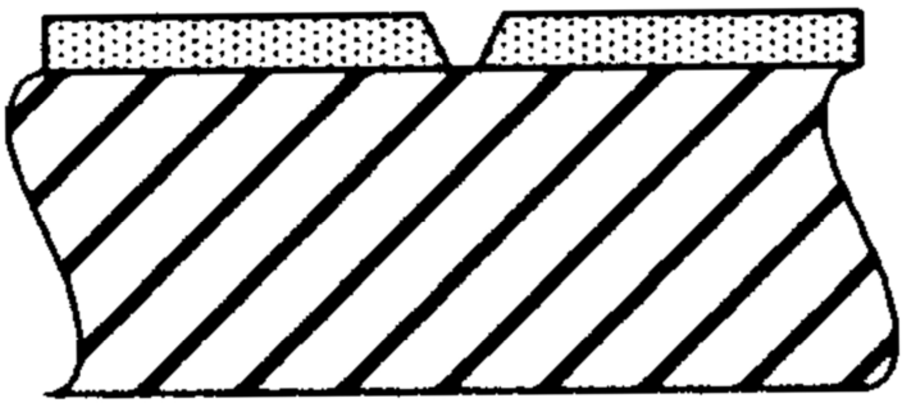


Figure 41.3-1 - Damage categories: Scratch

41.3.2.3 Notches

The damage does not extend through the full thickness of the part. The effects on the mechanical performance depend on the depth of the notch relative to the component thickness.

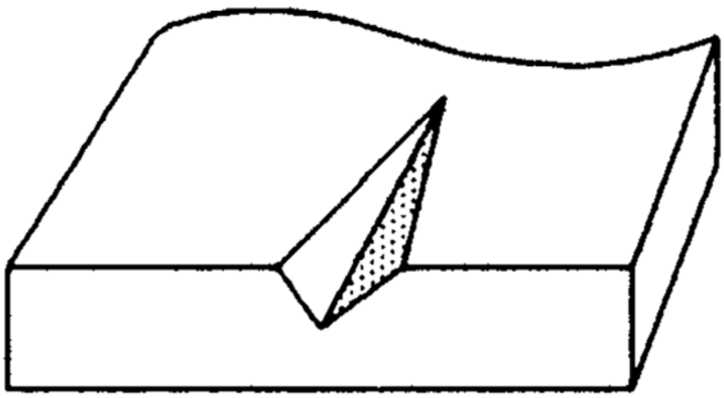


Figure 41.3-2 - Damage categories: Notch

41.3.2.4 Chipping

This is a local fracture with separation of the surface plies. The effect on the mechanical performance depends on the thickness of the part. Repair is necessary to prevent the structure from subsequent attack by external agents, e.g. humidity, radiation, atomic oxygen.

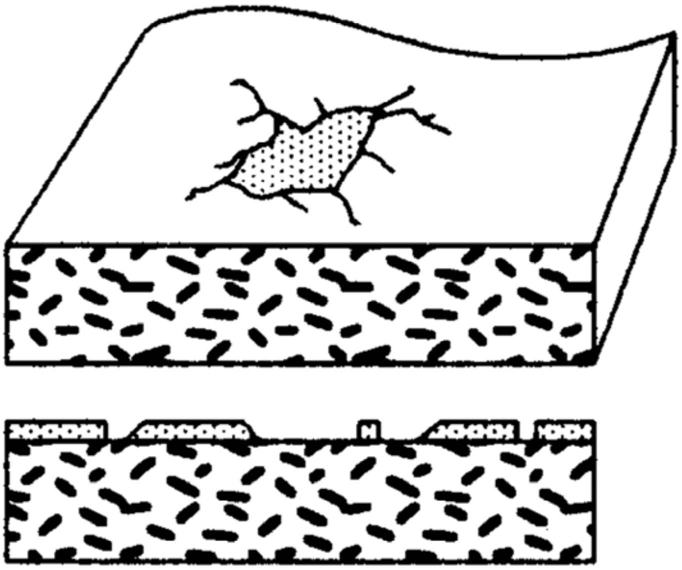


Figure 41.3-3 - Damage categories: Chipping

41.3.2.5 Debonding

An example is the separation of sandwich skin and the core. The effects depend on the size of the separated area. Repair of the damage zone is necessary if the separated area is greater than a specified value.

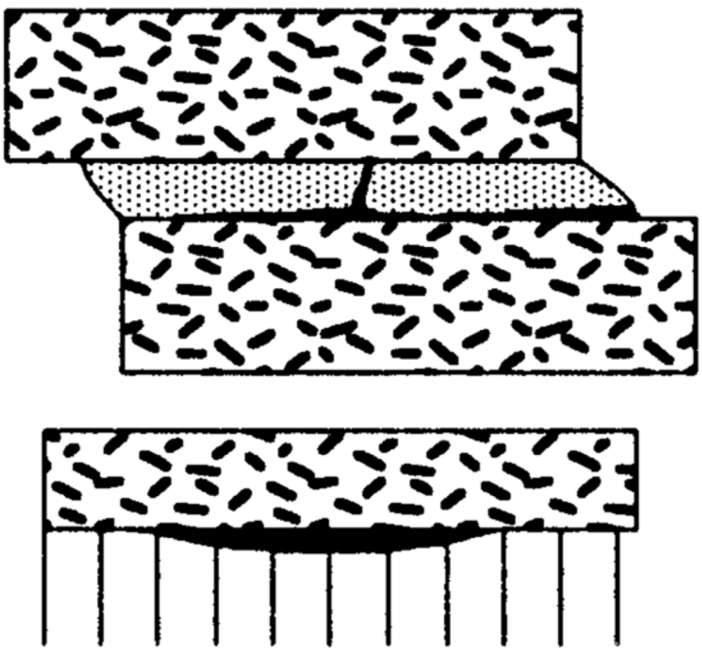


Figure 41.3-4 - Damage categories: Debonding

41.3.2.6 Delamination and dents

This occurs due to the separation or from a lack of adherence between two plies of a laminate. It is of major importance, especially in thin compression-loaded panels (stability failure) or in thick panels sub-jected to interlaminar shear loading. Delaminations are repaired to retain the mechanical performance.

The combination of delamination and debonding - dents - often occurs after an impact on the structure. Depending on the energy level of the impact, the fibres or the plies can be broken.

Figure 41.3-5 - Damage categories: Delamination

41.3.2.7 Perforation

Depending on the impact energy, a perforation can affect part or all of the component thickness. It has major effects on the compression properties of a structural component. Perforation also causes leaks in tanks or pressurised structures.

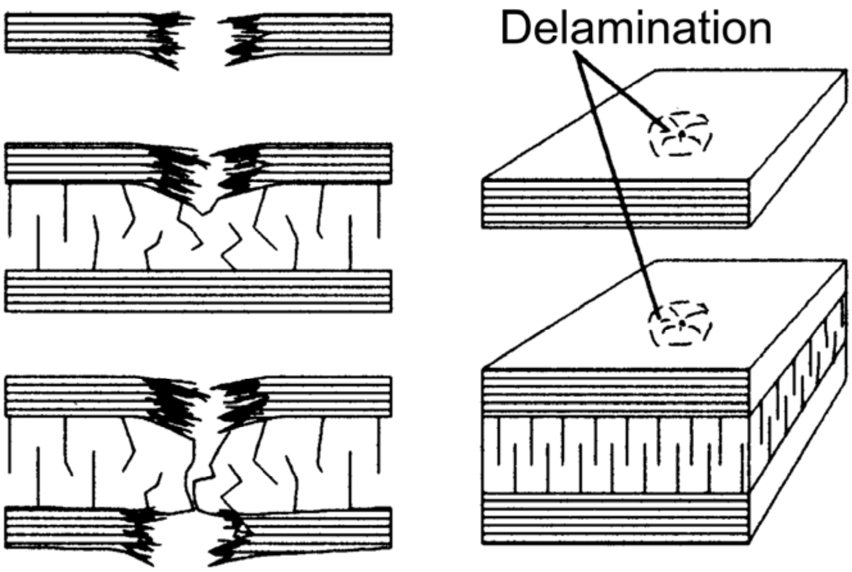


Figure 41.3-6 - Damage categories: Perforation

41.4 Basic repair concepts

41.4.1 General

The repair principles of the aircraft industry, which are based on the FAA regulations, state that:

'After the repair to a structural part made of composite materials, there should be no degradation in the ultimate design strength of the part for its remaining service life'.

41.4.2 Basic types of repairs

Repairs are usually made by reinforcing the damaged component with a metal or composite patch.

The principal methods are:

- Flush repairs, where patches are bonded or cured in place, [See: 41.5].
- External repairs, where patches are:
- bonded, [See: 41.6].
- bolted, [See: 41.7].

Typical repair concepts are shown schematically in Figure 41.4.1, Ref. [41-1].

41.4.3 Repair selection

The general approach within the aircraft industry is that the choice of a repair concept is defined in the appropriate structural repair manuals, and depends on the:

- type of damage, [See: 41.3].
- criticality of the damage related to the importance of the structural component, [See: 41.2].

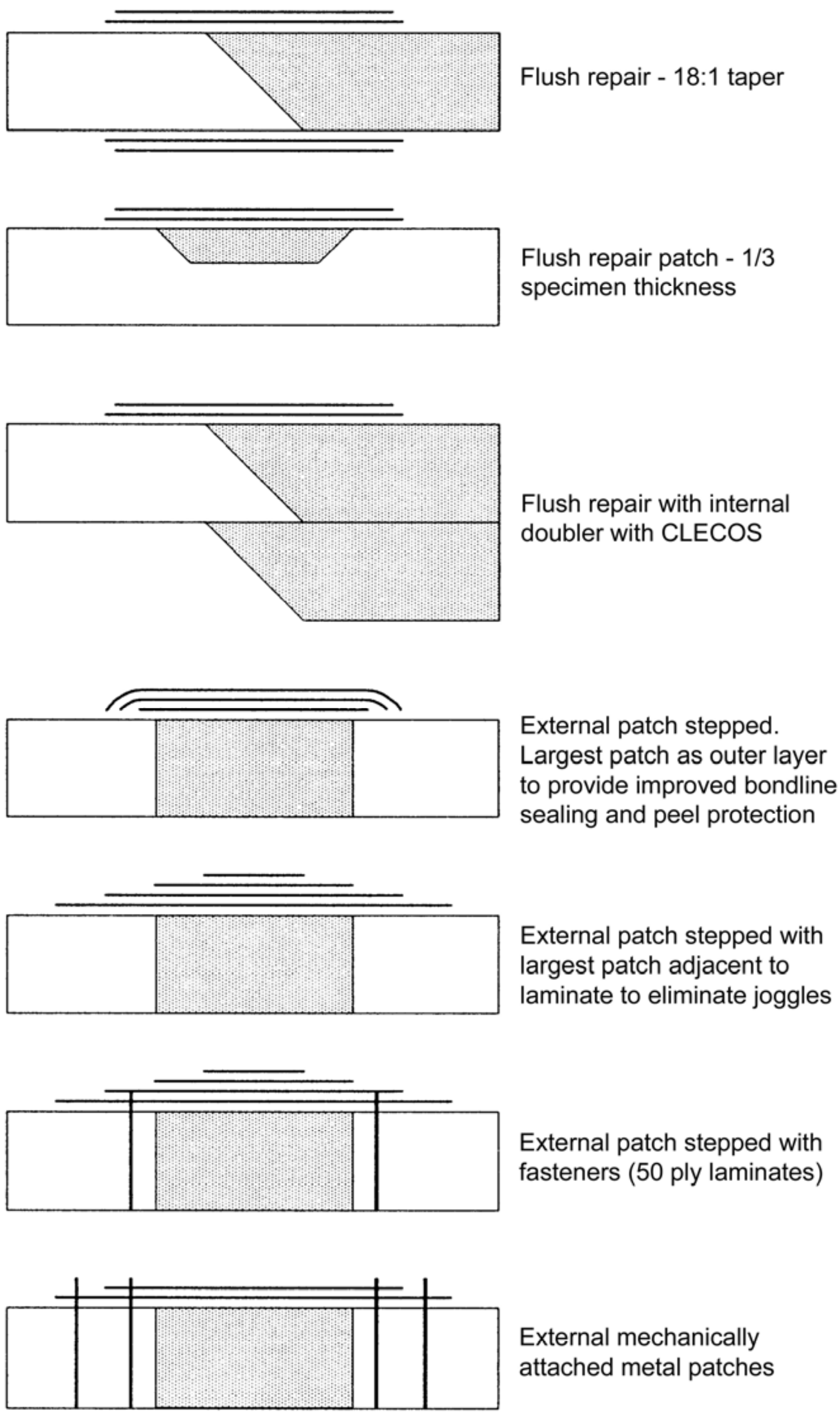


Figure 41.4-1 - Basic repair concepts

41.5 Flush repair concepts

41.5.1 Applications

Flush-bonded patches of composites can be necessary to rebuild the originally smooth surface of a damaged structural component.

41.5.2 Post-repair properties

Test results, Ref. [41-4], show that flush repairs provide the most effective restoration of the original strength, i.e. without defect.

The strength recovery is about 60 % for thick laminates, which is higher than the typically stated 40 % to 60 % of ultimate strength (design ultimate strength).

41.5.3 Design

The possible designs of a flush repair are, Ref. [41-5]:

- The removed material is replaced in the same orientation and the same stacking order that used in the original component; as shown in Figure 41.5.1.
- The removed material is replaced parallel to the surface; as shown in Figure 41.5.2.

The 'parallel method' is used by Airbus Industrie and provides the maximum bond strength between the component and the patch. This concept is also cheaper than the other method to implement.

41.5.4 Manufacture

Both types of repairs are cured in place using heat-blankets and special types of vacuum bags. Additional cover plies and the use of an adhesive film between the component and the patch are advised; as shown in Figure 41.5.3.

If the component has already been in-service, the laminate is dried in order to recovery the maximum strength.

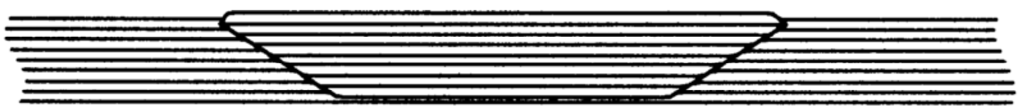


Figure 41.5-1 - Typical flush repair

Figure 41.5-2 - Flush repair, used by Airbus Industrie

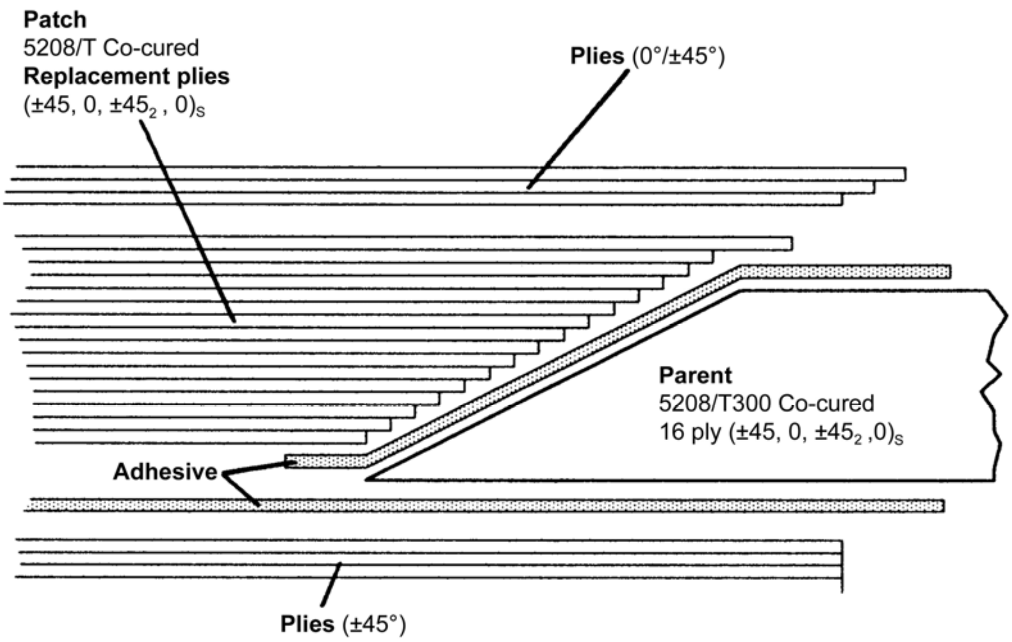


Figure 41.5-3 - Flush repair with adhesive film and cover plies

41.6 Bonded external repair concepts

41.6.1 Applications

An external patch offers an intermediate level of complexity and cost. It is usually used for lightly-loaded, thin laminates.

41.6.2 Post-repair properties

The strength recovery is typically between 25 % and 60% of the original strength, Ref. [41-4]. This indicates that the design strength (typically 40 to 60% of the ultimate strength without defects) is possible.

The curing process is a significant parameter regarding strength recovery; the higher the curing temperature, the higher the strength of a repaired component.

41.6.3 Design

The design varies depending on whether the plies are pre-cured or cured in place, [See: Manufacture].

41.6.4 Manufacture

Patches are either:

- Pre-cured; as shown in Figure 41.6.1, or
- Cured in place; as shown in Figure 41.6.2.

If the component has already been in-service, the repair area is dried before the patches are bonded in order to maximise strength recovery.

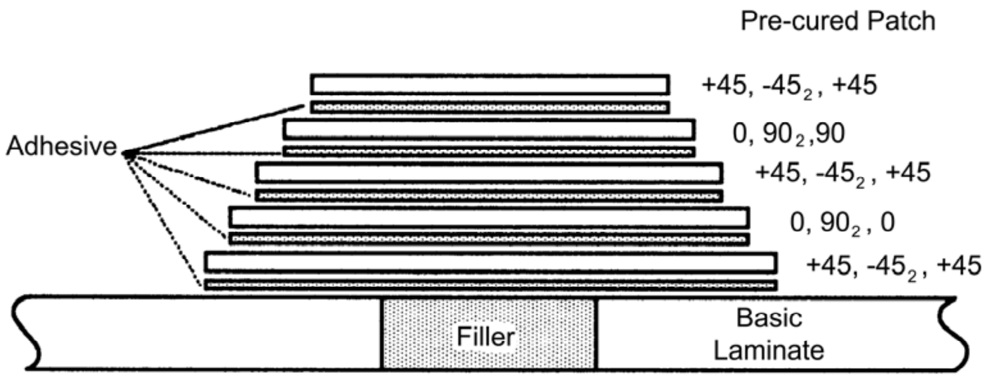


Figure 41.6-1 - External repair: Pre-cured patches

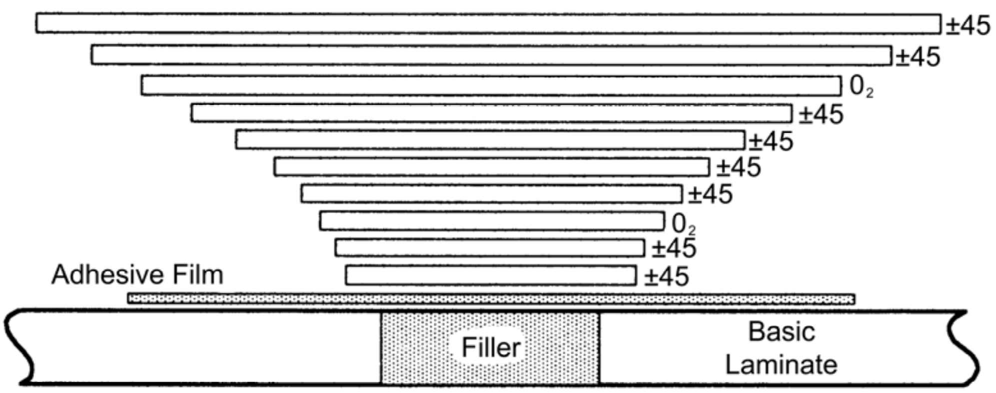


Figure 41.6-2 - External repair: Cured in place

41.7 Bolted external repair concepts

41.7.1 Applications

Bolted repairs are normally used for:

- Delamination in significant components, e.g. skin/stringer structures,
- Prevention of peeling within a bonded external patch.

Lightly-loaded, structural components or secondary parts can be repaired by this method.

41.7.2 Post-repair properties

For structural parts, the repair has to restore at least the ultimate design strength; typically 40 % to 60% of the undisturbed ultimate strength.

41.7.3 Design

The design varies depending on the:

- Location
- Use of a metal patch, or
- Use of a pre-cured composite patch; as shown in Figure 41.7.1.

Special care is taken when drilling the holes for fasteners to prevent additional delamination of the laminate.

A sealant is used between the CFRP component and the metal patches to inhibit galvanic corrosion problems.

Often blind fasteners are used because the access to both sides of a structural component is reduced. Blind fasteners have low pull-out strength; this type of repair is normally used for temporary repairs only.

41.7.4 Manufacture

Repairs to a damaged component are made by using metal or composite patches which are secured by:

- Bolting only.
- Bonding and bolting.

[See also: Design]

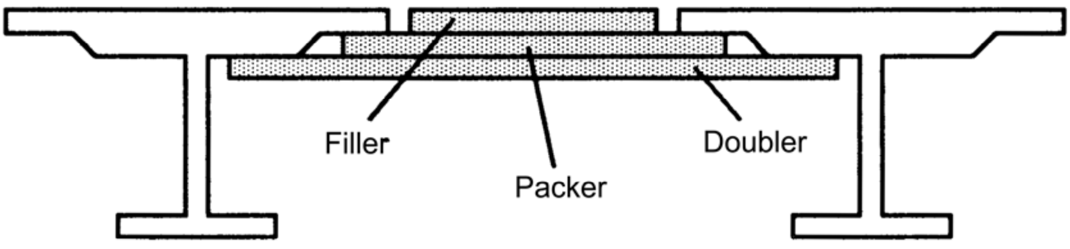


Figure 41.7-1 - Bolted and bonded repair using a composite patch

41.8 Sandwich structure repair concepts

41.8.1 No core damage

When the honeycomb or foam core of a sandwich structure is intact, the repair scheme is the same as for a thin laminated component accessible from only one side, [See: 41.6 and 41.7].

41.8.2 Core damage

When the core is crushed severely, it should be replaced. Otherwise the space is filled with a mixture of resin and microballoons.

41.8.3 Manufacture

The repair sequence for a sandwich panel with a damaged core depends on whether it is accessible from:

- One side, or
- Both sides.

If the sandwich panel is accessible from one side only, the area of damaged core is removed and a pre-cured composite plate, bonded to a honeycomb plug, is then fitted. The skin is then closed by a typical flush repair.

Figure 41.8.1 shows an example of a sandwich panel repair.

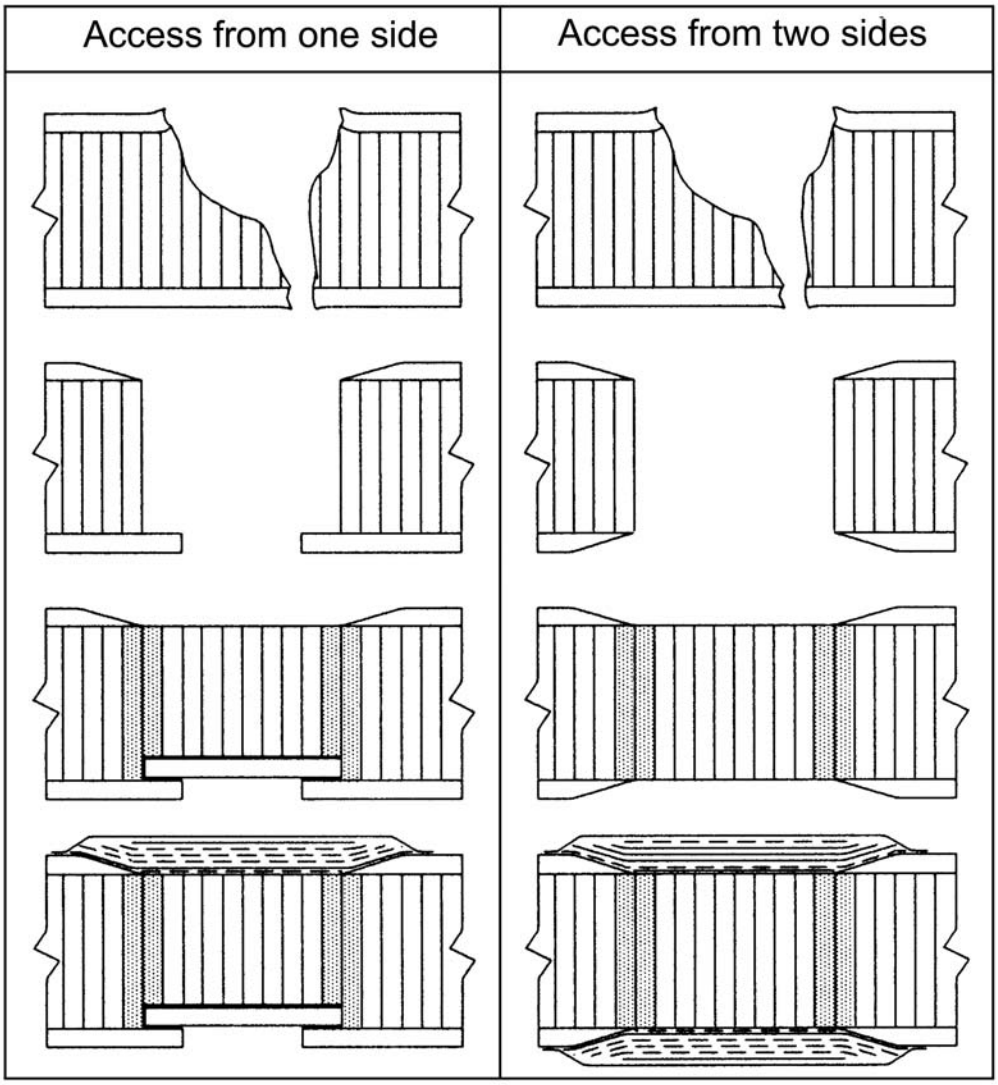


Figure 41.8-1 - Sandwich repair

41.9 References

41.9.1 General

[41-1] M. Torres & B. Plissonneau
'Repair of helicopter composite structure techniques and substantiations'
AGARD Conference Proceedings No. 402

[41-2] Th. Thiele
'Repair procedure for composite parts on the Alpha Jet'
AGARD Conference Proceedings No. 402

[41-3] Airbus Industrie
A320: Structural repair manual

[41-4] R.H. Stone
'Development of repair procedure for graphite/epoxy structures on commercial transports'
SAMPE Monograph No. 1 - Composite Repairs

[41-5] G. Lubin
'Handbook of composites'
Van Nostrand Reinhold Company, 1982

[41-6] J.F. Knauss & R.H. Stone
'Demonstration of repairability and repair quality on graphite/epoxy structural sub-elements'
SAMPE Monograph No. 1 - Composite Repairs

42 Basic characteristics of new advanced materials

42.1 Introduction

The basic characteristics of the various groups of new advanced materials in ECSS-E-HB-32-20 are described. It serves to highlight reasons for the development of the different groups of materials and the problems encountered in doing so. Information is presented and discussed in further detail in subsequent chapters, including:

- Metals and MMCs
- Technical ceramics and ceramic-based textiles
- CMCs
- Carbon-carbon

Design aspects of the various material groups are given, along with examples of their actual or intended application in space structures.

The majority of information is related to their high-temperature performance, gained from various reusable vehicle concepts.

Smart technologies and the various smart materials are also presented for space applications

42.2 Material groups

42.2.1 Metallic materials

Information on more conventional and well-developed materials such as aluminium and titanium alloys is not given in detail. The resumé of their characteristics is used as a starting point for the newer developments, including:

- Aluminium-lithium alloys, [See: Chapter 46].
- Dispersion strengthening
- Powder metallurgy alloys
- Superplastic alloys (SPF/DB)
- Metal matrix composites:

Reference is made to source documents for the established alloys in aerospace applications.

[See: Chapter 46 for aluminium; Chapter 47 for titanium]

42.2.2 Ceramic materials

42.2.2.1 General

Ceramics can be broadly grouped as:

- Reinforcing fibres, [See: 42.6]
- Monolithic, which is a large family of different materials also known as technical ceramics. These are often produced from powders, [See: Chapter 43].
- Fibre-reinforced composite, which are then classed by the matrix phase, e.g.
  - silicon carbide, [See: Chapter 52]
  - glass and glass-ceramic, [See: Chapter 53].
  - carbon, [See: Chapter 54]

42.2.2.2 High temperature applications

Fibre-reinforced ceramic composites and carbon-carbon materials offer attributes for use in high-temperature applications; notably in propulsion and thermal protection systems. Significant studies relating to materials development and evaluation occurred during reusable space plane programmes.

42.2.2.3 Dimensionally stable applications

The low thermal expansion characteristics offered by monolithic ceramics, various CMCs and carbon-carbon composites have led to their evaluation for structures needing dimensional stability over a wide range of temperatures from cryogenic upwards, e.g. supports for optical equipment, mirrors for space-based telescopes.

42.2.3 Material classification

42.2.3.1 High temperature capability

Materials can be classified by generic type, e.g. polymer, metallic or ceramic-based, [See: Chapter 1], or by temperature capability, e.g. those offering capabilities above 600°C and ultimately towards 2000°C.

Figure 42.2.1 provides a broad indication of ultimate temperature capabilities, which can be defined to a point where the materials are essentially stable. Beyond this point degradation accumulates and the materials residual properties can only be useful for a restricted duration.

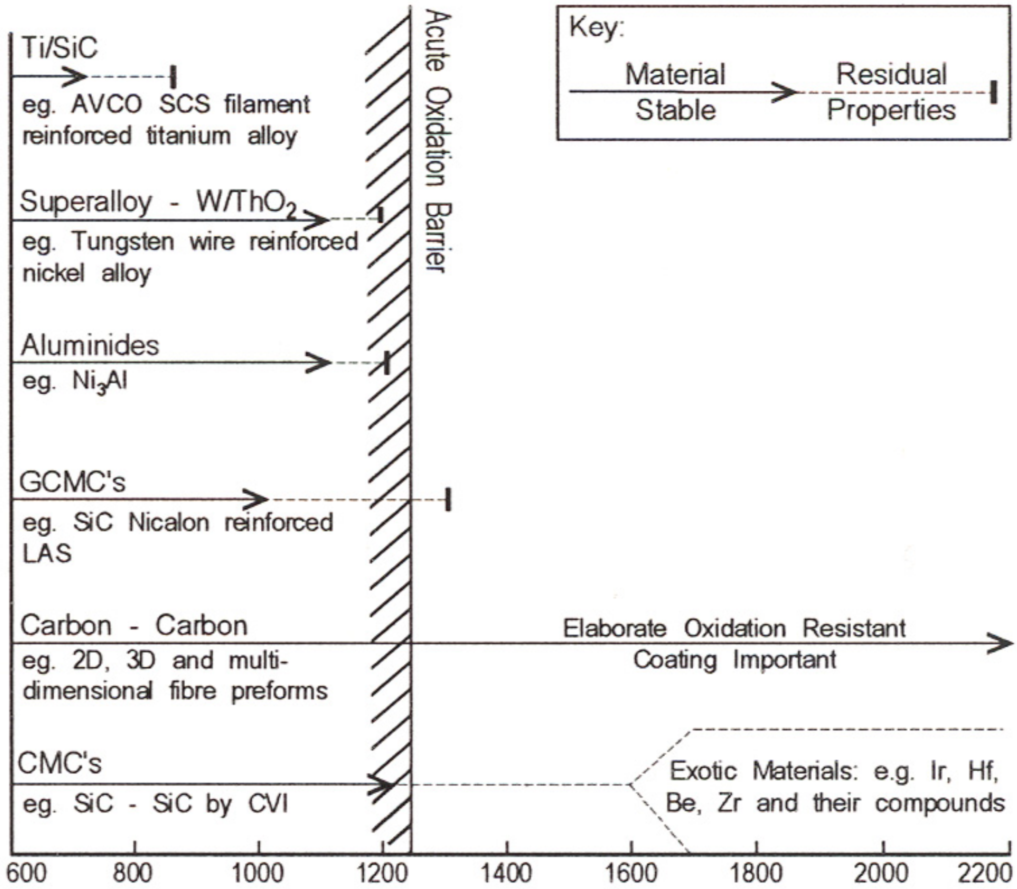


Figure 42.2-1 - Classification of advanced metallic and ceramic materials with temperature

Figure 42.2.2 shows specific strength against increasing temperature for different materials. Other than carbon-carbon composites, which show increasing strength with temperature, the trend is for a gradual strength reduction as temperature increases and the material options become more limited.

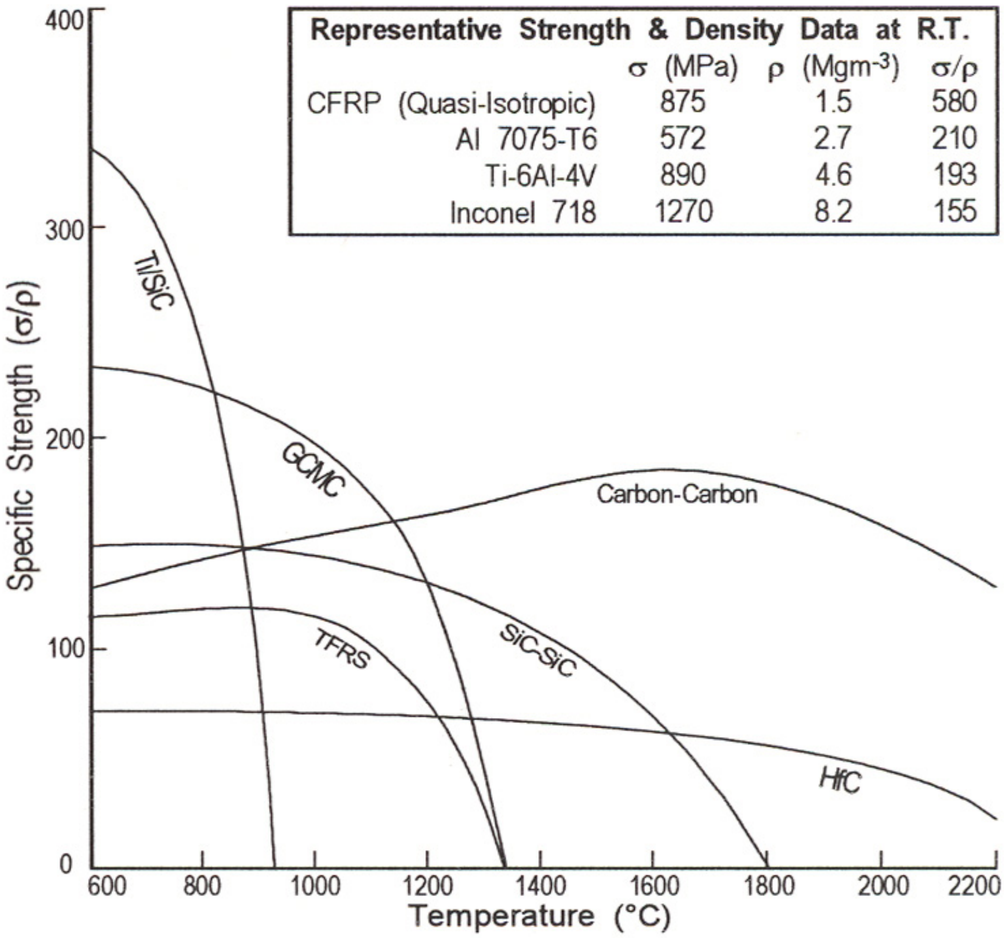


Figure 42.2-2 - Specific strength against temperature for advanced materials

42.3 Features of alloys and metal matrix composites

42.3.1 Structural alloys

The principal metal alloys of structural interest are aluminium, titanium and the superalloys, notably nickel-based. Supporting roles are provided by magnesium, copper, beryllium, intermetallics and refractory metals, Ref. [42-1], [42-2].

Alloys generally have a main constituent with additional alloying elements in varying proportions. The alloy composition, its production route and its heat treatment and mechanical working history determine the mechanical properties.

Examples of common aerospace alloys include:

- Aluminium [See also: Chapter 46]:
  - 2XXX Series, e.g. 2024 (Copper principal alloying element)
  - 7XXX Series, e.g. 7050 (Zinc principal alloying element)
- Titanium [See also: Chapter 47]:
  - Intermediate alpha-beta, e.g. Ti-6Al-4V

42.3.2 Properties

These alloys have good mechanical properties in their own right and are well-established, well-characterised materials which fulfil structural needs. However, the aerospace industries are always seeking greater efficiency and longevity in structures from improved materials. This provides an incentive to develop metal alloys beyond precipitation and mechanical hardening.

The material properties in which improvements are sought include, Ref. [42-3], [42-4], [42-5], [42-6], [42-7]:

- Greater strength, notably:
  - ultimate tensile strength (UTS)
  - yield strength (YS)
- Greater stiffness
- Higher temperature capability:
  - creep resistance
- Better fracture toughness
- Better fatigue resistance
- Better corrosion resistance
- Optimised thermal characteristics, e.g.
  - conductivity,
  - heat capacity, and
  - dimensional stability.

The requirements are usually application driven and maybe only a few selected parameters from the above list can be addressed at one time, it is impossible to improve all simultaneously. When attempts are made to improve some parameters, there is a strong possibility that others are sacrificed or reduced.

All alloys are optimised to enhance certain characteristics and a balance or compromise is reached on a range of engineering properties.

42.3.3 Material developments

The means of improving alloys or preparing composites are:

- Fine particle dispersions:
  - particle size: sub-micrometre (<1μm).
  - particle type: e.g. oxides.
  - particle volume fractions: 1% to 9%, typically.
  - usually classified as:
    - dispersion strengthened (DS), or
    - mechanically alloyed (MA).
- Discontinuous or particulate reinforced Composites (MMCs):
  - particle size: Micrometre range (1 to 50μm).

- reinforcement is achieved by the addition of ceramic particles, e.g.:
  - silicon carbide,
  - alumina,
  - boron carbide.
- Particle fractions within MMCs: 10% to 20%, typically.

- Continuous fibre or filament reinforced composites (MMCs):
  - Fibre diameter: 8μm to 200μm.
  - Analogous to fibre reinforced plastics but with a metal matrix providing significant strength and stiffness.
  - Fibre volume fractions within MMCs: 30% to 50%, typically.

42.3.4 Effects of reinforcements

The addition of particles and fibres has a disrupting effect on conventional alloy microstructures and the usual precipitation and grain characteristics of solidifying metal melts. As the volumetric content of reinforcement increases, the disruption becomes more acute and the chance of deleterious effects increases. As reinforcement contents increase, the most obvious property to diminish is overall material ductility in terms of plastic deformation, elongation to failure and reduction in area. This can present uncertainty to designers and engineers who expect or anticipate metals to have high ductility.

Dispersion-strengthened and particle-reinforced metals are treated as isotropic materials. Fibre-reinforced MMCs are anisotropic and are considered as a group of materials in their own right possessing property characteristics different from those of both metal alloys and fibre-reinforced plastics.

Low strains to failure (<1%) and lack of ductility are features of CFRP, MMC, intermetallic and ceramic materials. As the number of applications of these materials increases, design experience is being accumulated with low strain to failure materials.

42.3.5 Processing

In preparing metal matrix composites, a wide range of reinforcing materials has been tried with an equally large number of matrices. Only a small proportion of these have reached commercial maturity. The two main areas requiring attention are:

- Cost-effective production techniques for combining molten matrix with the reinforcement to give a controllable and reproducible composite product.
- Optimum compatibility between matrix and reinforcement to ensure that both function in unison to give a composite with enhanced properties.

The combining of molten metal with a large volume fraction of reinforcement is not without difficulties. The interface between the two phases has considerable importance and a balance is reached on interfacial bonding.

- Very good bonding usually results from a direct chemical reaction producing:
  - fibre degradation,
  - brittle fracture characteristics, and

— modest composite strength.

- Very poor bonding leads to:
  — low interfacial shear strengths,
  — lack of utilisation of reinforcement properties resulting in poor cohesion between fibre and matrix.

42.4 Features of ceramic and inorganic composites

42.4.1 Features

42.4.1.1 High temperature

For some space applications there is a need for materials which can provide a load bearing and structural function at high temperatures, i.e. 800°C to >2000°C.

42.4.1.2 Materials

The materials are principally inorganic (non-metallic) and ceramic compositions. In view of the structural implications, materials with benign and progressive fracture characteristics are preferred to monolithic, brittle materials, [See: Chapter 43 for conventional monolithic ceramics].

The concentration is on fibre-reinforced materials with a matrix of either:

- Glass,
- Glass-ceramic, or
- Ceramic.

Carbon-carbon composites also offer some unique capabilities.

42.4.1.3 Fracture

Composites achieve their benign fracture characteristics by ensuring that the matrix is de-coupled from the fibres. This provides a large interface area which acts as a crack stopping medium and allows fibre slippage and pull-out. In many respects the composites can be viewed as rigidised fibre (insulation) materials where sufficient integrity is provided for the composite to be classified as an engineering material.

The microstructures of these composites are complex and the matrix at any time contains a significant population of microcracks and microvoids. An optimised composite gives an element of "pseudo-ductility" and fails with noticeable fibre pull-out.

A monolithic ceramic has a nominal fracture toughness of approximately $4MPam^{-1/2}$, a "good" composite is usually in excess of 15 $MPam^{-1/2}$ and preferably 30 $MPam^{-1/2}$.

The term 'good composite' indicates a material which shows tolerance to thermal shock and thermal cycling and can sustain microcracking and minor impact damage without catastrophic failure.

42.4.2 Properties

Mechanical strengths are modest (100MPa to 400MPa), but these are retained at high temperatures. Ultimate failure strains can be in the range of 0.4% to 1.0%, compared with 0.2% or less for monolithic ceramics. Carbon fibre-reinforced carbon possesses characteristics which are suited to high-temperature applications. Its excellent thermal stability is retained at temperatures approaching 2000°C and is unique in that strength increases as the temperature rises. This is found in both the pyrolysed matrix and the fibres.

42.4.3 High-temperature use

42.4.3.1 Oxidation

Carbon-carbon composite would be ideal for most applications were it not for its poor oxidation resistance at temperatures exceeding 500°C. For prolonged use in oxidising environments, elaborate coatings and protection systems are used to give high thermal stability and high-temperature synthesis, with process temperatures between 1500°C and 2000°C, is necessary.

Other ceramic matrix composites offer high-temperature stability without the oxidation problems or the very high synthesis temperatures. In this there are varying degrees of success, influenced by the following facts:

- All ceramic oxidation-resistant fibres, such as silicon carbide or alumina, show increasing thermal degradation and performance loss at temperatures beyond 1200°C.
- The most successful de-coupling interface material between fibre and matrix is carbon which performs the mechanical requirements but is prone to oxidisation.
- Progressively reducing the process temperatures, e.g. from ceramic (silicon nitride, 1500°C) to glass-ceramic (LAS, 1100°C) to glass (borosilicate, 800°C) equally reduces the maximum use temperature.
- Avoiding mechanical damage of the fibres during processing favours low-pressure, reactive-consolidation techniques, but these tend to leave residual porosity (approximately 10%).
- High pressure consolidation eliminates porosity but poses severe limitations on component size and complexity.

42.4.3.2 Prolonged exposure

Above 1000°C, the composition and microstructure of ceramic composites can undergo chemical and diffusion reactions. Time and temperature are the determining parameters for deleterious composition changes to reach significant proportions and degrade the material beyond an acceptable point.

42.5 High temperature and interface phenomena

42.5.1 Effects of processing temperature

42.5.1.1 Metal and ceramic composites

In fibre-reinforced plastics, the fibres are stable and inert at the processing and operating temperatures. It is only the polymer matrix that chemically responds to its environment; it cannot chemically react with the fibres.

For metals and ceramics the processing and operating temperatures are far higher, greatly promoting chemical reactions. Molten metals are aggressive and the large surface area of fibre is susceptible to damage. Therefore processing with molten metals needs to be accomplished as quickly as possible. Ceramic processing also involves chemical reactions and diffusion mechanisms for matrix consolidation, e.g.:

- Sintering,
- Polymer pyrolysis, or
- Chemical vapour deposition, (CVD).

All of these methods rely on controlling the desired reactions and limiting the deleterious ones. Some examples where deleterious reactions have needed controlling are:

- Carbon fibre reinforced aluminium alloys: If molten aluminium remains in contact with carbon for too long, the brittle carbide, Al_3C_4, forms. This occurs on the fibre surface, severely degrades the fibre, and causes a brittle interface. Carbon and aluminium also have an electrochemical potential difference which can cause internal galvanic corrosion under hot-wet conditions.
- Silicon carbide Fibre Reinforced Aluminium Alloys: Very severe fibre degradation occurs if molten aluminium is kept in contact for too long, i.e. more than a few minutes. The SiC is converted to aluminium carbide. A more compatible choice for aluminium is Alumina (Al_2O_3).
- Silicon carbide fibre or filament reinforced titanium alloys: Placing silicon carbide into molten titanium leads to titanium carbide formation producing fibre degradation and a brittle interface. Only large diameter filaments can survive this, and then only if the fibres are coated with titanium diboride (TiB_2) or fibres with a carbon-rich surface.
- Tungsten fibre reinforced nickel superalloys: Tungsten is soluble in molten nickel giving brittle interfaces. A coating of thorium oxide (ThO_2) acts as a diffusion barrier to control the degradation.

42.5.1.2 Intermetallics

As confidence grows in using brittle materials, attention has been given to some which have traditionally been classified as undesirable when unintentionally present in conventional alloys. These include the intermetallics, notably titanium aluminide (Ti_3Al) and nickel aluminide (Ni_3Al). In their pure form, they are not usable owing to brittleness.

By 'doping' and 'micro-alloying', great improvements can be made to:

- Ambient ductility,
- High temperature strength retention,

- Reduction of oxidation embrittlement, and
- Improvement of creep resistance.

This effectively creates a new group of materials and can be taken a step further by using intermetallics as the matrices for composites.

The reinforcement of Ti_3Al with niobium (Nb) coated silicon carbide fibres has been studied. The niobium is selected as the interface to prevent a chemical reaction between the matrix and fibre. Equally it is a desirable microalloying element with Ti3Al for improving ductility, so its diffusion into the matrix should not be a hindrance.

42.5.2 High-temperature applications

42.5.2.1 Hydrogen-containing environments

In high-temperature applications, notably propulsion and thermal protection systems, the presence of hydrogen-oxygen fuel in the former and oxygen in the latter pose problems. The small size of hydrogen ions, atoms and molecules makes diffusion relatively easy within materials, notably metals, manifesting itself as unacceptable material embrittlement. This causes concern for the integrity and longevity of fuel tanks, pipelines, pumps and motors.

42.5.2.2 Oxygen-containing environments

The presence of oxygen at high temperatures always causes concern because there is a natural inclination for all metals to form oxides. In some cases, this is a desirable mechanism for passivating the surface of materials to render further oxidation impossible. Classic examples include:

- $Al \rightarrow Al_2O_3$,
- $SiC \rightarrow SiO_2$
- $AlN \rightarrow Al_2O_3$

For exposed carbon-carbon composites, oxidation is progressive and ultimately catastrophic.

Given that materials oxidise, it is usually the case that oxidation should be controlled or inhibited. This is usually done by:

- Careful material selection,
- Avoiding material constructions which, under thermal cycling, produce:
  - spalling,
  - porosity,
  - cracking, or
  - delamination.
- use of protective coatings, if necessary.

42.5.2.3 Temperature variations

At high temperatures materials are rarely used under isothermal conditions, i.e. there are always some heating and cooling cycles. Three phenomena contribute in determining the life expectancy of materials under non-isothermal conditions:

- Thermal fatigue or thermal cycling: Cyclic events have a cumulative effect. Material expansion is similar to mechanical loading (fatigue) causing initiation and propagation of damage.
- Thermal shock: Heating rate and localised temperature rises affect materials. Uneven heating gives rise to internal stresses due to differences in expansion.
- Coefficient of thermal expansion and material matching: Within a composite material, different phases can have differing expansion coefficients. Different materials within a joint or a coating and substrate can have different expansion coefficients. If these are heated over a wide temperature range, the internal stresses can become very high. Stress relief usually occurs through microcracking, spalling or even total fracture. Therefore, close CTE matching is beneficial if a large temperature range is expected.

42.6 Refractory and ceramic fibres

42.6.1 Introduction

42.6.1.1 General

Refractory and ceramic fibres can be used as:

- Reinforcements in composites, e.g.:
  - Carbon (rayon, pitch or pan), [See: Chapter 52; Chapter 54]
  - Silicon carbide
  - Alumina
  - Boron
- Thermal insulation, [See: Chapter 99].

Numerous types of fibres have been considered for reinforcing various metal and ceramic matrix phases. Those types which have been studied, primarily during the development of composites for high-temperature applications, are summarised. The majority are continuous fibres, although some whisker reinforcements have also been considered.

Not all the types of fibres described are commercially-available or have been successful in composite manufacture.

42.6.2 Types of refractory and ceramic fibre

Table 42.6.1 identifies refractory and ceramic fibres which can sustain temperatures of 1000°C or more and indicates their use in composites.

42.6.3 Use in composites

The majority of successful composite development has centred on:

- High strength carbon fibres
- Nicalon SiC fibres

All commercially-available reinforcing fibres, except carbon, tend to degrade progressively in terms of strength at temperatures beyond 1200°C to 1300°C. Properties are then a matter of assessing residual strengths and whether these are sufficient to be useful.

Table 42.6-1 - Reinforcing fibres available for use in inorganic composites

Fibre	Status	Comments
Carbon		
HS T300	†	Excellent mechanical properties [See: Chapter 2], with stability to 2000°C+, but will oxidise.
Silicon Carbide		
Nicalon Tyranno	† †	Based on fibre pyrolysis. Good handleability & tensile strength. Significant property loss above 1200 to 1300°C.
SCS filaments		Good thermal stability, but avoided as difficult to process.
Derivatives: MPDZ HPZ MPS	‡	Less thermally stable than Nicalon/Tyranno. Good handleability. Applicable to a limited number of matrix materials, mainly glass-ceramics.
Fiberamic		Under development, not yet available.
Alumina		
Fiber FP Safimax Saffil ALMAX	†	Alumina fibres with good thermal stability, but brittle with modest strength. Useful properties to 1500°C.
Sumitoma DENKA Nextel 312 Nextel 440 Nextel 480 PRD-166	 ‡ ‡ ‡ ‡	Modified alumina fibres with better tensile strength than high alumina, but with reduced thermal stability ~ 1400°C.
Zicar ZYF-100		Little known
Boron		
Boron Borsic		Good thermal stability, but avoided as difficult to process.
Key: † Widely used ‡ Gaining use in ceramic and glass-ceramic matrix composites		

42.6.4 Thermal stability

42.6.4.1 General

Thermal stability is often gained at the expense of tensile strength, as in the case of alumina (Al_2O_3) compared with silicon carbide (SiC). Table 42.6.2 gives details of the commercial fibres and some under development.

Table 42.6-2 - Continuous fibre and filament reinforcements used in MMCs and CMCs

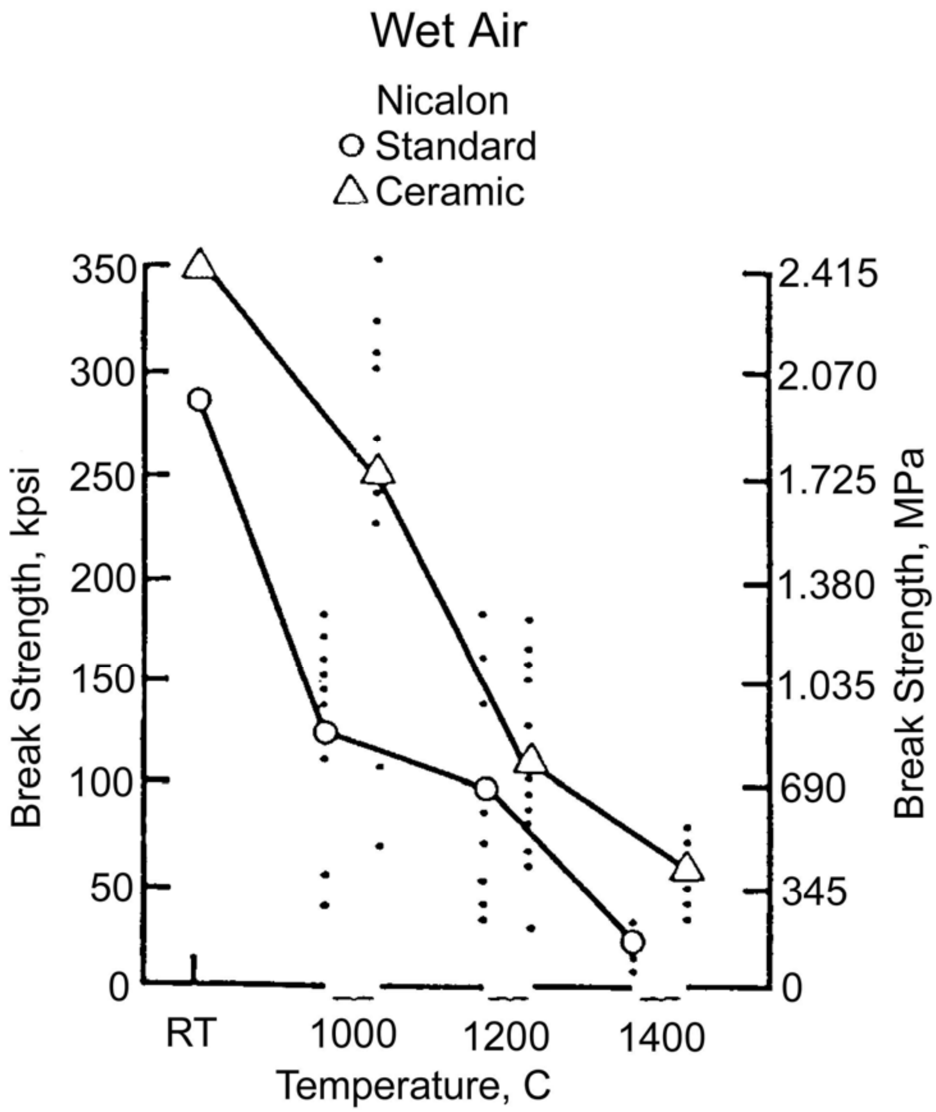
Specific Grade [Manufacturer]	Chemical Composition (%)	Density (kgm^{-3})	Fibre or Filament Diameter (μm)	Tensile Modulus at RT (GPa)	Tensile Strength at RT (MPa)	Tensile Strain to Failure %	Product Forms and Availability	Comments
CARBON								
High Strength grade. e.g. Toray T300	> 99C	1760	7	235	3500	1.5	Yarn and fabrics widely available. Can be braided. Net-shape preforms, (multi-directional).	Carbon fibres possess excellent thermal stability but will oxidise at low temperatures. They are used with carbon and silicon carbide matrices for high temperature applications.
SILICON CARBIDE								
Nicalon (Nippon Carbon)	54.3Si, 30C, 11.8O (β-SiC)	2550	15	196	2740	1.4	3 grades of yarn available. Limited standard fabrics.	Used in MMCs and CMCs. Stable to 1200°C, but will gradually lose its properties as temperatures > 1500°C to 1600°C. Grade NL200 specific to CMCs
SCS Filaments (AVCO, USA)	β-SiC deposited by CVD on carbon fibre substrate	3300	140	427	3450 +	~0.81	Continuous filament. Cannot be woven.	CVD fibre has good thermal stability. Used in MMCs but not CMCs. It Is difficult to process and tends to be avoided.
Tyranno (UBE, Japan)	Si-Ti-C-O: 44.2Si, 24.5C, 11.0Ti, 12.3 O (β-SiC + TiC)	2300	8-12	200-220	2800-3000	1.4-1.5	-	SiC fibre contains titanium and oxygen and is relatively new (1987) and untried. Combines well with aluminium alloys but untried for CMCs
MPDZ HPZ MPS (Dow Corning/ Celanese, USA)	47Si, 30C, 15N, 8O 59Sl, 10C, 28N, 3O 69Sl, 30C, 1O	2300 2350 2600 - 2700	10-15 10 10-15	175-210 140-175 175-210	1750-2100 2100-2450 1050-1400	0.8-1.2 1.2-1.7 0.5-0.8	Development product.	Variants of SiC compositions. MPS likely to have the highest thermal stability.
Fiberamic (Rhone-Poulenc, France)	?	?	15	220	1800	1.2	Development fibre Not yet available.	Under development in conjunction with SEP for use in MMCs and CMCs
ALUMINA								
Fiber FP (Du Pont)	>99 α-Al_2O_3	3950	20	380	1380	0.4	Yarn from Du Pont(USA). Special fabrics possible.	Good thermal stability and is almost pure alumina. Very brittle and tends to be avoided.
Alumina (Sumitomo, Japan)	$85Al_2O_3.15SiO_2$	3200	9-17	210-250	1800-2600	0.7-1.2	Little known.	Adding silica improves strength but lowers thermal stability.
Safimax SD	95 δ-Al_2O_3	3300	3	300	2000	0.67	Uncertain	Small diameter fibre, untried in CMCs
Saffil RF/RG (ICI)	96-97Al_2O_3	3300	3	300	2000	0.67	Bulk and matted fibre	Principally an Insulating fibre which can be used in some MMC composites.

Specific Grade [Manufacturer]	Chemical Composition (%)	Density (kgm^{-3})	Fibre or Filament Diameter (μm)	Tensile Modulus at RT (GPa)	Tensile Strength at RT (MPa)	Tensile Strain to Failure %	Product Forms and Availability	Comments
ALMAX [Mitsu mining Co. Japan]	$99.5Al_2O_3$ α-alumina	3900	10	330	1800	0.5	Various product forms available including yarn and fabric.	No information as yet on the use in composites

Specific Grade [Manufacturer]	Chemical Composition (%)	Density (kgm^{-3})	Fibre or Filament Diameter (μm)	Tensile Modulus at RT (GPa)	Tensile Strength at RT (MPa)	Tensile Strain to Failure %	Product Forms and Availability	Comments
ALUMINA - continued								
DENKA (Japan) Continuous Alumina	$80Al_2O_3$, $20SiO_2$	3100	10	170	1700	1.0	Various yarn and fabric forms available.	No information as yet on use in composites.
PRD-166 (Du Pont)	80 α-Al_2O_3 20 stabilised ZrO_2	4200	20	380	2070	0.6	Development product in Du Pont (USA).	New fibre yet to be released. Improved strength on Fiber FP.
ALUMINA-BORIA-SILICA								
Nextel 312 Nextel 440 Nextel 480 (3M)	$62Al_2O_3$,$14B_2O_3$, $24SiO_2$ 70 Al_2O_3, 2 B_2O_3, $28SiO_2$ 70 Al_2O_3, 2 B_2O_3, $28SiO_2$	2700 3050 3100	12 12 12	152 186 220	1720 2000 1900	1.13 1.07 0.86	Origin USA. A range of yarns and fabric available as standard products.	Modified alumina-silica fibres that provide a compromise between strength & thermal stability. Used in MMCs and CMCs. Property loss >1300°C.
ZIRCONIA								
Zircan ZYF-100 (USA)	Stabilised ZrO_2 with HfO_2 and Y_2O_3	5600 - 5900	4-6	?	?	?	?	Little known.
BORON								
Boron Filaments (AVCO)	CVD deposited boron on tungsten wire substrate	2470 - 2580	101-203	400	3500	0.88	Continuous filament. Cannot be woven	Good thermal stability, but brittle and difficult to process.
BORSIC								
Borsic Filaments (USA)	SiC coated boron on tungsten wire	2380 - 2610	101-203	400-414	2760	0.66-0.69	Continuous filament. Cannot be woven	Good thermal stability, but brittle and difficult to process.

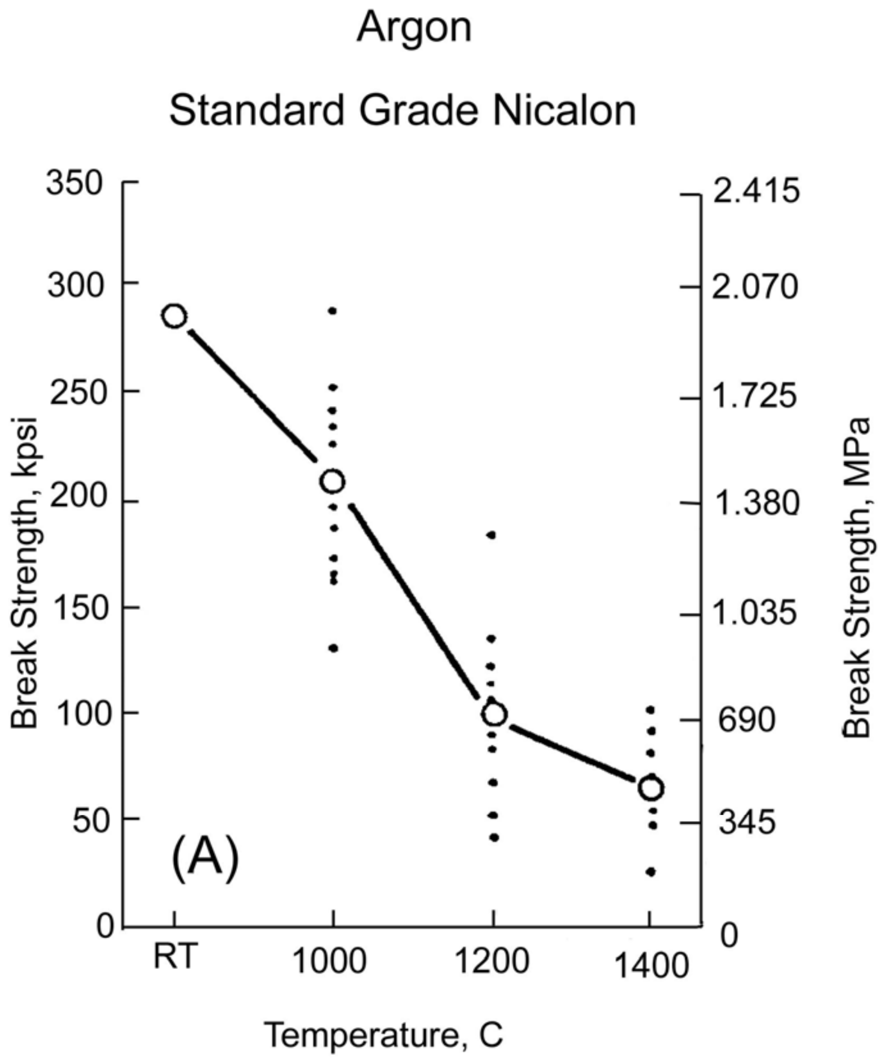
42.6.4.2 Silicon carbide fibres

Figure 42.6.1, Figure 42.6.2 and Figure 42.6.3 show the fibre tensile strength at various temperatures, for two grades of Nicalon fibre, Ref. [42-10].



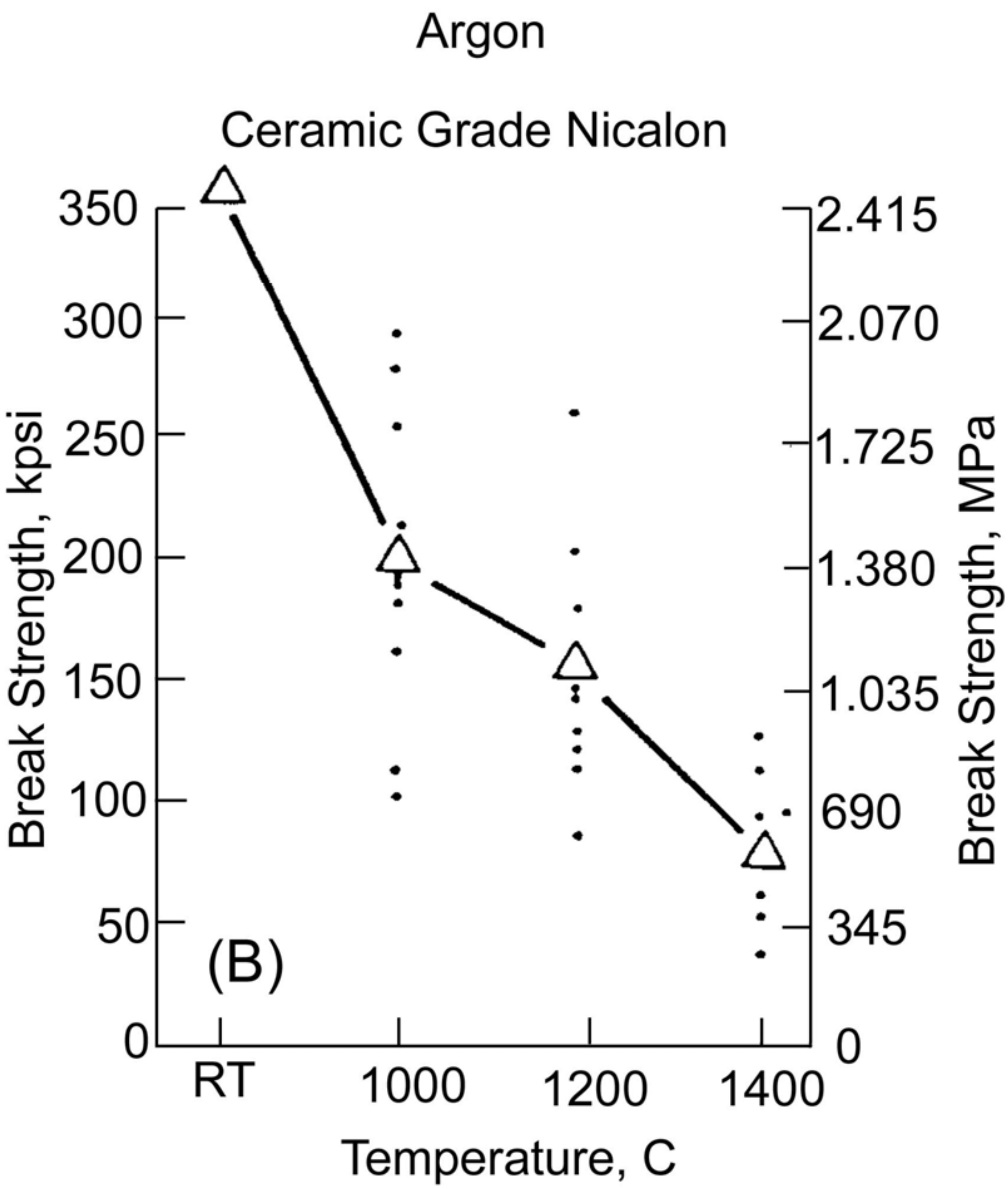
After heat treatments in flowing wet air furnace environment (12 hour soak)

Figure 42.6-1 - SiC Nicalon fibre tensile strength at elevated temperature



After heat treatment in flowing argon furnace environment (12 hour soak)

Figure 42.6-2 - SiC Nicalon standard grade fibre tensile strength at elevated temperature



After 12 hour furnace soak in flowing argon

Figure 42.6-3 - SiC Nicalon ceramic grade fibre: tensile strength at elevated temperature

The tensile strength of Nicalon fibres after prolonged exposure at various temperatures is shown in Figure 42.6.4 and Figure 42.6.5, Ref. [42-11].

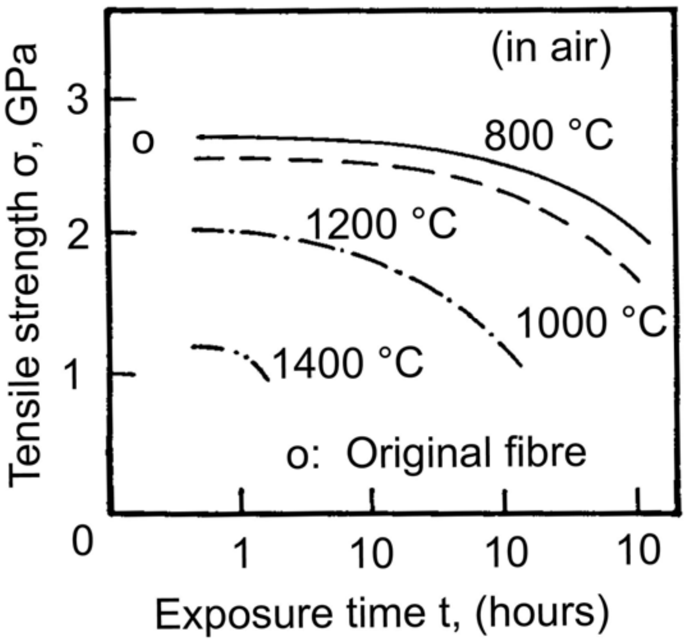


Figure 42.6-4 - SiC Nicalon fibre: tensile strength after high temperature exposure in air

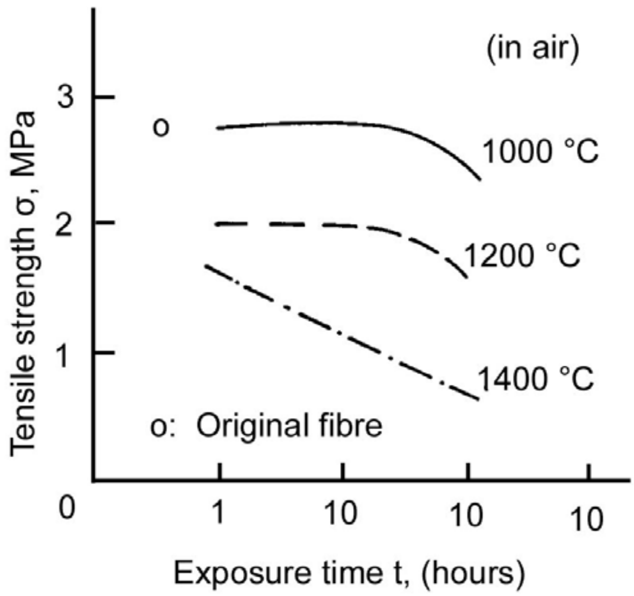


Figure 42.6-5 - SiC Nicalon fibre: tensile strength after high temperature exposure in argon

The high temperature tensile strength and stiffness of Tyranno and Nicalon SiC fibres are compared in Figure 42.6.6 and Figure 42.6.7, Ref. [42-12].

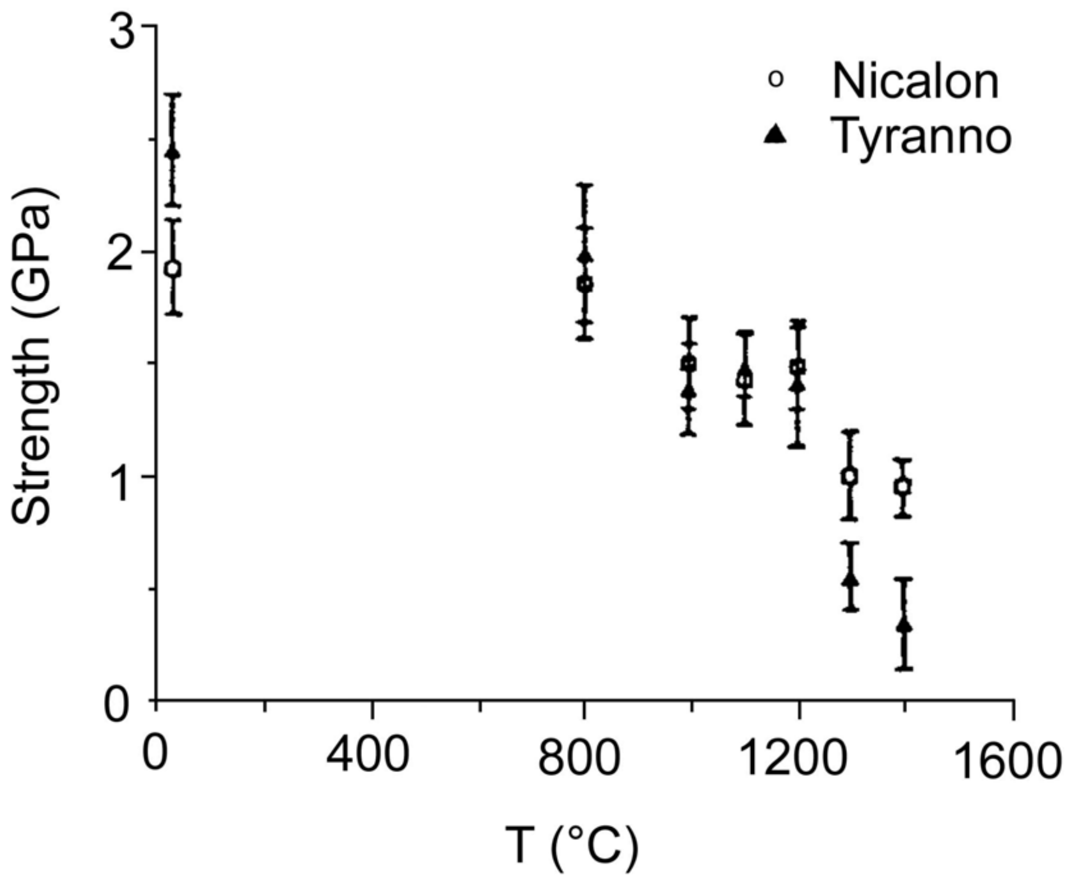


Figure 42.6-6 - SiC fibres Nicalon and Tyranno: tensile strength at elevated temperature

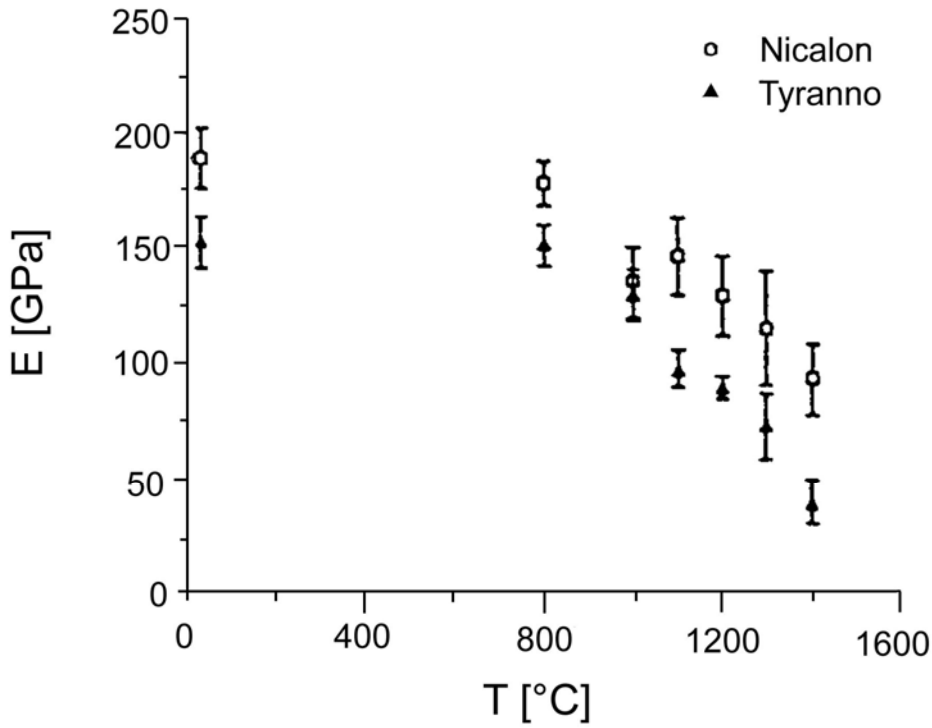


Figure 42.6-7 - SiC fibres Nicalon and Tyranno: modulus at elevated temperature

42.6.4.3 Alumina-based fibres

Figure 42.6.8 and Figure 42.6.9 compare the tensile strength and modulus of three grades of fibre at elevated temperatures, Ref. [42-12].

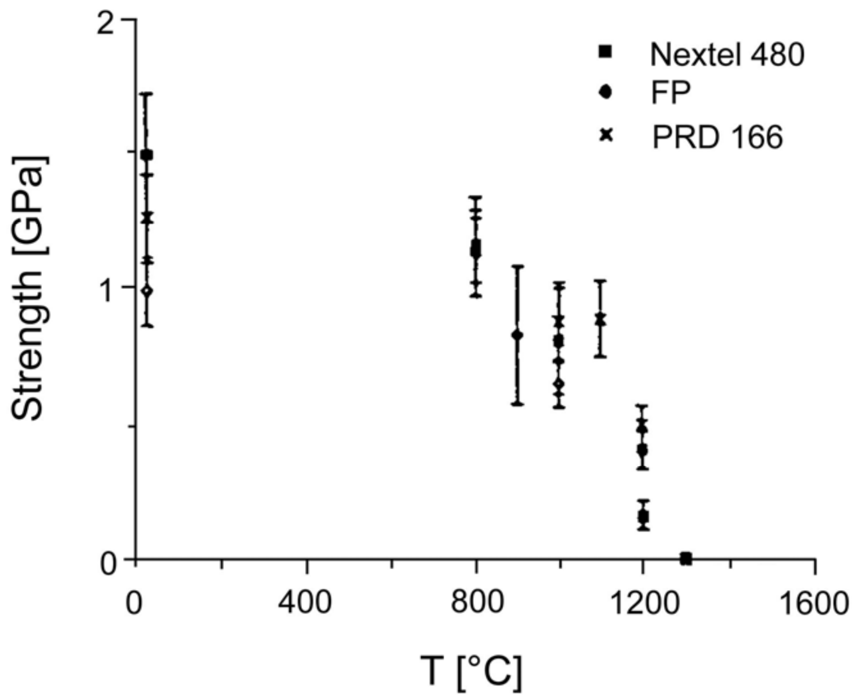


Figure 42.6-8 - Alumina fibres: tensile strength at elevated temperature

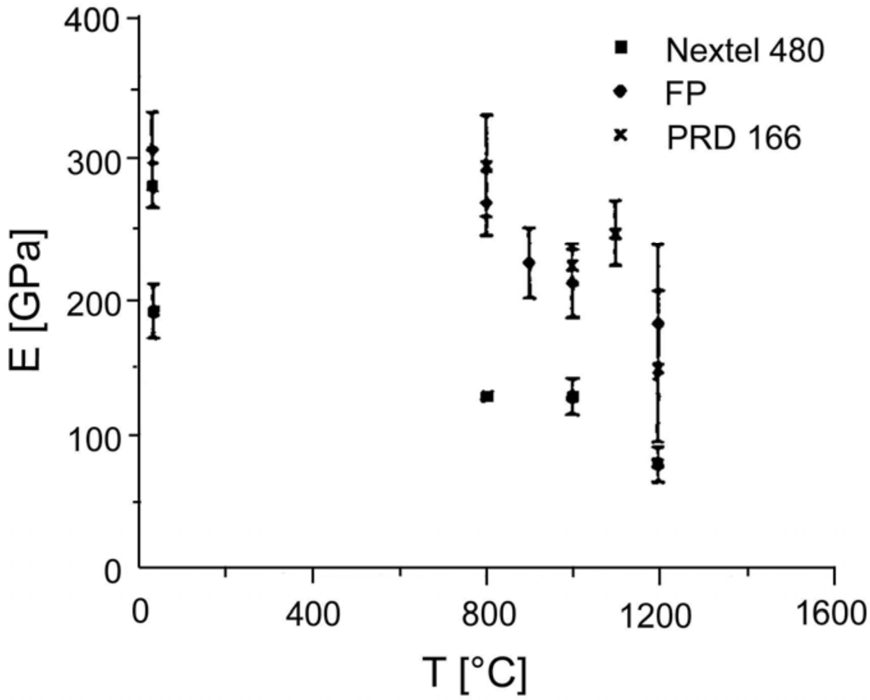


Figure 42.6-9 - Alumina fibres: modulus at elevated temperature

The effect of a high temperature treatment is shown in Figure 42.6.10 for alumina-based fibres PRD-166 and Fiber-FP. The stability of the two fibres after exposure to high temperatures is shown in Figure 42.6.11, Ref. [42-13].

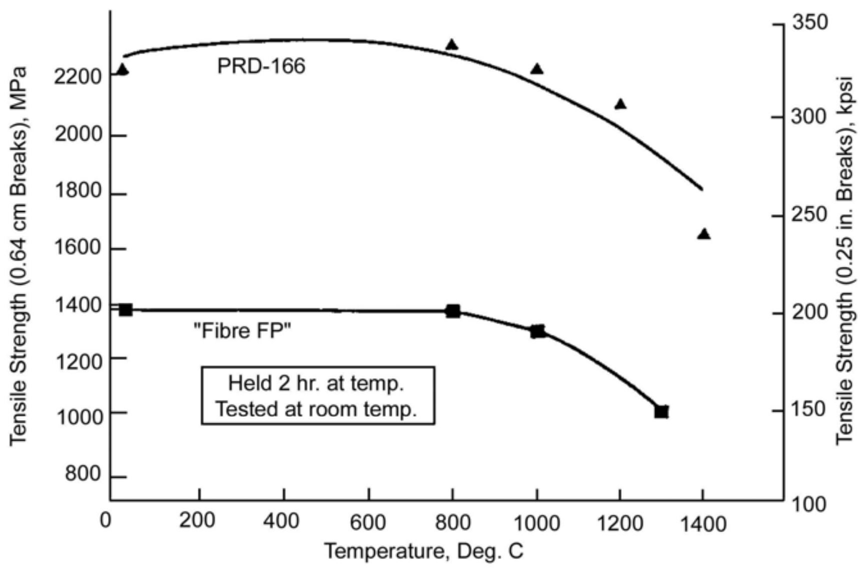


Figure 42.6-10 - Alumina fibres: tensile strength against exposure to elevated temperature

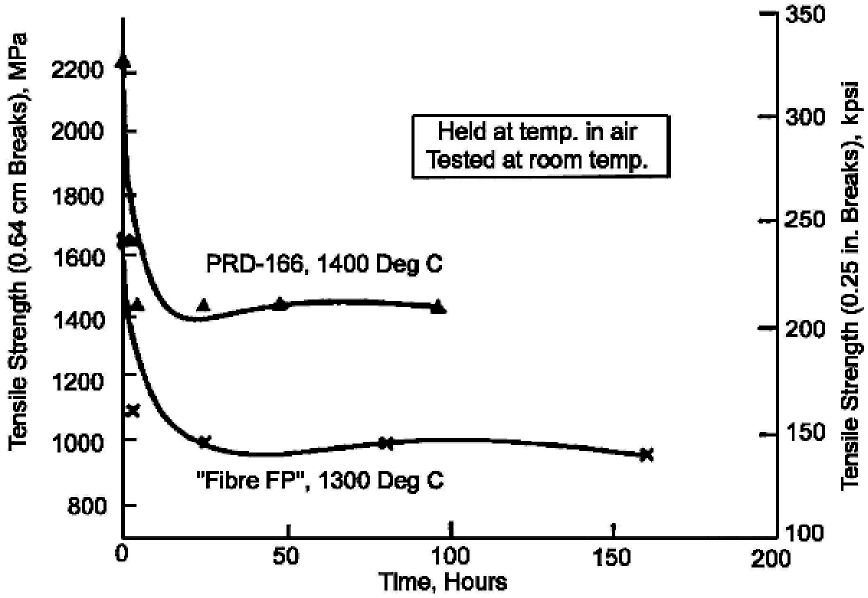


Figure 42.6-11 - Alumina fibres: tensile strength against exposure time

Figure 42.6.12 and Figure 42.6.13 show the high-temperature strength-stiffness stability of Alumina-Boria-Silica Nextel single filaments, Ref. [42-14].

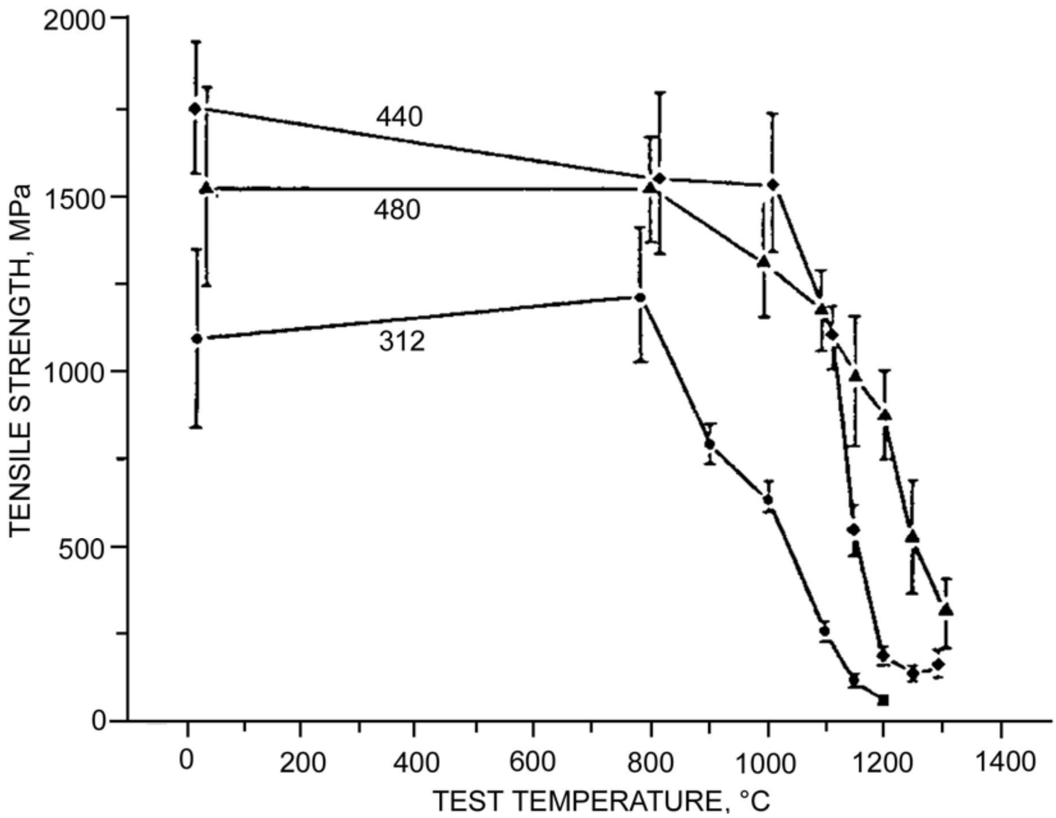


Figure 42.6-12 - Alumina-boria-silica single filament: tensile strength at elevated temperature in air

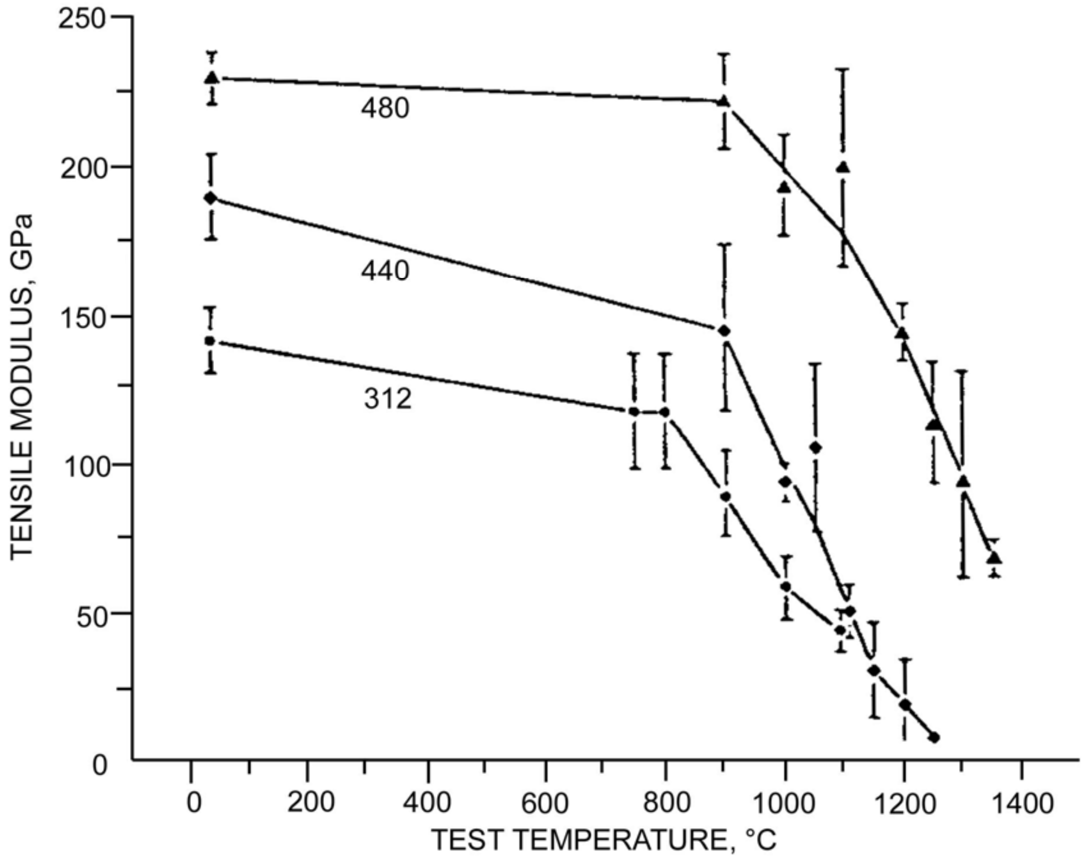


Figure 42.6-13 - Alumina-boria-silica single filament: tensile modulus at elevated temperature in air

42.6.4.4 Whisker reinforcements

Combined high thermal stability and high strength has to an extent been achieved with whiskers, e.g. high purity, stoichiometric, SiC whiskers. The problem is then to keep this good combination in a composite. To date, this has been far from successful because chemical reactions degrade the very small diameter (~1μm) whiskers.

42.6.4.5 Thermal expansion

Thermal expansion is an important characteristic of refractory composites. It should be similar for both fibre and matrix.

Figure 42.6.14 and Figure 42.6.15 give stiffness and CTE values for some fibres and matrices.

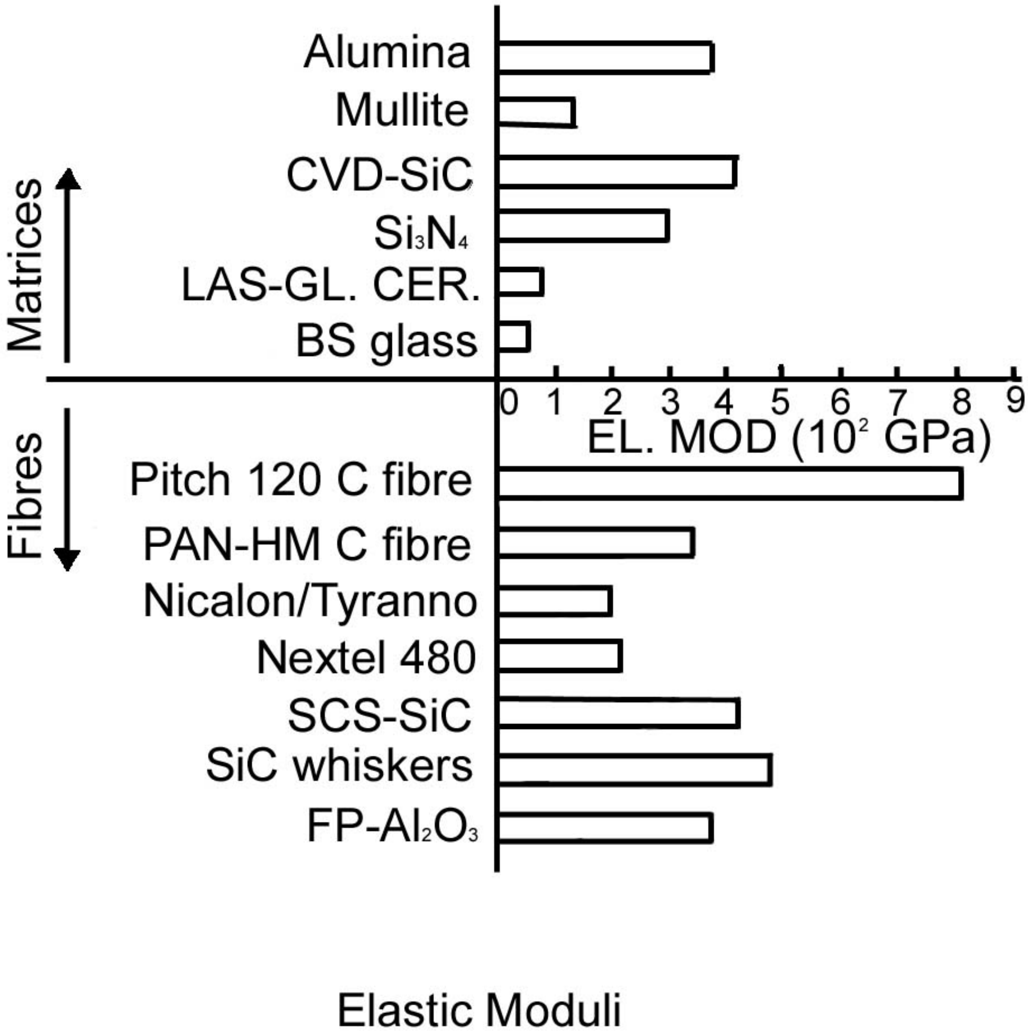


Figure 42.6-14 - Modulus for various fibres and matrix materials

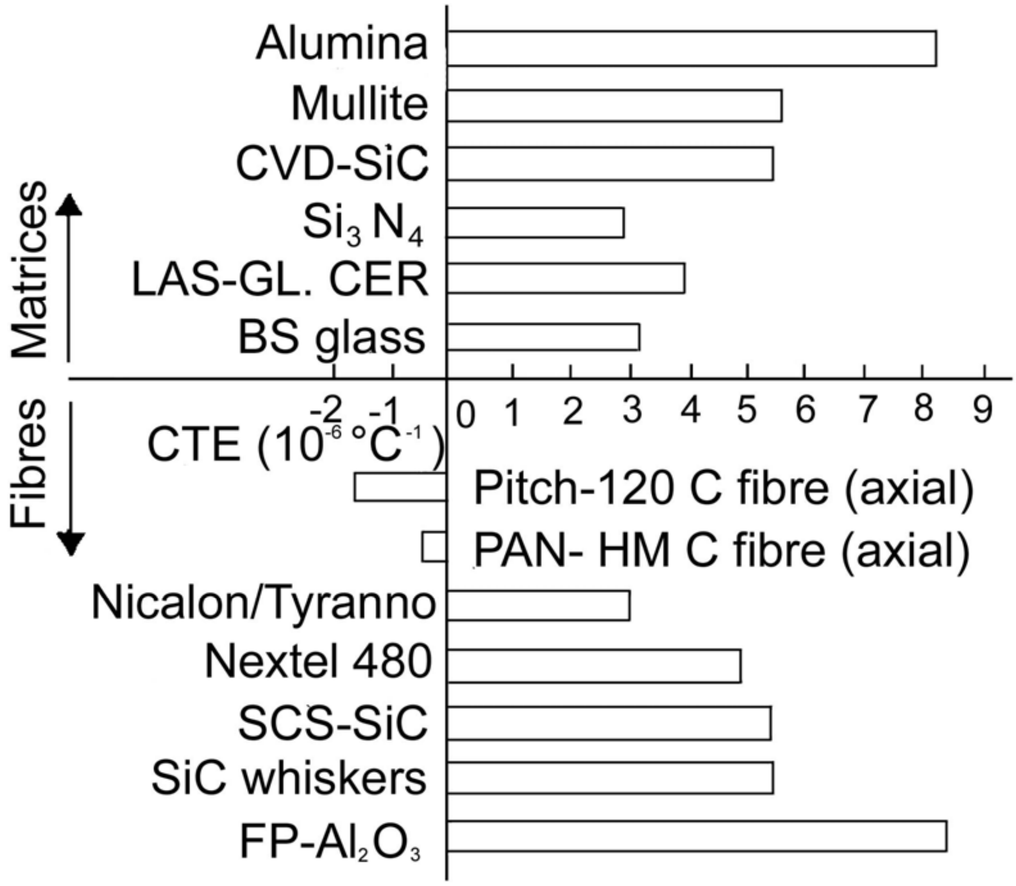


Figure 42.6-15 - Coefficients of thermal expansion for various fibres and matrix materials

42.7 References

42.7.1 General

[42-1] MIL-HDBK-5F: Metallic Materials and Elements for Aerospace Vehicle Structures. November 1990

NOTE Replaced by MMPDS-01, Ref. [42-15]

[42-2] Metal Handbook
ASM International, 10th. Edition, Volume 2
ISBN 0-87170-378-5(V.2), 1990

[42-3] M. Taya & R.J. Arsenault
'Metal Matrix Composites - Thermomechanical Behaviour'
Pergamon Press. ISBN 0-08-036983-9, 1989

[42-4] W.S. Johnson
'Metals Matrix Composites - Testing, Analysis and Failure Modes'
ASTM STP 1032, ISBN 0-8301-1270-X. 1989

[42-5] J. Fuller et al
'Developments in the Science and Technology of Composite Materials'
ECCM 4, September 25-28, 1990, ISBN 1-85166-562-5

[42-6] Symposium on High Temperature Composites
Proceedings of the American Society for Composites. June 1989
Technomatic Publishing Company. ISBN 87762-700-2

[42-7] S.G. Fishman & A.K. Dhingra
'Cast Reinforced Metal Composites'
Conference Proceedings. ASM International, September 1988
ISBN 0-87170-339-4

[42-8] Annual Conferences on Composites and Advanced Ceramic Materials
Published by American Ceramic Society
11th. Annual Conference, January 18-23, 1987. ISBN 0196-6219

[42-9] K.S. Mazdiyasui
'Fibre Reinforced Ceramic Composites - Materials, Processing and Technology'
Noyes Publication, 1990

[42-10] T.J. Clark et al
'Thermal Degradation of Nicalon SiC Fibres'
Proceedings of 9th Annual Conference on Composites and Advanced Ceramics, p576-588, 1985, ACS publication

[42-11] T. Ishikawa et al
'Strength & Structure of SiC Fiber after High Temperature Exposure'
Proceedings of the Symposium on High Temperature Materials Chemistry - IV, the Electrochemical Society, Vol. 88-5, p205-217

[42-12] D.J. Pyscher et al
'Strengths of Ceramic Fibers at Elevated Temperatures'
J.Am.Cer.Soc. Vol. 72, No. 2. p284-288. (1989)

[42-13] J.C. Romine
'New High-Temperature Ceramic Fiber'
Ceram. Eng. Sci. Proc., 8[7-8] p755-765 (1987)

[42-14] A.R. Holtz & M.F. Grether
'High Temperature Properties of Three Nextel Ceramic Fibers'
32nd International SAMPE Symposium, April 6-9, 1987, p245-256

[42-15] R. C. Rice et al.
'Metallic Materials Properties Development and Standardization (MMPDS)'
DOT/FAA/AR-MMPDS-01, January 2003
Website: www.hdbk-5.battelle.org/mil-hdbk2.htm

43
Technical ceramics

43.1 Introduction

43.1.1 General

43.1.1.1 Characteristics

'Ceramic' describes a large group of material compositions having diverse characteristics and properties. Ceramics are available in a variety of forms, e.g. powders for consolidation, fibres and whiskers of various lengths and diameters, pre-consolidated shapes (tubes and plates), and custom-shaped items.

[See also: 42.6 for ceramic reinforcing fibres for composites]

Many types of ceramics are successfully used in industry for a very wide variety of applications. They are typically employed for their electrical and thermal insulation; electrical and thermal conductivity; thermal stability; corrosion- and wear-resistance, or a combination of one or more properties. Some types of ceramics are used for applications that make us of their characteristics, such as bearing, friction and high stiffness. For extreme low-temperature applications, low thermal expansion encouraged evaluation of some ceramics for applications demanding high dimensional stability.

43.1.1.2 Terminology

Within the context of this handbook, technical ceramics are monolithic materials, without deliberate additions of fibre-reinforcement, hence excluding ceramic matrix composites, [See: 52]. Another means of distinguishing between monolithic and composite is that the mechanical properties of technical ceramics are usually stated on a statistical basis, i.e. Weibull.

Over the years, technical ceramics have also been known as:

- 'engineering ceramic' or 'fine ceramic', in order to differentiate between those offering characteristics suitable for engineering applications and other types used for construction products, sanitary ware, tableware, fibrous insulation products and refractories.
- 'advanced technical ceramic', which was adopted after extensive research and development into some new materials largely destined for aero engines.

Although the terms 'fine ceramic' or 'advanced technical ceramic' are still used in some organisations, 'technical ceramic' is now more common, Ref. [43-1], [43-2].

In addition to those types of monolithic technical ceramics used for industrial or engineering applications, the other divisions of ceramics commonly made are:

- Electronic technical ceramics: Although constituting a large proportion of the technical ceramic market, they are not considered as structural materials, e.g. chip carriers and device substrates.
- Coatings: Although many types of ceramics are used as coatings applied to substrate materials, these are not included here. Ceramic coatings are very complex mixtures, which are tailored to provide protection of the substrate against a number of environments, e.g. hot corrosion, chemical attack, erosion. Where ceramic-based coatings have been applied to substrate materials described in this handbook, they are mentioned in that chapter, e.g. thermal barrier coatings on Superalloys.
- Functional ceramics are not included here, i.e. dielectric, piezo- or pyro-electrical grades used for electronic devices such as sensors and igniters. Some functional ceramics are used in sensor systems and actuating devices for 'smart' technologies, [See: Chapter 90].
- Optical devices: Materials used solely for optical devices are not included, e.g. lenses, windows, fibre optics, because their selection is usually made on their optical performance rather than load-carrying ability. Some examples of materials that offer a combination of optical and load-bearing properties are described as 'speciality materials', [See: 43.16; 43.17].

43.1.2 Technical ceramics for space structures

43.1.2.1 Materials

The basic technical ceramic material families of prime interest for space structures are, [See: 43.2]:

- oxides,
- carbides,
- nitrides,
- carbon (elemental carbon)
- glass ceramics, also considered as 'structural glasses', e.g. commercially-available products such as Zerodur® and ULE®.

43.1.2.2 Application examples

Until recently, monolithic ceramic materials were of low interest within the space community for structural applications. In general, ceramics have the reputation of being brittle and intolerant of shock and vibrational loads. This, in part, led to the development of CMCs for reusable space plane technologies.

Low thermal expansion properties coupled with other inherent characteristics (electrical, optical or environmental resistance) have provoked a new interest in the potential use of some technical ceramics in space, particularly to meet the dimensional stability demands of systems operating at very high frequency.

Mass remains a design driver for all spacecraft, leading to interest in lightweight ceramic forms, such as foams, along with innovative mass-optimised designs, [See: 43.18; 43.19]

The wider use of ceramics is encouraged by a growing knowledge-base from applications in other industry sectors and also by the increasing number of internationally-recognised standards available, e.g. for testing.

43.2 Material groups

43.2.1 Composition

All technical ceramics are essentially synthetic materials produced either from modified minerals or by chemical processing. These wide ranges of materials tend to be grouped by their basic chemical composition, i.e.:

- Oxides
- Carbides
- Nitrides
- Elemental, such as carbon.
- Structural Glasses And Glass-Ceramics

Each of the composition groups contains a variety of materials of different formulation. Some are high-purity, single constituent grades whereas others are complex blends or chemical compounds. Within a single family, materials provide a range of mechanical or physical properties for use in a variety of applications.

Commercial ceramic products are described in a number of ways, i.e.:

- Mineral origin, e.g. steatite,
- Chemical formulation, e.g. magnesium silicate,
- Molecular formula, e.g. $MgO\ SiO_2$,
- Manufacturers' product name.

Manufacturers usually offer several grades of the same basic ceramic, often classed by purity or composition.

For completeness, all common types of commercial technical ceramics are listed in tables by their basic chemical composition. Those with 'greyed' backgrounds are of less interest for structural space applications and are not discussed further.

43.2.2 Oxides

Table 43.2.1 introduces some of the general features of common commercially-available oxide technical ceramics. Of these, materials that provide a useful combination of mechanical, electrical and thermal characteristics are of interest for aerospace engineering applications, e.g. grades of alumina, silica and zirconia.

Table 43.2-1 – Technical ceramics: Oxides - general features

Oxide Type	Features	Comments
Alumina Aluminium oxide Al_2O_3 [See: 43.6]	High stiffness. Hard and brittle. Properties retained to about 800°C, but reduce significantly above 1000°C. Chemical resistance to both acid and alkalis (except HF). Excellent electrical insulation. Medium shock and thermal cycling resistance.	Zirconia additions (ZTA) improve toughness and strength. Synthetic sapphire is a single crystal form of alumina; often used for optical applications. Translucent alumina is used for high-temperature, optical applications, e.g. lighting. Alumina fibres are used as reinforcement phase in metals and ceramics. Alumina-titanium carbide is used for cutting tools.
Beryllia Beryllium oxide BeO [See: 43.16]	Good chemical stability. Excellent electrical insulator. High thermal conductivity. IR transparent. Low thermal neutron absorption.	Toxic. Thermal management in electronics-electrical. Microwave applications, e.g. windows and radomes. Nuclear industry.
Cordierite Magnesium aluminosilicate	Moderate strength. Good electrical insulation. Low thermal expansion.	Porous types for catalyst supports. Impervious types for electrical uses.
Magnesia Magnesium oxide MgO	Moderate mechanical properties. Poor thermal shock resistance. High chemical resistance, including molten metals.	High-temperature refractory uses (up to 2400°C). Single crystal forms for optical uses.
Mullite $3Al_2O_3{\cdot}2SiO_2$	Low mechanical properties. Poor toughness.	Refractory material. Toughness can be improved by formulation and processing.
Silica Silicon dioxide SiO_2 [See: 43.11]	Excellent thermal shock resistance. Translucent or transparent depending on process route used.	Fused quartz or vitreous silica for optical uses. Fused silica for thermal shock resistance.
Steatite Magnesium silicate $MgO{\cdot}SiO_2$	Often a modified talc formulation. Abrasives. Dielectric properties.	Grinding-cutting media. Electronic-electrical uses.
Titania Titanium dioxide TiO_2	High dielectric properties. Wear resistance.	Electronic applications. Semiconducting type used as electrodes or to discharge static (process industries).
Zirconia Zirconium dioxide ZrO_2 [See: 43.14]	Blended with other oxides to stabilise microstructural phases: Fully stabilised. Partially stabilised (PSZ), including forms known as TZP 'tetragonal zirconia polycrystal' and TTZ 'transformation toughened zirconia'.	Fully stabilised materials are used as refractories. PSZ for thermal resistance. TZP for high strength and TTZ for engineering uses. At high temperatures PSZ becomes electrically conductive.

Key :	'Grey background': less of interest for space structural applications

43.2.3 Carbides

Table 43.2.2 gives some general features of commercially-available carbide technical ceramics. Whilst boron carbide can be considered for particular applications, silicon carbide has a range of properties of most interest for space applications, [See also: 43.12].

Table 43.2-2 – Technical ceramics: Carbides - general features

Carbide Type	Features	Comments
Boron carbide B_4C [See: 43.8]	Extremely hard. Lightweight. Relatively good fracture toughness.	Wear resistance uses. Impact resistance (armour). Neutron absorption.
Silicon carbide SiC [See: 43.12]	Mechanical properties vary with the processing route used. Thermal stability to very high temperatures (2200°C to 2800°C, depending on type). Hard. Good oxidation and hot-corrosion resistance, depending on purity. Impact resistance (armour). Wear resistance. Moderate thermal conductivity. Moderate electrical conductivity.	Various processing methods used to give a range of materials (from refractories to engineering materials). Used for dimensionally-stable structures in space-based and terrestrial telescopes.

43.2.4 Nitrides

Table 43.2.3 provides some general features of commercially-available nitride technical ceramics. The characteristics of different grades are largely due to their processing methods.

Table 43.2-3 – Technical ceramics: Nitrides - general features

Nitride type	Features	Comments
Aluminium nitride AlN [See: 43.7]	High thermal conductivity. Good electrical insulation. Powders prone to moisture uptake affecting electrical properties.	Thermal and electrical properties depend on purity. Heat management in electronic applications. Windows for visible to infra-red systems.
Boron nitride BN [See: 43.9]	Microstructure determines properties: Hexagonal form similar to graphite. Properties are anisotropic. Oxidises in air above 800°C. Cubic form is extremely hard.	Hexagonal (powders) used as high-temperature solid lubricant. Densified shapes for insulators and high-temperature refractories in inert atmospheres. Cubic form used for abrasives.
Silicon nitride Si_3N_4 [See: 43.13]	Good creep resistance. Good thermal shock resistance. High stiffness. Low thermal expansion. Not electrically conductive (pure). High temperature strength (1500°C in vacuum; disassociates at 1850°C in nitrogen) depends on processing used: RBSN 'reaction bonded silicon nitride' or 'reaction sintered': porous, moderate strength but high thermal shock resistance. Modified processing for fine, even porosity to improve strength. Hot-pressed or hot isostatic pressed materials: very high strengths, good fracture toughness and oxidation resistance (to 1200°C). Creep resistance depends on secondary, glassy phases. Sintered materials: strengths can approach hot pressed variants. Modified compositions can increase toughness at the expense of strength.	Strength properties depend on processing method. RBSN variants are used for refractory-type applications. Non-wetting by molten metals enables uses in metal and process industries. Hot-pressed or isostatic pressed are used for high-temperature engineering components, e.g. gas turbine and engine parts. Sintering enables production of shaped parts not possible by hot-pressing, but can compromise a level of mechanical performance.
Sialon Silicon aluminium oxynitride	High strength. Good fracture resistance. Excellent wear resistance. Good thermal shock resistance.	High-temperature wear applications, e.g. engines (combined mechanical properties and hardness). Also used for cutting tools and welding jigs. Conductive sialons are blended with compounds such as titanium carbide.
Key : 'Grey background': less of interest for space structural applications		

43.2.5 Elemental

43.2.5.1 Carbon

Carbon is used in its elemental forms and can be classed as a technical ceramic.

Carbon materials are erroneously known as 'graphite' in the USA. In the context of this handbook, the term graphite is used only for the soft, hexagonally-crystallised, allotrope of carbon.

Carbon can have a wide range of properties depending on its structure, from the extreme hardness of diamond (natural or synthetic) to glassy amorphous forms and soft graphites, [See: 43.10].

Compared with other technical ceramic groups, carbons have:

- Moderate mechanical properties can be retained to about 3000°C in inert or reducing atmospheres. In oxidising atmospheres and air, the upper limit is governed by the oxidation rate. This in turn is heavily influenced by the microstructure, hence: graphite (anisotropic), 400°C; lower for amorphous or crystalline carbons (isotropic). The upper use temperature is loosely defined as the point at which oxidation rates become exponential.
- Good electrical conductivity,
- Moderate thermal conductivity,
- Good thermal shock resistance.

Table 43.2.4 summarises some features of commercially-available carbon technical ceramics.

The majority of carbon-based structural components within space vehicles are composites, however, some discrete components made from carbon technical ceramics have flown, e.g. components in Space Shuttle and ISS, such as seals and bearings, Ref. [43-3].

Foams destined for structural roles are a recent innovation, e.g for spacecraft thermal management systems, [See: 43.10]

Table 43.2-4 – Technical ceramics: Carbons - general features

Carbon Type	Features	Comments
Graphite [See: 43.10]	Solid: Soft, weak material. Self-lubricating properties. Anisotropic properties. High temperature and corrosion resistance.	Lubricants for high or low temperatures. Anisotropy can be controlled by processing. Refractories and process industries.
	Porous: Exfoliated (graphite foil in USA). Foams.	Flexible 'sheet' for gaskets and seals. Thermal management systems.
Vitreous carbon	Non-porous, amorphous glassy form created from the thermal decomposition of a carbon-rich material, e.g. resins, pitch, tars. Combined strength and corrosion resistant.	Constituent of refractories, used as a binder matrix phase for other materials, e.g. carbon, graphite and SiC; especially 'carbon-graphites' (mechanical carbons). Specialist uses only, e.g. laboratory equipment.
Carbon-Graphites (mechanical carbons) [See: 43.10]	Combination of amorphous carbon and graphite. A proper mixture of the two materials is strong and hard with a low friction coefficient. Excellent corrosion resistance. Can operate at temperatures above 315°C for extended periods of time (depends on grade).	Materials can be created to achieve a balance of properties, i.e. range of mechanical carbon materials for a variety of wear-resistant uses, e.g. bearings, seals, pumps.
Pyrolytic carbon [See: 43.10]	Anisotropic properties. Higher strength compared with conventional polycrystalline graphite.	Produced by deposition onto substrates, often from chemical vapours, e.g. CVD process.
Siliconised carbon †	Compared with carbon alone: Increased hardness. Lower porosity. Improved oxidation resistance.	Silicon carbide surface layer formed by liquid or vapour infiltration processes. Processes adapted and improved for ceramic composite manufacture, [See also: Chapter 89].
Fibres [See: Chapter 2; 42.6]	Most common reinforcing fibre for advanced composite materials	Wide variety of fibre properties and characteristics depending on processing methods and treatments.
Diamond [See: 43.17]	Extremely hard. Abrasion and wear resistant. Developments in synthetic process techniques now enable large, simple shapes to be manufactured rather than just thin films or coatings deposited on substrates.	Abrasives and cutting tool industries. Wear protection (coatings and films). Optical applications. Thermal management.
Key:	† known as 'siliconized graphite' in the USA; 'Grey background': less of interest for space structural applications	

43.2.6 Glass-ceramics

Glass-ceramics, also known as structural glasses, are a group of materials that contain about 70% to 100% crystalline phases within a glassy 'matrix', [See: 43.15].

The main features include:

- High strength.
- Low thermal expansion.
- Good thermal shock resistance.
- Good corrosion resistance.
- Good wear resistance.
- Electrical insulation.
- Good dielectric properties.

Commercially-available compositions are generally tailored to optimise particular properties, e.g. extremely low thermal expansion or machinability, [See: 43.15].

Characteristics of interest for space use are low thermal expansion for dimensionally-stable structures combined with dielectric or electrical insulation, e.g. for antenna structures, [See: 43.15; 43.19].

43.2.7 Sources

There are numerous manufacturers and suppliers of technical ceramics worldwide. Manufacturers usually offer a comprehensive range of material types and a wide range of grades for any particular material. They usually also provide a comprehensive design-development and processing service for their materials. In general, commercial products have evolved over many years as applications moved from refractories to electronics and structural engineering applications. In recent years, some of the larger companies have been active in acquiring or creating licensing or distribution agreements with other organisations to enhance their product ranges, e.g. Boostec and CoorTek for silicon carbide in space applications.

Technical ceramics continue to be used mainly for some combination of their high-temperature, corrosion resistance and electrical characteristics, so available property data corresponds to these prime markets. For example, it is rare to have data on low-temperature performance from manufacturers.

Speciality materials, such as optical grade materials and glasses, tend not to be supplied by mainstream technical ceramic companies.

Table 43.2.5 summarises some examples of sources of commercial materials, including those providing specialist product forms, e.g. foams, [See also: Chapter 26 for foam cores in sandwich panels].

Table 43.2-5 – Technical ceramics: Sources - examples

Source [4]	Types of technical ceramics: Alumina	Aluminium nitride	Boron carbide	Boron nitride	Carbon	Silica	Silicon carbide	Silicon nitride	Zirconia	Glass ceramics	Speciality
Boostec (F)							⊙				
Bettini (I)	⊙						⊙	⊙	⊙		
Carbon Lorraine (F)					⊙						
Carpenter (USA)	⊙						⊙	⊙	⊙		
Ceradyne	⊙	⊙	⊙			⊙	⊙	⊙			
CeramTech	⊙						⊙	⊙	⊙		
Cercom		⊙	⊙				⊙	⊙			⊙
CoorsTek	⊙						⊙		⊙		
Corning Corp.										⊙	
Dynamic Ceramic (UK)	⊙						⊙	⊙		⊙	
ECM (D)							⊙				
Element Six [3] (UK, NL)											⊙
ERG Aerospace (USA)							⊙				
Fraunhofer IKTS (D)							⊙				⊙
GE Advanced Ceramics				⊙	⊙						
Graphite Metallizing (USA)					⊙						
HITCO (USA)					⊙						
Kubota								⊙			
Kyocera	⊙	⊙					⊙	⊙	⊙		⊙
MER Corp. (USA)					⊙						
Morgan Group [2] (UK)	⊙	⊙		⊙	⊙	⊙	⊙	⊙	⊙	⊙	⊙
NGK							⊙				
POCO Graphite Inc. (USA)					⊙		⊙				
Precision Ceramics	⊙	⊙		⊙			⊙	⊙	⊙	⊙	
Safran Group - Snecma (F)					⊙		⊙				
Schott (D)										⊙	
Schunk (D)					⊙						
SCT (F)	⊙								⊙		
SGL (D)					⊙						
St. Gobain [1] (F)	⊙					⊙	⊙	⊙	⊙		
Toshiba Ceramics							⊙				
TRL - Touchstone Research Lab. (USA)					⊙						
Ultramet (USA)					⊙		⊙				

Key: (1) St.Gobain includes former products of Norton, AnnaWerk, Cesiwid, Carborundum; (2) Morgan group includes Morgan Advanced Ceramics, Morgan Advanced Materials & Technology, Haldenwanger, Morgan Performance Materials; (3) Formerly DeBeers; (4) Sources are indicative only and not an exhaustive list.

43.3 Design aspects

43.3.1 Guidelines

43.3.1.1 General

Compared with conventional engineering materials, ceramics are brittle and this inherent feature should be taken into account in the design approach used, [See also: 43.15 – design with glass ceramics].

Historically, selection of technical ceramics for engineering applications was based on thermal or electrical performance needs first and then the ability to resist any mechanical loads arising. In demanding high-temperature environments, this meant that technical ceramics were only applied beyond the capabilities of conventional materials. For electrical insulation applications, use of metals is impossible, so ceramics were used where plastics (when invented) lacked adequate performance.

Over recent years, a growing impetus for the use of technical ceramics in mainstream applications has helped increase their knowledge base, e.g. standards for property evaluation, more reliable property data, improved processing and availability of design tools. These can now be applied to the more demanding, high-performance industry sectors, such as aerospace.

43.3.1.2 Material and process selection

Their inherent brittle fracture behaviour, and the consequences of this failure mode, make the space industry wary of ceramics and glasses. Therefore, all aspects of selection, design and verification are subject to review and approval by the final customer, [See: ECSS-Q-70-71].

Other than applications in equipment and electronics, proposed structural uses within European projects mainly exploited the extreme high-temperature performance of ceramic-based materials for passive TPS thermal protection systems and thermo-structural designs in reusable vehicles, [See: Chapter 70; Chapter 71].

The emphasis on ceramics has now changed to dimensionally stable structures, exploiting the low CTE and electrical characteristics. Although confidence in extreme high temperature performance is not an issue for such intended applications, lessons learnt in obtaining reliable property data and analysis techniques remain appropriate. Likewise, experience from other industry sectors can also be applicable.

43.3.1.3 Component design

The design and manufacture of technical ceramic components is similar to that of composites in that they are difficult to machine to final form. Therefore, technical ceramic near-net shape components tend to be designed, manufactured and supplied by companies with the expertise. Although technical ceramics companies do provide some stock shapes, the majority of their business is providing custom components for particular applications. Some general examples include: seals, feed-throughs, wear components in process industries, cutting tools, heater elements, rollers, kiln furniture, heatsinks and refractories such as crucibles and boats. Therefore manufacturers and suppliers of technical ceramics tend to work together with the engineers considering such materials for high-performance applications.

Good design of ceramic components, as with any other material, aims to exploit the beneficial characteristics whilst avoiding the bad, Ref. [43-30].

The various factors to consider during component design can be summarised as:

- Mechanical loads, [See also: 43.15 – design of glass ceramics]:
  - Use compressive loading and avoid tensile or shear loads.
  - Some materials are anisotropic, whereas others are not.
  - Point loading is detrimental, so distribute loads as evenly as possible.
- Physical:
  - Some materials are thermally conductive, whereas other are not.
  - Thermal expansion is generally much less for ceramics than other materials. In mixed material combinations thermal expansion mismatch needs consideration.
  - Some ceramics are electrically conductive, others are not (insulators).
  - Some materials become electrically conductive under certain conditions, e.g. high temperature (zirconia, silicon carbide).
- Shape:
  - Poor surface finish and features such as notches, sharp corners, slots and cut-outs acts as stress raisers and need to be avoided by using generous radii.
  - Large or sudden changes in section can promote high thermal stresses and temperature gradients.
  - Joints between ceramics and other materials need careful consideration, e.g. limiting thermal expansion mismatch or point loads.
  - Hole sizes, shapes and position need consideration.
  - Near-net shapes help avoid extensive post-manufacture machining operations that are difficult or expensive and can damage hard, brittle materials.
- Processing:
  - The maximum size of high-density, high-performance materials is generally limited by the production process used, e.g. volume constraints of hot-isostatic press chambers.
  - Processes need optimisation to avoid repeated high-temperature firing which can induce damage by thermal gradients or mismatch.

43.3.2 Property and performance evaluation

43.3.2.1 Test and analysis

An extensive development exercise has resulted in methods of determining the properties of technical ceramics for engineering applications, [See: Test methods].

Development of models that predict the performance of ceramics (both monolithic and composite) under various conditions aids design and analysis. An example is the NASA CARES software suite, which has been applied to ceramics destined for space applications, Ref. [43-4].

Originally access to the NASA suite of software programs was restricted to organisations within the USA. Providing that certain criteria are met, applications from European-based organisations are now welcomed, Ref. [43-49].

43.3.2.2 NASA CARES software

Developed by NASA, CARES 'ceramics analysis and reliability evaluation of structures' is a suite of integrated design software tools that provide a means to optimise the design of brittle material components using probabilistic analysis techniques. It incorporates fundamental mechanics theory and associated computational approaches for component design using isotropic brittle materials. CARES can be generally applied to designs involving the use of brittle materials (monolithic structural ceramics, glasses, intermetallics, and ceramic matrix composites) in a wide range of industry sectors and can be used in conjunction with commercially-available FEA packages, e.g. ANSYS® and ABAQUS®.

The suite of programs includes, Ref. [43-4]:

- CARES-Life, which was developed to predict the reliability and life of structures made from advanced ceramics and other brittle materials, e.g. glass, carbons and intermetallics.
- CARES-Creep, which is an integrated design program for predicting the lifetime of structural ceramic components subjected to multiaxial creep loads. It takes into account the time varying creep stress distribution (stress relaxation).
- Composite CARES (C/CARES), which was developed to address aerospace design issues relating to CMC ceramic matrix composites. The aim is to predict the time-independent reliability of a laminated structural component subjected to multiaxial load conditions.

CARES can predict the durability and lifetime of brittle materials used for structural applications in harsh environments, e.g. turbine parts, TPS, radomes, fuel cells and MEMS microelectromechanical systems for power generation and propulsion, Ref. [43-50], [43-51].

Further developments include enhancement of computational design methodologies for brittle structures, Ref. [43-4].

43.3.2.3 Test methods

Traditionally, test methods for technical ceramics were adapted from those used in the refractory, electrical or optical industries, i.e. the main consumers. International standards now cover aspects of high-performance technical ceramics for engineering applications. These are summarised in Table 43.3.1 with 'draft' standards listed in Table 43.3.2, Ref. [43-5], [43-6].

Table 43.3-1 – Technical ceramics: ISO standards - summary

Number	Date (1)	Title
ISO - Fine ceramics (advanced ceramics, advanced technical ceramics) (2)		
ISO 14703	Mar-2000	Sample preparation for the determination of particle size distribution of ceramic powders
ISO 14704	Aug-2000	Test method for flexural strength of monolithic ceramics at room temperature
	Jan-2004	ISO 14704 - Technical Corrigendum 1.
ISO 14705	Mar-2000	Test method for hardness of monolithic ceramics at room temperature.
ISO 15165	Oct-2001	Classification system.
ISO 15490	Jul-2000	Test method for tensile strength of monolithic ceramics at room temperature.
ISO 15732	Oct-2003	Test method for fracture toughness of monolithic ceramics at room temperature by single edge precracked beam (SEPB) method.
ISO 15733	Feb-2001	Test method for tensile stress-strain behaviour of continuous, fibre-reinforced composites at room temperature.
ISO 17561	Mar-2002	Test method for elastic moduli of monolithic ceramics at room temperature by sonic resonance.
ISO 17562	Oct-2001	Test method for linear thermal expansion of monolithic ceramics by push-rod technique.
ISO 17565	Dec-2003	Method for flexural strength of monolithic ceramics at elevated temperature.
ISO 18753	Aug-2004	Determination of absolute density of ceramic powders by pyknometer.
ISO 18754	Aug-2003	Determination of density and apparent porosity.
ISO 18755	Mar-2005	Determination of thermal diffusivity of monolithic ceramics by laser flash method.
ISO 18756	Dec-2003	Determination of fracture toughness of monolithic ceramics at room temperature by the surface crack in flexure (SCF) method.
ISO 18757	Dec-2003	Determination of specific surface area of ceramic powders by gas adsorption using the BET method.
ISO 20501	Dec-2003	Weibull statistics for strength data.
ISO 20502	Apr-2005	Determination of adhesion of ceramic coatings by scratch testing.
ISO 20507	Dec-2003	Vocabulary.
ISO 20508	Dec-2003	Determination of light transmittance of ceramic films with transparent substrate.
ISO 20509	Dec-2003	Determination of oxidation resistance of non-oxide monolithic ceramics.
ISO 20808	Feb-2004	Determination of friction and wear characteristics of monolithic ceramics by ball-on-disc method.
ISO 24370	2005	Test method for fracture toughness of monolithic ceramics at room temperature by chevron-notched beam (CNB) method.
Key: (1) Always confirm with standards organisations the version of a standard is current. (2) ISO 'fine ceramics' standards that cover fibre-reinforced composites are not included.		

Table 43.3-2 – Technical ceramics: ISO standards - drafts

Number (2)	Date (1)	Title
Draft standards - Fine ceramics (advanced ceramics, advanced technical ceramics) (3)		
ISO/CD 22197-1	-	Test method for air purification performance of semiconducting photocatalytic materials -- Part 1: Removal of nitric oxide.
ISO/CD 24235	-	Determination of particle size distribution of ceramic powders by laser diffraction method.
ISO/DIS 18452	-	Determination of thickness of ceramics films by contact-probe profilometer.
ISO/DIS 22214	Nov-2004	Test method for cyclic bending fatigue of monolithic ceramics at room temperature.
ISO/DIS 22215	Nov-2004	Test method for tensile creep of monolithic ceramics.
ISO/DIS 24369	Feb-2004	Determination of content of coarse particles in ceramic powders by wet sieving method.
ISO/DIS 24370	Jan-2004	Test method for fracture toughness of monolithic ceramics at room temperature by chevron notched beam (CNB) method.
ISO/FDIS 24369	-	Determination of content of coarse particles in ceramic powders by wet sieving method.
ISO/PRF 17092	-	Determination of corrosion resistance of monolithic ceramics in acid and alkaline solutions.
ISO/WD 23145	-	Determination of tap density of ceramic powders.
ISO/WD 23146	-	Test methods for determination of fracture toughness of monolithic ceramics- Single edge V-notch beam (SEVNB) method.

Key: (1) Date included for guidance only. Always confirm with standards organisations the version of a standard is current.

(2) ISO abbreviations: FDIS: Final draft international standard; DIS: Draft international standard; PRF: Proof of international standard; CD: Committee draft; WD: Working document.

(3) ISO 'fine ceramics' standards that cover fibre-reinforced composites are not included.

43.4 Processing aspects

43.4.1 Manufacturing processes

Owing to the high melting point, most technical ceramic materials are produced by consolidating the raw materials in the form of powders.

A few of the materials can be produced by deposition from a vapour phase.

Ceramic powders are available in a range of purities, particle sizes and size distributions, adapted to various processing techniques. Additives are used to aid blending (mixing) and as binders to aid shape retention in the green form prior to machining and firing. Some powder stock is blended wet, then spray-dried prior to processing into a green shape.

Various methods of producing shapes have evolved and there can be several variants of the same basic process. Process selection depends on size and shape of the component and on production rates. Some more sophisticated shaping processes include the densification step.

Figure 43.04.1 shows a schematic of the powder processing sequence, Ref. [43-1].

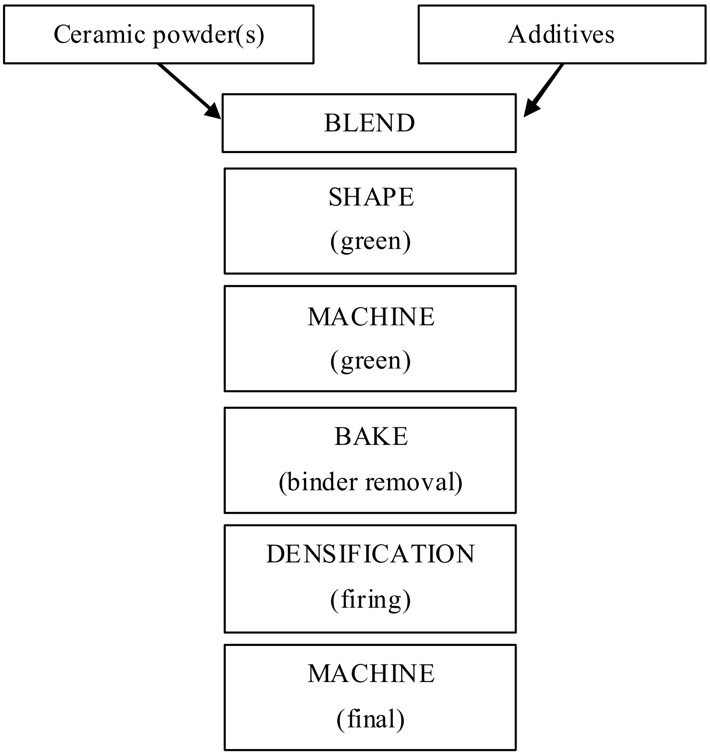


Figure 43.4-1 – Technical ceramics: Manufacturing processes

43.4.2 Health and safety

Some materials have known toxicological occupational health effects, e.g. beryllia and some man-made vitreous silicate fibres; nano-sized materials are also under investigation. Material suppliers provide material safety sheets that stipulate the necessary handling procedures and protection measures.

Ceramic processing involves working with fine powders that can easily become dispersed, carried in the air and ingested. In general, precautions are necessary to control the levels of airborne materials and also debris resulting from machining.

Standards organisations and occupational health authorities provide guidance on the monitoring and exposure levels for personnel working with ceramics, Ref. [43-41].

43.4.3 Green shapes

43.4.3.1 General

Green shapes are parts in an intermediate state following initial consolidation of blended powders but prior to high pressure and/or temperature processes that give the desired mechanical properties.

Process methods can be grouped by whether the blended powders are used dry or wet. Wet shaping processes use a variety of liquids, such as water or solvents. These, along with various additives, aim to produce an even suspension of ceramic particles within a slurry or slip. The liquids are removed before firing. Depending on the process, this can involve: evaporation, absorption by the mould substrate or a drying process applied to the green shape. Common processes for producing green shapes are summarised in Table 43.4.1, Ref. [43-1].

Table 43.4-1 – Technical ceramics: 'green' shape processes

	Dry pressing	Roll compaction	Injection moulding	Tape casting	Extrusion	Sol-Gel	Slip casting
Dry	⊙	⊙					
Wet			○ (1)	⊙	⊙	⊙	⊙
Key:	(1) Although feedstock is wet to enable injection, the green shape is dry on ejection.						

43.4.3.2 Dry pressing

Green shapes are made from dry powders that are placed in a mould and pressed. Variants on the basic technique are:

- Uniaxial compaction is a technique that uses a rigid mould and a punch to apply the compaction pressure. Production rates can be high for both simple and complex shapes. Using two punches operating in opposite directions on the same axis with a floating mould aids compaction and green compact removal.
- Wet-bag cold isostatic pressing, in which the powder is held in a rubber bag mould and submerged in a liquid. Consolidation is achieved by pressure transmitted through the liquid. This process is fairly slow and used for large items.
- Dry-bag cold isostatic pressing, similar to wet-bag in that powder in a rubber mould is pressurised via a liquid, but the rubber mould is integrated into the equipment. This enables automation and small components can be produced at high production rates.

43.4.3.3 Roll compaction

Spray-dried powders, containing about 5% to 10% of binders and plasticisers, are continuously fed between two rotating rollers. As the rolls come together, the high pressure compacts the powder. The tape produced possesses uniform high density and sufficient green strength and flexibility to be spooled.

43.4.3.4 Injection moulding

A granulated mixture of ceramic and organic binders is charged into the machine and the air removed. A heated charge is then injected into a cold die cavity, where it cools slightly prior to ejection.

Usually for making small, complex-shaped green components, injection moulding has also been used for prototyping complex engine parts, such as rotors and turbines in turbochargers.

43.4.3.5 Tape casting

Tape casting is based on the patented Park process and is also known as 'doctor blade' or knife casting'.

A slurry or slip is produced from a suspension of ceramic powder in a liquid along with additives appropriate to the materials and densification process, e.g. binders, plasticisers and deflocculants. The slip is then fed onto a polymer sheet that passes under a blade. The height of the blade controls the thickness of the as-cast sheet or tape. After drying, to remove the solvent, the green tape is then spooled.

The process is used for making continuous thin sheets or tapes of ceramic, often used as substrates for electronic devices.

43.4.3.6 Extrusion

A slurry is produced from ceramic powder mixed with water, an organic solvent or sometimes pitch. A variety of additives are used to adjust the flow characteristics of the mix and to ensure sufficient green strength after extrusion.

The slurry is then forced through the extrusion die to form continuous green compacts of constant cross section, e.g. tube, rod, channel and honeycomb sections.

43.4.3.7 Sol-Gel

The 'Sol' is a liquid colloidal solution containing dissolved metallic salts or organic compounds. A rigid 'Gel' is formed from the solution and is then dried prior to densification (firing). The basic technique has many variables, enabling a wide range of compositions and products, including: shapes, films, fibres and very low density, porous, aerogels

The process is used for exceptionally fine, pure ceramics, e.g. ultra fine grained alumina and some speciality compositions for functional electronic devices.

Owing to the reactive nature of the ceramic produced, the sintering temperatures are lower than those necessary for conventionally-produced materials.

Sol-Gels are also used in the manufacture of ceramic matrix composites, where the Sol is infiltrated into a fibre preform which acts as a deposition substrate, [See also: 88.16].

43.4.3.8 Slip casting

Slip (or slurry) is a liquid suspension of ceramic particles, usually in water, containing various additives to aid processing. This is poured into or over a porous mould (usually plaster), which absorbs some of the water near the interface. Excess liquid slip is then removed leaving the green shape in contact with the mould. The moulded thickness can be controlled by process parameters such as the liquid content of the slip or time in contact with the mould. The green shape is removed from the mould and dried prior to firing.

Slip casting is a widely-used traditional processing technique within the ceramics industry. It can produce large, complex-shaped items.

43.4.4 Densification

43.4.4.1 General

Following a drying process, the majority of green shapes are densified by sintering (firing). Depending on the material composition and temperature, the densification process is different.

43.4.4.2 Liquid-phase sintering

At the sintering temperature a small quantity of liquid phase forms, less than needed to fill all the voids. The remaining solid phase is soluble in the liquid and fills the voids by a combination of diffusion, dissolution and redeposition.

43.4.4.3 Viscous-flow sintering

Part of the material melts at the sintering temperature and flows by capillary action into the voids. After cooling, this phase can be crystalline or glassy.

43.4.4.4 Solid-state sintering

During sintering no liquid phase forms and consolidation is by solid-state diffusion only. Fine powders and high temperatures are necessary for this process.

43.4.4.5 Reaction bonding

Mixtures of suitable ceramic powders are subjected to conditions that enable a chemical reaction between the constituents, forming a densified product, e.g. high temperatures and reactive gaseous atmospheres. Reactions can occur in the solid, liquid or gaseous states.

Silicon carbide and aluminium nitride can both be produced by these methods, e.g. $C + Si \rightarrow SiC$; $2Al + N_2 \rightarrow 2AlN$.

43.4.5 Combined shaping and densification

43.4.5.1 Hot pressing

Powders are enclosed in a die, often made from graphite to enable induction heating. Heat and pressure are then applied. Depending on the material composition and temperature, densification can be liquid-phase or solid-state.

Hot pressing is limited to fairly simple shapes. Machining of densified shapes needs very hard tools.

43.4.5.2 HIP - hot isostatic pressing

The ceramic powders are sealed into a metal or glass capsule and then loaded into the HIP pressure vessel. High-pressure gas, usually argon, compacts the powder at high temperature. The sintering process can be liquid-phase or solid-state. The consolidated materials can approach full theoretical (i.e. voidless) density.

HIP is also used as a final or remedial process to close voids and increase the density of materials processed by other techniques.

Complex shapes can be consolidated effectively by HIP, but the capacity of the HIP pressure vessel limits the size of components. Machining of the densified shape needs very hard tools.

43.4.6 Vapour-phase techniques

43.4.6.1 General

There are several processes in which ceramic materials are deposited from a chemical atmosphere onto a substrate at high temperatures. The size and shape of components produced are limited by the process vessel dimensions.

Vapour-phase processes are also used for the manufacture of ceramic matrix composites, where the fibre preform acts as the deposition substrate, [See also: Chapter 88].

43.4.6.2 Chemical vapour deposition

The decomposition of a gas mixture contained in a pressure vessel at high temperature deposits high-purity material onto a substrate surface. Material properties tend to be anisotropic.

CVD is normally used to create thin-walled shapes or coatings. Process times are usually quite long, but can be decreased by a modified technique using a plasma around the substrate; known as PACVD, plasma-assisted CVD. CVI chemical vapour infiltration is a CVD technique in which the material is deposited on and between the fibres of a preform to create a ceramic matrix composite.

CVD is used to produce synthetic diamond materials and shapes for a variety of wear parts, thermal management and optical uses, Ref. [43-8]; [See also: 43.17].

43.4.6.3 Physical vapour deposition

One or more materials transform from the solid state into a vapour phase under vacuum and then condenses onto a substrate. PAPDV, plasma-assisted PVD, enables virtually any material to be deposited as a coating onto a non-porous substrate.

43.4.7 Machining

Although comprising of compacted abrasive powders, the strength of green shapes (green strength) is sufficient to enable them to be machined fairly easily without the need for expensive, hard cutting tools. The majority of features tend to be moulded into the shape rather than machined. Where necessary, machining is used in the green stage to reduce as far as possible the need to machine components after firing.

Densified materials are much more difficult to machine due to their hard, brittle nature. Machining operations are usually limited to finishing surfaces using very hard grinding media.

43.4.8 Joining

43.4.8.1 General

Techniques for joining ceramics can be classed as those used to assemble green shapes, prior to firing, or those used to assemble fully-densified materials (post-firing).

Some recent novel techniques involve diffusion bonding, the use of compatible interlayers and local infiltration to produce joints. Processes using microwave heating and ultrasonics are also under development for ceramics, Ref. [43-48].

43.4.8.2 Green shape assembly

Large items, often of complex shape, can be produced as a series of sub-components which are then joined prior to firing. Depending on the material and the process technique used to create the green part, a slip of the same, or a compatible material, can be used as 'adhesive'. After baking to remove moisture or solvent, the part is fired and the slip forms a bond.

Some ceramic-based adhesives can be used instead of slip provided that their firing temperature is compatible with the component densification temperature.

43.4.8.3 Fully-densified materials

The choice of assembly method largely depends on the application and service environment. The main problems to consider are avoiding point or uneven loads and thermal mismatch between the ceramic and adjacent parts; both of which can cause fracture in ceramics, Ref. [43-48].

Ceramic parts can be retained within metal components by shrink-fitting. This relies on the thermal expansion differences between metals and ceramics to produce a compression-type joint when the mixed-material component has cooled. This process is widely-used for locally increasing the wear resistance of engineering parts, such as thread guides in process machinery and for the assembly of spark-plugs.

Mechanical fastening can be used providing that point and uneven loads are avoided. Mechanical seals are used to position and retain ceramic parts between other materials, e.g. O-ring seals to position equipment windows.

Bonded joints are limited by the working temperature range of the adhesive. Ceramic-based adhesives can be used for bonded assemblies in high-temperature applications. These adhesives usually need firing, but this can be at a lower temperature than the densification process. Conventional adhesives can also be used providing that they tolerate the thermal conditions, adhere to the surfaces and have sufficient mechanical performance.

Some ceramics are assembled by brazing, where the metal-based filler is used as the adhesive, e.g. assembly of ceramic tips and inserts onto metal tool supports. To ensure the quality of brazed joints, the surfaces of ceramic parts are metallised with a thin metal-rich coating in order to improve adhesion with the braze filler. Thermal gradients created during brazing need to be minimised to avoid possible failures. To limit thermal gradients, assemblies are often heated prior to brazing and cooled slowly and evenly afterwards.

43.5 Applications

43.5.1 Industrial

Technical ceramics have proven success in many diverse applications covering a wide range of industry sectors, e.g. from aggressive environments in the petrochemical process industries to prosthetic biomedical implants.

43.5.2 Aerospace

43.5.2.1 General

Within the aerospace industry as a whole monolithic technical ceramics are widely used in equipment and payloads. Some applications are based solely on electrical or optical performance, e.g. electrical-electronic components and assemblies, sensors or optics; whereas others are, to some extent, load-bearing. These are often static loads but are increasingly being used in dynamic situations, e.g. pump parts, such as bearings, valves or seals; within combustion chambers; turbochargers; discs in braking systems.

Ceramics are now used for the mirrors and structures of scientific space instruments that need high stiffness and stability. Ceramics also offer interesting properties for cryogenic applications.

43.5.2.2 Environment – components

Technical ceramics are selected to resist the working environment throughout the service temperature range for the intended service life. Depending on the aerospace application, the environment for technical ceramic engineering components can range from high to low temperatures, and include contact with gases, fluids or solids, e.g. nozzles, pump parts, seals.

43.5.2.3 Environment – structures

For structural applications, the environments comprise:

- Earth, during manufacture and storage. Ceramics are rarely 100% densified, so retain some level of residual porosity. If not properly controlled, moisture and contaminant uptake is possible unless exposed surfaces are sealed. Sealing is an established process for technical ceramics in direct exposure to 'chemical' environments. A bake-out process is often used to remove contaminants before or after sealing.
- Launch; where conditions are largey dictated by the launch vehicle, e.g. vibration levels. For ceramic-type materials, the effects of dynamic loads need evaluation as do any interfaces between the ceramics and adjacent materials.
- Space environment (mission-related).

43.5.2.4 Environment – space

Although levels are mission-dependent, the general effects of the space environment on technical ceramics are summarised as, [See also: ECSS-Q-70-71]:

- Vacuum: provokes outgassing of residual, process-related, materials and moisture. Components to be exposed to vacuum are usually baked-out and sealed (glazed).
- Radiation: levels in space have no known effects on the characteristics of ceramics.
- Temperature: High-temperature performance is a main reason for selecting ceramics. However, the upper working temperature varies for different groups of materials, especially when combined with an aggressive chemical environment, e.g. carbon-based materials in oxygen-rich environments.
- Thermal cycling: can cause cracking in ceramics due to shape-effects and thermal gradients.
- Atomic oxygen: has no known effect on ceramics.

43.5.3 Materials

43.5.3.1 General

Whilst some materials are used for many different applications, others tend to be more limited to exploit a particular characteristic, e.g. IR, microwave or radar transparency.

Excluding the abrasive and cutting tools sector, manufacturers and standards organisations alike, tend to group technical ceramics by their usual type of applications, Ref. [43-5], e.g.:

- mechanical components,
- thermomechanical components,
- chemical components, include items used in industrial processing which are often in very aggressive environments (acids, alkalis, chemical slurries, liquid metals, abrasive). Corrosion, often at high temperatures, or wear resistance are the main selection factors.
- electrical insulators, as defined in IEC 672 EN60672-1, Ref. [43-7].
- electrical conductors, used in fuel cells, batteries, heating elements, gas sensors and electrodes; static discharge.
- functional electrical materials, which exhibit dielectric properties or characteristics, e.g. ferroelectric, piezo- or pyro-effects.
- magnetic materials.

Whilst all of these applications exist to some extent within the aerospace industry, only those which usually provide some degree of load-bearing capability combined with the specialist properties of ceramics are presented here. Materials considered for dimensionally-stable and optical structures are also included.

43.5.3.2 Mechanical components

Some technical ceramics commonly used for discrete, often simply-shaped, mechanical components are summarised in Table 43.5.1. These items usually provide a level of load-bearing combined with wear or corrosion resistance.

Table 43.5-1 – Technical ceramics: Materials - mechanical components

	Aluminas	Aluminium nitride	Boron carbide	Boron nitride	Carbons	Diamond (CVD)	Silicas	Silicon carbide	Silicon nitride	Zirconia	Glass ceramics
Bearings *	⊙	⊙			⊙(1)				⊙		
Bearings (ball roller)									⊙		
Bearings (sleeve)	⊙							⊙			
Injector pins (fuel)									⊙		
Pumps *	⊙				⊙(1)			⊙		○	
Seals * (shaft)	⊙				⊙(1)			⊙	⊙		
Seals * (mechanical)	○				⊙(1)			⊙		○	
Valves (ball)	⊙									⊙	○
Valves (pneumatic)	⊙									⊙	
Valves (trains)									⊙		

Key: * wear resistance; ⊙ primary material; ○ secondary material; (1) carbon-graphites

43.5.3.3 Thermomechanical components

Technical ceramics for high-temperature technologies are summarised in Table 43.5.2. These can be relatively large, complex-shaped items operating at high temperatures in hostile environments under dynamic or static load conditions. Carbon-carbon and ceramic matrix composites have also been evaluated for some of these applications, [See: Section XII].

Table 43.5-2 – Technical ceramics: Materials - thermomechanical components

	Aluminas	Aluminium nitride	Boron carbide	Boron nitride	Carbons	Diamond (CVD)	Silicas	Silicon carbide	Silicon nitride	Zirconia	Glass ceramics	Carbon-Carbon (2)	CMC(3)
Brake discs								○				⊙	
Combustion chambers (diesel)									⊙				
Gas turbines					⊙(1)				⊙				⊙
Jet engine (combustors)								⊙	⊙				
Jet engine (petals)													⊙
Rocket nozzles					⊙(1)			○	○			⊙	
TPS (active)												⊙	
TPS (passive)							⊙					⊙	⊙
Turbo charger (housings)								○	⊙				
Turbo charger (rotors)									⊙				
Valves (reciprocating engines)									⊙				

Key: TPS thermal protection system; ⊙ primary material; ○ secondary material;
(1) carbon-graphites; (2) [See: Chapter 54]; (3) [See: Chapter 52]

43.5.3.4 Electrical

The primary criterion in selecting technical ceramics for electrical and electronic uses is the insulating or electrical conductivity behaviour. Service temperature can affect this, e.g. zirconia becomes conductive at high temperatures.

Table 43.5.3 summarises some of those technical ceramics which are used as insulators or conductors. Only a few examples are provided for electrical and electronic applications from those that constitute a large proportion of the technical ceramic market.

Table 43.5-3 – Technical ceramics: Materials - electrical

	Aluminas	Aluminium nitride	Boron carbide	Boron nitride	Carbons	Diamond (CVD)	Silicas	Silicon carbide	Silicon nitride	Zirconia	Glass-ceramics
INSULATORS (1)	✸	✸	✸	✸			✸		✸	✸ (2)	✸
Electronic devices	⊙	○(3)				○(3)					
High-strength									⊙		
Machinable				⊙							⊙
Radomes	⊙						⊙				
CONDUCTORS					✸			✸		✸ (2)	
Batteries	⊙									⊙	
Electrodes					⊙						
Fuel cells	⊙									⊙	
Heating elements					○			⊙			

Key: (1) For a comprehensive list of insulators, See: IEC 672 EN60672-1: Definitions and classifications;
(2) Zirconia is conductive at high temperatures;
(3) high-thermal conductivity;
⊙ primary material; ○ secondary material

43.5.3.5 Dimensionally-stable and optical structures

Technical ceramics for optical applications can be grouped as:

- optical devices and components, which are transparent to particular wave lengths, e.g. windows, laser components.
- mirrors and support structures for optical systems; providing highly stabilty and minimising thermal expansion problems which can affect system performance, e.g. telescopes, mirrors, antennas.

Excluding glasses, Table 43.5.4 gives examples of technical ceramics that are considered for some optical applications, [See also: Case studies 43.18; 43.19].

Table 43.5-4 – Technical ceramics: Materials – optical and dimensionally-stable structures

	Aluminas	Aluminium nitride	Boron carbide	Boron nitride	Carbons	Diamond (CVD)	Silicas	Silicon carbide	Silicon nitride	Zirconia	Glass-ceramics
Laser components	⊙ (1)					○					
Mirrors							⊙	⊙(2)			⊙
Mirrors (X-ray)								⊙			
Optical (support structures)								⊙			⊙
Telescopes							⊙				⊙
Windows (visible and infrared)	⊙ (1)					○	⊙				
Key: ⊙ primary material; ○ secondary material; (1) ruby or sapphire; (2) mirrors for telescopes											

43.6 Aluminium oxide: Alumina

43.6.1 Typical characteristics

43.6.1.1 Standard grades

Commercially-available alumina materials are generally grouped by:

- Form, e.g. powders, granules or pellets, shapes or components.
- Purity and composition usually stated as the percentage of the Al2O3 content. Some grades also give a composition if an addition of another material is included, e.g. alumina + zirconia. Trace elements, or impurities, are rarely stated.
- Crystal size, in the micron range.

The majority of engineering grades are opaque white, although pinks and browns also exist. Some grades are translucent, [See: Special grades].

The purity and crystal size affect the properties of the densified materials.

Basic features of alumina materials include:

- Mechanical: high stiffness, hard and brittle; toughness and strength improved by Zirconia additions (ZTA-grades).
- Thermo mechanical: properties are generally retained to about 800°C, but reduce significantly above 1000°C; medium shock and thermal cycling resistance.
- Electrical: non-conductive; excellent electrical insulation.
- Environment: chemical resistance to both acid and alkalis (except HF).

43.6.1.2 ZTA – Zirconia toughened alumina

ZTA is higher in strength and toughness than conventional alumina grades. This result from a stress-induced transformation toughening effect achieved by incorporating about 10% to 20% fine zirconia particles uniformly throughout the alumina.

Typical characteristics include:

- Strength,
- Toughness,
- Wear resistance,
- High temperature stability,
- Corrosion resistance.

ZTA grades are used in mechanical applications, such as wear parts, valve seats, pump components, bearings, bushes.

43.6.1.3 Special grades

Translucent alumina is used for higher-temperature, optical uses, such as lighting.

Synthetic sapphire is a single crystal form of alumina, which is used in optical applications, e.g. laser systems and windows for imaging systems [See also: 43.16].

43.6.2 Typical properties

43.6.2.1 General

The properties stated by manufacturers of alumina materials depend on the form in which the materials are supplied and also their main applications. It is unusual to have a complete set of mechanical, thermo-mechanical, physical and electrical properties for any particular grade of material.

43.6.2.2 Powders

Powder properties often state an average powder size, although some give particle size distributions. Density values can be for loose powder, tamped or green state (compacted but not fired). Some suppliers give an indicative bulk density for a densified (fired) material.

43.6.2.3 Shapes and components

Density values are given along with a typical porosity level, which can be stated as % apparent or open (interconnecting pores) or % closed and interconnecting pores.

For some applications, porous alumina grades are needed, e.g. thermal insulation and catalytic supports.

Commonly-stated mechanical properties are some combination of:

- Hardness (on various scales),
- Compressive properties (strength and modulus),
- Flexural strength,
- Fracture toughness.

Tensile properties are less common because suppliers assume that applications avoid using ceramics under tensile or bending loads.

Thermal characteristics often stated are a coefficient of thermal expansion, thermal conductivity and a maximum use temperature, usually in air. Some also provide a thermal shock value.

Electrical characteristics stated can include dielectric properties and volume resistivity at various temperatures.

Table 43.6.1 summarises the properties of some examples of densified commercial alumina materials, [43-12].

The grades presented, taken from a much wider range of alumina products, are all used in load-bearing, mechanical applications rather than for electrical or thermal characteristics alone.

The large number of grades available has evolved over the years to meet particular applications in various industry sectors. In general, this is to achieve a balance between the wear, chemical, mechanical and other characteristics. Grades have also been developed for specialist applications, e.g. ballistic protection.

Although the materials cited all come from one source, it also illustrates the range of materials that are available from other major technical ceramics suppliers.

Table 43.6-1 – Technical ceramics: alumina – typical properties

Property	Examples of commercially-available alumina grades †								
	HILOX™ 991 MAC-A990S	HILOX™ 965 MAC-A955S	HILOX™ 961 MAC-A960S	HILOX™ 882 MAC-A840S	SINTOX™ FL MAC-A950R-6	SINTOX™ FF MAC-A950R-5	SINTOX™ FC MAC-A950R-4	SINTOX™ AL MAC-A997R	DERANOX™ 975 MAC-A975R
Alumina content (%, typical)	99	95 - 96	96	84	95	96	95	99.7	97.5
Grain size (μm)	-	-	2.8	-	4	6	4	-	4
Bulk density (fired) (kg/m^3)	3900	3790	3880	3530	3720	3700	3720	3200	3800
Porosity apparent (5) (%)	0	0	0	0	0	0 (4)	0	20(3)	0
Vickers (GPa, Hv0.5kg)	-	-	-	-	14.6	12.5	14.6	-	15
Rockwell (R45N)	86	83	84	76.4	-	78		-	85
Compressive strength (MPa)	-	-	2100	1800	2500	2000	2500	-	2500
Flexural strength (1) (MPa)	370	283	376	271	325	320	325	150	350
Young's modulus (GPa)	-	336	354	250	330	325	330	-	340
Fracture toughness (MPa $m^{1/2}$)	-	-	-	-	5.9	4.5 (2)	5.9	-	3.6
Thermal conductivity (WmK, 20°C)	-	-	21	15	20	21	20	-	24
Coefficient of thermal expansion, 20°C to 1000°C ($\times10^{-6}$ /°C)	-	8.2	8.2	8.2	7.5	7.5	7.5	9.0	8.1
Applications (examples)	Wear parts, pumps, shafts, bearings, seats (chemical environment)	Wear parts, pumps, shafts, bearings, seats; valve seats; pneumatic valve sliders	Wear parts, pumps, shafts, bearings, seats	Automotive water pump parts	Dynamic pump parts for corrosive media; wear parts	Aerospace parts (non outgassing);	Dynamic pump parts for corrosive media; wear parts	Laser reflectors	Dynamic and static components; pumps, valves; chemical or abrasive environment

Key: † SINTOX, HYLOX and DERANOX are trademarks of Morgan Advanced Ceramics. (1) 3-point bend (ASTM C1161), 20°C; (2) K_{IC} (SENB); (3) Porous grade for laser reflectors; pumping chambers; (4) Non outgassing for aerospace applications; (5) Fully dense.

43.6.2.4 ZTA – Zirconia toughened alumina

Table 43.6.2 summarises properties of an example of a ZTA commercially-available material compared with an alumina grade used for similar mechanical applications, Ref. [43-12], [43-32].

Table 43.6-2 – Technical ceramics: ZTA zirconia toughened alumina – typical properties

Property	Comparison of commercially-available grades of alumina with ZTA †	
	HILOX™ 965	ZTA
Composition	Alumina	Zirconia-toughened alumina
Zirconia content (%, typical)	0	not stated
Grain size (µm)	-	2
Bulk density (fired) (kg/m^3)	3790	4050
Porosity apparent (%)	0	-
Hardness, Knoop (GPa, 1000g)	-	14.4
Rockwell (R45N)	83	85
Compressive strength (MPa)	-	2900
Flexural strength (1) (MPa)	-	450
Flexural strength (2) (MOR, MPa)	283	-
Young's modulus (GPa)	336	360
Fracture toughness (MPa m$^{1/2}$)	-	5 to 6
Thermal conductivity (WmK, 20°C)	-	27
Coefficient of thermal expansion, 20°C to 1000°C (×10^{-6} /°C)	8.2	8.3
Key: † ZTA - CoorsTek product; HILOX is a tradename of Morgan Advanced Ceramics; (1) 3-point bend (ASTM C1161), 20°C; (2) ASTM F417		

43.7 Aluminium nitride

43.7.1 Typical characteristics

General features of aluminium nitride ceramics include:

- high thermal conductivity
- good electrical insulation.

The thermal and electrical properties depend on purity. Powder forms are prone to moisture uptake that can affect electrical properties. Some typical applications include uses in the semiconductor processing industry, heat management in electronics and windows for visible to infra-red systems.

43.7.2 Typical properties

43.7.2.1 General

Table 43.7.1 summarises properties for some examples of commercially-available aluminium nitride materials, Ref. [43-14], [43-15].

43.7.2.2 Shapal™ machinable grade

Shapal™ is produced by Tokuyama, Japan but is available in Europe from distributors. The Shapal™ grades are high-purity, machinable aluminium nitrides, which offer strength and thermal conductivity. Shapal™-M is a machinable grade and Shapal™-SH 15 is translucent to visible and IR light.

The thermal conductivity is not as high as standard aluminium nitride, but is about 50 times greater than that of machinable glass ceramics, [See also: 43.15]

The ability to machine Shapal™ with conventional carbide tools means that more complex shapes can be made compared with standard aluminium nitride ceramics.

Applications are similar to standard grades, e.g. substrates for semiconductors; heat sinks and insulating parts for high-power electronics; window materials for infrared and radar applications; refractories (molten metals).

Table 43.7-1 – Technical ceramics: Aluminium nitride – typical properties

Property	Examples of commercially-available aluminium nitride grades †						
	CERALLOY™ 1370DP	CERALLOY™ 1370CS	CERALLOY™ 13701E	AN 215	AN 217	AN 216A	AN 2000
AlN content (%, typical)	98.0	99.0	99.8	-	-	-	99.9 (2)
Grain size (μm)	10	10	4	-	-	-	-
Bulk density (fired) ($\times10^3$ kg/m^3)	3300	3300	3260	3400	3400	3400	3200
Porosity apparent (%)	-	-	-	-	-	-	-
Hardness (kg/mm^2)	1110	1000	1100	-	-	-	-
Vickers (GPa, Hv0.5kg)	-	-	-	10.8	10.4	10.4	11.2
Rockwell (R45N)	-	-	-	-	-	-	-
Compressive strength (MPa)	-	-	-	-	-	-	-
Flexural strength (MPa)	200	200	330	290	310	310	220
Young's modulus (GPa)	320	320	320	320	320	320	310
Fracture toughness (MPa m$^{1/2}$)	3.0	3.0	2.5	-	-	-	-
Thermal conductivity (WmK, 20°C)	170	170	80	150	160	150	67
Coefficient of thermal expansion, 20°C to 1000°C ($\times10^{-6}$/°C)	6.2	5.6	4.9	5.4 (1)	5.4 (1)	5.3 (1)	5.2 (1)
Applications (examples)	Sintered; Semiconductor components; microwave parts	Hot pressed; Semiconductor components	Hot pressed; Semiconductor components; Gas distribution	Semiconductor devices (IC packages)	Semiconductor devices (IC packages)	Semiconductor devices (IC packages)	Semiconductor processing industry; Plates for uniform heating; High temperature supports

Key: † CERALLOY is a tradename of Ceradyne Inc., AN grades are from Kyocera Corp.; (1) CTE 40°C to 800°C; (2)High purity grade.

43.8 Boron carbide

43.8.1 Typical characteristics

Boron carbide has a low density (2500 kg/m3) and a hardness approaching that of diamond.

General features of densified boron carbide include:

- Low density.
- High modulus.
- Extremely hard.
- Susceptible to thermal shock.

Excluding abrasives, typical applications are for wear resistance, e.g. nozzles (processing equipment), dies, precision engineering parts. It also has some specialist applications such as impact resistance (ballistic armour) and in nuclear shielding (neutron absorption).

Boron carbide can be processed in a powder form or produced by vapour-phase techniques.

43.8.2 Typical properties

Table 43.8.1 summarises the properties of some examples of commercially-available boron carbide materials, Ref. [43-14].

Table 43.8-1 – Technical ceramics: boron carbide – typical properties

Property	Examples of commercially-available boron carbide grades †		
	CERALLOY™ 546	CERALLOY™ 546-3E (1)	CERALLOY™ 546-4E
B_4C content (%, typical)	98.5	98.5	99.5
Grain size (µm)	15	15	15
Bulk density (fired) ($\times 10^3$ kg/m^3)	2500	2500	2500
Porosity apparent (%)	-	-	-
Hardness (kg/mm^2)	3200	2800	3200
Vickers (GPa, Hv0.5kg)	-	-	-
Rockwell (R45N)	-	-	-
Compressive strength (MPa)	-	-	-
Flexural strength (MPa)	410	410	410
Young's modulus (GPa)	460	460	460
Fracture toughness (MPa m$^{1/2}$)	2.5	2.5	2.5
Thermal conductivity (WmK, 20°C)	90	90	90
Coefficient of thermal expansion, 20°C to 1000°C ($\times 10^{-6}$ /°C)	5.6	5.6	5.6
Applications (examples)	Hot pressed; ballistic armour; semiconductor processing; wear parts.	Hot pressed; semiconductor processing.	Hot pressed; ballistic armour; semiconductor processing; wear parts.

Key: † CERALLOY is a tradename of Ceradyne Inc.; (1) High-purity, single phase, low porosity grade.

43.9 Boron nitride

43.9.1 Typical characteristics

43.9.1.1 General

Boron nitride is commercially available as:

- Hexagonal boron nitride. (H-BN);
- Cubic boron nitride (C-BN).
- Pyrolytic boron nitride (PBN), produced by a vapour-phase process.

The characteristics of H-BN and C-BN, dictated by their crystal structures, are totally different.

43.9.1.2 H-BN - hexagonal boron nitride

H-BN has a graphite-like crystal structure. The lattice layers and plate-like structure provide good lubricating properties. H-BN is usually formed by hot pressing because of its resistance to sintering.

Features of H-BN include:

- Hot-pressed blanks can be machined, using conventional techniques, to make complex-shaped parts,
- Anisotropic properties, when produced by hot pressing,
- Good electrical properties (dielectric strength; resistivity).
- High chemical resistance (resistant to most molten metals, glasses, salts),
- Excellent lubricating properties,

Excluding lubricants, H-BN is used for shaped parts in materials processing industries, including HIP-produced ceramics, due to its non-wetting capability; high-temperature electrical insulators (about 850°C in air due to its tendency to oxidise; about 1350°C in inert or reducing atmospheres).

43.9.1.3 C-BN - cubic boron nitride

The structure of C-BN is the same as diamond and its characteristics are very similar. General features of C-BN include:

- Hardness approaching that of diamond,
- Excellent wear resistance,
- High thermal conductivity,
- Good chemical inertness.

Excluding abrasives, C-BN is used for cutting tools, wear-resistant parts, coatings and substrates for high-power electronic devices.

43.9.1.4 PBN - pyrolytic boron nitride

PBN is a high-purity, pyrolytic material formed by a vapour deposition process. The structure of the deposited PBN is hexagonal, as shown in Figure 43.9.1, Ref. [43-17].

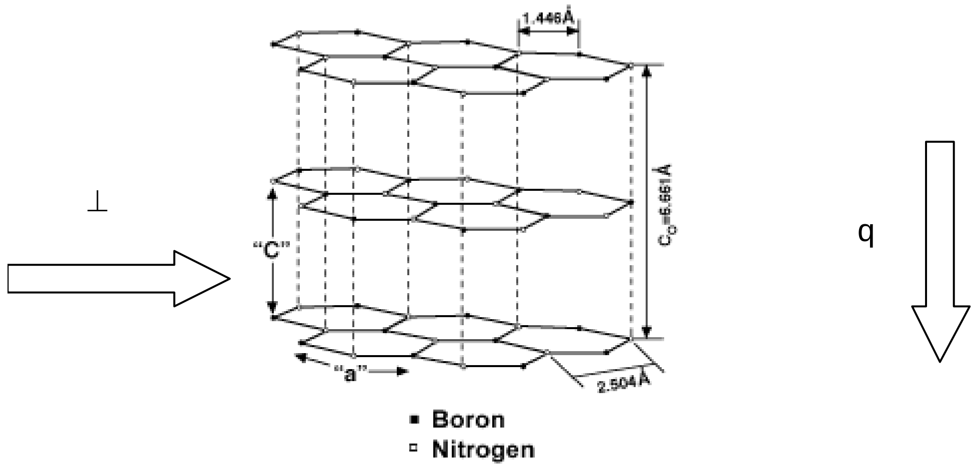


Figure 43.9-1 – Technical ceramics: pyrolitic boron nitride – structure

As with H-BN, PBN is highly anisotropic in its electrical and mechanical properties, but very resistant to thermal shock. It is also a very good electrical insulator and has very high thermal conductivity. The material is stable in inert and reducing atmospheres up to 2800°C, but only to 850°C in oxidising atmospheres.

Its general features include, Ref. [43-12]:

- Low density,
- High thermal conductivity,
- Low thermal expansion,
- Good thermal shock resistance,
- High electrical resistance,
- Low dielectric constant and loss tangent,
- Good chemical resistance.

PBN is used for thin-walled electrical insulators, heating element parts and components for the semiconductor processing industry.

43.9.2 Typical properties

Table 43.9.1 summarises properties for some examples of commercially-available boron nitride materials, Ref. [43-12], [43-16], [43-17].

43.9.3 Space application examples

43.9.3.1 Combat™ boron nitride grades

Several of the St. Gobain (formerly Carborundum Corp.) Combat™ boron nitride grades have been used in inserts in Hall effect thrusters, Ref. [43-16].

Hall effect thrusters were invented in the USA and developed into the SPT - stationary plasma thruster in the ex-USSR. The thrusters are small rocket engines that use a powerful magnetic field to accelerate a low density plasma and so produce thrust.

Combat™ grades M and M26, along with HP and AX05, have been fabricated into a wide variety of thruster inserts. Each grade has demonstrated sputter erosion resistance, secondary ion emission, gas absorption and outgassing that are well within the necessary limits, Ref. [43-16].

Table 43.9-1 – Technical ceramics: boron nitride – typical properties

Property	Direction (2)	COMBAT™ A Grade	COMBAT™ HP Grade	COMBAT™ AX05 Grade	COMBAT™ ZSBN Grade	COMBAT™ M Grade (40%BN + 60% Silica)	HBR Grade™ (calcium borate)	PBN
	Examples of commercially-available boron nitride grades †							
BN content (%, typical)	-	-	-	-	-	40	94	-
Grain size (μm)	-	-	-	-	-	-	-	-
Bulk density (fired) (×10³ kg/m³)	-	1920	1900	1910	-	-	2000	2185
Porosity (%)	-	2.9	15.3	14.2	-	-	11	-
Hardness Knoop (kg/mm²) (1)	-	19.9	16.4	4.2	100	-	-	-
Vickers (GPa, Hv0.5kg)	-	-	-	-	1.4	-	-	-
Rockwell (R45N)	-	-	-	-	-	-	-	-
Compressive strength (MPa)	II	143.3	30.1	17.9	31.7	-	68.9	255
	⊥	186.6	44.5	23.4	36.8	-	62.0	331
Flexural strength (MPa	II	75.8	43.7	17.9	106.9	-	51.7	193
	⊥	113.0	60.2	21.4	144.1		41.3	
Young's modulus (GPa)	II	-	-	-	70.8	-	62.0	23.4
	⊥	-	-	-	70.5	-	48.2	-
Fracture toughness (MPa m$^{1/2}$)		-	-	-	-	-	-	-
Thermal conductivity (WmK, 20°C)	II	19.4	27.0	78.0	22.6	-	55	-
	⊥	33.7	29.0	130.0	40.2	-	33	-
Coefficient of thermal expansion, (×10^{-6} /°C) 20°C to 1500°C	II	11.85	2.95	1.0	-	-	3	-
	⊥	3.2	0.87	0.3	-	-	4	-
Applications (examples)		Electrical insulators; high-purity materials processing; Thermo-electrical (radar parts, antenna windows)	Electrical insulators; high-purity materials processing; Thermo-electrical (radar parts, antenna windows); Thruster inserts	Electrical insulators; high-purity materials processing; Thermo-electrical (radar parts, antenna windows); Thruster inserts	High-temperature mechanical components (fixtures, jigs, bearings, valves)	Thermoelectrical; thermomechanical (materials processing, thermal shock - and, chemical resistance); Thruster inserts	Electrical insulators; high-purity materials processing; Vacuum parts;	Electrical insulators; High-purity materials processing (shapes, crucibles).

Key: † COMBAT is a tradename of Saint Gobain, HBR is a tradename of GE Advanced Ceramics; (1) average values; (2) Pressing or crystal direction; (3) Other GE Advanced Ceramic grades available, including 'composite' mixture BN+AlN grade; PBN data from Morgan Performance Materials.

43.9.3.2 Sintec Keramic boron nitride grades

Boron nitride grades from Sintec Keramic (D), have been used in the propulsion system on the SMART-1 space probe, Ref. [43-45]:

- BN composite, used in the plasma chamber.
- PBN parts, used in the electrical feed of the PPS thruster.

Launched September 2003, SMART-1 was the first ESA space probe to go to the Moon. Its aims were to test the advanced technology needed for future scientific planetary missions. This involved testing miniaturisation technology whilst exploring the Moon from orbit, Ref. [43-46].

43.10 Carbon

43.10.1 Typical characteristics

43.10.1.1 General

Carbon materials are available in a variety of forms with widely differing characteristics, e.g.:

- Graphite, including densified and porous types.
- Carbon-Graphites
- Vitreous (or amorphous) carbon
- Diamond
- Pyrolytic carbon
- Siliconised carbon
- Fibres for reinforcing composites, which have a wide range of properties and characteristics, [See also: Chapter 2; 42.6].
- Carbon-carbon composites, [See also: Chapter 54]
- Foams
- Nano materials, e.g. carbon black (particles); CNT nanotubes; CNF nanofibres, [See also: Chapter 102].

Carbon materials are erroneously described as ‘graphite’ in the USA. In the context of this handbook, the term graphite is used only for the soft, hexagonally-crystallised, allotrope of carbon.

43.10.1.2 Graphite

Graphite is a relatively soft and weak material because of its plate-like crystalline structure. It possesses good self-lubricating capabilities, making it useful at temperatures where other lubricants are not reliable, e.g. at low or high-temperatures. In the vacuum of space, graphite loses its lubricating properties and is abrasive. It can however be combined with other lubricants known to perform correctly in the space environment, [See: ECSS-Q-70-71].

The properties of graphite are anisotropic if the flake structure is fully aligned. Not all commercially-available grades of graphite are aligned, so their properties are more isotropic.

Graphite is used in a structural, or semi-structural, role in many industry sectors for handling and processing corrosive chemicals, often at high temperatures, e.g. heat exchangers, large tubes and vessels, Ref. [43-21], [43-22].

Apart from densified forms of graphite, porous types are also available, e.g.:

- Exfoliated graphite (also called 'graphite foil' in the USA) is a binderless, flexible sheet material produced by expanding an interlocked graphite flake structure by over 100 times in the 'thickness' direction. The exfoliation process effectively forms an interlocked, porous material with a layered structure that renders it flexible. The composition is pure graphite with a low level of residue from materials used in the exfoliation process. Properties are anisotropic due to the alignment of layers parallel to the surface. Both the electrical and thermal conductivity are high in-plane but lower for the out-of-plane direction. Exfoliated 'flexible' graphite is used for high-temperature gaskets and seals (asbestos replacement) and thermal interfaces. It is also under evaluation for applications such as electronics (EMI shielding), vibration damping and applications in the electrochemical industries, Ref. [43-42]
- Carbon foams, which are rigid porous graphitic materials, are under consideration for spacecraft thermal management systems, Ref. [43-23], [43-24], [43-25]. The properties of the foams, e.g. density, strength and thermal or electrical characteristics, can be adapted during manufacture to suit various applications.

43.10.1.3 Vitreous (amorphous) carbon

Vitreous carbon is a non-porous, amorphous, glassy form of carbon which possesses combined strength and corrosion resistance. It is rarely used as a monolithic material, except in laboratory apparatus, but it is a major constituent in:

- Carbon-based refractories, as a solid matrix phase used to bind graphite flakes or other constituents, e.g. silicon carbide.
- Carbon-Graphites, also known as mechanical carbons.

43.10.1.4 Carbon-Graphites

Also known as mechanical carbons, carbon-graphites are a combination of amorphous carbon and graphite. There are a range of materials available, varying in composition of these two phases. The aim is to produce a material which is strong and hard, but with a low coefficient of friction. Some grades provide excellent corrosion resistance, whereas others can operate for extended periods at temperatures above 315°C. The maximum use temperature is usually determined by the oxidation rate in an oxidising environment, e.g. air. In non-oxidising atmospheres (inert or reducing) and vacuum, the maximum use temperature is about 3000°C.

Carbon-graphites are, in general, chemically-inert, temperature resistant, light-weight, resilient, dimensionally stable and impermeable to gases and liquids, Ref. [43-19]. Depending on the processing, some carbon-graphite grades can exhibit anisotropic properties, though less so than pyrolytic carbons. In these grades, the flakes in the graphitic phase are aligned to enable the material characteristics to be directionally optimised, e.g. conductivities and oxidation resistance.

Components can be moulded to shape or machined to size with close tolerances. Items can also be impregnated with a variety of substances, from resins to metal compounds to enhance performance or permeability. They can be plated, vulcanised to rubber, cemented or shrunk-fit into housings or retainers, Ref. [43-19].

Mechanical carbon materials are typically used in a variety of bearings, seals and pumps. Some examples of aerospace applications include: Space Shuttle; Mars Lander; aircraft turbine engine main shaft seals (high-speed, high-temperature, cyclic conditions); shaft seals to prevent hot gases affecting bearings.

43.10.1.5 Pyrolytic carbon

Pyrolytic carbon is produced by vapour-phase techniques in which the carbon is deposited onto substrates (CVD). The materials have anisotropic properties, but are stronger than conventional polycrystalline graphite.

43.10.1.6 Diamond

Diamond is a well-known hard, wear-resistant material. Natural diamonds are limited by the small available sizes to applications such as precision bearings in small equipment or abrasives and cutting tools. Developments in synthetic processing techniques now enable large, simple shapes to be manufactured rather than just thin films or coatings deposited on substrates. Consequently more potential applications now exist, e.g. optical and thermal management components, [See: 43.17].

43.10.1.7 Siliconised carbon

Also known as 'siliconized graphites' in the USA, these materials have a silicon carbide surface layer formed by liquid or vapour infiltration processes. Compared with carbon alone, they have increased hardness, lower porosity and improved oxidation resistance. Similar processes are used to manufacture ceramic matrix composites, [See also: Chapter 88].

43.10.1.8 Nano materials

Carbon nano materials can be broadly grouped as:

- Particles in a variety of shapes and sizes, e.g. carbon black which is an established industrial product commercially-available in many different grades, Ref. [43-56].
- Structures, e.g. CNT carbon nanotubes; CNF carbon nanofibres of various forms and aspect ratios, e.g. several tens of nanometres in diameter, several tens of micrometers long. Their appearance can be described as platelets, herringbone, screw-type. Papers and foams can be made from mixtures of CNT and CNF, Ref. [43-53]. All these materials are recent innovations and still in the early stages of development and evaluation, Ref. [43-54].

Carbon black is produced by subjecting hydrocarbons, such as heavy residual fuel-oil feedstock, to extremely high temperatures in a carefully controlled combustion process. Fine adjustment of the production process parameters results in a different size and structure of the carbon black and provides the range of commercial grades. The primary carbon particles are of the order of 12nm to 75nm in size, although these are processed to form 'aggregates' the finest of which tend to be micron-size, e.g. 0.1µm to 0.6 µm, Ref. [43-56]. Carbon black is an established and widely-used industrial product. Some typical application examples include:

- Reinforcing agent in elastomers, e.g. vulcanised rubber products, such as rubber tyres for vehicles; drive belts; hoses; gaskets and seals, [See also: 75.3].
- Polymers, e.g. conductive packaging, films, fibres, mouldings, pipes, semi-conductive cable compounds, conductive resins and similar compounds.
- Coatings, e.g. pigments, provide conductivity, UV protection.
- Toners and printing inks.

Carbon nano materials are essentially extremely fine filler substances that, when added in small quantities, are seen as a means of enhancing the characteristics of engineering materials by some amount, e.g. electrical and thermal conductivity of polymers, ceramics, glasses, Ref. [43-53].

In composites, incorporating nano materials of various compositions aims to improve the performance of the matrix phase, e.g. mechanical and thermo physical properties of polymer resins, Ref. [43-55].

[See also: Chapter 102]

43.10.2 Typical properties

43.10.2.1 General

Table 43.10.1 compares the basic properties of 100% carbon, 100% graphite and two carbon-graphite materials, Ref. [43-18].

Table 43.10-1 – Technical ceramics: carbon types – typical properties

Property [(2)]	Carbon 100%	Carbon-graphite (70%:30%)	Carbon-graphite (30%:70%)	Graphite 100%
Apparent density (kg/m^3)	1700	1720	1750	1800
Hardness	100	85	65	40
Compressive strength (MPa)	300	208	145	55
Flexural strength (MPa)	62	62	52	28
Modulus of elasticity (GPa)	21	17	14	10
Thermal conductivity (W/m°C)	5	9	12	85
Temperature limit [(1)], in air (°C)	315	315	315	455

Key: (1) The temperature limit is determined by the oxidation rate in air; (2) Depending on the processing, properties of carbon-graphites can be anisotropic due to the alignment of the graphite phase. Non-aligned phases give more isotropic characteristics.

Excluding reinforcement fibres and carbon-carbon composites, of the various types of carbons available, those considered for aerospace applications are:

- carbon-graphites, which are used in certain load-bearing aerospace applications;
- carbon foams, which are under consideration for thermal management systems on spacecraft.

43.10.2.2 Carbon-Graphites

The properties of some commercially-available grades of carbon-graphite materials are summarised in Table 43.10.2, Ref. [43-19], [43-20].

Table 43.10-2 – Technical ceramics: carbon-graphite grades – typical properties

Property	Examples of commercially-available carbon-graphite grades †							
	MAM&T P-5N	MAM&T CJPS	MAM&T P-03	MAM&T P-7454	EK 2000	EK 2209	EK 3205	EK 24
Composition (%, typical)	-	-	-	-	-	-	-	-
Additions (1)	-	-	-	-	Resin	None	Antimony	None
Grain size (μm)		-	76		-	-	-	-
Bulk density (fired) (×10³ kg/m³)	1850	1770	1800	2000	1820	1770	2250	1700
Apparent porosity (%)	10	-	8		2.5 (2)	2.5 (2)	2.5 (2)	8.0 (2)
Hardness (Shore Scleroscope)	90	65	75	90	-	-	-	-
Hardness (Rockwell B, HR5/100)	-	-	-	-	110	110	115	105
Compressive strength (MPa)	275.8	179.3	137.9	138	200	200	260	180
Transverse strength (MPa)	86.2	55.1	55.2	48	-	-	-	-
Flexural strength (MPa)	-	-	-	-	75	65	95	60
Young's modulus (GPa)	20.7	13.8	12.4	-	27.0	26.0	30.0	18.0
Fracture toughness (MPa m$^{1/2}$)	-	-	-	-	-	-	-	-
Thermal conductivity (WmK, 20°C)	-	-	71	-	14	14	20	14
Coefficient of thermal expansion, 20°C to 200°C (×10^{-6} /°C)	-	-	-	-	5.0	3.8	4.0	4.1
Applications (examples)	Seal components in cryogenic pumps in Space Shuttle main engines	Rudder speed brake assembly on Space Shuttle	Rocket nozzle Mars Lander (initial)	Environmental recirculating pump on space station ISS (system to remove CO_2)	Highly loaded seals and bearings	Highly loaded seals and bearings	Highly loaded seals and bearings	Dry running (3)
Key † MAM&T grades from Morgan Advanced Materials & Technology and used in space applications (stated); Selected carbon-graphite EK grades from SGL Carbon, for high-load applications; (1) Impregnated; (2) Open porosity, %; (3) Several different 'dry running' grades available.								

Carbon-Graphite materials are used mainly in sealing and bearing applications, where material selection involves consideration of loads, speeds, temperatures, mating materials, cost constraints and projected volumes.

Many carbon-based material types are available, with various modifications for particular designs and environments, e.g. for high temperatures or corrosive materials. Depending on the particular grade, carbon-graphites can be manufactured in a variety of shapes and sizes, e.g. cylinders up to 530 mm diameter × 200 mm high; plates up to 600 mm × 325 mm × 130 mm thick, typically.

Some grades have safety approvals for use within specific environments, e.g. oxidising conditions, food contact, potable water.

43.10.2.3 Foams

Porous forms of carbon include flexible, exfoliated graphite, [See: Graphite] and rigid foams.

Conducted as part of an on-going AFRL program in the USA, the potential uses of carbon foams in spacecraft thermal management systems is under evaluation by Northrop Grumman Space, Ref. [43-23].

Spacecraft thermal management systems comprising of light-weight, thermally-conductive materials are considered necessary to accommodate high heat dissipation in military and communications payloads. These applications are increasingly using densely-packed electronics for extended mission lives, Ref. [43-23].

Optimised designs for spacecraft radiators using carbon foams can potentially simplify radiator structures. Reducing the need for doublers and heat pipes to dissipate the heat generated by the electronics whilst meeting, or lowering, the mass budget for the thermal management system.

Properties of the carbon foams under consideration by Northrop are given in Table 43.10.3, Ref. [43-23], [43-24]. All the carbon foams cited are of US origin.

The properties of carbon foams are anisotropic.

Table 43.10-3 – Technical ceramics: carbon foams – typical properties

Property [5]	Examples of development carbon-foams †					
	TRL-Touchstone Research Laboratory		MER Corporation	Poco Graphite [1, 2]		Ultramet
	Grade [4] not stated	**CFOAM™ 25** [3]	Grade [4] not stated	Grade [4] not stated	**POCO™ HTC**	Grade [4] not stated
Composition	Coal-based product		Pitch precursor	Pitch precursor		CVD pyrolytic graphite on reticulated vitreous carbon
Bulk density (kg/m³)	160 to 800	400	160 to 620	500	900	230 to 610
Total porosity (%)	-	-	-	-	61	-
Open porosity (% of total)	-	-	-	-	95	-
Pore size (μm, typical dia.)	-	-	-	-	350	-
Compressive strength (MPa)	-	>15	5.5	2.1	5.9	-
Compressive modulus (GPa)	-	0.84	-	-	-	-
Young's modulus (GPa)	0.07 to 0.7	0.84	0.18 to 5.0	1.4	-	2.3 to 10.2
Thermal conductivity (WmK) in-plane out-of-plane	0.1 to 100 - -	25 - -	5 to 210 - -	150 - -	- 245 70	100 to 125 - -
CTE, (ppm/K) in-plane (50°C to 150°C) (600°C to 800°C) out-of-plane (50°C to 150°C) (600°C to 800°C)	1.5 to 2 - - - -	5.8 - - - -	- - - - -	1.5 to 2 - - - -	- 1.02 3.26 −1.07 1.31	1.5 to 2 - - - -
Comments	Large sizes available Panel sizes (approx.): 1.1 m × 0.6 m × 25 mm thick.	Mechanical properties can be modified to meet particular applications; Grade 25 is promoted for composite tooling	High thermal conductivity; high compressive strength Panel sizes (approx.): 250 mm × 250 mm × 25 mm thick.	High thermal conductivity; producible product Panel sizes (approx.): 250 mm × 250 mm × 37 mm thick.	Thermal management material; high thermal conductivity; laminated to form panels and enclosures; RF shielding. Panel sizes (approx.): 300 mm × 300 mm × 37 mm thick.	Uniform microstructure; high stiffness Panel sizes (approx.): 400 mm × 350 mm × 12 mm thick.
Key	† All sources cited are of US origin; POCO FOAM and POCO HTM are trademarks of Poco Graphite Inc. (USA); CFOAM is a trademark of TRL – Touchstone Research Laboratory (USA). (1) Licensed manufacturer of thermally conductive graphite foam developed by Oak Ridge National Labs. (USA); (2) POCO FOAM grade: Outgassing (ASTM E-595): TML total mass loss = 0.031%; VCM volatile condensible matter = 0.017%; (3) CFOAM fire resistance 'pass' ISO, MIL and ASTM standards; (4) Range of properties for foam materials from source; Ref. [43-23]; (5) Properties are anisotropic					

43.11 Silicon oxide: Silica

43.11.1 Typical characteristics

Silica-based materials are usually grouped as either:

- Fused silica, which is used for its thermal shock resistance.
- Fused quartz (vitreous silica), which is used for optical applications.

Both types of materials possess low thermal conductivity and high electrical resistivity and are virtually inert to the majority of aggressive chemicals over a wide range of temperatures. The main exceptions are:

- HF (hydrofluoric acid), which reacts at all temperatures.
- Phosphoric acid above 200°C.
- Caustic oxides react slowly at ambient temperatures but increases with temperature.
- Some liquid metals.
- Some metal oxides above 800°C.

Silica-based materials are available as:

- castables and slips,
- various standard shapes and forms (tubes, plates, discs), e.g. plate sizes up to 1.2m × 0.6m × 10mm thick; tubes 0.8 m × 1.5 m long.
- components.
- fibrous forms (insulation products), [See also: Chapter 99].
- powders or microspheres, which are used as fillers and additives for other materials, e.g. potting compounds.
- nano materials, which are under evaluation for many varied applications, e.g. fine particulate additives in the polymer matrix phase of composites, Ref. [43-55].

Uses of silica-based materials are wide ranging, from components in materials processing industries and optical applications to radomes and nose cones, e.g. Patriot missile system.

For missile nose cones, the design drivers are transparency to various microwave energies, coupled with mechanical strength to enable the missile system to withstand erosion and the large temperature excursions while flying at hyper-velocities through the atmosphere, Ref. [43-14].

The Space Shuttle Orbiter uses silica for external windows and within the TPS, [See also: 71.5].

43.11.2 Typical properties

Table 43.11.1 summarises the properties for some examples of commercially-available silica materials, Ref. [43-13], [43-14].

Table 43.11-1 – Technical ceramics: silica – typical properties

Property	Examples of commercially-available silica grades †				
	VITREOSIL™	SPECTROSIL™	THERMO-SIL™ HS (3)	THERMO-SIL™ UHS (3)	THERMO-SIL ISOMOLDED™ (3)
Silica content (%, typical)	99.95	99.9999 99.8(1)	-	-	-
Grain size (µm)	-	-	-	-	-
Bulk density (fired) ($\times10^3$ kg/m^3)	2150	2210 (2) 2150	2005	1970	2020
Porosity apparent (%)	-		9	10	7.5
Vickers (GPa, Hv0.5kg)	-		-	-	-
Rockwell (R45N)	-		-	-	-
Compressive strength (MPa)	2000		50	207	75.9
Flexural strength (MPa)	-		18.6	56.2	11.4
Young's modulus (GPa)	-	74	37.2	38.2	34.5
Fracture toughness (MPam$^{1/2}$)	-		-	-	-
Thermal conductivity (WmK, 20°C)	-	2	0.84	0.75	0.62
Coefficient of thermal expansion, 20°C to 1000°C ($\times10^{-6}$ /°C)	0.54	0.54	0.6	0.5	0.8
Applications (examples)	Materials processing industries, inc. semiconductors; electrical insulators.	Materials processing industries, inc. semiconductors; electrical insulators.	High-precision shapes (materials process industry); corrosive environments.	Thin-walled detailed shapes by shell casting	Shapes (size and mass); Materials process industry (high-temperature precision platens, load-bearing support parts)

Key: † VITREOSIL and SPECTROSIL are tradenames of St. Gobain Quartz, THERMO-SIL and ISOMOLDED are tradenames of Ceradyne Inc.; (1) Range of purities: High-purity transparent grade, purities all exceed 99%, but vary with opacity; (2) Transparent grade; (3) Average values;

43.12 Silicon carbide

43.12.1 Typical characteristics

43.12.1.1 General

The general characteristics of densified silicon carbide include:

- Low density,
- Hardness and wear resistance,
- High strength,
- Low thermal expansion,
- High thermal conductivity,
- Useful electronic properties,
- Chemical resistance; good oxidation and hot-corrosion resistance, depending on purity and processing route,
- Thermal Shock resistance,
- High temperature strength, depending on the processing route,
- Thermal stability to very high temperatures (2200°C to 2800°C, depending on the grade).

The performance of commercially-available grades of silicon carbide is strongly linked to their processing routes, which determine the microstructure, e.g. grain size, residual porosity levels and residual free elements. This, in turn, dictates their mechanical characteristics and high-temperature environmental resistance.

43.12.1.2 Processing

The processing routes use various techniques to compact and densify powders or deposit material from a vapour-phase, Ref. [43-1]:

- Hot pressing of pure silicon carbide to produce high density materials needs both a high temperature and high pressure, e.g. above 2000°C at 2 GPa. Additions of alumina or boron carbide can reduce the pressure necessary.
- Pressureless sintering (moderate temperatures, no pressure) uses fine-particle-size silicon carbide powders with additions of other materials to act as sintering aids.
- Reaction bonded or sintered materials vary in composition and process details. These are usually fully dense, but can contain some free silicon. Applications include wear-parts and high-temperature precision components. To improve the high-temperature capability of reaction-bonded materials, a nitriding process to convert any free silicon to silicon nitride results in materials known as 'nitrogen-bonded silicon carbide', Ref. [43-47].
- Silicon-infiltrated silicon carbide is a process where silicon carbide powder is mixed with carbon or graphite then compacted into a green shape. Silicon, as either a liquid or vapour phase, infiltrates the green shape and reacts with the carbon to form silicon carbide which bonds the silicon carbide particles together. Usually around 10% residual, unreacted, silicon remains in the densified item. This affects the high-temperature performance, i.e. the melting point of pure silicon is about 1400°C. A similar process is used to manufacture Cesic® in which a

carbon felt rather than carbon powder is used to react with the liquid silicon to form silicon carbide.

- Chemical vapour processes, either CVD deposition or CVI infiltration, rely on the reduction of silicon- and carbon-containing chemicals at high temperatures. These techniques produce high-purity silicon carbide with high oxidation resistance. Deposition can be on a substrate to form a protective coating, e.g. oxidation protection of porous or non-porous substrate materials; whereas infiltration can be used to densify green shapes by producing silicon carbide within interstices. CVI processes are used to create silicon carbide-based ceramic matrix composites, [See: 88.22].

43.12.2 Typical properties

Table 43.12.1 summarises the properties of some commercially-available silicon carbide grades produced by different process routes, [43-12], [43-14], [43-27].

Of these materials, SiC 100™, Cesic® and SiC 54 have been extensively evaluated for space applications, [See also: 43.18].

Tubes of sintered SiC (Hexalloy) and silicon infiltrated carbon felt are under evaluation for the GAIA optical bench truss structure, Ref. [43-109].

Table 43.12-1 – Technical ceramics: silicon carbides – comparison of materials properties produced by different process routes

Property	Examples of commercially-available silicon carbide grades †								
	PUREBIDE™ (6)	CERASIC™	PUREBIDE™ (6)	CERALLOY™ 146-IS	SiC 100™	Cesic®	SiC 54S®	Performance SiC™	CERATREX™ SC-3P
Processing method	Self-sintered	Pressureless sintering	Reaction bonded	Hot pressed	Isostatic pressed + sintered	Silicon infiltration	Isostatic pressed + Sintered	CVD (1)	CVD (1)
Silicon carbide content (%, typ.)	-	-	-	99.3	0% free Si	-	-	99.9995	99.9995
Grain size (μm)	-	-	-	-	5	-	-	2 - 10	-
Bulk density (kg/m^3)	2650 3100	3150	2900 3100	3150	>3100	2650	3150	3210	3200
Porosity (apparent, %)	-	-	-	-	<3.5	-	1.5	-	-
Porosity (open, %)					0		0		
Hardness (Vickers)	2000 3000	-	2200 3000	-	-	-	-	-	-
Vickers (GPa, Hv0.5kg)	-	-	-	-	2.2	-	2.3	2.8	-
Vickers (GPa, Hv0.3kg)	-	-	-	2.3	-	-	-		2.8
Rockwell (R45N)	-	-	-	-	-	-	-	-	-
Compressive strength (MPa)	483 3790	-	552 2751	-	3000	-	2200	-	-
Flexural strength, (MPa): 20°C 1300°C 1450°C	103 449 - -	450 - 450	- - -	375 - -	450 450 450	>130 - -	365 (2) - -	450 (2) 560 (2) -	375 - -
Young's modulus (GPa): 20°C 1300°C	124 414 -	420 -	152 - 345 -	440 -	420 -	225 -	415	450 (2) 430 (2)	440 -
Fracture toughness ($MPam^{1/2}$)	-	3.5	-	2.5	3.5	>4.6	4	2.94 (3)	3.1
Thermal conductivity (WmK, 20°C)	-	170	-	115	180	160	110	250	200
CTE, ($\times 10^{-6}$ /°C)	-	4.5	-	4.8 (5)	4.6 (5)	0.003 (7)	3.8 (8) 4.4 (9)	4.5 (4)	4.5 (5)
Applications (examples)	Abrasive; corrosive, e.g. high-pressure, high—temperature process chemicals-	Wear parts; corrosion resistance, e.g. automotive parts, material process industries	High friction; high temperature, e.g. fluid handling, high-speed rotation parts, aerospace.	Corrosion resistance; wear resistance	Space-destined applications: telescope and optical system structures and supports. e.g. ARAGO, Herschel, OSIRIS, ROCSAT, VLT-NAOS, SPICA, GAIA, NIRSpec (JWST).	Space-destined applications: telescope and optical system structures and supports. e.g. NIRSpec (JWST).	Space and avionics applications. Optical and electro-optical systems, e.g. NIRSpec (JWST).	Corrosion resistance; semiconductor material process industries	Corrosion resistance; wear resistance; near-net shape to 400 mm OD.

Key: † PUREBIDE & HALSIC: Morgan AM&T; Performance SiC: Morgan Advanced Ceramics (CVD Div.); CERASIC: Toshiba Ceramics Co.; CERATREX & CERALLOY: Ceradyne; SiC 100: Boostec Industries (F); Cesic® ECM (D); SiC54S: Bettini (I); (1) Chemical vapour deposition; (2) 4-point bend test; (3) microindentation test; (4) 20°C to 950°C;(5) 20°C to 1000°C; (6) Range of properties for various commercial grades; (7) 20K to 100K; (8) 20°C to 400°C; (9) 20°C to 800°C.

43.12.3 Materials for space applications

43.12.3.1 General

The majority of silicon carbide materials are used for their thermo-mechanical properties. Applications, depending on the grade, range from refractories to high-performance engineering materials and CMC ceramic matrix composites, [See: Chapter 52].

43.12.3.2 Monolithic ceramics

In addition to high temperature applications, the low thermal expansion coupled with high strength and stiffness make some silicon carbide grades of interest for dimensionally-stable structures, such as in ground- and space-based optical structures and systems. Some grades of silicon carbide have been extensively evaluated for such applications, e.g.

- Cesic®
- SiC 54S®
- SiC 100®

43.12.3.3 Foams

Whilst the majority of commercially-available silicon carbide grades are densified materials (low porosity), some specialist suppliers produce deliberately, open-pore materials, Ref. [43-28], [43-39], [43-59], [43-60], e.g.:

- Ultramet (USA).
- Schafer Corp (USA)
- Duocel™ from ERG Materials and Aerospace Corporation (USA)
- SiC LigaFill™ foams from Fraunhofer IKTS (D)

Ceramic foams were originally developed as filters for molten metal about 40-years ago. During processing, an open cell structure forms that resembles that of polyurethane foam but in which the pore walls (known as ligaments) are hollow. These hollow walls are then infiltrated to produce solid ligaments that provide a substantial increase in strength, Ref. [43-39].

SiC foams are now considered to have potential uses in space structures as possible replacement materials for glass-ceramics or beryllium and as a competitor to conventional silicon carbide used for some large, lightweight mirrors and structures, Ref. [43-29], [43-39], [43-57], [43-58], [43-59], [43-61] and as cores for sandwich panels, [See also: Chapter 26].

The majority pf SiC foams are of USA origin. A summary of properties for Duocel™ of interest for sandwich panel cores is provided, [See: Table 26.3.7].

Within Europe, Fraunhofer IKTS (D) has developed SiC LigaFill™ foams, Ref. [43-39]. High-strength LigaFill™ foams contain between 70% and 90% pores, with cell sizes in the range of 10 to 60 pores per inch (4 to 25 pores per centimetre, i.e. pore diameters from 0.4 to 2.5 mm), typically. Fracture loads, determined by indentation tests, are in the range of 900N to 7500N, depending on the foam structure. High-temperature resistance is to about 1300°C, Ref. [43-39]. Sandwich panels, comprising of silicon carbide skins (mat or plates) can be assembled to foam cores by a paste technique, Ref. [43-39].

Another product Ceranet™, also developed by Fraunhofer IKTS, is not foam but a lightweight silicon carbide material with a net-like structure.

43.12.4 Cesic®

43.12.4.1 Introduction

Cesic® is high-performance technical ceramic material from ECM (Munich, D). It contains a mixture of silicon carbide, silicon and carbon phases after processing, Ref. [43-57]. The material was evaluated for the development of the optical bench of the NIRSpec instrument on the JWST James Web Space Telescope, [See: 43.18] and also mirrors for NIRSpec, Ref. [43-58].

43.12.4.2 Material composition

Cesic® uses a felt of short, chopped randomly-orientated fibres as a precursor material. During the high-temperature processing stage, the carbon reacts with liquid silicon to form silicon carbide. Cesic® can be considered a composite rather than a ‘conventional’ technical ceramic because of the initial fibrous content and the mixture of resulting phases. The material properties of Cesic® are isotropic, which makes it more like a ‘conventional’ technical ceramic than a composite that has anisotropic characteristics conferred by a continuous fibre-reinforcement. The processing stages, which determine the final material composition and structure, also share similarities with those used in the manufacture of continuous fibre-reinforced CMC shaped-components, [See: Chapter 52].

43.12.4.3 Material processing sequence

The basic manufacturing process sequence for Cesic® is summarised in Figure 43.12.1.

CARBON BLANK
Carbon felt infiltrated with phenolic resin.
Moulded under pressure to form a blank.
Vacuum heat-treatment.
Carbon blank size: 1100 mm × 1100 mm × 80 mm

⇩

ASSEMBLY OF BLANKS
(bonding)

⇩

MACHINING OF BLANKS
(CNC milling and 'pre-holes')

⇩

INSPECTION
(green body)

⇩

SILICON INFILTRATION
[See: Figure 43.12.2]
1600°C process temperature
Vacuum
Transformation of Si to SiC by reaction with carbon (matrix and fibres)
Capacity: 2.4 m diameter; three 1.2 m levels

⇩

EXCESS SILICON REMOVAL
(sand-blasting)

⇩

INSPECTION
(cracks; geometry)

⇩

FINAL MACHINING
EDM technique
Grinding (diamond tools)

⇩

FINAL INSPECTION
Non-destructive test (dye penetrant; ultrasonic)
Geometry
Mass

Figure 43.12-1 – Cesic®: Basic processing sequence

The Cesic® process is known as 'direct-to-shape' because the carbon green-body shape is produced close to the final size and shape of the final structure.

'Direct-to-shape' implies better dimensional control than other processes described as 'near-net shape'.

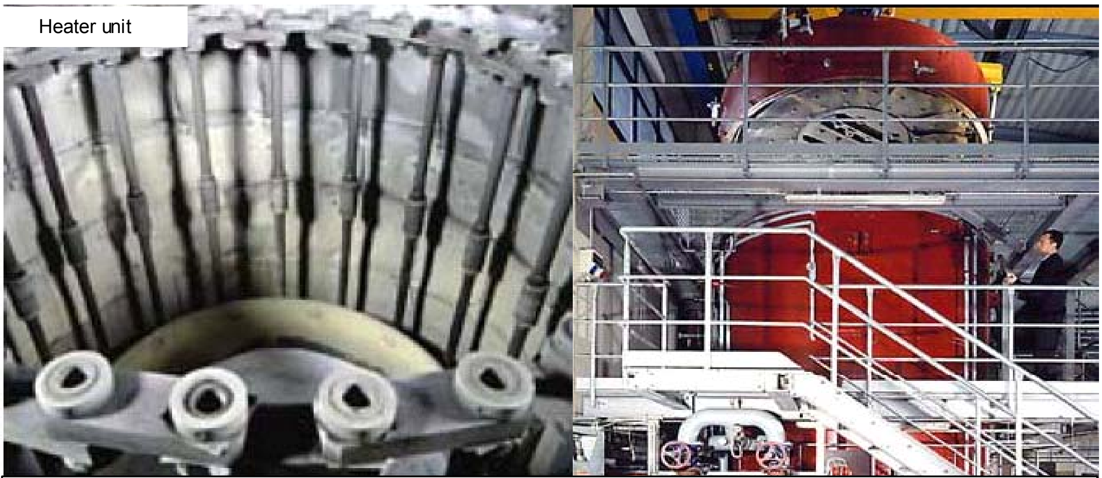
During the infiltration process, the transformation from carbon to silicon carbide that occurs is a 'solid-to-solid' reaction; hence no deformation occurs due to effects such as gravity. Whereas some ceramics, processed by different methods, exhibit large dimensional changes during manufacture, Cesic® undergoes a low, controlled shrinkage of only 0.2%. This enables close-tolerance structural parts to be produced and reduces the need for machining.

Depending on the particular structure being made, additional processes can be used, Ref. [43-57], e.g.

- mirrors, which have a surface preparation step prior to coating and a polishing stage after coating. The optical quality is also established during the final inspection.
- small components do not always need assembly of multiple carbon blanks; maximum blank thickness 80mm.
- large structures can be produced as sub-assemblies, which are then joined after silicon infiltration, final machining and inspection steps.

43.12.4.4 Process equipment

The infiltration furnace, installed at ECM in Germany, is shown in Figure 43.12-2. This equipment enables infiltration of components up to 2.4 m diameter.



Cesic ® silicon infiltration facilities at ECM (Munich, Germany) capable of processing up to 2.4 m diameter structures.

Figure 43.12-2 – Cesic®: Silicon-infiltration processing equipment

43.12.4.5 Machining

EDM and wire erosion machining is possible because Cesic® is electrically conductive. The EDM technique offers a number of advantages over grinding, e.g. fast and accurate, so less expensive. For large areas, the tolerances achievable are flatness <10µm; position ±20µm. Combining wire erosion machining (preliminary) with EDM (finishing) achieves tolerances of ±10µm for an 7mm hole.

43.12.4.6 Material characteristics

The material properties were determined during the design-development of an optical bench; a dimensionally-stable structure for the JWST/NIRSpec programme, [See: 43.18]. Properties, related to its potential use for mirrors for NIRSpec, were also established, Ref. [43-58].

The material properties are isotropic and are not affected by the processing conditions, e.g. infiltration runs or direction, and are also independent of surface condition.

The properties are reproducible between different production runs using different batches of green-body carbon blocks; as demonstrated by tests on samples taken from different material batches.

Isotropy was demonstrated by samples taken from different orientations, i.e. samples denoted as x-, y- and z-directions.

The tests conducted included, Ref. [43-57]:

- coefficient of thermal expansion, from RT to 20K
- 4-point bending to an ASTM standard.
- Biaxial test, at RT and low temperature (liquid nitrogen).

43.12.4.7 Mechanical properties

Table 43.12.2 summarises the mechanical properties of Cesic®. The 4-point bending tests use two different sample sizes; both of which are relatively large compared with those used often used for ceramic material test campaigns.

Table 43.12-2 – Cesic®: Mechanical properties

Property (1)	Value	Comment
Density (kg/m^3)	2650	Ref. [43-57]
Modulus, E (GPa) (2)	225	
Specific bending stiffness, E/ρ (MPa)	>13.7	
Strength (MPa)	>130	
Fracture toughness, K_{IC}	>4.6	
Bending strength, at RT (MPa): 'large sample':		ASTMC1341 (150mm × 30 mm × 3mm); 97 samples tested, Ref. [43-57].
Mean	111.1	
Maximum	152.3	
Minimum	80.7	
SD	15.7	
Weibull	11	
Bending strength, at RT (MPa): 'small sample':		ASTMC1341 (80mm × 30 mm × 3mm); 21 samples tested, Ref. [43-57].
Mean	148.9	
Maximum	171.6	
Minimum	128.1	
SD	13.0	
Weibull	13.6	
Directionality (bending strength):		4-point bending strength, 'small sample', Ref. [43-57].
x-direction (MPa)	147.4	
y-direction (MPa)	151.3	
z-direction (MPa)	146.9	
Bi-axial bending strength, at RT, (MPa)	164.2	Ref. [43-57]
SD	22.2	
Bi-axial bending strength, at LN2 temp, (MPa)	162.9	Preliminary tests, Ref. [43-57].
SD	20.4	
Key: (1) SD – standard deviation; (2) ExInertia/M ratio higher than other advanced materials, d = 2.65		

43.12.4.8 Thermal properties

Table 43.12.3 summarises the temperature-related properties of Cesic®. The CTE of Cesic® is near-zero over a wide temperature range; down to cryogenic levels.

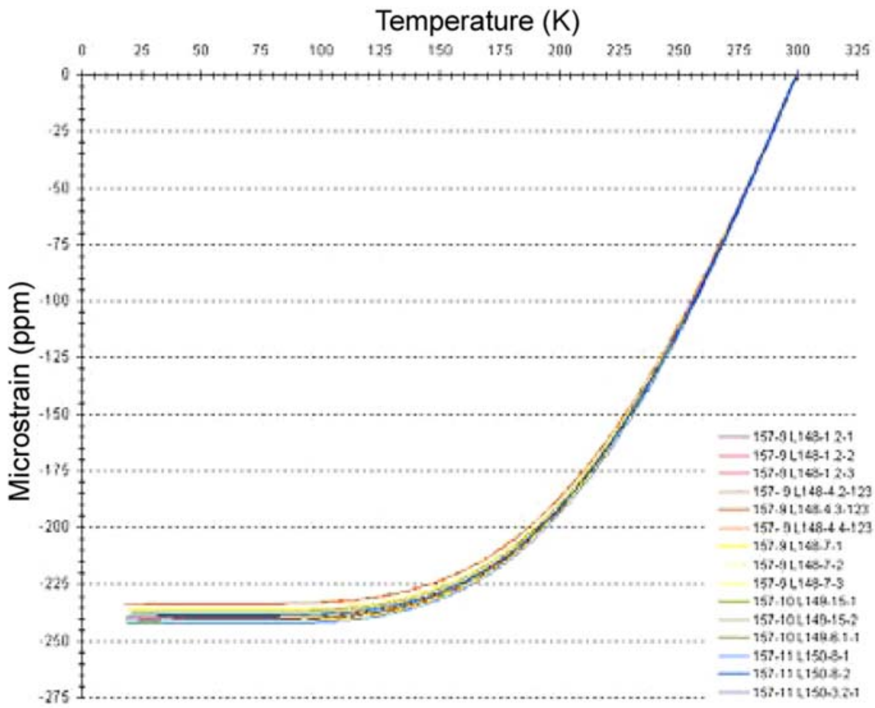
Table 43.12-3 – Cesic®: Thermal properties

Property [1]	**Value**	**Comment**
Coefficient of thermal expansion, ($\times 10^{-6}$/°C): from 20K to 100K	<0.003	Michelson interferometry technique, 15 samples tested
Thermal conductivity (W/mK): at 300K at 40K	 160 35	

The results of CTE tests on materials produced from three different batches, i.e. infiltration of different batches of carbon, and tested in different directions are shown in Figure 43.12.3, Ref. [43-57]. The average value determined from the curves is 238.6μm/m with a maximum variation of 5.3μm/m at 20K.

The samples for z-direction tests were joined prior to silicon infiltration because the thickness limit of the carbon blank is 80mm (test sample size: 250mm × 30mm × 10mm).

Given that the CTE in the z-direction are the same as those in the other directions, where samples did not contain joints, this shows that the joining method developed by ECM does not affect the material expansion or contraction characteristics.



CTE measurement on 15 samples from 3 batches of materials tested in *x*-, *y*- and *z*-directions, where z-direction samples contained joints made prior to silicon infiltration.

Figure 43.12-3 – Cesic®: Coefficient of thermal expansion

43.12.4.9 Applications: Panels

The combination of the material characteristics and the 'near-net-shape' manufacturing process of Cesic® enable both structural shapes and integrated, complex-shaped assemblies to be produced.

For example: I-beams for rigidity, large thin webs (less than 1.5 mm thick) and plates, along with open-backed structures, flat or curved panels with high dimensional surface tolerances.

Such structural designs are typical of those used for dimensionally-stable structures, such as mirrors and support structures, optical benches or other similar equipment supports.

Figure 43.12.4 shows an example of a 1m2 integrally-stiffened panel made of Cesic®.

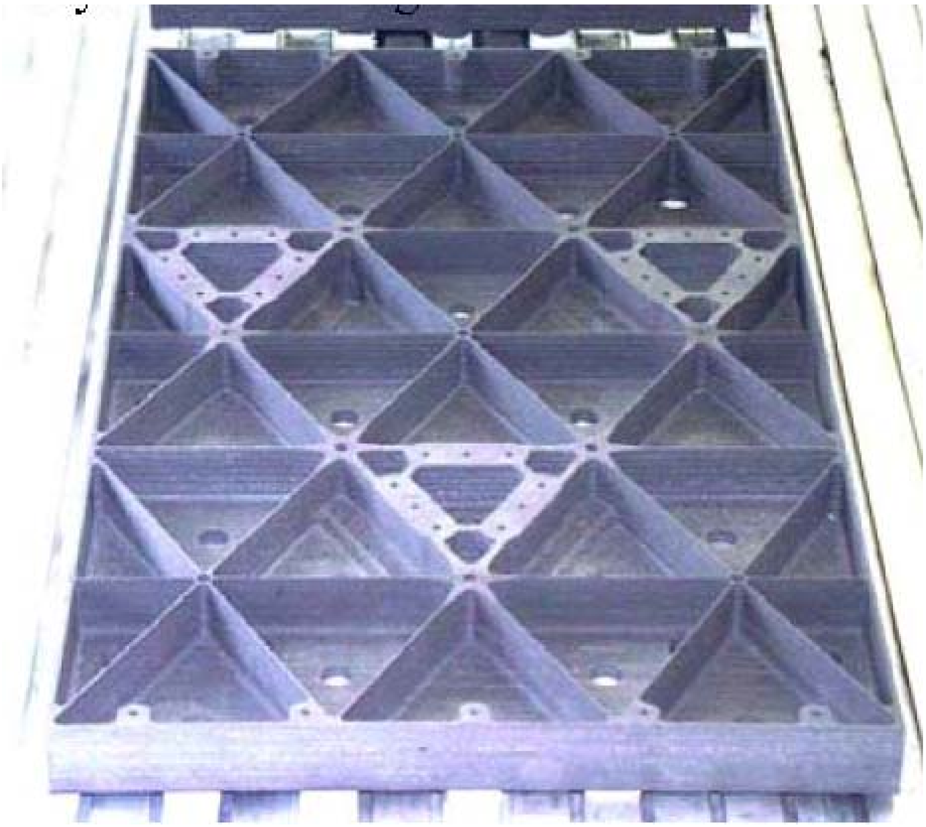


Figure 43.12-4 – Cesic® ceramic: Example of integrally-stiffened panel

43.12.4.10 Applications: Mirrors

An ESA-funded study compared Cesic® with Bettini SiC-54S for the types of mirrors envisaged for the NIRSpec programme, [See also: SiC-54S]. Not only the materials were evaluated, but optical coatings, manufacturing of test mirrors and performance at cryogenic temperatures were also investigated, Ref. [43-58].

43.12.4.11 Applications: Optical bench

In the design-development of NIRSpec, an optical bench demonstrator component measuring 1045mm × 600mm × 100mm was manufactured successfully, [See: 43.18]

43.12.5 SiC 54S®

43.12.5.1 Introduction

Produced by Bettini in Italy, SiC 54® has been evaluated for dimensionally-stable space structures.

An ESA-funded study compared Bettini SiC-54 with ECM Cesic® with for the types of mirrors envisaged for the NIRSpec programme, Ref. [43-58].

43.12.5.2 Material composition

The raw powder is a spray dried α-SiC (Norton standard product) containing >97.7% SiC with <0.04% free Si along with other materials and trace elements. Each batch of raw powder is subjected to incoming quality control to determine chemical and thermal analyses, humidity and physical features of powder, e.g. particle size distribution, surface area.

43.12.5.3 Material processing sequence

Figure 43.12.5 summarises the overall manufacturing process for SiC54S from incoming raw material to final inspection of finished item, Ref. [43-63]. Each step in the process is accompanied by rigorous quality control procedures.

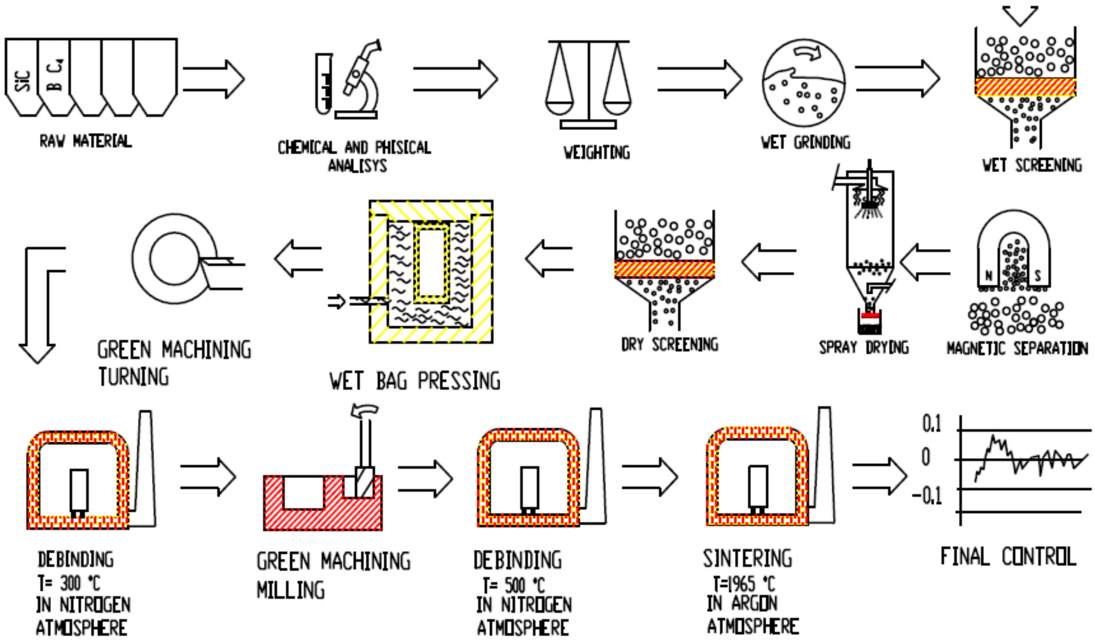


Figure 43.12-5 – SiC 54S® ceramic: Process sequence

The basic process was used to manufacture test mirrors for the NIRSpec programme. To improve the surface polishing capability, a coating was applied to the surface to meet NIRSpec micro-roughness demands, Ref. [43-58].

Snecma's proprietary isothermal chemical vapour infiltration (ICVI-SiC) coating process was used to deposit a dense, homogeneous SiC layer on the SiC54 substrates. The process is conducted at a lower temperature compared with other chemical vapour processes, Ref. [43-58].

43.12.5.4 Process equipment

Compaction of the prepared powder to form a green shape is accomplished by wet-bag, cold isostatic pressing. The green shape is densified by sintering at 1965°C in an inert (argon) atmosphere. The sintering process is accompanied by a witness sample for quality control purposes.

43.12.5.5 Machining

Prior to densification by sintering, machining is carried out on the green body either after the cold isostatic pressing step (turning) or after the debinding step (milling).

43.12.5.6 Material characteristics

The good stability, low shrinkage and low residual porosity of SiC54S® make it of interest for optical applications.

43.12.5.7 Mechanical properties

The mechanical properties of SiC54S are summarised in Table 43.12.4. The values stated are a combination of manufacturers' data and those determined during the NIRSpec mirror study, Ref. [43-58], [43-63].

Table 43.12-4 – SiC 54S®: Mechanical properties

Property	Test standard	Value
Porosity:		
Open (%)		0
Closed (%)		1.5
Bulk density (g/cm^3)	EN 623-2	≥ 3.15
Flexural strength, 4-point, (MPa)	EN 843-1	365
Batch 1	EN 843-1	369.5 ± 61.4
Batch 2	EN 843-1	364.1 ± 64.7
P_f (1) = 63.21%	-	390.28
Bending strength (4-point)		225 (4)
Specific strength, σ/ρ	-	71
Allowable strength, σ_{adm}	-	82 (3)
Compressive strength (MPa)	DIN 51067T1	2200
Young's modulus (GPa):	ENV 843-2	415 ± 1.7 (2)
Specific stiffness, E/ρ	-	132
Allowable modulus, E_{adm}	-	400 (3)
Hardness, Vickers HV 0.5 (GPa)	ENV 843-4	2.3
Fracture toughness, K_{IC} ($MPa\ m^{1/2}$)	DIN 51109	4
Weibull modulus	ENV 843-5	7
Poisson's ratio	ENV 843-2	0.15 ± 0.001 (2)
Allowable Poisson's ratio, ν_{adm}	-	0.16 (3)

Key: (1) P_f probability of failure; (2) Based on 3 samples; (3) Mirror design value; (4) Corresponds to 63% P_f probability of failure on increased load area $3200 mm^2$.

43.12.5.8 Thermal properties

The thermal properties of SiC54S are summarised in Table 43.12.5. The values stated are a combination of manufacturers' data and those determined during the NIRSpec mirror study, Ref. [43-58], [43-62].

Table 43.12-5 – SiC 54S®: Thermal properties

Property	Test standard	Value
Thermal conductivity (W/mK):		
20°C	EN 821-2	110
300K	-	130
100K	-	48
Coefficient of thermal expansion (10^{-6}/°C):	EN 821-1	
20°C to 400°C	EN 821-1	3.8
20°C to 800°C	EN 821-1	4.4
Coefficient of thermal expansion (ppm/K):	(1)	
RT	(1)	2.2
RT to 35K	(1)	1.0
Heat capacity (J/kg K):	(2)	
300K	(2)	660
200K	(2)	392
100K	(2)	106
35K	(2)	2
Max. service temperature (°C)	-	1300

Key: (1) Measured by Michelson Interferometry technique (Galileo Avionica, Italy); (2) Special test fixture for measuring thermal capacity between 4K and 293K.

43.12.5.9 Applications: Mirrors

Figure 43.12.6 shows the SiC54S–ICVI SiC coated mirror prototype design to meet the NIRSpec design requirements, Ref. [43-58]:

- First eigenfrequency: >300 Hz
- Mass: <1.5 kg, including rear mount collar
- Design load: ≈ 50 g (30 g simultaneously along directions (+x, +y, +z) or (+x, +y, -z).
- Operating temperature: 35K, with qualifications 20K.

The mirror is 170mm diameter, with a rear integral mounting collar.

The mirror surface is spherical with a 600mm radius of curvature. The open back face of the mirror is flat with a depth at the edge of 30mm. The cells are equilateral triangles.

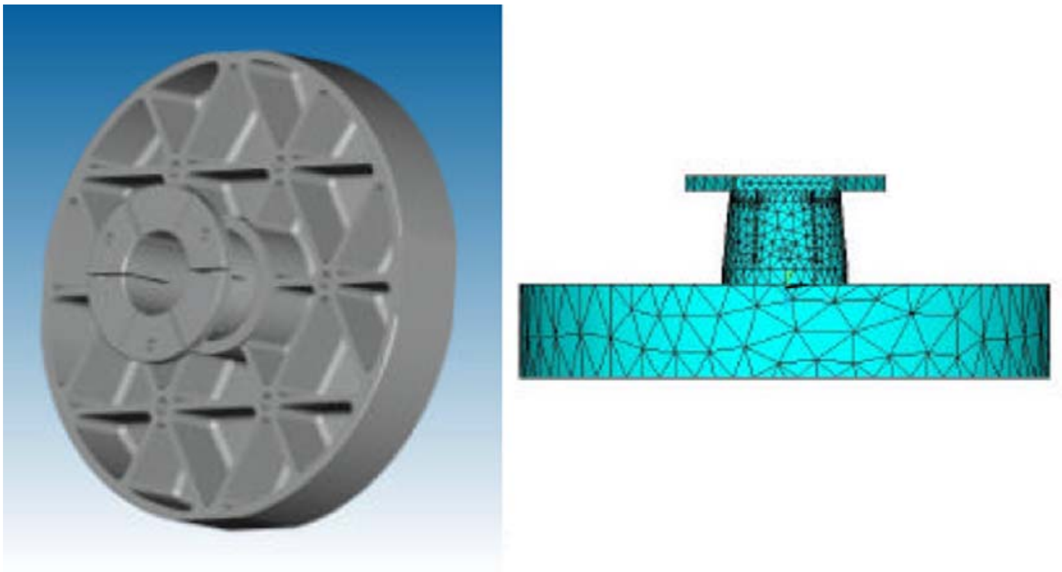


Figure 43.12-6 – SiC 54S®: NIRSpec prototype mirror

43.12.6 SiC-100®

43.12.6.1 General

As of August 2004, CoorsTek (USA) and Boostec (France) have a licensing agreement that covers a wide range of design, processing and manufacturing technologies associated with silicon carbide mirrors and structural systems for optical applications, Ref. [43-27]. This includes high-performance, space-based telescopes for earth observation, astronomy and defence. Some examples include:

- HERSCHEL (Demonstrator breadboard, Primary mirror)
- ROCSAT 2
- ROSETTA
- OWL
- FIRST
- ALADIN
- ARAGO
- NIRSpec – James Web Space Telescope (Optical bench, Optical assembly)
- VLT-NAOS
- GAIA

43.12.6.2 Source

SiC-100® has been developed by EADS Astrium SAS together with the manufacturing company Boostec S.A., France.

43.12.6.3 Chemical composition

SiC-100 is cold-isostatically pressed, sintered monolithic silicon carbide, Ref. [43-110]. The crystal structure of SiC-100 is the alpha form, with carbon atoms in an hexagonal grid and the silicon atoms occupy the tetrahedral sites of the structure. The material is poly-crystalline and fully isotropic. The mean grain size is about 5 µm.

Key advantages for the production of high-performance components or systems include:

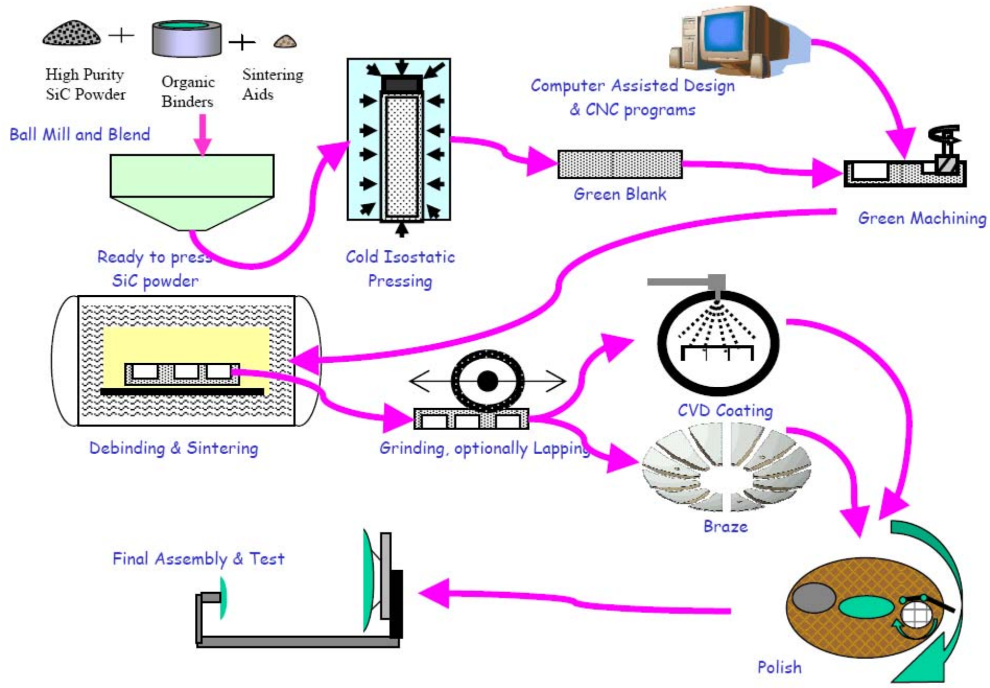
- Nearly pure SiC, no secondary phase.
- Isotropic physical properties.
- High mechanical strength and stiffness, insensitivity to mechanical fatigue.
- High thermal conductivity and low thermal strain.
- High stability.
- High resistance to aggressive environments.

43.12.6.4 Material processing sequence

The manufacturing of SiC-parts and components from powder is a well-established and qualified process, comprising:

- SiC powder preparation,
- Isostatic pressing,
- Green forming, also called green machining,
- Sintering,
- Grinding,
- Milling,
- Polishing,
- Inspection.

Figure 43.12.7 shows the step-by-step SiC-100 production process, Ref. [43-111].



Reproduced courtesy of EADS Astrium GmbH

Figure 43.12-7 – SiC-100®: Production process - schematic for Herschel primary mirror

43.12.6.5 SiC powder preparation

Sintered silicon carbide (SiC) raw material is produced by mixing high purity SiC with sintering aids and binder additives which aid production of large-scale products from this fine-grained material. Initially, the powder is ball milled to a precise sub-micron size distribution. The sintering aids comprise less than 1% by weight of the sintered material.

43.12.6.6 Isostatic pressing

The process involves filling a sealable container, such as a rubber bag, with SiC powder. The rubber bag is usually supported by a skeletal structure, typically a steel box, with perforations enabling access by the isostatic pressure media, generally water, to the full surface area of the bag. This assembly is placed into a pressure vessel where it is subjected to hydraulic pressure of about 2100 bars.

43.12.6.7 Green forming (green machining)

This process, using CNC machine tools, provides near-net shaping of non sintered SiC to its final design. Near-net shaping in the green state is generally used because SiC can be machined 20 to 30 times faster compared with the fired state. Features can be machined in the green state if their tolerances for linear shrinkage are in the range ± 0.2% to 1%. There are a few limitations on the green forming of SiC-100. The material in the pressed state is friable. It has the mechanical strength of chalk, being quite brittle and low in strength. Maintaining machine tools is crucial for the accurate machining of light-weight structures. Aspect ratios (height to thickness) approaching 80 to 1 have been achieved for light-weight ribs. Advanced machining techniques using high-speed (up to 20,000 rpm) 3 and 5 axis milling heads with harmonically-optimised tools have significantly reduced machining times.

43.12.6.8 Sintering

Sintering techniques have improved dramatically in the last 30 years. New furnace technology with computer controlled zones, optimised gas flow, and vacuum environments now enable sintering of very large and complex products. The challenge is to produce a uniform temperature during the sintering process that peaks in excess of 2100°C whilst maintaining an environment which removes binders and prevents oxygen exposure. SiC powder is kept under vacuum or an inert atmosphere during the entire sintering process to avoid degradation. The sintering process typically takes between 20 hours and 120 hours, depending on the size and complexity of the load.

During the process, there are 3 basic stages: binder burnout up to 500°C, densification (shrinkage of about 20%) up to 2100°C, then cool-down. The process needs to be controlled precisely. Not reaching the full sintering temperature for the required time can produce a part that is not fully dense. Exceeding the sintering conditions can cause excessive grain growth, which could be detrimental to material properties.

43.12.6.9 Grinding

Grinding of sintered SiC is similar to grinding of other types of ceramic materials. Abrasive diamond wheels are suitable tools for the purpose. Typically, grinding is done with a large flow of water-based coolant to prevent excessive heat. For optical applications, grinding is avoided in order to maintain the ultimate strength of the material measured in the 'as fired' surface condition. As with most ceramics, grinding can cause sub-surface damage that can lower the strength by 50% or more when compared with an as-fired or highly-polished surface. However, lapping and polishing operations can remove the damage caused by grinding and so recover the strength.

43.12.6.10 Milling

Design details such as interface attachment holes and recessed mounting surfaces are milled in accordance with precise geometric and dimensional requirements. CNC milling machines with up to 5 axes are generally used to perform these operations. Machining times for these operations are relatively long. Only diamond tooling with substantial coolant is effective for milling.

43.12.6.11 Polishing

Most optical polishing techniques can be applied to SiC optics. Conventional abrasive polishing with pads and pitch along with advanced techniques such as computer-controlled polishing, ion beam figuring and magneto-rheological finishing have been demonstrated. Diamond is the only abrasive medium that is effective in polishing. Optimised removal rates can be comparable with removal rates for more traditional optical substrate materials.

43.12.6.12 Inspection

Several in-process and final inspection steps are applied to SiC-100 optical projects. Although some special products are subject to design-specific testing and qualification requirements, the usual inspection steps are:

- Green body qualification: The raw material is qualified by using test coupons for density, pore distribution and strength.
- As-pressed blank: The blank is visually inspected for cracks and measured for dimensional accuracy.
- As-sintered part: The sintered part undergoes a dye penetrant inspection for cracks and porosity.

- Ground /machined parts: Dye penetrant inspection for cracks. Dimensional and geometrical inspection measurements.
- Proof testing: Final parts can undergo proof testing and/or vibration testing prior to integration into the optical subsystem.

Table 43.12.6 summarises the manufacturing size constraints (as of 2006). It gives the practical size limitations, determined by available process facilities, in terms of finished part size, i.e. after shrinkage and machining, Ref. [43-111].

Table 43.12-6 – SiC 100®: Manufacturing size constraints - practical size limitations for finished part

Process	Size limitations (m)	
Isostatic pressing	Cylindrical:	1.0
	Rectangular:	1.0 × 1.7
	Rectangular:	0.6 × 2.0
Green machining	Cylindrical:	1.5
	Rectangular:	1.0 × 3.0
Sintering	Rectangular:	1.4 × 1.8
Grinding	Cylindrical:	3.5
	Rectangular:	1.0 × 5.0
CVD SiC	Cylindrical:	1.5
Brazing	Cylindrical:	3.5

(Status 2006)

43.12.6.13 Material properties

Material properties are given in Table 43.12.7, Ref. [43-110]. Typical characteristics of SiC-100 are stated on the Boostec webpage.

Table 43.12-7 – SiC 100®: Typical characteristics

Typical characteristics		Conditions	Units	Value
Chemical	Free Silicon		wt %	0
Physical	Crystal structure		-	alpha SiC
	Mean grain size		10^{-6} m	5
	Total porosity		vol %	< 3.5
	Open porosity		vol %	0
	Apparent bulk density		kg / m³	3100
	Theoretical density		kg / m³	3210
Thermal	Coefficient of thermal expansion	20°C to 500°C	10^{-6}/°C	4.0
		20°C to 1000°C	10^{-6}/°C	4.6
		20°C to 1400°C	10^{-6}/°C	5.2
	Thermal conductivity	20°C	W/m K	180
		500°C	W/m K	68
		1000°C	W/m K	40
	Specific heat	20°C	J/kg K	680
		500°C	J/kg K	1040
		1000°C	J/kg K	1180
	Maximum thermal shock		°C	325
Mechanical	Vickers hardness	500 g load	GPa	22
	Mechanical strength (3-point bend)	20°C	MPa	450
		1000°C	MPa	450
		1400°C	MPa	450
	Weibull modulus	20°C	-	10
	Compressive strength		MPa	3000
	Young's modulus		GPa	420
	Shear modulus		GPa	180
	Poisson's ratio		-	0.16
	K_{IC} toughness	20°C	MN $m^{-3/2}$	3.5
Electrical	Electrical resistivity		Ohm m	105
Service	Maximum working temperature	Air	°C	1450
		Inert atmosphere	°C	1800

Status: (30.05.2006)

43.12.6.14 Outgassing

The results of outgassing tests, conducted in accordance with ECSS-Q-70-01 and ECSS-Q-ST-70-02, are given in Table 43.12.7, Ref. [43-112].

Table 43.12-8 – SiC 100®: Micro-VCM test E544 - outgassing values

	TML (%)	RML (%)	CVCM (%)
SiC-100	0.01	0.00	0.00

The measured outgassing properties of SiC-100 were within those stated in ECSS-Q-ST-70-02. No FTIR spectrum was found from the condensed material.

43.12.6.15 Assembly technologies

Conventional processes, such as adhesive bonding with epoxy or using mechanical fasteners (bolting), are used to assemble SiC parts. These technologies offer micrometric stability performance sufficient for most structural applications or fixings.

For mirror development, the stability demand is much more severe, so others processes are used. Two different assembly technologies for SiC-100 have been achieved in parallel, Ref. [43-111], [43-113]:

- Ceramic bonding.
- Brazing.

Both of these assembly methods are now well-established or qualified for space use.

43.12.6.16 Ceramic bonding

Ceramic bonding is a process that enables assembly of separate 'green body' parts before sintering to produce a pure SiC item at the end of the process. The technique uses adhesive containing SiC particles to bond the two green bodies together. Green body machining of the full assembly can then proceed in the usual manner.

After sintering, a monolithic SiC part is obtained and the bonded interface can hardly be detected with the naked eye. This technique needs milling and sintering facilities capable of handling the final item dimensions. The maximum size that can currently be manufactured is about 1.5 m in diameter. An example of a product joined by ceramic bonding is the 1 m diameter 'Demonstration Mirror'.

43.12.6.17 Brazing

For larger monolithic pieces, brazing is the best solution. The brazing process enables of a large number of sintered SiC parts to be joined in a single step. The pieces to be brazed are positioned using jigs. During the process the braze material fills the gap between the pieces by capillary action. The operation is carried out at high temperature under a non-oxidizing atmosphere. The thickness of the brazed joints is typically around 50 μm.

The major advantages of brazing are the ease of processing and the absence of any distortion arising from the brazed joints during service. The entire assembly behaves as if it was made of a single SiC element. Limited by the dimensions of available brazing furnaces, the maximum size that can currently be achieved is about 3.8 m in diameter, e.g. 3.5 m diameter for the HERSCHEL primary mirror made of 12 brazed segments.

Examples:

- HERSCHEL Demonstrator Breadboard.
- HERSCHEL primary mirror.
- NIRSpec Optical Bench for James-Webb-Space Telescope (JWST).

43.12.6.18 Design guidelines

Some general design guidelines for the manufacturing of SiC-100 components are, Ref. [43-111]:

- Thickness > 0.5 mm, < 20 mm.
- Keep thickness transitions to less than an 8 to 1 ratio.
- Web thickness minimum 0.5 mm with aspect ratio (height to thickness) less than 80 to 1.
- Exterior chamfers 0.5 mm to 2 mm.

- Fillets > 1.5 mm for bottom of pockets.
- Fillets > 3 mm for vertical.
- As-fired (no post-sinter machining) ± 1% linear tolerance for pockets and non-critical features.

Actual product design parameters have many factors that determine the prudent design rules. An optimal design is achieved by evaluation on a case-by-case basis.

43.12.6.19 Applications: 1m diameter Demonstration Mirror

A 1 m diameter 'Demonstration Mirror', shown in Figure 43.12.8, was manufactured for visible wavelength applications, with two important requirements, Ref. [43-113]:

- Light weight
- Representative of the actual needs of optical devices, in order to demonstrate the feasibility of large diameter mirrors.

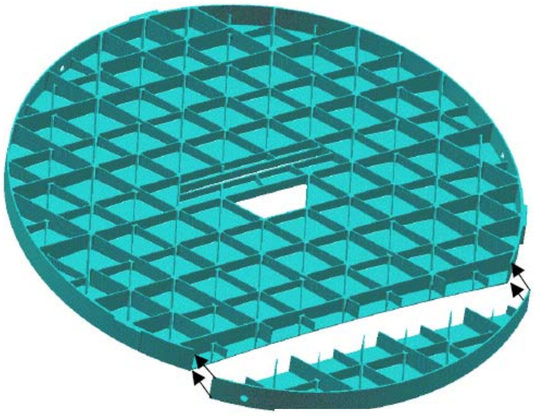
Reproduced courtesy of EADS Astrium GmbH

Figure 43.12-8 – SiC-100®: 1m diameter 'Demonstration Mirror' – ready to polish

The visible wavelength range implies that a SiC-CVD chemical vapour deposition layer is necessary before polishing and coating in order to achieve the desired surface roughness.

The 1 m mirror has a concave parabolic shape (primary reflector of Cassegrain telescope). The rear side is light weight. It is an optimised open back structure, which is the easiest way of machining the sintered SiC blank.

The mirror was manufactured from green parts that are joined in the green state by ceramic bonding, as shown in Figure 43.12.9.



Reproduced courtesy of EADS Astrium GmbH

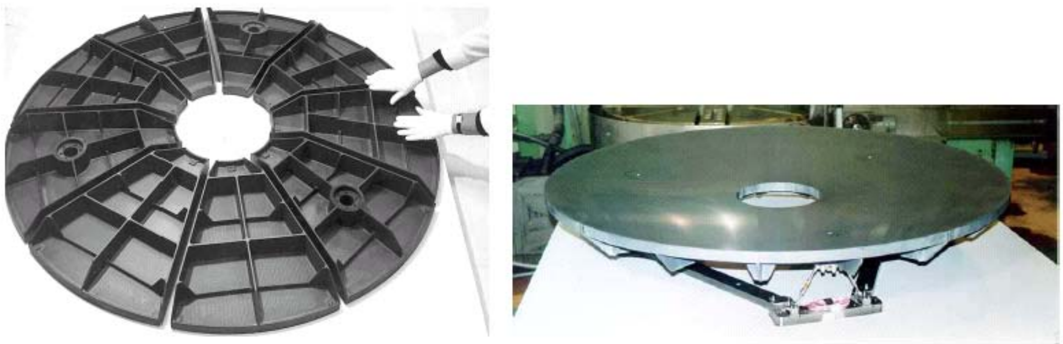
Figure 43.12-9 – SiC-100®: 1m diameter 'Demonstration Mirror' – green part joining by ceramic bonding

Other features of the final mirror are, Ref. [43-113]:

- 15.3 kg mass after grinding, equivalent to less than 20 kg/m² with lateral interfaces. An optimisation of supporting points by analysis enabled a mass reduction of 50 %.
- Stiffness of the mirror over 280 Hz (mirror alone) and 175 Hz with supporting devices.
- 20 g static load, design for sine, random and acoustic environment.
- Design for visible wavelength WFE ≤ 32 nm RMS.

43.12.6.20 Applications: 1.35 m HERSCHEL Demonstrator Breadboard

The brazing technique for joining was successfully verified by manufacturing of the HERSCHEL Demonstrator Breadboard, which is composed of 9 single segments, as shown in Figure 43.12.10, Ref. [43-113].



Reproduced courtesy of EADS Astrium GmbH

Figure 43.12-10 – SiC-100®: 1.35 m diameter HERSCHEL Demonstrator Breadboard – before and after brazing

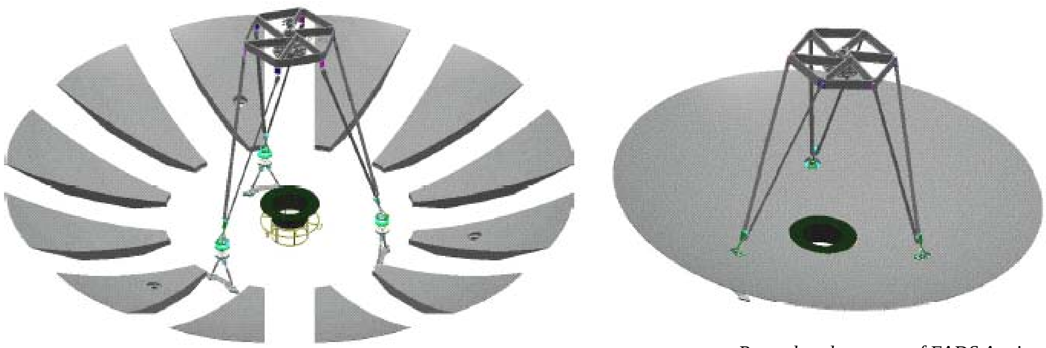
The manufactured monolithic mirror was successfully tested in vibration and low temperature environment (110 K). The vibration levels were 29 g in lateral direction and 27 g in axial direction.

During the thermal test, no distortion resulting from brazing or from CTE variation between segments was measured within an accuracy of 0.3 µm, Ref. [43-113].

Owing to the thinness of the brazed joint, it has no influence on the polishing process and does not create any discontinuity. Brazed pieces have been polished without problems even for optical accuracies in a visible wavelength application.

43.12.6.21 Applications: 3.5 m HERSCHEL Primary Mirror

The 3.5 m diameter primary mirror blank for the HERSCHEL telescope was fabricated by brazing together 12 silicon carbide (SiC) segments, as shown in Figure 43.12.11, Ref. [43-113].



Reproduced courtesy of EADS Astrium GmbH

Figure 43.12-11 – SiC-100®: 3.5 m diameter HERSCHEL Primary Mirror – manufactured by brazing 12 SiC segments

To provide mechanical stability, temporary stiffening ribs were positioned on the internal surface of the segment, both during the manufacturing of the segments and whilst the mirror blank was being assembled. These were in addition to the external surface stiffeners that are part of the final mirror configuration.

Once the assembly process was completed, the mirror blank was machined by grinding to remove the temporary stiffening ribs, to reduce the thickness of the mirror shell to 3 mm, and to achieve the correct shell profile ready for polishing, as shown in Figure 43.12.12, Ref. [43-113]. The machining process reduced the mass of the mirror blank from 720 kg to 240 kg.

Reproduced courtesy of EADS Astrium GmbH

Figure 43.12-12 – SiC-100®: 3.5 m diameter HERSCHEL Primary Mirror – after assembly

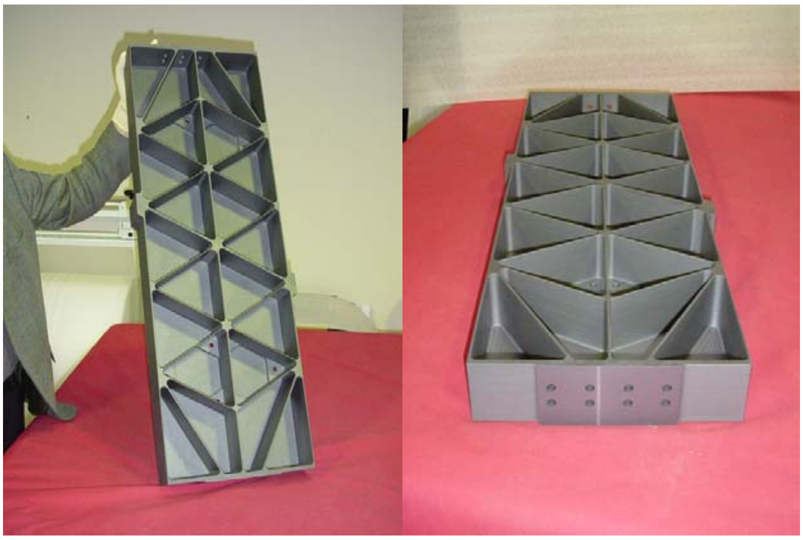
43.12.6.22 Applications: NIRSpec Optical Bench Breadboard

During a preparatory technology development activity for the JWST NIRSpec instrument, a technology demonstrator (the Optical Bench Breadboard) was successfully manufactured and tested, Ref. [43-114]. Objectives were to manufacture and test an optical bench breadboard model, representative in dimensions, shape and design, for the actual size NIRSpec instrument optical bench.

The study objectives were to:

- validate the complete manufacturing sequence including joining by brazing,
- demonstrate the feasibility of the all-ceramic SiC concept for operation at 30 K,
- provide design figures for the actual NIRSpec bench design.

Figure 43.12.13 shows the successfully tested 'Optical Bench Breadboard', which was manufactured in SiC-100 in two halves then joined by brazing.

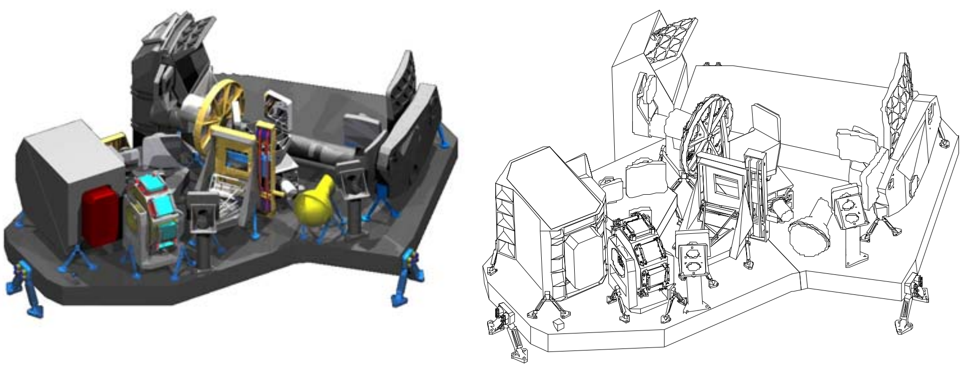


Reproduced courtesy of EADS Astrium GmbH

Figure 43.12-13 – SiC-100®: NIRSpec Optical Bench Breadboard

43.12.6.23 Applications: NIRSpec Optical Assembly for James Webb Space Telescope

NIRSpec 'near-infrared multi-object spectrograph' is one of three astronomical instruments on board the future James Webb Space Telescope (JWST). Its configuration, shown in Figure 43.12.14, is a dispersive spectrograph capable of observing astronomical objects in the 1 µm to 5 µm wavelength range, Ref. [43-115].



Reproduced courtesy of EADS Astrium GmbH

Figure 43.12-14 – SiC-100®: Optical Assembly of NIRSpec for James Webb Space Telescope

The NIRSpec optical bench is a large planar structure (1.8 m × 1.25 m) made of SiC-100. It carries mirrors, filters, mechanisms and the 'Focal Plane Assembly'. A unique feature of the 'Spectrograph' is its athermal design, where most of its components, particularly mirrors, mechanisms and structure, are made from the same material in order to avoid distortion resulting from thermo-elastic mismatch.

The large base plate is a joined structure manufactured from two monolithic parts brazed together.

43.12.6.24 Applications: High precision components

Other high-precision components made of SiC-100 for space applications are described on the Boostec website.

43.13 Silicon nitride

43.13.1 Typical characteristics

43.13.1.1 General

The general characteristics of silicon nitride include:

- high stiffness,
- low thermal expansion,
- good creep resistance,
- good thermal shock resistance,
- not electrically conductive (pure).

Silicon nitride can be used up to 1500°C in vacuum. It disassociates at 1850°C in nitrogen atmospheres.

43.13.1.2 Sialon

Sialon is a modified form of silicon nitride in which aluminium and oxygen are substituted for silicon and nitrogen to produce 'silicon aluminium oxynitride'. In general they possess good strength, fracture and wear resistance. Applications include wear parts and high-temperature engine components. The addition of titanium carbide produces conductive sialons.

43.13.1.3 Processing

The performance of commercially-available silicon nitride grades is affected by the processing route. This determines the microstructure, e.g. grain size, residual porosity levels, secondary phases, and consequently the mechanical characteristics and high-temperature resistance. The characteristics of silicon nitride materials produced by different processing routes can be summarised as:

- Sintered materials can have strengths approaching those of hot pressed variants and shaped parts can be made that are not possible by hot pressing. Modified compositions can increase toughness at the expense of strength.
- RBSN 'reaction bonded silicon nitride', or 'reaction sintered', are usually porous and have moderate strengths but high thermal shock resistance. Uses tend to be for refractories, exploiting their non-wetting by molten metals. Modified processing can produce fine, even dispersions of porosity improving the strength.
- Hot-pressed or hot-isostatically-pressed materials have very high strengths, good fracture toughness and good oxidation resistance (to 1200°C). Creep resistance depends on the presence of glassy, secondary phases. Uses include precision high-temperature engineering components, e.g. gas turbine and engine parts.

43.13.2 Typical properties

Table 43.13.1 compares the properties of some commercially-available silicon nitride materials produced by different process routes, Ref. [43-12], [43-14], [43-31].

Table 43.13-1 – Technical ceramics: silicon nitrides – comparison of materials properties produced by different process routes

Property	Examples of commercially-available silicon nitride grades †						
	Dynacer SSN	SSN (MAC SSNS)	RBSN (1) (MAC RBSNS)	CERALLOY™ 147-01B	Dynacer RBSN	CERALLOY™ 147-31N	CERALLOY™ 147-5
Processing method	sintered	sintered	reaction bonded	reaction bonded	reaction bonded	sintered reaction bonded	hot pressed
Silicon nitride content (%, typ.)	-	99	99	72	-	92	>98.5
Grain size (μm)	-	-	-	-	-	-	-
Bulk density (kg/m³)	3250	3200	2500	2300	2500	3200	3300
Porosity apparent (%)	-	0	20 (open)	-	-	-	-
Hardness							
Vickers (GPa, Hv0.5kg)	-	16	-	-	-	-	-
Vickers (kg/mm², Hv0.3kg)	1500	-	-	800	1100	1800	1800
Compressive strength (MPa)	2000	>3000	650	-	550	-	-
Flexural strength, 20°C (MPa)	650	650	200	190	190	700 to 800	930
Young's modulus, 20°C (GPa)	290	280	170	175	170	310	320
Fracture toughness (MPam$^{1/2}$)	8.0	-	-	2.5	3.0	6.0	6.0
Thermal conductivity, 20°C (WmK)	25	15	12	14	16	26	35
CTE, ($\times 10^{-6}$ /°C) 20°C to 1000°C	3.0	3.3	3.1	3.2	3.0	3.1	3.2
Applications (examples)	not stated	Mechanical parts (bearings – ball and rollers); High-temperature jigs and fixtures	Materials process industries (refractories); high temperature jigs and fixtures	Industrial (insulators, sputtering targets); Net shape parts	not stated	Automotive components; bearings; wear parts; Net shape parts	Wear parts
Key: † MAC grades are tradenames of Morgan Advanced Ceramics; CERALLOY is a tradename of Ceradyne Inc. Dynacer = Dynamic Ceramic Ltd. (1) Low density porous grade.							

43.14 Zirconium oxide: Zirconia

43.14.1 Typical characteristics

43.14.1.1 General

Pure zirconia undergoes phase transformations with increasing temperature that can result in significant dimensional changes, e.g. about 7.5% volume reduction between 1000°C to 1100°C. For this reason it is not used commercially as a technical ceramic in its pure form.

Zirconia usually occurs with hafnium oxide, and this tends to remain in commercial compositions because it is difficult to remove totally. Deliberate additions of other oxides, e.g. magnesium oxide or yttria, stabilise the crystal structure of zirconia and so avoid the volumetric changes. This enables a range useable technical ceramics to be produced for different applications.

Commercially-available types of zirconia are grouped by whether they are:

- Fully stabilised: used largely as refractories because of their resistance to attack by many molten metals.
- Partially stabilised: PSZ grades have combined strength and toughness, making them suitable for engineering components.

43.14.1.2 PSZ partially-stabilised zirconia

Additions of other oxides can modify and stabilise the crystal structure of zirconia throughout the intended service temperature range, so limiting the dimensional changes associated with phase changes.

At high temperatures PSZ becomes electrically conductive

The commercially-available grades of PSZ can also be grouped as:

- TZP ‘tetragonal zirconia polycrystal’, which have high strengths but moderate usage temperatures.
- TTZ ‘transformation toughened zirconia’ undergoes a phase change that results in beneficial compressive stresses, e.g. surfaces undergoing machining or at crack tips.

43.14.2 Typical properties

43.14.2.1 PSZ partially-stabilised zirconia

Table 43.14.1 compares the properties of some commercially-available PSZ grades, Ref. [43-12], [43-31], [43-32].

Table 43.14-1 – Technical ceramics: partially stabilised zirconia – typical properties

Property	Examples of commercially-available partially-stabilised zirconia grades †					
	Z 500 (MAC 965R)	CoorsTek TTZ	DURA-Z™	CoorsTek YTZP	CEROXIDE™	Z900 (MAC –Z940R)
Zirconia content (%, typical) (3)	96.5	-	-	-	-	94.0
Stabilised	3.5% MgO	MgO	MgO	Y_2O_3	3.0% Y_2O_3	5.4% Y_2O_3 0.25% Al_2O_3
Grain size (μm)	60	-	-	-	-	<1
Bulk density (kg/m³)	5700	5720	5720	6020	6080	6000
Porosity apparent (%)	0	-	-	-	0	0
Hardness: Vickers (GPa, Hv0.5kg) Rockwell (HR45N)	 11 78	 11.8 77	 11.8 77	 12.7 81	 - 84	 12.5 80
Compressive strength (MPa)	2000	1750	1750	2500	-	-
Flexural strength, 20°C (MPa)	550	620	758	900	1000	900
Young's modulus, 20°C (GPa)	200	200	200	210	205	200
Fracture toughness ($MPam^{1/2}$)	8.4	11	11	13	-	10.0
Thermal conductivity (WmK, 20°C)	2.5	2.2	2.2	2.2	2.9	2.9
CTE, ($\times 10^{-6}$ /°C) 20°C to 800°C	10	10.1 (2)	10.2	10.3	9.5 (1)	9
Applications (examples)	Process equipment parts: (pumps, valves) in corrosive or abrasive sectors.	Industrial applications	Industrial applications	Industrial applications	Industrial applications	Process equipment parts: (pumps, valves) in corrosive or abrasive sectors.

Key: † MAC grades are tradenames of Morgan Advanced Ceramics; CEROXIDE is a tradename of SCT – Societe des Ceramiques Techniques, (F); DURA-Z is a tradename of CoorsTek. (1) 20°C to 600°C; (2) 20°C to 1000°C; (3) includes hafnium oxide content.

43.15 Structural glass: Glass ceramics

43.15.1 Introduction

43.15.1.1 General characteristics

Glass ceramics are formed from molten glass which is then crystallised by heat treatment. They consist of mixtures of several oxides which form complex, multiphase microstructures. In general the properties can be tailored by controlling the crystalline structure of the glass matrix. Unlike conventional sintered ceramics, glass ceramics do not contain porosity which can adversely affect mechanical performance.

Applications for glass ceramics are many and varied; from domestic cookware and appliances, to industrial uses and static and dynamic aerospace engineering components, e.g. radomes, engine parts and mirror substrates for terrestrial or space telescopes.

Glass ceramics are stable at high temperatures, have near-zero coefficients of thermal expansion and are resistant to many forms of high-temperature corrosion and oxidation.

43.15.1.2 Composition

The main types of glass ceramic materials are:

- LAS - lithium-aluminium-silicate (beta spodumene), which has near-zero thermal expansion up to about 430°C. Whilst the high silica content gives low expansion characteristics it does lower the strength.
- MAS - magnesium-aluminium-silicate (cordierite), which in general, is both stronger and more corrosion resistant than LAS. A modified, multiphase version of MAS which contains aluminium titanate has corrosion resistance up to about 1100°C.
- AS - aluminium-silicate (aluminous keatite), which is produced by leaching the Li from LAS prior to forming. It also has near-zero thermal expansion up to about 430°C. It has been evaluated for dynamic parts in turbine engines because of its combined strength and corrosion resistance.

43.15.2 Commercially-available glass and glass ceramics

43.15.2.1 Products

The main commercial products can be summarised as:

- Zerodur®, produced by Schott AG. Zerodur is a well-known glass ceramic within the space industry for applications such as mirror substrates and optical benches, [See also: 43.19].
- ULE®, produced by Corning Corp. ULE is a titania-silicate glass used within the space industry for applications such as mirrors.
- Macor®, which is produced by Corning Corp. but is available throughout Europe from distributors.
- Chemical-machinable grades.
- Glass-bonded Mica, also known as a 'ceramoplastic',

Some examples of commercially-available glass ceramics evaluated for space applications include:

- Hubble Space Telescope: ULE for main mirror; Zerodur for secondary mirror.
- ELT extremely large telescopes (Zerodur)
- SST (Zerodur)

43.15.2.2 Zerodur®

Schott AG produces Zerodur®, a glass ceramic with an extremely low thermal expansion coefficient. General characteristics are, Ref. [43-35]:

- near-zero thermal expansion with 3D homogeneity,
- high internal quality,
- good processing behaviour,
- polishable to a high accuracy,
- easily coated,
- low Helium permeability,
- non-porous,
- good chemical stability.

Zerodur® has been evaluated for applications such as lightweight mirrors for astronomical telescopes, both space-based and terrestrial, including ELT projects for extremely large telescopes (20m to 100m). Commercially-available grades of Zerodur® glasses are categorised by, Ref. [43-35], [43-36]:

- thermal expansion characteristics, from Class 0 to 2.
- internal quality is determined by the presence and size of inclusions (number and maximum dimension), striae and bulk stress (measured by birefringence).

For optical applications, the Zerodur® grades are known as 'standard' or 'special', where Class 0 to 3 stipulates the internal quality, Ref. [36]. Zerodur® M is a variation intended for applications where processing involves cooling rates more than 0.1K/min, which can affect the CTE values of components. Whilst Zerodur® can be used up to 600°C (mechanical parts and windows), a modified material known as Zerodur® K20 is intended for applications up to 850°C. This material has an expansion coefficient of 1.5 × 10-6 /K at room temperature and 2.0 × 10-6 /K (20°C to 700°C).

Table 43.15.1 gives the basic properties of Zerodur® standard grade, Ref. [43-35]. Standard grade Zerodur® covers materials up to expansion Class 2.

Table 43.15-1 – Technical ceramics: Zerodur® glass – typical properties

Property	Schott AG. commercially-available Zerodur® glass ceramic †
Composition	not stated
Grain size (μm)	-
Bulk density (kg/m³)	2530
Porosity apparent (%)	0
Hardness: Knoop (0.1/20)	620
Tensile strength, MOR (MPa)	-
Compressive strength, 20°C (MPa)	-
Flexural strength, 20°C (MPa)	-
Young's modulus, 20°C (GPa)	90.3
Shear modulus, 20°C (GPa)	-
Fracture toughness (MPam$^{1/2}$)	-
Thermal conductivity, 20°C (Wm K)	1.46
Coefficient of thermal expansion, (×10^{-6} /K)	0
Expansion Class, (×10^{-6} /K), 0°C to 50°C: Class 2 Class 1 Class 0	 0 ± 0.10 0 ± 0.05 0 ± 0.02
Applications (examples)	Astronomy mirrors, inc. GTC - Gran Telescopio Canarias (42 inch mirror); ROSAT; SST – solar telescope.
Key: † Zerodur is a tradename of Schott AG (D);	

43.15.2.3 ULE®

Corning Inc. ULE® 'ultra low expansion' is a range of titanium silicate glasses which have particular characteristics suitable for applications needing thermo-mechanical stability combined with optical performance. ULE® has been evaluated for applications such as lightweight mirrors for astronomical telescopes, both space-based and terrestrial, and spacecraft applications. Although the basic characteristics of the materials are the same, the different commercially-available grades of ULE® are categorised by, Ref. [43-33]:

- thermal expansion characteristics, which state guaranteed maximum limits (linear coefficient of thermal expansion; delta CTE variations in axial and radial directions of a part),
- optical retardation, which is measured by birefringence,
- inclusion quality, i.e. the guaranteed maximum numbers of seeds, bubbles and opaque inclusions.

The main grades of ULE® glasses are known as, Ref. [43-33]:

- Premium, which has the lowest CTE variations, optical retardation and inclusions.
- Mirror, which exhibits slightly larger CTE variations and birefringence than premium grade, but the presence of inclusions are divided into 'critical' and 'non-critical' zones and the size and number present are different.
- Standard, where the CTE and birefringence values are the same as 'mirror' grade and the inclusions are the same as 'mirror – non-critical zone'.
- Tooling, which has no limits given for CTE, optical or inclusions.

For EUVL 'extreme ultraviolet lithography' (semiconductor manufacturing) applications, a further ULE® glass grade is under development, i.e. improved material straie, which affect surface roughness, and CTE homogeneity, Ref. [43-34].

ULE® glasses can be machined by milling and by water-jet. Components can be assembled using high-temperature joining techniques. Sandwich structures can be manufactured that offer the same mechanical and thermoelastic performance of monolithic structures are possible, e.g. for light-weight mirror applications.

Table 43.15.2 gives the basic properties of ULE® glass, Ref. [43-33].

Table 43.15-2 – Technical ceramics: ULE® glass – typical properties

Property	Corning Corp. commercially-available ULE® glass †
Composition	titanium silicate
Grain size (µm)	-
Bulk density (kg/m^3)	2210
Porosity apparent (%)	0
Hardness: Knoop (kg/mm^2, 200g load)	460
Tensile strength, MOR (MPa)	49.8
Compressive strength, 25°C (MPa)	-
Flexural strength, 25°C (MPa)	-
Young's modulus, 25°C (GPa)	69.6
Shear modulus, 25°C (GPa)	29.0
Fracture toughness ($MPam^{1/2}$)	-
Thermal conductivity, 25°C (Wm °C)	1.31
Coefficient of thermal expansion, ($\times10^{-9}$ /K)	0 (0 ppb/°C)
Δ CTE maximum variation, ($\times10^{-9}$ /K) 5°C to 35°C	± 30 (± 30 ppb/°C)
Applications (examples)	Astronomy mirrors, e.g. 4.2 m primary mirror for Lowell Observatory's Discovery Channel Telescope.
Key: † ULE is a tradename of Corning Corporation (USA);	

43.15.2.4 Macor®

Macor is a machinable glass ceramic which can be machined in its fired state using conventional tools. Its composition is a proprietary oxide mix containing Si, Mg, Al, K, B and F, Ref. [43-12]. Some of its characteristics include:

- machinable, stock bar available,
- strength, 345 MPa (compressive), 89 MPa (flexural),
- modulus, 68 GPa,
- high-temperature resistance similar to conventional glass ceramics, e.g. 800°C (continuous), 1000°C (no load),
- thermal conductivity, typically 1.46 Wm/K,
- thermal expansion, typically 9.3×10^{-6} /°C from RT to 1500°C,
- electrical properties similar to conventional glass ceramics.
- If properly baked-out, Macor can meet outgassing requirements.

Some examples of aerospace applications include, Ref. [43-12]:

- Shaped parts on Space Shuttle Orbiter, e.g. retaining rings at hinge points, windows and doors.
- NASA's space-borne gamma radiation detector uses large pieces of Macor, in which frame corners are joined by a combination of machined (butt-lap) mechanical joints and a sealing glass.

43.15.2.5 Chemical-machinable grades

Chemical-machinable grades are photo-sensitive glasses in their initial state. After sensitising with light to create a pattern, they can be chemically machined or etched to form the component. The part can either be used 'as etched', i.e. as glass, or fired to convert it to a glass ceramic. Fired glass-ceramics can be used where precise tolerances are needed or where a close match to the thermal expansion characteristics of metals is necessary, e.g. computer equipment, such as disk drive heads and printers.

43.15.2.6 Glass-bonded Mica

Also known as a 'ceramoplastic', glass-bonded mica can be moulded and machined (using carbide tools) like a plastic but their properties are similar to ceramics. Processing involves heating a green compact mix of glass and mica until the glass flows, then shaping by a moulding process. Densification occurs during moulding, so no further firing is necessary after machining. The low shrinkage of these materials ensures close dimensional tolerances on moulded parts. Thermal expansion is similar to metals, so enabling mixed-material assemblies to be used from cryogenic temperatures up to about 700°C, depending on the grade. An example of a commercially-available grade is MicaTherm® HT from Morgan Advanced Ceramics, which can be used continuously up to about 450°C, Ref. [43-12]. Applications include domestic, industrial and aerospace components, beyond the capability of polymer-based materials and especially where non-flammability, non-offgassing of toxic fumes and non-outgassing properties are stipulated.

43.15.3 Glass ceramics for space applications

Glass ceramics are processed as molten glasses with controlled crystallisation. The main constituents are silicon oxide and alumina but also lithium oxide, zirconia and titania. The processing of the melt occurs in a similar way as with the processing of glass, however the melt is finally transferred by temperature treatment into the crystalline or ceramic condition. The result is a glass-similar product with controllable material properties. A typical characteristic of glass ceramic is a negative coefficient of thermal expansion within the temperature range between -50°C and 300°C.

The very low thermal expansion of glass ceramics makes these materials good candidates for the design of precise and highly dimensionally stable optical instruments and structures.

Different types of glass ceramics have been proven in the design and manufacture of optical instruments for space. Amongst others, Zerodur®, Zerodur M®, ULR® 'ultra low expansion glass' and Fused Silica, [See: 43.11], are used for the manufacture of optical mirrors and reflectors but also for support structures, such as optical benches, [See: 43.19].

Glass ceramics have also been considered as the matrix phase of composites, [See: Chapter 53].

Figure 43.15.1 shows the coefficient of thermal expansion of different glass ceramics applied for optical instruments in space as function of temperature.

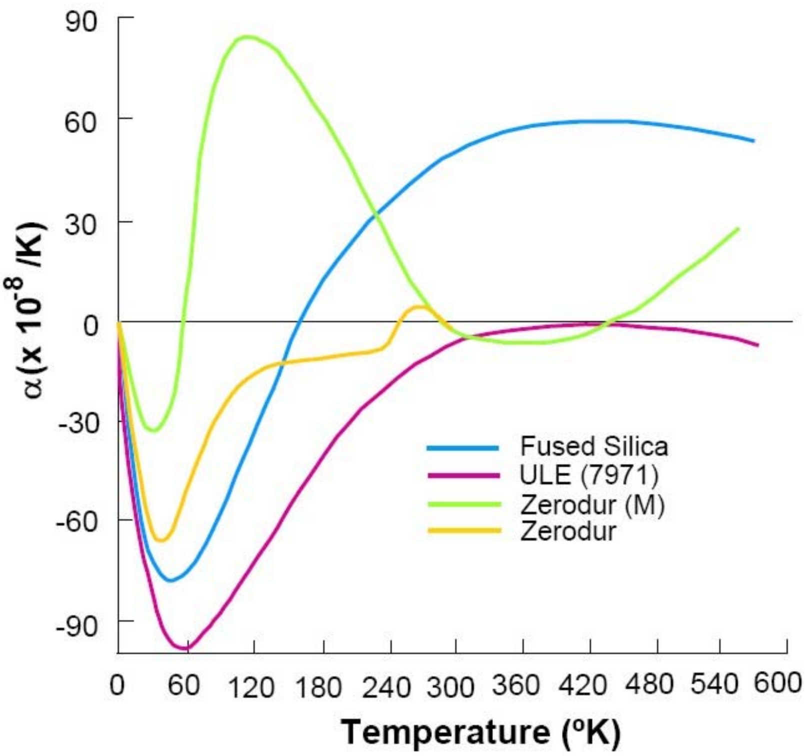


Figure 43.15-1 - Glass ceramics: Coefficient of thermal expansion with temperature - materials for optical instruments in space

43.15.4 ZERODUR®

43.15.4.1 General

Schott AG produce several grades of material. Zerodur glass ceramic is an inorganic, non-porous material, made by a process of controlled volume crystallisation. It contains a crystallised phase and a residual glass phase which together determine the properties of the material. Zerodur contains 70 to 78 weight percent crystalline phase with a high quartz structure. This crystalline phase has a negative linear thermal expansion, whilst that of the glass phase is positive. The crystals have an average size of about 50 nm.

Zerodur has an extreme low coefficient of thermal expansion, which even can become zero or slightly negative in some temperature ranges. Another unique characteristic of Zerodur is its exceptional good homogeneity. Even in large material blocks, the fluctuations of mechanical and thermal properties are nearly not measurable. A good transparency in the range of about 400nm to 2300nm enables a verification of the inner quality of Zerodur. If needed, bubbles or inclusions in Zerodur are detectable.

43.15.4.2 Processing

The Zerodur manufacturing sequence is based on established and proven methods used in the production of high homogeneity optical glasses; as shown in Figure 43.15.2.

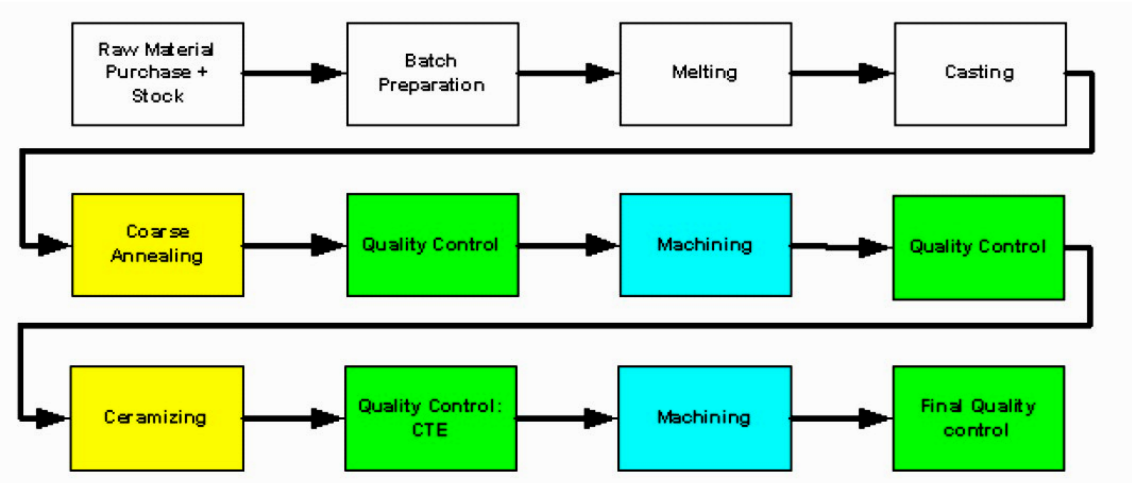


Figure 43.15-2 - Zerodur®: Production process schematic

The melt is cast into moulds and inserted into annealing ovens to cool down the raw casting to room temperature in a controlled way for several weeks. In the ceramisation process the material is heated up again to achieve controlled nucleation and growth of the crystal phase; as shown in Figure 43.15.3.

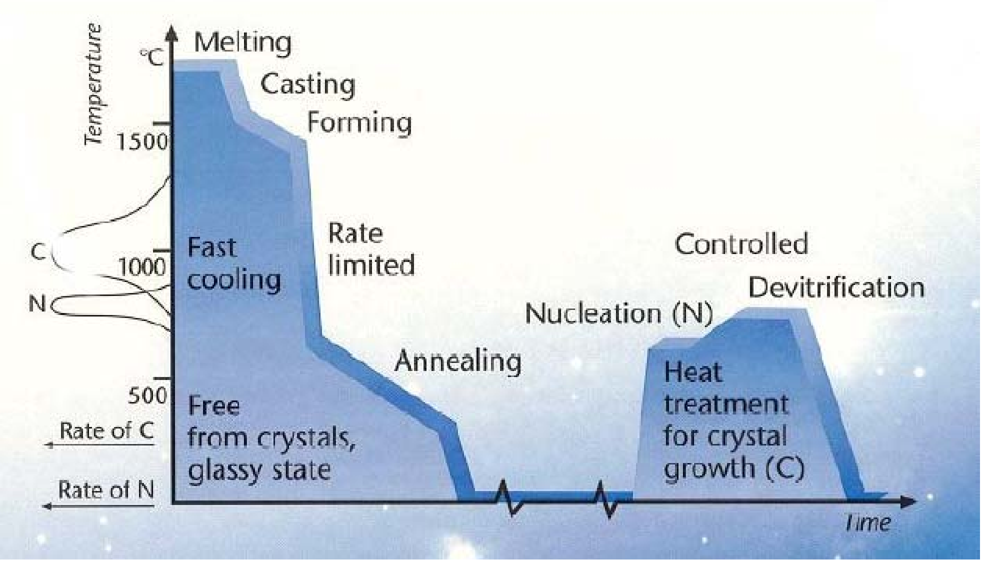


Figure 43.15-3 - Zerodur®: Annealing process

The ceramisation process has a large influence on the final homogeneity of CTE within the blank. Depending on the size of the part, it can take up to several months, Ref. [43-64]. After ceramisation the material can be processed to its final shape.

43.15.5 ZERODUR®: Properties

43.15.5.1 General

A summary of engineering data for Zerodur is given in Figure 43.15.4, Ref. [43-86].

PROPERTY	UNITS	VALUE
Mechanical		
Density	gm/cc	2.53
Hardness	Knoop	620
Tensile Strength	kpsi	-
Modulus of Elasticity	psi x 10^6	13
Flexural Strength	kpsi	-
Compressive Strength	kpsi	-
Poisson's Ratio	-	0.243
Fracture Toughness	MPa $m^{1/2}$	-
Electrical		
Dielectric Strength	ac volts/mil	-
Dielectric Constant	(at 1 MHz)	7.4
Volume Resistivity	ohm-cm^2/cm	2 x 10^{13}
Thermal		
Coefficient of Thermal Expansion	x10^{-6}/°C	0.0+/-0.1
Thermal Conductivity	W/m°K	1.46
Specific Heat	cal/g°C	0.8
Max Working Temp	°C	600
Shock Resistance	°C diff.	
Optical		
Index of Refraction	(Ordinary ray, N_o,c-axis)	1.54
	(Extraordinary ray, N_e,c-axis)	n/a
Birefringence		n/a
Transmission Band		0.30 - 2.5

All properties are at room temperature unless otherwise noted.

Engineering data are representative, and are not intended as absolute nor warrantable. Manufacturer's Data shown is blended from multiple sources and therefore illustrates the marketplace.

Figure 43.15-4 - Zerodur®: Summary of engineering data

43.15.5.2 Mechanical properties

The mechanical properties of Zerodur are:

- Density: 2.53 g/cm^3), Ref. [43-87]
- Young's modulus, E : 90 GPa, Ref. [43-95]
- Poisson's ratio, ν : 0.24, Ref. [43-95]
- Fracture toughness, K_{IC} : 0.9 MPa $m^{1/2}$, Ref. [43-83], [43-85], [43-96]
- Bending strength and Weibull parameters: Values are dependent on the surface condition, [See: Design].
- Flatness: up to λ/20, Ref. [43-87]

43.15.5.3 Optical properties

The optical properties of Zerodur are, Ref. [43-87]:

- Refractive Index, Ref. [43-97]:
  — nd = 1.5424 (587.6nm).
  — ng = 1.5544 (435.8nm).
- Transmission; as shown in Figure 43.15.5 for a 5 mm thick sample, Ref. [43-87].

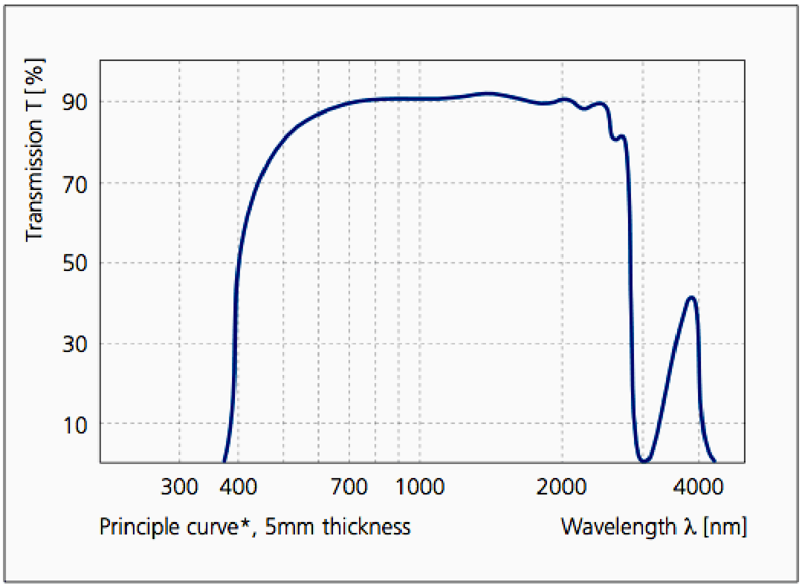


Figure 43.15-5 - Zerodur®: Optical transmission curve

43.15.5.4 Electrical properties

The electrical properties of Zerodur are, Ref. [43-87]:

- Dielectric constant:
  — 8.0 (1kHz).
  — 7.4 (1 MHz).
- Loss factor tan δ:
  — 29×10^{-3} (1kHz).
  — 15×10^{-3} (1 MHz).

43.15.5.5 Thermal properties

The thermal properties of Zerodur are, Ref. [43-87], [43-95]:

- CTE coefficient of thermal expansion, α: $0 \pm 0.10 \times 10^{-6}$/K, over temperature range 0°C to 50°C, Ref. [43-87]
- Thermal conductivity, λ: 1.46 W/mK, Ref. [43-95]
- Specific heat, c_p: 0.80 J/gK, Ref. [43-95]
- Operating temperature, *Tmax*: up to 600°C, Ref. [43-87]

The thermal expansion characteristics as a function of temperature, Ref. [43-70], [43-74], [43-87], [43-102] are shown in Figure 43.15.6 for 0K to 900K, Ref. [43-87].

Zerodur is compared with different materials in Figure 43.15.7, Ref. [43-74], [43-102] and Figure 43.15.8, [43-74].

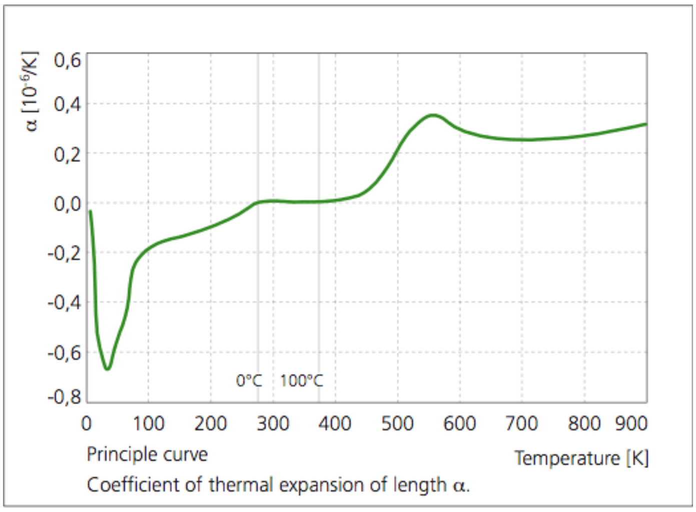


Figure 43.15-6 - Zerodur®: Thermal expansion – cryogenic behaviour

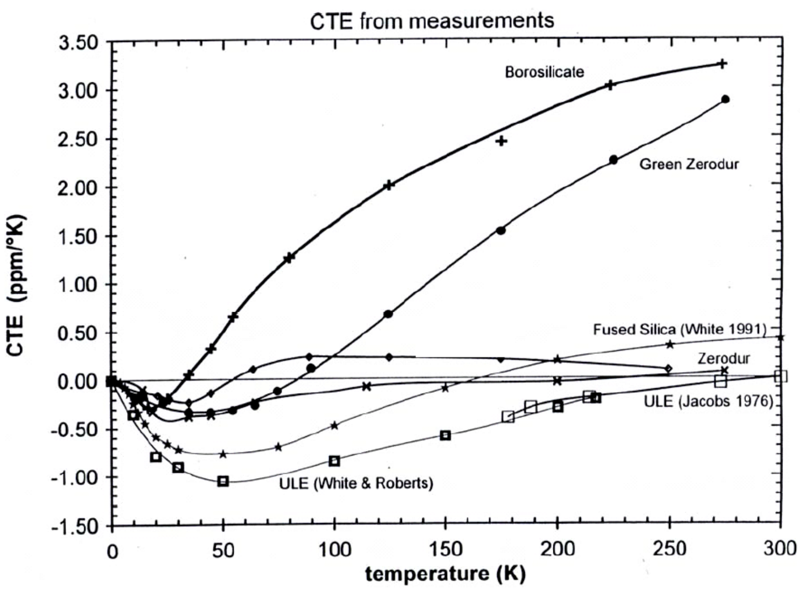


Figure 43.15-7 - Zerodur®: Coefficient of thermal expansion – different glass ceramics

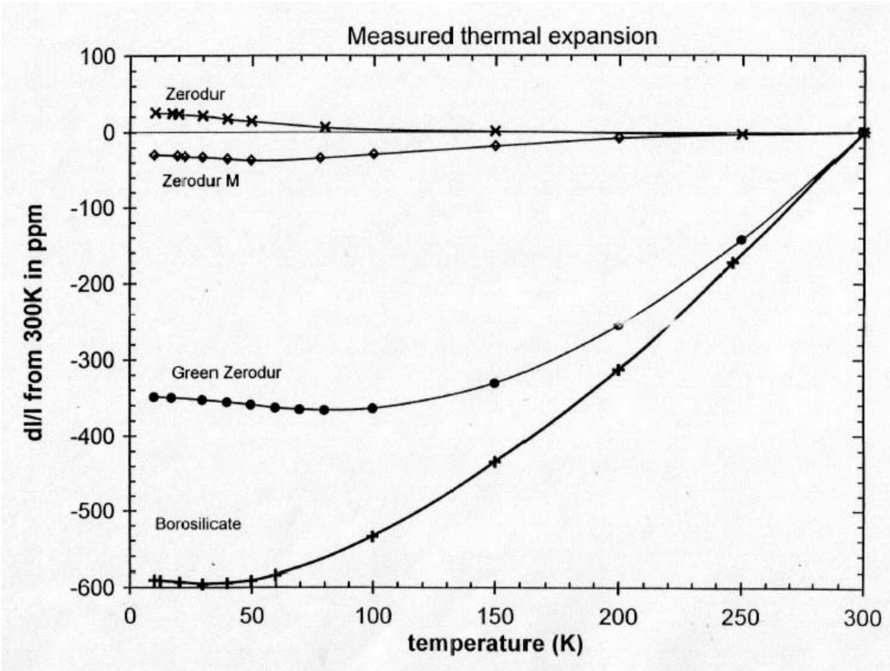
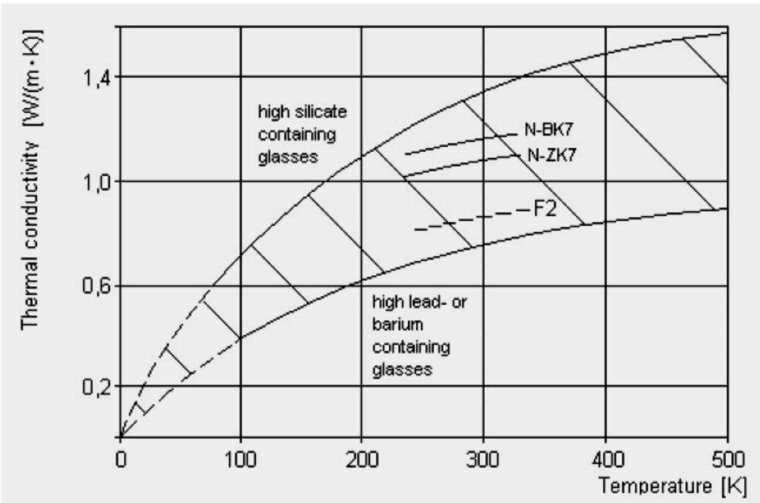


Figure 43.15-8 - Zerodur®: Thermal expansion of different glass ceramics



In Figure 43.15.9, the thermal conductivity is plotted as function of temperature for different glasses, Ref. [43-94]. The values of thermal conductivity for Zerodur are located between the two limiting curves in the diagram.

Range of thermal conductivity of glasses with curves at temperatures between 0K and 500K. The data for Zerodur® are near to the upper limiting curve 'high silicate containing glasses', Ref. [43-94].

Figure 43.15-9 - Zerodur®: Thermal conductivity characteristics of different glasses

43.15.6 ZERODUR®: Design

43.15.6.1 General

Zerodur® is a widely-applied glass ceramic material with excellent performance also in space. Material properties and design principles for this material are well documented. Only a rough outline of the usual design procedure is presented here. Detailed information is given in Ref. [43-90], [43-94], [43-96], [43-103], [43-104].

43.15.6.2 Design strength

In general, the strength of glass and glass-ceramics is not a material property like Young's modulus because it is dependent upon several factors, Ref. [43-96]:

- microstructure of the surface which is tension stressed by the load applied,
- area of the surface exposed to tensile stress,
- rate of stress increase,
- environmental conditions

A mechanical failure of glass occurs if two conditions occur together, firstly the presence of tensile stress at the surface and secondly the presence of a flaw in the region of the tensile stress. In the case of large flaws, i.e. more than about 0.5 mm as an order of magnitude, or at too high tensile stresses glass breaks immediately or within short duration.

If flaws are small, e.g. well below 0.5mm or of microscopic dimensions, such flaws grow slowly under the influence of the tensile stress. If microcracks exceed a critical length their growth accelerates rapidly and the material fails. The largest microflaw within a tensile stress loaded area determines the strength of the material.

Example: Conservative bending strength values for long-term applications of optical glass and Zerodur are 8 MPa and 10 MPa respectively. The values are valid for normal surface conditions without scratches and flaws.

43.15.6.3 Mechanical analysis: Mathematical model

The mathematical model which is applied for the mechanical lay-out of glass ceramics is based on the Weibull theory, Ref. [43-96].

The Weibull probability function $F(\sigma)$, i.e. probability of failure at bending stress σ, is defined by:

$$F(\sigma) = 1 - \exp\left(-(\sigma/\sigma_0)^\lambda\right) \qquad [43.15\text{-}1]$$

where the Weibull parameters are:

σ_0	*Characteristic strength, $F(\sigma_0)$ = 63.21%*
λ	*Weibull factor, given by the slope of the Weibull line and provides a measure for the scatter of distribution.*

The distribution function is widely used in product lifetime statistics and enables deriving of predicted failure rates for collectives of identical parts. Based on test results obtained under well-defined conditions, design strengths for loads and conditions defined by special application requirements can be calculated.

43.15.6.4 Calculation procedure

The calculation procedure described is a short summary of the procedure published in Ref. [43-82]. The notation used is adopted from the source document.

The evaluation of strength test results provides $\sigma_0(S_L, 63\%, R)$ and λ for a particular glass type and surface condition.

$\sigma_0(S_L, 63\%, R)$ is the characteristic strength for the laboratory test surface area S_L and stress increase rate R. In the model employed the design strength depends on the parameters:

S_V	*the area of the tensile stress loaded surface*
R_V	*stress increase rate or*
t_V	*the stress load duration time (when constant)*
F_V	*the admissible probability of failure*

The design strength is derived from the laboratory strength by dividing it by f_{FOS}, the so called factor of safety:

$$\sigma_k = \sigma_0 / f_{FOS} \tag{43.15-2}$$

The factor of safety f_{FOS} is determined as a product of the area factor, probability factor and fatigue factor.

The formulae for the individual factors are derived on the basis of the Weibull model using the laws on probability:

$$f_{FOS} = f_A \times f_P \times f_F \tag{43.15-3}$$

where:

f_A	*area factor*
f_B	*probability factor*
f_f	*fatigue factor*

The area factor, f_A, is determined using the expression:

$$f_A = (S_V / S_L)^{1/\lambda} \tag{43.15-4}$$

Eqn. [43.15-4] assumes constant stress within the loaded area. It is a conservative approach since in many cases the tensile stress has a maximum value that decreases with distance away from that maximum.

For a more precise calculation, $S_{eff,V}$ is used. This is obtained by weighting the maximum stress with the stress distribution function instead of S_V.

The probability factor, f_p, is determined using the expression:

$$f_p = \frac{1}{(\ln \frac{1}{1-F_V})^{1/\lambda}} \qquad [43.15\text{-}5]$$

The fatigue factor, f_F, is determined using the general expression:

$$f_F = \left(\frac{t_{eff,V}}{t_{eff,L}} \right)^{1/n} \qquad [43.15\text{-}6]$$

Where:

$t_{eff,V}$	*effective loading time for the application*
$t_{eff,L}$	*effective loading time at laboratory*
n	*environmental stress corrosion constant*

Ref. [43-82] gives a more detailed description of parameters $t_{eff,V}$ and $t_{eff,L}$.

In the special case of a stress load constant in time, Eq [43.15-6] reads explicitly:

$$f_F = (t_V \times f_A \times f_P \times R \times (n+1)/\sigma_0)^{1/n} \qquad [43.15\text{-}7]$$

For application cases with time-varying loads $t_{eff,V}$ is calculated instead of t_V by using a weighting function that describes the load variation with time.

The stress corrosion constant, n, has been determined for several glass types. If no experimental results are available, n can be estimated according to the expression:

$$n = 38 - 2.6 \times \alpha \qquad [43.15\text{-}8]$$

Where:

α	*coefficient of thermal expansion, in units 10^{-6}/K usually at (-30°C, +70°C)*

The derivations of the formulae and more detailed information about the calculation of the factors *fA*, *fP* and *fF* are given in Ref. [43-82].

Table 43.15.3 show the measured results for the characteristic bending strength, σ_0, Weibull factor and stress corrosion constant, *n*, for Zerodur, Ref. [43-96].

The test procedure was according to DIN 52292-1 Double ring method R 30-6 with a stress increasing rate of 2 MPa/s, room climate conditions and a test area of 113 mm².

Failure of probability curves of bending strength for different surface conditions are shown in Figure 43.15.10 and Figure 43.15.11 and as a function of test area in Figure 43.15.12, Ref. [43-96].

Bending strength under constant load is shown in Figure 43.15.13, Ref. [43-96].

Table 43.15-3 - Zerodur®: Characteristic strength, Weibull factor and stress corrosion factor

Material	Surface condition	Characteristic strength σ_0 [MPa]	Weibull factor λ	Stress corrosion constant n / Medium
ZERODUR®	SiC 600	108.0	16.0	51.7 Air 50% [15]
ZERODUR®	SiC 320	71.3	12.4	59.2 Air 50% [16]
ZERODUR®	SiC 230	57.5	15.7	30.7 Water [16]
ZERODUR®	SiC 100	53.6	18.7	
ZERODUR®	D 15 A	130.6	10.6	(for all
ZERODUR®	D 35	78.7	15.7	surfaces)
ZERODUR®	D 64	64.0	12.5	
ZERODUR®	D 151	53.7	22.7	
ZERODUR®	D 251	48.8	11.1	
ZERODUR®	Opt. polish	293.8	5.3	
ZERODUR®	D 64 etched	219.8	6.0	
ZEROOUR®	SiC 600 at 77K	192.7	10.7	
glassy ZERODUR®	SiC 320	66.6	16.7	
ZERODUR® M	D 64	63.9	12.5	
ZERODUR® M	D 64 etched	291.6	3.8	

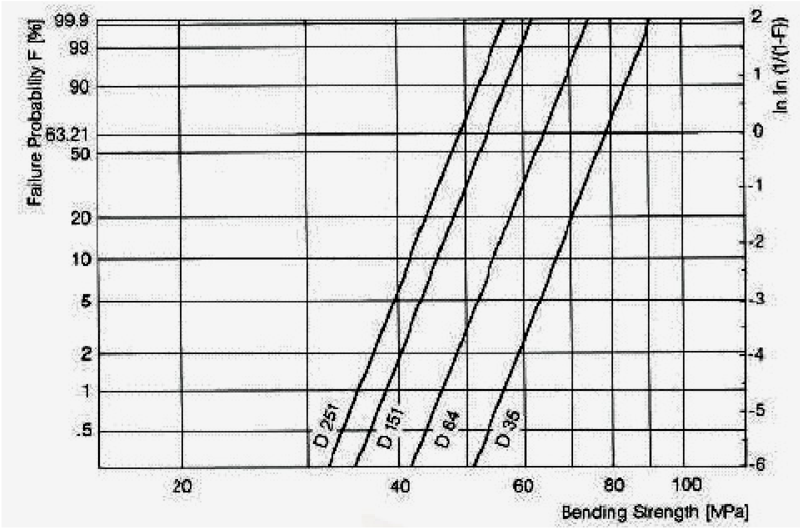


Figure 43.15-10 - Zerodur®: Failure probability for test surfaces processed with bonded grit of different sizes

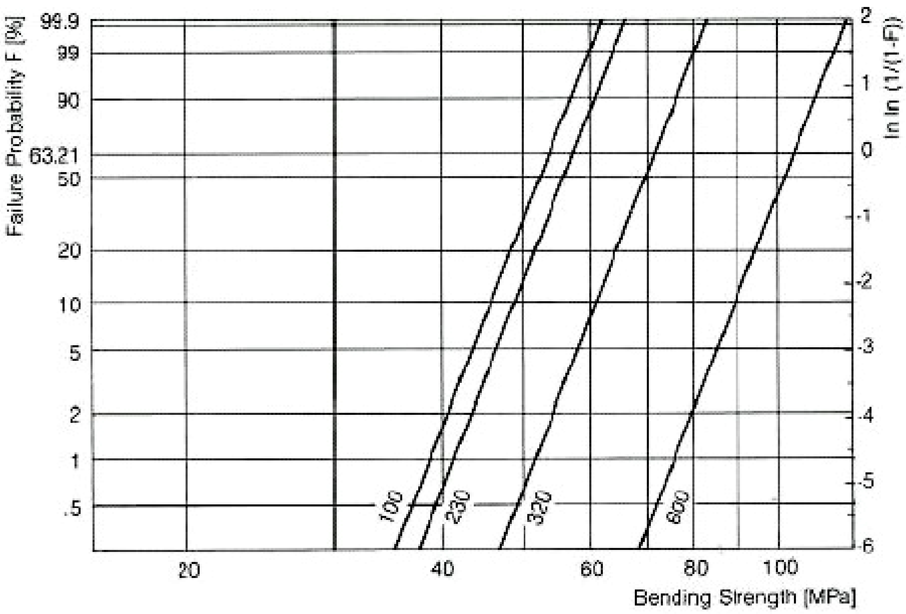
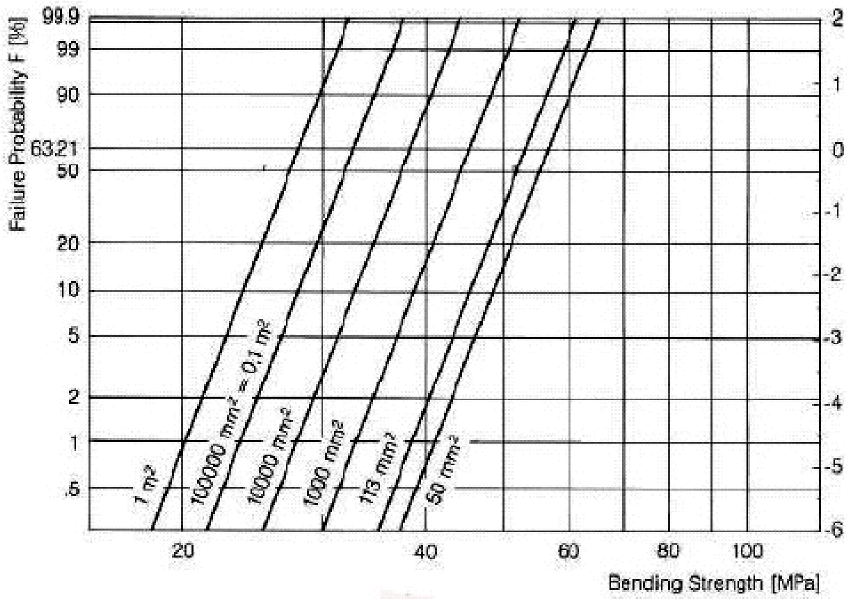


Figure 43.15-11 - Zerodur®: Failure probability of Zerodur for test surfaces processed with loose grit of different sizes



The values for the test surface area SL = 113 mm2 are measured by test. Test conditions: Rate of stress increase 2MPa/s; test surface processed with D 151, air as surrounding medium. The values for the other test surfaces are calculated.

Figure 43.15-12 - Zerodur®: Failure probability, F, as a function of test surface area, SL

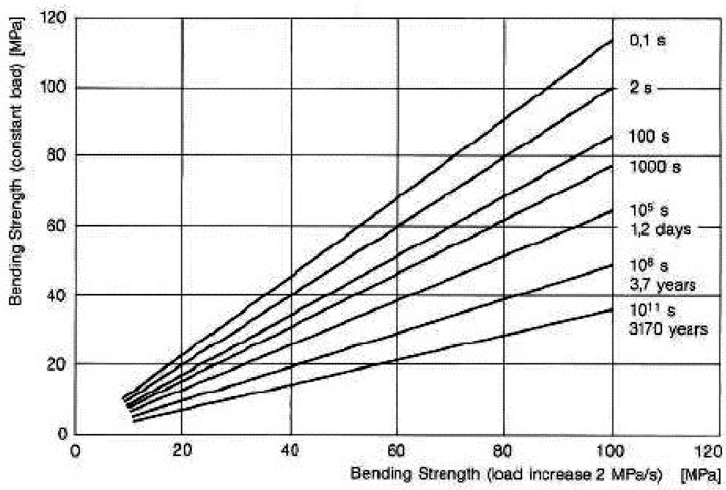


Figure 43.15-13 - Zerodur®: Bending strength under constant load

43.15.6.5 Fracture toughness

In the 'mathematical model' presented, the strength of optical glass was considered from a statistical point of view by measuring the bending strength of samples and subsequently estimating the failure probabilities.

Looking at a single flaw in a material, the maximum bending strength depends on the size of the flaw and geometry in the material. For example in case of a flaw with a short depth in a thick plate with tensile forces acting normal to the crack plane one can define a stress intensity factor KI by the expression:

$$K_I \approx 2\sigma_0 \sqrt{a} \qquad [43.15\text{-}9]$$

Where:

σ_0 *the nominal stress perpendicular to the stress plane*

a *depth of the flaw.*

A flaw results in fracture if:

$$K_I \geq K_{IC} \qquad [43.15\text{-}10]$$

K_{IC} is the critical stress intensity factor for crack mode I (tensile forces normal to the crack plane, crack propagation perpendicular to the forces). K_{IC} is a material constant. For glasses without additional strengthening the value is typically ≤ 1.

Table 43.15.4 provides some fracture toughness values for some glasses, Ref. [43-83], [43-85], [43-96].

Table 43.15-4 - Zerodur®: Fracture toughness of some glasses

Glass	K_{IC} [MPa m$^{1/2}$]
N-BK7	1.1
F5	0.9
ZERODUR®	0.9
SF6	0.7

For a given nominal stress, the plate will break for a critical depth ac determined by:

$$a_c = \left(\frac{K_{IC}}{2\sigma_0} \right)^2 \qquad [43.15\text{-}11]$$

Example:

For the characteristic strength of Zerodur of samples with D64 surface condition $\sigma_0 \approx 64$ MPa, (taken from Table 43.15.1) and Zerodur $K_{IC} \approx 0.9$ MPam$^{1/2}$, the critical flaw size, a_c, is approximately 49µm.

Most glasses exhibit slow crack growth for a stress intensity factor well below the critical value. The most important sub-critical crack growth occurs in the presence of water (amounts less then 10 mg per m^2). The velocity of the crack can be described using, Ref. [43-84]:

$$\frac{da}{dt} = A\left(\frac{K_I(a)}{K_{IC}}\right)^n \qquad [43.15\text{-}12]$$

Where:

a	*the depth of the crack,*
A	*a constant*
n	*stress corrosion constant.*

Typical values for the stress corrosion constant of Zerodur are given in Table 43.15.1.

Typically, the sub-critical crack grow can start from 0.25 K_{IC} .

43.15.7 ZERODUR®: Application examples

Zerodur® has been a favoured material for more than 30 years for mirror substrates of terrestrial and orbital telescopes. Owing to the ability to produce large amounts with reproducible quality, it is suited for future ELT 'extremely large telescopes' projects in the 20m to 100m class.

Some other typical applications include:

- Terrestrial astronomical telescopes, [See: 43.xx].
- Optical bench for LISA pathfinder mission, [See: 43.xx].
- Hubble space telescope secondary mirror.
- JWST - James Webb space telescope (successor to Hubble).
- Mirror substrates and mandrels for X-Ray telescopes.
- Optical elements for comet probes.
- Ring laser gyroscopes.
- Lightweight honeycomb mirror supports.

43.15.8 ULE® Ultra low expansion titanium-silicate glass

43.15.8.1 General

ULE® is produced by Corning Corp. Corning Code 7972 Ultra Low Expansion glass is a titania-silicate glass with a composition of 92.5% SiO_2 and 7.5% TiO_2.

The main characteristics can be summarised as:

- Very low thermal expansion.
- High temperature shock resistance.
- High operating temperature.

Such properties have made ULE of interest for use in various applications needing dimensional precision, e.g. tool reference blocks, components, lasers, solid and lightweight mirror blanks (large astronomical telescopes; space satellites). Both small and large items can be made successfully, e.g. from a few millimetres to several metres.

43.15.8.2 Processing

ULE is made by flame hydrolysis. Pure liquid silicon tetrachloride and titanium tetrachloride are mixed and the vapours are delivered to a furnace, where they react chemically and the glass droplets deposit on a spinning turntable. The resulting material is a transparent glass ceramic with extremely low coefficient of thermal expansion (near-zero at RT) providing dimensional stability. CTE variation within each batch are typically <15 ppb/°C, radially and axially.

43.15.9 ULE®: Properties

43.15.9.1 General

Table 43.15.5 gives a summary of engineering data, Ref. [43-106].

Table 43.15-5 - ULE®: Summary of engineering data

PROPERTY	UNITS	VALUE
Mechanical		
Density	gm/cc	2.2
Hardness	Knoop	49
Tensile Strength	kpsi	-
Modulus of Elasticity	psi x 10^6	9.8
Flexural Strength	kpsi	-
Compressive Strength	kpsi	-
Poisson's Ratio	-	.17
Fracture Toughness	MPa $m^{1/2}$	-
Electrical		
Dielectric Strength	ac volts/mil	-
Dielectric Constant	(at 1 MHz)	-
Volume Resistivity	ohm-cm^2/cm	-
Thermal		
Coefficient of Thermal Expansion	$x10^{-6}$/°C	0.0+/-.03
Thermal Conductivity	W/m°K	1.35
Specific Heat	cal/g°C	
Max Working Temp	°C	800
Shock Resistance	°C	
All properties are at room temperature unless otherwise noted.		
Engineering data are representative, and are not intended as absolute nor warrantable. Manufacturer's Data shown is blended from multiple sources and therefore illustrates the marketplace.		

43.15.9.2 Mechanical properties

The mechanical properties of ULE are, Ref. [43-108]:

- Ultimate tensile stress, *MOR*: 49.8 MPa.
- Elastic Modulus, *E*: 67.6 GPa
- Shear modulus, *G*: 29.0 GPa
- Knoop hardness, 200 g load: 460 kg/mm^2
- Density, ρ: 2.21 g $/cm^3$
- Poisson's ratio, ν: 0.17
- Softening point (estimated): 1490°C
- Density (25°C): 2.21 g/cm^3, Ref. [43-107]

43.15.9.3 Optical properties

The optical properties of ULE are, Ref. [43-107]:

- Refractive index, Ref. [43-107]
  - nD = 1.4828 (589nm)
  - nF = 1.4892 (486nm)
- Transmission; as shown in Figure 43.15.14 for a 10 mm thick sample, Ref. [43-107].

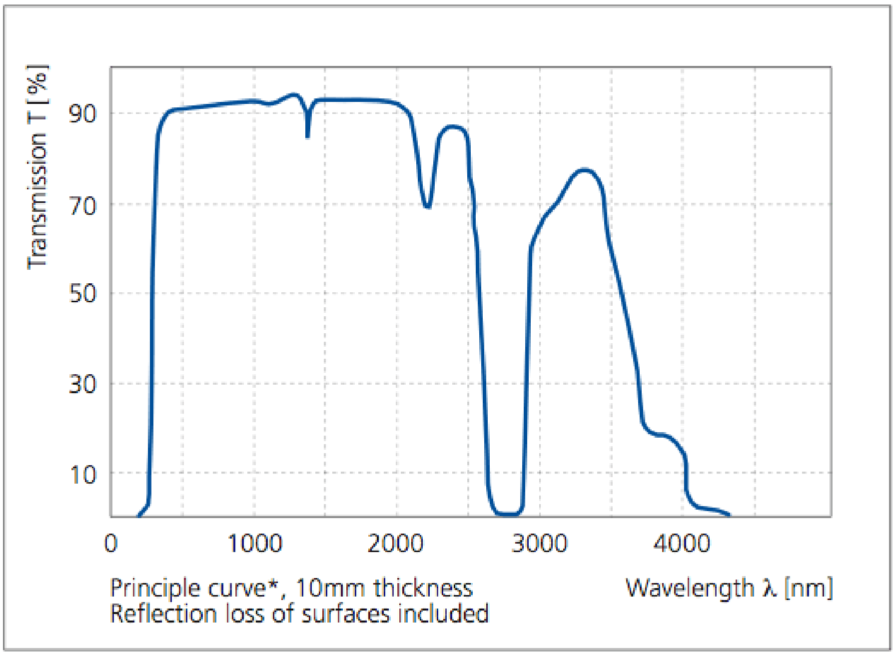


Figure 43.15-14 - ULE®: Transmission curve

43.15.9.4 Electrical properties

The electrical properties of ULE are, Ref. [43-107]:

- Dielectric constant: 3.99 (100Hz; 25°C)
- Loss Factor: 0.00005 (100Hz; 25°C)
- Volume resistivity (D.C.; 200 °C;100Hz; R): 1011.6 Ωcm, Ref. [43-108]

43.15.9.5 Thermal properties

The thermal properties of ULE are, Ref. [43-107], [43-108]:

- Coefficient of thermal expansion (5°C to 35°C): 0 ±0.10 × 10^{-9}/K
- CTE maximum variation (5°C to 35°C): ±30 × 10^{-9}/K
- Thermal conductivity, K: 1.31 W/mK, Ref. [43-108]
- Specific heat, C_p (mean): 767 J/kgK, Ref. [43-108]
- Thermal diffusity, D: 0.0079 cm^2/s, Ref. [43-108]

The thermal expansion characteristics as a function of temperature are shown in Figure 43.15.15 for -100°C to +100°C, Ref. [43-106].

[See also: Figure 43.15.7 and Figure 43.15.8 in which ULE is compared with different materials].

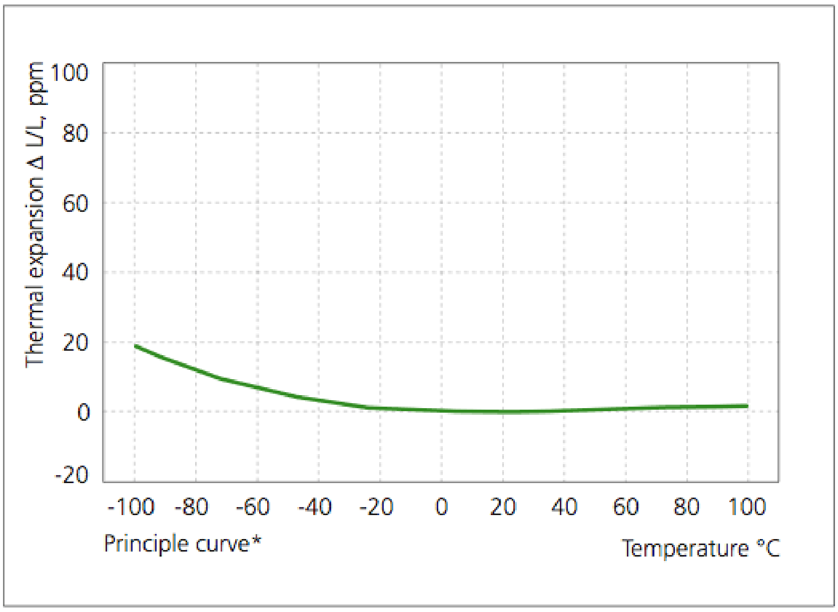


Figure 43.15-15 - ULE®: Thermal expansion characteristics

43.15.10 ULE®: Design

The design approach and principles applied for ULE are the same as those described for Zerodur, [See: Zerodur: Design].

43.15.11 ULE®: Application examples

43.15.11.1 General

ULE zero-expansion glass was originally developed for the space industry to ensure the critical performance of optical systems at the extreme temperatures encountered during space missions.

Many of the major observatories world-wide have primary mirrors made of ULE.

Some typical applications include:

- Ultra low expansion substrates

- Length-standards
- Lightweight honeycomb mirror mounts
- Astronomical telescopes
- Precision measurement technology
- Laser cavities

43.15.11.2 Hubble space telescope – main mirror

The Hubble main telescope mirror used ULE glass as a structural material. To reduce weight, the front and back mirror plates were fused to a honeycomb core design. The 'light-weighting' techniques used for the Hubble mirror have been extended to other space programs and many ground-based astronomy applications. The weight of these large mirrors, therefore, is reduced to one eighth the weight of an equal sized solid mirror.

43.15.11.3 Future applications

Current and future space-based telescopes envisage mirrors that are less than 10 percent of the weight of an equal sized solid mirror.

The technology has been adapted for use in the GOES Weather Satellites.

43.16 Speciality materials

43.16.1 Introduction

Although classed as technical ceramics, there are a wide variety of other materials that offer particular characteristics of potential interest for use in optical equipment and structures, including:

- Beryllia.
- Sapphire.
- Synthetic diamond.

43.16.2 Beryllia

Beryllium oxide, BeO, is an electrical insulator with a high thermal conductivity of about 250 Wm/K. These properties have made it of interest in the thermal management of electronics. It is also used in microwave applications, for items such as windows and radomes. The low thermal neutron absorption makes it useful in the nuclear industry.

An example of a commercial >99.5% pure BeO grade is Ceradyne Ceralloy™ 418, a high thermal conductivity material for use in applications needing heat transfer coupled with dielectric properties, e.g. electronic and microwave tubes and IR high-intensity arc lamps, Ref. [43-38].

Whilst offering some interesting characteristics, the known human toxicity of beryllia means that very specialised handling is needed during processing. This was a major incentive for the development of alternative high thermal conductivity ceramics, e.g. aluminium nitride grades.

43.16.3 Sapphire

Synthetic sapphire is an essentially 100% pure, transparent form of alumina consisting of single-crystal (or polycrystalline), hexagonal, alpha-alumina. The characteristics vary with respect to the optical axis of the crystal.

The general characteristics include:

- Hardness approaching diamond, e.g. Mohs 9,
- Strength, ~2000 MPa (compressive); ~900 MPa (flexural),
- Stiffness, ~400GPa,
- Fracture toughness, ~2 MPa m1/2,
- Wear resistance (scratch and abrasion),
- Wide optical transmission band, e.g. from UV to near-IR,
- High melting temperature, approximately 2050°C ,
- High electrical resistance (insulator),
- Chemically inert,
- Good corrosion resistance, except to some very hot caustic compounds,
- High thermal conductivity, i.e. better than copper at cryogenic temperatures.

Numerous grades of sapphire are commercially-available, these are usually classed by their intended application, e.g. for mechanical or optical applications (then by light transmission frequency).

Sapphire is used for precision mechanical components (bearings, wear-parts, coatings) and within optical systems, e.g. laser systems and windows for imaging systems. Product forms include standard shapes, such as rods, tubes and substrates, along with custom shapes, e.g. opto-electronic devices and LCD screens.

43.16.4 Synthetic diamond

43.16.4.1 General characteristics

The general characteristics of synthetic industrial diamond include:

- Polycrystalline material,
- Very hard (Mohs 10),
- High stiffness,
- High thermal conductivity,
- Low CTE,
- Good chemical resistance (acids and bases),
- Electrical insulator,
- Useful optical properties, e.g. transparent to visible, infrared and UV.

- These combined properties have made synthetic diamond of interest for a wide range of industrial applications, e.g. abrasives and cutting tools to coatings and shaped parts for optical imaging systems and, recently, tweeter cones in loud-speakers, Ref. [43-9].
- The main types of synthetic diamond can be grouped as:
- Abrasives,
- Coatings,
- Shaped parts.

43.16.4.2 Coatings

Coatings are deposited onto other materials, usually to improve wear and abrasion resistance. Depending on the proprietary processes used, diamond or DLC 'diamond-like coatings' can be deposited onto polymers, metals, ceramics and glass, Ref. [43-12].

43.16.4.3 Shaped parts

Proprietary CVD chemical vapour deposition techniques are used to create polycrystalline diamond shapes up to a few mm thick, Ref. [43-9], [43-12].

[See also: 43.17].

43.16.4.4 Developments

Boron-doped diamond has p-type semiconductor characteristics, with potential uses in the electronics industry, Ref. [43-37].

Single-crystal synthetic diamond, in useable sizes, is under development as a potential replacement for silicon in high-temperature, high-power electronics, Ref. [43-37].

43.17 CVD diamond

43.17.1 Introduction

Since natural diamond flew as a crucial component in one of the Pioneer Venus experimental payloads in the mid-1970s, diamond technology has advanced considerably.

Using proprietary CVD chemical vapour deposition techniques, thin plates and shapes in high-purity synthetic diamond can now be produced. This has greatly increased the potential applications for diamond, as many were impractical due to size and shape using natural material.

Excluding abrasives, some examples include, Ref. [43-8], [43-9], [43-10], [43-11]:

- Optical applications, e.g. windows for missile systems, uses in high-power laser systems, infrared imaging and process control and analysis, high-power microwave systems.
- Thermal management products; e.g. substrates for high-power electronic devices.
- Wear parts, e.g. bearings, seals and cutting tools for advanced materials such as MMCs and CMCs.
- Cutting blades for precision industrial and medical uses.

- Parts for analytical and research equipment, operating at very high pressures (0.5Mbar) and extreme temperatures (both high and cryogenic).

43.17.2 Typical characteristics

Most synthetic CVD diamond is polycrystalline material and many of its final properties are influenced by the grain structure, e.g. size, orientation, intergranular purity and presence of micro stresses. These are in turn largely controlled by the process conditions.

Although single-crystal synthetic diamond is available, the sizes available are much smaller and so of less interest for thermo-mechanical engineering applications.

All diamond products are extremely hard and abrasion resistant, but the bulk mechanical, optical and thermal properties of materials from different sources can vary significantly.

Commercially-available CVD diamond materials can be grouped as, Ref. [43-8]:

- Optical grades: optically transparent with disc thicknesses from 0.3 mm to 1.2 mm and diameters typically up to 120 mm,
- Mechanical grades: darker in appearance with better mechanical properties, i.e. strength. Plates up to typically 140 mm diameter.
- Thermal grades, which are a compromise between thermal conductivity and cost.

43.17.3 Typical properties

43.17.3.1 General

Table 43.17.1 gives some typical properties for a commercial optical CVD diamond grade; Diafilm™ OP, produced by Element Six, Ref. [43-9].

43.17.3.2 Mechanical properties

The microstructure of CVD diamond is linked to the synthesis techniques and growth conditions. In thicker products it can also vary during the processing; in particular the grain size at the growth surface is coarser than that at the nucleation surface. Consequently the strength has a strong dependence on the thickness, and the critical flaw size is related to the grain size on the face tested. Strength values obtained from tests show good reproducibility.

Table 43.17-1 – Technical ceramics: CVD diamond – typical properties

Property	Diafilm™ OP	Comments
Density (kg/m^3)	3515	
Mechanical:		
Hardness (GPa)	81 ± 18	
Fracture toughness (MPa $m^{1/2}$)	5.3 to 7.0	
Young's modulus (GPa)	1000 to 1100	
Poisson's ratio	0.1	
Tensile strength (MPa): Nucleation surface Growth surface	 800 [11] 400 [23]	 Fine grain Coarse grain
Physical:		
Refractive index	2.375	at 10μm
Thermal expansion coefficient (ppm/K)	1.0 4.4	at 300 K at 1000 K
Thermal conductivity (W/m K)	1900 to 2200 1100	at 300K at 500K
Thermal shock *FOM* (1) (x 10^3 W/m)	1000 (approx.)	[See: Key]
Specific heat capacity (J/gK)	0.52	300K
Dielectric constant, D	5.68 ± 0.15	35 GHz
Loss tangent (10^{-6})	8 to 20	145 GHz
Environmental:		
Rain impact DTV (m/s)	525	2 mm drop size
Sand erosion (mg/kg)	21 ± 6	at 100m/s C300/600 sand

Key: (1) $FOM = S(1-\nu)\, k / \alpha\, E$; where: α = thermal expansion coefficient; E = Young's modulus; ν = Poisson's ratio; S = strength; k = thermal conductivity

For optical and mechanical grades, the Weibull modulus (for tensile strength), determined from the results of 3-point-bend fracture tests, is given in Table 43.17.2, Ref. [43-8]. Values corrected for thickness variation are also included. A high value indicates a reproducible strength. For comparison purposes, typical values for other competitive materials are: sapphire: 2.1, zinc sulphide: 5.4, silicon carbide: 10, Ref. [43-8].

Table 43.17-2 – Technical ceramics: CVD diamond – Weibull modulus

CVD diamond grade	Growth surface (thickness corrected)	Nucleation surface
Mechanical	9.8 (11.6)	6.5
Optical	11.6 (23.1)	11

Typical stiffness values for material grades at room temperature are shown in Table 43.17.3, Ref. [43-8].

Table 43.17-3 – Technical ceramics: CVD diamond – Young's modulus

CVD diamond grade	Room temperature Young's modulus (GPa)
Mechanical	1166
Optical	1133

Young's modulus for CVD diamond materials is linked to the occurrence of defects. Materials with high concentrations of micron-size voids and microcracks give the lowest values, emphasising the need for rigorous process control. It also shows the importance to designers of accurate property values for material grades, Ref. [43-8].

The effect of increasing temperature on modulus is more-or-less insignificant between room temperature and 700°C; a 5% loss typically. At higher temperatures, the modulus drops as the diamond begins to oxidise.

43.17.3.3 Optical properties

[See: Application examples for some thermo-optical properties of CVD diamond]

43.17.3.4 Electrical properties

[See: Application examples for some dielectric properties of CVD diamond]

43.17.4 Application examples

43.17.4.1 Pioneer Venus mission

Natural diamond was used for the window of the infrared radiometry experiments carried by the large probe of Pioneer Venus because of its resistance to the planet's extremely hostile atmosphere, Ref. [43-8]. The Pioneer Venus multiprobe comprised of a bus that carried one large and three small atmospheric probes. In early-December 1978, all the probes entered the Venus atmosphere at about 11.5 km/s and then decelerated. Parachute deployment occurred at about 47 km altitude. The large probe, about 1.5m overall diameter, carried 7 science experiments, within a sealed spherical pressure vessel (≈0.7m diameter), Ref. [43-43], including an infrared radiometer to measure distribution of infrared radiation, which had a natural diamond window, Ref. [43-8].

Although an example of the use of natural (single-crystal) diamond, it demonstrates the material's combined optical and severe environmental resistance in space. After 25+ years of further research and development, synthetic polycrystalline diamond materials can now attain such characteristics, but in much larger, useable sizes.

43.17.4.2 Missile infrared imaging windows

One application which exemplifies diamond's combination of optical properties and mechanical performance in hostile environments is IR imaging windows for air-launched missiles. These are dome-shaped structures, about 70 mm diameter, forming the nose cone of the missile. As well as performing their aerodynamic and IR optical roles during the short, high-velocity missile flight, they resist environmental degradation for much longer periods while mounted on the carrier aircraft.

The important properties for materials in this role are:

- good optical transmission characteristics in the imaging system waveband,
- high strength and stiffness, to support high flight loads (on-aircraft and after deployed),
- precise shape and good geometrical tolerances, for accurate imaging,
- good environmental resistance to the impact of rain and sand at high velocities,
- good thermal shock resistance to cope with the high thermal gradients generated during acceleration and manoeuvres.

Materials traditionally used for LWIR long wavelength infrared (8μm to 14μm) detection systems are Zinc Sulphide and Germanium. These have poor erosion resistance to rain and sand, so coatings are applied to improve their performance.

A design-development exercise manufactured optical quality CVD diamond windows in the form of free-standing domes, 70mm diameter and over 1mm thick. The radius of each surface was machined to ± 1μm, Ref. [43-8].

In addition to IR, diamond also has high transparency in other spectral ranges (wavelengths from 0.3μm to 3.0μm and 7.0μm to >500μm), enabling it to be used in multispectral windows, e.g. combined visible, IR imaging and laser ranging systems. Other materials tend to have a much more limited transparency range, e.g. 0.4μm to 11.5μm for ZnS, depending on the grade and 1.8μm to 23.0μm for Ge.

For missiles, the thermo-mechanical characteristics of CVD diamond are superior to other materials. Diamond has very high thermal conductivity and low thermal expansion, which makes it very resistant to thermal shock. Thermal shock for CVD diamond can attain a FOM value ($\times10^3$ W/m) of 1000, whereas Ge is 6.1 and ZnS grades are about 2.5. The maximum extended window operating temperature (in air) for CVD diamond is 1023K, compared with 873K for ZnS. Above these temperatures, mass loss occurs due to oxidation.

The environmental erosion resistance of CVD diamond is considerably better than other materials for this application. Impacts from high-velocity water drops, simulating operational rain impact, can seriously affect the mechanical strength of windows and reduce their transmission capabilities, hence imaging quality.

Repeated sand-grain impact erodes the material surface and degrades the optical transmission characteristics. The hardness and abrasion resistance of CVD diamond makes it the most resistant of all the IR materials, including sapphire (used for 3μm to 5μm spectral band); as shown in Figure 43.17.1, Ref. [43-8].

Retained transmission for CVD diamond is different depending on which production surface is exposed to sand erosion, i.e. upper bound is 'nucleation surface'; lower bound is 'growth surface'.

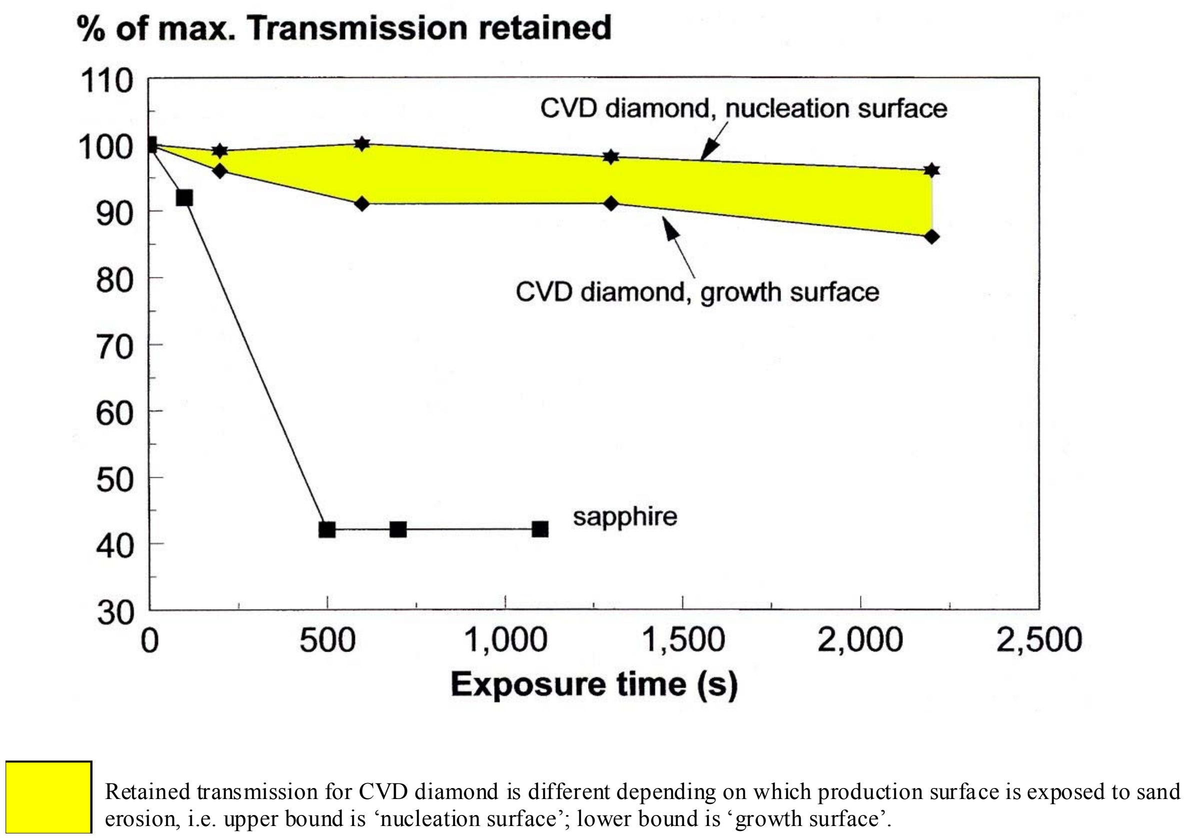


Figure 43.17-1 – Technical ceramics: CVD diamond – sand erosion

43.17.4.3 High-power laser systems

A common problem for high-power laser windows made from conventional materials, such as Zinc Selenide (ZnSe), is thermal distortion of the beam. This is caused by thermally-induced refractive index gradients where the window is heated by the transmitted beam, Ref. [43-8], [43-44].

The characteristics of optical grade CVD diamond (high thermal conductivity, low absorption coefficient, low temperature coefficient of refractive index) minimise these effects, enabling its use in high-power CO_2 lasers systems for exit windows, output couplers, beam splitters and lenses, Ref. [43-8], [43-44].

Table 43.17.4 compares the typical properties of CVD diamond with ZnSe, materials of interest for high-power laser systems, Ref. [43-8].

Table 43.17-4 – Technical ceramics: CVD diamond – typical optical properties for high-power laser systems

Property	CVD diamond (optical grade)	ZnSe
Thermal conductivity (W/mK)	2000	17
Absorption, at 10.6µm (/cm)	0.05 (1)	0.0005 (2)
dn/dT (10^{-6} /K)	10	57
Key: (1) Values range from 0.1 to 0.03; (2) Although the bulk absorption of ZnSe is lower, in practice the applied coating system results in considerably higher temperature rises in ZnSe windows than in coated CVD diamond under test comparable conditions.		

43.17.4.4 Microwave windows

Low dielectric loss materials are used for the output windows of high-power microwave tubes. These are also structural components as they act as vacuum barriers. All window materials suffer heating effects to some extent in these applications, and in practice, this is countered by cooling systems. For synthetic diamond windows, thermal conductivity decreases with increasing temperature, which can provoke a thermal runaway effect. However, this can be countered using simple water-cooled edge systems. For severe cases with sapphire windows it can involve the use of expensive cryogenic coolants, Ref. [43-8].

As a result of development work on improving the dielectric properties of CVD diamond (in the mm wave band), it is now used for Gyrotron tubes operating from 70GHz to 170GHz, with output powers in excess of 1MW, Ref. [43-8].

For the application considered, the particular demands to be met included:

- Size: window 100 mm diameter and 1.6mm to 2.3mm thick, depending on microwave frequency.
- Dielectric loss: relatively uniform over the window area to avoid 'hot spots'.
- Hermetic joining of the diamond window into a metal flange to sustain high vacuum.

Modelling of the thermal performance of CVD diamond windows transversed by a 1.2 MW beam showed that for materials with a dielectric loss value of tan $\delta < 10^{-4}$, the central temperature rise was less than 240°C with a simple water-cooled edge, which is tolerable for diamond.

Table 43.17.5 gives some typical properties of commercially-available Diafilm® dielectric grade CVD diamond, Ref. [43-8].

For high-power Gyrotron tube applications, the predicted temperature rise in dielectric grade materials is under 15°C. This means that CVD diamond dielectric grades can perform above 1MW, and also in continuous wave mode at powers around 0.5 MW without the need for cryo-cooling.

Table 43.17-5 – Technical ceramics: CVD diamond – typical dielectric properties for high-power microwave systems

Property (2)	Diafilm™ (1) (dielectric grade) (3)	Comments
Loss tangent, tan δ ($\times 10^{-6}$)	20 to 50	Typical values, maximum value 100×10^{-6}
Equivalent absorption, (/cm)	0.0014 to 0.0035	Typical values, maximum value 0.007/cm
Relative permittivity, εr	5.68	Typical value
Key: (1) Diafilm is a tradename of Element Six (UK, NL); (2) Data measured at frequency = 145 GHz (wavelength, λ = 2.1 mm); (3) available sizes: up to 120mm diameter and 2.25mm thick.		

43.17.4.5 Cutting blades

The ability to retain a sharp edge during repeated use, coupled with hardness and wear-resistant properties has made CVD diamond attractive for precision cutting blades for both industrial and medical uses, including, Ref. [43-8]:

- fibre optic assembly, where a diamond cutting blades produce clean cleavage of optical-grade glass filaments.
- medical applications, e.g.
  - Ultra-sharp diamond scalpels for opthalmic surgery.
  - Laser scalpels, where the diamond blade is combined with a laser cauterising system.

Ultramicrotamy for the preparation of very thin, typically 100nm thick, slices of tissue for clinical analysis.

43.18 Cesic®: NIRSpec optical bench

43.18.1 Introduction

Under ESA contract, Alcatel Space (F) and ECM (D), designed, manufactured and tested a flight-representative optical bench made of Cesic®. This was an application for the NIRSpec instrument on the James Web Space Telescope.

The optical bench was successfully submitted to intensive shaker vibration tests, up to 80 g, without problem and showed very high stability when tested down to a temperature of 30 K.

43.18.2 Material characteristics

43.18.2.1 Ceramics

Ceramic materials are of interest for space instruments because of their characteristics, including:

- High specific stiffness and large mechanical dimensional stability (no residual deformation), and thermal and thermo- elastic performances.
- High thermal conductivity, high $\lambda/\alpha a$ ratio enabling through thermal conduction, good thermo-elastic behaviour, homogeneity, low coefficient of thermal expansion and remarkable cryogenic behaviour.

[See: 43.1]

43.18.2.2 Cesic®

From amongst the many different types of ceramic materials, Cesic® from ECM in Germany was selected by Alcatel Space as the best candidate for use in the development of structures and mirrors of future space instruments. Knowledge of the material itself, along with all the associated processes, enabled Cesic® to be considered as a mature technology.

Cesic® is a ceramic made of SiC, Si and C. It is made by the reaction, at high temperature between a C-C green-shape and liquid silicon, Ref. [43-57].

[See also: 43.12]

43.18.3 Development of optical bench

43.18.3.1 NIRSpec

During the NIRSpec development phase for the JWST James Web Space Telescope project, a large design-development programme was initiated under ESA contract, with Alcatel Space, in France, and ECM, in Germany.

43.18.3.2 Development programme

The aim of the developments programme was to:

- Manufacture and test an optical bench representative of NIRspec in dimensions, shape and design in order to:
  - demonstrate the feasibility of the concept,
  - validate the entire manufacturing processes.
- Based on an elementary characterisation of Cesic® material, to demonstrate that the material is homogenous enough to avoid any unwanted distortion at cryogenic temperatures.
- Perform vibration tests to demonstrate that:
  - The equipped bench can support the mechanical launch environment without damage.
  - Validate loads introduced by the bench to equipment due to its damping behaviour.
- Verify bench stability under tests down to the cryogenic working temperature of 30 K.

43.18.3.3 Demonstrator structure

An optical structure, fully representative of the NIRspec optical bench, except for size, was designed, dimensioned and manufactured successfully. The bench produced was 1045mm × 600mm × 100mm and the mass was 23kg.

The design and size constraints used for the bench were the optical and launch loads, but also took into account the flexibilty of manufacturing parts with Cesic®; notably by producing a half-closed back design to maximise stiffness, I-shaped stiffeners, thin ribs to minimise mass, C-C blocks at interface junctions and holes cut using EDM electro-discharge machining techniques.

Figure 43.18.1 shows the main features of the demonstrator optical bench produced.

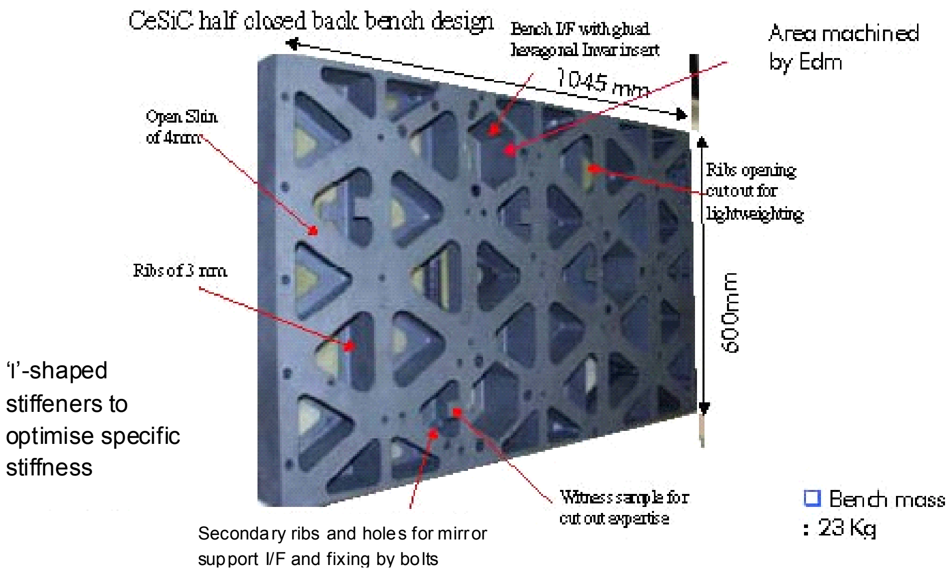


Figure 43.18-1 – NIRSpec optical bench development: Cesic ceramic demonstrator structure

43.18.3.4 Mechanical testing

To support the design and validate the proper sizing, representative technology samples were tested up to ultimate loads; as shown in Figure 43.18.2.

The tests results confirmed the strength of the joint area and showed no loss of stiffness and strength. The test results also showed good correlation with the FEM finite-element model, including failure loads.

Representative fully-instrumented bench cells have also been tested up to ultimate load.

Figure 43.18.3 show the test set-up. These tests established a large safety factor margin for the designed flight bench: factor of safety 3.

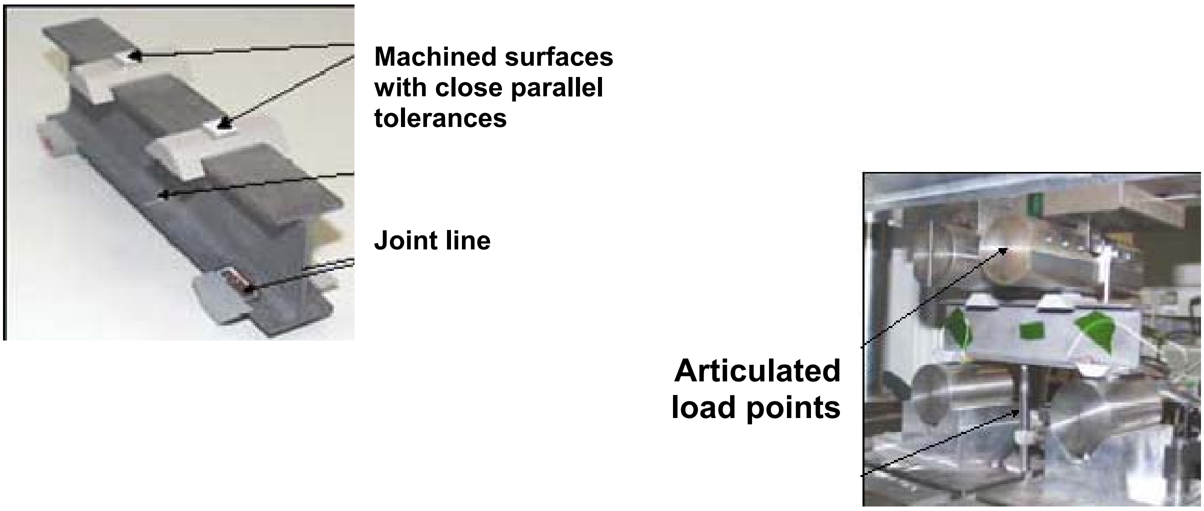


Figure 43.18-2 – NIRSpec optical bench development: Test set-up and I-beam sample with joint line

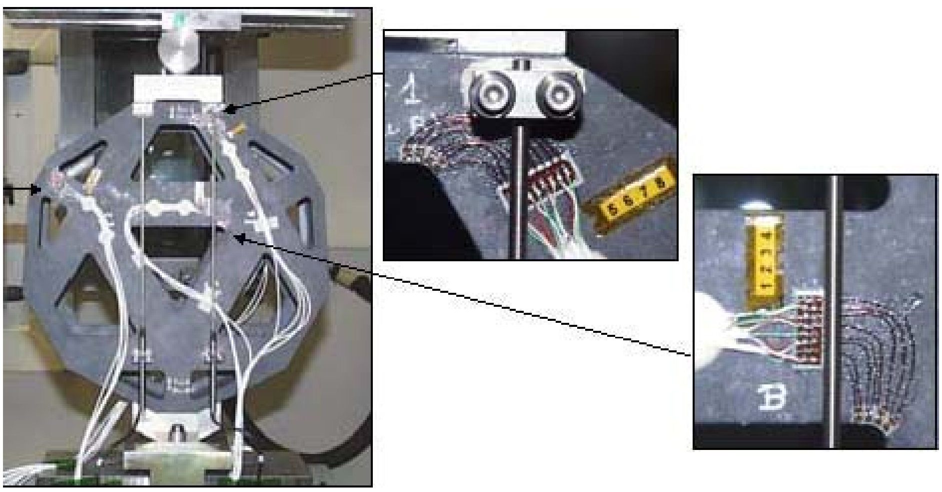


Figure 43.18-3 – NIRSpec optical bench development: Test set-up for instrumented bench cells

43.18.3.5 Vibration testing

The entire bench was tested under vibration. The objectives of the optical bench vibration tests were to:

- Identify the dynamics behaviour of such representative flight bench, e.g. amplification, damping coefficient.
- Validate the stiffness of the bench.
- Validate the strength and identify the eventual margin for the allowable strength necessary for the sizing of a future NIRspec flight bench.

To establish the strength and margins, the bench underwent vibration testing up to the maximum sinus level possible by the shaker, i.e. more than 80g at shaker level. The vibration levels imposed on the test bed exceed the qualification loads experienced by a full-sized flight bench, the bench was therefore qualified for flight.

Figure 43.18.4 shows the bench installed on the shaker.

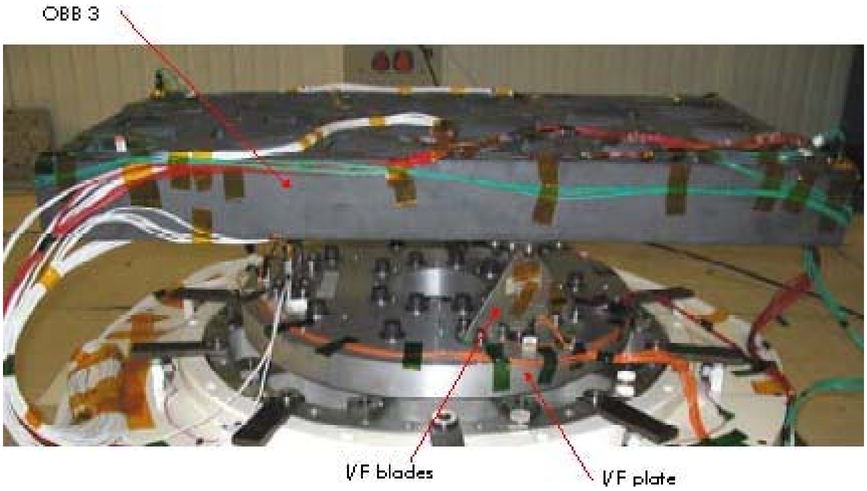


Figure 43.18-4 – NIRSpec optical bench development: Test set-up for 'shaker' vibration tests

The FEM prediction was corroborated by the test results, which confirmed the material properties and also the ability to predict stiffness, stress and strength behaviour .

The bench eigenfrequency is above 500Hz and the amplification factor, measured during OBB sine test, is less than Q=30, so limiting loads induced on critical equipment under vibration and notching.

43.18.3.6 Cryogenic testing

The stability of the optical bench was demonstrated by testing at cryogenic temperatures; from 300K down to 30K. The test was performed in CSL (Belgium) in focal V chamber. The bench was cooled through cryo-helium panels by a radiative and conductive link. Figure 43.18.5 shows the bench installed in the cryogenic test chamber.

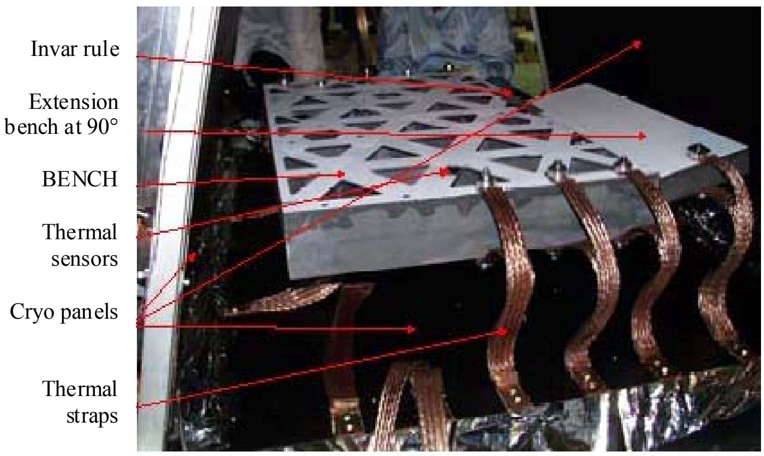


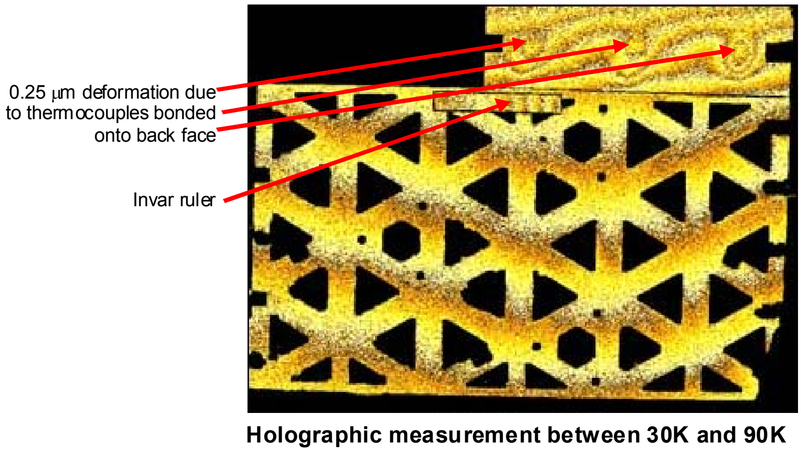
Figure 43.18-5 – NIRSpec optical bench development: Test set-up for cryogenic temperature test

During the test, the in-plane contraction and the out-of-plane stability was measured by a speckle holographic interferometric method. This technique provides an accuracy of around 1μm on the whole cycle with a resolution of 0.1μm. The measurement was performed by HOLO3.

During the cryogenic test, all the criteria were met successfully, i.e.:

- Temperature of less than 30 K was reached by OBB.
- Thermal contraction was as predicted from measurement of samples:
  - contraction was homogeneous on the whole bench surface; as shown in Figure 43.18.6.
  - bench deformation was zero from 100K to 30K; as shown in Figure 43.18.7, where all the curves from different positions are combined to also show that the contraction was homogeneous over the surface.
- Out-of-plane deformation was quasi-zero between 293K and 30K; as shown in Figure 43.18.8. This enables good optical alignment to be maintained during cool down from 300K to 40K. No hysteresis was observed during the entire thermal cycle.

The overall results of the cryo testing demonstrated that the bench structure made of Cesic® was highly stable. It was concluded that Cesic® is a suitable material for making dimensionally-stable structures operating under cryogenic conditions.



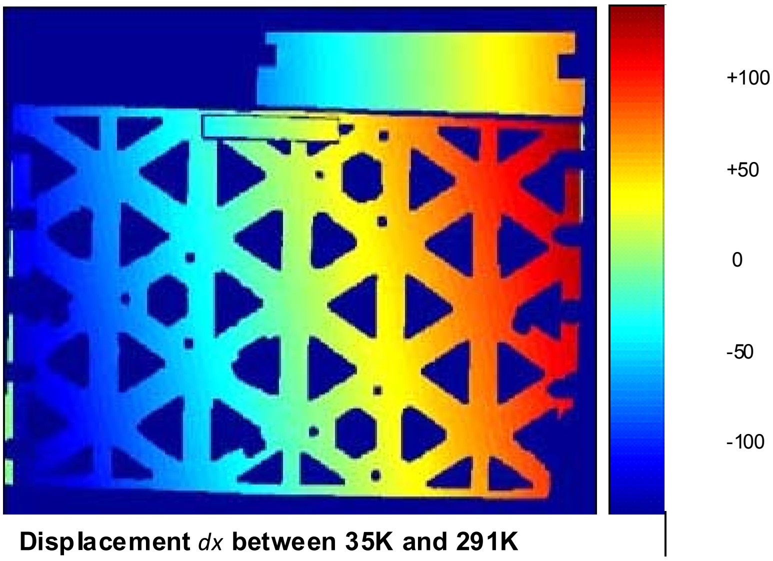


Figure 43.18-6 – NIRSpec optical bench development: Thermal contraction under cryogenic test

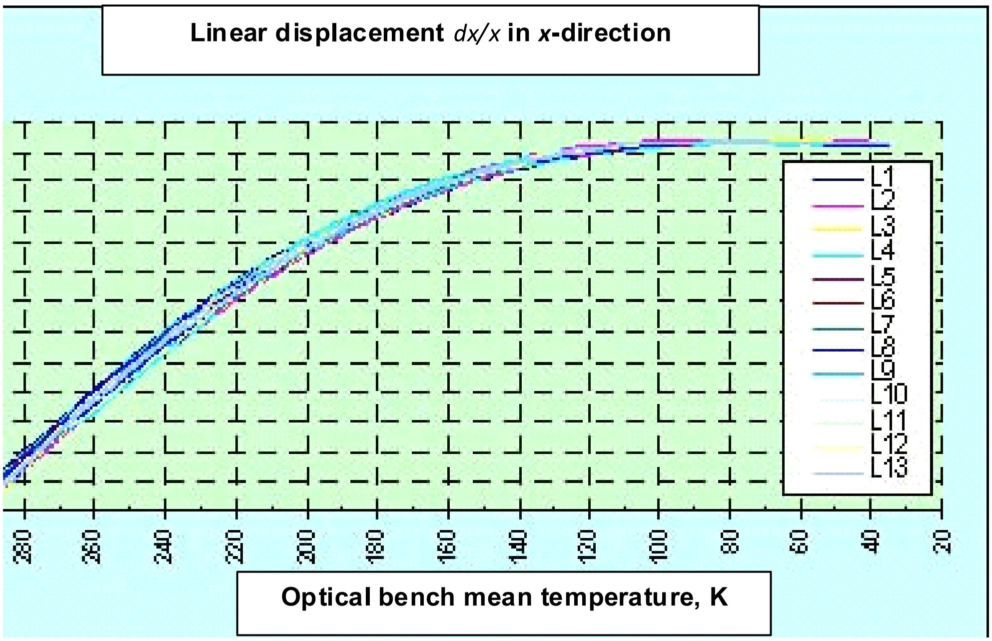


Figure 43.18-7 – NIRSpec optical bench development: Linear displacement under cryogenic test

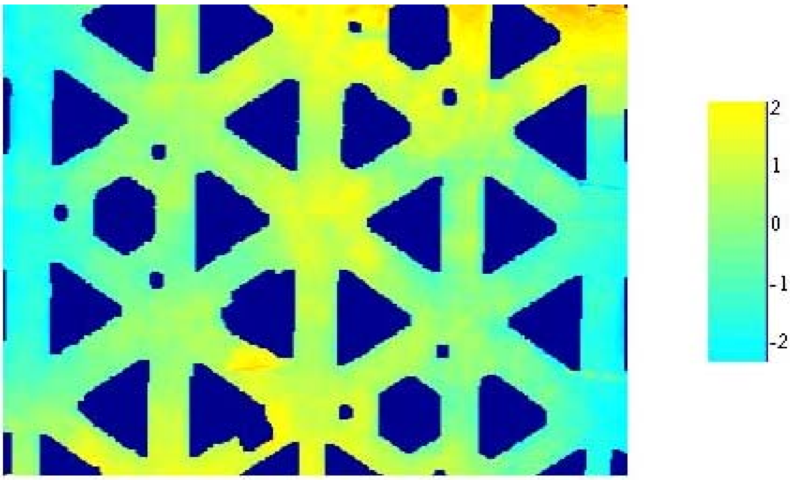


Figure 43.18-8 – NIRSpec optical bench development: Out-of-plane displacement under cryogenic test

43.18.4 Conclusions

The large development study, performed jointly by Alcatel Space and ECM and supported by ESA, demonstrated that Cesic® is a mature technology which can be considered for applications needing:

- Structural optimised-shaped components, e.g. mechanical performance combined with the processing flexibility to produce mass-efficient components, i.e. very high *EI/M* ratio,
- Mechanical properties, e.g. high strength, insensitive to fatigue, good surface finishes, at joints and interfaces, bolted joints under high loads.
- Processing using an established material production route, along with accurate machining by EDM to provide precision interfaces.
- Mirror manufacturing capability, i.e. able to produce large lightweight and stiff mirrors; mirror with integrated filtering MFD.
- High-stability performance under cryogenic temperatures for dimensionally stable structures, e.g. optical and telescope supports.
- Cost and time-efficient manufacturing.
- Potential to evolve further, e.g. manufacturing of more complex shapes and structures; enhanced properties.

43.19 Zerodur®: Applications case study

43.19.1 Introduction

For over 30 years, Zerodur® has been a material favoured for mirror substrates of terrestrial and orbital telescopes. More than 200 astronomical mirror blanks with diameters between 1m and 2m have been produced, Ref. [43-68].

The ability to produce large amounts with reproducible quality makes it suitable for future ELT 'extremely large telescopes' in the 20m to 100m class.

Modern fabrication technologies enable the production of structures with more than 65% weight reduction. Owing to its good homogeneity of coefficient of thermal expansion, Zerodur is also used as a mandrel material for shaping mirror shells, such as those envisaged in future X-ray telescope projects

The material has been used in a large number of optical instruments for different space missions, including the secondary mirror of the Hubble Space Telescope. It is also intended to be used in its successor; JWST 'James Webb Space Telescope'.

Some further applications for space include:

- Mirror substrates for X-ray telescopes.
- Optical elements for comet probes.
- Ring laser gyroscopes.
- Lightweight honeycomb mirror supports.

43.19.2 Terrestrial mirrors

A general overview on 100 mirror telescopes made of Zerodur is given in Ref. [43-68].

Table 43.19.1 gives an overview of large terrestrial mirror blank projects, Ref. [43-68].

Table 43.19-1 – Zerodur®: Overview of terrestrial astronomical telescope mirrors

Project/Site	Dimensions of Primary Mirror Blank	CTE [$10^{-6}K^{-1}$] specification	ΔCTE [$10^{-6}K^{-1}$] specification	samples per blank	Completed
Keck I / Hawaii	43 segments dia. 1900 mm thickness 76,5 mm	+/- 0.10	≤ 0.02	18 (9 from top and 9 from bottom, on two circles)	1990
Keck II / Hawaii	42 segments dia. 1900 mm thickness 75,8 mm				1993
HET / Texas	96 hexagons, width 1019 mm, thickness 56 mm	+/- 0.15	≤ 0.01	4 (2 from top and 2 from bottom per raw casting)	1995
GTC / La Palma	42 hexagons, width 1622 mm, thickness 83,5 mm	+/- 0.05	≤ 0.02	12 (6 from top, 6 from bottom)	2002

43.19.3 Optical bench

Figure 43.19.1 shows a breadboard of an optical bench made of Zerodur for the LISA Pathfinder Mission. Zerodur was selected as a structural material because its thermally-stable behaviour met the extreme requirements to detect gravitational waves.

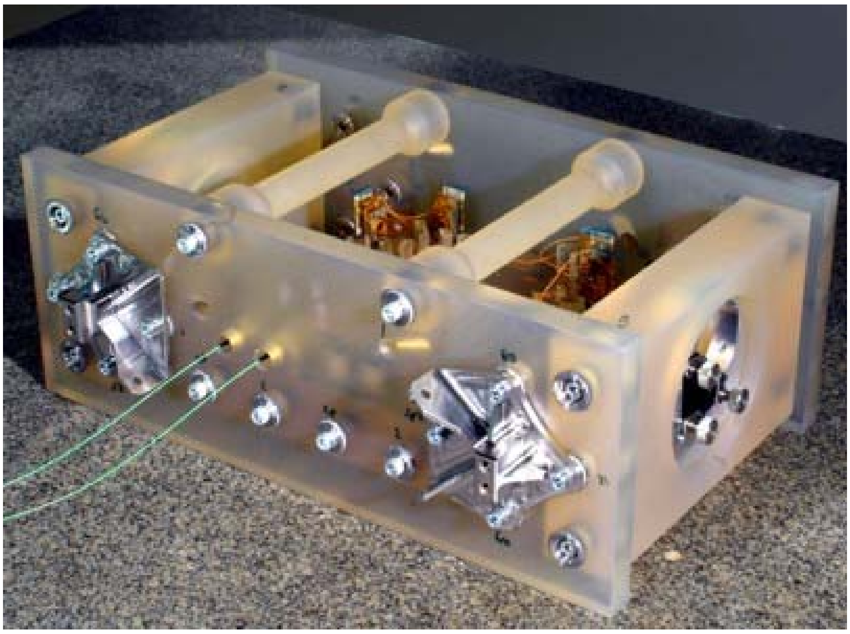


Figure 43.19-1 – Zerodur®: Optical bench for LISA pathfinder mission

43.20 References

43.20.1 General

[43-1] R. Hussey & J. Wilson – RJ Technical Consultants
'Advanced Technical Ceramics – Directory and Databook'
Chapman & Hall, ISBN 0 412 80310 0 (1998)

[43-2] Think Ceramics – Technicshe Keramiker website
(www.keramverband.de)

NOTE Technical ceramic materials classed according to ENV 12212; listed under C ... as per IEC 672.

[43-3] Morgan Advanced Materials and Technology, (USA):
Private communication (2005)

[43-4] CARES software: Extract from NASA website (June 2005): website:
http://www.grc.nasa.gov/WWW/LPB/cares/index.html

[43-5] S.J. Schneider Jnr: Chair ISO/TC 206 – Fine ceramics
'Assessment of international standardisation needs for ceramic materials – Status of ISO/TC 206 Fine ceramics',
ISO Bulletin (Feb 2003)

[43-6] ISO website: www.iso.ch

[43-7] EN60672: Ceramic and glass insulating materials

[43-8] R.S. Sussmann: Element Six – De Beers Ind. Diamonds (UK)
'Applications of Diamond Synthesized by Chemical Vapour Deposition';
Chapter 2 in 'Hard Materials', Editor R. Riedel. Published by Wiley-VCH, (2000), p574 - 622

[43-9] Element Six (NL; UK) (website: www.e6.com)
'Diafilm™ OP datasheet' (2004)

[43-10] H.P. Godfried et al: Drukker International/Element Six (NL)
'Use of CVD diamond in high-power CO2 lasers and laser diode arrays';
In 'Advanced High-Power Lasers' proceedings of SPIE Vol. 3889 (2000), p553-563. ISBN 0277 786X/00

[43-11] C.S. Pickles & R.S. Sussman: Element Six (UK)
'Diamond: a laser's best friend'
Physics World (July 2000), p25-26

[43-12] Morgan Group (UK)
Advanced Ceramics: www.morganadvancedceramics.com
Boron nitride: www.performancematerial.com
Carbons: www.morganamt.com
Silicon carbide (CVD): www.performancematerial.com

[43-13] Saint Gobain Quartz (website www.quartz.saint-gobain.com)

[43-14] Ceradyne Inc. (website www.ceradyne.com)

[43-15] Kyocera Corp. (website www.kyocera.co.jp)

[43-16] St. Gobain (F): Boron nitride (website www.bn.saint-gobain.com)

[43-17] GE Advanced Ceramics (website www.advceramics.com)

[43-18] J. Boylan: Morgan Advanced Materials & Technology
'Carbon-Graphite Materials'
Materials World, Vol. 4, no. 12 (December 1996), pp. 707-708

[43-19] J. Boylan: Morgan Advanced Materials & Technology
Private communication (June 2005)

[43-20] SGL Carbon (D) website (website www.sglcarbon.com)

[43-21] Carbone Lorraine (France) website (www.chem.carbonelorraine.com)

[43-22] Schunk (D) website (www.schunkgraphite.com)

[43-23] Dr. E. Silverman: Northrop Grumman Space Technology (USA)
'Multifunctional Carbon Foam Development for Spacecraft Applications'
SAMPE Journal, Vol. 41, No. 3 May/June 2005

[43-24] Poco Graphite Inc. (USA) website (www.poco.com)

[43-25] M.M. Rowe et al: TRL – Touchstone Research Laboratory, (USA); website (www.trl.com)
'Case studies of carbon foam tooling'
SAMPE Journal, Vol. 41, No. 4 July/August 2005.

[43-26] Toshiba Ceramics (Japan) website (www.tocera.co.jp)

[43-27] Boostec (France) website (www.boostec.com)

[43-28] Ultramet (USA) website (www.ultramet.com)

[43-29] Schafer Corp (USA) website (www.schafercorp.com)

[43-30] Dynamic Ceramic Ltd. (UK) website (www.dynamcer.com)

[43-31] SCT (France) website (www.sct-ceramics.com)

[43-32] CoorsTek (USA) website (www.coorstek.com)

[43-33] Corning Corporation (USA) website (www.corning.com)

[43-34] B. Ackerman et al: Corning Corporation (USA)
'Improved characteristics of ULE® glass for meeting EUVL needs'; SPIE Conference 2003

[43-35] Schott AG (D) website (www.schott.com)

[43-36] Zerodur® - Schott Lithotec AG (D) website (www.schott.com)

[43-37] A. Yarnell
'The many facets of man-made diamonds'
Chemical and Engineering News, February 2, 2004

[43-38] MatWeb website (www.matls.com)

[43-39] LigaFill™ ceramics foams: Fraunhofer Institute for Ceramic Technologies and Sintered Materials website (www.ikts.fhg.de)

[43-40] J. Adler et al: Fraunhofer Institute IKTS, Dresden (D).
'SI-SIC LigaFill™ foams and related net-like structures: New lightweight and low-cost materials for spaceborne applications', Proceedings of EUROMAT 99, Vol. 1 - Materials for Transportation Technology; Peter-J. Winkler (Editor)
ISBN: 3-527-30124-0 (April 2000)

[43-41] Dr. M.A. Moore et al: Morgan Crucible Co. Ltd. (UK)
'High temperature insulating refractories: Technical, economic, health & safety and environmental issues'
5th European Conference on Industrial Furnaces and Boilers', Espinho, Porto, Portugal, April 2000

[43-42] Xiangcheng Luo et al: New York State University, USA
'Electronic applications of flexible graphite'
Journal of Electronic Materials, Vol. 31, No.5 (2002)

[43-43] Pioneer Venus mission –from NASA website (2005)
http://nssdc.gsfc.nasa.gov/planetary/pioneer_venus.html

[43-44] H.P. Godfried et al: Drukker Int. /Element Six (NL, UK)
'Use of CVD diamond in high-power CO2 lasers and laser diode arrays'
Proceedings of SPIE 'Advanced High-power Lasers', 1-5 November 1999, Osaka, Japan.
Vol. 3889 (2000) ISBN 0277-786X 00

[43-45] Sintec Keramik (D) website (www.sintec-keramik.com)

[43-46] B.H. Foing et al.
'SMART 1 mission, technologies and science: with solar power to the Moon'
Presented at 5th IAA International Conference on Low Cost Planetary Missions, 24th September 2003

[43-47] Ceram Research (UK) website (www.ceram.co.uk)

[43-48] Dr. W. Hanson & Dr. J. Fernie – TWI (UK)
'Ceramic Joining – An overview'
Materials World, Vol. 6 No. 9 pp. 524-36, September 1998

[43-49] Dr. Noel N. Nemeth: NASA- Glenn Research Centre (USA)
NASA-CARES: Private communication (September 2005)

[43-50] N.N. Nemeth et al: NASA- Glenn Research Centre (USA)
'Lifetime Reliability Prediction of Ceramic Structures under Transient Thermomechanical Loads'
NASA TP-2005-212505 (September 2005)

[43-51] N.N. Nemeth et al: NASA- Glenn Research Centre (USA)
'Fabrication and Probabilitic Fracture Strength Prediction of High-aspect-ratio Single Crystal Silicon Carbide Microspecimens with Stress Concentration'
Pre-publication issue: Journal of Thin Solid Films (2005-2006)

[43-52] Galileo Avionica (I)
'Trade-off of Mirror Materials (Cesic®, SiC 54) Maunfacturing of Test Mirrors and Characterisation at Cryogenic Temperatures'
Executive Summary (June 2004)
ESTEC Contract No. 16355/02/NL/VD

[43-53] FutureCarbon GmbH (D)
Presentation (September 2004)

[43-54] Neue Materialien Wurzburg GmbH (D)
Presentation (September 2004)

[43-55] T. Mahrholz et al: DLR (D)
'Fibre reinforced Nanocomposites for Spacecraft Structures'
Proceedings of European Conference on Spacecraft structures, Materials and Mechanical Testing ESA-ESTEC Noordwijk (NL) 10-12 May, 2005.
ESA-SP-581 (August 2005)

[43-56] Cabot Corporation (USA)
PDF file: 'Fundamentals of Carbon Black'

[43-57] M. Krödel: ECM (Munich, Germany)
'Cesic® - Engineering material for optics and structures'
Proceedings of European Conference on Spacecraft structures, Materials and Mechanical Testing ESA-ESTEC Noordwijk (NL) 10-12 May, 2005.
ESA-SP-581 (August 2005)

[43-58] Galileo Avionica
'Trade-off Mirror Materials (Cesic and SiC-54) Manufacturing of test mirrors and characterisation at cryogenic temperatures'
Executive Summary (June 2004)
ESA-ESTEC Contract No. 16355/02/NL/VD

[43-59] A. Novi et al.
'Lightweight SiC foamed mirrors for space applications'
Optomechanical Design and Engineering 2001
(Ed. A. E. Hatheway), Proceedings of SPIE Vol. 4444, 2001

[43-60] ERG Materials and Aerospace Corporation (USA)
'Duocel Silicium Carbide Foam Data sheets'

[43-61] A. Obst: ESA-ESTEC (NL)
'Design of sandwich structures for space applications'
Chapter 18 "Theory and applications of sandwich structures"
Ed. R.A. Shenoi et al., University of Southampton, UK, 2005

[43-62] Bettini Spa (I): Information and data on SiC54S does not appear on the website and is by request only.

[43-63] Dr Luca Mazzucchi: Bettini Spa (I)
'SiC 54S Fabrication'
Private communication (February 2006)

[43-64] Hans Bach (Editor)
'Low Thermal Expansion Glass Ceramics',
Springer-Verlag, 1995

[43-65] W.A Plummer & H.E Hagy
'Precision Thermal Expansion measurements on Low Expansion Optical Materials'
Applied Optics, Vol. 7, No. 5, p. 825-831, 1968

[43-66] R. Mueller et al
'Ultra precision Dilatometer System for Thermal Expansion measurements on Low Expansion Glasses'
Proceedings of 12th Thermal Expansion Symposium, Pittsburgh/PA.
(Editors: P.S. Gaal & D.E. Apostolescu) 1997

[43-67] Ina Mitra et al
'Optimized Glass-ceramic Substrate Materials for EUVL Applications'
Proceedings of SPIE 5374, Emerging Lithographic Technologies VIII (2004)

[43-68] P. Hartmann & H.F. Morian
'100 Years of Mirror Blanks' Proceedings of 2nd Workshop on Extremely Large Telescopes, Proc. Bäckaskog workshop on ELT

[43-69] R. Jedamzik et al
'Homogeneity of the coefficient of linear thermal expansion of ZERODUR®'
Proceedings SPIE Vol. 5868 Optical Materials and Structures Technologies II (2005) p. 241-251

[43-70] O. Lindig & W Pannhorst
'Thermal expansion and length stability of ZERODUR® in dependence on temperature and time'
Applied Optics, Vol. 24, No. 20, p. 2330-3334, 1985

[43-71] F. Bayer-Helms et al
'Längenstabilität bei Raumtemperatur von Proben der Glaskeramik ZERODUR®'
Metrologia 21, p. 49-57, 1985

[43-72] R. Haug et al
'Length variation in ZERODUR® M in the temperature range from –60°C to +100°C'
Applied Optics, Vol. 28, No. 19, 1989

[43-73] R. B. Roberts et al
'Thermal properties of ZERODUR® at low temperatures' Cryogenics 22, p. 566, November 1982

[43-74] J.H. Burge et al
'Thermal expansion of Borosilicate glass, ZERODUR®, ZERODUR® M, and unceramized ZERODUR® at low temperatures'
Applied Optics, Vol. 38, p.7161, 1999

[43-75] J. W. Baer & W. P. Lotz
'Figure testing of 300 mm ZERODUR® mirrors at cryogenic temperatures'
Proceedings of SPIE Cryogenic Optical Systems and Instruments IX, Vol. 4822, 2002

[43-76] Schott AG: ZERODUR® glass ceramic
Catalogue 10162 e 09043.0

[43-77] K.Schilling SCHOTT Internal report 303/91 Mainz 1991

[43-78] H.Richter & G.Kleer: Fraunhofer Institut für Werkstoffmechanik, Freiburg (D)
Report V24/83 (1983)

[43-79] M.J.Viens
NASA Technical memorandum 4185 (1990)

[43-80] F. Kerkhof et al:
Glastech. Berichte 54 (1981) No. 8 p. 265 to 277

[43-81] G. Exner & O.Lindig
Glastech Berichte 55 (1982) No. 5 p. 107 to 117

[43-82] G.Exner, Schott Glaswerke, Mainz (D)
Glastech Berichte 56 (1983) Nr. 11, p.299-312

[43-83] H. Bach & N. Neuroth (Editors)
'The properties of optical glass'
Springer Verlag 1998

[43-84] S.M. Wiederhorn, L.H. Bolz
'Stress corrosion and static fatigue of glass'
J. Am. Ceram. Soc. 53, 543-548 (1970)

[43-85] M.J. Viens
'Fracture toughness and crack growth parameters of ZERODUR®'
NASA Technical Memorandum A969903 (1990)

[43-86] INSACO Inc. (USA) http://www.insaco.com/MatPages/zerodur.asp

[43-87] Praezisions Glas & Optik GmbH, Iserlohn (D)
http://www.pgo-online.com/intl/katalog/zerodur.html

[43-88] SCHOTT AG, Mainz (D): Data sheet from website
http://www.schott.com/optics_devices/english/
2TIE25 - Striae in optical glass

[43-89] SCHOTT AG, Mainz (D): Data sheet
2TIE26 - Homogeneity of optical glass

[43-90] SCHOTT AG, Mainz (D): Data sheet
2TIE27 - Stress in optical glass

[43-91] SCHOTT AG, Mainz (D): Data sheet
2TIE28 - Bubbles and inclusions in optical glass

[43-92] SCHOTT AG, Mainz (D): Data sheet
2TIE29 - Refractive index and dispersion

[43-93] SCHOTT AG, Mainz (D): Data sheet
2TIE30 - Chemical properties of optical glass

[43-94] SCHOTT AG, Mainz (D): Data sheet
2TIE31 - Mechanical and thermal properties of optical glass

[43-95] SCHOTT AG, Mainz (D): Data sheet
2TIE32 - Thermal loads on optical glass

[43-96] SCHOTT AG, Mainz (D): Data sheet
2TIE33 - Design strength of optical glass and Zerodur®

[43-97] SCHOTT AG, Mainz (D): Data sheet
2TIE34 - RoHS - frequently asked questions July 2005

[43-98] SCHOTT AG, Mainz (D): Data sheet
2TIE34 - RoHS Annex - present status

[43-99] SCHOTT AG, Mainz (D): Data sheet
2TIE34 - RoHS Hazardous substances in optical glass

[43-100] SCHOTT AG, Mainz (D): Data
2TIE35 - Transmittance of optical glass

[43-101] SCHOTT AG, Mainz (D): Data sheet
2TIE36 - Fluorescence of optical glass

[43-102] SCHOTT AG, Mainz (D): Data sheet
2TIE37 - Thermal expansion of ZERODUR

[43-103] SCHOTT AG, Mainz (D): Data sheet
2TIE38 - Lightweighting of ZERODUR

[43-104] SCHOTT AG, Mainz (D): Data sheet
2TNE03 - Material safety data sheet abbreviation explanation

[43-105] SCHOTT AG, Mainz (D): Data sheet
2TNE04 - Test report for delivery lots

[43-106] INSACO Inc. (USA)
http://www.insaco.com/MatPages/corning_ule_glass.asp

[43-107] Praezisions Glas & Optik GmbH, Iserlohn (D)
http://www.pgo-online.com/intl/katalog/ule.html

[43-108] Corning Corp. (USA): Semiconductor Optics
http://www.corning.com/semiconductoroptics/technical_information/
<SEARCH for ULE>
Corning International – Europe, Wiesbaden (D); email -
cieurope@corning.com

[43-109] A.A. van Veggel: Eindhoven Technical University/ TNO (NL)
'Experimental Evaluation of the Stability and Mechanical Behaviour of Contacts in Silicon Carbide for the Design of the Basic Angle Monitoring System of GAIA'
Proceedings of European Conference on Spacecraft structures, Materials and Mechanical Testing ESA-ESTEC Noordwijk (NL) 10-12 May, 2005.
ESA-SP-581 (August 2005)

[43-110] Boostec S.A. website: http://www.boostec.com/index.htm
Material characteristics:
http://www.boostec.com/SiCTypicalCharacteristics.pdf
Other high-precision components: http://www.boostec.com/real.htm

[43-111] S. Williams & P. Deny: CoorsTek Inc (USA)/ Boostec Industries (F),
'Overview of the production of sintered SiC optics and optical sub-assemblies',
Optical Materials And Structures Technologies II (OEI402),
SPIE Optics and Photonics 2005, San Diego, CA

[43-112] G. van Papendrecht
'Outgassing test on Silicon carbide samples'
Materials Report Number: 3959, QMC report number: QMC 2004/099, 21 June 2004, Materials Physics and Chemistry Section, Materials and Processes Division, ESTEC

[43-113] Castel D. (Astrium), Denaux D. (Astrium), Bourgoin M. (Boostec), Mercier K. (CNES Toulouse)
'New Technology for Manufacturing Very Large SiC Mirror'

European Conference on Spacecraft Structures, Materials and Mechanical Testing, Toulouse, (France), 11-13 December 2002

[43-114] Optical Bench Breadboard,
Near Infrared Spectrograph for JWST,
Final Report, Doc. No.: NGST-ASD-TR-007, dated 18.3.2004

[43-115] SI-01 NIRSPEC Design Report to ESTEC,
Doc. No. NIRS-ASD-RP-0001, dated 01.10.2004

43.20.2 Sources

Data are for illustrative purposes only and cannot be used for design.

All data presented and cited within the text are summarised from information provided directly by material suppliers or obtained from their websites (to end-2005).

43.20.3 ECSS documents

[See: ECSS website]

ECSS-Q-70-71	Data for the selection of space materials and processes, previously ESA PSS-01-701.
ECSS-Q-ST-70-01	Cleanliness and contamination control
ECSS-Q-ST-70-02	Thermal vacuum outgassing test for the screening of space materials; previously ESA PSS-01-702

43.20.4 Other standards

ISO standards	Fine ceramics (advanced ceramics, advanced technical ceramics), [See: 43.3]
EN60672-1	Ceramic and glass insulating materials - Part 1: Definitions and classification
EN60672-2	Ceramic and glass insulating materials - Part 2: Methods of test
EN60672-2	Ceramic and glass insulating materials - Part 3: Specification for individual materials

[See also: 81.9 for CEN ceramic-related standards]

44 Magnesium alloys and their composites

44.1 Introduction

44.1.1 General

The main magnesium alloys are listed, together with their chemical compositions and typical mechanical properties.

Both discontinuously reinforced and continuous fibre reinforced composites are described, along with potential applications for both.

44.1.2 Features

The basic features of magnesium alloys include:

- Low density (1740 kg/m^3), which makes Mg attractive.
- Low thermal stability, giving limitations in terms of thermal performance (usually to temperatures below 100°C, but new alloys extend range towards 200°C).
- High damping capacity, good resistance to vibration fatigue.
- High CTE, e.g. 27 to 28 × 10^{-6} /K between 20°C and 300°C.
- Processing, where the c.p.h. crystal structure of most magnesium alloys means that extruded and sheet products are more difficult to manufacture than aluminium alloys, hence more costly. Good fluidity of magnesium casting alloys means that a wide range of near-net shape components are available.

[See also: ECSS-Q-70-71]

44.1.3 Alloy classification

Table 44.1.1 provides the ASTM system of alloy and temper designations for magnesium alloys. Conventional alloy specifications, plus supplier product codes, are provided in Ref. [44-6], [44-20].

Table 44.1-1 - Magnesium alloys: ASTM designation system - alloys and tempers

First part	Second part	Third part	Fourth part
Indicates the two principal alloying elements	Indicates the amounts of the two principal alloying element	Indicates minor variations to the basic designation	Indicates condition (temper)
Consists of two code letters representing the two main alloying elements arranged in order of decreasing percentage (or alphabetically if percentages equal)	Consists of two numbers corresponding to rounded percentages of the two main alloying elements, arranged in same order as alloy codes in first part	Consists of a letter of the alphabet assigned in order as compositions become standard	Consists of a letter followed by a number (separated from the third part of the designation by a hyphen)
A - aluminium B - bismuth C - copper D - cadmium E - rare earth F - iron G - magnesium H - thorium J - strontium K - zirconium L - lithium M - manganese N - nickel P - lead Q - silver R - chromium S - silicon T - tin V - gadolinium W - yttrium X - calcium Y - antimony Z - zinc	Whole numbers	Letters of alphabet except I and O	F-as fabricated O-annealed, recrystallised (wrought products only) H-strain hardened H11 (slight) to H18 (fully) - strain hardened H21 to H28 - strain hardened and partially annealed (H21 softest) H31 to H38 - strain hardened and stabilised to prevent age softening T1- cooled and naturally aged T3- solution heat treated and cold worked T4- solution heat- treated T5- cooled and artificially aged T6-solution heat-treated and artificially aged T7- solution heat treated and stabilised T8- solution h.t. cold worked and artificially aged T9- solution h.t. artificially aged and cold worked T10- cooled artificially aged and cold worked

44.1.4 Environmental aspects

44.1.4.1 General

Magnesium is resistant to attack by alkalis but is attacked by most acids. Similarly, magnesium is attacked by common acidic salts such as chloride solutions found in the form of seawater. Therefore, unless a magnesium component is to function in an innocuous environment, it should not be used in service without some form of protective treatment.

Magnesium alloys are not prone to intergranular attack since the grain boundaries are cathodic compared with the actual grains and would therefore be cathodically protected if exposed to a corrosive environment.

In addition, most magnesium alloys, with the exception of the magnesium-aluminium alloys, are not susceptible to stress corrosion cracking under normal conditions and at stresses up to 0.2% Proof Stress values, Ref. [44-9].

[See also: ECSS-Q-ST-70-36]

Cathodic impurities, e.g. iron, nickel, copper, can have a significant effect on the corrosion behaviour of unprotected magnesium alloys and are therefore controlled to low levels in alloys specified for aerospace and defence applications.

Conversely, surface contamination, for example by minute cathodic particles resulting from abrasive blasting with cast iron or steel shot, may result in high corrosion rates.

Galvanic corrosion can also arise at dissimilar metal attachments, such as nuts, bolts and rivets, unless the necessary precautions are taken.

44.1.4.2 Protection systems

A number of well proven protective coating systems and techniques are available and these are generally based on anodising or a chemical conversion treatment (usually chromating) followed by painting, Ref. [44-9]. However, chromating is being phased out due to health and safety legislation concerning chromate baths and a number of new protective systems have been developed, such as Tagnite®, Keronite® and Oxsilan®. The designer therefore needs to consult the material, component supplier or specialist treatment companies at an early stage.

44.1.5 Machining

The flammability of finely divided magnesium in the form of machining swarf or grinding dust means that due care must be exercised when machining magnesium alloys.

Likewise, there is potential for hydrogen generation should water based cutting fluids be used. However, magnesium alloys show very good free cutting and may be machined faster, without cutting fluids and at lower power than aluminium alloys, allowing complex shapes with close tolerances to be easily manufactured.

Guidance on machining parameters and the relevant safety procedures are well documented, Ref. [44-10], although this will normally be the responsibility of the component supplier.

44.1.6 MMC - metal matrix composites

Considerable activity on discontinuously reinforced (particulate) magnesium-based MMCs occurred during the 1980's and 1990's and some materials were commercialised, [See also: 44.3]. However, the cost of production of these materials has resulted in their withdrawal, although production could be re-instated if the demand justifies the high set-up costs.

Work continues on continuous fibre-reinforced MMCs, mainly in the USA where much of the activity is restricted, with the primary interest being on low CTE materials for electronics packaging or substrates for space mirrors.

An indication of the technical development and commercial availability of MMCs is provided on the website of the MMC-Assess project.

44.1.7 Material availability

44.1.7.1 General

Many of the established magnesium alloys contained Thorium as a major alloying element. Owing to concerns over residual radioactivity, these alloys have been withdrawn from regular commercial supply and are therefore not included in this handbook.

Also, whilst 'super light' magnesium lithium alloys were developed in the 1960's (and used in the US Saturn programme) and the higher lithium alloys have a b.c.c. structure which is more amenable to extrusion or rolling, difficulties in the foundry procedures and concerns over corrosion resistance in aqueous environments have limited the use of these alloys. Further work is continuing in Germany, Ref. [44-13], and Japan but no new alloys have as yet been registered commercially.

44.1.7.2 Sources

Table 44.1.2 lists the major commercial sources of magnesium alloys. Alloys and product forms vary between suppliers, Ref. [44-6].

NOTE Further information on magnesium suppliers is available from: International Magnesium Association (www.intlmag.org) and DMTC - Israeli Consortium for the Development of Magnesium (www.dmtc.org.il).

Table 44.1-2 - Magnesium alloys: Sources - examples

Supplier	Website
Alubin (Israel)	www.alubin.com
AMTS, Wrought Product Division (Israel) (1)	www.magnesium-technologies.com
Dead Sea Magnesium (Israel)	www.dsmag.co.il
Hydro Magnesium (Norway)	www.hydromagnesium.com
Magnesium Elektron (UK)	www.magnesium-elektron.com
Otto Fuchs (D)	www.otto-fuchs.com
Timminco (Canada)	www.timminco.com
Key: (1) Links with Chemetall GmbH (D) for surface treatment.	

44.1.8 Future developments

Much of the development activity on magnesium alloys in recent years has centred on applications in the automotive industry, where weight reduction has been seen as a means to improving fuel efficiency. Because of the good castability and economics of high volume processes, the thrust of this has been on alloys for pressure diecasting. The drawbacks with existing casting alloys have been twofold: poor high temperature properties and low room temperature ductility.

Alloys have now been introduced with improved properties both at room temperature and in elevated temperature creep resistance.

Whilst diecastings may not have been normally considered for aerospace applications, the improvements in process technology have resulted in the availability of high-quality components with close tolerance near net shape. Also, whilst high initial tooling costs are involved, the overall cost of the components can still be competitive with alternative manufacturing routes even at low volumes, Ref. [44-15]. Some of the new diecasting alloys are therefore given, [See: 44.2].

There has also been much activity in attempts to improve the economics of production of wrought magnesium products which have traditionally been more costly than aluminium products due to the lower processing speeds of magnesium alloys.

Magnesium sheet products produced by twin-roll casting are becoming available and work is also progressing on improved extrusion processes.

A new high strength extruded alloy, Elektron 675, has recently been announced by Magnesium Elektron and, whilst this is still undergoing final development, its tensile properties at 200°C are better than 2024 or 7075 aluminium alloys which makes it of potential interest for aerospace applications.

Further work continues at various Universities and research centres on alloys for higher temperature applications, super-light Mg-Li alloys and on Mg-based MMCs. However, the economics of production mean that few of these are likely to reach commercial availability.

44.2 Magnesium alloys

44.2.1 Chemical composition

Table 44.2.1 indicates the nominal chemical compositions of many magnesium alloys. The compositions are classed by the method used for processing and the alloys are listed using their ASTM designations and UNS numbers where registered, Ref. [44-20], [44-21], covering:

- Sand and permanent mould castings
- Die castings
- Wrought products

[See also: Table 44.1.1 for ASTM designation system]

Some proprietary alloys are also included together with some recent alloys, Ref. [44-1], [44-7], [44-8].

Variations in alloy chemical composition can exist in different specification systems for ‘equivalent’ alloys. Care should be exercised when comparing alloys across different standards systems, Ref. [44-6].

Table 44.2-1 - Magnesium alloys: Nominal chemical compositions

Alloy		Composition, wt%						Notes
ASTM No.	UNS No.	Al	Mn	Zn	Zr	Rare earths	Other	(a)
Sand and permanent mould castings								
AZ63A	M11630	6	0.15	3	-	-	-	
AZ81A	M11810	7.6	0.13	0.7	-	-	-	
AZ91E	M11918	8.7	0.13	0.7	-	-	-	(b)
AZ92A	M11920	9	0.1	2	-	-	-	
EQ21A	M12210	-	-	-	0.7	2.25(c)	1.5 Ag	
EZ33A	M12330	-	-	2.55	0.7	3.25	. . .	
K1A	M18010	-	-	-	0.7	. . .	. . .	
QE22A	M18220	-	-	-	0.7	2.15(c)	2.5 Ag	
WE43B	M18432	-	-	-	0.7	3.0(d)	4.0 Y	
WE54A	M18410	-	-	-	0.7	3.5(d)	5.2 Y	
ZC63A	M16331	-	0.25	6	-	-	2.7 Cu	
ZE41A	M16410	-	-	4.25	0.7	1.25	-	(e)
ZK51A	M16510	-	-	4.55	0.7	-	-	
Die castings								
AM20	-	2.0	0.40	0.20 max	-	-	-	
AM50A	M10500	4.9	0.26	0.20 max	-	-	-	
AM60B	M10602	6	0.24	0.20 max	-	-	-	
AE42	-	4	0.50 max	0.20 max	-	2.5	-	
AS21	-	2	0.4	-	-	-	1.0 Si	
AS41B	M10412	4.25	0.35	0.12	-	-	1.0 Si	
AZ91D	M11916	9	0.15	0.7	-	-	-	(b)
Wrought products								(f)
AZ10A	M11100	1.2	0.2	0.4	-	-	-	E
AZ31B	M11310	3	0.2	1	-	-	-	F, S, E
AZ61A	M11610	6.5	0.15	0.95	-	-	-	F, E
AZ80A	M11800	8.5	0.12	0.5	-	-	-	F, E
LA141A	M14141	1.25	0.15	-	-	-	14 Li	S, E
M1A	M15100	-	1.6	-	-	-	0.3 Ca	E
WE43B	M18432	-	-	-	0.7	3.0(d)	4.0 Y	F, E
WE54A	M18410	-	-	-	0.7	3.5(d)	5.2 Y	F, E
ZE10A	M16100	-	-	1.25	-	0.17	-	S
ZK31	-	-	-	3	0.7	-	-	F, E (g)
ZK60A	M16600	-	-	5.5	0.7	-	-	F, E
ZM21	-	-	1.0	2.0	-	-	-	S, E

Recent alloys								Notes
Sand and permanent mould castings								
Elektron 21	M12310	-	-	0.3	0.7	4.5(h)	-	
MRI-201S	-	-	-	-	-	-	-	(j)
MRI-202S	-	-	-	-	-	-	-	(j)
Die castings								
AE44	-	4	0.50 max	0.20 max	-	-	-	(k)
AJ52X	-	4.9	0.3	-	-	-	1.3 Sr	(l)
AJ62	-	6.3	0.4	-	-	-	2.5 Sr	(m)
AS31	-	3.5	0.50 max	0.20 max	-	-	1.0 Si	(k)
MRI-153M	-	-	-	-	-	-	-	(j)
MRI-230D	-	-	-	-	-	-	-	(j)
Wrought products								
Elektron 675	-	-	-	-	-	-	-	(n)

Key:

(a) Magnesium alloys containing thorium (code letter H) additions have been removed from this Handbook, due to concerns over residual radioactivity, but may still be available to special order for replacement purposes.

(b) High purity (low Fe, Ni,Cu) versions for improved corrosion resistance

(c) Rare earth elements are in the form of didymium (a mixture of rare earth elements made chiefly of neodymium and praseodymium).

(d) Rare earths are 2.0 to 2.5% and 1.5 to 2.0% Nd for WE43A and WE54A, respectively, with the remainder being heavy rare earths.

(e) Elektron RZ5

(f) F, forging; S, sheet and plate; E, extruded bar, shape, tube, and wire.

(g) Elektron ZW3

(h) Proprietary alloy, Magnesium Elektron, rare earths are 3.0% Nd, 1.5% Gd

(j) Proprietary alloy, Dead Sea Magnesium, composition not published

(k) Proprietary alloy, Hydro Magnesium

(l) Proprietary alloy, Noranda Magnesium, typical composition

(m) Proprietary alloy, Noranda Magnesium, licensed to Magnesium Elektron

(n) Development alloy, Magnesium Elektron, composition not published

Many magnesium alloys have also been registered with SAE International for an aerospace, AMS, standard. Like many former defence standards these apply to specific temper conditions and forms and are too extensive to be included in this handbook.

European standards and specifications (EN) are being developed and adopted. These are progressively superseding the various National standards in member states, Ref. [44-6].

Aerospace EN specification designations are controlled by AECMA and also apply to a particular alloy, conditions and forms, e.g.**prEN 2731** Magnesium alloy MG-C46001-T6 - Sand casting

44.2.2 Mechanical properties

44.2.2.1 Room temperature properties

Table 44.2.2 gives typical room temperature mechanical properties for a variety of magnesium alloys. Recent alloys are included, Ref. [44-1], [44-7], [44-8].

[See also: Table 44.1.1 for alloy designation system]

Table 44.2-2 - Magnesium alloys: Typical mechanical properties, at room temperature

Alloy	Tensile strength	Yield strength			Elongation in 50mm	Shear strength
		Tensile	Compressive	Bearing		
	MPa	MPa	MPa	MPa	%	MPa
Sand and permanent mould castings						
AZ63A-T6	275	130	130	360	5	145
AZ81A-T4	275	83	83	305	15	125
AZ91E-T6	275	145	145	360	6	145
AZ92A-T6	275	150	150	450	3	150
EQ21A-T6	235	195	195	-	2	152
EZ33A-T5	160	110	110	275	2	145
K1A-F	180	55	-	125	19	55
QE22A-T6	260	195	195	-	3	-
WE43B-T6	250	180	187	-	7	162
WE54A-T6	280	205	172	-	4	150
ZC63A-T6	210	125	-	-	4	-
ZE41A-T5	205	140	140	350	3.5	160
ZK51A-T5	205	165	165	325	3.5	160
Elektron 21-T6	280	170	168	-	5	172
MRI-201S-T6	260	170	190	-	6	-
MRI-202S-T6	250	150	145	-	7	-
Die castings						
AM20	206	94	74	-	16	-
AM50A	220	120	113	-	6 - 10	-
AM60B	220	130	130	-	6 - 8	-
AS21	230	120	106	-	12	-
AS41	240	130	-	-	10	-
AE42	237	134	103	-	8 - 10	-
AZ91D	230	150	165	-	3	140
AE44	245	142	-	-	10	-
AJ52X	232	145	-	-	5	-
AJ62	227	138	-	-	7	-

Alloy	Tensile strength	Yield strength			Elongation in 50mm	Shear strength
		Tensile	Compressive	Bearing		
	MPa	MPa	MPa	MPa	%	MPa
AS31	216	130	-	-	8	-
MRI-153M	250	170	170	-	6	-
MRI-230D	235	180	180	-	5	-
Sheet and plate						
AZ31B-H24	290	220	180	325	15	160
LA141A-T7	145	125	-	-	23	-
ZE10A-H24	234	152	-	-	6	-
ZM21-H14	250	165	-	-	5 - 8	-
Extruded bars and shapes						
AZ10A-F	240	145	69	-	10	-
AZ31B-F	260	200	97	230	15	130
AZ61A-F	310	230	130	285	16	140
AZ80A-T5	380	275	240	-	7	165
LA141A-T7	139	108	-	-	23	. . .
M1A-F	255	180	83	195	12	125
WE43B-T6	270	190	-	-	10	-
WE54A-T6	275	190	-	-	10	-
ZK31-F	305	225	200 - 250	-	8	145
ZK60A-T5	365	305	250	405	11	180
ZM21-F	230	150	-	-	8	-
Elektron 675 (a)	400	300	-	-	9	-
Forgings						
AZ61A-F	275	160	130 - 165	-	7	-
AZ80A-T5	290	200	-	-	6	-
WE43B-T6	280	180	-	-	7	-
WE54A-T6	295	195	-	-	6	-
ZK31-F	290	205	165 - 215	-	7	-
ZK60A-T5	365	305	250	405	11	180
Key: (a) - Development alloy - provisional data						

44.2.2.2 Low temperature properties

Magnesium alloys do not undergo ductile-brittle transition and low temperature ductility show only small falls compared with room temperature values. Some examples of tensile properties at –196°C are given in Table 44.2.3.

Table 44.2-3 - Magnesium alloys: Typical low temperature tensile properties at -196°C

Alloy	Yield strength	Tensile strength	Elongation
	MPa	MPa	%
Cast alloys			
AZ91C-T6	180	310	1.7
AZ92A-T6	195	320	0.8
EZ33A-T5	-	154	5
QE22A-T6	233	359	2.4
ZE41A-T5	-	245	5
Elektron 21-T6	-	270	7
Wrought alloys			
ZK31-F	-	460	6

44.2.2.3 Elevated temperature properties

Typical tensile properties of a number of magnesium alloys at temperatures up to 300°C are given in Table 44.2.4.

Table 44.2-4 - Magnesium alloys: Typical tensile properties at elevated temperatures

Alloy	Test temperature							
	150 °C		200 °C		250 °C		300 °C	
	YS (MPa)	UTS (MPa)	YS (MPa)	UTS (MPa)	YS (MPa)	UTS (MPa)	YS (MPa)	UTS (MPa)
Sand and Permanent Mould Castings								
AZ63A-T6	-	165	-	110 (a)	-	-	-	55 (b)
AZ92A-T6	-	195	-	115 (a)	-	-	-	55 (b)
EQ21A-T6	180	211	170	191	152	169		132 (b)
EZ33A-T5	85	140	75	130	70	110	55	85
QE22A-T6	-	208	-	-	-	-	-	80 (b)
WE43B-T6	185	260	170	245	145	215	115	155
WE54A-T6	195	255	183	241	175	230	140	180
ZE41A-T5	120	170	100	135	80	110	55	85
MRI-201S	170	245	-	-	-	-	-	-
MRI-202S	145	220	-	-	-	-	-	-
Elektron 21-T6	165	255	160	240	145	185	-	-
Diecastings								
AE42	95	155	-	-	-	-	-	-
AS21	75	130	-	-	-	-	-	-
AS41	85	152	-	-	-	-	-	-
AZ91D	110	159	-	-	-	-	-	-
AJ62	-	-	-	-	-	-	-	-
MRI-153M	135	190	-	-	-	-	-	-
MRI-230D	150	205	-	-	-	-	-	-
Extrusions								
AZ80A-T5	-	235	-	-	-	-	-	69 (b)
WE43B-T6	185	245	170	235	130	210	-	-
WE54A-T6	185	250	175	240	165	230	-	-
ZK31-F	100	150	50	125	15	100	-	-
ZK60A-T5	-	180	-	-	-	-	-	41 (b)
Elektron 675	-	-	290	380	-	-	-	-
Sheet								
AZ31B-H24	95	150	60	105	40	65	-	48 (b)

Key: (a) Tested at 205°C; (b) Tested at 315°C

Magnesium alloys generally show a rapid fall off in strength above 150°C and much of the development work has centred on improving this situation. The sand casting alloys based on rare earth additions, e.g. WE43 and WE54, have largely overcome the problems with earlier alloys and the newer alloy, Elektron 21, shows good promise as a lower cost alloy with good corrosion resistance.

WE54 shows a loss of ductility after >5000 hours at elevated temperatures and where this might be a problem alloy WE43 is preferred.

For extruded products the WE alloys also show the best performance amongst the established alloys but recent work by Magnesium Elektron has resulted in a new alloy, Elektron 675, with high-temperature strength exceeding that of many aluminium alloys. Preliminary data for this alloy is provided, [See: Table 44.2.4].

The WE alloys and Elektron 21 also show the best longer term creep properties, as shown in Table 44.2.5

The newer diecasting alloys have been developed for improved elevated temperature properties and although the short term tensile properties are not much different from AZ91 they show improved longer term creep resistance.

Table 44.2-5 - Magnesium alloys: Typical elevated temperature creep properties

Alloy	**Test Temperature**		
	150°C	**200°C**	**250°C**
	Stress (MPa) to produce 0.2% strain in 1000 hours		
Sand and Permanent Mould Castings			
EQ21A-T6	134	52	19
EZ33A-T5	-	57	20
WE43B-T6	-	110	37
WE54A-T6	-	170	35
ZE41A-T5	80	-	-
Extrusions			
ZK31-F	17	15.5	-
Sheet			
AZ31B-H24	17	-	-
Stress (MPa) to produce 0.1% strain in 100 hours			
Sand and Permanent Mould Castings			
AZ92A-T6	-	12	-
WE43B-T6	-	150	-
ZE41A-T5	-	40	-
Elektron 21-T6	-	100	-
Diecastings			
AE42	65	40	20
AS21	38	30	-
AS41	15	15	-
AZ91D	20	5	-

44.2.2.4 Fatigue and fracture toughness properties

Fatigue data for magnesium alloys has, for many years, been limited, Ref. [44-7]. This has become a requirement for their application in automotive and aerospace sectors. However, magnesium alloys are similar to other alloys in that fatigue strength depends on tensile strength, Ref. [44-11]. The ratio is not so well defined as for some other metals since whilst solid-solution strengthening increases the fatigue strength of magnesium alloys, cold working and precipitation strengthening produce little improvement in fatigue strength at longer lives.

When compared with data for A357 aluminium alloy, *S-N* curves for AZ91E and WE43 show that although A357 performs well at low cycles the situation changes so that WE43 has the better properties at high cycles. The lower strength alloy, AZ91E, has lower properties at low-cycles but at high cycles the difference compared with WE43 is less marked, Ref. [44-11].

Room temperature fatigue strengths of some magnesium alloys are given in Table 44.2.6 together with fracture toughness K_{Ic} values.

Table 44.2-6 - Magnesium alloys: Typical fatigue and fracture toughness properties at room temperature

Alloy	Fatigue strength (a)	Fracture toughness, K_{1C}
	MPa	MPa√m
Sand and Permanent Mould Castings		
AZ91E-T6	70	13.2
AZ81A-T4	70	-
EQ21A-T6	100 - 110	16.4
EZ33A-T5	60	-
WE43B-T6	80	15.9
WE54A-T6	97	14.3
ZE41A-T5	100	15.1 - 16.3
Elektron 21-T6	115 - 120 (b)	15
Diecastings		
AZ91D	70	-
AM50A	70	-
AM60B	70	-
Extrusions		
ZK31A-F	122	45.6
WE54A-T5	145	15 - 17
Elektron 675 (d)	200 (b)	-
Sheet		
AZ31B-H24	100 (c)	28

Key:

(a) 5×10^7 cycles, rotating bend test

(b) 5×10^7 cycles, pull-pull test, $R = 0.1$ (ASTM E466)

(c) 5×10^7 cycles, cantilever bend test

(d) Development alloy, provisional data

In addition to alloy manufacturers fatigue data, a study considered the fatigue performance of several recent alloys (AM50, AM60, AE42, WE43 and RZ5), Ref. [44-7]:

- *S-N* curves for round bar and flat specimens.
- IST - incremental step test and hysteresis effects.
- Stress-life functions (stress amplitude versus fatigue life) for round and flat specimens of WE43 and RZ5 alloys.

44.2.2.5 Damping capacity

Magnesium alloys in general show higher damping capacity than most common structural metals.

In particular magnesium alloy K1A shows a specific damping capacity around twice that of the Cu-Mn damping alloys and six times better than grey cast iron, shown in Table 44.2.7.

Magnesium K1A therefore finds aerospace applications where vibration damping is important.

Table 44.2-7 - Magnesium alloys: Damping capacity of selected alloys and other metals

Alloy	Specific damping capacity at 10% of yield stress
Cast alloys	
K1A	60
AZ91E	0.2
AZ80A-T4	0.02
EQ21A-T6	0.22
EZ33A-T5	4.5
WE43B-T6	0.09
WE54A-T6	0.17
ZE41A-T5	1
Other metals	
1100 aluminium	0.3
2011 aluminium alloy	0.25
Other Al alloys	<0.2
Grey cast iron	10
Cu-Mn damping alloys	20 - 40

44.3 Discontinuously reinforced magnesium composites

44.3.1 Types of discontinuous reinforcement

Discontinuous reinforcements for magnesium alloys are grouped as:

- Particulates, using additions silicon carbide or boron carbide particles
- Short ceramic fibres, using alumina or carbon.
- Whiskers, using silicon carbide.

44.3.2 Effect of particulate additions

The addition of particulates to a base magnesium alloy:

- Increases the initial tensile modulus,
- Increases yield and tensile strengths, provided modest additions of particles are made.
- Reduces elongation, for the optimised composites with 12 to 15 volume percentage particles.
- Reduction of CTE, from 26 × 10-6 /°C to 20 × 10-6 /°C, for a 20% addition of silicon carbide particles.

44.3.3 Processing

44.3.3.1 General

In the USA, atomised ZK60A alloy in powder form has been consolidated with both SiC and boron carbide particles.

Magnesium Elektron (UK), prefer the cheaper melt stirring route to create the composite using ZC63 and AZ91 casting alloys or variants on ZC71 wrought alloy, Ref. [44-17].

Short fibre and whisker reinforced composites are usually prepared by melt infiltration of preforms by squeeze casting or pressure diecasting techniques, Ref. [44-14].

44.3.3.2 Occupational health and safety

Many types of ceramic whiskers and polycrystalline fibres have been evaluated by the World Health Organisation's International Agency for Research on Cancer (Monograph 43, 1988) as possibly carcinogenic to humans, and are subject to occupational health and safety risk control measures in some jurisdictions.

Exposure to respirable dusts containing these materials should be avoided. Such dusts are not likely to be released from MMCs in their normal handling and use, but control measures need to be taken if the materials are machined, cut or subject to abrasion.

Refer to manufacturers' and suppliers' Safety Data Sheets for further health and safety information.

44.3.4 Properties

Representative properties of the SiC-reinforced AZ61 alloy composite are given in Table 44.3.1.

Table 44.3-1 - SiC particulate reinforced AZ61 magnesium alloy: Typical mechanical properties

Material	Modulus (GPa)	0.2% PS (MPa)	UTS (MPa)	Elongation (%)
AZ61	44	190	290	18
AZ61 + 2%SiC	60	242	310	2.0
AZ61 + 5%SiC	66	261	321	1.2

For high-strength, high-rigidity applications, Magnesium Elektron MELRAM 072 contains 12%SiC (10µm mean particle size) using a matrix based on the ZC71 alloy.

To improve the extrudability and room tempertature ductility, the matrix composition was modified to Mg-4.5%Zn-0.5%Mn, and the extruded product given a cold drawing operation, which also improved dimensional accuracy. This material was renamed MELRAM 072TS.

Typical mechanical properties for both variants are shown in Table 44.3.2, Ref. [44-6].

Table 44.3-2 - SiC particulate reinforced magnesium alloy: Typical mechanical properties

Material (1)	Modulus (GPa)	YS (MPa)	UTS (MPa)	Elongation (%)
MELRAM 072 ZC71 + 12%SiC	63	370	398	1.5
MELRAM 072TS Mg-4.5%Zn-0.5%Mn + 12%SiC	63	311	344	4
Key (1): MELRAM – Magnesium Elektron, development MMC (1998).				

44.4 Continuously reinforced magnesium composites

44.4.1 Types of composite

The types of continuous reinforcement considered for magnesium alloys are:

- carbon fibre,
- silicon carbide fibre.

Work has centred on continuous carbon fibre reinforced magnesium alloys.

Table 44.4.1 shows examples of MMCs, Ref. [44-2], [44-3], [44-4], [44-5], [44-16], [44-18].

Table 44.4-1 - Continuous fibre reinforced magnesium MMC materials: Examples

Fibre	Matrix	Comments
Carbon fibre		
T300	Mg-1Al	Squeeze cast. Susceptible to galvanic corrosion.
M40	Mg 99.85	Gas pressure infiltration
K63B12	AZ31	Gas pressure infiltration
P55	AZ91C	Design for near zero CTE. Laser absorptivity tests.
P100	AZ31B	-
	AZ61A	-
Silicon carbide		
SCS-2	ZE41	Cast rod.

44.4.2 Features

Carbon fibre-reinforced magnesium MMCs offer:

- high specific properties, and
- possibility of near-zero thermal expansion characteristics.

Consequently they have been considered as having potential uses in aerospace applications, Ref. [44-2], [44-16], [44-18].

44.4.3 Processing

The starting point for all continuous fibre-reinforced magnesium composites is a fibre preform. The most popular production method is filament winding although weaving, knitting and variants thereof are also employed. To some extent the chosen method depends on the type of carbon fibre used.

The magnesium matrix is incorporated by molten metal techniques which depend upon the chosen type of preform. Thus, to prevent damage to filament wound preforms these are usually gas pressure infiltrated whilst woven or knitted preforms are usually more robust and squeeze casting or pressure diecasting techniques may also be employed.

In carbon-Mg MMC's the properties are dictated primarily by the reinforcement, the matrix serving mainly to hold the fibres together. The important factors are therefore the nature of the bonding between fibre and matrix and the avoidance of any degradation of fibre properties to interaction with the matrix. These are therefore prime factors in the selection of the matrix alloy although prior treatments may also be applied to the fibre surfaces.

44.4.4 Properties

Table 44.4.2 gives general mechanical property data on magnesium MMC materials, Ref. [44-1], [44-3], [44-4], [44-18].

The main interest in carbon fibre-reinforced magnesium composites for space applications appears to be in materials with low CTE coefficient of thermal expansion, together with high thermal conductivity.

Published information is sparse, but data from US-based work indicates CTE of 1.67ppm/K with conductivity of 180W/mK compared with 0.5ppm/K and 1.5W/mk for fused silica and 26ppm/K and 122W/mK for pure magnesium, Ref. [44-16].

Table 44.4-2 - Magnesium continuous reinforced MMCs: Typical mechanical properties

Composite	Fibre vol. (%)	Typical properties UTS (MPa)	*E* (GPa)	Failure strain (%)	Comments
Carbon fibre					
P55/AZ91C+AZ31B faces	12.7	367	85.5	0.29	Single ply
P55/AZ91C+AZ31B faces	23.3	543	135	0.48	Three ply
P100/AZ91C+AZ31B faces	28.4	574	237	0.42	Single ply
P100/AZ91C+AZ61A faces	37.0	489	290	-	RT/Tensile
		284	258	-	RT/Compressive
		294	247	-	149°C/Compressive
		214	263	-	260°C/Compressive
T300/MgAl1	45	440	105	-	Longitudinal
		150	30	-	Transverse
M40/Mg	65	1500	235	-	-
Silicon carbide					
SCS-2/ZE41	34	1000	169.6	0.83	675°C/5 mins
	46	1524	209.6	0.88	675°C/10 mins
	50	1331	230.3	0.78	
	37	1379	180.6	0.95	

44.4.5 Environmental resistance

44.4.5.1 Galvanic corrosion

Magnesium MMCs suffer from one severe problem in that they are highly susceptible to galvanic corrosion; this is due to the potential difference between fibre and matrix.

A study on the corrosion of T300 carbon fibres in Mg-1Al alloy showed corrosion occurred in a laboratory environment and corrosive penetration was in the order of 100μm per year, Ref. [44-2].

Further studies have shown, however, that both the selection of matrix alloy and fibre coating techniques can reduce detrimental corrosion effects.

Galvanic corrosion can be controlled by proper and adequate protection methods, such as painting. Such protection methods rely on being intact and not disrupted during the entire manufacturing, assembly, storage and service life of the finished item, Ref. [44-19].

Whilst the mechanical and thermal expansion of these materials shows promise for space applications, considerable effort is needed to reduce the corrosion aspects and production costs.

[See also: ECSS-Q-70-71]

44.5 Potential applications

44.5.1 Alloys

The very low density of magnesium is an obvious attraction, and although the stiffness and strengths of the alloys are modest, these are good when considered on a 'specific' basis i.e. strength to weight ratio. Satellite applications have used AZ31, AZ61 (AZM), AZ80 and ZK60 alloys.

A Mg-Li development alloy, Ref. [44-12], has a very low specific gravity of 1.35, although it never reached commercial status and is now obsolete. However, work continues on other Mg-Li alloys and composites based upon them, Ref. [44-13], [44-14].

[See also: ECSS-Q-70-71]

Conventional alloy developments have increased properties at elevated temperatures, e.g. RZ5 (ZE41A) to 130°C; WE43 and WE54 to 300°C (short-term) and 250°C (long-term) and the newer alloy Elektron 21 shows excellent promise. These alloys are finding uses in applications such as transmission casings for military, civil aircraft and helicopters, Ref. [44-8].

Magnesium Elektron have recently announced the development of a new extruded alloy, 'Elektron 675', which is claimed to have greater strength at temperatures above 100°C than wrought 2000-series and 7000-series aluminium alloys. At 200°C, yield strength is 290MPa, and the ultimate tensile strength is 380 MPa. This is over 100% stronger than 2024 aluminium, and more than 200% stronger than 7075.

44.5.2 Composites

Improvements in stiffness can be achieved by the addition of fibres and particles to give composites.

Specialised components can be made in continuous fibre composites, mostly with carbon fibre for dimensional stability. However, for long-life structures, the corrosion characteristics of magnesium-based composites causes concern and the economics of production of these materials has mitigated against their development and wider availability.

The field of metal-matrix composites is still relatively new and rapidly developing. To assist the engineer in updating information on technology and potential suppliers a Thematic Network entitled MMC-Assess has been established with E.C. funding and is administered by the University of Vienna. It provides engineers with the status of these materials.

44.5.3 Space

44.5.3.1 General

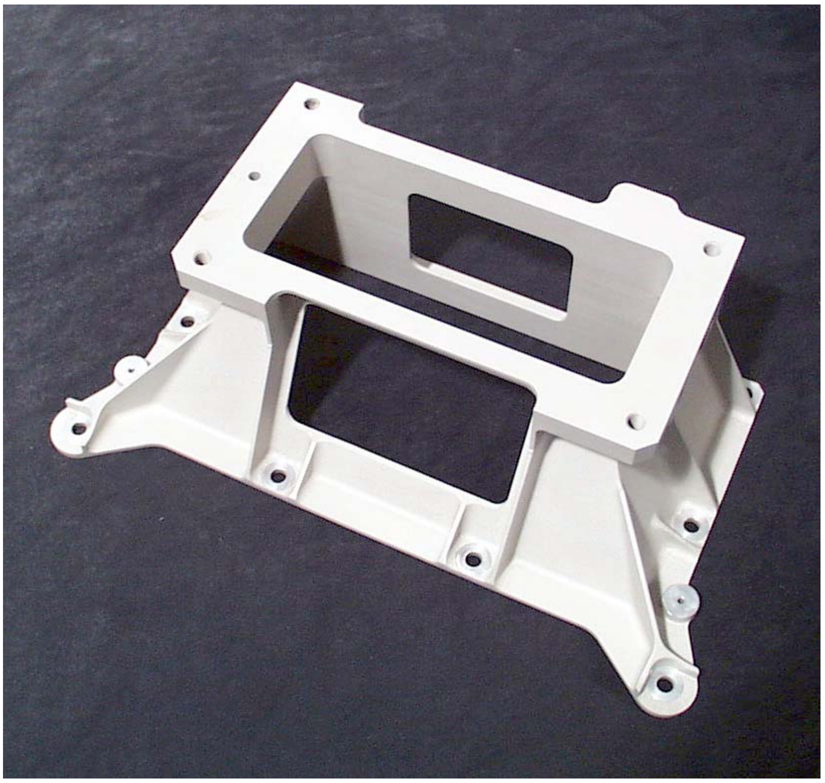
Magnesium alloys are most likely to be used in the form of machined castings, forgings or extruded rod or bar. However, as in the case of aluminium alloy aerospace components, there is a growing trend to use extruded billet for forging stock or for precision machining for longer components.

Given the mandatory limitations imposed on the use of magnesium materials in space, possible applications remain:

- Dimensionally-stable tubes and struts.
- End-fittings and connections for lattice structures.
- Mirror substrates.

44.5.3.2 Satellite

Figure 44.5.1 shows a magnesium alloy momentum wheel bracket for a satellite. This item is 250 mm × 150 mm × 80 mm and was machined from ZK60A-T5 extruded bar and treated with ALGAN 2 plus OXSILAN® MG 0611, Ref. [44-22].



This item is 250 mm × 150 mm × 80 mm and was machined from ZK60A-T5 extruded bar and treated with ALGAN 2 plus OXSILAN® MG 0611, Ref. [44-22].

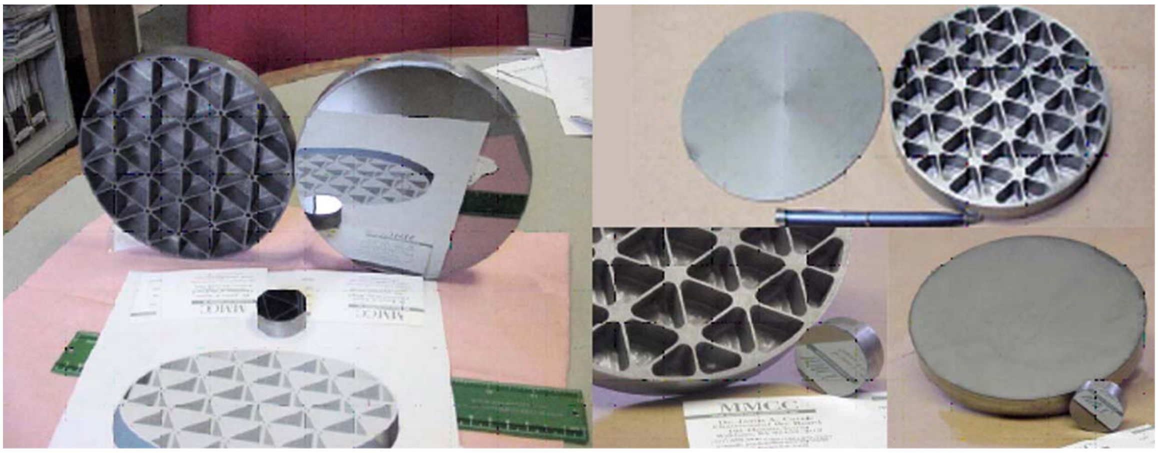
Figure 44.5-1 - Magnesium alloys: Momentum wheel bracket for satellite - Example

44.5.3.3 Mirrors

Magnesium-based composites are under consideration for light weight mirrors. MetGraf Mg™ is a tradename of MMCC (USA)

Figure 44.5.2 shows an example of a carbon-reinforced magnesium alloy lattice structure used to support silicon-based reflectors, Ref. [44-16].

Attachment of Si and SiC membranes to
Lightweighted MetGraf Mg substrates with pre and post polishing options



Pre-polished Si membrane bonded to lightweighted substrate.

Areal density = 9.7 kg/m^2

SiC membrane bonded to Mg/Carbon lightweighted substrate.

Part to be Si coated and polished

Figure 44.5-2 - Carbon fibre-reinforced magnesium alloy: Space mirrors (experimental) - Example

44.6 References

44.6.1 General

[44-1] 'Properties and Selection: Non-ferrous Alloys and Special Purpose Materials'
ASM Handbook, 10th Edition, Volume 2
ASM International. ISBN 0-87170-378-5(V.2)

[44-2] B.J. Maclean et al
'Thermal Mechanical Behaviour of Graphite/Magnesium Composite'
Martin Marietta Aerospace Proceedings of Symposium on Mechanical Behaviour of Metal Matrix Composites
Dallas, Texas. February 16th to 18th, 1982

[44-3] I.W. Hall
'Corrosion of Carbon/Magnesium Metal Matrix Composites'
University of Delaware Scripta Metallurgica. Vol. 21, 1987

[44-4] D.J. Chang et al
'Compressive Properties and Laser Absorptivity of Unidirectional Metal Matrix Composites'
The Aerospace Corporation Report No. SD-TR-8681
30th September 1986

[44-5] Engineered Materials Handbook. Volume 1. COMPOSITES
ASM International, Metals Park, Ohio

ISBN 0-87170-279-7(v.1)

[44-6] B. Hussey & J. Wilson: RJ Technical Consultants, UK
'Light Alloys Directory & Databook'
Chapman & Hall (1998). ISBN 0 412 80410 7

[44-7] H-J. Ertelt: ISD – Institute for Statics & Dynamics, D
'Some Fatigue Properties of Magnesium Alloys'
Proceedings of ESA European Conference on Spacecraft Structures, Materials & Mechanical Testing, Braunschweig, D. 4-6 November, 1998. ESA-SP-428, p263-268

[44-8] Extract from Magnesium Elektron website: www.magnesium-elektron.com (April 2003).

[44-9] Data Sheet 256, 'Surface Treatments for Magnesium Alloys in Aerospace & Defence'
Magnesium Elektron, 1997: www.magnesium-elektron.com

[44-10] Data Sheet 254, 'Machining Magnesium Alloys'
Magnesium Elektron, 2000: www.magnesium-elektron.com

[44-11] 'Fatigue and Fracture'
ASM Handbook, volume 19
ASM International. ISBN 0-87170-385-8

[44-12] NASA SP–5028,
'Technical and Economic Status of Magnesium-Lithium Alloys', Paul D. Frost, August 1965
http://ntrs.nasa.gov/archive/nasa/casi.ntrs.nasa.gov/19650020351_1965020351.pdf

[44-13] F. W. Bach et.al.
'SFB 390 Prject A4: Density Reduced Magnesium Alloys with Increased Ductility'
University of Hanover, Germany
http://www.iw.uni-hannover.de/sfb/sfb390/englisch/index_e.html

[44-14] S. Kudela
'Magnesium- lithium matrix composites – an overview'
Int. J. of Materials & Product Technology, Vol. 18, Nos 1/2/3, 2003, p91-115

[44-15] A. A. Luo
'Recent magnesium alloy developments for elevated temperature applications'
International Materials Reviews, 2004, Vol. 49, No 1, p13-30

[44-16] J.A.Cornie, L. Ballard, E. Chen & S. Zhang
'Development of graphite fiber reinforced magnesium alloys for lightweight mirror substrates and zero CTE metering structures'

National Space & Missile Materials Symposium: Seattle, 21-25 June 2004
LINK

[44-17] J.F.King, T.E.Wilks & G.D.Wardlow
'Properties and applications of magnesium MMC materials'
IMA 53, conf. Proc., Ube, Japan, 1996, p77-82

[44-18] A. Schoberth
'Overview on continuous fibre reinforced light metals'
MMC VIII, Metallic Composites & Foams, 26/27.11.2001, The Institute of Materials, London
http://mmc-assess.tuwien.ac.at/public/cont_fibers.pdf

[44-19] A.Schoberth
'Continuous carbon fiber reinforced Mg-alloys'
paper in 'mmc-data' section of MMC-Assess website
http://mmc-assess.tuwien.ac.at/index1.htm

[44-20] ASTM B275-04: Standard Practice for Codification of Certain Nonferrous Metals and Alloys, Cast and Wrought
ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, USA

[44-21] ASTM E 527-83 (Reapproved 2003): Standard Practice for Numbering Metals and Alloys (UNS)
ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, USA

[44-22] AMTS (Israel) – private communication (2005)

44.6.2 Sources

Alubin: www.alubin.com
AMTS, Wrought Product Division: www.magnesium-technologies.com
Dead Sea Magnesium: www.dsmag.co.il
Hydro Magnesium: www.hydromagnesium.com
Magnesium Elektron: www.magnesium-elektron.com
Otto Fuchs: www.otto-fuchs.com
Timminco: www.timminco.com

44.6.3 Organisations

International Magnesium Association : www.intlmag.org
Israeli Consortium for the Development of Magnesium: www.dmtc.org.il
MMC-Assess: mmc-assess.tuwien.ac.at

44.6.4 ECSS standards

[See: ECSS web site]

ECSS-Q-70-71	Data for the selection of space materials and processes; previously ESA PSS-01-701
ECSS-Q-ST-70-36	Material selection for controlling stress-corrosion cracking; previously ESA PSS-01-736
ECSS-Q-ST-70-37	Determination of the susceptibility of metals to stress-corrosion cracking; previously ESA PSS-01-737

44.6.5 ASTM standards

[See: ASTM web site]

ASTM B275-04	Standard Practice for Codification of Certain Nonferrous Metals and Alloys, Cast and Wrought
ASTM E 527-83	Standard Practice for Numbering Metals and Alloys (UNS). Reapproved 2003

45 Copper alloys and their composites

45.1 Introduction

Copper alloys are not considered for structural applications, unless the application has exacting electrical or thermal conductivity requirements.

The mechanical properties of the alloys can be improved by using reinforcement, but the conductivity often suffers as a result.

Some copper alloys exhibit a shape memory effect. These alloys, sometimes known as 'smart materials', can be used as actuators, [See: 91.5].

45.2 Copper alloys

45.2.1 Precipitation hardening

The range of copper alloys is large, Ref. [45-1]. Those alloys which are of preliminary interest include the precipitation-hardened systems which have enhanced strengths without loss of conductivity. These alloys include:

- Cu-Be,
- Cu-Ag,
- Cu-Zr,
- Cu-Cr.

An example is Narloy Z.

These alloys form the baseline for many industrial applications, but can be considered as lacking in certain areas of property optimisation; most notably high-temperature creep resistance (~500°C) and dimensional stability. This has initiated some reinforced copper and composite developments appropriate to space applications.

45.3 Copper ODS alloys

45.3.1 Types and effects of dispersions

Adding very fine dispersions of alumina (Al_2O_3) and titanium diboride (TiB_2), in low concentrations, has achieved significant improvements in selected properties over precipitation hardened systems.

Copper ODS alloys are prepared by a powder route.

45.3.2 Properties

Whilst maintaining good conductivity, the hardness, wear resistance and creep resistance have been improved. This is most noticeable in the retention of strength to higher temperatures than the conventional alloys, as shown in Figure 45.3.1.

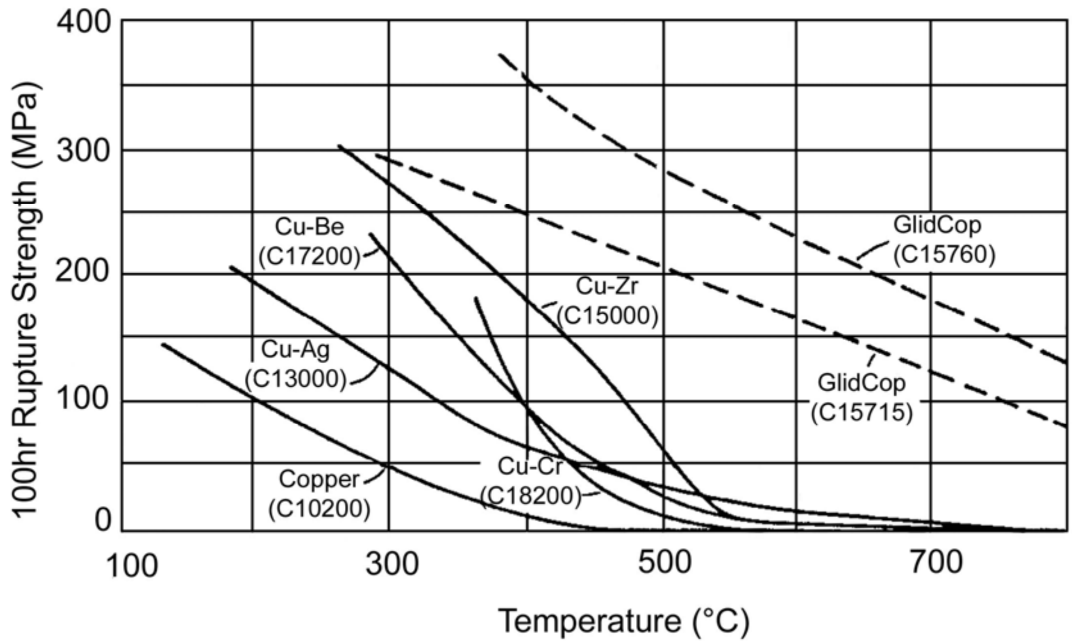


Figure 45.3-1 - Copper ODS alloys: Elevated temperature stress-rupture properties of Glidcop ODS alloys compared with some high conductivity copper alloys

Table 45.3.1 gives the ambient temperature properties attainable by ODS alloys, whilst Table 45.3.2 and Table 45.3.3 provide data at 538°C.

Table 45.3-1 - Copper ODS alloys: Ambient temperature tensile properties

Material/Temper [See: Key]	Modulus (GPa)	Proof stress $\sigma_{0.2}$ (MPa)	UTS (MPa)	Strain to failure (%)
Cl5710: Cu + 0.2 wt.%. $Al_2 0_3$, †				
As fabricated	105	270	325	20
Cold worked 98.5% RA	106	540	565	-
15720: Cu + 0.4 wt.%. $Al_2 0_3$, †				
As fabricated	113	365	470	19
Cold worked 98% RA	113	585	615	3.5
Cl5735: Cu + 0.7 wt.%. $Al_2 0_3$, †				
As fabricated	123	420	485	16
Cold worked 56% RA	123	565	585	10
Cl5715, Glidcop Al-15: Cu + 0.3 wt.%. $Al_2 0_3$ ‡				
As hipped	130	255	360	26
As extruded	130	331	413	20
Extruded + 98% cold work	130	613	661	6
Cl5725, Glidcop Al-25: Cu + 0.5 wt.%. $Al_2 0_3$, ‡				
As hipped	130	295	410	18
As extruded	130	345	4,34	21
Extruded + 98% cold work	130	613	675	6
Glidcop Al-25: Cu + 0.5 wt.%. $Al_2 0_3$, ‡				
As hipped	130	413	517	13
As extruded	130	655	737	6
Sutek MXT-5 Cu + 5 vol.% TiB_2 *ƒ*				
Extruded and cold rolled 8% reduction in thickness	-	620	675	7

Key: † ASM Metals Handbook, 10th Edition, 1990.
‡ Manufacturers data, SCM Metal Products Inc, USA.
ƒ Manufacturers data, Sutek Corp, USA.

Table 45.3-2 - Copper ODS alloys: Short term properties at 538°C

Material	Temper ③	YS $\sigma_{0.2}$ (MPa)	UTS (MPa)	*e* (%)
C15715,GlidCop Al-15: Cu-0.3 wt.%Al_2O_3 ①	As HIP	85	85	~1
C15725,GlidCop Al-25: Cu-0.5 wt.%Al_2O_3 ①	As HIP	105	120	~2
Sutek MXT-5A: Cu-5 vol.% TiB_2 ②	Unknow n	145	145	~5

Key: ① Manufacturers data: SCM Metal Products Inc., USA.
② Manufacturers data: Sutek Corporation, USA.
③ HIP: Hot isostatic pressing

Table 45.3-3 - Copper ODS alloys: Creep strength at 538°C

Material (Temper not stated)	100 hr Creep rupture strength (MPa)
C15715, GlidCop Al-15: Cu-0.3 wt.%Al_2O_3	190
C15760, GlidCop Al-60: Cu-1.1 wt.%Al_2O_3	260

45.4 Discontinuously reinforced copper composites

45.4.1 General

There is virtually no published information on the use of particle or discontinuous fibres to reinforce copper.

45.4.2 Problems

Indications are that:

- Additions of inorganic, insulative phases such as SiC, Al2O3 and B4C noticeably reduce conductivity characteristics.
- Addition of particles to precipitation-hardened systems is likely to affect the microstructure of the matrix alloy.
- The addition of particles to pure copper does not give substantial improvements in strength to match precipitation-hardened or ODS alloys.
- Problems arise in obtaining satisfactory bonding between particle and matrix, Ref. [45-2].

45.5 Continuously reinforced copper composites

45.5.1 General

These materials have been studied in the USA to provide support for the space programmes.

45.5.2 Types and effects of reinforcements

45.5.2.1 Carbon fibre

Interest has centred on carbon fibre (graphite) reinforced materials using pitch fibres (Amoco P100).

The pitch fibres contribute to:

- Thermal conductivity: The pitch fibres increase the conductivity beyond that possible with copper alone.
- Very low coefficient of thermal expansion, albeit anisotropic.

45.5.2.2 Tungsten filaments

Composites with 10% tungsten filaments have been proposed for combustion liners where:

- hoop strength can be increased by 90% with only a 4% loss in conductivity.

This suggests opportunities for the copper wall thickness to be reduced to increase its heat transfer coefficient without sacrificing strength.

45.5.3 Properties

45.5.3.1 General

Detailed information on materials is awaited.

45.5.3.2 Thermal characteristics

Table 45.5.1 presents the thermal characteristics of fibre-reinforced copper compared with carbon fibre/aluminium composites.

Table 45.5-1 - Continuous fibre reinforced copper: Thermal conductivity of carbon/Cu and carbon/Al composites

Material ‡	Thermal conductivity (W/m K)		CTE (ppm/°C)		Density (kg/m^3)
	Long.	Trans.	Long.	Trans.	
Pure Copper †	398	398	16.5	16.5	8960
P100/Copper (UD)	454	140	2.0	11.7	5600
P100/Copper (0°/90°)$_s$	305	305	5.76	5.76	5700
P100/6063 Al (UD)	339	94	1.62	26.6	2450
P100/6063 Al (0°/90°)$_s$	216	216	5.04	5.04	2470

Key: † Properties given for comparative purposes.
‡ Nominally 40% carbon fibre.

45.6 Potential applications

Copper is not considered a structural material because its density is too high with respect to its strength and stiffness. Its attributes mainly relate to its excellent electrical and thermal conductivity.

For practical systems using these conductivity characteristics there is a need to improve the mechanical properties, notably wear resistance and high-temperature stability. Unfortunately most methods of achieving this tend to impair the conductivity.

Two materials with possible uses in space are oxide dispersion strengthened (ODS) alloys and carbon (pitch) fibre reinforced copper.

Potential applications include:

- Rocket engine combustion liners.
- Heat exchangers and radiators.
- Space power systems.

45.7 References

45.7.1 General

[45-1] Metals Handbook, Volume 2, Tenth Edition
Properties and Selection: Non-ferrous Alloys and Special Purpose Materials
ASM International, ISBN 0-87170-378-5(v.2), 1990

[45-2] J.F. Mason
'The development of High Conductivity/Thermally Stable Copper-based Composites'
Work Order No. 17. BNF-Fulmer Report R1176/10/1
November 1990 for ESTEC Contract 7090/87/NL/PP