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Guidelines for carbon and other advanced fibre prepreg procurement specifications

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ABSTRACT

This guideline contains information to assist in the preparation of a specification for the procurement of advanced composite prepregs. The guideline is biased particularly towards the special requirements of carbon-fibre-reinforced plastics for use in spacecraft structures.

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CHAPTER 1: PURPOSE OF THE GUIDELINE

This document is intended as a general guide to organisations preparing specifications for the procurement of particular thermosetting-resin-impregnated reinforcing fibre systems (prepreg). These are primarily epoxy-, bismaleimide- and polyimide-matrix-based materials. The parameters that it is necessary to control and/or monitor for qualification and batch control of a prepreg are described. This guideline is not intended to be a definitive prepreg specification. In many cases, it may not be necessary to specify all the parameters outlined. It is left to the judgement of those preparing specifications to include only what is really necessary, thereby avoiding excessively complex specifications and the unduly high cost of complying with them.

Whilst this guideline concentrates on the procurement of thermosetting-resin prepreg, it is, in many respects, applicable to thermoplastic-matrix prepregs as well. Thermoplastic prepregs are chemically stable, having an infinite shelf-life at room temperature. This document is not relevant to non-polymeric matrix prepregs.

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CHAPTER 2: DESCRIPTION, SCOPE AND CLASSIFICATION OF PREPREG

This guideline is aimed primarily at purchasers of prepreg containing carbon fibres. Many of the parameters described, however, 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, require similar specification control.

The commonest type of prepreg is the unidirectional variety. A unidirectional prepreg contains a parallel planar array of fibres, usually several fibres thick, suitably preimpregnated with either a partially cured or an uncured 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 may be defined by reference to appropriate specifications for these constituents. (See, for example, ESA PSS-56 Issue 1 (April 1981) : Guidelines for a resin procurement specification.)

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 and/or stiffness characteristics. The classification of a prepreg may 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. Alternatively, the type of resin and/or fibre may be used as a basis for prepreg classifications. In either case, the material supplier should provide quidelines on the range of applicability of a particular prepreg and its constituents.

Section 1 noted the recent availability of thermoplastic matrix prepregs. These are supplied in unidirectional and fabric forms, but do not possess the same intrinsic physical characteristics of thermoset prepregs such as drape and tack. A procurement specification of thermoplastic matrix prepregs contains fewer requirements than one for thermoset prepregs, since material content and distribution are important but factors relating to incomplete matrix chemistry are less critical.

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CHAPTER 3: DEFINITION AND BACKGROUND

Owing to the multiplicity of factors that can influence prepreg properties, the procurement of prepreg and the preparation of a suitable specification can be a bewildering task. The relevant factors are outlined in Appendix 1 and are explained where necessary; bracketed numbers in the body of this document, e.g. (A.1.1.), refer to the paragraph in Appendix 1 in which the particular term is defined.

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CHAPTER 4: PREPREG REQUIREMENTS FOR QUALIFICATION AND BATCH TESTING

It is envisaged that all the requirements in this section will be satisfied for the qualification of a prepreg to a specification. Many of the requirements will be necessary for batch release to a specification, and these are indicated in Section 4.6. It is usually the responsibility of the user to define for himself 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 system and fibre in the prepreg will have been qualified, batch released and accepted according to appropriate specifications.

4.1 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.

4.1.1 Reeled Material

It is usually required that each reel of prepreg be legibly and durably marked by label or tag that is so securely attached that it remains in place until all the material on the reel has been used. The markings should include at least:

4.1.1.1 Material specification and issue number.

4.1.1.2 Prepreg batch number and reel number.

4.1.1.3 Fibre and resin designation, and their proportions.

4.1.1.4 Recommended storage conditions and expiry date under ambient conditions and in a refrigerated store, account being taken of environmental history prior to delivery.

4.1.1.5 Length and width of reeled material.

4.1.2 Sheet Material

Each bag of sheet material should be labelled as set out in Paragraph 4.1.1 and, in addition, each sheet within the bag should be labelled as set out in Subparagraphs 4.1.1.1., 4.1.1.2 and 4.1.1.3, sheet number being substituted for reel number in 4.1.1.2.

4.2 RELEASE DOCUMENTATION

It is the usual practice for information additional to that set out in Section 4.1 to be supplied as release documentation accompanying each batch or shipment (whichever is the smaller) of material released to a specification. This information usually includes many of the following:

4.2.1 Manufacturer's name and material code.

4.2.2 Material specification and issue number.

4.2.3 Manufacturer's prepreg batch number and any other identification markings required.

4.2.4 Date of manufacture.

4.2.5 Purchase order number.

4.2.6 Fibre type and batch number(s).

4.2.7 Resin type and batch number.

4.2.8 Fibre/resin content.

4.2.9 Prepreg mass per unit area.

4.2.10 Length and width of reeled material or sheet size and number of sheets of sheet material.

4.2.11 Required shipping and storage conditions and expiry date for storage at a specified sub-zero temperature and at shop-floor ambient temperature.

4.2.12 Laminate properties derived from prepreg from the batch.

4.2.13 Volatile content and type. (This information is usually only supplied where it is important, e.g. for prepreg for use in spacecraft component fabrication.)

4.2.14 Inspection record, including location and nature of any rejected material.

4.2.15 Any special order requirements.

4.2.16 Differential scanning calorimetry (DSC), High Pressure Liquid Chromatography (HPLC), Infra-Red Spectroscopy and Dynamic Mechanical Analysis (DMA) are used for investigating the cure properties of prepreg materials. Data from these tests can provide confirmation of the expected cure properties. Chromatography and Spectroscopy are used for batch testing, whereas DSC and DMA are more appropriate for prepreg qualification.

4.3 Packaging

Both faces of a prepreg must be protected by nonmigratory, noncontaminating backing materials. These materials should be crease-free, readily removable from the prepreg (without damaging the prepreg) under defined conditions, and substantially dimensionally stable over the anticipated storage temperature and humidity ranges. The backing material should be readily distinguishable from the prepreg, and the upper and lower faces of the material should be identified by different colour backing materials. It is recommended that backing materials should be marked on the external surface (visibly when rolled in the case of tape) with continuous 0° axis lines about 1 cm apart, and with agreed prepared identification and batch markings at specified intervals along the entire length. The backing material should be trimmed to the prepreg edge and cut cleanly. There should be no risk of contamination by the marking material.

Prepreg tape should be wound into reels of a core diameter which is usually not less than 20 cm for 125-μm-thick carbon tape. Thicker and higher-modulus tape, e.g. boron, will require a larger core diameter. The tape should be contained within the reel edges and wound in such a manner as to prevent wrinkling or buckling of the inner layers. Each reel of tape should be supplied in its own sealed, moisture-proof plastic bag. Sheet material should be supplied in a sealed, moisture-proof plastic bag adequately protected and supported to prevent distortion and damage.

For thin prepreg, the backing material should be sufficiently compliant to permit draping of the prepreg without wrinkling.

4.4 PREPREG CONSTRUCTION

This section outlines the construction parameters of a prepreg that are commonly specified. Bracketed numbers (e.g. A.1.1) refer to definitions of terms given in Appendix 1.

4.4.1 General

It is the usual practice to include a brief description of the major characteristics of the prepreg, e.g. 'the prepreg should consist of unidirectionally aligned' (if appropriate) 'continuous carbon fibre tows' (A.1.1, A.1.2, A.1.3), 'uniformly spread, impregnated and wetted with the resin system. Sheet and unrolled tape prepreg should lie substantially flat, the tows being straight and parallel to each other throughout and' (unless otherwise ordered) 'parallel to the edge of the prepreg'.

Any batch restraints on the fibre and resin constituents within a prepreg batch (as outlined in A.1.8) are usually defined at this stage.

4.4.2 Thickness

The thickness to be defined is the nominal thickness of the prepreg when the latter is cured to a specified fibre-volume-fraction laminate.

4.4.3 Cohesion

It is usually required that a prepreg should have a sufficient degree of cohesion (A.1.l6) at a specified shop-floor ambient temperature to permit handling, trimming and laying of the material without affecting the fibre distribution, orientation and degree of consolidation.

4.4.4 Gaps and Slits

The maximum cumulative gap of slit width between tows is usually specified as a function of prepreg width. The maximum length of a single gap or slit of the maximum permissible width may be defined per unit area of the prepreg.

4.4.5 Splicing

Splices in the prepreg are usually treated as defective areas, as described in Section 4.5. Splices of carbon fibre tow are usually permissible when the two lengths of tow being joined are of similar mass per unit length and the number of splices does not exceed a defined maximum per unit mass of tow. If necessary, the minimum and maximum acceptable overlap length of a splice may be specified.

4.4.6 Dimensions

Prepreg may be processed to a particular width or slit from wide-stock material. In either case, a width will be specified, as will a tolerance. It is conventional practice to specify a nil negative tolerance and a positive tolerance up to 2%, although other tolerances may be agreed between the purchaser and the manufacturer. Close tolerances on width can be very critical for narrow unidirectional prepreg used in filament winding where precision in lay-up is of paramount importance. The minimum length and the tolerance on length of a prepreg (which may also be expressed in terms of mass) are usually defined on a purchase order.

4.4.7 Resin Content (A.1.11)

The resin content can be defined as a resin mass per unit area or as a percentage of the mass of the prepreg. In either case, any resin size applied to the tow should be included in the resin content. When a resin content is expressed as a mass per unit area, it is common practice when the resin is used in conjunction with carbon fibre to allow a ±10% tolerance on the value. When resin content is expressed as a percentage of the prepreg mass, a tolerance of ±3% or less on the value is appropriate.

4.4.8 Fibre Content (A.1.11)

The fibre content can be expressed as a mass per unit area and/or a volume per unit area of the prepreg. In the case of carbon fibre, a tolerance of approximately ±5% on fibre mass is usually allowable. This tolerance is also appropriate for aramid and glass fibres.

4.4.9 Prepreg Mass

Prepreg mass should be defined as a mass per unit area. The range of tolerance allowed on the value is somewhat variable, depending on the precise manner of specification of fibre and resin contents, but is commonly about ±6% for carbon-fibre prepreg.

4.4.10 Volatile Content (A.1.2O)

It is common practice to specify a total volatile content in a prepreg. The quantity will depend on the application and processing techniques. The maximum allowable content may typically vary between 1 and 2% for epoxies and BMI.

4.4.11 Tack

It is common practice to specify an acceptable degree of tack in the prepreg. This is usually a fairly qualitative assessment, defined in terms of the parameters outlined in A.1.17.

4.4.12 Edges

If so desired, a specification may contain a requirement that there should be no resin-rich area at the edge of the prepreg.

4.4.13 Flammability, Toxicity and Safety

Recent industrial and environmental legislation in several European countries has led to restrictions concerning the use of various flammable, toxic or otherwise potentially hazardous materials. Where such materials are incorporated into a product, the supplier is required by law to inform a customer of that fact and to appraise him of the precautions to be taken when the product is used. If so desired, part or all of such legal requirements can be incorporated into a specification. For special end uses, the customer may restrict the constituents that the supplier may incorporate into the prepreg.

All safety-related details shall be specified in a Safety Data Sheet.

4.4.14 Shelf Life and Shop Life (A.1.18, A.1.19)

If so desired, minimum shelf and shop lives under defined storage conditions can be included in a prepreg specification. Such a condition in a specification should be the subject of close consultations between the manufacturer and the customer in order to ensure that the requirement is feasible, practicable and not excessively expensive. Similarly, the constraints on re-lifing (see A.1.18) should likewise be defined only after consultation between manufacturer and customer.

Note: Acceptable tack is frequently the parameter defining the usability of a prepreg.

4.4.15 Fabrics

For prepregs based on fabrics, there are descriptive parameters for each commercial product, these include:

* Weave type
* Areal weight (g/m^2)
* Warp characteristics: ends/cm and yarn denier (or tex).
* Weft characteristics: picks/cm and yarn denier (or tex).

The warp and weft yarns shall be perpendicular to each other on conventional 0°/90° fabrics. All warp and all weft yarns need to be parallel with each other in the prepreg. The maximum allowable misalignment shall be less than 5 mm over 500 mm taken widthwise or lengthwise; this implies that any misalignment should not be discernible to the human eye.

4.4.16 High-pressure Liquid Chromatography (HPLC)

HPLC is an analytical technique for determining the chemical constituents of the prepreg matrix. Testing a batch of prepreg makes it possible to take the results and a reference trace (form of fingerprint) for the same prepreg to provide a means of comparison on product consistency. Similar reasoning can be used for applying infrared spectroscopy.

4.4.17 Differential Scanning Calorimetry (DSC)

DSC provides a means of assessing the cure characteristics of the supplied prepreg batch. Like HPLC it is an analytical technique providing data on which to make comparisons. It will provide:

* Reaction start temperature
* Heats of polymerisation
* Temperature at peak maximum heat of polymerisation
* Glass transition temperature (T_g) of cured prepreg.

4.4.18 Dynamic Mechanical Analysis (DMA)

DMA can be used on uncured prepreg and cured laminates to quantify some of their mechanical properties over a range of temperatures. DMA provides a measurement of storage modulus and loss modulus in a shear or flexural mode. From these it is feasible to judge the cured material for consistency on expected modulus and modulus retention at elevated temperatures. The glass transition temperature (T_g) of cured material can also be determined. There are techniques complementary to DMA, such as TBA (Torsional Braid Analysis) and other torsional testing techniques.

4.5 PREPREG QUALITY AND DEFECTS

Prepreg material as supplied inevitably exhibits some defects. Defective material is a significant area (to be defined by agreement between manufacturer and customer) containing discontinuous tows (excluding allowable tow splices, as defined in Paragraph 4.4.5), prepreg splices, cured or hardened resin particles, fibre-starved areas, excess resin or resin-starved areas, gaps or slits in excess of those defined in Paragraph 4.4.4, whorls, fuzz balls, any imperfection in fibre alignment due to tow slackness (such as kinks, waves or twisted fibres), foreign material, contamination and any other features that could adversely affect its performance.

To reject material containing any defects is impracticable, and it is usually permissible for defective prepreg to be supplied to a specification, provided certain conditions are met. Typically, these are:

* That the beginning and end of each defective length of prepreg should be clearly marked by a suitable flag placed close to one edge of the backing material.

* That each continuous length of released material should satisfy the purchaser's minimum length requirement on defect-free material.

* That, for reeled material, the location of the beginning and end of each defective length, given as a distance into the reel, should be stated on an inspection sheet supplied with each flagged reel.

* That, for sheet material, the quantity of defective lengths on each sheet should be stated on an inspection sheet supplied with each bag of sheet material.

Failure to comply with these conditions should be sufficient cause to justify the rejection of the prepreg by the purchaser. Defective lengths incorporated in prepreg released to a specification should not be regarded as part of the amount delivered for payment purposes.

4.6 BATCH ACCEPTANCE TESTING BY THE SUPPLIER

4.6.1 Test Parameters

It is usual for the selection of the qualification requirements to be tested for each prepreg batch. Typically, the following would be tested at the beginning and end of each batch, the results being reported in the release documentation (see Section 4.2.):

* Gel time (usually specified in the resin procurement specification) (A.1.14).

* Resin content (The resin extracted for this measurement may be used, if so desired, for the identification of the resin, hardener system(s) and/or catalysts, and their proportions.) (A.1.11).

* Fibre content (A.1.11).

* Volatile content (A.1.20).

* Tack (A.1.17.).

* Laminate properties (see Section 5).

Additionally, each roll of prepreg tape or bag of prepreg sheet would usually be tested once for prepreg mass per unit area.

If a large batch is purchased, i.e. more than one thousand metres, intermediate testing within the batch is appropriate. One in ten rolls should be tested, each roll being 100 metres in length.

Continuous visual inspection is usually required during production of a prepreg batch to ensure that the prepreg quality requirements set out in Section 4.5. are met.

Suppliers checks and procedures should be examined and approved by the purchaser or user.

4.6.2 Batch Acceptance/Rejection and Retest

The material should satisfy all the requirements laid down by the specification for acceptance. Failure to meet any of the requirements should be cause for rejection of a material batch.

Should the results obtained in a particular property test fail to meet the specified requirement, it is conventional practice to permit one retest in respect of that property. For uncured prepreg, duplicate sets of specimens are taken from opposite ends of the batch for retest, and both should meet the requirement. The results of both the original test and the retest should be included in the release documentation. Failure to meet the cured laminate requirements on retest should be cause for batch rejection, unless the customer agrees that both test and retest failures were demonstrably not due to a basic fault in the prepreg.

4.7 INCOMING INSPECTION BY THE USER

It is common practice for a user to check incoming material by retesting for some or all the batch-acceptance requirements. This is at the user's discretion and is not normally incorporated into a procurement specification.

4.8 TREND ANALYSIS BY THE USER

When a specific prepreg system is regularly purchased over a long period, it is recommended that a trend analysis be maintained on the properties obtained from the prepreg and its composites. Experience has shown that products with the same designation can be 'improved' by the supplier over a period of time. Whilst the minimum properties of the specification are always exceeded there can be subtle increases in composite strength and modulus values. This may introduce problems in some structural designs if the phenomenon goes undetected.

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CHAPTER 5: LAMINATE REQUIREMENTS

It is the usual practice to specify minimum physical and mechanical properties for a laminate produced from the prepreg by means of a defined cure schedule. As noted in Section A.1.15, this defined cure schedule is for specification purposes and need not correspond to the component production cure; it must, however, be within acceptable temperature limits. A number of curing-cycle repetitions having no adverse influence on the laminate's mechanical properties may be incorporated, if required. The following parameters and their interrelationships should be defined:

* Cure time (with allowable range);
* Cure temperature (with allowable range);
* Permissible range of heat-up and cool-down rates;
* Curing-pressure range and/or cycling;
* Pre- and post-cure dwell phases and their purpose.

If so desired, the tooling, prepreg stacking sequence, release and bleed cloth layers and stack assembly for preparing test laminates may all be specified. The sample-cutting pattern from the laminate may also be specified. It is the conventional practice to manufacture unidirectionally reinforced laminates for specification testing purposes. Special tests may be required in the case of woven fibre prepregs.

5.1 QUALIFICATION TESTING

Against defined test standards agreed between the supplier and purchaser, various of the following mechanical and physical properties should be determined at ambient temperature and humidity. If so required, additional elevated-temperature and/or humidity conditions may be prescribed.

* 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 performance.

The major part of batch testing concentrates on mechanical testing, because this provides data that are relevant to structural design and provides confidence in the material. As noted in Section 4.4, analytical techniques such as DSC and DMA exist. These are used to provide supportive evidence on laminate material consistency and rely on accumulated experience to provide a basis for interpreting the results.

5.2 BATCH AND INCOMING INSPECTION TESTING

The following is suggested as a practical minimum for realistic quality control testing of a prepreg, both for qualification and for batch and incoming acceptance purposes. Usually, a minimum of five individual tests for each property would be carried out, the individual results being recorded as part of the release documentation. Values quoted below are typical for carbon-fibre prepreg.

* **Fibre volume fraction:** to be within a specific tolerance - usually about ±3% of a defined figure that is usually 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, 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%.

However, other laminate tests may be defined to reflect the service requirement, e.g. compression tests and tests at elevated temperatures.

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CHAPTER 6: TEST METHODS

Unfortunately, it is not feasible to give totally 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/ANSI, but even this is not exhaustive. For some properties, e.g. prepreg tack, no national standard test methods have been published; the nonstandard ones that do exist have often been written especially for specification purposes. National and European standards that could be applicable are listed in Appendix 2.

This situation is far from ideal; one set of widely accepted standards would be preferable. However, all will provide satisfactory data provided supplier and user are in agreement on a common test method. Many of the individual standards for each test method are regularly updated and the most recent versions should be used.

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CHAPTER 7: SPECIFICATION STRUCTURE

There is a growing tendency for specifications to be written on what is described as the 'fiche-sheet' principle. The fiche-sheet structure requires the drawing-up of a master document in which ordering, delivery, qualification, acceptance, batch-release and test-method procedures are set out, but which does not include specific numerical property data requirements. A separate document — which is the 'fiche sheet' — is issued as a supplement to the master document and lists the required property data for a specific material. This 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 requires a special test or when various manufacturers have to be persuaded to supply a material to the same specification procedures.

For these reasons and others, most prepreg is still ordered to a specific material specification, drawn up by the manufacturers and the customer in collaboration.

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APPENDIX 1
DEFINITIONS AND BACKGROUND

Where appropriate, the definition is followed by an explanation of the importance of the parameter being defined in determining property consistency in a prepreg or laminate.

A.1.1 Tow

A loose, untwisted bundle of individual filaments, usually identified by fibre type (in terms of fibre tensile strength and elastic modulus) and the number of individual filaments, often in terms of thousands of filaments.

A.1.2 Filament

A continuous discrete fibre with an effective diameter — in the case of carbon fibres — in the range 6 to 10 μm. The cross-section is not necessarily circular. Other fibres are of different sizes, e.g. glass: 6 to 13 μm, aramid: $\sim$12 μm, alumina: $\sim$20 μm, boron: l00 to 200 μm and silicon carbide: 10 to 20 μm or 100 to 140 μm, depending on source.

A.1.3 Continuous filament tow

Tow produced by a continuous process without true twist.

A.1.4 Batch of fibre

A batch of fibre tow is that amount which is produced by the conversion of a number of precursor tows under standard, controlled, processing-plant conditions in one continuous operation, including any surface treatment and sizing of the fibre.

Changes in plant processing conditions (e.g. speeds, tensions and temperatures) affect the final mechanical properties of the fibre. Changes in surface treatment and sizing of the fibre can have a dramatic effect on the shear properties of laminates incorporating the fibre. Consistency in laminate properties thus requires unchanged conditions for several fibre manufacturing process parameters, and the consistency can only be completely assured within a particular fibre batch. Modern production methods usually produce sensibly consistent fibre properties from batch to batch, but some batch-to-batch variation can, and does, occur. Changes can be caused by changing the batch of PAN precursor used for carbon-fibre production. Where more than one batch of precursor has been used in one carbon-fibre batch, this should be stated by the supplier. Moreover, consistency in the properties of the carbon fibres derived from the different precursor batches should be tested for, and the results should be quoted on the release documentation.

A.1.5 Fibre surface treatment

Carbon fibres are usually given a post-carbonisation surface treatment by either a chemical or an electrochemical method. This surface treatment enhances the bond between the fibres and the resin matrix in the cured laminate. This enhanced bond improves the shear and compressive properties of the laminates, but if the surface treatment is overdone, brittle laminates can result.

A.1.6 Size

A size is often added to a fibre tow (in the case of carbon, after surface treatment). This size improves the handling characteristics of the tow and reduces filament damage during subsequent processing, e.g. weaving or prepreg manufacture. When a prepreg with an epoxy-resin matrix is to be manufactured, an epoxy-resin size containing no curing agents is usually specified. Size content with respect to the tow is frequently in the range 0.5 to 1.0% by weight.

A.1.7 Batch of resin

A batch of resin in either film or liquid form shall be a quantity of resin produced from one mix of resins, resin modifiers and curing agents.

Changes in the resin can affect certain properties of a cured laminate, such as shear strength and stiffness, water absorption, and properties at elevated temperature. Additionally, changes in the resin mix can affect the cure parameters necessary to ensure the manufacture of a sound, void-free laminate with a required fibre-volume fraction. Comments similar to those made regarding within-batch and batch-to-batch variation in Section A.1.4 apply to resin batches.

A.1.8 Batch of prepreg

A batch of prepreg is a quantity, irrespective of width, that is produced under 'no-change conditions' in one continuous operation of the impregnating plant from one batch of resin mix and one batch of fibre. A batch is expected to conform to a fixed manufacturing process and to have homogeneous properties within prescribed tolerances over its whole width and length. A maximum allowable length for a prepreg batch is sometimes specified.

Under exceptional conditions, a batch of prepreg may contain more than one fibre batch. Where this is the case, it is usual to specify some or all of the following requirements:

* Each batch of fibre to be incorporated in the prepreg shall have been used previously to supply at least one batch of prepreg to the same specification containing only that fibre batch.

* The previous batch or batches of prepreg shall have satisfied all the physical, mechanical and construction requirements of the specification.

* The release documentation accompanying the prepreg batch shall indicate the number of fibre batches used and the individual batch numbers of the previous prepreg batches containing these fibre batches.

Economic necessity sometimes requires the use of more than one fibre batch in a prepreg batch. Insistence on a single fibre batch within a prepreg batch could result in delivery delays or cost increase or both. If the precuations outlined are taken, it is possible to manufacture a sensibly consistent prepreg batch incorporating more than one fibre batch.

A.1.9 Tape

Prepreg of a continuous length including tape splices in the fibre direction (the position of which must be indicated by the tape supplier) and of any width, supplied on a reel.

A.1.1O Sheet

Prepreg of any width, supplied as flat sheet in the form of cut lengths.

A.1.11 Fibre and resin content

The fibre and resin contents are the amounts of the constituents contained in the prepreg, expressed as percentages by mass or volume. Procurement specifications for prepregs usually specify the fibre and resin contents in terms of mass per unit area, since this is the parameter that is usually controlled by the manufacturer for the prepreg and its constituents. Sometimes, the fibre volume per unit area of prepreg is specified in addition to the mass per unit area. Various ratios of fibre to resin may be specified for a prepreg, depending on the desired fibre content in the cured laminate and the flow characteristics during the cure of the resin.

A.1.12 Resin flow

Resin flow is a measure of the amount of resin exuded from the prepreg during cure processing. Resin flow is a measure of the transient reduction in viscosity that takes place in thermosetting systems as the temperature is increased, prior to gelation. Resin flow has an important effect on the production process and varies widely from system to system.

At one time, production of sound laminates relied on removal (bleed) of a substantial quantity of resin from a prepreg stack during cure. Increasing emphasis is now placed on resin systems that require little (low-bleed systems) or no (nil-bleed systems) resin removal for the production of sound laminates. Which type of resin is being used obviously influences the specified fibre/resin ratio in a prepreg.

A.1.13 Thickness

The thickness of prepreg is usually specified as a nominal value in the cured laminate, when cured to a particular value (often 60%) of fibre volume fraction in the laminate. Common unidirectional carbon-fibre prepreg thicknesses are 50 μm, 100 μm, 125 μm and 250 μm. Fabric-based prepregs vary in thickness over the range 50 to 450 μm depending on tow size and weave type. The thinner prepregs are more difficult to manufacture and require low-filament-count tow; they are therefore usually more expensive.

A.1.14 Gel time

The gel time of a resin is the exposure period required at a prescribed temperature to convert the resin from a fluid to a defined partial-cure stage. Resin bleed during cure can only occur substantially before gelation.

A.1.15 Cure cycle

The cure cycle defines the relationship between time, temperature and pressure required to achieve the correct laminate cure. The cycle may include defined heat-up and cool-down rates, isothermal holds for specified periods, and application and removal of negative and/or positive pressures at defined times or temperatures. The optimum cure cycle is usually specific to a particular type of prepreg, its layup and the component and tool design. Small variations in the prepreg (in particular in the resin) can have a dramatic effect on the cure cycle and bagging sequence needed to achieve a satisfactory laminate.

Note: Laminates may be cured so as to achieve, for example, the minimum degree of outgassing or other desired properties, rather than the optimum mechanical properties. Hence, any cure cycle incorporated as part of a testing schedule in a prepreg specification may be simpler than that for component production practice.

A.1.16 Cohesion

The requirement that the constituent components of the prepreg shall remain united in the required format for the specified storage and handling conditions.

A.1.17 Tack

Tack is a measure of the stickiness of the prepreg in relation to itself, the release or backing material with which the prepreg has been protected during storage and transit, and mould surfaces. A prepreg with very low tack ('dry') will be difficult to stack and laminate in such a way that the precured stack remains stable during layup and cure. A prepreg with a very high tack will be difficult to separate from its backing film without damage. Tack is probably the parameter most important in determining the acceptability or otherwise of a prepreg to a laminator on the shop floor.

A.1.18 Shelf life

The shelf life of a prepreg is the period of time during which it retains the specified flow, tack and cure characteristics when stored under specified conditions. Most prepregs require refrigerated storage in order to achieve useful shelf lives. A consequence of this is the desirability of refrigerated transit from manufacturer to user. Removal from refrigerated store during normal manufacturing operations should be noted on the prepreg batch record, and appropriate adjustments to the remaining shelf life made. It is sometimes possible to re-life a time-expired prepreg batch, but the technical requirements vary from prepreg to prepreg, and the procedure should be approached with caution.

A.1.19 Shop or working life

The shop life of a prepreg is that period following removal from the specified storage conditions and attaining shop-floor temperature for which the prepreg remains workable in terms of tack, and flow and cure characteristics. The shop life may be a few days or a few weeks, depending on the material.

A.1.20 Volatile content

The volatile content of a prepreg is a measure of the mass loss from a sample subjected to prescribed test conditions. The volatile loss is an indication of the solvent content of the prepreg remaining from either the resin compounding process or a solution prepregging operation.

Prepregs with a high volatile content tend to produce heavily voided laminates, owing to the vaporisation of the usually low-boiling-point solvent within the resin constituent during cure. High volatile content can also affect the resin flow characteristics.

A.1.21 Outgassing and offgassing

Depending on the application, there may be restrictions on the gaseous products released from the finished component in operational vacuum conditions, which could contaminate other equipment (outgassing), or on the allowable gaseous contamination of the air during preparatory or operational conditions for manned spacecraft (offgassing).

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APPENDIX 2
COLLATED EUROPEAN AND NATIONAL TEST METHODS APPLICABLE TO PREPREG PROCUREMENT SPECIFICATIONS

A.2.1 AMERICAN (ASTM) STANDARDS FOR HIGH-MODULUS FIBERS AND COMPOSITES

A.2.1.1 ASTM C613-67 (1985)
Test method for resin content of carbon and graphite prepregs by solvent extraction.

A.2.1.2 ASTM D2344-84
Test method for apparent interlaminar shear strength of parallel fiber composites by short-beam method.

A.2.1.3 ASTM D3039-76 (1982)
Test method for tensile properties of fiber-resin composites.

A.2.1.4 ASTM D3355-74 (1980)
Test method for fiber content of unidirectional fiber-resin composites by electrical resistivity.

A.2.1.5 ASTM D34IO-87
Test method for compressive properties of unidirectional or crossply fiber-resin composites.

A.2.1.6 ASTM D3518-76 (1982)
Practice for in-plane shear stress-strain response of unidirectional-reinforced plastics.

A.2.1.7 ASTM D3529-76 (1982)
Test method for resin solids content of carbon-fiber/epoxy prepreg.

A.2.1.8 ASTM D3530-76 (1982)
Test method for volatiles content of carbon-fiber/epoxy prepregs.

A.2.1.9 ASTM D3531-76 (1982)
Test method for resin flow of carbon-fiber/epoxy prepreg.

A.2.1.10 ASTM D3532-76 (1982)
Test method for gel time of carbon-fiber/epoxy prepreg.

FIBRES

A.2.1.11 ASTM D3800-79 (1985)
Test method for the density of high-modulus fibers.

A.2.1.12 ASTM D3379-75 (1982)
Test method for tensile strength and Young's modulus for high-modulus single-filament materials.

GENERAL

A.2.1.13 NASA TS NHB-8060.1A
Test methods for outgassing and flammability.

A.2.2 EUROPEAN STANDARDS

A.2.2.1 EN 61 (77)
Glass-fibre-reinforced plastics: determination of tensile properties.

A.2.2.2 EN 62 (77)
Glass-fibre-reinforced plastics: standard atmospheres for conditioning and testing.

A.2.2.3 EN 63 (77)
Glass-fibre-reinforced plastics: determination of flexural properties, three-point method.

A.2.2.4 ESA PSS-01-702
A screening test method employing a thermal vacuum for the selection of materials to be used in space.

A.2.2.5 ESA PSS-01-721
Flammability testing.

A.2.2.6 ISO 1183 (70)
Plastic method for determining the density and relative density (specific gravity) of plastics excluding cellular plastics.

Additionally, the following standards are available from AECMA (Association Européenne des Constructeurs de Matériel Aérospatial). The status of individual standards is indicated. (a = issued; b = draft as at mid 1990.)

A.2.2.7 prEN 2000
Quality assurance requirements for manufacture and procurement of EN-aerospace standard products. (a)

A.2.2.8 prEN 2310
Requirements and methods for testing inflammability for the qualification of non-metallic materials. (b)

A.2.2.9 prEN 2329
Test method for the determination of mass per unit area of woven-textile glass-fibre preimpregnate. (a)

A.2.2.10 prEN 2330
Test method for the determination of the percentage of volatile matter in woven-textile glass-fibre fabric preimpregnate. (a)

A.2.2.11 prEN 2331
Test methods for the determination of the resin content of woven-textile glass-fibre fabric preimpregnate. (a)

A.2.2.12 prEN 2332
Test method for the determination of the resin flow of woven-textile glass-fibre fabric preimpregnate. (a)

A.2.2.13 prEN 2374
Production of test panels for glass-fibre-reinforced mouldings and sandwich constructions. (a)

A.2.2.14 prEN 2375
Sampling procedures for reinforced plastics made from resin-preimpregnated materials. (a)

A.2.2.15 prEN 2377
Test method for the determination of interlaminar shear properties for glass-fibre-reinforced plastics. (a)

A.2.2.16 prEN 2378
Fibre-reinforced plastics: determination of water absorption by immersion in demineralised water. (a)

A.2.2.17 prEN 2489
Fibre-reinforced plastics: determination of the effects of liquid chemicals. (a)

A.2.2.18 prEN 2557
Test method for the determination of mass per unit area of a carbon-fibre preimpregnate. (a)

A.2.2.19 prEN 2558
Test method for the determination of percentage of volatile matter in a carbon-fibre preimpregnate. (a)

A.2.2.20 prEN 2559
Test method of the determination of resin content of a carbon-fibre preimpregnate. (a)

A.2.2.21 prEN 2560
Test method for the determination of resin flow of a carbon-fibre preimpregnate. (a)

A.2.2.22 prEN 2561
Test method for the determination of longitudinal tensile properties of unidirectional carbon-fibre-reinforced epoxy-resin composites. (a)

A.2.2.23 prEN 2562
Test method for the determination of flexural properties of unidirectional carbon-fibre-reinforced epoxy-resin composites. (a)

A.2.2.24 prEN 2563
Test method for the determination of interlaminar shear strength of unidirectional carbon-fibre-reinforced epoxy-resin composites. (a)

A.2.2.25 prEN 2564
Test method for the determination of fibre content of a carbon-fibre-reinforced resin laminate. (a)

A.2.2.26 prEN 2565
Preparation of unidirectional carbon-fibre-reinforced resin panels for test purposes.(a)

A.2.2.27 prEN 2597
Test methods for the determination of transverse tensile properties of unidirectional carbon-fibre-reinforced epoxy-resin composites. (a)

A.2.2.28 prEN 2746
Fibre-reinforced plastics: determination of flexural properties by three-point bend method. (a)

A.2.2.29 prEN 2747
Glass fibre reinforced plastics: determination of tensile properties – Three-point bend method. (b)

A.2.2.30 prEN 2833
Spécification technique pour l'approvisionnement des préimprégnés en fibre de verre (b).

A.2.2.31 prEN 2850
Test method for the determination of longitudinal compressive properties of unidirectional carbon-fibre-reinforced epoxy-resin composites (b).

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