

CHAPTER 5 PREPREG MATERIALS CHARACTERIZATION

5.1 INTRODUCTION

The processability and properties of high performance composites depend upon the composition of the fiber/resin preimpregnated materials (prepregs) from which they are manufactured. In general, prepregs consist of "modified" or surface-treated glass, graphite, or aramid fibers impregnated with 28-60 weight-percent of a reactive and chemically-complex thermoset resin formulation or a thermoplastic resin. A typical thermoset resin formulation may contain, for example, several different types of epoxy resins, curing agents, diluents, rubber modifiers, thermoplastic additives, accelerators or catalysts, residual solvents, and inorganic materials, plus various impurities and synthetic by-products. Furthermore, such resins are often "staged" or partially reacted during the prepregging process and may undergo compositional changes during transport, handling, and storage. Although less likely to undergo compositional changes, polymer molecular weight (MW), molecular weight distribution (MWD), and crystalline morphology have major effects on the processability and properties of thermoplastic prepregs and composites. Inadvertent or minor changes in resin composition may cause problems in processing and have deleterious effects on the performance and long-term properties of composites.

Modern analytical techniques and detailed knowledge relating to fibers, fiber surface treatments, and resin types and formulations are needed to characterize prepregs and composite materials. Characterization involves the identification and quantification of the fiber, fiber surface, and major resin components and should include information about the presence of impurities or contaminants. For thermoset resins and composites, characterization should include a description of the nature and extent of the prepreg resin reaction and the thermal/rheological and thermal/mechanical behavior. In the case of thermoplastics, the polymer molecular weight distribution, crystallinity, and time/temperature viscosity profile should also be analyzed. However, few laboratories are equipped or have the knowledgeable technical personnel to characterize prepregs and composites completely, and few studies have been published describing how variations in fiber type and resin chemistry/morphology affect the physical properties and long-term performance of composites. Also, until recently, prepreg compositions were considered proprietary, processing conditions were only recommended, and acceptance was based primarily upon mechanical testing of fabricated specimens. The purpose of this chapter is to provide an overview of characterization techniques and, more specifically, to address the application of state-of-the-art techniques for the chemical and physical characterization of resins and prepreg materials used in the manufacture of high performance organic matrix composites.

5.2 CHARACTERIZATION TECHNIQUES - OVERVIEW

According to a recent survey (Reference 5.2(a)), the most widely utilized techniques for the characterization and quality assurance of composite material precursors are -

1. High Performance Liquid Chromatography (HPLC)
2. Infrared (IR) Spectroscopy
3. Thermal Analysis
4. Rheological Analysis

HPLC and IR spectroscopy provide the capability for rapid screening and quality control fingerprinting of individual resin constituents as well as of the prepreg resin and, therefore, may be used advantageously by both the prepregger and composite manufacturer (References 5.2(b) - (f)). Thermal analytical techniques, such as thermal gravimetric analysis (TGA), differential thermal analysis (DTA), differential scanning calorimetry (DSC), thermal mechanical analysis (TMA), dynamic mechanical analysis (DMA), and torsional braid analysis (TBA) are not strictly chemical analysis techniques; however, they provide useful information relating to the composition and processability of resins (Reference 5.2(g)). Similarly, rheological and dielectric techniques are used frequently to evaluate the chemoviscosity properties of

thermoset resins during cure (Reference 5.2(h)), and there is increasing interest in applying such techniques for process monitoring and process control of both thermoset and thermoplastic resins.

Many of the chemical and physical analytical techniques described in Chapter 4 are also applicable to the characterization of prepreg materials. HPLC, IR spectroscopy, thermal analytical techniques and rheological methods are described in Section 5.5.

5.3 SAMPLING

Prepregs are commonly described by the purchaser's requirements which may include the manufacturer's trade name, resin type (e.g., 250°F) and lot number, fiber type and form (tape, fabric, roving, etc.), prepreg lot and roll numbers, and date of manufacture. The shipping date and expected shelf life are also usually designated along with recommended processing conditions. Generally, prepregs are shipped as rolls of impregnated woven fabric or unidirectional tape. (Typical widths are 38" (~97 cm) for woven aramid and glass, 42" (~107 cm) for woven graphite, and 12" (~30 cm) for tapes.) The prepreg layers are separated by thin, removable plastic or coated paper films which are removed when the prepregs are analyzed.

To check uniformity, it is recommended that sections be cut from the center and each side of the front-end (first off) of designated prepreg rolls. The amount and number of samples required for a particular analysis or test depends upon the homogeneity of the resin and the uniformity of the prepreg fibers. For some techniques, such as HPLC, 0.5 to 2.0 grams of prepreg may be needed to provide a representative sample. Other techniques (e.g., DSC) which utilize relatively small specimens (10 to 20 milligrams) may demand multiple specimens to provide an "average" value or test result.

Care must be taken not to contaminate or in any way alter samples during handling and storage. The samples should be placed in clean, dry, sealable containers and be carefully labeled. The containers must not react with the samples and precautions must be taken not to expose the prepregs to moisture nor allow them to stand unrefrigerated for long periods of time. For reactive prepreg resins such as epoxies, it is recommended that the prepregs be stored in hermetically sealed packaging at -0°F (-18°C). Upon removing the containers from cold storage, they should be allowed to achieve room temperature before opening to prevent condensation of moisture on the samples.

5.4 PHYSICAL CHARACTERISTICS AND PROPERTY TESTS

5.4.1 Physical description of reinforcement

The physical description of the reinforcement used in a composite should be described using the standard definitions of ASTM D 3878 (Reference 5.4.1). The filaments in the prepreg should be uniformly wetted by the resin. No particulates should be observable upon visual examination.

5.4.1.1 Alignment

In unidirectional prepregs, the filament bundles must be parallel to the longitudinal direction of the prepreg within an angle of 0.5° when examined visually using appropriate aids to measure angular alignment.

5.4.1.2 Gaps

Any gap within or between filament bundles in unidirectional prepregs generally should comply with the specifications. General guidelines are provided as follows:

- a. No gap should exceed 0.030 inch (0.76 mm) in width.

- b. The length of any portion of the gap with an average width of 0.030 inch (0.76) should not exceed 24 inches (0.61 m).
- c. Gaps in line with each other and no more than one inch (25 mm) apart should be considered as one gap, regardless of number.
- d. Gaps with excessive width or length should be considered defective and will be the basis for flagging or replacing the prepreg.

5.4.1.3 *Width*

Width tolerance for unidirectional prepreg tape should be as specified, typically ± 0.030 inch (0.76 mm).

5.4.1.4 *Length*

The length of each roll of prepreg should be provided. Limits on the length of prepreg on any single roll should be specified. Alternatively, suppliers and users may agree on limits for the prepreg weight or area per roll.

5.4.1.5 *Edges*

Acceptable waviness of edges should be specified. A typical acceptance value for any 12-inch (30-cm) length of tape should be 0.030 inch (0.76 mm) from the edge when measured with an appropriate straight edge.

5.4.1.6 *Splices*

Prepreg splices may be permitted on any roll of tape where processing is continuous without change in fiber or resin batch. Such splices should be appropriately marked as a nonconforming area.

5.4.2 **Resin content**

The resin content of prepregs may be determined by extracting the resin from the prepreg fibers using a solvent in which the resin material is fully soluble and the fibers are not dissolved. Soxhlet extraction procedures are described in ASTM C 613 (Reference 5.4.2(a)). Procedures for determining the resin content of carbon fiber-epoxy prepregs are provided in ASTM D 3529 (Reference 5.4.2(b)). Special procedures and solvents may be required to extract high molecular weight or thermoplastic resins. An alternate procedure for determining the resin content in epoxy resin prepregs is outlined in Section 5.5.1.

5.4.3 **Fiber content**

Procedures used to determine resin content often provide information about the fiber content of prepregs. Alternatively, acid digestion methods (ASTM D 3171) may be applied to remove the matrix resin from the fibers as long as the fibers are not degraded (Reference 5.4.3). Digestion methods are not preferred for graphite and aramid fiber prepregs since such fibers are subject to oxidative degradation. Section 5.5.1 describes a procedure for determining the fiber and resin contents of glass, carbon, and aramid fiber/epoxy resin prepregs.

5.4.4 **Volatiles content**

The volatiles content of prepregs may be determined by ASTM D 3530 (Reference 5.4.4). Thermogravimetric analysis (TGA) may also be applied to estimate weight percent volatiles in a prepreg.

5.4.5 Moisture content

The moisture content of prepregs may be determined by coulometry in accordance with ASTM D 4019 (Reference 5.4.5) or by automated moisture meters based on the Karl Fischer titration method.

5.4.6 Inorganic fillers and additives content

The quantitative determination of inorganic fillers and additives in the prepreg resin requires considerable care. For example, the weight percent inorganic fillers and additives in a prepreg resin may be determined according to the procedure described in Section 5.5.1. Assuming that the organic resin material is fully soluble in tetrahydrofuran (THF) and that the inorganic fillers and additives are insoluble, the solution prepared in step 6 can be centrifuged to precipitate the insoluble components. The precipitate is washed at least three times with the solvent, dried, and then weighed.

5.4.7 Areal weight

The areal weight (mass per unit area) of a prepreg material may be determined using ASTM D 3776 (Reference 5.4.7).

5.4.8 Tack and drape

Tack refers to the ability of a prepreg to adhere to itself or to other material surfaces and is a key factor in determining prepreg suitability for component/part fabrication. There is no quantitative method for measuring tack. Subjective terms such as high, medium, and, low are often used in describing tack. Although there is no generally accepted method for measuring tack, some composite manufacturer's use a Monsanto Tack Tester™ to obtain a relative index for prepreg tack. Drape is also a subjective term which relates to the ease of handling and conforming prepregs to complex surfaces.

5.4.9 Resin flow

Resin flow under specified test conditions relates to the composition, extent of reaction, and/or morphology of the prepreg resin, as well as the resin content. Prepreg processability and resin content in the processed laminate are affected by resin flow. Test conditions (temperature, pressure, layers of prepreg, number of bleeder plies) depend upon the type of resin. The resin flow of prepreg materials may be determined by ASTM D 3531 (Reference 5.4.9).

5.4.10 Gel time

Gel time relates to the chemical composition and extent of reaction of thermosetting prepreg resins. Prepreg processability is affected by the resin gel time. The test temperature depends upon the type of resin. The gel times of prepregs may be determined by ASTM D 3532 (Reference 5.4.10).

5.5 TEST METHODS

The following methods are examples of analytical techniques that can be used for prepreg characterization.

5.5.1 Resin extraction procedure for epoxy resin prepregs

This procedure is applicable for determining the fiber and resin contents of glass, carbon, and aramid fiber/epoxy resin prepregs. Solutions prepared according to this procedure can be used directly for HPLC analysis provided appropriate grade solvents are used. Recommended sampling, specimen handling procedures, and standard laboratory safety procedures should be followed.

1. Cut a rectangular specimen (approx. 1 g) from prepreg section and weigh on analytical balance (± 0.001 g or better). Record weight as W_o (grams).
2. Place specimen in 25 mL Erlenmeyer flask (fitted with a ground-glass stopper) and add about 20 mL THF (tetrahydrofuran, fresh, HPLC grade, distilled-in-glass, with no inhibitor added).
3. Stopper the flask and allow the specimen to soak in the THF for at least 4 hours.
4. Place flask on vortex mixer and agitate for 1 minute.
5. Carefully decant the THF solution into a 50 mL volumetric flask. The fibers should remain bunched together in the 25 mL flask.
6. Add about 10 mL THF to rinse the fibers in the 25 mL flask, mix on the vortex mixer, and decant the THF into the 50 mL volumetric flask containing the primary solution (step 5).
7. Repeat step 6.
8. Add THF to fill the volumetric flask to the 50 mL mark.
9. Carefully remove the graphite fibers from the 25 mL Erlenmeyer flask (using forceps), wrap fibers in Kimwipes™ or equivalent, place in labeled paper envelope, place the envelope in fume hood air stream, and allow fibers to dry overnight. Alternatively, residual THF may be removed by placing the envelope with fibers in a vacuum oven (fitted an appropriate vapor trap) set at 40°C and maintaining a vacuum for at least 1 hour.
10. The fibers are removed from the Kimwipes™ and weighed on an analytical balance. Record the fiber weight as W_f (grams).
11. Calculate the concentration of the resin solution (see step 8) and record concentration as C_o ($\mu\text{g}/\mu\text{L}$). This concentration will be useful in the analysis of HPLC data.

$$C_o = \frac{(W_o - W_f)}{0.050} \mu\text{g}/\mu\text{L} \quad 5.5.1(a)$$

12. Mix resin solution (from step 11) on vortex mixer and immediately filter about 4 mL of the resin sample solution through a 0.2 μm Teflon™ membrane filter into a dry, clean glass vial. Immediately cap the vial to prevent contamination and solvent loss. This solution will be used for HPLC analysis. The remaining (unfiltered) solution in the flask can be used to determine soluble resin content and insoluble content (steps 16 and 17).
13. The extractable resin content and fiber content, not corrected for the presence of volatiles and insoluble components in the prepreg resin and remaining on the fibers, are calculated -

$$\text{wt- \% extractable resin} = 100\% \times \frac{(W_o - W_f)}{W_o} \quad 5.5.1(b)$$

$$\text{wt- \% fiber} = 100\% - \text{wt \% extractable resin} \quad 5.5.1(c)$$

14. Glass fibers may be placed in a muffle furnace and heated at 650 to 800°C to remove nonextractable surface material. After cooling to room temperature, the fibers are reweighed and their weights are recorded as W_f .

15. The amount of nonextractable fiber surface material in glass fiber prepreps is calculated -

$$\text{wt- \% nonextractables} = 100\% \times \frac{(W_f - W_f')}{W_o} \quad 5.5.1(d)$$

16. Insoluble content. The extractable or THF-soluble resin content may also be determined by filtering the solution prepared in step 8 through a 0.2 μm Teflon™ membrane filter. Using a volumetric pipet,

an aliquot (e.g., 10 mL) of the filtered solution is transferred to a pre-weighed aluminum pan (weight W_A) which is then placed into a fume hood to evaporate the solvent. A stream of filtered air or nitrogen can be directed over the surface of the pan to accelerate evaporation. After 9 mL or more of the solvent is removed leaving an oily residue of resin, the pan can be placed in a vacuum oven and heated at about 50°C for several hours to remove residual solvent. After cooling to room temperature, the pan is reweighed (W'_A) and the resin content is calculated -

$$\text{wt- \% soluble resin} = 100\% \times (W'_A - W_A) \times \frac{5}{W_o} \quad 5.5.1(e)$$

Differences in the weight-percent resin determined using Equations 5.6.1(b) and 5.6.1(e) may be attributed to the presence of volatiles and insoluble (nonfibrous) components in the prepreg.

17. Insoluble content. The amount of insoluble components may be determined by the following procedure. An aliquot of the solution from step 12 can be centrifuged to precipitate the insoluble components. The precipitate is washed at least three times with the solvent, dried, and weighed.

5.5.2 Procedure for HPLC/HPSEC analysis of glass, aramid, and graphite fiber prepreps

Mix resin solution (prepared in Section 5.5.1, step 12) on a vortex mixer and immediately filter about 4 mL of the resin sample solution through a 0.2 μm Teflon™ membrane filter into a dry, clean glass vial.

Immediately cap the vial to prevent contamination and solvent loss. The sample is now ready for HPLC analysis.

If the HPLC analysis is not run immediately, the sample solution should be kept in a cool, dark location. If care is taken during storage, the THF solution will remain stable and may be analyzed weeks after its preparation with no apparent effect on the HPLC analysis.

5.5.2.1 Reverse phase HPLC analysis

The epoxy resin prepreg analysis can be run using any of a number of commercially available HPLC instruments. An integrator/recorder or state-of-the-art HPLC data analysis system is recommended for data acquisition, plotting, and reporting. HPLC operating conditions were selected for simplicity and compatibility with most commercial HPLC equipment.

HPLC System: Waters Associates model-244 instrument with M6000A solvent delivery systems, M720 system controller, 710B WISP auto-injection system, M440 UV detector, and M730 data module. Similar systems available from other manufacturers may also be used.

Solvents: Acetonitrile (distilled-in-glass) and reagent grade water prepared from distilled water using a Millipore Milli-Q2 (Millipore Corp., Bedford, MA) or equivalent water purification system. Purging the solvents with helium is recommended.

Column: Waters Associates $\mu\text{Bondapak C18}$. (Similar columns available from other manufacturers may also be used).

Injection Volume: 10 mL

Flow Rate: 2.0 mL/min

Mobile Phase (solvent program):

Time	% Acetonitrile	% Water	Curve
0	45	55	*
12 min	100	0	7
16 min	100	0	*
20 min	45	55	6

Detector: UV 254nm Run Time: 20 minutes

5.5.2.2 Size Exclusion Chromatography (SEC) analysis

The SEC analysis of the prepreg resins can be run using HPLC instrumentation as described above.

Solvent: THF (distilled-in-glass) A helium purge should be maintained on THF for optimum results.

Columns: IBM SEC type A and type C, 5 micron (columns from other manufacturers, such as the Waters μ Styragel 1000, 500, 100, 100 \rightarrow , are also acceptable).

Injection Volume: 10 μ L

Flow Rate: 1 mL/min

Detector: UV 254nm

Run Time: 15 minutes

Calculations: Integrated peak areas are converted to area percentages (% area).

5.5.3 Procedure for Fourier transform infrared spectroscopy (FTIR)

Several droplets of the resin/THF solution (prepared in Section 5.5.1, step 12) are placed on the surface of a polished salt plate (preferably KBr). The sample is analyzed as soon as the THF has evaporated. A Perkin Elmer model 1550 or 1700 FTIR spectrometer with model 7500 computer or an equivalent instrument is used to scan and record the spectrum (500 to 4000 cm^{-1}) of the salt plate with and without the sample on its surface. The analysis should be conducted with the salt plate and sample in a purged, dry nitrogen atmosphere at room temperature. Depending upon the sample, 100 to 200 scans of the spectrum may be required to optimize spectral resolution. It also may be necessary to deposit more or less sample on the salt plate. The spectrum of the sample obtained by subtracting that of the salt plate is plotted, reported, and stored on a computer disk.

5.5.4 Procedure for differential scanning calorimetry (DSC)

This test can be performed using a DuPont Instruments 9900 Thermal Analyzer/Controller and model 912 DSC accessory or an equivalent instrument.

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Specimen:	Prepreg (10 to 30 mg) in an aluminum sample pan
Reference:	Empty sample pan
Heating Rate:	10°C/min
Temperature Range:	Room temperature to 350°C
Atmosphere:	Dry nitrogen gas purge
Data Handling:	Data is stored on a computer disk and a plot of heat flow dH/dt ($\mu\text{W}/\text{sec}$) vs temperature ($^{\circ}\text{C}$) is produced.
Heat of Reaction:	The calibration routine and integration program provided with the thermal analyzer is used to calculate heats of reaction ΔH of thermoset prepreg resins.
Glass Transition:	A cooling device attached to the DSC cell may be needed to facilitate glass transition temperature T_g measurements of thermoset prepreg resins; i.e., it is often necessary to initiate temperature scans at -50°C or lower since such resins typically have T_g values below room temperature. The thermal analyzer may have a software routine to assist in determining T_g values.

5.5.5 Procedure for dynamic mechanical analysis (DMA)

A single ply of prepreg is cut into a 1.1 cm x 1.7 cm strip and the strip is mounted in a DuPont model 982 or 983 DMA accessory. A DuPont 9900 or 1090 controller is used to run the test and plot the results. Equivalent instruments may also be used.

Heating Rate:	5°C/min
Temperature Range:	Room temperature to 350°C
Atmosphere:	Dry, nitrogen gas purge
Data Handling:	Data is stored on a computer disk and a plot of storage modulus and $\tan \delta$ is plotted as a function of temperature.
Glass Transition:	The temperature of the damping peak maximum is assigned as the T_g value.
Gelation:	Gelation occurs when the Young's modulus starts increasing rapidly (several orders of magnitude) over a narrow temperature range. Gelation temperature depends upon heating rate and mechanical frequency. Therefore, both heating rate and frequency should be included when DMA gelation temperatures are reported.
Gelation Time:	In the isothermal mode, the time to gelation is determined by rapidly heating a sample to the desired temperature, holding the temperature constant and monitoring the change in Young's modulus with time. Gelation time is defined as the time it takes for the modulus to start rapidly increasing (several orders of magnitude).

5.5.6 Procedure for rheological characterization

A Rheometrics Dynamic Spectrometer (RDS) or equivalent system is used for this test. Samples are prepared by cutting three 25-mm diameter circles from a single ply of prepreg. The three plies are stacked and placed between the rheometer's parallel plates.

Heating Rate:	2°C/min
Temperature Range:	Room temperature to the onset of gelation (for thermosets)
Atmosphere:	Air or a blanket of nitrogen gas
Geometry:	25-mm diameter parallel plate
Gap:	Typically 0.8 mm, but may be adjusted according to sample characteristics.
Data Reporting:	Shear moduli (storage and loss) and complex viscosity are plotted as a function of temperature.

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