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**ATOMIC ENERGY
OF CANADA LIMITED**



**L'ENERGIE ATOMIQUE
DU CANADA, LIMITEE**

RADIATION-CURABLE PREPREG COMPOSITES

COMPLEXES D'IMPREGNES TRAITABLES PAR IRRADIATION

**Chris B. Saunders, Lawrence W. Dickson, Ajit Singh,
Alistair A. Carmichael, Vince J. Lopata**

**Whiteshell Nuclear Research
Establishment**

**Etablissement de recherches
nucléaires de Whiteshell**

Pinawa, Manitoba R0E 1L0

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RÉSUMÉ

Une application possible du traitement au faisceau d'électrons dans la fabrication des complexes est le traitement des imprégnés en fibres de carbone. Ces imprégnés traitables thermiquement, fibres de carbone ou tissus préimprégnés de résine polymère liquide, servent couramment dans l'industrie aéronautique. On a conçu un préimprégné traitable par irradiation pour répondre aux spécifications de propriétés mécaniques et physiques d'une grande société de construction aéronautique. Les études de caractérisation ont montré que la fraction de gel maximale du polymère traité s'est manifestée à une dose d'environ 50 kGy et a varié de 91 à 97% selon le type d'atmosphère (air ou azote) et la pression (100 à 1000 kPa) pendant l'irradiation. Seuls les groupes d'acrylates de la résine ont contribué pour une large part aux réactions de traitement. Le polymère a été amorphe, le point (température) de ramollissement étant d'environ 235°C et le coefficient de dilatation thermique linéaire de $1,3 \times 10^{-4}$ m/(m°C) entre 25 et 150°C (30 à 50 kGy). En outre, on a commencé les essais mécaniques et physiques préliminaires des complexes d'imprégnés.

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ABSTRACT

A potential application of electron-beam processing in composite manufacturing is curing carbon-fibre prepregs. These thermally curable prepregs, carbon fibres or fabrics preimpregnated with liquid polymer resin, are commonly used in the aircraft industry. A radiation-curable prepreg has been designed to meet the mechanical- and physical-property specifications of a leading aircraft manufacturing company. Characterization studies showed that the maximum gel fraction in the cured polymer occurred at a dose of about 50 kGy and varied from 91 to 97%, depending on the type of atmosphere (air or nitrogen) and the pressure (100 to 1000 kPa) during irradiation. Only the acrylate groups of the resin took a significant part in the curing reactions. Some carbon dioxide was produced during radiation curing. The polymer was amorphous with a softening point of about 235°C and a linear thermal expansion coefficient of $1.3 \times 10^{-4} \text{ m}/(\text{m} \cdot ^\circ\text{C})$ between 25 and 150°C (30 to 50 kGy). Preliminary mechanical and physical testing of the prepreg composites has also been started.

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CONTENTS

	<u>Page</u>
1. INTRODUCTION	1
2. MATERIALS AND METHODS	3
3. RESULTS AND DISCUSSION	4
3.1 FT-I SPECTROSCOPY	4
3.2 GELATION	5
3.3 THERMAL CHARACTERISTICS	5
3.4 VISCOSITY AND DENSITY	6
3.5 LAMINATE IRRADIATIONS AND MECHANICAL TESTING	6
4. CONCLUSIONS	7
ACKNOWLEDGEMENTS	7
REFERENCES	7
TABLES	10
FIGURES	12

1. INTRODUCTION

Fibre-reinforced plastic composites are being used for many industrial applications, both nonstructural and load bearing. The industries that use these composites extensively include the aircraft and aerospace industry, the electrical and electronics industry, various process industries for corrosion-resistant tanks and pressure vessels, the construction industry, and the commercial products and sporting equipment industries. The use of composites in the automotive industry, presently at a low level, is also expected to increase (1).

At present, radiation processing is not used extensively in the commercial fibre-reinforced composite manufacturing industry. Nevertheless, electron-beam (EB) processing does seem to have potential applications for several reasons. These reasons include the following:

1. The continuing search for new matrix-fibre systems that can be easily processed and have the required mechanical and chemical properties. Thermoplastic composites are easily processed, but they often do not have the high-temperature properties of thermosets required for many aircraft, aerospace and automotive applications. Radiation processing can convert a thermoplastic resin to a thermoset polymer. The processing ease of thermoplastics can be exploited while still obtaining the high-temperature properties of thermosets.
2. The importance of automation of processes using fibre-reinforced composites to increase their use in high-volume consumer-oriented industries such as the automotive industry. Production speed is of primary importance and extensive cure cycles limit production. EB processing should eliminate long cure cycles for some free radical-initiated resin systems, which would significantly increase production speeds.
3. Radiation processing can be easily retrofitted to many types of process lines, such as pultruders, pulformers and extruders. Radiation processing can also be an off-line process to treat selected products as required from a large processing facility, providing production flexibility.
4. The advantages that have been identified over the past 25 years in the radiation-processing industry for the production of plastics, include improved rate control, reduced curing times and curing at ambient temperature without the need for chemical initiators and solvents.

One potential application for electron-beam processing of composites is for curing carbon-fibre prepregs. These prepregs, carbon-fibres or fabrics preimpregnated with liquid resin, are used in the aircraft industry for hand lay-up application, producing a high-strength, low-weight construction material. Epoxy resins are commonly used to manufacture these prepregs, but the curing cycle is both time consuming and expensive.

Previous reviews of the radiation-induced polymerization of epoxy compounds have been written by Williams (2), Omel'chenko and Bokalo (3) and Laricheva (4). Williams (2) presented a short discussion of the research on the radiation polymerization of cyclohexene oxide, ethylene oxide and propylene oxide. Omel'chenko and Bokalo (3) noted that radiation homopolymerization of epoxy compounds does not occur readily, but copolymerization occurs more easily. Chemical curing of epoxies may proceed by either cationic or anionic mechanisms depending on the nature of the curing agent, but most epoxies polymerize by cationic mechanisms under the influence of high-energy radiation. Radiation-induced cationic polymerization of epoxy compounds is inhibited by trace quantities of water because of proton transfer from the chain-propagating epoxy cation to water and, thus, most epoxies are not currently good candidates for industrial-scale radiation polymerization.

One method for imparting good radiation curing properties to an epoxy resin is to acrylate the terminal epoxy groups of an epoxy oligomer. Gotoda and co-workers (5,6) and Gotoda, Miyashita and Takeyama (7) studied the radiation-induced polymerization of epoxy-acrylate prepolymers in the presence and absence of several vinyl monomers; they found that an acrylic acid-based epoxy acrylate cured at a dose of ≤ 10 kGy. Kumanotani and co-workers (8) determined that electron-beam curing (dose rate = $7 \text{ kGy}\cdot\text{s}^{-1}$) of epoxy acrylates gave a larger amount of cross-linking than ultraviolet (UV) curing (dose rate = $0.2 \text{ kGy}\cdot\text{s}^{-1}$). Pyun and co-workers (9) studied the use of electron beams for curing of a methacrylic ester of a low molecular-weight diglycidyl ether epoxy resin. Rot, Shiryaeva and Chernyakov (10) investigated the use of a radiation-hardened epoxy acrylate as a coating for wood. Omel'chenko and co-workers (11) showed that an acrylic acid modified epoxy oligomer cured by irradiation in the presence of a sensitizer to a dose of 200 kGy had a gel fraction of 97.2%. Videnina and Bratslavskaya (12) determined that epoxy acrylate mixtures containing 20 to 50% aliphatic epoxy acrylates (remainder aromatic epoxy acrylates) radiation cured with electron beams (dose rate = $8 \text{ kGy}\cdot\text{s}^{-1}$) gave the best physical and mechanical properties. Yakovleva and co-workers (13) studied the electron-beam curing of several resins, including an epoxy acrylate. Thalacker and Boettcher (14) determined that the thermal stability of electron-beam-cured acrylated epoxies increases with the number of acrylated epoxide groups present in the parent compound. Thompson, Song and Wilkes (15) found that electron-beam curing of the diglycidyl methacrylate of bisphenol-A to a dose of 100 kGy gives a polymer with a gel fraction of about 98%. Additional discussion of radiation curing of epoxies may be found in Dickson and Singh (16).

The objective of this experimental program is to design and manufacture a radiation-curable, polymer-carbon fibre prepreg that meets the mechanical- and physical-property specifications set by a leading manufacturer of aircraft components. The program is divided into two phases: designing the prepregs and characterizing the selected resin system, and mechanical and physical testing of the radiation-cured material. This paper describes our current work involving characterization of the selected resin system and preliminary mechanical testing of the prepregs; it also outlines our future testing program.

2. MATERIALS AND METHODS

A resin system recommended by the resin supplier, combining an epoxy diacrylate oligomer (50% by mass), a polybutadiene diacrylate oligomer (30%) and the multifunctional monomer dipentaerythritol monohydroxypentaacrylate (20%), was selected for preparing our preregs. Table 1 lists the relative product characteristics for these compounds (17). The polybutadiene diacrylate was included in the resin formulation to improve the flexibility and impact resistance of the epoxy diacrylate polymer. Dipentaerythritol monohydroxy pentaacrylate was added to improve the weatherability and the flexibility of the formulation.

A plain weave carbon fabric, produced from polyacrylonitrile precursor (PAN) using a thermal decomposition process, was selected from the prepreg aircraft manufacturer's qualified products list. Table 2 lists the product specifications for the carbon fabric. The mechanical properties of carbon fibres are not deleteriously affected by irradiation when irradiated in a vacuum to a dose of as much as 50 MGy (18). This excellent radiation stability makes these fibres well suited for radiation-curable preregs.

The preregs were prepared using a standard bead hot-melt process (19). The selected resin content in the composite was $35 \pm 2\%$.

A Gammacell-220 (dose rate = 17 kGy/h) was used to irradiate all samples in either air or nitrogen atmospheres at ambient temperature over a pressure range of 100 to 1000 kPa. The dose range was 0 to 300 kGy.

The first phase of the experimental program, the polymer characterization study included:

1. Fourier-transform infrared (FT-IR) spectroscopy to study the resin structure and the radiation-curing mechanism. A hydrogen purged photoacoustic cell was used with a Bomen IMH4700L IR spectrometer to analyze 3-mm-diameter, 2-mm-long samples irradiated in air from 1 to 300 kGy. In photoacoustic spectroscopy (PAS) the sample is placed in the cell with a very sensitive microphone and excited with monochromatic energy in the UV to IR range. In the relaxation process, the energy is released in the form of heat, expanding the air in the cell. This expansion is recorded by the microphone. By changing the frequency of light, a series of spectra are obtained that give information about the sample as a function of depth. PAS is often used to study the chemistry of polymer coatings.
2. Linear thermal expansion of cured samples (dose = 30 to 50 kGy) using a Theta apparatus and a Lindberg furnace.
3. X-ray diffraction (XRD) analysis using a Rigaku Rotoflex D/Max system to examine the crystallinity of the cured polymer (dose = 30 to 50 kGy). The minimum cooling rate was 1°C/min from a 300°C maximum sample temperature.

4. Examining the effects of irradiation (dose ≤ 50 kGy), atmosphere (air or nitrogen) and pressure (100 to 1000 kPa) on the gel fraction of the cured polymer. Standard soxhlet extraction techniques, using chloroform as the solvent, were used for these experiments.
5. Determination of the viscosity of the selected resin formulation as a function of temperature. The resin viscosity is an important variable for prepreg manufacturing, affecting the flow-out properties and the resin wetting characteristics in the prepregging process. A Haake RV3 rotary viscometer was used for the measurements.

The second phase of the experimental program is the characterization and the mechanical and physical testing of the radiation-cured carbon-epoxy prepregs. All tests will be done by the accepted procedures of the American Standard of Testing and Materials (ASTM). The prepreg physical tests will include measuring the resin content, gel fraction, volatile content, moisture content, resin flow and ply thickness. Table 3 lists the mechanical tests and specifications for a 14-ply laminate made of carbon fabric-epoxy prepregs (20). Tests will be conducted over the temperature range of 20 to 130°C (Table 3) with samples being irradiated in a vacuum bag assembly. Each property listed in Table 3 will be measured for the radiation-cured laminate and the results will be compared with the specifications.

3. RESULTS AND DISCUSSION

3.1 FT-IR SPECTROSCOPY

Figure 1 shows the spectra for the three components that were combined to produce the resin. The actual chemical structures of these compounds and their various additives are proprietary information of the resin supplier. The peaks common to each component can be associated with the acrylated structure ($R-\overset{\text{O}}{\parallel}{\text{C}}-\text{O}-\text{R}$ and $\text{R}=\overset{\text{O}}{\parallel}{\text{C}}-\text{O}-\text{R}$ at 1165 cm^{-1} , 1250 cm^{-1} and 1730 cm^{-1}), various unsaturated hydrocarbon structures ($\text{CH}=\text{CH}_2$ at 983 cm^{-1} and 1450 cm^{-1} ; $\overset{\text{H}}{\text{C}}=\text{C}-\text{O}-$ at 1635 cm^{-1}), and the CH_3 , CH_2 and CH peaks (2850 cm^{-1} to 3060 cm^{-1}). The broad peak at 3436 to 3550 cm^{-1} in both the epoxy diacrylate spectrum (a) and the dipentaerythritol monohydroxypentaacrylate spectrum (c) can be associated with a polyether structure (21). The remaining peaks unique to each spectrum, below 1800 cm^{-1} , identify various saturated and unsaturated fragments of the components' structure.

Figure 2 illustrates the qualitative effect of gamma irradiation in air on each of the three resin components. The irradiation dose was 100 kGy. The following general conclusions can be drawn from these spectra:

1. The amount of unsaturation, corresponding to peaks below 1800 cm^{-1} , is reduced in each of the components upon irradiation. The carbon-carbon double bond is a common reaction site for

radiation-produced free radicals during both polymerization and crosslinking. Acrylated peaks (1165 cm^{-1} , 1250 cm^{-1} and 1730 cm^{-1}) are reduced significantly, confirming that this fragment of each component takes an active part in the curing reactions.

2. Carbon dioxide (CO_2) (peak at 2300 to 2350 cm^{-1}) has been identified as a radiolysis product in both the polybutadiene diacrylate (Figure 2b) and the dipentaerythritol monohydroxypentaacrylate (Figure 2c) (22). The origin of the CO_2 is as yet unconfirmed. It may be produced during irradiation of the various acrylates. Atmospheric oxygen may also be involved in its production. Further experiments are required to identify the CO_2 -producing reactions and to determine the appropriate irradiation conditions to minimize its production.
3. Irradiation affects the CH_2 and CH_3 peaks (2850 cm^{-1} to 3060 cm^{-1}) of each spectrum (Figures 2a, 2b and 2c) differently. The epoxy diacrylate peaks (Figure 2a) remain unchanged. The polybutadiene diacrylate peaks (Figure 2b) are all reduced, suggesting that CH_2 and CH_3 may be involved in reactions to produce methane and hydrogen gases, which have not yet been identified. Only the CH_2 peak at 2903 cm^{-1} is reduced upon irradiation of the dipentaerythritol monohydroxypentaacrylate.
4. The polyether peaks (3436 to 3550 cm^{-1}) in Figures 2a and 2c are unchanged with irradiation.

3.2 GELATION

Figure 3 illustrates the effect of irradiation on the gel fraction of the cured polymer that was irradiated under pressure (100 or 1000 kPa) in either an air or a nitrogen atmosphere. The radiation dose to initiate gelation in air—the gel point—was 2 to 3 kGy. The gel point of the samples irradiated in a nitrogen atmosphere was slightly lower than 2 kGy. The gel fraction increased rapidly with dose, reaching its constant maximum level at a dose of 50 kGy in both air and nitrogen. The maximum gel fraction in a nitrogen atmosphere was about 97% and was independent of the pressure during irradiation (100 to 1000 kPa). The maximum gel fraction in air at 100 kPa pressure was about 94%. Increasing the pressure to 1000 kPa reduced the gel fraction over the entire dose range (2 to 50 kGy), with the maximum level being about 91%. This gel fraction reduction is related to both the sample surface area and the concentration of dissolved oxygen in the resin. Atmospheric oxygen diffuses further into the samples at elevated pressures, which would inhibit the cross-linking of the polymer by reacting with the free radicals produced during irradiation.

3.3 THERMAL CHARACTERISTICS

Differential thermal analysis of the radiation-cured polymer (dose = 30 to 50 kGy) in an argon atmosphere showed a characteristic endotherm at about 230°C . X-ray diffraction analysis of these samples, which were heated to 300°C and cooled at $1^\circ\text{C}/\text{min}$, showed that the polymer was amorphous and unchanged from the control samples, confirming that the endotherm was not a result of crystallization of the polymer. The X-ray scattering curve given in Figure 4 was typical of all of the samples analyzed. It is the

traditional curve for liquids and amorphous solids, showing only one broad peak (23). The polymer was not expected to crystallize because, as with most acrylated polymer formulations, the bulky side groups in the polymer structure makes molecular ordering, and hence crystallization, difficult (24).

Linear thermal expansion analysis confirmed that the differential thermal analysis (DTA) peak was due to polymer softening. Figure 5 is a plot of the linear expansion of the cured polymer (30, 40 and 50 kGy) as a function of temperature. The polymer softening point, about 235°C, is unaffected by irradiation. The slope of each curve below 150°C, the linear thermal expansion coefficient, is also unaffected by irradiation up to 50 kGy. The expansion coefficient is approximately 1.3×10^{-4} m/(m°C) for all three polymer samples (30, 40 and 50 kGy). The elucidation of the effect of irradiation on the thermal expansion of the polymer above 150°C requires further investigation. The curves in Figure 6 do not have any rapid slope increases, suggesting that the cured polymer may not have a detectable glass transition temperature (T_g).

Figure 6 plots the linear thermal expansion of the 50-kGy sample as a function of temperature during both heating and controlled cooling at 1°C/min in a vacuum. The maximum temperature of the samples was either 220°C (below the softening temperature) or 250°C (above the softening temperature). The figure illustrates how the expansion varied at a particular temperature depending on the thermal history of the sample. At a given temperature (27 to 250°C), the increased sample length during the cooling period was always less than the expansion during heating. This observation suggests that the polymer shrinks slightly as a result of the thermal cycle. As shown in Figure 6, a 1.3% reduction in the sample length was measured when the samples were heated to either 220 or 250°C and then cooled. Reheating a sample to 250°C did not shrink it further.

3.4 VISCOSITY AND DENSITY

The viscosity of the unirradiated resin was approximately 1 Pa•s at 80°C and 0.1 Pa•s at 200°C. The viscosity limit for a hot-melt pre-pegging process is 0.1 Pa•s to ensure proper fabric wetting and resin flow-out.

The density of the unirradiated resin and irradiated polymer (30 to 50 kGy) was about 1110 kg/m³. Varying the curing conditions (dose, atmosphere and pressure) did not significantly affect the polymer density.

3.5 LAMINATE IRRADIATIONS AND MECHANICAL TESTING

A prepreg manufacturer has impregnated carbon fabric with our specified resin formulation. These prepreps have been received and lamination and processing experiments have been started. Laminate sheets have been cured in the gamma cell using the vacuum bag assembly and samples are being cut from them for mechanical testing. The average resin content in the prepreg is about 36% and the cured ply thickness is 0.2 mm as specified in Table 2.

4. CONCLUSIONS

A radiation-curable polymer-carbon fibre prepreg composite has been designed to meet the mechanical- and physical-property specifications set by a major aircraft company. The selected resin formulation is a mixture of epoxy diacrylate (50%), polybutadiene diacrylate (30%) and dipentaerythritol monohydroxypentaacrylate (20%) resins. A plain-weave carbon fabric was selected for the prepreg material. A hot-melt process was used to impregnate the fabric with about 36% (by mass) of the resin.

The resin radiation cures through crosslinking reactions involving the acrylated groups in the resin components. The epoxy group takes little or no part in the curing reactions. Carbon dioxide has been identified as a radiolysis product in both the polybutadiene diacrylate and the dipentaerythritol monohydroxypentaacrylate.

The maximum gel fraction for the cured polymer, obtained at a dose of 50 kGy or greater, depended on the atmosphere and the pressure during irradiation. The maximum gel fraction in air varied from 94% at 100 kPa pressure to 91% at 1000 kPa pressure. The maximum gel fraction in nitrogen was 97% (100 to 1000 kPa). The polymer gel point was at 2 to 3 kGy. X-ray diffraction confirmed that the polymer was amorphous, even at a cooling rate of 1°C/min. The polymer softening point was about 235°C and the linear thermal expansion coefficient was about $1.3 \times 10^{-4} \text{ m}/(\text{m}\cdot^\circ\text{C})$ (30 to 50 kGy) up to 150°C. Heating the cured polymer to between 220 to 250°C and cooling it at 1°C/min caused the polymer to shrink 1.3%. The physical and mechanical testing of the irradiated prepreg laminates has been started.

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TABLE 1

RELATIVE PRODUCT CHARACTERISTICS FOR SELECTED COMPOUNDS

Property ¹	Dipentaerythritol monohydroxypenta- acrylate	Polybutadiene diacrylate	Epoxy diacrylate
Abrasion Resistance	X		X
Adhesion	X	X	X
Chemical Resistance	X		X
Flexibility	X	X	
Hardness	X		X
Heat Resistance	X		X
Impact Resistance		X	
Low Shrinkage		X	X
Water Resistance		X	X
Weatherability	X		
Molecular Weight	525	3000	428
Reactive Esters (%)	97.5	99+	98+
Density (kg/m ³ @ 25°C)	1190	940	1200
Viscosity (Pa*s @ 25°C)	15.25	0.01-0.02	~1000
Resin Composition (wt.%)	20	30	50

¹ X means that when the compound is included in a resin formulation, the specified property is imparted to the cured polymer.

TABLE 2

CARBON FABRIC SPECIFICATIONS AND FIBRE PROPERTIES

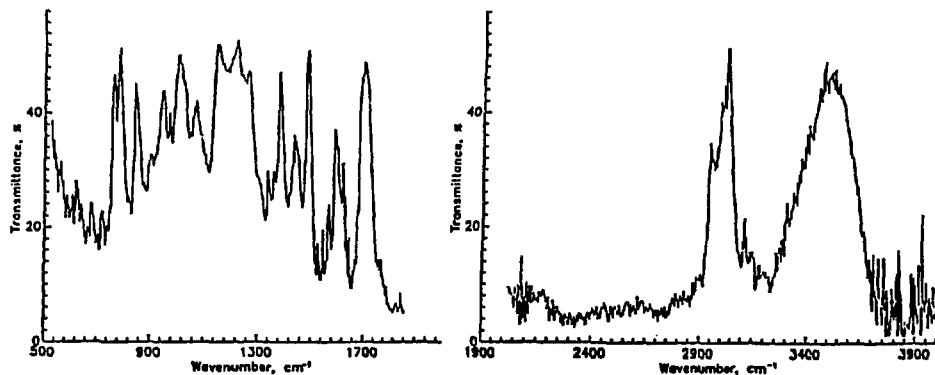
Specification/Property	Value
Fabric Weave	Plain
Filaments/Tow	3000
Fabric Area Weight (g/m ²)	193
Cured Fabric Thickness (mm)	0.2
Fibre Tensile Strength (MPa)	3790
Fibre Tensile Modulus (GPa)	234
Fibre Tensile Strain (%)	1.5
Fibre Density (kg/m ³)	1800
Prepreg Resin Content (wt.%)	35 ± 2.0

TABLE 3

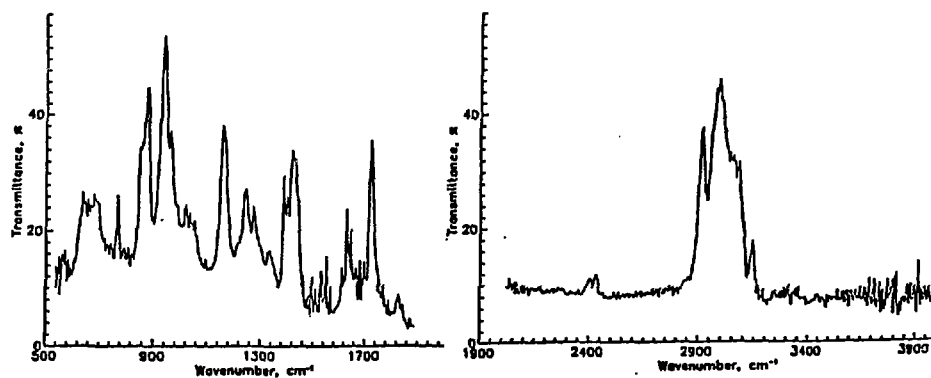
INDUSTRIAL CARBON FABRIC - EPOXY PREPREG LAMINATE SPECIFICATIONS¹

Property ²	Temperature ³ (°C)	Minimum ⁴ Average	Minimum Value
Ultimate Tensile Strength (MPa)	20-130	1275	1100
Tensile Modulus (GPa)	20-130	145	117
Tensile Strain (m/m) x 10 ⁶	20	9500	7500
	130	9500	7500
Ultimate Compression Strength (MPa)	20	1140	1030
	70	1070	900
	130	930	760
Compression Modulus (GPa)	20-130	131	97
Compression Strain (m/m) x 10 ⁶	20	8500	-
	70	7700	-
	130	7000	-
Wet Compression Strength (MPa) ⁵	20	483	345
	70	414	310
	130	241	172

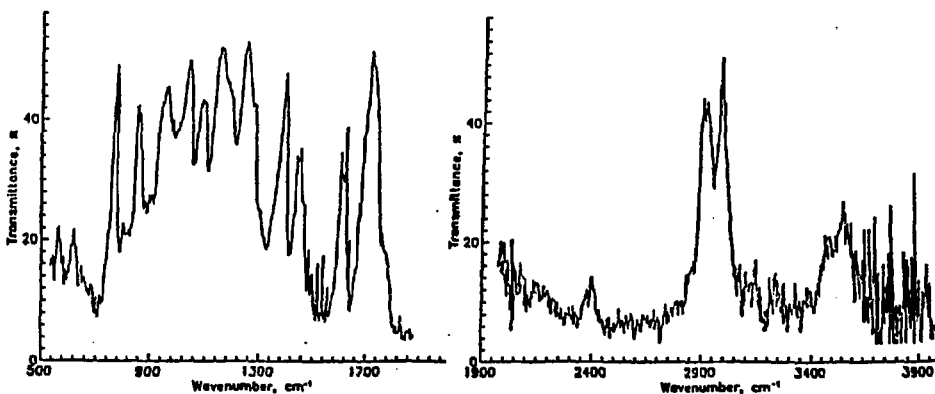
- 1 The laminate contains 14 plys, all in the same fibre orientation ($\pm 1^\circ$).
- 2 All properties will be measured using the American Standard of Testing and Materials approved methods.
- 3 The time specified at the test temperature prior to the test is 10 ± 3 min for all dry tests and 2 ± 1 min for all wet tests.
- 4 At least five specimens are to be tested.
- 5 Specimen conditioning: soaked in water at 71°C for 14 d prior to the test.



(a)

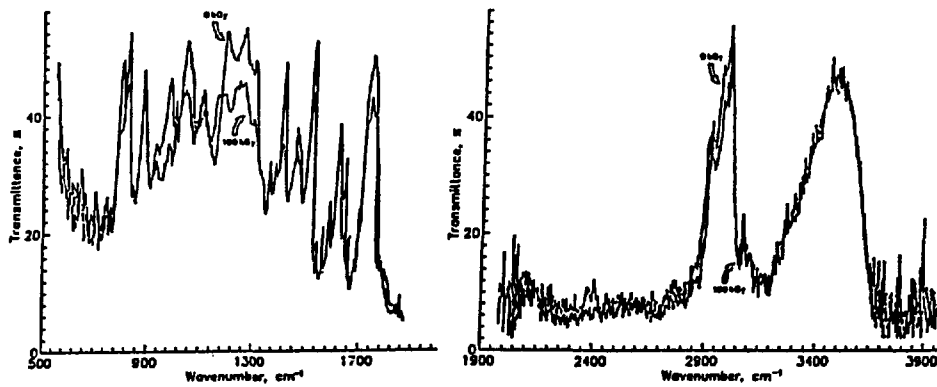


(b)

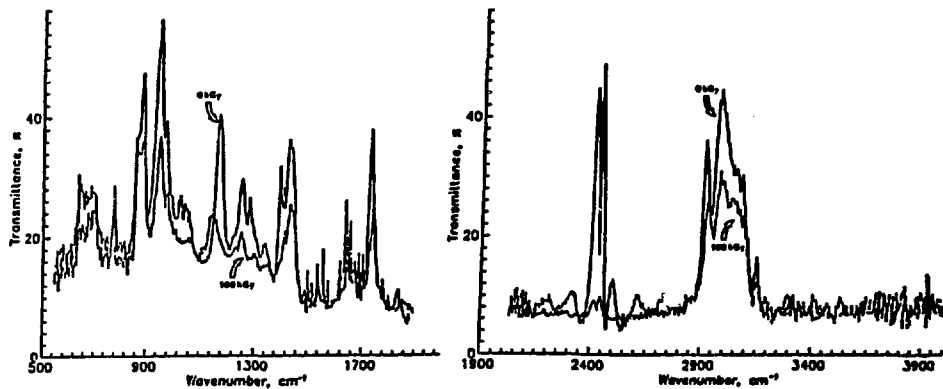


(c)

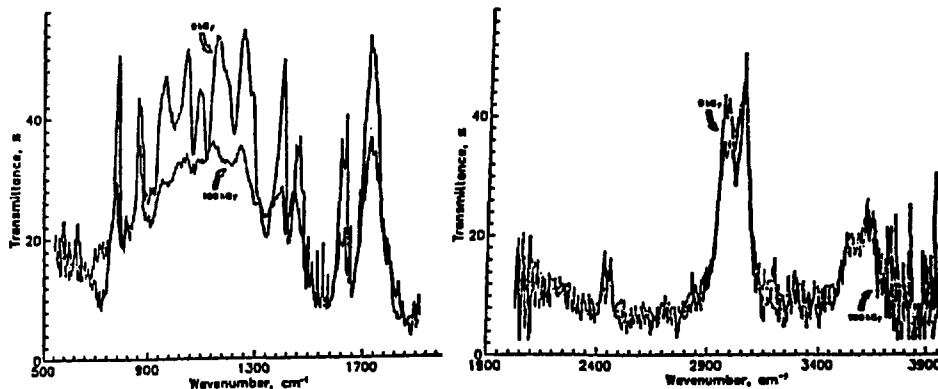
FIGURE 1: Photoacoustic Infrared Spectra of the Unirradiated Resin Component:
(a) Epoxy Diacrylate,
(b) Polybutadiene Diacrylate, and
(c) Dipentaerythritol Monohydroxypentaacrylate



(a)



(b)



(c)

FIGURE 2: Comparison of the Photoacoustic Infrared Spectra of the Irradiated and Unirradiated Resin Components:
(a) Epoxy Diacrylate,
(b) Polybutadiene Diacrylate, and
(c) Dipentaerythritol Monohydroxypentaacrylate

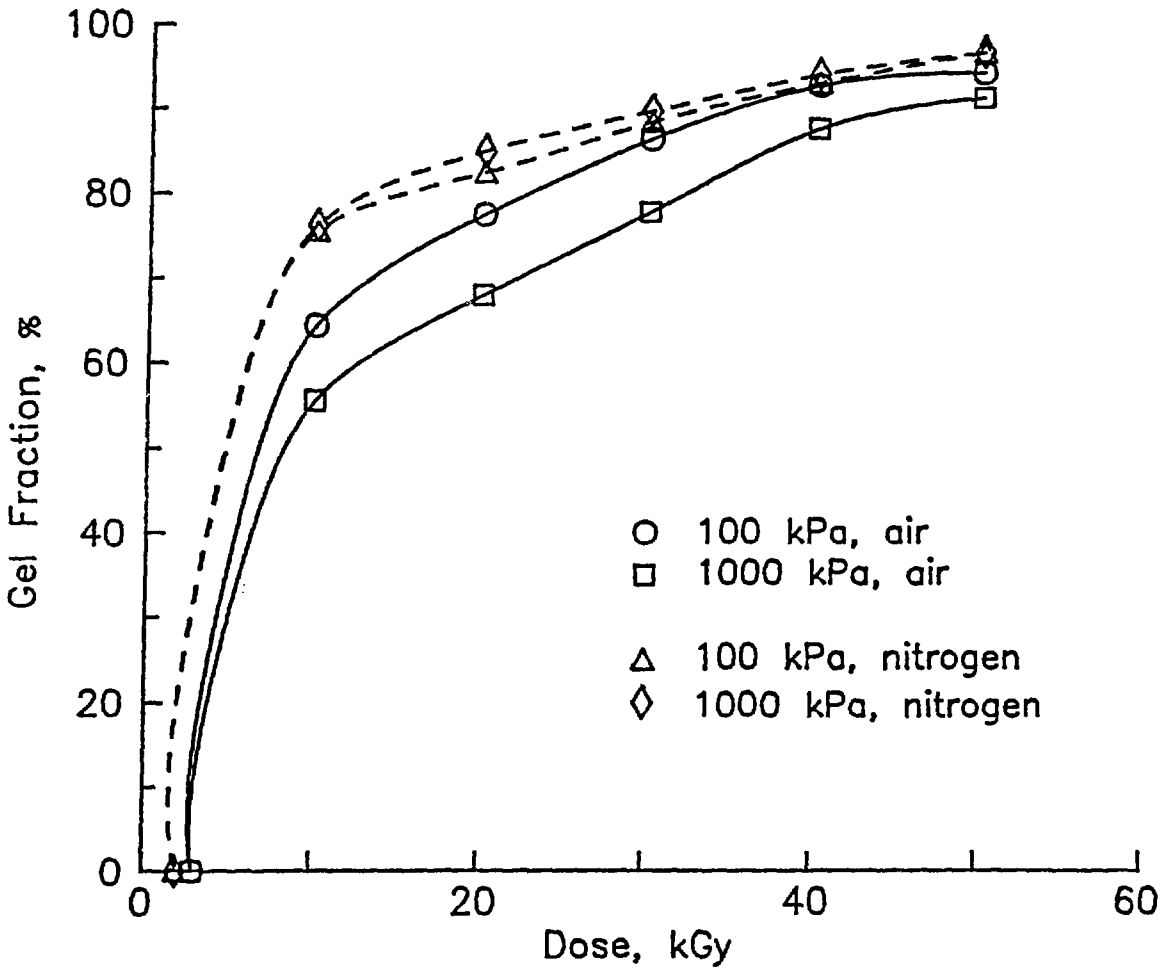


FIGURE 3: Gel Fraction Versus Irradiation Dose for the Prepreg Polymer

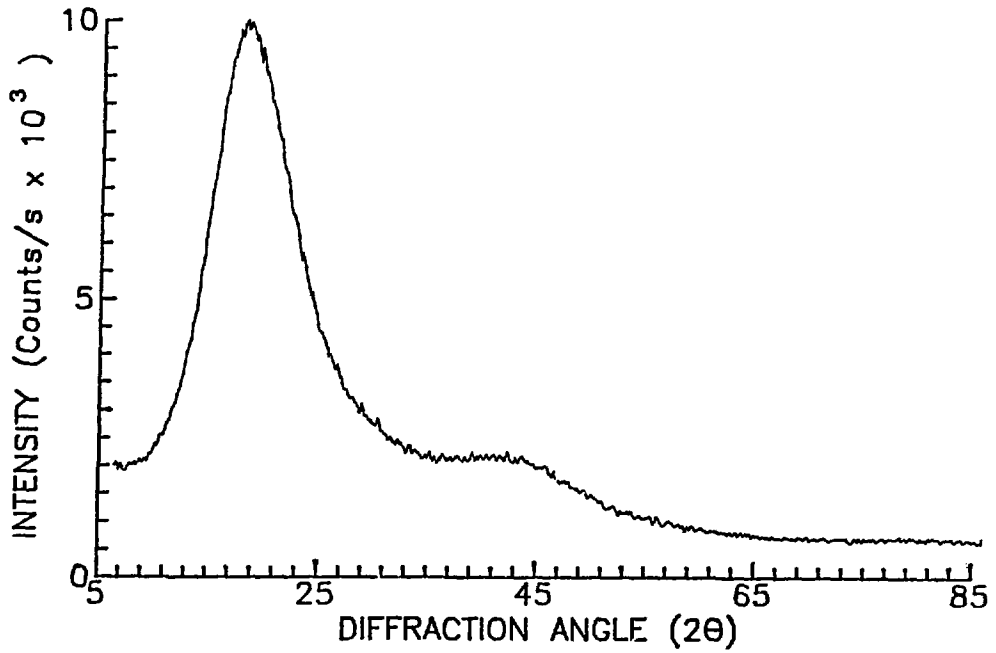


FIGURE 4: X-ray Diffraction Curve; Intensity Versus Diffraction Angle for the Cured Prepreg Polymer Cooled from 300°C at 1°C/min

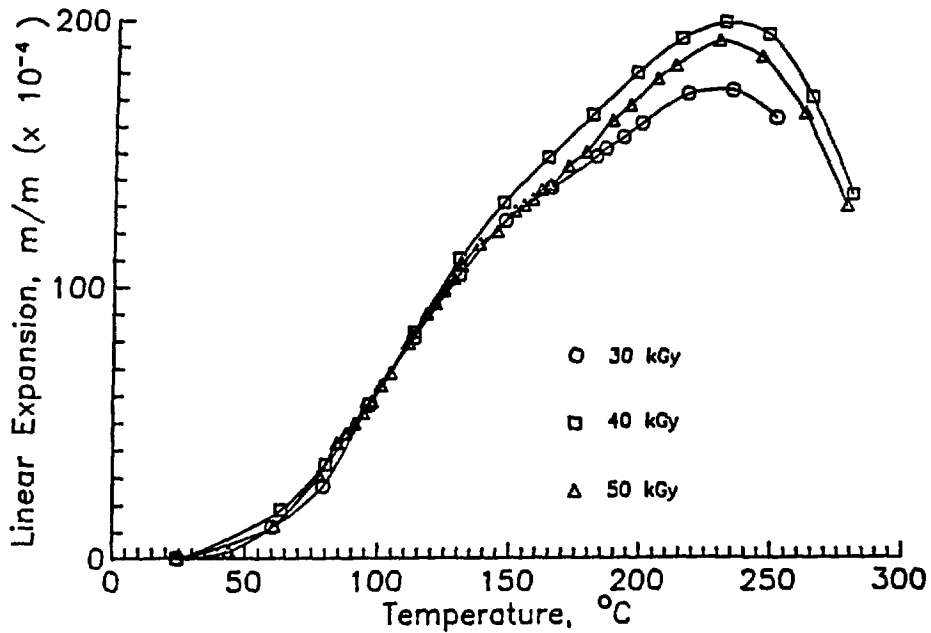


FIGURE 5: Linear Thermal Expansion of the Cured Prepreg Polymer Versus Temperature

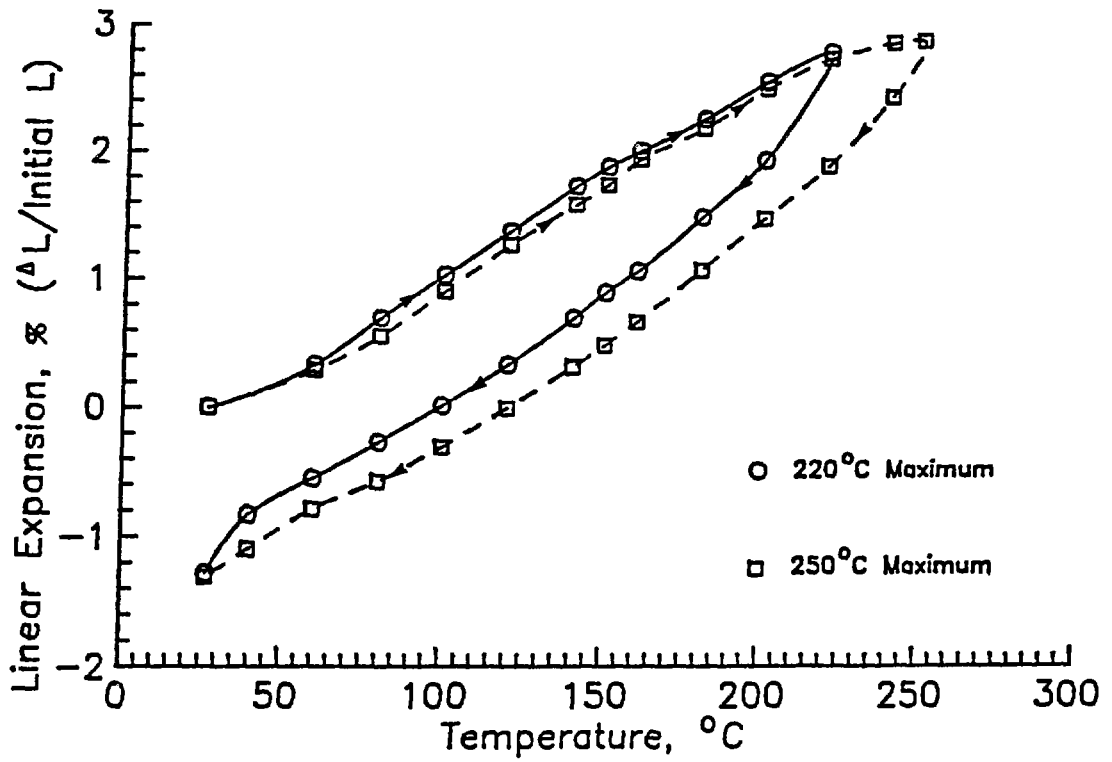


FIGURE 6: Linear Thermal Expansion Versus Temperature; Controlled Heating and Cooling Rates (1°C/min)

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