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CRITICAL PARAMETERS FOR ELECTRON BEAM CURING OF CATIONIC EPOXIES AND PROPERTY COMPARISON OF ELECTRON BEAM CURED CATIONIC EPOXIES VERSUS THERMAL CURED RESINS AND COMPOSITES

Christopher J. Janke, Robert E. Norris, Kenneth Yarborough
Oak Ridge Centers for Manufacturing Technology
Oak Ridge National Laboratory*
Oak Ridge, Tennessee 37831-8048

Stephen J. Havens
Oak Ridge Institute for Science and Education
Oak Ridge, Tennessee 37831-0117

Vincent J. Lopata
Atomic Energy of Canada Limited, Whiteshell Laboratories (AECL)
Pinawa, Manitoba R0E 1L0
Canada

ABSTRACT

Electron beam curing of composites is a nonthermal, nonautoclave curing process offering the following advantages compared to conventional thermal curing: substantially reduced manufacturing costs and curing times; improvements in part quality and performance; reduced environmental and health concerns; and improvements in material handling. In 1994 a Cooperative Research and Development Agreement (CRADA), sponsored by the Department of Energy Defense Programs and 10 industrial partners, was established to advance electron beam curing of composites. The CRADA has successfully developed hundreds of new toughened and untoughened resins, offering unlimited formulation and processing flexibility. Several patent applications have been filed for this work. Composites made from these easily processable, low shrinkage materials match the performance of thermal cured composites and exhibit: low void contents comparable to autoclave cured composites (less than 1%); superb low water absorption values in the same range as cyanate esters (less than 1%); glass transition temperatures rivaling those of polyimides (greater than 390°C); mechanical properties comparable to high performance, autoclave cured composites; and excellent property retention after cryogenic and thermal cycling. These materials have been used to manufacture many composite parts using various fabrication processes including hand lay-up, tow placement, filament winding, resin transfer molding and vacuum assisted resin transfer molding.

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DISCLAIMER

KEY WORDS: Electron Beam Curing; Epoxy Resins; Toughened Epoxy Resins

1. INTRODUCTION

Electron beam curing of Polymer Matrix Composites (PMCs) is a nonthermal, nonautoclave curing method that uses either high-energy electrons or electron-generated X-rays to initiate polymerization and crosslinking reactions in suitable resin systems. The electron beam curing of PMCs offers many advantages compared to baseline thermal curing including: reduced curing times, lower cost tooling, shelf stable resin systems, lower health risks because of fewer volatiles and the use of less hazardous chemicals (no hardeners are required in the resin formulation), reduced thermal stresses, curing at selectable temperatures, and offers the potential of making unique material combinations incorporating tailored resin systems.

Electron beam curing offers many other advantages compared to conventional thermal cured PMCs. Complex part shapes can be made with inexpensive tooling, and part throughput is extremely fast. Conventionally cured composites require the use of expensive tools that have to withstand tremendous autoclave pressures and elevated curing temperatures. These tools are often heavy and expensive and require labor-intensive disassembly and cleaning between each use. In many cases, the design of a composite structure must be compromised to allow removal of tools from cured parts. Electron beam processing allows the use of low cost fabrication tools made from reusable or disposable materials.

Interest in electron beam curing of PMCs over the last few years has dramatically increased due in part to its potential as a major cost-reduction technology for producing PMCs. Cost analysis studies from several independent industrial and governmental organizations on electron beam curing has consistently shown that cost savings from 25% to 65% can be realized depending on the size, shape and quantity of the part being manufactured (1).

In 1994 a Cooperative Research and Development Agreement (CRADA), sponsored by the Department of Energy Defense Programs and 10 industrial partners, was established to advance electron beam curing of PMC technology. Over the last several years a significant amount of effort within the CRADA has been devoted to the development and optimization of resin systems and PMCs that meet the performance of thermal cured composites. This highly successful materials development effort has resulted in a broad family of high performance, electron beam curable cationic epoxy resin systems possessing a wide variety of excellent processing and property profiles. Hundreds of new resin systems, both toughened and untoughened, offering unlimited formulation and processing flexibility have been developed and evaluated in the CRADA Program. Several patent applications have been filed for this work.

Electron beam curable cationic epoxy resin systems developed within the CRADA are friendly to the environment and greatly reduce the amount of waste generated in composite fabrication processes. Hazardous and carcinogenic chemical curing agents (hardeners) are not required, providing essentially unlimited shelf life and pot life for the electron beam resins. In addition, excess resin from manufacturing lines can be recovered and reused in subsequent fabrication runs. Conventional resins exhibit pot life of only a few hours after the addition of hardeners, and they cannot be reused. Prepreg materials made with conventional resins exhibit shelf life of only a few months, even when kept in refrigerated storage.

Electron beam curable cationic epoxy prepregs can be stored indefinitely at room temperature if sunlight and ultraviolet (UV) exposure is avoided. No oxygen inhibition problems or special handling procedures are required when using these electron beam curable resin systems. The relative cost of these materials are comparable to their thermally curable epoxy variants.

Electron beam curable cationic epoxies developed within the CRADA are easily processable and have yielded excellent thermal, mechanical, and moisture resistance properties including (2):

- Improved mechanical properties compared to thermal autoclave cured materials.
  PMCs made from electron beam curable cationic epoxy resins have exhibited some mechanical properties exceeding those of Fiberite's, autoclave cured, 977-2 and 977-3, toughened epoxy PMCs.

- Cryogenic and thermal cycling of electron beam curable cationic epoxy laminates showed excellent retention of properties.
  Mechanical properties of PMCs, made from electron beam curable cationic epoxies, after cryogenic and thermal cycling were unaffected and in some cases actually increased in value.

- Void contents comparable to autoclave cured composites.
  Many PMCs fabricated from electron beam curable cationic epoxy resin systems using conventional hand lay-up and filament winding processes have had less than 1% void content.

- Glass transition temperatures rivaling those of polyimides.
  Glass transition temperatures, (tan delta T_g's), of electron beam curable cationic epoxy resins and their PMCs have demonstrated values higher than 390°C; these are the highest T_g's ever reported for any commercially available epoxy resin systems. Surprisingly, these T_g's also exceed those of some polyimides.

- Superb low water absorption values in the same range as cyanate esters.
  Some of these electron beam curable cationic epoxy resin systems have exhibited water absorption values of less than 1% after 48 hour water boil. Thermal cured epoxies normally absorb water in the 3 - 6% range.

- Lower shrinkage.
  Shrinkage for electron beam curable cationic epoxy resins have been measured in the range of 2.2 - 3.4%. Thermal cured epoxies are generally in the range of 4 - 6% and electron beam curable acrylate resins are notoriously high in the range of 8 - 20%.

- Electron beam curable cationic epoxy resin systems have been processed using several, conventional fabrication methods.
  Many composite parts manufactured via hand lay-up, tow placement, filament winding, resin transfer molding (RTM) and vacuum assisted resin transfer molding (VARTM) have been produced using these materials, thus demonstrating their fabrication versatility.

In previous work (1-4) it was shown that one must optimize several variables for successfully electron beam curing of cationic epoxy resin systems used in PMC applications. If these variables are not fully optimized, then these materials will suffer from incomplete cure and inferior properties. In summary, these variables include:

1.1 Different Initiator Effects In our work we found that diaryliodonium salts are more efficient than triarylsulfonium salts assuming the same counterion (this same trend has also been confirmed by other investigators in the field of ultraviolet curing of cationic cure epoxies). Within the diaryliodonium (or triarylsulfonium) salt families, having the same counterion, differences in cure effectiveness exist. Initiators incorporating larger counteranions are generally more efficient than smaller, more tightly bound counteranions. Generally, the cure effectiveness generally decreases in the order:

$$SbF_6^- > AsF_6^- > PF_6^- > BF_4^-$$

In the CRADA we have identified four initiators (out of over 25 evaluated) that are extremely efficient in terms of electron beam cure effectiveness and these materials have formed the basis of most of our work.

Our studies have shown that there is significant differences in the thermal properties (i.e., T_g, service temperature) of electron beam cured cationic epoxy resin systems incorporating the various initiators. The T_g's of resins incorporating very efficient initiators do not change even after several thermal postcures. On the other hand, the T_g's of resins having inefficient initiators do change after thermal postcure. However, and more importantly, even after several thermal postcures the T_g's for these resin systems incorporating less efficient initiators never approach the T_g's of the same resin systems incorporating highly efficient initiators. The T_g's of electron beam cured cationic epoxy resins with very efficient initiators have been equal to or exceed those same identical epoxies which have been thermally cured with boron trifluoride monoethylamine catalyst. Additionally, the more efficient the initiator the less electron beam dose is required to cure the material.

1.2 Initiator Concentration Effects For most electron beam cured cationic epoxies the optimum initiator concentration is in the range of 1 - 3 phr. We have also determined that at optimum initiator concentration, the dose required to cure is at a minimum.

1.3 Electron Beam Dose Effects The majority of resins and PMC's cure at doses ranging from 70 - 250 kGy. The specific dose depends on the specific resin used, the initiator used and its concentration, fiber type, and the percent fiber content. Normally 150 kGy has been sufficient to cure most PMCs. Overdosing (i.e. up to 2 -3 times the dose needed to cure the materials) cationic cure epoxies has very little effect on their glass transition temperature and flexural modulus, E' values.

1.4 Special Precautions We have found that several materials inhibit the electron beam cationic cure of these epoxy materials. Materials which should be avoided include: Active nitrogen's (amines, hydrazines, etc.); Anionic surfactants; Calcium carbonate and basic clays; Alkaline materials; and Strong anions (Cl^-, Br^-, OH^-). Direct UV or solar radiation should be avoided because these radiation sources will prematurely initiate polymerization.

2. EXPERIMENTAL

2.1 Materials

2.1.1 Electron Beam Cured Cationic Epoxy Resin Unidirectional Prepregs All unidirectional prepreg material consisted of proprietary electron beam curable cationic epoxy resin systems (Electron Beam Cured Resins 1 -5) from Oak Ridge, and was processed at YLA, Inc. in Benicia, CA using IM7-GP-12K carbon fiber from Hercules, Inc.

2.1.2 Manufacture of Electron Beam Cured Cationic Epoxy Resin Unidirectional Laminates Each panel was 30.5 cm x 30.5 cm and consisted of 16 plies of unidirectional prepreg material. All panels were prepared using conventional lay-up techniques. Intermediate debulks were conducted under vacuum bag pressure every 4 plies at room temperature for 15 minutes. The final debulk and bleed cycle was performed under vacuum bag pressure at 70°C for 1 hour.

After the panels were laid up they were shipped to AECL in vacuum sealed bags for subsequent electron beam curing. Several days later, the panels were electron beam cured at room temperature under vacuum bag pressure at a dose per pass of 50 kGy for a total dose of either 150 kGy or 250 kGy.

2.1.3 Property Testing of Electron Beam Cured Cationic Epoxy Resin Unidirectional Laminates Several different tests were performed on the electron beam cured cationic epoxy resin unidirectional laminates. Percent void volume was measured by acid digestion (ASTM D 3171); glass transition temperature via DMA (T_g, tan delta; ASTM D 4065 and D 4092); O° flexural strength and modulus properties (ASTM D 790, data normalized to 62% fiber volume); O° compressive strength and modulus properties (ASTM D 695, data normalized to 62% fiber volume); and O° interlaminar shear strength properties (ASTM D 2344) for several electron beam cured IM7-GP-12K/Resin X (Resin X materials refer to Electron Beam Curable Cationic Epoxy Resins 1-5) unidirectional tape specimens.

2.1.4 Electron Beam Cured and Thermally Cured Filament Wound Cylinders All electron beam cured filament wound cylinders consisted of IM7-GP-12K carbon fiber from Hercules, Inc. and a proprietary electron beam curable cationic epoxy resin system (Electron Beam Cured Resin 6) from Oak Ridge. Two thermally cured epoxy resins were used as controls for comparison to the electron beam cured cationic epoxy resin system discussed above, including: 1. Union Carbide ERL-2258 cured with meta-phenylenediamine hardener (Thermal Cured Resin 1) and; 2. Dow Tactix 123 cured with 3 phr of Elf Atochem ATO Boron Trifluoride Monoethylamine Complex (Thermal Cured Resin 2).

2.1.5 Manufacture Of Electron Beam Cured And Thermally Cured Filament Wound Cylinders Hoop wound composite cylinders having part diameters of 15.24 cm and part thickness' of 0.3175 cm were produced at room temperature by wet filament winding using the three resins discussed above. After the cylinder containing the electron beam cured resin 6 was manufactured, it was shipped to AECL where it was electron beam cured while being rotated at room temperature for a total dose of 150 kGy. The cylinders containing the thermal cured resins (Thermal Cured Resin 1 and Thermal Cured Resin 2) were cured in an oven for 3 hours at 121°C , 3 hours at 150°C , then 4 hours at 177°C.

2.1.6 Property Testing Of Filament Wound Composites Properties testing of the electron beam cured and thermally cured filament wound composite parts included: Percent void volume using acid digestion (ASTM D 3171); percent fiber volume content (ASTM D 3171; glass transition temperature via DMA (T_g, tan delta; ASTM D 4065 and D 4092); O° tensile properties (similiar to ASTM D 2290, data normalized to 75%), cycled and uncycled; 90° flexure properties (similiar to ASTM D 790), cycled and uncycled; and O° interlaminar shear strength properties (ASTM D 2344), cycled and uncycled. Cycled specimens were treated to the following conditions before being tested at room temperature: samples were placed in liquid nitrogen dewar at -194°C for 30 minutes; allowed to warm to room temperature for 30 minutes; placed in oven at 121°C for 30 minutes; then, allowed to warm to room temperature for 30 minutes; the cycle was repeated 3 times.

2.2 Electron Beam Accelerator All electron beam irradiation's were performed at the Whiteshell Laboratories of AECL using the I-10/1 Electron Accelerator (10 MeV; 1 kW). The instantaneous dose rate was 1.5 Mgy s^{-1}.

2.3 Dynamic Mechanical Analysis (DMA) DMA was performed using a Rheometric Solids Analyzer RSA II. The resin and PMC samples were trimmed to fit a dual cantilever tool, with an initial stress and strain of zero. The applied strain used for measuring the theological properties was set at 0.1%. The temperature was raised at 2.5°C per step with a dwell time of 15 seconds. The frequency of the applied strain was 6.28 radians per second. Sample size was normally 3 mm x 7 mm x 55 mm. Measurements were obtained using the software provided by Rheometrics.

3. RESULTS AND DISCUSSION

One of the goals within the CRADA was to match the performance properties of Fiberite's, autoclave cured, IM7/977-2 and IM7/977-3 PMC laminates. Table 1 shows that many of the properties match surprisingly well and in some cases exceed the goals of the CRADA. Table 1 summarizes the property results of the electron beam cured, O° unidirectional laminates including: percent void volume using acid digestion; glass transition temperature via DMA (T_g, tan delta); O° flexural strength and modulus properties; O° compressive strength and modulus properties; and O° interlaminar shear strength properties for several electron beam cured IM7-GP-12K/Resin X (Resin X materials refer to Electron Beam Cured Cationic Epoxy Resins 1-5) unidirectional tape specimens. Also in Table 1 are the comparative property values, from recent Fiberite marketing data sheets, for Fiberite's autoclave cured IM7/977-2 and IM7/977-3 composite laminates.

Void volume for the electron beam cured composite laminates were all less than 2%, with two of the materials having less than 1% voids. These results were very encouraging. In effect, we were able to duplicate the low percent void contents that are typically associated with autoclave (high temperature/high pressure) cured composites by using only vacuum bag pressure and electron beam curing at room temperature. The glass transition temperatures (tan delta) for all of the electron beam cured laminates exceeded 200°C, and two of the electron beam cured laminates, Electron Beam Resin 1 and Electron Beam Resin 2, exceeded 390°C; these are the highest T_g's ever reported for any commercially available epoxy resin systems. Surprisingly, these T_g's also exceed those of some polyimides.

The flexural strength and flexural modulus properties for the electron beam cured laminates in Table 1 were also unexpectedly high in view of the fact that these materials were processed using a nonthermal, nonautoclave process. All five of the electron beam cured laminates exceeded the flexural properties of the 977-2 laminates. In addition four out of the five electron beam cured laminates exceeded the flexural properties of the 977-3 laminates. Additionally, the compression strength and compression modulus properties determined thus far on the Electron Beam Resin 1 laminates are well within the experimental error of the corresponding values for the 977-2 laminates. The compression properties for Electron Beam Resin 1 laminates were also within 3-7% of the modulus and strength values, respectively for the 977-3 laminates. The interlaminar shear strength properties of the electron beam cured laminates are about 70-80% of the corresponding values for the 977-2 laminates. We fully expect these shear properties and hot/wet properties to continue to increase for the electron beam cured materials when further work is completed in the areas of processing optimization and fiber sizing.

Another goal within the CRADA was to develop low viscosity resins for use in wet filament winding, resin transfer molding, and vacuum assisted resin transfer molding processes. Table 2 shows that the electron beam curable cationic epoxies that have been developed perform very well in comparison to their thermally curable variants.

The results for the filament wound laminates in Table 2 show that electron beam curable cationic epoxy resins can be used to make laminates having less than 1% void contents, T_g's around 200°C, and high mechanical properties compared to conventional, thermally cured epoxy resins. In addition, the mechanical property values of these electron beam cured laminates after cryogenic and thermal cycling exhibited excellent property retention.

Table 2 summarizes the property results of the electron beam cured and thermally cured filament wound composite properties including: Percent voids by acid digestion; Percent fiber volume content by acid digestion; glass transition temperature via DMA (T_g, tan delta); O° tensile properties, cycled and uncycled; 90° flexure properties, cycled and uncycled; and O° interlaminar shear strength properties, cycled and uncycled. Cycled specimens were treated to the following conditions before being tested at room temperature: samples were placed in liquid nitrogen dewar at -194°C for 30 minutes; allowed to warm to room temperature for 30 minutes; placed in oven at 121°C for 30 minutes; then, allowed to warm to room temperature for 30 minutes; the cycle was repeated 3 times.

As seen in Table 2 two thermal cured epoxy resins, Thermal Cured Resins 1 and 2, were used to fabricate filament wound laminates for the purposes of comparing their mechanical properties with an electron beam curable cationic epoxy resin system, Electron Beam Resin 6. The results in Table 2 indicate that the room temperature tested values for O° tensile strength, 90° flexure strength, and O° interlaminar shear strength of the electron beam cured filament wound laminates all exceeded the values of both of the thermally cured laminates.

Table 2 also shows that cryogenic and thermal cycling of the electron beam cured or thermal cured filament wound laminate specimens several times from liquid nitrogen temperatures, -194°C, to elevated temperatures, 121°C, had no deleterious effect on any of the mechanical properties tested in Table 2. In some cases these properties actually increased in value. This result is very encouraging and reinforces the concept of using electron beam cured cationic epoxy materials for applications involving cryogenic and/or thermal cycling (e.g., space).

CONCLUSIONS

The CRADA has successfully developed and optimized a broad family of high performance, electron beam curable cationic epoxy resin systems possessing a wide range of excellent processing and property profiles. Hundreds of new resin systems, toughened and untoughened, offering unlimited formulation and processing flexibility have been developed and several patents have been filed for this work. PMCs made from these easily processable, low shrinkage materials have been demonstrated to exhibit: low void contents which are comparable to autoclave cured composites (less than 1%); superb low water absorption values in the same range as cyanate esters (less than 1%); glass transition temperatures rivaling those of polyimides (greater than 390°C); mechanical properties comparable to high performance autoclave cured PMCs; and excellent property retention after cryogenic and thermal cycling. These materials have been used to manufacture several composite parts using various fabrication processes including hand lay-up, tow placement, filament winding, resin transfer molding and vacuum assisted resin transfer molding.

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Table 1

Property Comparison Of Electron Beam Cured Versus Thermal Cured IM7/Resin (X) Unidirectional Laminates (Data Normalized to 62% fiber volume)

Resin Systems	Fiberite 977-2 (Fiberite Marketing Literature Data)	Fiberite 977-3 (Fiberite Marketing Literature Data)	Electron Beam Resin 1	Electron Beam Resin 2	Electron Beam Resin 3	Electron Beam Resin 4	Electron Beam Resin 5
Cure Conditions	Autoclave Cured (6 hrs. @ 350°F @ 85 psi)	Autoclave Cured (3 hrs. @ 355°F @ 85 psi)	250 kGy	150 kGy	150 kGy	150 kGy	150 kGy
Void Volume, %	Not Reported	Not Reported	1.77	0.72	1.24	0.64	1.18
Tg, °C (Tan Delta)	200	190/240	396	392	232	212	212
O° Flexural Strength, MPa (ksi)	1641 (238)	1765 (256)	1986 (288)	2006 (291)	1793 (260)	1765 (256)	1710 (248)
O° Flexural Modulus, GPa (msi)	147 (21.3)	150 (21.7)	196 (28.5)	163 (23.6)	163 (23.7)	154 (22.3)	150 (21.8)
O° Compressive Strength, MPa (ksi)	1580 (230)	1680 (244)	1565 (227)				
O° Compressive, Modulus, GPa (msi)	152 (22)	154 (22.3)	149 (21.6)				
O° Interlaminar Shear Strength, MPa (ksi)	110 (16)	127 (18.5)	77 (11.2)	79 (11.5)	79 (11.5)	89 (12.9)	77 (11.2)
Hot/Wet O° Interlaminar Shear Strength*, MPa (ksi)		89 (12.9)	61 (8.8)				
* 1 week immersion in H_2O @ 160°F, tested at 220°F							

Table 2

Property Comparison Of Electron Beam Cured Versus Thermal Cured IM7-GP-12K/Resin (X) Filament Wound Laminates (15.24 cm dia.; 0.3175 cm thickness)

Resin Systems	Electron Beam Resin 6	Thermal Cured Resin 1 (Oven Cured)	Thermal Cured Resin 2 (Oven Cured)
Cure Conditions	150 kGy	3 hours @ 121°C, 3 hours @ 150°C, then 4 hours @ 177°C	3 hours @ 121°C, 3 hours @ 150°C, then 4 hours @ 177°C
Void Volume, %	< 1 %	< 1%	< 1%
Percent Fiber Content	75.8	77.8	74.6
Tg, °C (Tan Delta)	192	218	165
O° Tensile Strength, MPa (ksi) - Norm. to 75% F.V.	2358 (342)	1986 (288)	1538 (223)
O° Tensile Strength, Cycled*, MPa (ksi) - Norm. to 75% F.V.	2337 (339)	2379 (345)	1565 (227)
90° Flexure Strength, MPa (ksi) - Concave up	70.3 (10.2)	68.3 (9.9)	57.2 (8.3)
90° Flexure Strength, Cycled*, MPa (ksi) - Concave up	77.9 (11.3)	77.2 (11.2)	64.1 (9.3)
O° Interlaminar Shear Strength, MPa (ksi)	73.8 (10.7)	57.2 (8.3)	47.6 (6.9)
O° Interlaminar Shear Strength, Cycled*, MPa (ksi)	82.7 (12.0)	56.5 (8.2)	46.9 (6.8)

* Cycling Procedure: Specimens placed in liquid nitrogen (-194°C) for 30 min.; Allowed to warm to room temp. for 30 min.; Placed in oven @ 121°C for 30 min.; Allowed to warm to room temp. for 30 min.; Cycle repeated 3 times.

CONCLUSIONS

The CRADA has successfully developed and optimized a broad family of high performance, electron beam curable cationic epoxy resin systems possessing a wide range of excellent processing and property profiles. Hundreds of new resin systems, toughened and untoughened, offering unlimited formulation and processing flexibility have been developed and several patents have been filed for this work. PMCs made from these easily processable, low shrinkage materials have been demonstrated to exhibit: low void contents which are comparable to autoclave cured composites (less than 1%); superb low water absorption values in the same range as cyanate esters (less than 1%); glass transition temperatures rivaling those of polyimides (greater than 390°C); mechanical properties comparable to high performance autoclave cured PMCs; and excellent property retention after cryogenic and thermal cycling. These materials have been used to manufacture several composite parts using various fabrication processes including hand lay-up, tow placement, filament winding, resin transfer molding and vacuum assisted resin transfer molding.

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