

TOUGHENED EPOXY RESINS CURED BY ELECTRON BEAM RADIATION

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ABSTRACT

Preliminary research has determined that conventional epoxy resins can be cured by exposure to Electron Beam (EB) radiation as provided by a high energy/power electron beam accelerator to provide materials with high glass transition temperatures and mechanical properties comparable to thermally cured epoxies. A cationic photoinitiator at a concentration of 1-3 parts per hundred of the epoxy resin is required for this process. However, these materials tend to be brittle. In order to increase the fracture toughness (K_{IC}) of the EB cured materials, a variety of toughening agents were incorporated into the epoxy resin/photoinitiator blend in concentrations ranging from 5 - 30 percent. High molecular weight thermoplastics such as Udel P-1700, Radel A-200, and Ultem 1000 and hydroxy-terminated polysulfone oligomers nearly doubled K_{IC} with no reduction in glass transition temperature. Viscosities of the thermoplastic/epoxy resin/photoinitiator blends were extremely high. Reactive flexibilizers and rubbers also provided tougher materials, but sometimes with reduction in glass transition temperature. For example, a blend of 10 percent Shell Kraton FG 1901 X (a maleic anhydride terminated SEBS copolymer) with

*Managed by Lockheed Martin Energy Research Corporation for the U.S. Department of Energy under contract DE-AC05-96OR22464.

**Managed by Lockheed Martin Energy Systems Incorporated for the U.S. Department of Energy under contract DE-AC05-84OR21400.

Dow Tactix 123 (bisphenol A epoxy) containing 3 phr (4-octyloxyphenyl) phenyliodonium hexafluoroantimonate was irradiated at a total dosage of 150 kGy. Glass transition temperature ($\tan \delta$) of the cured material was 171 °C and the K_{IC} was 0.92 MPa·m^{1/2}.

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

1. INTRODUCTION

Previously the authors described a process by which conventional epoxy resins can be cured by electron beam (EB) radiation at selectable temperatures to provide materials with high glass transition temperatures (essentially the same as with thermal curing) (1). A cationic photoinitiator at a concentration of 1-3 parts per hundred of the epoxy resin is required. This process was developed as part of a Cooperative Research and Development Agreement (CRADA) sponsored by the Department of Energy Defense Programs and ten industrial partners. One of the primary objectives of this project is the development of high performance EB curable matrix resins for use in DOE Defense Programs and United States aircraft and aerospace industries. Although epoxy resins cured by this process have flexural strengths and moduli comparable to thermally cured epoxy resins, they tend to be somewhat brittle. Resins intended for use in aircraft, aerospace, and many other applications must have sufficient toughness and impact resistance to endure many years of harsh service. What is needed is a means by which EB cured cationic epoxies can be toughened and still retain the good thermal and mechanical properties of the original composition. Additionally, the toughening agent must not interfere with the cationic curing mechanism and must be miscible with or form a stable suspension with the epoxy resin-photoinitiator composition.

A variety of toughening agents and flexibilizers have been used with thermally cured epoxy resins, which typically use an amine coreactant or hardener for crosslinking. These tougheners include: rubbers or elastomers, either as discrete particles or reacted with epoxy resin (2-7), epoxy functionalized flexibilizers (8), engineering thermoplastics (9-12), and amine or hydroxy-terminated thermoplastic oligomers (13-16). There are no references in the literature to the use of these or any other materials to toughen an EB curable cationic epoxy composition (patents are pending). Our approach has been the evaluation of a wide variety of toughening agents in a baseline EB curable cationic epoxy composition, with the expectation that the results obtained would be applicable to higher performance epoxy compositions. EB irradiation of a mixture of Dow Tactix 123 (a high purity, low viscosity diglycidyl ether of bisphenol A) containing three parts per hundred (4-octyloxyphenyl) phenyliodonium hexafluoroantimonate (OPPI) resulted in a cured material with a glass transition temperature of 182 °C (1). The initial low viscosity of this composition (~ 6000 cP at 25 °C) made it very amenable to the addition of toughening agents. The addition of high molecular weight thermoplastics in particular significantly increased the viscosity of the epoxy resin composition. Compositions of the epoxy resin, cationic photoinitiator, and toughener were cured using a high energy EB accelerator. Thermal and mechanical properties, including fracture toughness (K_{IC}), were determined.

2. EXPERIMENTAL

2.1 Materials

2.1.1 Epoxy Resins Commercial epoxy resins were obtained from the manufacturer. These are described in Table 1.

2.1.2 Photoinitiator (4-Octyloxyphenyl) phenyliodonium hexafluoroantimonate (OPPI) was purchased from General Electric Silicones Company as Aryl Fluoroantimonate, Product 479-2092 C.

2.1.3 Toughening Agents Samples of commercial materials were obtained from the various manufacturers (Table 1). Hydroxy-terminated polysulfone oligomers were either generously provided by NASA Langley Research Center or prepared as described in the literature (17).

2.2 Epoxy-Toughening Agent Blending Many of the toughening agents were easily stirred into the epoxy resin at approximately 80°C along with the photoinitiator to form a homogeneous mixture. Udel P-1700 and the hydroxy-terminated polysulfone oligomers were slowly dissolved in the epoxy at 140-150°C with vigorous stirring. The viscous solutions were allowed to cool to approximately 80°C before the photoinitiator was added. Other high molecular weight thermoplastics such as Radel A and Ultem 1000 were dissolved in methylene chloride, the solution mixed with the epoxy resin, and the methylene chloride removed under vacuum at elevated temperature. Solid Kraton rubber was either similarly blended into the epoxy using toluene or melt blended into the epoxy at 190-200°C for 30 minutes using a high shear mixer. Again, the photoinitiator was not added until the temperature cooled to approximately 80°C. Nylon particles such as Orgasol 2001 and polyimide particles such as IMITEC X-902 were insoluble and dispersed to form a suspension. Rubber particles such as Paraloid EXL-2330 were blended into the epoxy as a dispersion in acetone, with subsequent removal of the acetone under vacuum at elevated temperatures.

2.3 EB Accelerator All electron beam irradiation was performed at the Whiteshell Laboratories of AECL using the I-10/1 Electron Linear Accelerator (Energy, 10MeV; Power, 1kW).

2.4 Fracture Toughness The previously degassed toughened epoxy-photoinitiator mixture was poured hot into an aluminum mold (specimen thickness, 6.25 mm) which was treated with release agents. Thickness of the side plates was 1.6 mm. Specimens were placed in a vacuum oven maintained at approximately 80°C to remove any remaining bubbles, allowed to cool, then irradiated in multiple passes of 50 kGy per pass for a total of 150 kGy. Cured panels were cut into 12.5 x 90 mm test specimens and fracture toughness (K_{IC}) determined according to ASTM E 399 using 6-10 specimens per test condition.

2.5 Dynamic Mechanical Analysis Dynamic Mechanical Analysis (DMA) was carried out using a Rheometrics Solids Analyzer RSA. II. The sample was placed in a dual cantilever tool, with an initial stress and strain of zero. The strain used for measuring the rheological properties was set at 0.1 percent. The temperature was raised at 2.5°C per step with a dwell time of 12 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 for the instrument.

2.6 Scanning Electron Microscopy (SEM) Micrographs of the fracture surfaces were taken using a JEOL JSM-35 CF Scanning Electron Microscope operated at 25 kV.

3. RESULTS AND DISCUSSION

Untoughened EB cured bisphenol A-based epoxy resin was quite brittle. The addition of any toughening agent resulted in some increase in fracture toughness (K_{IC}). Addition of high molecular weight, high glass transition temperature (T_g) thermoplastics to the uncured resin composition resulted in increased toughness with little reduction and some increased T_g 's in the cured resin (Table 2). Only small decreases in moduli were observed. In several cases (Radel and Ultem) increasing toughener concentration was ineffective. Concentrations of Udel up to 20 percent were increasingly effective. However, the viscosity of the toughened epoxy resin composition increased exponentially with Udel concentration (e.g. 7000 and 50,000 cP for 10 and 20 percent solutions, respectively, of Udel in Tactix 123 at 60°C). Udel was more useful since it could be dissolved into the epoxy without the use of solvents. Insoluble nylon and polyimide particles (Orgasol and IMITEC) did not substantially increase resin viscosity, but did increase toughness without decreasing T_g or modulus.

In general the hydroxy-terminated polysulfone oligomers were more effective as their concentration and molecular weight increased (Table 3). For the same degree of toughness, viscosities of the hydroxy-terminated polysulfone compositions were comparable to high molecular weight thermoplastic compositions. For example, the viscosity of the 30 percent solution of 6000 g/mole polysulfone oligomer in Tactix 123 was approximately 29,000 cP at 60°C. For the most part the addition of reactive flexibilizers only increased toughness by 30-40 percent (Table 4), often with decreased T_g 's. However the low viscosity flexibilizers kept the viscosities of the resin compositions low and suitable for resin transfer molding (RTM) or filament winding applications. Tactix 123 containing 10 percent Vikoflex 7170 had a viscosity of 3600 cP at 24°C. The effect of rubbers and elastomers was varied (Table 5). All aided in increasing toughness, but many significantly reduced T_g . Of particular interest is the 10 percent mixture of Kraton FG1901X with Tactix 123. Fracture toughness of the EB cured composition was more than double that of untoughened Tactix 123 (control) and T_g and initial service temperature were slightly higher than for the control. No low temperature transitions were observed in the DMA curves. However, when the Kraton concentration was increased to 20 percent, a slight decrease in fracture toughness and the appearance of a transition at 86°C in the DMA curve occurred. Viscosity of the 10 percent Kraton FG1901X Tactix 123 mixture was 1100 cP at 60°C. Kraton FG1901X is a styrene-ethylene-butylene-styrene (SEBS) block copolymer which is terminated with maleic anhydride. This rubber was specially designed for the toughening of epoxy resins.

Weight gain after 48 hours in boiling water was also determined for a number of the specimens. Values were typically low (1-1.5 percent). Incorporation of toughening agents such as Udel P-1700 and Radel A-200 resulted in less water absorption than obtained with untoughened epoxy resin.

For comparison purposes, the T_g (tan delta) of the thermally cured 977-3 toughened resin system is reported as 190°C and the fracture toughness is 0.90 MPa·m^{1/2}. Weight gain when exposed to 65°C and 85% relative humidity to equilibrium is approximately 1.1%.

Fracture surface of the untoughened epoxy specimens were smooth and glassy when viewed under an optical microscope (60 X). This morphology is also seen in the SEM of this surface (Figure 1). The fracture surface of the thermoplastic (Udel P-1700) toughened specimens also looked smooth, but there is some indication of “tough crack initiation.” The SEM of these fracture surfaces is also much rougher (Figure 2). The fracture surface of a specimen toughened with core shell rubber particles (Rohm and Haas Paraloid EXL-2330) exhibits crack front growth which suggests a ductile-brittle advance of the fracture (run-arrest). A very rough surface with evidence of “pull out” is seen in the SEM (Figure 3). The specimen toughened with Kraton rubber exhibited an extremely rugged fracture surface morphology with evidence of large voids created by “pull out” of large pieces of material. The fracture appears to be a ductile failure. The SEM of this surface also exhibits large irregular morphology (Figure 4). This probably accounts for the high fracture toughness of this composition.

4. CONCLUSIONS

Thermoplastics, hydroxy-terminated polysulfone oligomers, reactive flexibilizers, rubbers, and elastomers all increased the fracture toughness of an EB cured epoxy resin composition. The use of thermoplastics such as Udel P-1700 and rubbers such as Kraton 1901X provided significant increases in fracture toughness without adverse reduction of glass transition temperature and modulus.

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Table 1. Description of Materials

Epoxy Resins	Description
Tactix 123	Dow Tactix 123: High purity, low viscosity diglycidyl ether of bisphenol A; Epoxy equiv. wt., 172-176; Viscosity at 25°C, 4400-5600 cP.
Tactix 556	Dow Tactix 556: Glycidyl ether of the condensation product of dicyclopentadiene and phenol; Epoxy equiv. wt., 215-235; Viscosity at 85°C, 1000-1500 cP.
Tactix 742	Dow Tactix 742: Triglycidyl ether of tris (hydroxyphenyl)methane; Epoxy equiv. wt., 150-170; Viscosity at 79°C, 2860 cP.
DER 661	Dow D.E.R. 661: Solid diglycidyl ether of bisphenol A; Epoxy equiv. wt., 500-560.
ERL 2258	Union Carbide ERL 2258 (Discontinued): 50:50 Blend of bis (2, 3-epoxy cyclopentyl) ether isomers (ERL 4205) and diglycidyl ether of bisphenol A.
Toughening Agents	Description
Udel P-1700 NT	Amoco Udel P-1700 NT: Polysulfone; Glass Transition Temperature (T _g) 190°C.
Radel A-200 NT	Amoco Radel A-200 NT: Polyethersulfone; T _g , 220°C.
Ultem 1000 - 1000	General Electric Ultem 1000 - 1000: Polyetherimide; T _g , 220°C.
Hydroxy - Terminated Polysulfone Oligomers	Polysulfone oligomers based on the Udel P-1700 backbone were synthesized by the reaction of a calculated excess of bisphenol A with bis(4-chlorophenyl)sulfone in the presence of potassium carbonate. T _g 's for calculated molecular weights of 1500, 2650, 5360, 6000, and 8890 g/mole were 125, 146, 170, 176, and 180°C, respectively.
Orgasol 2001	Elf Atochem Orgasol 2001 UD NAT 2: Nylon 12 in ~ 5 μm particles; mp, 175°C.
Vikoflex 7010	Elf Atochem ATO Vikoflex 7010: Methyl epoxy soyate; Viscosity at 25°C, 80 cP.
Vikoflex 7170	Elf Atochem ATO Vikoflex 7170: Epoxidized soybean oil; Viscosity at 25°C, 380 cP.
Vikoflex 7190	Elf Atochem ATO Vikoflex 7190: Epoxidized linseed oil; Viscosity at 25°C, 660 cP.
Vikolox 14	Elf Atochem ATO Vikolox 14: 1,2-Tetradecane oxide.
DER 736	Dow D.E.R. 736: Polyglycoldiepoxy; Epoxy equiv. wt., 175-205; Viscosity at 25°C, 30-60 cP.
Heloxy 32	Shell Heloxy Modifier 32: Polypropylene glycol glycidyl ether; Epoxy equiv. wt., 305-335; Viscosity at 25°C, 45-90 cP.

Table 1. Description of Materials Continued

Heloxy 68	Shell Heloxy Modifier 68: Neopentyl glycol diglycidyl ether; Epoxy equiv. wt., 130-140; Viscosity at 25°C, 13-18 cP.
Heloxy 71	Shell Heloxy Modifier 71: Dimer acid diglycidyl ester; Epoxy equiv. wt., 390-490; Viscosity at 25°C, 400-900 cP.
Heloxy 505	Shell Heloxy Modifier 505: Castor oil polyglycidyl ether; Epoxy equiv. wt., 550-650; Viscosity at 25°C, 300-500 cP.
Poly bd 600	Elf Atochem ATO Poly bd 600 VM 312051: Internally epoxidized 1,3-butadiene homopolymer; Viscosity at 25°C, 68 cP.
Tone 301	Union Carbide Tone 301: ϵ -Caprolactone triol; Hydroxyl equiv. wt., 98-103; Viscosity at 25°C, 2250 cP.
PY 322	Ciba-Geigy PY 322: Modified bisphenol A glycidyl ether.
Dow XU 71790.04L	Dow XU 71790.04L: Bisphenol A glycidyl ether reacted with acrylic rubber.
EXL -2330	Rohm and Haas Paraloid EXL-2330: Methacrylate-butadiene core shell rubber
Epon 58134	Shell Epon 58134: Bisphenol A glycidyl ether blended with castor oil glycidyl ether and a polyurethane resin.
Kraton FG 1901X	Shell Kraton FG 1901X: Styrene-ethylene-butylene-styrene block copolymer terminated with maleic anhydride.

Table 2. Toughening of Radiation Cured Epoxy Resins with Engineering Thermoplastics^{1,2}

Toughening Agent	Toughening Agent Concentration (%)	Epoxy Resin	Glass Transition Temperature (°C)		Initial Service Temperature ³ (°C)	Weight Gain (48h H ₂ O Boil) (%)	Fracture Toughness, K _{IC} (MPa·m ^{1/2})
			Tan Delta	G''			
Control	None	Tactix 123	163	145	120	1.41	0.411 (25°C)
Udel P-1700 NT	10	Tactix 123	168	156	132	1.25	0.680 (25°C)
Udel P-1700 NT	20	Tactix 123	158	144	128	0.95	0.746 (25°C)
Radel A-200 NT	10	Tactix 123	177	162	141	1.38	0.549 (25°C)
Radel A-200 NT	20	Tactix 123	168	143	129	1.37	0.494 (25°C)
Ultem 1000-1000	10	Tactix 123	175	157	136	1.37	0.696 (25°C)
Ultem 1000-1000	20	Tactix 123	189	174	155	1.69	0.691 (25°C)
Orgasol 2001	20	Tactix 123	163	138	79	2.49	0.667 (25°C)
IMITEC X-902	10	Tactix 123	161	144	124	1.63	0.774 (25°C)
IMITEC X-902	20	Tactix 123	161	146	128	1.81	0.481 (25°C)
Udel P-1700 NT	20	ERL 2258	164		87		0.755 (25°C)
Udel P-1700 NT	11	Tactix 556/ ERL 2258 (44.5:44.5)	157		75		0.805 (25°C)
Udel P-1700 NT	10	Tactix 742/ ERL 2258 (45:45)	106		65		0.643 (25°C)

1. All formulations contained OPPI at a concentration of 3 phr.
2. All specimens were cured using a total dosage of 150 kGy.
3. Initial service temperature is defined as the temperature at which the modulus is at 50% of its value at 25°C.

Table 3. Toughening of Radiation Cured Epoxy Resins with Hydroxy-Terminated Polysulfone Oligomers^{1,2}

Hydroxy-Terminated Polysulfone Oligomer (Mn, g/mole)	Toughening Agent Concentration (%)	Epoxy Resin	Glass Transition Temperature (°C)		Initial Service Temperature ³ (°C)	Weight Gain (48h H ₂ O Boil) (%)	Fracture Toughness, K _{IC} (MPa·m ^{3/2})
			Tan Delta	G''			
Control	None	Tactix 123	163	145	120	1.41	0.411 (25°C)
1500	5	Tactix 123	173	160	137	1.06	0.508 (25°C)
1500	20	Tactix 123	155	143	132	1.21	0.536 (25°C)
1500	30	Tactix 123	160	145	120	2.56	0.557 (25°C)
2650	20	Tactix 123	149	134	122	1.57	0.700 (25°C)
5360	20	Tactix 123	150	134	123	2.38	0.731 (25°C)
6000	5	Tactix 123	171	156	130	1.15	0.475 (25°C)
6000	20	Tactix 123	159	150	135	1.26	0.593 (25°C)
6000	30	Tactix 123	161	149	134	1.64	0.705 (25°C)
8890	20	Tactix 123	154	136	122	1.77	0.738 (25°C)

1. All formulations contained OPPI at a concentration of 3 phr.
2. All specimens were cured using a total dosage of 150 kGy.
3. Initial service temperature is defined as the temperature at which the modulus is at 50% of its value at 25°C.

Table 4. Toughening of Radiation Cured Epoxy Resins with Reactive Flexibilizers^{1,2}

Toughening Agent	Toughening Agent Concentration (%)	Epoxy Resin	Glass Transition Temperature (°C)		Initial Service Temperature ³ (°C)	Weight Gain (48h H ₂ O Boil) (%)	Fracture Toughness, K _{IC} (MPa·m ^{1/2})
			Tan Delta	G''			
Control	None	Tactix 123	163	145	120	1.41	0.411 (25°C)
DER 736	10	Tactix 123					0.569 (25°C)
Heloxy 32	10	Tactix 123					0.487 (25°C)
Heloxy 68	10	Tactix 123	158	135			0.534 (25°C)
Heloxy 71	10	Tactix 123					0.529 (25°C)
Heloxy 505	10	Tactix 123					0.480 (25°C)
POLY bd 600	10	Tactix 123	166	151	123	1.28	0.594 (25°C)
Vikoflex 7010	10	Tactix 123	139	121	101		0.592 (25°C)
Vikoflex 7170	10	Tactix 123	151	133	117	2.02	0.598 (25°C)
Vikoflex 7170	20	Tactix 123	134		113		0.559 (25°)
Vikoflex 7190	10	Tactix 123	172		137		0.479 (25°C)
Vikolox 14	10	Tactix 123	130		112	1.12	0.491 (25°C)
Tone 301	10	Tactix 123					0.810 (25°C)
Vikoflex 7170	10	Tactix 556	175		150		0.555 (25°C)

1. All formulations contained OPPI at a concentration of 3 phr.
2. All specimens were cured using a total dosage of 150 kGy.
3. Initial service temperature is defined as the temperature at which the modulus is at 50% of its value at 25°C.

Table 5. Toughening of Radiation Cured Epoxy Resins with Rubbers and Elastomers^{1,2}

Toughening Agent	Toughening Agent Concentration (%)	Epoxy Resin	Glass Transition Temperature (°C)		Initial Service Temperature ³ (°C)	Weight Gain (48h H ₂ O Boil) (%)	Fracture Toughness, K _{IC} (MPa·m ^{1/2})
			Tan Delta	G"			
Control	None	Tactix 123	163	145	120	1.41	0.411 (25°C)
PY 322	20	Tactix 123	132	119	107	1.06	0.630 (25°C)
Dow XU 71790.04L	20	Tactix 123	166	151	121	2.00	0.565 (25°C)
Dow XU 71790.04L	30	Tactix 123/ Tactix 556 (28:42)	176	161	137	2.22	0.411 (25°C)
EXL-2330	10	Tactix 123	132	132	109	2.59	0.585 (25°C)
EXL-2330	15	Tactix 123	129	111	92	2.47	0.600 (25°C)
EXL-2330	20	Tactix 123	122	104	81	2.19	0.572 (25°C)
Epon 58134	10	Tactix 123	183		158		0.618 (25°C)
Kraton FG 1901X	10	Tactix 123	171		131		0.919 (25°C)
Kraton FG 1901X	20	Tactix 123	86/176		111		0.841 (25°C)

1. All formulations contained OPPI at a concentration of 3 phr.
2. All specimens were cured using a total dosage of 150 kGy.
3. Initial service temperature is defined as the temperature at which the modulus is at 50% of its value at 25°C.

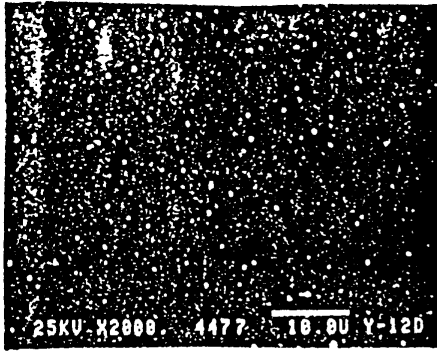


Figure 1



Figure 2



Figure 3



Figure 4

Figures 1-4. Scanning Electron Photomicrographs of: 1. Untoughened Tactix 123. 2. Tactix 123 toughened with 10% Udel P-1700. 3. Tactix 123 toughened with 10% Paraloid EXL-2330. 4. Tactix 123 toughened with 10% Kraton FG-1901X, respectively.