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MECHANICAL-PROPERTY CHANGES OF STRUCTURAL COMPOSITE MATERIALS AFTER LOW-TEMPERATURE PROTON IRRADIATION: IMPLICATIONS FOR USE IN SSC MAGNET SYSTEMS*

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INTRODUCTION

Long-term physical, mechanical, electrical, and other properties of advanced composites, plastics, and other polymer materials are greatly affected by high-energy proton, neutron, electron, and gamma radiation. The effects of high-energy particles on materials is a critical design parameter to consider when choosing polymeric structural, nonstructural, and elastomeric matrix resin systems. Polymer materials used for filled resins, laminates, seals, gaskets, coatings, insulation and other nonmetallic components must be chosen carefully, and reference data viewed with caution. Most reference data collected in the high-energy physics community to date reflects material property degradation using other than proton irradiations. In most instances, the data were collected for room- temperature irradiations, not 4.2 K or other cryogenic temperatures, and at doses less than 10^8-10^9 Rad. Energetic proton (and the accompanying spallation-product particles) provide good simulation fidelity to the expected radiation fields predicted for the cold-mass regions of the SSC magnets, especially the corrector magnets. We present here results for some structural composite materials which were part of a larger irradiation-characterization of polymeric materials for SSC applications.

EXPERIMENTAL

Specimens were supplied to BNL and SSCL by the vendors in either 2.5x.25x.25 or 2.5x.25x0.125 inch lengths. After characterization they were irradiated in liquid helium with 200-MeV protons to nominal doses of either 18^8 or 10^9 rad at the Brookhaven Radiation Effects Facility. The specimen temperatures during radiation did not rise above 20 K. They were then stored in liquid nitrogen until ready for mechanical testing at 4.2 K. Prior to the mechanical tests, the specimens were annealed at room temperature for one week. Standard ASTM short-beam-shear

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DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED tests were employed for the mechanical-property response to the irradiation. For experimental details, dosimetry, references, etc. for these and other materials in the program, see Refs. 1 and 2.

RESULTS

The data for the structural composites are presented in Table 1. The following Results and Observations are based upon these data.

Material #1, Specimen Group #1 (10^8 + 10^9 Rad): This thermoset composite is manufactured by Spaulding Composites. The standard deviation was low and constant, with the specimen color remaining the same after cold irradiation. Failure of the specimens was concentrated in the center with delamination and migration to the ends. Strength after 10^8 Rad increased 5%, and 13.5% after 10^9 Rad. It is reported that this material however is being modified by the original manufacturer as certain constituents of the formulation have been identified as "undesirable" for SSC and prime contractor applications.

Material #2, Specimen Group #2 and #3 (10^8 and 10^9 Rad): This thermoset composite material is manufactured by Allied Signal Corp. The "C" version indicates "cured" and the "P" version "post-cured". The purpose of including two (2) levels of cure was to see if strength would be affected following cold irradiation exposures. As discussed previously, proton irradiation like electron and other forms continue crosslinking of advanced composite materials further than generic materials which lose strength by degradation sooner. All specimens showed a slight darkening with failure concentrated in the center and migrating to ends. All standard deviations remained the same. The "C" version went up 5% after 10^8 Rad, the "P" version down 5% after 10^8 Rad, and up 5% after 10^9 Rad.

Material #3, Specimen Group #4 (10^9 Rad): This thermoset, a vinylester pultrusion is manufactured by Creative Pultrusion Corp. The specimens were cut like Material #3. Dark local discolorations were observed with multiple delaminations across specimens. Standard deviation stayed the same and strength dropped 25% after 10^9 exposures.

Material #4, Specimen Group #5 (10^8 + 10^9 Rad): This thermoset is manufactured by a convertor, Franklin Fiber Lamitex Corp. The "G-10" material specimens were cut from NEMA-grade laminate. As predicted the composite material strength went up 5% after 10^8 Rad, and dropped 58% after 10^9 Rad. All failures (single) were in the center of specimens. Standard deviation of 10^8-Rad specimens was 1/2 of unirradiated and 10^9 standard deviation was X2. Discoloration was localized for 10^8 Rad, and much darker in 10^9 samples.

Material #5, Specimen Group #6 (10^8 Rad): This thermoset is manufactured by Franklin Fiber also. Strength of this "G-11" material was the same after 10^8 Rad. A slight darkening was observed with multiple delaminations along the specimens. Standard deviation stayed the same.

Material #6, Specimen Group #7 (10^9 Rad): This thermoset composite is manufactured by Franklin Fiber also. It is a formulation of a highly crosslinked advanced-composite resin system. The strength went up 7% after testing. Fractures were not visible and standard deviation went up 0.2%. No color changes were recorded.

Material #7, Specimen Group #8 (10^9 Rad): This toughened thermoset composite is manufactured by Bryte Corporation. The mechanical strength decreased by 14% following the irradiation. No color change was recorded. Standard deviation went up .30 and failure was in center of specimens. Specimens were wet indicating a "leaching" of the toughening agent.

Material #8, Specimen Group #9 (10^9 Rad): This thermoset composite submitted as per Material #10. Failure of specimens in center. Standard deviation was up 0.43 without unirradiated test data.

OBSERVATIONS

The short-beam-shear test was used to screen all materials. Materials were selected from very generic thermoset chemistry categories like polyesters, vinylester to epoxies to phenolicotriazine, cyanate ester, polyimide and bismaleimide. Resin injection, resin transfer molding, liquid molding, compression, transfer, bulk molding compound, pultrusion, hand-lamination and other forms of thermoset materials and processes were considered during the selection process. Cost of materials and cost to process was also an important criterion. As always, safety and environmental issues entered into the selection process.

The high-energy-physics community industry standard has been "G-10". It is known that because of the curing agent or hardener system used, the radiation resistance at high levels (10^8-10^9 Rads) will be poor. Also the mechanical and physical properties of the material exceed the requirements for some SSC applications. So it is suggested that instead of machining "G-10" for a non-structural application, one might select a more radiation-resistant material that can be "liquid molded", poured, or resin injected. Such applications include stand-offs, insulators, supports, etc.

Table 1. Structural Composites

Specimen Group	Manufacturer of Material	Type	Color Change	Strength After Cold Irradiation* (CI)	KSI After (CI)	Sample Condition after (CI)
1	Spaulding	Spaulrad	Same	Up 5% after 10^8	9.2	Failure in center and
			Same	Up 13.5% after 10^9	10.0	Migrates to sample end
2	Allied S.	Cryorad "C"	Slight darken	Up 4% after 10^8	4.8	Migrates to sample end
3	Allied S.	Cryorad "P"	Slight darken	Down 5% after 10^8	3.9	Failure in center and
			Same	Up 5% after 10^9	4.3	Migrates to sample end
4	Creative Pultrusions	1625	Local discolor, dark	Down 25% after 10^9	4.4	Multiple delams
5	Franklin Fiber	G10CR	Local discolor, dark	Down 58% after 10^9	20.8	Single failure in center of all samples
			Slight darken	Same after 10^8	8.3	
6	Franklin Fiber	G11	Slight darken	Same after 10^8	9.7	Multiple delams
7	Franklin Fiber	221CR		Up 7% after 10^9**	5.2	Fractures not visible
8	Bryte	EX1524	NA	Down 14% after 10^9**	8.1	Single (wet leak) failure in center
9	Permaglas	TE630		10^9 (No unirr.)	13.4	Single failure in center

*All mechanical tests are short-beam shear.

**Unirradiated baseline data supplied by CTD.

The "structural composites" testing section of the program also proved that most of the more highly crosslinked polymeric and composite materials continued to get stronger (continued crosslinking). The more generic non-aromatic cured (aliphatic) lost strength and this is an indication that molecular bonds were broken or severed. In all cases except "G-10" the loss was less than 50%. If we use the same selection criterion as "adhesive systems" and "filled and unfilled resin systems", we could select the materials tested for applications and radiation zones where we know they will not degrade greater than a certain amount. In other words, if we plan to use a material as a "neat" polymer or composite in a known radiation environment, we can select the appropriate material for that radiation zone.

J. Morena is preparing a design guide[3] that will be available for circulation shortly. The Design Guide will list all common and known nonmetallic materials along with their radiation-resistance levels. This Guide should assist with the selection of composite and polymeric materials for specific radiation-environment applications.

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The information analyzed and reported herein is based upon the estimated performance of a material as it is envisioned for use in actual production applications and environments. It should be noted, however, that ACE, Inc., ACMLC, Inc., John J. Morena, BNL, SSCL and others involved in the subject screening and testing program assume no responsibility for the interpretation or misrepresentation of any data, information, or analysis resulting from the reported results of these tests.

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