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Evaluation of ionic liquid epoxy carbon fiber composites in a cryogenic environment

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ABSTRACT

A novel ionic liquid epoxy (ILE) was used to fabricate carbon fiber composite discs which were then subjected to biaxial strain testing in liquid nitrogen. The ILE composite showed a greater strain-to-failure at cryogenic temperatures when compared to a commercial epoxy. This result is likely an effect, as shown in micrographs, of the strong ILE bonding with the carbon fibers as well as it exhibiting plastic deformation at the fracture surface.

Introduction

Material properties, such as strength to weight ratio and high stiffness, make carbon-fiber composites attractive for space applications such as tanks for cryogenic liquid containment. This work focuses on the problem of the fiber/epoxy interface where mismatch at cryogenic temperatures can lead to detrimental microcracking and leaking [1–3]. Here a novel epoxy, 1,3-bis(glycidyl) imidazolium bis((trifluoromethyl)sulfonyl)imide [4] is assessed for potential in making carbon fiber composites for cryogenic applications. Unlike commercial bisphenol based epoxy monomers, the ionic liquid resin is based on a unique heterocyclic imidazolium cation with a large coordinated anion. Systematic development and testing [5,6] of the ionic liquid epoxy (ILE) has continually shown improved cryogenic properties when compared to commercial counterparts, the implication being that a carbon-fiber composite material could be fabricated and subjected to mechanical cycling at cryogenic temperatures without loss of strength or fatigue failure. Here particular attention is paid to failure modes such as temperature induced embrittlement of the epoxy matrix, microcracking, and adhesion between the epoxy and fibers. Comparison with a traditional/commercial epoxy is made to illustrate the observed differences in behavior.

Experimental procedure

The composite samples consisted of four layers of carbon fiber fabric, Hexcel® T300 style 824 with 1000 unsized fibers per tow and a plain weave pattern, stacked in a 0/0/0/0 ply orientation. Commercial layups were prepared using Epon® 828 epoxy resin with Huntsman® hardening agent (100:42 mix ratio). Biaxial strain gages were then placed on the top layer of fabric and aligned with the fibers. The epoxy-soaked fabric and strain gages were then vacuum bagged and cured at a temperature of 100 °C for two hours. The IL was mixed with the hardening agent, APB-N 1,3-bis-(3-aminophenoxy)benzene, (2:1 mix ratio) and cured at a temperature of 150 °C for three hours. The final cured composite sample thickness was approximately 0.58 mm. The flat composite sheets, with the strain gages centered, were then fashioned to 76 mm diameter discs.

Ball-on-ring biaxial strain testing, typically applied to ceramics [7], was used to evaluate the composite discs. Output from the biaxial strain gages was recorded using a reader, model 8000-8-SM by Micro-Measurements®, connected to a PC. Testing involved placing a prepared disc into the holder beneath the suspended ball. For cryogenic testing the apparatus was put in a stainless steel dewer which was filled with liquid nitrogen (LN2) to well cover the composite disc. Once thermal equilibrium was achieved, as determined by the strain gages, the readings were zeroed and the ball was pressed onto the disc. The hydraulic pressure load

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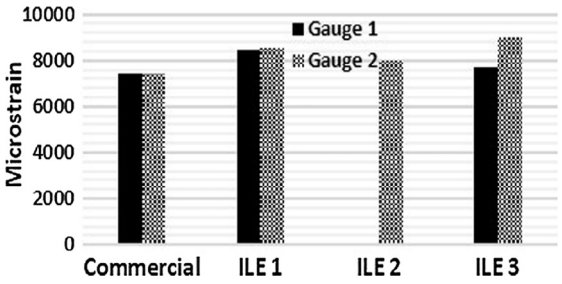

Fig. 1. Biaxial strain at failure in LN2.

was systematically increased with data recorded until disc failure, a severe step change of deflection accompanied by a loud percussive bang. This culminated in a clearly visible crack extending radially from the center to the edge of the disc. The resultant fractures were examined using scanning electron microscopy (SEM).

Results

In LN2, the commercial epoxy composite disc failed at 7411 microstrain, while the IL epoxy composite discs failed at an average of 8344 microstrain, Fig. 1. This is a ~12.6% improvement over the commercial epoxy.

Discussion

Insight to the measured differences can be found by comparing SEM micrographs of the fractured samples. Figs. 2a, b and 3a, b compare, respectively, fracture surfaces of the commercial and ILE composites. A significant difference in fiber adhesion by the

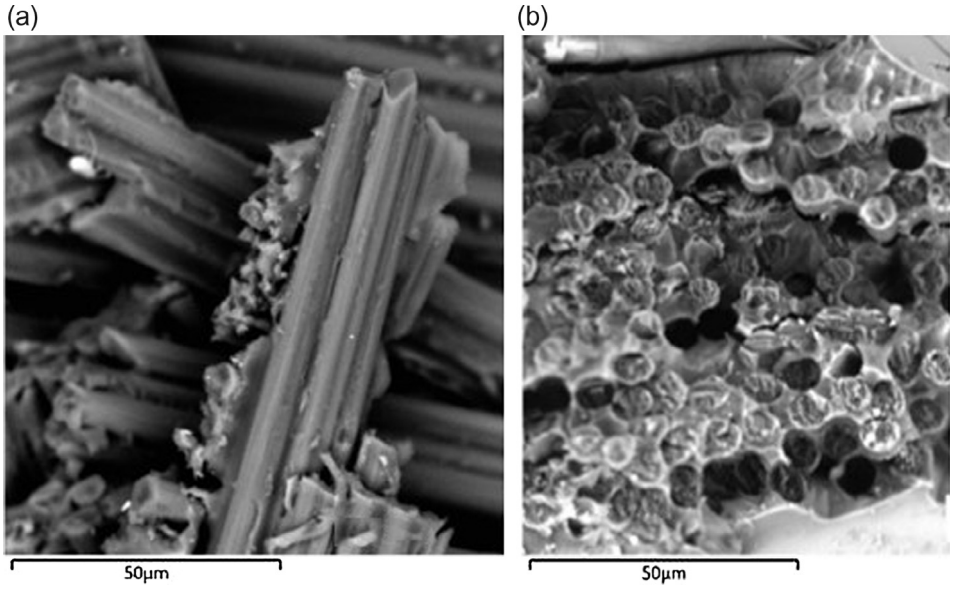

Fig. 2. Fracture surfaces of the commercial epoxy composite.

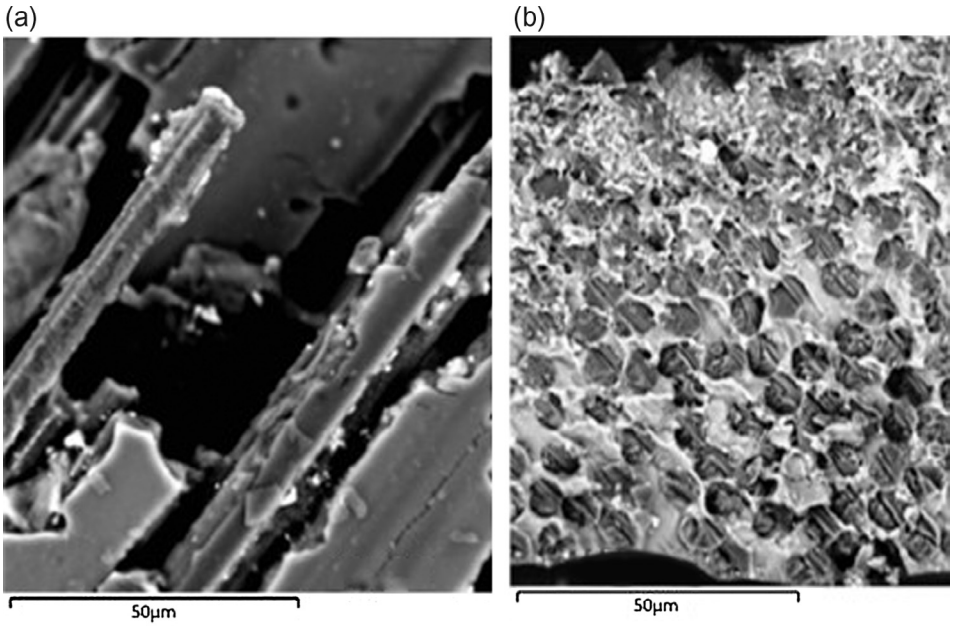

Fig. 3. Fracture surfaces of the ionic liquid epoxy composite.

two epoxies is seen. Little, if any, of the commercial epoxy is seen adhering to the carbon fibers, Fig. 2a; the fracture path is interfacial between the fiber and the epoxy. In Fig. 3a the ILE is seen adhering to the fibers and the fracture path goes through the epoxy.

In cross-section the commercial composite shows considerable fiber pullout, Fig. 2b, again indicating poor interfacial adhesion; this is not seen in the ILE composite, Fig. 3b. Secondly, and not immediately obvious, the commercial epoxy exhibits a glassy-type fracture while the ILE appears to show plastic deformation with multiple tears between the fibers. This yielding indicates the ILE remained ductile, lengthening the fracture path and absorbing more energy at cryogenic temperatures, important for toughness and preventing microcracking. Differential strains developing between fiber and matrix at cryogenic temperatures are thus lower and less likely to contribute to debonding at the fiber interface. These test results and observed microstructures, despite the small sample base, trend with enhanced cryogenic material properties seen in our previous ILE studies (5–6).

Conclusions

Biaxial strain testing demonstrated that ionic liquid epoxy makes tough, high strength composite materials that perform well at cryogenic temperatures. This is attributed to a combination of bonding between the carbon fibers and ILE and the ILE matrix maintaining ductility at LN2 temperatures. These two attributes will abate microcracking at cryogenic temperatures under nominal load conditions as well as in cases where a tank is cycled between cryogenic and room temperatures

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