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ACCEPTED MANUSCRIPT

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.

Keywords

Ionic liquid epoxy; carbon fiber composite, biaxial strain testing, cryogenic fluids 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 dertrimental 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 1,000 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 0.58mm. The flat composite sheets, with the strain gages centered, were then fashioned to 76mm 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 was systematically increased with data recorded until disk 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 disk. 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, Figure 1. This is a ~12.6% improvement over the commercial epoxy.

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