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Abstract

The present study investigates the mixed mode interlaminar fracture toughness of six polymer matrix composite materials at both room temperature and cryogenic liquid nitrogen temperature. The single leg bending test employed gives a mix of Mode I (opening) and Mode II (in-plane shearing) cracking. The tested thermoplastic-matrix composites were shown to have total mixed mode toughnesses 3.1–4.3 times higher than those of representative thermoset resin composites.

1. Introduction

Knowledge of the damage resistance and damage tolerance of polymeric composites is essential to the design of aerospace structures from these materials [1]. Cryotanks are one example [2-5].

From a materials standpoint, thermoplastic matrix resins provide new automated manufacturing opportunities, moisture resistance, as well as much greater toughness, at least at room temperature [6]. Oliver and Johnson [7] compared Mode I fracture of IM7/PETI-5 and IM7/977-2 at temperatures ranging from -196 °C to 160 °C and found the IM7/PETI-5 system had higher Mode I interlaminar fracture toughness (G_{IC}) at all temperatures, and may have slightly increased G_{IC} at -196 °C relative to ambient temperature.

Hohe *et al.* [8] and Chen *et al.* [9] provide reviews of composites for cryogenic applications, including both thermoset and thermoplastic systems. Grogan *et al.* characterized microcracking and other damage from cryogenic cycling of polyether ether ketone (PEEK) composites with IM7 and AS4 fibers [10]. Flanagan et al. also studied PEEK composites with AS4 and IM7 fibers and investigated their permeability for cryogenic applications [11].

In the present study, the mixed Mode I – Mode II interlaminar fracture toughness of six materials was evaluated at both room temperature and cryogenic liquid nitrogen (LN_2) temperature. Four thermoplastic composite materials and two state-of-the-art thermoset composite materials were included. The single leg bending (SLB) test [12] was used since its fixturing has no moving parts and samples can be fully submerged in LN_2.

2. Experimental

2.1 Materials

Four thermoplastic composite materials and two thermoset composite materials were selected as shown in Table 1.

Table 1. Materials tested in this study.

Material ID	Description
Epoxy 8552/IM7	Baseline toughened epoxy resin (Hexcel® Hexply® 8552 epoxy) matrix with Hexcel® Hextow® IM7 carbon fiber
Epoxy 5320-1/IM7	Baseline toughened epoxy resin (Syensqo Cycom® 5320-1 epoxy) matrix with Hexcel® Hextow® IM7 carbon fiber
PEEK/AS4	Semicrystalline thermoplastic PEEK resin (Toray Cetex® TC1200) matrix with Hexcel® Hextow AS4 carbon fiber
LM-PAEK/T800	Semicrystalline thermoplastic low-melt polyaryletherketone (LM-PAEK) resin (Toray Cetex® TC1225) matrix with Torayca™ T800 carbon fiber
PEKK/AS4	Semicrystalline thermoplastic polyetherketoneketone (PEKK) resin [Syensqo APC (PEKK)] matrix with Hexcel® Hextow® AS4 carbon fiber
PEI/AS4	Amorphous thermoplastic polyetherimide (PEI) resin [Syensqo APC (PEI)] matrix with Hexcel® Hextow® AS4 carbon fiber

2.2 Panel Fabrication

For prepreg tapes that were 305 mm (12 inch) wide, plies were simply laid between two 305 mm × 305 mm caul plates. Materials that were not the full 305 mm width were stacked so that seams where tapes met would not align from layer to layer. The panels were approximately 5 mm thick with a film crack starter 102 mm (4 inch) wide at one edge at the mid-plane. That insert was Kapton® polyimide (for thermoplastic composites) or Teflon™ (for thermoset composites). Each finished layup was then processed via autoclave according to manufacturer recommendations.

2.3 Sample Preparation

Fabricated panels underwent ultrasonic C-scans. In a few cases, porosity was observed; areas with porosity were not used for testing. Coupons for SLB testing were cut 23 mm (0.9 inch) wide and 279 mm (11 inch) in the fiber direction. The SLB method in cryogenic testing is not standardized yet because the testing is not a univeral industrial need, but these dimensions match those of the End-Notched Flexure test standardized as ASTM D7905 [13]. Samples were rinsed with water to remove debris from the wet saw cutting, dried in a vacuum oven at 90°C at low pressure for 24 hours and stored in a dry environment until tested. The thickness and width of the first, sixth, and eleventh coupons were measured at three locations along the length of the coupon with a micrometer (Mitutoyo 389-714) and averages were tabulated.

2.4 SLB Test Method

The test utilizes a beam-like specimen with two arms on one end of the beam and a crack length a, as shown in Figure 1. The test protocol mimics that in ASTM D7905, end-notched flexure.

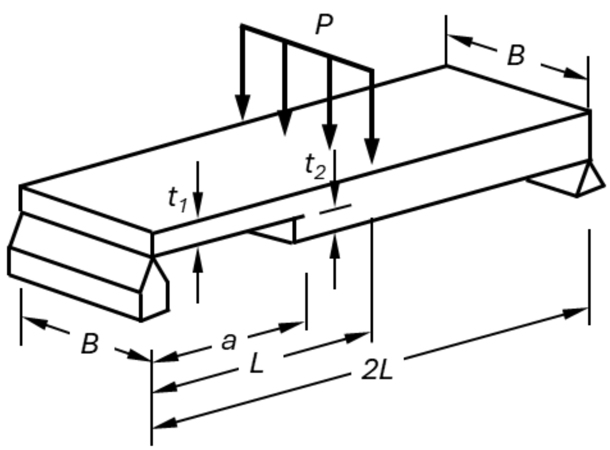

Figure 1. Schematic of SLB test and testing coupon.

The ratio of the mode II interlaminar sliding sheare, G_{II}, to the total strain energy release rate of mode I and mode II, G_T (where $G_T = G_I + G_{II}$), is determiend from the SLB test. In this study, where cracking occurred along the mid-plane, the mixed-mode ratio, G_{II}/G_T was 0.40 [14].

Samples were loaded in displacement control, with a loading rate of 0.5 mm/min (0.02 inch/min) and an unloading rate of 1.3 mm/min (0.05 inch/min). Force-displacement data were recorded throughout the duration of the tests. The specimens were pre-cracked by loading at a location 30 mm (1.2 inch) beyond the insert. The length of the resulting pre-crack was measured from an ultrasonic C-scan of the specimen. An average of five positions along the crack front were used. For the actual test, the load was applied 30 mm beyond the pre-crack. Prior to the fracture test, specimen compliance was determined by loading at two other locations relative to the pre-crack. Loads for these compliance calibration runs were limited such that the applied strain energy did not exceed 40% of the fracture value. Then, the three compliances were fitted to an empirical expression:

$$C = A + ma^3 \quad (1)$$

Where C is compliance, A is the intecept and m is the slope obtained from the regression analysis.

Finally, the total strain energy release rate, G_T was calculated from the expression

$$G_T = 3mP^2a^2/2B \quad (2)$$

where B is the average width of the specimen, and P is the peak load during the fracture test. The effective crack length, a, employed in this equation is back-calculated using Equation 1.

In several cases, specimens could be re-used for another increment of crack growth. Tests were conducted at room temperature (21–23°C or 70–74°F) or immersed in liquid nitrogen (-196°C or -321°F) (Figure 2).

2.5 Sample Preparation

Samples exhibiting fracture toughness values closest to the average for the material and condition were selected for optical microscopy. The edge of each SLB sample was polished for ten minutes to remove the white paint used for the markings during mechanical testing.

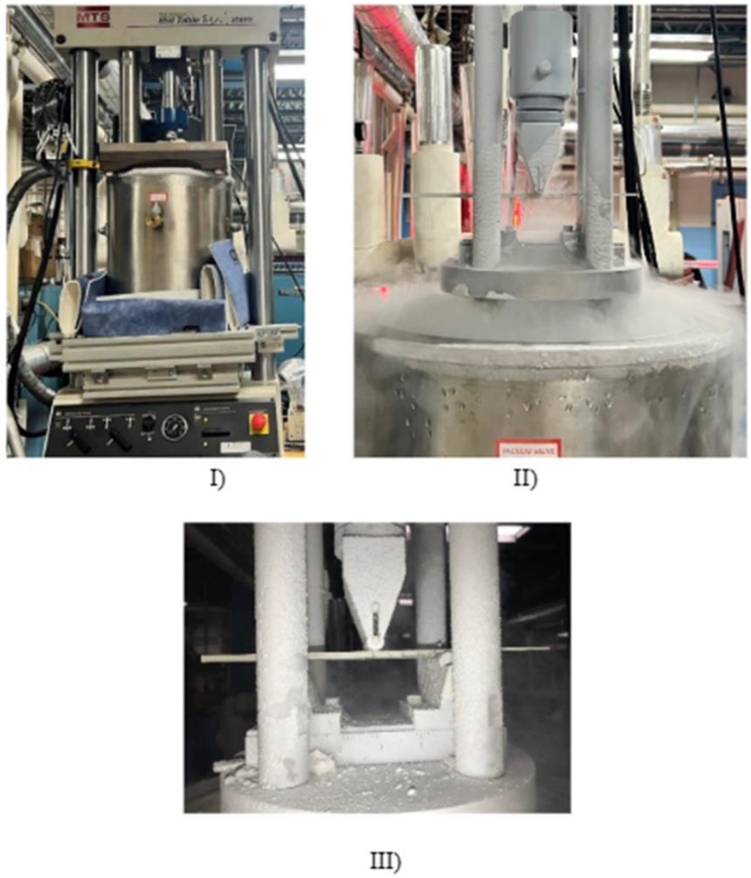

Figure 2. SLB cryogenic temperature test setup. (I) The sample was submerged in LN_2 for testing. (II–III) The sample was removed from LN_2 tank after testing.

3. Results and Discussion

3.1 Mixed Mode I – Mode II Fracture Toughness

The testing conducted here allows two sets of comparisons: thermoset vs. thermoplastic and room temperature vs. liquid nitrogen.

It has been previously shown that (Mode I) composite interlaminar toughness tracks with resin toughness [6]. Our SLB testing (Figure 3) reveals that the thermoplastic composites show mixed mode fracture toughnesses approximately 3.1–4.3 times higher than the thermosets. This should translate to better damage resistance and damage tolerance in structures. The LM-PAEK composite exhibited the highest mixed Mode I – Mode II fracture toughness among the materials tested.

Somewhat surprisingly, all six of the tested materials retain their toughness at LN2 temperature as shown in Figure 3 (and elaborated in the Appendix).

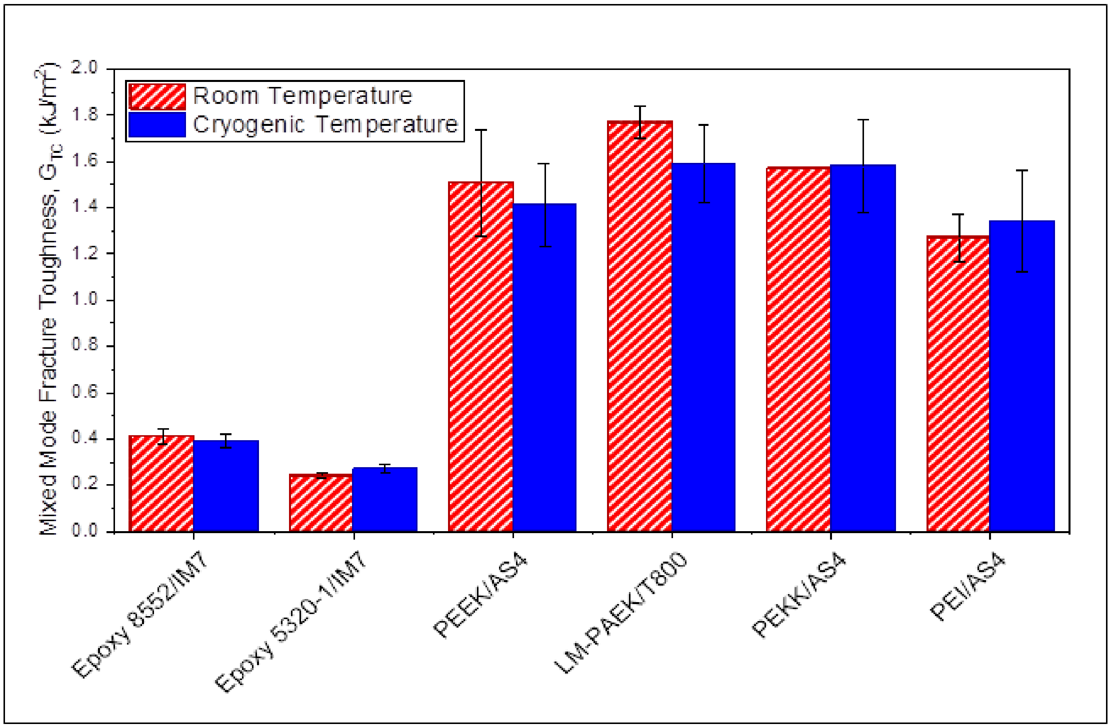

Figure 3. Mixed Mode I – Mode II fracture toughness of the tested polymer composites.

To understand this, the interlaminar fracture was further investigated. Some raw data are shown in Figure 4. In both cases (thermoset and thermoplastic), the load drop appears more acute at the cryogenic temperature, as might be expected if the resin were behaving in a more brittle manner. The G_T calculation, however, is based on the maximum load irrespective of the shape of the curve.

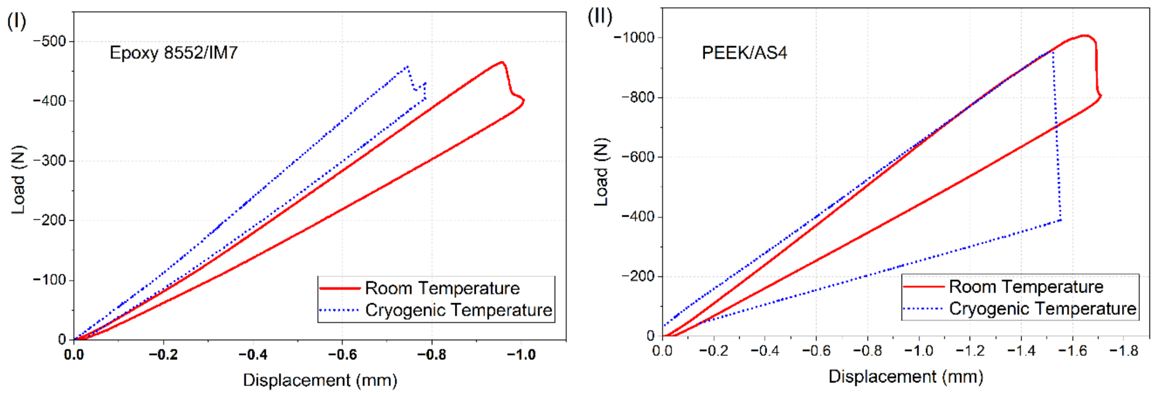

Figure 4. Load-displacement plot of (I) epoxy 8552/IM7 and (II) PEEK/AS4 tested at room temperature and cryogenic temperature.

Figure 5 and Figure 6 compare the appearances of some of the interlaminar cracks as viewed from the specimen edge. As shown in Figure 5, cracking in Epoxy 8552/IM7 seemed to follow a relatively straight path with minimal evidence of plastic deformation. This is consistent with a cross-linked molecular structure. The LM-PAEK composite shown in Figure 6 exhibits evidence of a resin-rich region between plies and some topography indicative of ductile fracture, especially at room temperature.

Interlaminar fracture depends on the resin toughness (modulated by constraint effects), and on the fiber/matrix interface and the loading mode. Considering first the resin, the respective crystallinities of each of the thermoplastics are probably determined during processing and are unlikely to change. We observe that even at room temperature, all the resins are deep in the glassy phase and find, like Oliver and Johnson [7], that further cooling may have little effect.

With regard to the interphase, it is at least possible that resin shrinkage below room temperature could improve contact with the fibers in a unidirectional layup.

Finally, it is important to note that in Mode II delamination fracture, the first step may be formation of tensile cracks [15]. It is highly likely that the resin tensile modulus and strength are substantially increased at the low temperature. Nikonovich et al. [16] actually noted an increase in impact strength for pure PEEK in the beta-transition region (-195 °C).

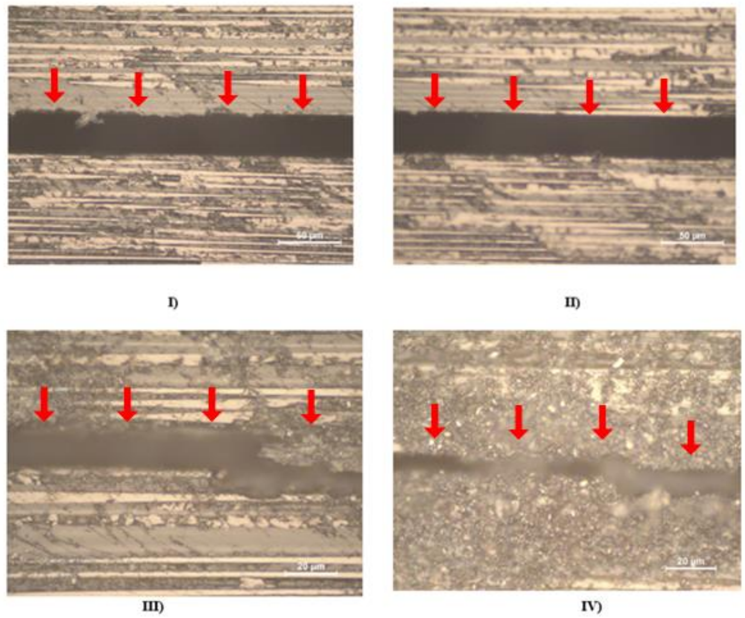

Figure 5. Crack apperance for Epoxy 8552/IM7 after (I, II) room temperature testing and (III, IV) after cyrogneic testing (LN_2).

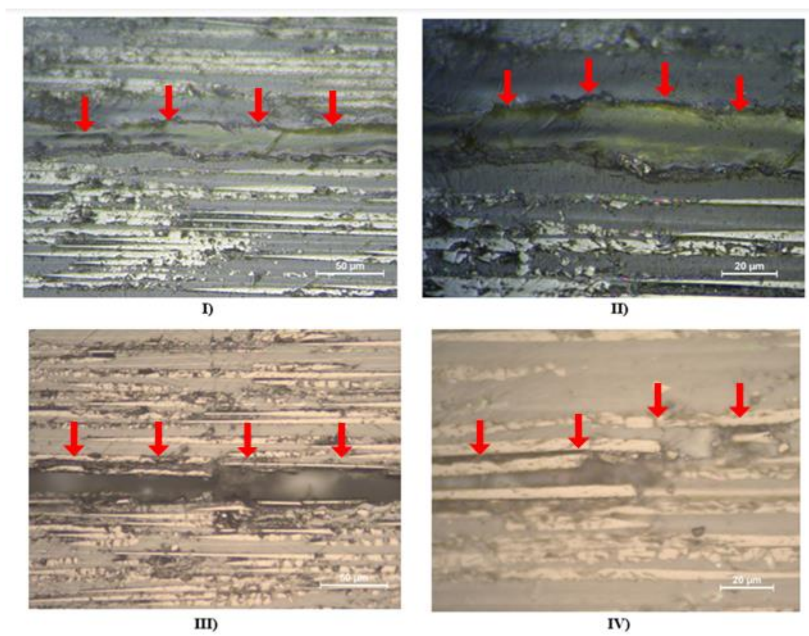

Figure 6. Crack apperance for LM-PAEK/T800 after (I, II) room temperature testing and (III, IV) after cryogenic testing (LN_2).

4. Conclusion

The SLB mixed-mode test was shown to be suitable for use under cryogen immersion. The four thermoplastic composites evaluated in this study outperformed two state-of-the-art thermoset composites in fracture toughness. All six materials were shown to have cryogenic mixed-mode toughnesses comparable to the room temperature values. The higher cryogenic toughness of thermoplastic composites provides improved damage resistance and damage tolerence in cryogenic structural applications, along with the potential fabrication advantages of thermoplastics, such as unlimited out-time and applicability of advanced tape-laying technique. These findings, used along with permeability, fatigue, and other measurements, are helpful in understanding materials for cryotank and other aerospace applications.

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6. Appendix

Looking at Figure 3, of the six materials, three have a higher toughness at LN_2 temperature, and for three, toughness at LN_2 temperature is lower. Although the differences appear to fall within the standard deviation, it was of interest to perform a quantitative analysis.

In order to compare the means of two samples that have different variances – there is no reason to think the two conditions lead to identical variances, and for several of the thermoplastics, the observed variances differ by severalfold – the appropriate test would seem to be Welch's t-test. Table A1 shows the statistic and two-tailed probability (against the null hypothesis that for each material, there is no difference between the toughness at room temperature and that at the cryogenic condition). As anticipated, the differences are not statistically significant. Note that PEKK/AS4 was excluded from this analysis due to the limited number of room temperature specimens.

Given the limited size of the data set, it should not be considered "safe" to assume that cryogenic data are not needed, and other materials may behave differently.

Table A1. Mixed Mode I – Mode II fracture toughness of thermoplastic and thermoset polymer composites.

Material	Room Temperature		Cryogenic Temperature		Difference	
	G_{TC}, kJ/m^2 (in-lb/in²)	Number of specimens	G_{TC}, kJ/m^2 (in-lb/in²)	Number of specimens	t-statistic	p
Epoxy 8552/IM7	0.41 ± 0.02 (2.33 ± 0.14)	5	0.39 ± 0.03 (2.21 ± 0.15)	4	1.23	>0.3
Epoxy 5320-1/IM7	0.24 ± 0.01 (1.37 ± 0.08)	5	0.27 ± 0.02 (1.52 ± 0.10)	5	2.62	>0.05
PEEK/AS4	1.51 ± 0.22 (8.61 ± 1.28)	5	1.41 ± 0.18 (8.07 ± 1.02)	4	0.70	>0.5
LM-PAEK/T800	1.77 ± 0.06	5	1.59 ± 0.17	5	2.18	>0.05

	(10.10 ± 0.37)		(9.07 ± 0.99)			
PEKK/AS4	1.57 (8.94)	1	1.58 ± 0.20 (9.02 ± 1.14)	6	--	--
PEI/AS4	1.26 ± 0.10 (7.22 ± 0.59)	5	1.34 ± 0.22 (7.65 ± 1.27)	5	0.69	>0.5