

## **Loading Rate Sensitivity of Glass Fiber-epoxy Composite at Ambient and Sub-ambient Temperatures**

**B.C. Ray**

Department of Metallurgical and Materials Engineering, National Institute of Technology, Rourkela- 769008, India

### **ABSTRACT**

Little information regarding the effects of weight fraction of constituent phases of freeze-thaw response in polymer composites at different loading rates and temperatures has been published to date. The present experimental study uses 3-point flexural test to qualitatively assess such effects for 55, 60 and 65 weight percentages of E-glass fibers reinforced epoxy composites during cryogenic and after thawing conditions. The specimens were tested at a range of 0.5 mm/min to 500 mm/min crosshead speed to evaluate the sensitivity of mechanical response during loading at ambient and sub-ambient (-80°C temperature). These shear strength values are compared with the testing data of as-cured samples. The findings are explainable at lower range of crosshead speed. The data obtained at higher loading rate suggest further investigation to establish the nature.

**KEY WORDS:** epoxy, composites, weight fraction, cryogenic temperature, loading rate, shear strength

## INTRODUCTION

Different loading conditions are probable in many of the applications where fiber-reinforced polymer (FRP) composites find use as potential and promising materials. Damage and failure on a small scale in cryogenic liquid-storage systems is expected to grow and spread with freeze–thaw cyclic and it may eventually cause bulk failure in structure that were not designed to account for cryogenic microcracking and residual stresses induced by thermal shock and thermal cycling [1]. The effect of varying loading rate on mechanical properties of fiber-reinforced polymer composites has been investigated and reported a variety of contradictory observations and conclusions [2]. The heterogeneous nature polymer composite that the local microstructure near the crack tip plays an important role in the crack blunting phenomena [3]. The polymer composites are characterized by a greater level of microcracking and delamination at sub-zero temperature because of higher residual thermal stresses [4]. The freeze-thaw exposure can result in significant changes in thermo mechanical response of polymer composite [5]. Brittle thermoset epoxy resin can undergo a limited extent of deformation prior to failure. The ductility of a matrix resin may become a limiting factor at high strain rate for composite strength. Epoxy resin is more ductile than its composite at low strain rate [6]. Delamination and microcracking are some of the most frequently observed damage phenomena that can develop in polymer composites when they are subjected to cryogenic conditioning [7]. A very large thermal expansion mismatch can result in debonding at the fiber/matrix interface and/or a possible matrix cracking due to thermal stress [8]. The fiber/matrix interfacial behavior is based on

mechanical principles with the assumptions made at either the level of fiber/matrix adhesion or using the surface chemistry phenomena [9]. Epoxy resin and E-glass fiber are reported to be loading rate sensitive [10]. The use of polymer composites in safety-critical applications leads to uneasiness since the mechanical response is not well established in freeze-thaw condition in different load bearing applications. The present work aims to study the role of interface in freeze-thaw response at different loading rates and also by considering the weight fraction effect of constituent phases of glass/epoxy composite.

## **EXPERIMENTAL**

Araldite LY-556, an unmodified epoxy resin based on Bisphenol-A and hardener (Ciba-Geig, India) HY-951, aliphatic primary amine were used with woven roving E-glass fibers treated with silane based sizing system (Saint-Gobain Vetrotex) to fabricate the laminated composites. Three fiber weight percentages 55, 60 and 65% were targeted in the laminate fabrication. They were cured for 48 hours at room temperature. The laminates were cut into short beam shear (SBS) test specimens by diamond cutter. The SBS 3-point bend tests were conducted to determine the interlaminar shear strength (ILSS) of composites. The test specimens were suddenly exposed to  $-80^{\circ}\text{C}$  temperature in a ultra low freezing chamber for 2 hours to attain the temperature of freezer. One batch of treated samples was tested in 3-point bend flexural mode almost at that temperature. The other batch was allowed to thaw ambient temperature ( $30^{\circ}\text{C}$  temperature) for 1 hour. The SBS bend test was carried out on the freeze-thaw conditioned specimen at ambient

temperature. The mechanical flexural tests were performed at 0.5, 2, 10, 50, 100, 200 and 500 mm/min crosshead speeds. The untreated as-cured composite specimens were also tested in a 3-point bend test at ambient temperature with those crosshead speeds. All samples were kept in desiccators for several hours to stabilize the variation in weight before freeze-thaw conditioning. The same procedure was followed for untreated specimens. An Instron1195 tensile testing machine was used to perform SBS tests in accordance with ASTM D 2344-84 standard. Multiple samples were tested at each point of experiment and the average value was reported.

## **RESULTS AND DISCUSSION**

The effects of different crosshead speeds on ILSS value at  $-80^{\circ}\text{C}$  temperature ( $\blacklozenge$ ), at room temperature after thawing ( $\blacksquare$ ) and at room temperature of untreated samples ( $\bullet$ ) of glass-epoxy laminates (fiber = 0.55 weight fraction) shown in Fig.1. The loading speed sensitivity is clearly evident upto 50 mm/min crosshead speed. The variation of ILSS value with crosshead speed for all three conditions of glass/epoxy composite (fiber = 0.6 weight fraction) is drawn in Fig.2. Figure 3 corresponds to the variation of shear strength with crosshead speed for 65% weight percentage fiber reinforced epoxy composites. A common observation is noticed for all weight fraction reinforcement phase is that laminates show higher ILSS value with more crosshead speed upto 50 mm/min for three situations and shear value drops with more loading speed. The localized failure depends on the local microstructure and defect concentration. The microstructure of polymer matrix is

influenced by temperature. Cryogenic microcracking may induce further defect in the matrix and/or interface. The onset of microcracking was detected from acoustic emission and it was further confirmed by microscopic examination of polished specimen edges [11]. The restriction of not breaking covalent bonds during deformation means that polymers are able to undergo only a limited amount of plastic deformation [12]. It is generally believed that the polymer is likely to be tough if homogeneous yielding does occur. Unfortunately, failure mechanisms in polymer composites in presence of different residual stresses tend to be localized and produce inhomogeneous plastic deformation. The microcrack density under the influence of freeze-thaw condition will be increased to a threshold value where stress redistribution may limit the initiation of new crack. Then matrix cracking becomes a macroscopic form of prevalent damage and it eventually lead to delamination failure of composite. The loading rate sensitivity at higher crosshead speed here appears to be complex nature of mixed fracture modes. This needs to be verified by a systematic study. Intermolecular forces and stress relaxation at the crack tip of an epoxy resin at cryogenic temperature is found to play an important role for higher fracture toughness [13]. Thermal contraction at the bulk epoxy matrix at low temperature may induce residual stresses at the fiber/polymer interface. The high and complex nature of residual stresses at cryogenic temperature and also at ambient temperature after thawing may possibly result in larger debonded interfaces. The specimens which were tested at  $-80^{\circ}\text{C}$  temperature experienced down-thermal shock (ambient to cryogenic temperature). The up-thermal shock (cryogenic to ambient temperature) of  $110^{\circ}\text{C}$  temperature gradient

was experienced by glass/epoxy laminates during thawing by ambient environment. Higher crosshead speed during testing restricts and/or minimizes the relaxation processes at the crack tip. Thermal stress-induced cracks may possibly grow without blunting at a steady rate. That could reduce the interlaminar strength at higher loading rate.

## **CONCLUSIONS**

It is reasonable to conclude that the cryogenic hardening is evident at lower range of crosshead speed. The effects result in higher ILSS values. Cryogenic compressive residual stresses are not nullified during thawing and results in higher ILSS value in compared to the non-treated samples. The ILSS values start decreasing at higher loading rate for all three situations. It may possibly be attributed to the less prevalent relaxation process at the crack tip. The crack blunting may happen to be less common occurrence at higher rate of loading. The nature of variations of ILSS value with the crosshead speed is found to be of almost same pattern for different weight fractions reinforcement phase in glass-epoxy composites.

## **ACKNOWLEDGEMENTS**

The fund for the study from MHRD Project, Govt. of India is sincerely acknowledged.

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## Figure Captions

- Figure 1** Variation of ILSS of glass-epoxy composites (fiber=0.55 weight fraction) with crosshead speed at ambient temperature (●), after thawing at ambient temperature (■), and cryogenic temperature (◆).
- Figure 2** Variation of ILSS of glass-epoxy composites (fiber=0.60 weight fraction) with crosshead speed at room temperature (●), after thawing at ambient temperature (■), and cryogenic temperature (◆).
- Figure 3** Variation of ILSS of glass-epoxy composites (fiber=0.65 weight fraction) with crosshead speed at room temperature (●), after thawing at ambient temperature (■), and cryogenic temperature (◆).



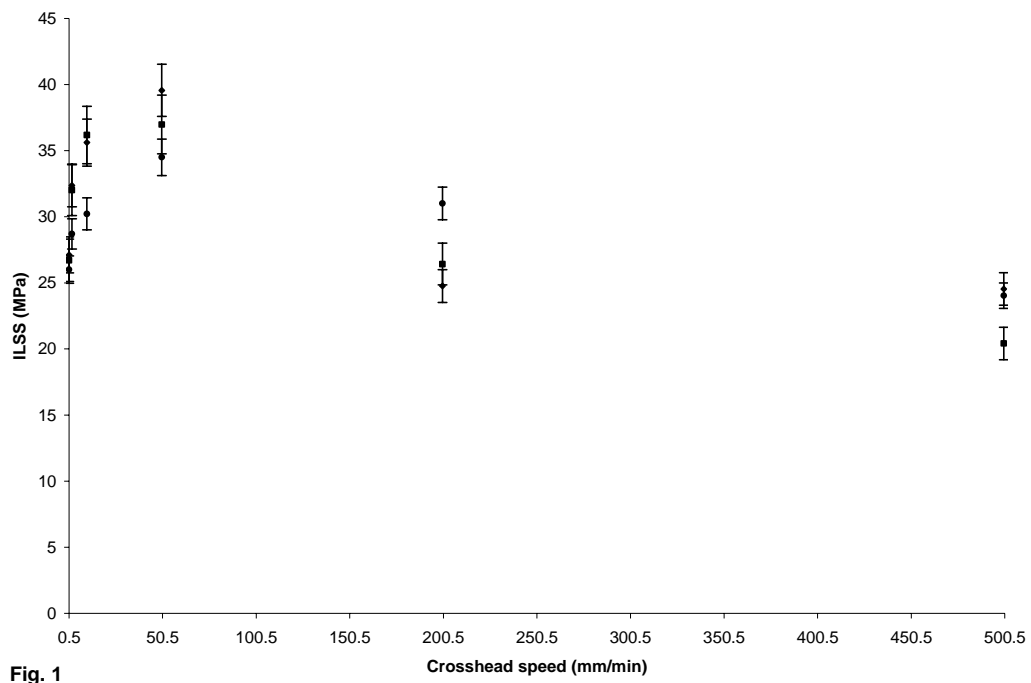


Fig. 1

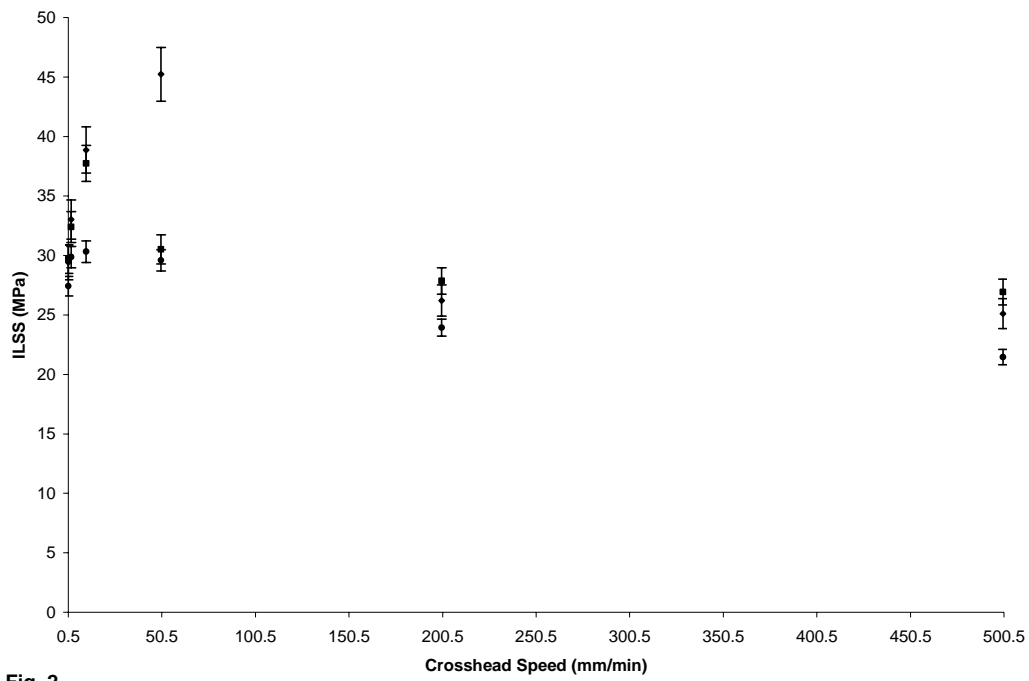


Fig. 2

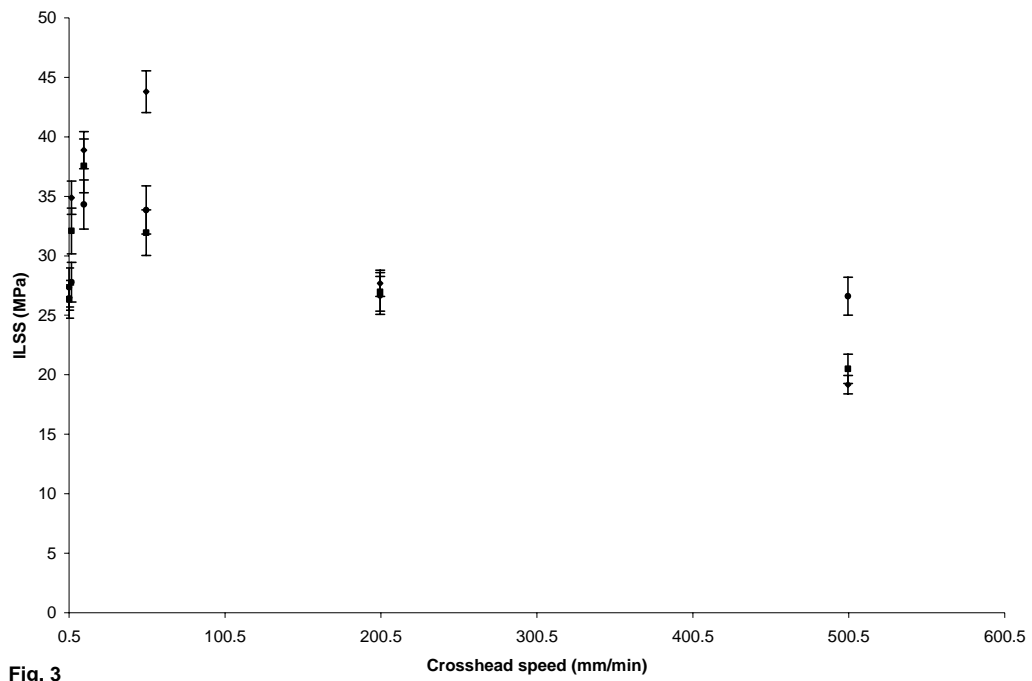


Fig. 3