

## **Freeze-thaw Response of Glass-polyester Composites at Different Loading Rates**

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### **ABSTRACT**

Present literature suggests the need for investigating and characterizing the freeze-thaw response of polymer composites under different loading rates and also at ambient and sub-ambient temperatures. The present experiment has been carried out with three weight fractions (i.e. 0.55, 0.6 and 0.65 ) glass fibers reinforced polyester composites. The short beam shear specimens were suddenly exposed to – 80°C temperature for 2 hours and then 3-point bend test was conducted instantaneously at that temperature. Another batch of samples was allowed to thaw at ambient temperature for 1 hour and tested on flexural mode at ambient temperature. The testing was carried out for a range of 0.5 to 500 mm/min crosshead speed. The shear strengths at cryogenic temperature and at thawing temperature were compared with the room temperature tested data of shear value of laminates.

**KEYWORDS:** glass fiber, composites, mechanical behavior, crosshead speed, interface, freeze-thaw.

## INTRODUCTION

The need in development of hypersonic vehicles has led to the evaluation of lightweight materials and structures for cryogenic tankage. Polymer composites are steadily replacing conventional metallic structures in improving aircraft and spacecraft performance [1]. The volumetric expansion of water when it freezes, results in stress concentration at the defect tip that may synergistically interact with residual tensile stresses in a laminated polymer composite at low temperature to nucleate a crack [2]. The thermal expansion of fiber composites undergoes great variations as a function of thermal stresses [3]. The mechanical behavior of polymer composite materials at ultra-low temperature must be investigated in order to increase the reliability of space transportation systems. The effect of sudden and/or seasonal freeze- thaw cycling on matrix cracking in a laminate is of vital importance for structural durability and reliability. The freeze-thaw effect on the overall integrity of the retrofit system is an important concern. The subzero temperatures and freeze-thaw exposure may result in significant changes in thermomechanical response of fiber-reinforced polymer (FRP) matrix composites. Mechanical performance as well as mechanisms of fracture and failure may be affected by such active environmental exposure. The effect at the constituent level may be hypothesised to be due to the cumulative effect of matrix hardening and stiffening as well as the formation cryogenic microcracks parallel to fibers and at the fiber-polymer interphase [4]. The final mechanical properties of composite materials are mainly determined by the properties of the interface and the state of adhesion between fiber and polymer matrix. Matrix shrinkage during cure,

Poisson's effects and mismatched coefficients of thermal expansion between constituent phases as well as the neighboring ply layers in a laminate may cause microcracking and delamination failures in a composite [5-8]. The fundamental understanding of strain-rate effect on mechanical properties of laminated composite is particularly important during impact loading applications of composite materials especially when glass fibers are strain rate and temperature dependent reinforcing phases [9-10]. High loading condition has always been a cause for concern that mechanical properties of FRP composites may be poor at high rates of strain. The very recent literature review [11] in the area suggests that the effect of varying loading rate on the tensile, compressive, shear and flexural properties of fiber-reinforced composite materials is very contradictory and also inconclusive in nature. Hence, the need for investigating further in the pursuit of eliminating the digagreement, which currently exist regarding the effect of loading rate on mechanical behavior of FRP composite. The properties of fiber-matrix interface are an important parameter in controlling matrix- fiber stress transfer [12]. Past research explored the role of fiber-matrix adhesion in determining freeze-thaw response at constant loading. The present work is original in that it focused on the role of interface in freeze-thaw response at various loading rates and with varied percentages of constituent phases of polymer composites. It should be emphasised that the freeze-thaw exposure causes matrix microcracking in resin-rich regions which most often leads to local zones of weakness. These local zones of weakness may change the fracture/ failure mechanisms in the loading process [4]. The final performance of a fiber- reinforced composite depends on the fiber properties, the

extent of resin cure, resultant matrix properties and the nature of fiber- polymer matrix interface [13].

## **EXPERIMENTAL**

An unsaturated polyester resin was 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. 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<sup>0</sup>C temperature in an ultra-low freezing chamber for 2 hours. One batch of samples was tested instantaneously in a 3-point flexural mode almost at that temperature. The other batch was allowed to thaw at ambient temperature (30<sup>0</sup>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 room temperature with the same range of crosshead speed. All samples were kept in desiccators for many days to stabilize the variation in weight before freeze-thaw conditioning. The same procedure was followed for untreated specimens. An Instron 1195 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**

Figure 1 shows the effect of crosshead speed on ILSS value of glass/ polyester composites (0.55 fiber weight fraction) at ultra-low freezing temperature, at thawing temperature, and also at room temperature. It is evident here that the nature of curves for all temperature is different at above and below 50mm/min crosshead speed. The ILSS values are higher at freezing and thawing temperature as compared to untreated samples upto about 50 mm/min crosshead speed. These could be attributed to the cryogenic hardening of polymer matrix phase. Here the matrix phase percentage is 45 by weight. A sharp and continuous fall in ILSS value is noticed for higher loading rate. Figure 2 corresponds to the glass/polyester composites (fiber 0.60 weight fraction). Here the variations in ILSS value for three types of situations are of different natures unlike figure 1. The shear strength value at cryogenic temperature for all loading rate is higher in compared to other two cases. The lower percentage of polymer phase in the composite is probably more susceptible to have greater percentage of damage during thawing by the effect of thermal shock. This may possible debond interfacial areas. The more interfaces in the present composites are prone to damage by thawing cycle. Here, it is also evident that the ILSS values are low for all cases at 500mm/min crosshead speed. Figure 3 shows the variation of ILSS value for glass/ polyester composites (fiber 0.65 weight fraction) with crosshead rate at different temperatures. The specimens,

which were tested at cryogenic temperature, show higher shear strength for almost all loading rates. The more percentages of interfacial area here are strongly affected during thawing. It may lead to generate more interfacial cracking. The damage may possible be accelerated because of poor fibre-polymer adhesion or improper/insufficient wetting. Here the effect of cryogenic micro cracking of matrix- interface on damage mechanics is not strongly influence at ultra-low freezing temperature. The different cross-link density of polymer chains can produce a highly nonhomogeneous local stress field. FRP composites generally contain randomly spaced micro voids, incipient damage sites and microcracks with statistically distributed sizes and directions. Therefore, the local strength in the material varies in a random fashion, so the failure sites do not necessary coincide with the maximum stress location. The damage mechanisms are time dependent processes and they are responsible for the time sensitivity of the constitutive and fracture behavior of the material [14]. The local microstructure near the crack tip plays an important role in the blunting phenomena. The crack advances by coalescing with microvoids. The crack tip is then resharpened. The basic mechanism of crack tip opening and growth involves the formation and growth voids ahead of crack tip. It should be emphasised here that the severity of blunting decreases with decreasing temperature as void formation is suppressed. It is important to note that a change in loading rate can change failure modes. The ductility of a resin matrix could become a limiting factor at high loading rate for the composite strength. The specimens tested at a lower temperature are characterized by a greater order of microcracking and

delamination. Thus, the fracture mechanisms in fiber-polymer composites are a function of temperature and environment. Brittle thermo set polyester resin may undergo a small order of microdeformation prior to failure. Polyester resin and E-glass fibers have been found to be loading rate sensitive [15]. A plastic deformation zone ahead of crack tip region may possible be formed by matrix deformation and micro cracking. A weaker of interfacial bond may result in a low flexural strength of the laminate. The deteriorated integrity can cause low strength at high loading. All phenomena are possibly contributing the observed non-linear mechanical behavior of FRP composites under freeze-thaw conditioning.

## **CONCLUSIONS**

The effects of ultra-low freezing and thawing of glass-polyester composites for varied weight fraction constituents at different loading rates are experimentally investigated. Interface characterization by the freeze-thaw cycle is qualitatively assessed in the present study. Loading rate sensitivity is strongly evident at lower range of crosshead speed (0.5 to 50 mm/min) and ILSS values are found to increase in all situations with more loading speed in the range. Thereafter, the fall in ILSS value is observed with higher crosshead speed. There are some experimental data of ILSS value reflect the weakening effect of thermal shock ( either due to sudden exposure to  $-80^{\circ}\text{C}$  temperature or by thawing effect). It may be stated that the hardening effect of matrix by cryogenic temperature is noticed at lower crosshead speed and results of specimen tested at higher loading rate indicate the damage of polymer matrix by cryogenic microcracking.

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## Figure Captions

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

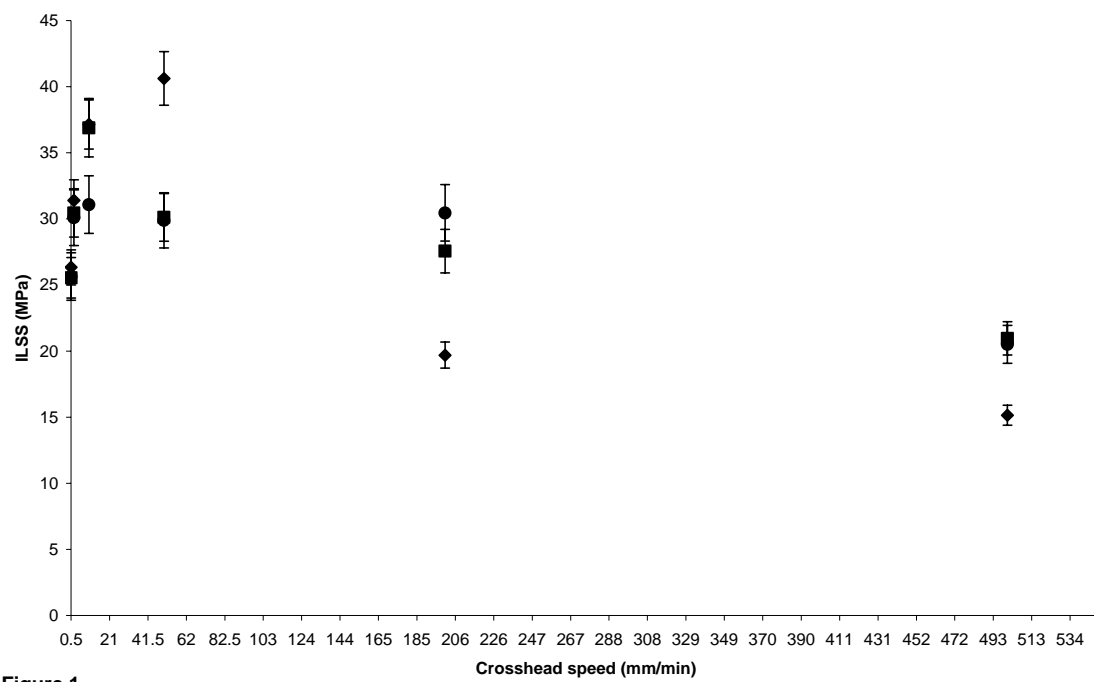


Figure 1

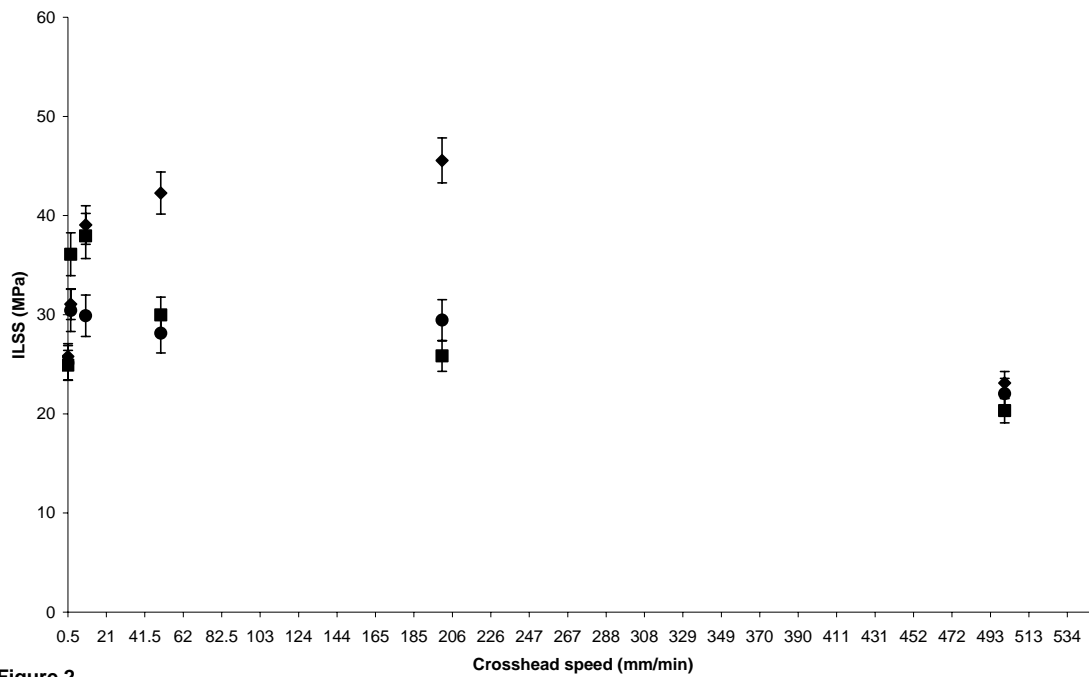


Figure 2

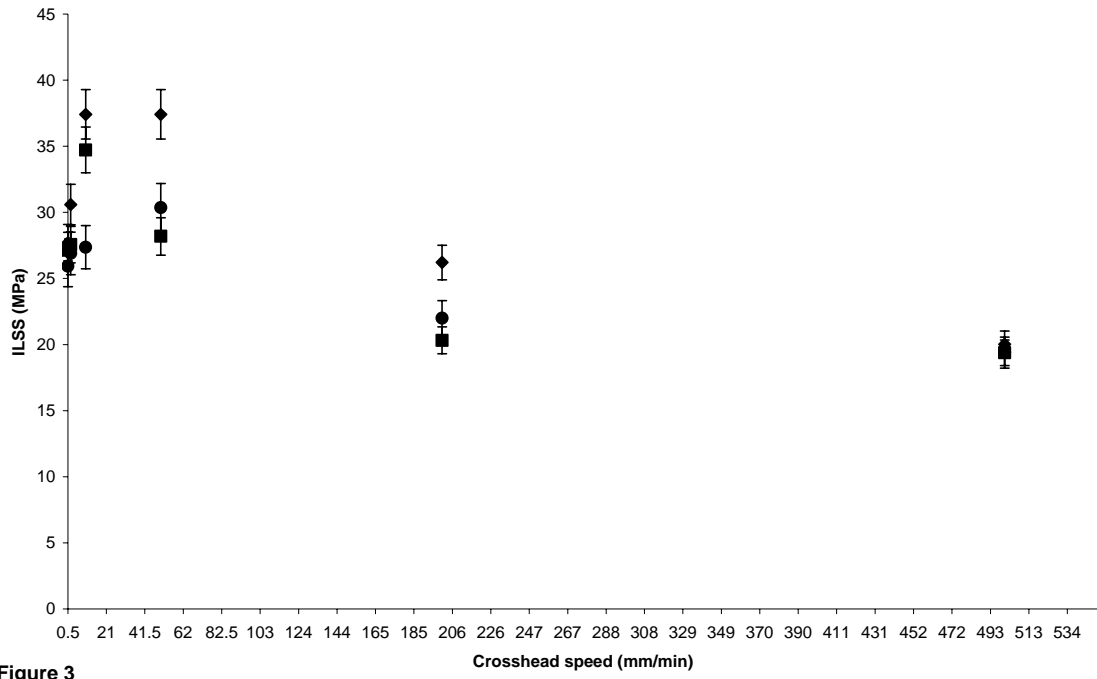


Figure 3