

## **Loading Rate Effects on Mechanical Properties of Polymer Composites at Ultra-low Temperatures**

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**ABSTRACT:** E-glass fibers of 55, 60 and 65 weight percentages were reinforced with epoxy matrix to prepare the laminated composites. They were exposed to  $-40^{\circ}\text{C}$ ,  $-60^{\circ}\text{C}$ , and  $-80^{\circ}\text{C}$  temperatures for different times. The 3-point bend test was conducted on the conditioned samples at those temperatures. Mechanical test was carried out at 2 mm/min and 500 mm/min crosshead speeds. The main emphasis of the investigation was to evaluate the roles of percentage matrix phase and interfacial areas on the interlaminar shear failure mechanism of glass/epoxy composites at ultra-low temperatures for different loading speeds. The mechanical performances of the laminated specimens at low temperatures were compared with room temperature property. The loading rate sensitivity of the polymer composites was appeared to be inconsistent and contradictory at some points of conditioning time and as well as at a temperature of conditioning. Phenomena may be attributed by low-temperature hardening, matrix cracking, misfit strain due to differential thermal coefficient of the constituent phases and also by enhanced mechanical keying factor by compressive residual stresses at low temperatures.

**Key words:** composites; failure; fracture; mechanical properties; shear; ultra- low temperature

## **INTRODUCTION**

The cryogenic properties of polymers are recently drawing attention with new development in space and electronic technologies. Although mechanical strength of most polymers increases or remains same as temperature is decreased, the elongation to failure decreases to extremely low values at cryogenic temperatures.<sup>1</sup> This behavior restricts the use of most polymeric materials at low temperatures. It has been proposed that the local intermolecular rearrangement results in relaxation at low temperature region. The relaxation phenomena are indicating a considerable dependence on morphology of polymers. There appears to be no definite relationship between the chemical structure of polymers and cryogenic behaviors. Polymers which are able to change their main chain bond angles seem to excel in cryogenic properties. These polymers are flexible and may undergo deformation even when their segmental motions are frozen at cryogenic temperatures.<sup>2</sup> A dynamic mechanical relaxation occurs in polymer molecules due to heat transfer between the intermolecular mode and the intramolecular mode. The physical properties of polymer materials depend severely on frequencies of excitation.<sup>3</sup> Glass fiber reinforced plastics are extensively employed in many different applications. E-glass fibers and epoxy resin are known to be highly loading rate sensitive.<sup>4</sup> Thus the mechanical properties of most of polymer matrix composites are rate sensitive. The mechanical properties of E-glass/epoxy composite are rate sensitive at low range of strain rate. It seems that the greater the strain rate and the loading velocity, the greater are the stiffness and ultimate strength of the composite material.<sup>5</sup> Failure strength of glass/epoxy composites increases manifold and

failure strain reduces sharply at also high range of strain rate.<sup>6</sup> Scanning electron microscopy of their work does not reveal any significant difference between the failure mechanisms at low and high strain rates. The effect of varying loading rate on tensile, compressive and flexural properties of fiber reinforced composites is reported to be contradictory and not so conclusive.<sup>7</sup> Hence the need exists in the pursuit of eliminating all disagreements in this area of research.

The need for the development of hypersonic vehicles has led to the evaluation of lightweight materials and structures for cryogenic tankage. Polymer composites are steadily replacing the conventional materials for improving performance and durability. Past research has explored mostly the effect of low temperatures on mechanical behavior of polymer composites at constant loading. The present investigation is original, in that it emphasizes on the role of fiber/polymer interface on mechanical performance at low and high loading rates for a range of ultra-low temperatures and also with the varied percentages of constituent phases of glass/epoxy laminates. The use of composites in safety-critical applications lead to uneasiness since the mechanical response is not well understood in complex environmental and service conditions.

## **EXPERIMENTAL PROCEDURES**

Woven fabric glass fibers were used with epoxy resin for fabrication of laminated composites by hand lay up method. Three weight percentages of glass fibers (55, 60 and 65 wt %) were targeted to prepare the specimens. They were cured at room temperature for 24 hours and then they were dried at a 50°C temperature oven for a

sufficient time unless the variation of weight change was almost negligible. The moisture-free polymer composites were exposed to  $-40$ ,  $-60$  and  $-80^{\circ}\text{C}$  temperatures for different time durations (1, 4, 9, 16 and 25 hours). The short beam shear (SBS) test specimens were immediately tested at each point of freezing time and at each low temperature. The test was carried out at 2 mm/min and 500 mm/min crosshead speeds for each point of experiment. The SBS test was conducted as per ASTM standard (D2344-84). About 10 samples were tested at each level of experiment and their average value was reported here. The ILSS (interlaminar shear strength) value was calculated as follows,

$$\text{ILSS} = 0.75P_b/wt$$

where  $P_b$  is the breaking load,  $w$  the width of specimen, and  $t$  the thickness of specimen.

The fractured surfaces were characterized by scanning electron microscope to observe changes in the polymer matrix and also at the fiber/matrix interfaces.

## **RESULTS AND DISCUSSION**

Figure 1 shows the variation of shear values of 0.55 weight fraction glass fibers reinforced epoxy composites with time of exposure time at  $-40$ ,  $-60$  and  $-80^{\circ}\text{C}$  temperatures for 2 mm/min and 500 mm/min crosshead speeds at each temperature of experiment. The stress at delamination failure was found to increase slightly with increased crosshead speed at some points of conditionings for three temperatures. The ILSS value increased with more conditioning time for a same loading rate. Here no statistically significant variations of shear value were found

with change of conditioning temperature. Greater value of shear value at longer conditioning time may possibly be attributed by enhanced key and lock principle at the fiber/polymer interfaces.

The variation of shear values of 0.60 weight fraction glass fibers reinforced epoxy composites with time of exposure time at  $-40$ ,  $-60$  and  $-80^{\circ}\text{C}$  temperatures for 2 mm/min and 500 mm/min crosshead speeds at each temperature of experiment is shown in Figure 2. It was more consistently found that the ILSS value is higher at higher loading rate for all conditioning temperatures. This consistency can be contributed by better adhesion at the interface. Here it was found that the shear values increased slightly with more conditioning time.

Figure 3 shows the variation of ILSS values of 0.65 weight fraction glass fibers reinforced epoxy composites with time of exposure time at  $-40$ ,  $-60$  and  $-80^{\circ}\text{C}$  temperatures for 2 mm/min and 500 mm/min crosshead speeds at each temperature of experiment. The most exceptional observation here was no significant increase in ILSS values with conditioning time unlike above two percentages of fibers reinforced composites. Here more percentage of fibers in the composites has resulted more interfacial areas and thus more matrix damages were inducted by misfit strain.

Scanning electron micrograph (Figure 4) shows the fractured surface of the ultra-low temperature treated glass/epoxy specimen. It reveals total loss of adhesion at the fiber/polymer interface and massive matrix cracking.

It is important to note that a change in the loading rate can change failure modes. A plastic deformation zone ahead of the crack tip may be formed by matrix

deformation and matrix microcracking. The deteriorated integrity can cause low strength at high loading.<sup>8</sup> The ductility or failure strain of a matrix resin may become a limiting factor for the composite strength.<sup>4,9</sup> The extent to which fiber content affects the rate sensitivity is yet to be established. The failure mode changes from fiber brittle failure to brittle failure with considerable matrix damage as the crosshead rate increases. This brings about a resulting increase in energy absorption.<sup>10,11</sup> The composite sensitivity to strain rate is mostly driven by the resin behavior.

## **CONCLUSION**

The present findings may lead to the following conclusions,

Loading rate sensitivity seems to be controlled by the area of interfaces and the percentage of polymer matrix phase present in composites.

Its nature of variation appears to be dependent on testing temperature and the rate at which specimens are tested.

Reasonably high loss of integrity at some points of experiment in ultra-low temperature could be a possible cause of inconsistent variation of mechanical strength.

Less conditioning time yields predictable mechanical behavior. It may possibly be attributed by the lesser damage in the polymer matrix.

Contradictory and inconsistent variation of shear strength with loading speed at higher condition times for all experimental ultra-low temperatures could be contributed by massive matrix crackings.

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## References

1. Nielsen, L. E. Mechanical Properties of Polymers and Composites, Marcel Dekker, New York, 1975.
2. Yano, O.; Yamaoka, H. Prog Polym Sci 1995, 20, 585.
3. Hayakawa, R.; Tanabe, Y.; Wada, Y. J Macromol Sci Phys 1973, B8, 445.
4. Jang, B. Z. Advanced Polymer Composites: Principles and Applications, ASM International, Materials Park, OH, 1994.
5. Fereshteh-Saniee, F.; Majzoobi, G. H.; Bahrami, M. J. Mater Proce Tech 2005, 162-163, 39-45.
6. Majzoobi, G. H.; Fereshteh-Saniee, F.; Bahrami, M. J. Mater Proce Tech 2005, 162-163, 76-82.
7. Jacob, G. C.; Starbuck, M.; Fellers, J. F.; Simunovic, S. J Appl Polym Sci 2004, 94, 296.
8. Ray, B. C. J Reinf Plast & Compos In Press.
9. Ray, B. C. J Reinf Plast & Compos 2005, 24, 713.
10. Okoli, O. I. Compos Struc 2001, 54, 299.
11. Okoli, O. I.; Smith, G. F. Compos Struc 2000, 48, 157.



## FIGURE CAPTIONS

**Figure 1** Changes of ILSS values with freezing time for glass/epoxy composites (containing 55% weight fiber) at  $-40^{\circ}\text{C}$  temperature for 2 mm/min (●) and 500 mm/min (●), at  $-60^{\circ}\text{C}$  temperature for 2 mm/min (▲) and 500 mm/min (▲), and at  $-80^{\circ}\text{C}$  temperature for 2 mm/min (◆) and 500 mm/min (◆) crosshead speeds.

**Figure 2** Changes of ILSS values with freezing time for glass/epoxy composites (containing 60% weight fiber) at  $-40^{\circ}\text{C}$  temperature for 2 mm/min (●) and 500 mm/min (●), at  $-60^{\circ}\text{C}$  temperature for 2 mm/min (▲) and 500 mm/min (▲), and at  $-80^{\circ}\text{C}$  temperature for 2 mm/min (◆) and 500 mm/min (◆) crosshead speeds.

**Figure 3** Changes of ILSS values with freezing time for glass/epoxy composites (containing 65% weight fiber) at  $-40^{\circ}\text{C}$  temperature for 2 mm/min (●) and 500 mm/min (●), at  $-60^{\circ}\text{C}$  temperature for 2 mm/min (▲) and 500 mm/min (▲), and at  $-80^{\circ}\text{C}$  temperature for 2 mm/min (◆) and 500 mm/min (◆) crosshead speeds.

**Figure 4** Scanning micrograph shows extensive fibers pull-out and massive matrix crackings of the low temperature treated specimen.

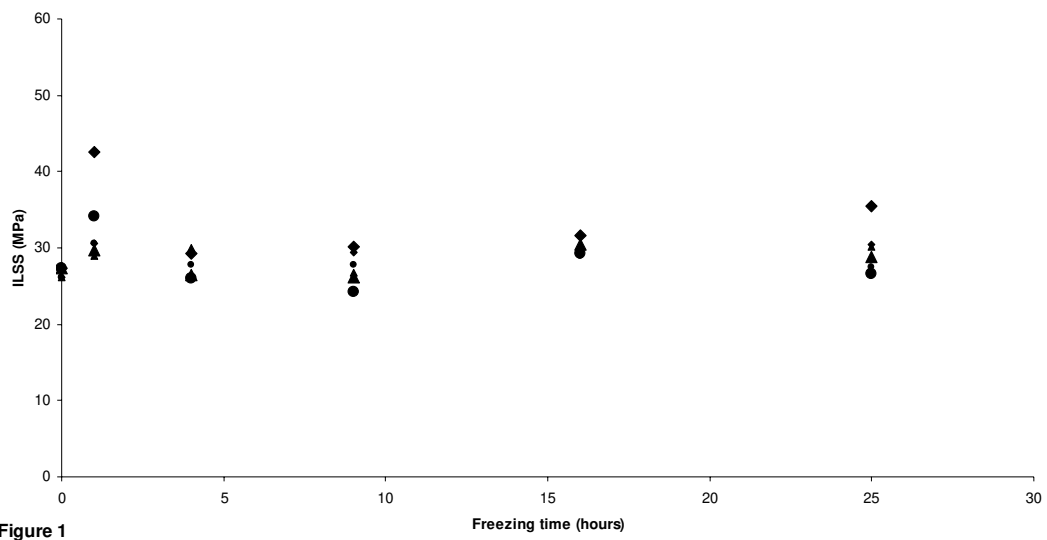


Figure 1

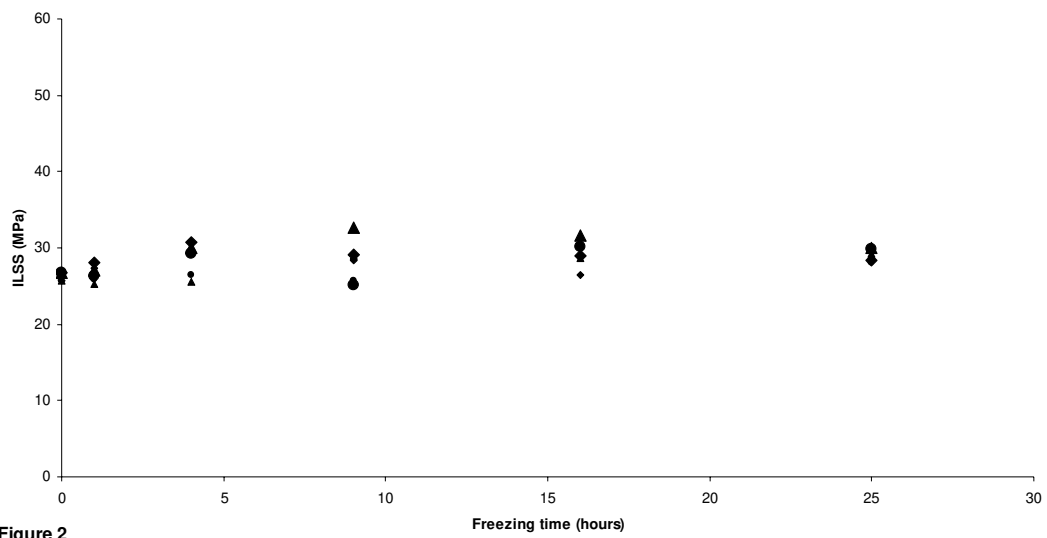


Figure 2

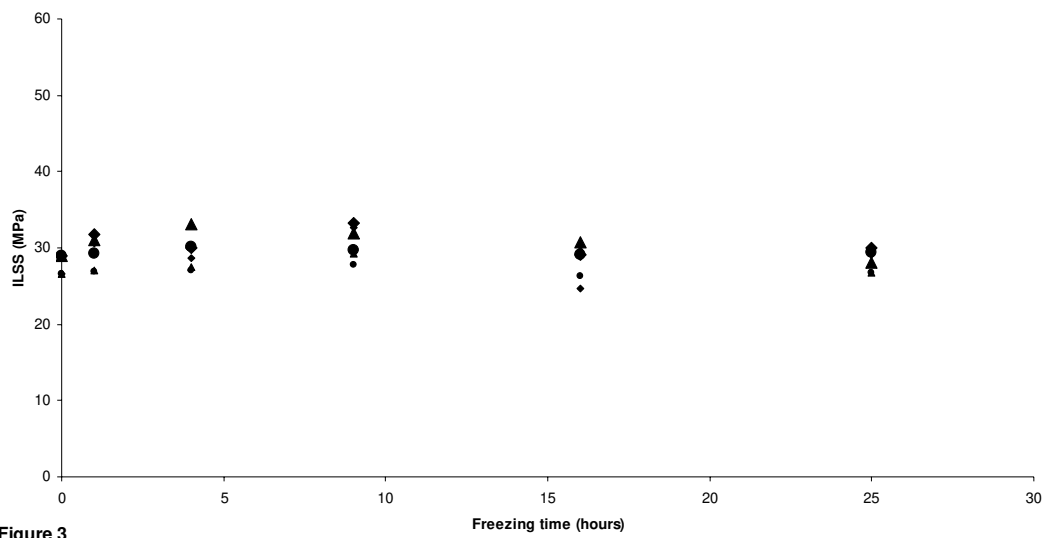


Figure 3

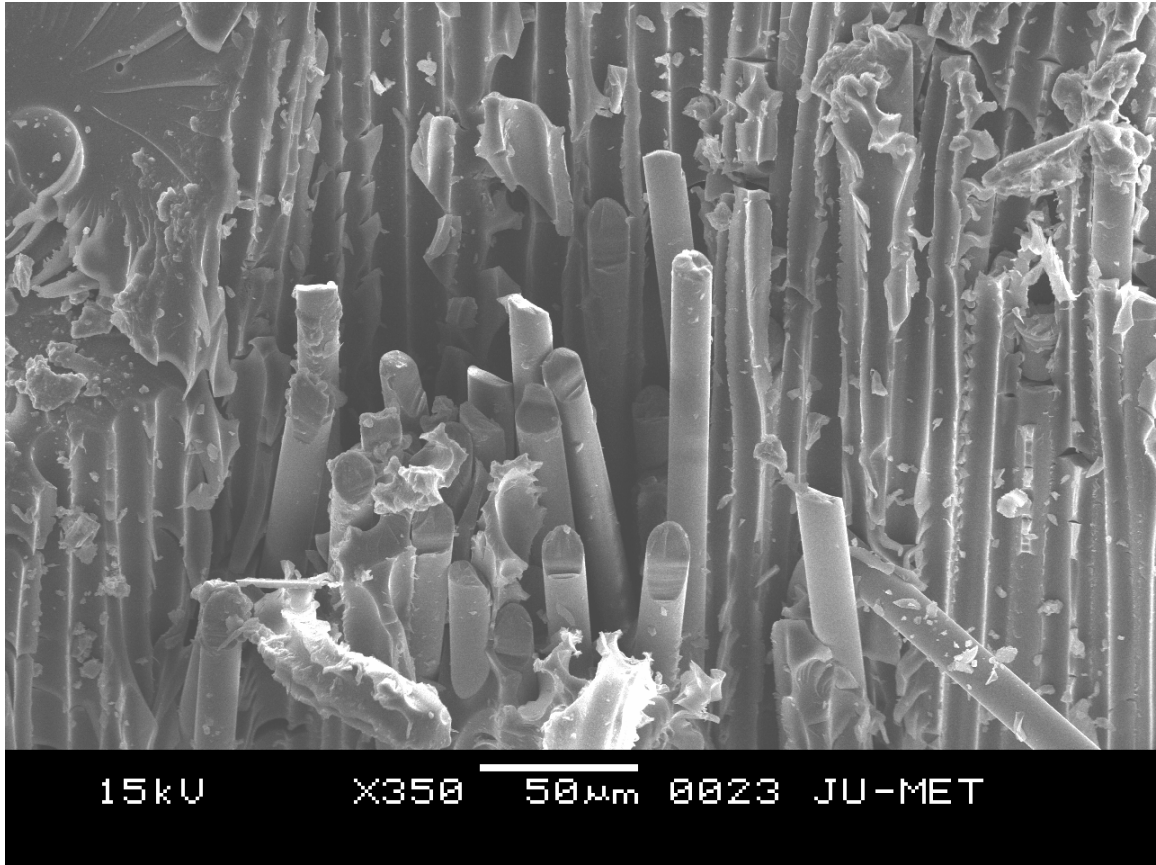


Figure 4