Paper Presented in "Second International Conference on Recent Advances in Composite Materials" (ICRACM-2007), Feb 20-23, 2007, India Habitat Centre, Lodhi Road, New Delhi India.

MECHANICAL BEHAVIOR OF FRP COMPOSITES AT LOW TEMPERATURE

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Abstract: Experimental studies have been carried out to understand the mechanical behavior of glass/epoxy composite laminates at cryogenic conditions and at different loading rates. This study uses 3-point flexural test to qualitatively assess and compare such behavior for 50 weight percentage of E-glass fibers reinforced epoxy composites during cryogenic and ambient conditions. The specimens were tested at 2 mm/min, 50 mm/min, 100 mm/min, 200 mm/min and 500 mm/min crosshead speeds to evaluate the sensitivity of mechanical response during loading at these conditions. The mechanical performances of the laminated specimens at cryogenic conditions were compared with room temperature property by using SEM photographs. Phenomenological behavior of these materials may be attributed by polymer stress relaxation, cryogenic matrix hardening, matrix cracking, resin/fiber debonding and misfit strain due to differential thermal coefficient of the constituent phases and also by enhanced mechanical keying factor by compressive residual stresses at cryogenic temperatures.

1. INTRODUCTION

Nowadays polymer composite materials are in massive demand for applications in the field of aerospace vehicles, automobile parts, satellites, sports goods, robots, and thermal insulation structures like cryostats for low temperature technology, hydrogen technology tanks, in superconductivity and also in biomedicine for body compatible implants [1, 2]. These materials exhibit exceptionally good characteristics such as low density, high specific strength, good anticorrosion properties, fatigue resistance and low manufacturing costs. These materials have received increased attention for applications in cryogenic environment [3]. Recently one area identified as potential source for significant weight reduction is the replacement of traditional metallic cryogenic fuel tanks with advanced polymeric matrix composite (PMC) tanks. Carbon-fiber/epoxy-resin composites have been evaluated for cryogenic tankage in RLV (Reusable Launch Vehicle) [4].So now Polymer composites are contenders for use in reusable launch vehicle components. Mostly such components are cryogenic fuel tanks, cryogenic fuel delivery lines, and parts of the cryogenic side of turbo-pumps [5]. Glass reinforced polymer composites are used mainly in thermal insulation, electrical insulation, and structural support, as well as in permeability barriers, which provide minimal structural support in superconducting magnets at low temperatures [6, 7]. The properties that support polymer composite materials for cryogenic applications are strongly dependent on the factors including the matrix and fiber material and their volume fractions, the fiber orientation, the applied stress levels and strain rates, as well as the loading conditions and the nature of fiber polymer interface [8]. Interface is the heart of the composite. The local response of fiber matrix interface within the composite plays an important role in determining the gross mechanical performance [9]. It provides a means of stress transfer from fiber to fiber through the matrix. In cold conditions, high residual stresses can build up within the fibrous composite materials due to different coefficients of thermal expansion of the fiber and the matrix and at low temperatures the polymer matrix experiences embrittlement which can also affect the properties of the composite [10 ,11]. But with change in temperature, or when the component comes from the cold condition to the normal ambient condition, it may affect the residual stresses leading to either deterioration or enhancement of mechanical properties. It is generally recognized that the bond strength variation at the interface greatly affects the integrity of composite materials. The bond strength depends on quality of interfacial adhesion. The non-zero state of residual thermal stresses at low temperatures is the underlying cause of microcracking in composites and these microcracks propagate results in transverse cracks. When the transverse crack develops further, the crack deflects through the interface between layers and delamination initiates. The delaminations connect the microcracks in adjacent layers and provide leakage paths. The combined cryogenic and elevated temperature thermal cycle produces substantially greater amounts of damage in polymer composites [12, 13]. Epoxy resin and E-glass fiber are reported to be loading rate sensitive [14]. This sensitivity is controlled by the area of the interfaces and the percentage of polymer matrix phase present in composites [15].The ductility of a matrix resin may become a limiting factor at high strain rate for composite strength [16]. Epoxy resin is more ductile than it's composite at low strain rate. So to increase the reliability of polymer composites it is necessary to understand the mechanical behavior of these composites at low temperature. The main objective of this paper is to investigate the mechanical behavior of cryogenically conditioned glass/epoxy composites at different loading rates.

2. EXPERIMENTAL

2.1 Material

Araldite LY-556, an unmodified epoxy resin based on Bisphenol-A and hardener (Ciba-Geigy, 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.

2.2 Fabrication of composites

The glass fiber/epoxy composite laminates were fabricated by wet lay-up method; the glass fiber woven cloth of required dimension was laid over a mould and then catalyzed epoxy resin was poured absorbed over the reinforcement. The wet composite was rolled, to distribute the resin and to remove the air pockets. The sequence was repeated until the desired thickness was obtained. The layered structure was allowed to harden on cure. It was cured at room temperature for 48 hours. After curing, the laminate was cut into the required size for 3-point bend (Short-Beam Shear) test by diamond cutter. Then stability test was done for the composite laminates. Here the laminates were weighed and then heated in an oven at 50 ^{o}C. The weight is intermittently checked till we get a stable weight, that is, with further heating there is no change in the weight of the composite.

2.3 Cryogenic Conditioning

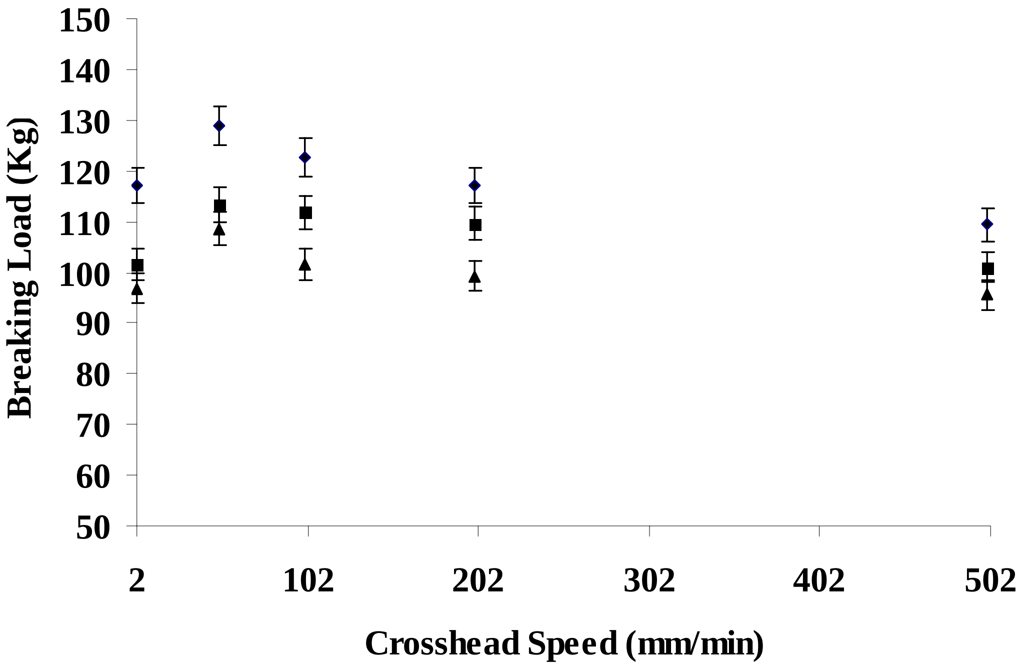
After the stability test the samples were allowed to come back to the ambient temperature. Then they were kept in the desiccators so that there is no further absorption of moisture. The samples to be cryogenically treated were exposed to liquid nitrogen environment (77K) for one hour. After the exposure one batch of samples were taken out and kept at room temperature for one hour. Another batch of samples was tested in 3-point bend test immediately after exposure to cryogenic temperature.

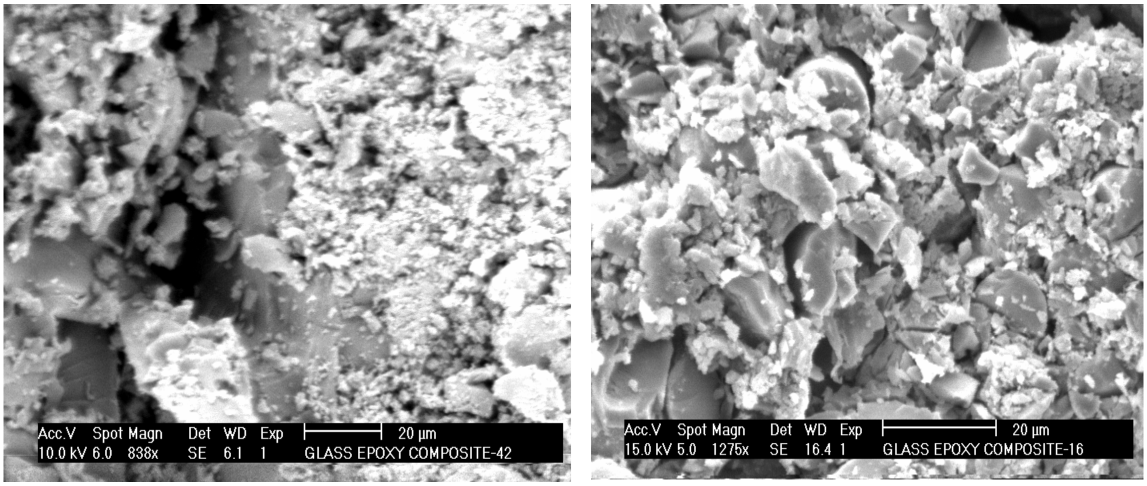
2.4 3-point bend test

The 3-point bend tests were carried out for first batch of samples immediately after exposure to cryogenic temperature. The former samples after exposure to room temperature and the untreated as-cured samples were tested in 3-point bend test at room temperature. All the mechanical flexural tests were performed at 2, 50, 100, 200 and 500 mm/min crosshead speeds. Then breaking load and strain at maximum load was measured from stress vs strain graphs for all the samples.

3. RESULTS AND DISCUSSION

The breaking load values at cryogenic temperature (♦), ambient temperature after exposure to cryogenic temperature (■) and ambient temperature of untreated samples (▲) are plotted against the cross head speeds of 2 mm/min, 50 mm/min, 100 mm/min, 200 mm/min and 500 mm/min for glass/epoxy laminates in figure 1. From the graph we note that the breaking load is maximum for cryogenically treated laminates than the untreated laminates for all the loading rates. This may be due to cryogenic hardening of the matrix phase at low temperature. The polymer chains get frozen due to which the deformation process is reduced results in less polymer relaxation i.e. it get hardened. Residual stresses build up due to differential thermal contraction between fiber and matrix during sudden cooling from room temperature to cryogenic temperature (figure 2(a)). When the load is applied, these stresses greater than the shear strength of the resin is readily generated and failure of the resin phase will result or fracture is induced when the stress induced stress intensity factor exceeds the fracture toughness of the resin. By controlling the molecular structure the performance of the resin can be optimized [17, 18].

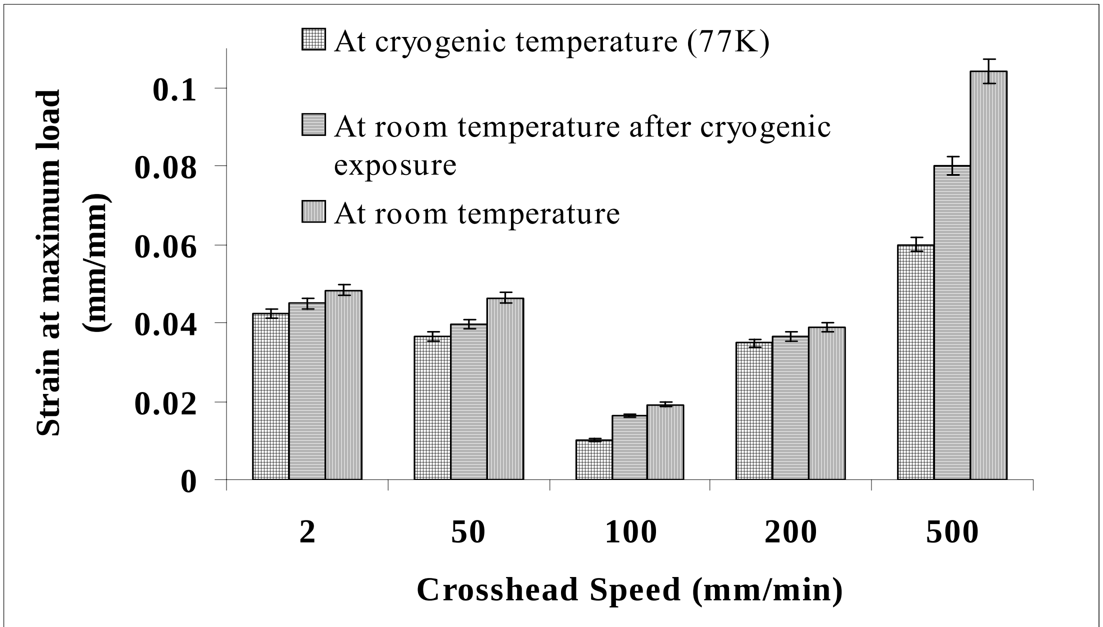




(a) (b)

Figure 2. Scanning micrographs showing (a) Differential contraction of epoxy matrix (b) Matrix cracking at cryogenic temperature (77K)

The cryogenic conditioning causes differential contraction and increases the resistance to debonding due to induced mechanical keying factor [19]. Hence, the load bearing capacity was irreversibly improved. However, it was found that the nature of the curve is different at above and below 50 mm/min crosshead speed. The breaking load increases with the crosshead speed upto 50 mm/min but reduces above. The lower value of breaking load at lower speed may be attributed to high failure strain at low strain rates so load increases with increases in speed. At low crosshead speed the laminate gets more time for failure to takes place which results in more deterioration causing reduction in the load bearing capacity. But at crosshead speeds above 50 mm/min the curve is opposite. Here the time available for the failure to takes place is very less; it is more like an impact force. So the matrix may be unable to transfer load properly to the fibers which leads to matrix cracking (figure 2(b)).



The higher crosshead speed restricts the relaxation process at the crack tip, so that the stress induced cracks may grow without blunting results in lowering of breaking load at higher crossl [illegible] tation factor [illegible] s that at all [illegible] e and maximum at room temperature. This may be attributed to cryogenic hardening of the matrix and massive microcracking at cryogenic temperature. Due to hardening, the brittleness of the matrix increases which leads to low strain values at maximum load.

Figure 3. Bar graph showing strain at maximum load for different crosshead speeds at cryogenic temperature (77K), at room temperature after cryogenic exposure, at room temperature

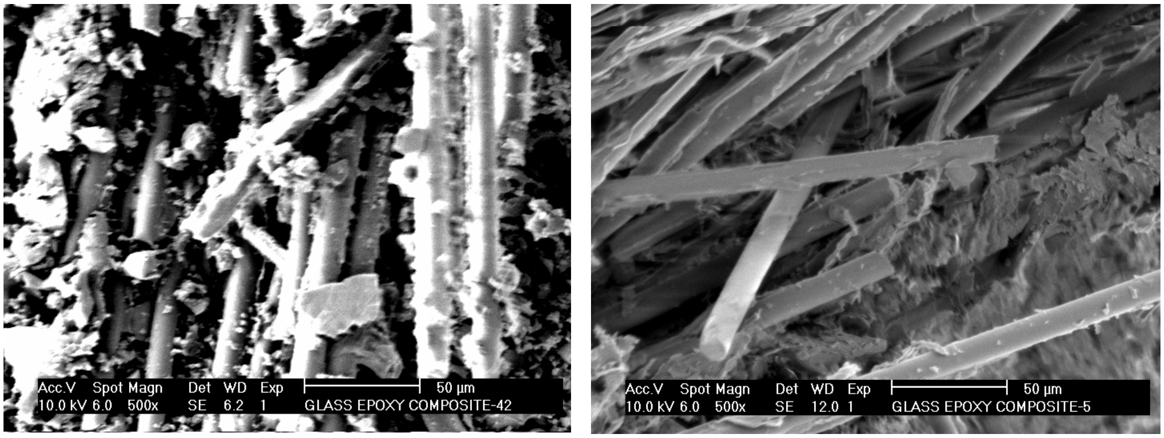


Figure 4. Scanning micrographs showing matrix cracking and debonded surfaces at (a) Cryogenic temperature (77K) (b) Room temperature

This will lead to resin/fiber debonding when the laminate is stresses further by cooling and loading in cold state. The characteristic of the interfacial adhesion is strongly influenced by the presence of residual stresses. However, some of the stresses developed by differential expansion/contraction are relaxed by viscoelastic flow or creep in polymer matrix [21]. The fracture toughness of the resin is controlled by stress relaxation at the crack tip and the strength of the molecular chains at cryogenic temperature [22]. The figure 1 also shows variation of breaking load with different cross head speeds at room temperature after exposure to cryogenic conditioning. Here relaxation of the stresses generated due to the applied load takes place after the cryogenic treatment at room temperature. This reduces the breaking load but higher than that of untreated laminates. At lower loading rates, redistribution of the load applied between the fiber and matrix takes place. Higher loading rates may reduce the stress relaxation processes at the crack tip resulting in lowering of the breaking load. Due to this relaxation phenomenon the brittleness decreases to some extent which leads to higher strains at maximum load but lower than the untreated laminates. Figure 4 shows that the cryogenically treated laminates have greater amount of matrix cracking and debonded surfaces than untreated laminates.

4. CONCLUSIONS

It is shown in the experiment that the breaking load values for the cryogenically conditioned laminates are higher than untreated laminates for all loading rates. This may be due to cryogenic hardening of the matrix and mechanical keying factor due to compressive residual stresses. This also results in lower strain values at maximum load for cryogenically conditioned laminates than the untreated laminates. Also the breaking load increases at with crosshead speed below 50 mm/min and exactly opposite nature was found above this speed. This can be attributed to less stress relaxation process at the crack tip. Large debonded surfaces were found in cryogenically treated laminates.

5. ACKNOWLEDGMENTS

Authors are expressing their deep gratitude to National Institute of Technology, Rourkela for the given infrastructural facilities to complete the project. The experimental work was assisted by Mr. Samir Pradhan and his cooperation is highly appreciated. The detailed and comprehensive study of this present investigation has already been under review process in the Journal of Reinforced Plastics and Composites.

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