

**MICROSTRUCTURAL ASSESSMENT OF FREEZE-THAW IMPACT ON CARBON-GLASS/EPOXY  
COMPOSITES**

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**ABSTRACT**

Cracks and debonded areas, nucleated by high residual stresses as a result of the changing temperature, can easily propagate at the weaker interface. The differential change in the condition of constituent phases (fibre, matrix resin and interphase) of fibrous polymer composites in freeze-thaw environments can result in a significant mismatch among constituents and this eventually leads to the evolution of localised stress and strain fields in composites. The situations could easily result in the nucleation of delaminating micro-cracks in the composites. The specimens were tested at 2, 50, 100, 200 and 500 mm/min crosshead speed to evaluate the microstructural integrity of glass, carbon and glass/carbon reinforced epoxy composites during 3-point loading at ambient, cryogenic ( $-50^{\circ}\text{C}$  &  $-80^{\circ}\text{C}$ ), fast thaw and slow thaw conditions. The SEM micrographs show changing failure behavior at different situations.

**1. INTRODUCTION**

The invincibility of composites has been their worst myth. Thermal shocks and fatigue are very common in many applications of polymeric composites. Epoxy resins are now increasingly used in engineering applications due to their excellent properties. A critical aspect of using fiber reinforced polymer matrix composites in various applications is their performance in changing environments. Having a vast field of application, it is subjected to different loading rates, a large range of temperatures and also a varied environmental condition. The effect of sudden and/or seasonal freeze-thaw cycling on matrix cracking in a

laminated is of vital importance for structural durability and reliability. Many works have been carried out to understand the dramatic changes that occur in the structure and properties of composite materials when they are exposed to low temperatures. Potholing or localized surface degradation, delamination and micro-cracking are some of the more dramatic phenomena that may occur as a result of cryogenic cycling [1].

Polymer composites are steadily replacing the traditional metallic structures for improving aircraft and spacecraft performance. The volumetric expansion of water when it freezes, results in stress concentration at the defect tip that may synergistically interact with residual stresses in a polymer composite at low

temperature to nucleate a crack [2]. The interaction between the surfaces of fibers and polymer matrix may create various chemical and morphological inhomogeneities. It is obvious that the mechanical performance of fibrous polymer composites are critically influenced by the micromechanical behavior of both the interface and the interphase regions. The interface is defined at the atomic scale as the layer of the immediate chemical bond between fiber and matrix resin. The interphase is created by local changes of the polymer matrix in the vicinity of a reinforcement fiber.

Hybridization provides materials designers with a better degree of freedom in tailoring composite to achieve a greater balance of stiffness and strength, increased failure strain, better damage tolerance, improved ability to absorb impact energy, and a significant reduction in cost [3].

## 2. EXPERIMENTAL

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 silane treated E-glass and PAN based epoxy compatible high strength carbon (M/S Carr Reinforcement Ltd., UK) fibers to fabricate the hybrid composite by hand lay up method. It was allowed to cure for 48 hours at room temperature.

The samples were exposed to  $-50^{\circ}\text{C}$  and  $-80^{\circ}\text{C}$  in a ultra low freezing chamber for 3 hours to attain those temperatures separately. One batch of treated samples was tested at that temperature. One out of other batch was allowed to thaw in freezer (slow thawing) and the other one in ambient temperature (fast thawing). The scanning electron microscopic examination was carried out on broken samples. The specimens were tested at different crosshead velocities.

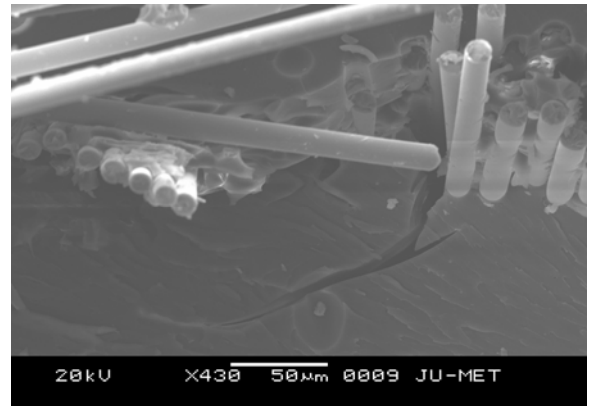
## 3. RESULTS AND DISCUSSION

The heterogeneous nature and very dissimilar expansion/contraction behavior, thermal stresses are easily generated in such composites and causes changing modes of failure in freeze-thaw conditioned glass-carbon/epoxy laminated composites.

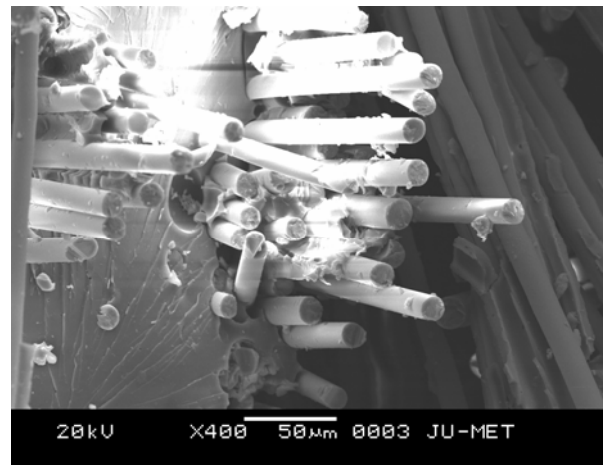
The fracture mechanisms in fiber-polymer composites are a function of temperature and environment. The damage mechanisms are time-dependent processes and they are responsible for the time sensitivity of the constitutive and fracture behavior.

Figures 1 and 2 are revealing matrix cracking and fiber breakage under the influence of freeze-thaw conditioning. The failure mode changes from fiber

brittle failure to brittle failure with considerable matrix damage, as the crosshead rate increases. The composite sensitivity to strain rate is mostly driven by the resin behavior [4]. It is important to note that a change in the loading rate can change failure modes. A plastic deformation zone ahead of a crack tip may be formed by matrix deformation and matrix micro-cracking. The deteriorated composite integrity can cause low strength at high loading. SEM study at some instants reveals a total loss of adhesion at the interface and also massive matrix cracking.



**Figure 1.** SEM micrograph is showing matrix cracking in carbon/epoxy composite

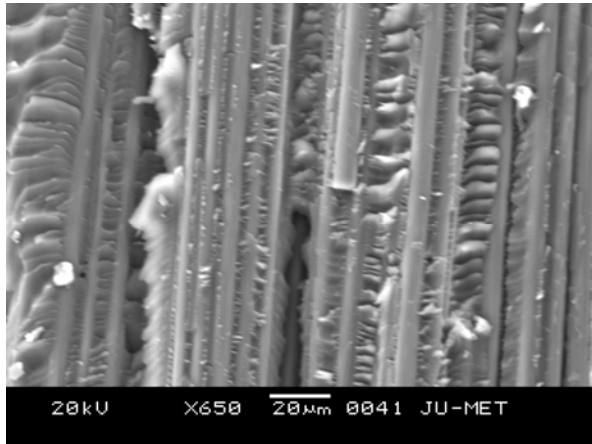


**Figure 2.** Fiber breakage and pull-out are evident in glass/epoxy system

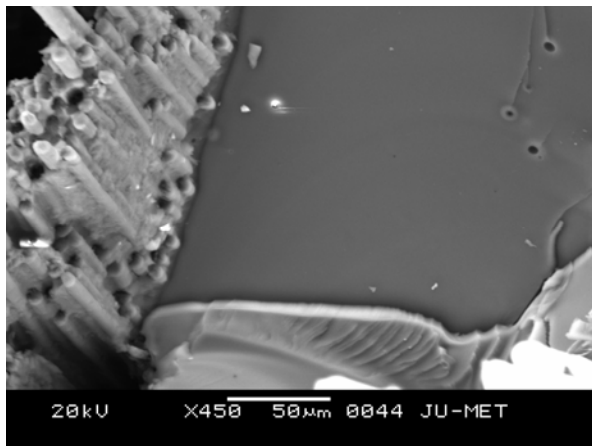
It is generally believed that the polymer is likely to be tough if homogeneous yielding does occur. Unfortunately, mechanisms in polymer composites in the presence of different residual stresses tend to be localized failure and produce inhomogeneous plastic deformation. The micro-crack density under the influence of freeze-thaw condition will be increased to

a threshold value where stress redistribution may limit the initiation of new crack [5].

Figures 3-5 are indicating the states of reinforcement fibers and matrix resins after freeze-thaw conditionings. Massive matrix cracking and potholing of resin matrix are observed to be dominating failure mechanism in such harsh and hostile environments. The weaker matrix phase is likely to be more prone to fail under such condition. The structural integrity and overall behavior of fiber reinforced polymer composites are strongly influenced by the stability of fiber/polymer interfacial region. The lateral distribution of the accumulated residual deformation stresses is heterogeneous, entailing strain localization. These strain localization phenomena may often lead to crack initiation via fiber breakage, microvoid formation and/or de-bonded interfaces [6].

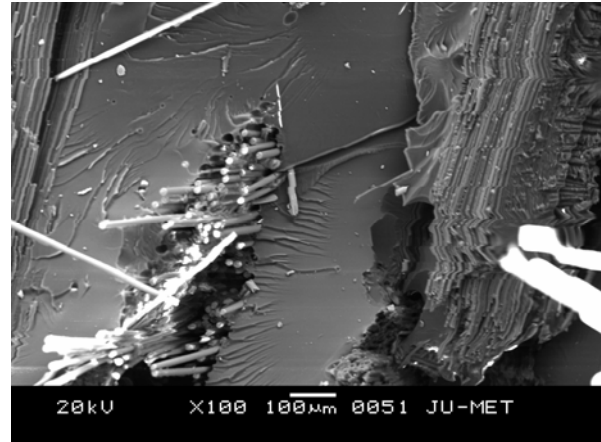


**Figure 3.** Cohesive damage instead of adhesive is prevalent in carbon/glass hybrid laminate



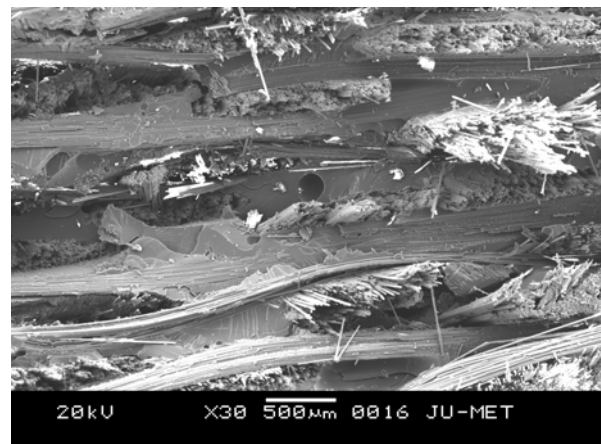
**Figure 4.** Potholings are evident in resin rich area and also in surrounding matrix resin in hybrid composite

The differential strains due to freeze-thaw conditioning may strongly alter the physical, chemical and mechanical characteristics of the material at different scale. The macroscopic behavior of composites depends not only on properties of individual constituents but also on elastic-plastic interaction between the different phases

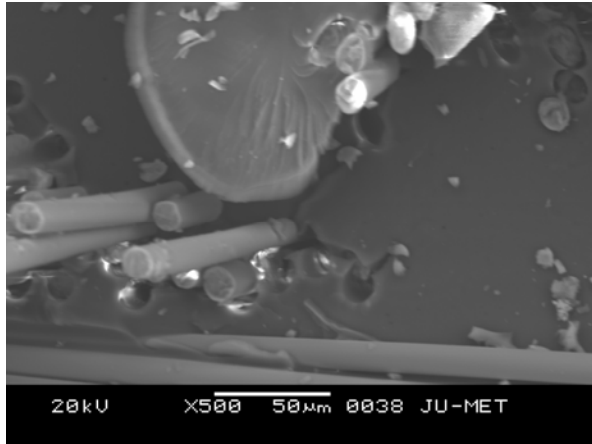


**Figure 5.** Adhesive failure is leading to matrix damage in glass/epoxy composite

Hybrid fiber composites are generally fabricated from glass fibers that are added to brittle carbon fiber to enhance the fracture toughness resulting from the toughening mechanisms associated with the ductile fibers, while maintaining a high strength and high modulus gained from carbon fibers. The effect of toughening depends on how the hybrid fibers are mixed and the ply lay-up is arranged [7].



**Figure 6.** Micrograph is showing matrix void in carbon/glass fibers reinforced epoxy laminated composite



**Figure 7.** De-adhesion and fibers pull-out are demonstrated in carbon/glass hybrid composite

Figures 6 and 7 are successively showing void and micro-void formation in the matrix resin and a loss of integrity at the fiber/matrix interface. Subzero temperature and freeze-thaw conditions can induce matrix hardening, matrix micro-cracking and fiber-matrix bond degradation. The present investigation has focused on possible microscopic view of failure modes in different individual as well as in combined state of fibers reinforced polymer composites under the influence of fast and slow free-thaw conditions.

#### 4. CONCLUSIONS

Contradictory and inconsistent variation of failure behavior at different freeze-thaw conditionings and also at different loading rates could be attributed by many factors, such as, resin relaxation, state of interfaces, post-curing phenomena, stresses relaxation and development, crazing and cracking in the matrix resin and also micro-void formation because of differential contraction/expansion among constituent phases. The presence of stronger interface (carbon/epoxy) and weaker interface (glass/epoxy) in hybrid composites induces more complex failure behavior. It necessitates a better and more critical understanding through more extensive investigation. A

fast-growing area of particular interest addresses the possible use of structural composite materials in cold regions. The progress has been slowed down by a lack of critical performance data.

#### ACKNOWLEDGMENTS

Authors are happy to extend their sincere gratitude to Prof. P C Chakraborty of Jadavpur University for helping in SEM examination. Authors remain grateful to Mr. M Madhan, Information Centre, NIT, Rourkela for continuous support in collecting literature.

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