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A comparative study of the mechanical performance of Glass and Glass/Carbon hybrid polymer composites at different temperature environments

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Abstract. Glass Fiber Reinforced Polymer (GFRP) composites have been widely accepted as high strength, low weight structural material as compared to their metallic counterparts. Some specific advanced high performance applications such as aerospace components still require superior specific strength and specific modulus. Carbon Fiber Reinforced Polymer (CFRP) composites exhibit superior specific strength and modulus but have a lower failure strain and high cost. Hence, the combination of both glass and carbon fiber in polymer composite may yield optimized mechanical properties. Further the in-service environment has a significant role on the mechanical performance of this class of materials. Present study aims to investigate the mechanical property of GFRP and Glass/Carbon (G/C hybrid) composites at room temperature, in-situ and ex-situ temperature conditions. In-situ testing at +70°C and +100°C results in significant loss in inter-laminar shear strength (ILSS) for both the composites as compared to room temperature. The ILSS was nearly equal for both the composite systems tested in-situ at +100°C and effect of fiber hybridisation was completely diminished there. At low temperature ex-situ conditioning significant reduction in ILSS was observed for both the systems. Further at -60°C G/C hybrid exhibited 32.4 % higher ILSS than GFRP. Hence this makes G/C hybrid a better choice of material in low temperature environmental applications.

1. Introduction

Fiber reinforced composites are one of the most commonly used structural materials in today's world. The most promising properties include high strength to weight ratio, high stiffness, low density, high corrosion, high endurance limit etc. Composites find various applications in aerospace industry, automobile industry, sports and goods, marine equipment etc. In these applications, during service life composites are exposed to various environmental conditions. Some harsh environmental conditions increase the vulnerability of degradation in mechanical properties. The properties of the fiber reinforced composite are governed by the individual property and behaviour of matrix, fibers and fiber/matrix interface in a particular environment. Due to their wide use in structural application the composites are commonly exposed to high temperature. During high temperature conditioning noticeable physical and chemical changes occur in the polymer network. Disintegration of short range ordering and thermo-oxidative degradation are observed at elevated temperature. It is inferred that these changes influence the performance of composite at high temperature. Due to difference in thermal expansion co-efficient of the matrix and reinforcement, thermal stresses get induced in the composite which can result in formation of micro-cracks at the interface. The interface hence becomes more susceptible to hostile reactions at elevated temperature environment [1]. The common failure modes at high temperature include fiber fracture, matrix cracking and fiber/matrix debonding. On exposing the unidirectional graphite composite at elevated temperature it was observed that the ILSS decreased by 30 % [2] Investigation on CFRP composite at -196°C for 555 hrs showed 20 % degradation in tensile strength compared to that at room temperature [3]. Due to increasing application of Fiber Reinforced Polymer (FRP) composites in aerospace vehicles for cryogenic fuel tanks, it is necessary to understand their behaviour at cryogenic temperature. The most commonly recorded damage mechanisms at cryogenic temperature are localised surface degradation,



delamination and generation of micro-cracks in polymer matrix and/or at the fiber/matrix interface [4] [5]. These failure mechanisms start degrading the properties of the composite at microscopic level which further extends to a noticeable proportion. A recent development in the fiber reinforced composite is the hybrid composite. This composite can have two or more types of reinforcements or matrix hybridisation with some nanoparticle. Hybrids are conceptually fabricated to take advantage of all the constituent reinforcements. As glass fibers have low specific strength and specific modulus, some volume fraction of carbon fibers (having high specific strength and modulus) should be incorporated in GFRP composite to enhance the mechanical properties and result in a better composite compared to GFRP. But due to low strain to failure and high cost of carbon fibers a balance should be maintained between the cost and performance of the resulting composite. Mechanical properties of E-glass fiber, carbon fiber and epoxy are shown in table 1.

Table 1. Mechanical Properties of E-glass fiber, carbon fiber and epoxy.

Material	Tensile strength (MPa)	Young's Modulus (GPa)	Density (g/cm ³)	Thermal Exp. Co- efficient (mm/ ^o C)
E-glass	2000	80	2.58	5.4
Carbon	2900	525	1.85	2
Epoxy	85	3.5	1.2	1.5-10

The present investigation deals with comparing the mechanical property of high and low temperature conditioned GFRP and hybrid with the unconditioned samples at room temperature. Also comparison is made between the performance of GFRP and glass/carbon hybrid.

2. Materials and experimental methods

2.1 Materials

Present investigation includes fabrication of two types of composite systems i.e. Glass fiber reinforced polymer composite (GFRP) and Glass/carbon fiber reinforced hybrid composite (G/C hybrid) Diglycidyl Ether of Bisphenol A (DGEBA) based epoxy resin was used as matrix and triethylene tetra amine (TETA) was used as hardener. Glass fibers and carbon fibers used in the study were manufactured by Saint Gobain and Toray industries, Japan, respectively. Epoxy resin and hardener were procured from Atul Industries Ltd, Gujarat under the trade name of Lapox L-12 and K-6 respectively. The epoxy to hardener ratio was taken as 10:1 as per the supplier's prescribed standard. GFRP composite laminate was fabricated with 14 layers of woven glass fibers fabric (40 vol. % of reinforcement) by hand layup method. Similarly Glass/carbon (G/C) hybrid contained 5 layers of woven fabric glass fiber (20 vol. %) and 4 layers of woven fabric carbon fiber (21 vol. %) with alternate sequence, keeping glass fiber at both ends by hand layup method. The laminates were then cured in a compression moulding press at 60°C temperature for 20 minutes at pressure of 1 MPa. After removing the laminates, they were dried in an oven at 50 °C for 3 hrs to remove moisture and other volatile entities. Samples were cut using diamond cutter as per the required dimensions as shown in figure 1(a) and 1(b). Samples were then post cured in an oven at 140°C for 6 hrs.

2.2 Experimental methods

2.2.1 Short Beam Shear (SBS) test. The test was performed with 3 point bending fixture in Instron 5967 UTM (shown in figure 1(c)) using ASTM D2344-84 standard specimens to evaluate the apparent ILSS. The specimens were tested at room temperature condition, high temperature and low temperature. In-situ high temperature testing of samples was done in the environmental chamber of Instron 5967 at +70°C and +100°C with the equilibration time of 10 minutes. Ex-situ high temperature conditioning of samples was done in an oven at +70°C and +100°C for 60 hrs. The samples were

allowed to cool in an oven till the temperature comes down to room temperature and then removed out for testing. Ex-situ low temperature conditioning of samples was done at 0°C and -60°C in an ultra-low chamber for 48 hrs. The specimens were removed, brought to room temperature and then tested. Some samples were also conditioned in a liquid nitrogen bath for 8hrs and 24 hrs. The samples were drained and tested immediately. All testing was conducted with loading rate of 1 mm/min.

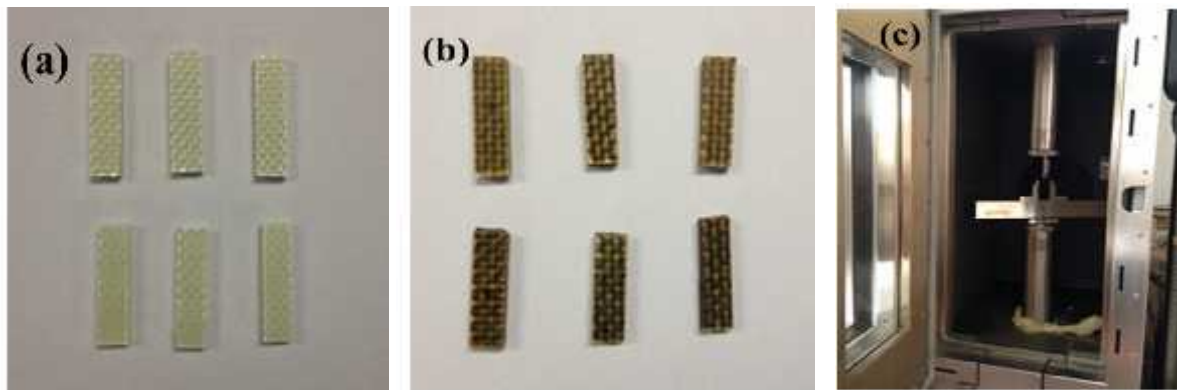


Figure 1. (a) GFRP standard specimens, (b) G/C hybrid specimens and (c) Environmental chamber of Instron 5967 UTM with 3 point bend fixture.

2.2.2 Scanning Electron Microscopy analysis. The fractured surfaces of the tested samples were further analysed for determining the failure mechanisms using JEOL-JSM 6480 LVSEM at 20KV.

3. Results and discussions

3.1 In-situ high temperature conditioning

The samples were tested at +70°C and +100°C with the equilibration time of 10 minutes. The ILSS obtained was compared with that at room temperature.

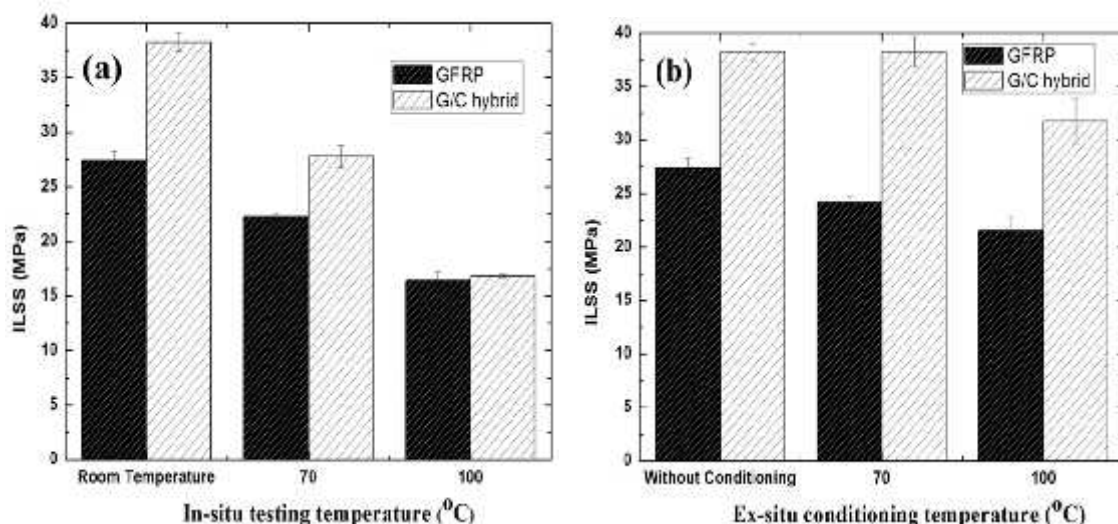


Figure 2. ILSS of GFRP and G/C hybrid samples (a) tested at +70°C and +100°C with 10 min equilibration time and (b) conditioned at +70°C and +100 °C for 60 hrs, then tested at room temperature.

It is observed from figure 2(a) that the ILSS of GFRP was decreased at +70°C and +100°C by 18.9 % and 40 % respectively as compared to that at room temperature. Similarly for glass/carbon hybrid the decrease was 27 % and 56 % at +70°C and +100°C respectively. At elevated temperatures thermal

stresses are induced due to differential thermal expansion of fiber and matrix. These stresses get released on the cost of micro-cracks in the matrix and at the fiber/matrix interface. At low loading rate as used here, these micro cracks get time for coalescence and turn into potentially enough crack to cause reduction in mechanical properties of the composite. It is further observed that ILSS of hybrid is 28.2 % higher over GFRP at room temperature and there is not much difference in ILSS of both the systems at +100°C. The high ILSS in the hybrid samples w.r.t GFRP may be drawn from the presence of carbon fibers which inherently exhibit a higher strength and better interfacial bonding with epoxy. At temperature close to the glass transition of the polymer (T_g is around +120 °C for this polymer), i.e. at +100 °C the mechanical response obtained is mostly matrix dominated. At this stage, the state of the matrix is not capable for effective load transfer. Hence, irrespective of the fiber system the ILSS obtained was nearly same.

3.2 Ex-situ high temperature conditioning

The samples were conditioned at +70°C and +100°C for 60 hrs. ILSS of the conditioned samples was compared with that of samples tested at room temperature. It is observed from figure 2(b) that the ILSS of GFRP decreased at +70°C and +100°C by 11.8 % and 21.5 % respectively as compared to that at room temperature. Similarly for glass/carbon hybrid the decrease was 0.05 % and 17 % at +70°C and +100°C respectively. The quality of interface is poor in GFRP due to weak glass/epoxy adhesion as compared to strong carbon/epoxy adhesion in hybrid. At high temperature the glass/epoxy interface further degrades at a faster rate and acts as stress raisers. These stress raisers ultimately transform into potential micro cracks after certain exposure of time.

3.3 Ex-situ low temperature conditioning

The samples were conditioned at 0°C and -60°C for 48 hrs. ILSS of conditioned samples was compared with that of ILSS at room temperature.

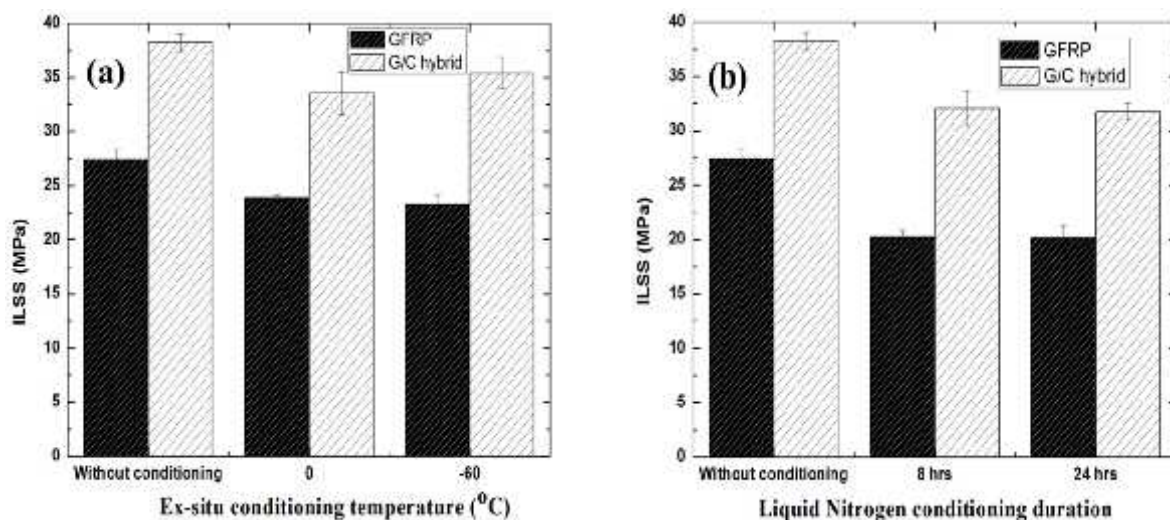


Figure 3. ILSS of GFRP and G/C hybrid samples conditioned (a) at 0°C and -60°C for 48 hrs and (b) in liquid nitrogen for 8 hrs and 24 hrs.

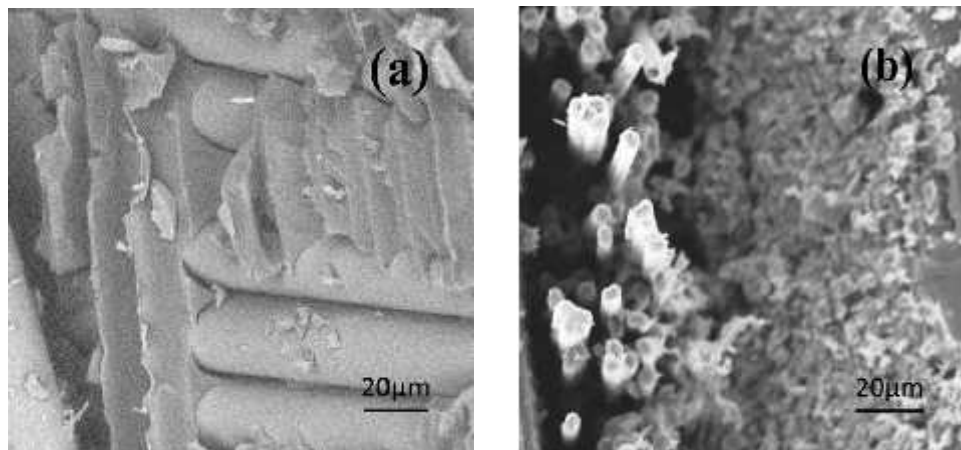
It can be seen from figure 3(a) that in case of GFRP there is reduction in ILSS at 0°C and -60°C by 13 % and 15 % as compared to that at room temperature. Similarly in case of hybrid the decrease at 0°C and -60°C was 12.4 % and 7.5 % with respect to ILSS at room temperature. Under low temperature exposure, polymer matrix becomes too hard and brittle to yield during load transfer from matrix to fibers. Hence this gives rise to formation of micro-cracks at the interface (being a weak region). Thus ILSS decreases. At -60°C the ILSS of hybrid is 34.2 % more than that of GFRP. Hence it can be noted that at cryogenic temperature the performance of hybrid is better than GFRP.

3.4 Liquid Nitrogen conditioning

The samples were kept in liquid nitrogen i.e. at -196°C for 8 hrs and 24 hrs. ILSS of the conditioned samples was compared with that of samples tested at room temperature. It can be seen from figure 3(b) that in case of GFRP there is reduction in ILSS for 8 hrs and 24 hrs by 26.2 % and 26.5 % as compared to that at room temperature. Similarly in case of hybrid the decrease for 8 hrs and 24 hrs was 16.2 % and 17 % with respect to ILSS at room temperature. It should be noticed that for both GFRP and hybrid samples there is a very significant reduction in ILSS for 8 hrs in comparison to ILSS at room temperature, which is then getting saturated (not much difference in ILSS for 8hrs and 24 hrs). The reason may be again attributed to micro crack density formed due to thermal stresses. When the samples were exposed to liquid nitrogen, micro cracks were immediately formed in the material. This micro crack density then gets saturated and no further decrement in ILSS takes place with further exposure time.

3.5 Fractography analysis

After 3 point bend test, fractured surfaces of samples were analysed under Scanning Electron Microscope (SEM) to study various failure mechanisms. Figure 4(a) shows the fracture surface of GFRP tested at room temperature. Initiation of failure could be fiber/matrix debonding at the interface, which upon further loading results in detachment of fibers from the matrix. Extensive fiber imprints on polymer matrix and attachment of matrix ligands on fibers were observed. Figure 4(b) shows fracture surface of G/C hybrid composite. The surface shown here represents matrix region near bundle of carbon fibers. Cohesive failure of matrix is clearly evident and can be attributed for higher interfacial strength of carbon fibers with epoxy resin. Figure 4(c) shows fracture surface of G/C hybrid composite tested at $+70^{\circ}\text{C}$ with 10 minutes equilibration time. Bare fibers with little polymer adherent on their surfaces is visible. This indicates that at high temperature failure is mainly due to loss of integrity of composite because of separation of matrix from fiber surface. Figure 4(d) shows fracture surface of liquid nitrogen conditioned GFRP sample. At this temperature, larger debonded interfaces in terms of delaminated surfaces were observed.



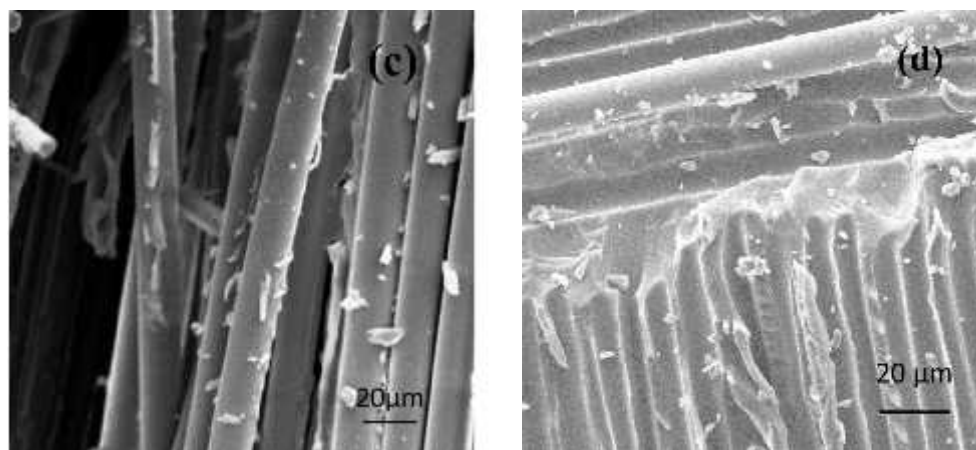


Figure 4. Scanning electron micrographs of fracture surfaces of (a) GFRP tested at room temperature, (b) G/C hybrid composite tested at room temperature, (c) G/C hybrid composite tested for in-situ at +70°C with 10 min equilibration time and (d) Liquid nitrogen conditioned GFRP for 24 hrs.

4. Conclusions

The influence of room temperature, above room temperature and below room temperature on interfacial mechanical property was studied for GFRP and G/C hybrid. The following conclusions can be drawn from the present investigation.

- At room temperature condition though the ILSS of G/C hybrid is more than that of GFRP by 28.2 %, yet the rate of degradation in ILSS for G/C hybrid is more than that of GFRP at high temperature. At +100°C the ILSS of both GFRP and G/C hybrid is found to be nearly equal due to negligible contribution from fiber at elevated temperature and the interfacial property of both the composite materials is governed by the matrix.
- While in-situ conditioning due to attainment of soft glassy state in matrix, stress transmittance through interface becomes less efficient, hence low ILSS is obtained. In case of ex-situ testing although spending more time at high temperature, the material regains its original properties upon cooling to room temperature. Loss in ILSS may now be attributed to the irreversible changes like formation, coagulation and growth of micro cracks at high temperature due to differential thermal changes between fiber and matrix.
- Decrease in ILSS is more in case of low temperature conditioning of GFRP than G/C hybrid. The ILSS of hybrid is 34.2 % (in contrast to 28.2 % without conditioning) more than that of GFRP when conditioned at -60°C for 48hrs.
- Liquid nitrogen conditioning for 8 hrs showed decrease in ILSS of GFRP and G/C hybrid by 26.2 % and 16.2 % respectively. Further conditioning for 24 hrs showed negligible change in ILSS due to saturation of density of micro-cracks with long exposure time. Hence it can be noted that at low temperature the performance of hybrid is better than GFRP.

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