

Investigation on the tensile properties of glass fiber reinforced polymer composite for its use as a structural component at cryogenic temperature

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Abstract

Polymer composites, especially glass fiber reinforced polymer (GFRP) are finding ever-increasing applications in areas such as superconductivity, space technology, cryogenic rocket engines, and cryogenic storage vessels. Various components made of polymer composites are much lighter than their metallic counterparts but have equivalent strength for ultra-low temperature applications. In this paper, we have investigated the tensile properties of an indigenously prepared unidirectional cylindrical hollow composite tube for its use as a neck of the cryogenic vessel. XRD and SEM of the tube are completed before cryogenic conditioning to ascertain the fiber and resin distribution in the matrix. The result shows that for composites, after 15, 30, 45, and 60 minutes of cryogenic conditioning at 77K in a liquid nitrogen bath, the strength and modulus increase significantly with the increase of strain rate and reach the optimum value for 45 minutes of conditioning.

The results are encouraging as they will be helpful in assessing the suitability of GFRP in the structural design of epoxy-based components for cryogenic applications.

Keywords: GFRP, epoxy resin, composite, tensile, cryogenic

1. INTRODUCTION

Fiber-reinforced polymer composites in general and glass fiber-reinforced polymer (GFRP) in particular are used in many industries requiring extensive characterization and understanding of their behavior in ambient conditions [1-3]. The response of the material can change drastically when exposed to extremities both within and beyond the Earth's atmosphere that require structures to operate at ultra-low temperatures in the cryogenic range. Liquid propellant tanks for space programs, satellite launch vehicle structures; aircraft structures at cruising altitudes; support elements and devices operating at cryogenic temperatures, and arctic exploration structures are some of the areas where composite structures have to encounter extremely low temperatures.

Hence, it is necessary to characterize composites at cryogenic temperature to know the effect of low temperatures on composite materials towards designing the structural components based on polymer composites to be operated down to cryogenic temperature.

Composites are composed of two different materials having resultant properties far superior to that of individual materials. Composites are macroscopically homogeneous, i.e., the distinguishable behavior of the components cannot be seen through the naked eye and hence will act together as a unit [4, 5]. The main constituent of a composite is reinforcement which is responsible for load bearing whereas the matrix is the medium that binds the

reinforcement and the interface is the common surface that contacts the reinforcement and matrix. Different composites can be categorized based on the type of matrix medium [6]. Glass-Fibers and epoxy resin are considered for our composite preparation. Glass fibers are the most versatile industrial materials as they are readily available and possess some useful bulk properties such as resistance to any form of chemical attack, hardness, stability, transparency, and inertness. In addition, they also show good fiber properties such as stiffness, flexibility, and strength [7]. Epoxy resin is a kind of reactive prepolymer referred to as poly polyepoxides [8]. Glass fiber-reinforced polymeric (GFRP) composites are utilized in the manufacturing of composite materials [9]. The strength of the fiber and its modulus play a crucial role in the physical properties including chemical stability and mechanical behavior in the resultant composites.

Matrix and the interface acting between the fiber and the matrix are responsible for the proper transfer of the stress [10].

The proper composition along with the orientation of the fibers made the GFRP properties comparable to steel, with stiffness higher than that of aluminum, and specific gravity one-quarter that of steel [11]. Mechanical properties of the composites can be improved by different glass fiber reinforcements like the woven mat, longitudinal, chopped mat, etc. [12]. The present work comprises the fabrication of typical glass-fiber composite tubes for use at the neck of a cryogenic container followed by their characterization for determining physical and tensile properties and also testing its optimum strength before and after cryogenic

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temperature conditioning during different time. Thermal stability, modulus, area of the elastic and plastic region, ductility, stiffness, strength at yield, and strength at break of the composites after liquid nitrogen conditioning are the main properties considered for the characterization.

2. MATERIALS AND EXPERIMENTAL METHOD

2.1. Materials

The special type of epoxy, EP3 is provided by a Fiberglass fabricator, Hoogly-712 233, India. The fabrication of composite tubes is done in collaboration with Industrial Molder and Fabricator, Howrah, India. The appropriate sample size is prepared by cutting one of the tubes with the help of *Discoplan-TS*, a precision cutting and grinding machine for mineralogical, petrographic, and ceramic thin sections supplied by Aimil Ltd, New Delhi, India.

2.1. Glass Fiber Reinforced Polymer (GFRP)

Glass fiber is isotropic in nature [13, 14]. E-glass, S-glass, C-glass, and AR-glass are popular kinds of glass fibers. Lightweight, well resistant to water and chemicals, low cost, and high strength are the main characteristics of glass fiber. Relatively low cost compared with other types of FRPs makes glass fiber most suitable for composite making. GFRP laminates are manufactured from plain woven E-glass laminates. The chemical composition of glass-fiber in wt% is SiO_2 (55.0), Al_2O_3 (14.0), TiO_2 (0.2), B_2O_3 (7.0), CaO (22.0), MgO (1.0), Na_2O (0.5), K_2O (0.3) [15]. The major drawbacks of glass fiber are low elastic modulus, and low resistance to alkaline with long-term strength due to stress rupture. On the other hand, lightweight to the high strength of glass-fiber (E-glass) is a very attractive property for which E-glass will always be in demand for industry and researchers. Fig 1(a) shows the geometrical illustration of the composite tube with its dimension. Fig 1(b) shows the top view (left one) and side view (left one) of the GFRP-EP3 composite tube

2.3. Epoxy Resin

Epoxy resins are the most commonly used substrates due to their good mechanical characteristic, thermal properties, corrosive resistance, versatility, and durability [16, 17]. EP3-type epoxy is used for fabricating the desired composites. It is a kind of synthetic resin that could be utilized as an adhesive, coating, casting plastics, and matrix resin for FRPs. EP3 is a breakthrough in epoxy adhesive technology featuring the properties like high shear and high cure speed. It offers superior resistance to impact, thermal shock, corrosion, creep, vibration, and stress fatigue cracking while maintaining moisture and thermal resistance even for cryogenic application down to -196°C (77K).

EP3 produces durable and exceptionally tough bonds which are remarkably resistant to serve thermal cycling and many chemicals including water, oil, and fuels. The hardened adhesive is a superior thermal insulator. EP3 offers the convenience of a heat cure with no mixing prior to use and uniquely favorable bonding performance for

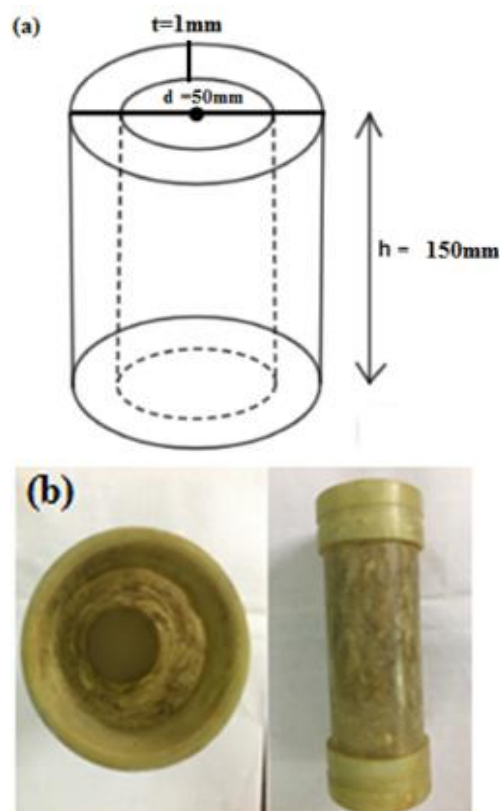


Fig .1. (a) Dimension of the composite tube and Fig1 (b) Pictorial top view(left one) and side view(right one) of the composite tube.

even the most difficult applications in the aerospace, composite, and chemical industries.

2.4. Fabrication of GFRP-EP composites

Glass fiber is available in the woven form but for enhancing its mechanical strength at low temperatures and giving it a perfect shape with reasonable strength, glass fiber-impregnated resin is introduced [18,19]. The entire fabrication is accomplished with the help of a professional Industrial Moulder and fabricator. The epoxy used for the fabrication of the composite is EP3. The W/W ratio for the fabrication of reinforced glass fiber cloth is 70% fiber cloth with 30% Epoxy Resin.

The method of manufacturing experimental glass fiber composite tubes can be described as follows.

A male mandrill is first layered with epoxy resin and then wrapped with a glass fiber cloth. The packing is tightened followed by layering with epoxy resin and then clamping with a female mandrill. The sequence is repeated to achieve the desired thickness. The assembled system then undergoes a heat-curing process [20]. The composite tube is further allowed to be hardened at room temperature. The composite is cut into the required size by an electrical saw in a thin and polished section preparation unit for tensile testing as per ASTM D638 standard [21]. After that, a piece of composite is weighed and then heated in a muffle furnace to ensure the desired percentage of Epoxy resin and glass fiber following ASTM D2584 standard [22].

The dimensions of the GFRP composite tube manufactured are given in Table 1.

TABLE I
DIMENSION OF THE COMPOSITE TUBE

Dimension	Values
Length(mm)	150
Thickness(mm)	1
Inner Diameter(mm)	48
Outer Diameter(mm)	50

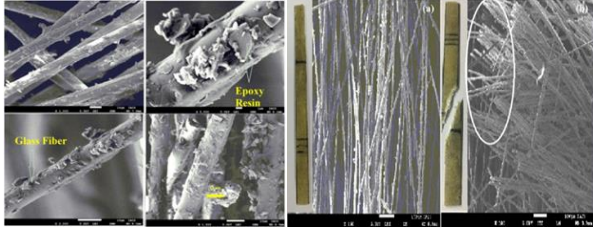


Fig. 2. (a) GFRP-EP3 composite, (b) SEM image on fibers orientation of the untreated samples(left one) and Cross-sectional view of fibers head of the untreated fiber composite after (right one) tensile test.

2.5. Physical properties

Knowledge of physical properties for determining the structural morphology of the sample is a very useful guideline to know the distribution of fiber in the matrix [23] and hence Scanning Electron Microscope (SEM) and X-ray diffraction (XRD) are performed on the sample tube at room temperature are completed to ascertain the fiber and resin distribution in the matrix of the materials to be used for subsequent tensile testing with or without cryogenic conditioning .

2.5.1. Scanning Electron Microscope (SEM)

Surface morphologies of glass fiber-impregnated resin composite films are studied by means of a scanning electron microscope (SEM: JEOL Model JSM-6490) with an accelerating voltage of 15 kV. Fibrous mesh of glass fiber is mounted on aluminum stubs and gold coated to avoid electrical charging during the examination. All the SEM images are taken at the magnification of 5 KX. Fig 2(a) shows the epoxy resin embedded in the fiber of the matrix and Fig 2(b) exhibits the cross-sectional view of the fractured fiber after the tensile test.

2.5.2. X-Ray Diffraction (XRD)

X-ray diffraction (XRD) study is carried out using PHILIPS SHIFFERT 3710 diffractometer supported by Lynx Eye super-speed detector and Ni-filtered Cu-K α ($\lambda=0.15406$ nm) radiation is generated at 40 kV/ 40 mA. The diffractometer is operated with a scan speed of 0.5s for steps of 0.02 in 2θ (degree) at room temperature. Figure 3(a)&(b) shows the GFRP with EP3 and normal GFRP.

2.6. Cryogenic conditioning

Five samples are prepared from one of the composite tubes for testing. The size of the specimen is 100mm x 20mm with a gauge length of 40mm. One sample is kept untreated at room temperature (RT) and other samples are immersed in liquid nitrogen in four different Dewar for cryogenic conditioning (CT) for 15, 30, 45, and 60 minutes to allow sufficient time for the change of properties [24].

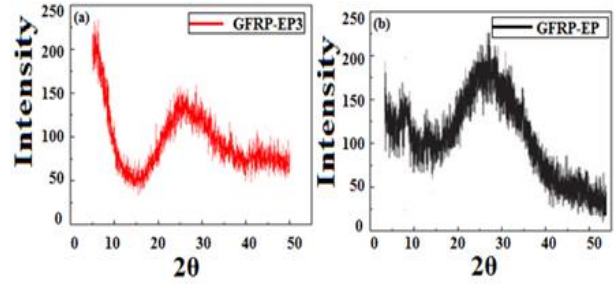


Fig. 3. (a) GFRP with EP3 laminates (b) Normal GFRP laminates.

Then the treated four samples are withdrawn from liquid nitrogen and kept in the desiccator so as to protect them from absorption of moisture. The conditioned samples are then promptly transferred to the testing system [25].

2.7. Tensile properties

The static tensile properties are obtained using a computer-controlled universal testing machine, INSTRON 5582 with a 100kN load cell as shown in Fig 4(a). ASTM D638 standard is followed for the testing. Room temperature (RT) is recorded as $25\pm 3^{\circ}\text{C}$ and the specimens are loaded at a constant speed of 1mm/min until breaking. To reduce the slippage problem, tabs from plane glass-fiber laminates are introduced on the GFRP-EP3 specimens. For data acquisition, Blue Hill 2 software is used. The tensile test is then performed to measure the characteristic curve, the modulus, and ultimate strength at yield and breakpoint [26, 27]. The average deformation energy for five samples is analyzed with optical devices.



Fig. 4. (a) Universal Tensile Machine(top) Bottom Fig 4(b) Types of delamination and Fractured surface of the untreated composite before and after tensile loading of GFRP-EP3 composite.

Fig 4(a) is the pictorial view of the Universal Tensile Machine (top) Fig 4(b) shows the types of fracture developed in untreated composite specimens before (middle) and after tensile loading(bottom) Figure 5 (a) represents Tensile stress vs Strain, and Figure 5(b) represents Load vs. Deflection of the GFRP-EP3 composite without conditioning Figure 6(a) to Figure -9(a) represent Tensile stress vs Strain of GFRP-EP3 composite with conditioning for 15 minutes, 30 minutes, 45 minutes, and 60 minutes respectively. Figure 6(b) to Figure -9(b)

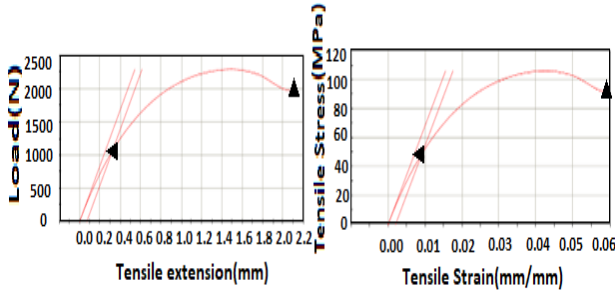


Fig. 5. (b) Tensile stress vs Strain, (a) Load vs. Deflection of GFRP-EP3 composite without conditioning.

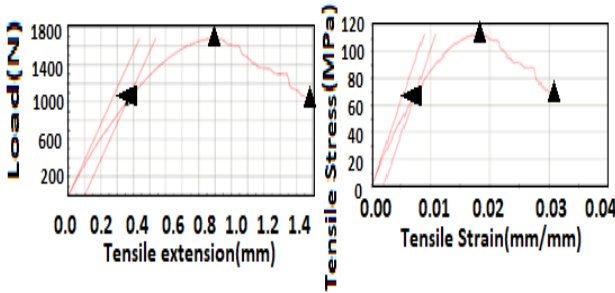


Fig. 6. (b) Tensile stress vs Strain, (a) Load vs. Deflection of GFRP-EP3 composite conditioned for 15 minutes

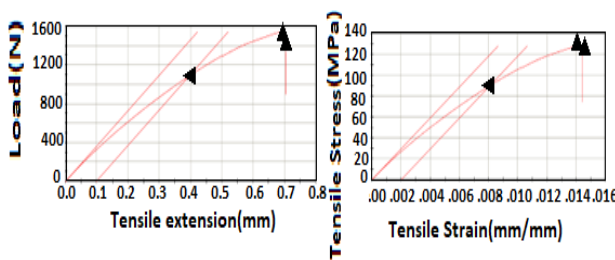


Fig. 7. (b) Tensile stress vs Strain, (a) Load vs. Deflection of GFRP-EP3 composite conditioned at 30 minutes.

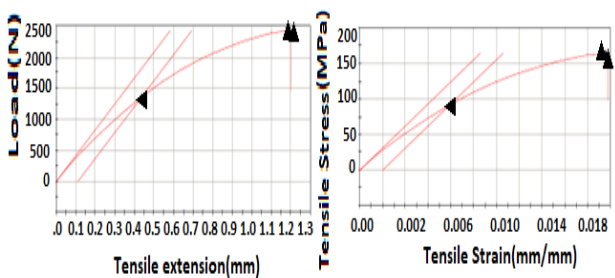


Fig. 8. (b) Tensile stress vs Strain, (a) Load vs. Deflection of GFRP-EP3 composite conditioned at 45 minutes.

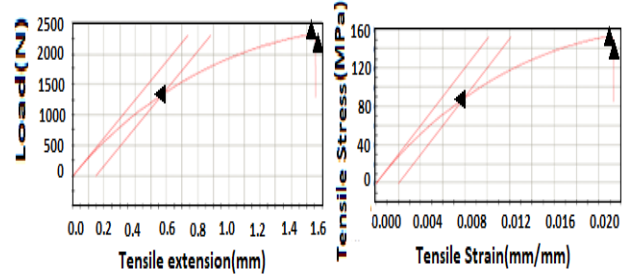


Fig. 9. (b) Tensile stress vs Strain, (a) Load vs. Deflection of GFRP-EP3 composite conditioned for 60 minutes.

TABLE 2
TENSILE PROPERTIES OF THE COMPOSITE SAMPLES BEFORE AND AFTER CRYOGENIC CONDITIONING FOR 15, 30, 45, AND 60 MINUTES

Composite sample (GFRP-EP3)	Young's Modulus (MPa)	Tensile Stress at Tensile Strength (MPa)	Tensile Extension at Break (mm)	Load at Break (kN)
Unconditioned	6835.82	90.85	2.08	1.97
Conditioned for 15 minutes	12699.14	113.65	1.48	1.00
Conditioned for 30 minutes	14885.56	128.39	0.70	1.49
Conditioned for 45 minutes	15532.91	164.15	1.20	2.35
Conditioned for 60 minutes	15112.47	152.21	1.55	2.13

represent Tensile stress vs Strain of GFRP-EP3 composite with conditioning for 15 minutes,30 minutes,45, minutes and 60 minutes respectively

Tensile properties of the composite samples before and after cryogenic conditioning for 15, 30, 45, and 60 minutes are given in Table 2.

3. RESULT AND DISCUSSION

SEM and XRD are carried out at room temperature for the composite material to ascertain its structural orientation with respect to resin and fiber before going for tensile properties determination.

Fig 2(a) gives an SEM image where we clearly visualize glass fiber and the epoxy resin on it. The microscopy of the composites is performed using a scanning electron microscope and proper impregnation of glass fiber with epoxy resin. The retention of epoxy Fig 2.(a) on glass fiber is observed on the bamboo shoots like fiber [28]. For characterization, fibers of the composite are dismantled from composite laminates. The traces of epoxy over glass fiber indicate the improved thermal stability of composites. This finding can also be confirmed by other thermal analyses like TGA, low-temperature thermal conductivity, etc. Generally, the thermal stability is improved due to the strong interface of epoxy resin with glass fiber. Even the resin reduces the void content from the reinforced matrix in the heat cure process. The matrix interface improves the mechanical properties of the composites. The developed composites (fiberglass-reinforced polymer) are ideal materials for applications in cryogenic temperatures. Fig

2(b) shows the fracture type of the untreated composite (right one) before and after continuous tensile loading, and the nature of delamination of the fiber composite(right one).

The orientation and loading of glass fibers in the epoxy resin and the identification of peaks of reinforced glass fiber and its characteristics in the matrix are further studied using x-ray diffraction analysis, as shown in Figure 3. Fig 3(a) shows the glass fiber-impregnated resin and Fig 3(b) shows plane glass fiber for comparison with examined reinforced polymer. X-ray diffractograms of oriented glass fiber fig 3(a) are represented by the majority of elements. Fig 3(a) also shows a peak at Bragg's s angle $2\theta=26^\circ$, affirming the crystalline silica in the composite. It can be seen that the glass fiber and epoxy resin do not have any other identifiable XRD materials that are amorphous in nature [29, 30]. This unidentifiable peak appears for composites' development of fiber loading and orientation with resin. Though Fig 3(b) shows the sharp peak at $2\theta=27^\circ$ of silica it has a similarity with the examined reinforced polymer. Therefore, it is established that the dispersion of glass fiber is uniformly distributed in the matrix and hence no specific peak in Fig 3(a).

Tensile properties are investigated with cryogenic conditioning for different durations of time as well as for a sample without cryogenic treatment The results are discussed below

After tensile loading, types of fractures to failures, and delamination of fibers are also studied. The influence of liquid nitrogen treatment on the strength of composites is studied. Table 2 shows the result of Young's modulus from the stress-strain curve of epoxy-based glass fiber-reinforced polymer before and after cryogenic conditioning for 15, 30, 45, and 60 minutes. The stress-strain curve and load-deflection curve of different temperatures are plotted which clearly indicate the enhancement of strength of the composite after cryogenic temperature conditioning (CT-C) for up to 45 minutes but beyond that duration, the modulus is slightly decreased for 60 minutes of conditioning and hence 45 minutes duration is considered as the optimum duration of treatment. It can be seen from Table-2 that Young's modulus of GFRP/epoxy composites after CT-C is increased up to 45 minutes conditioned to a great extent from its strength at room temperature (RT). The possible mechanism may be due to the mobility of the molecules of the polymer chain when the temperature is decreased and the binding forces between the molecules increase at low temperatures, this happened when the samples are conditioned for 45 minutes. After CT-C, the relaxation polymeric chain of the composite seems to be completely arrested resulting in increased tensile properties, like modulus, stiffness, and strength. So, it is revealed that Young's modulus of GFRP-EP will be higher after CT-C at 45 minutes [31- 36]

Table-2 also exhibits the tensile strength and load at break from the load-tensile extension curve of epoxy-based glass fiber reinforced composite polymer tube, before and after CT-C at different times. Modulus, tensile strength, and load after CT-C at 77k are higher than at RT. The reason behind the increase in strength and load is the binding forces between the molecules of the epoxies which becomes stronger at 77k conditioning. So, compared to

Epoxy Resin, GFRP, GFRP-EP and CFRP, the modulus and strength of the GFRP-EP3 composite after CT-C is higher [37-39].

4. CONCLUSION

The tensile properties of GFRP-EP3 laminates before and after CT –C at different durations have been evaluated in this article. The tensile-related mechanical properties are increased after cryogenic conditioning. At room temperature, the loss in tensile strength is mainly due to the epoxy resin softening of the composites, and the stiffness loss is attributed to the straightening of glass fibers as the epoxy resin softens. Results of low-temperature conditioning indicate that there is a considerable effect on the tensile strength between conditioned and unconditioned composites but there is a significant reason behind the duration of conditioning. The higher duration of the condition results in a higher modulus. It is clear that the increase of tensile modulus in the specimen with prior treatment with liquid nitrogen is two times higher than the unconditioned one and 45 minutes duration is considered as the optimum duration. The load at break is higher incase of 45 minutes of conditioning than 60 minutes of cryogenic conditioning. (table 2). In addition, treated laminates show brittle behavior. The stress-strain curve demonstrates that the area of the plastic region of the conditioned composite is larger than the unconditioned one, but its deformation is very abrupt due to increased brittleness in the treated specimen. The stress-strain, load vs. temperature, and load vs. deflection graphs reveal that for composites, after 45 minutes of cryogenic conditioning at 77K in a liquid nitrogen bath, the strength and modulus increase significantly with the increase of strain rate. Therefore, the present findings highlight the structural survivability of GFRP-EP3 at extremely low temperatures in the cryogenic range as well as at room temperature as results will be helpful in assessing the suitability of GFRP in the structural design of epoxy-based components for extremely low-temperature environments. Hence, GFRP-EP3 can safely be used for many industrial applications both at room temperature and cryogenic temperature including its use as a neck of cryogenic vessel.

NOMENCLATURE

- RT**- Room Temperature
- CT**- Cryogenic Temperature
- CT-C**- Cryogenic Temperature Conditioning
- CFRP**-Carbon Fiber Reinforced Polymer
- MWCNT**-Multi Wall Carbon Nano Tube
- SEM**-Scanning Electron Microscope
- XRD**-X-Ray Diffraction
- UTM**-Universal Tensile Machine
- KN** – Kilo Newton
- EP3**-Special type epoxy resin
- ASTM**- American Society for Testing and Materials
- FRP**—Fiber Reinforced Plastic

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