An assessment of flexural performance of liquid nitrogen conditioned glass/epoxy composites with multiwalled carbon nanotube

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Abstract

In the present investigation, alteration in flexural performance of glass/epoxy composite (GE) and CNT (0.1%, 0.3% and 0.5%)-GE composites due to liquid nitrogen conditioning is studied for various time lengths. The epoxy resin is first modified by 0.1, 0.3 and 0.5 % MWCNT, which was then used along with E-glass fibers to fabricate laminates. Flexural strength and modulus were evaluated by 3 point bend test. Out of these four compositions, the maximum strength in the as-fabricated condition was obtained for GE composite with 0.1 % CNT, which is 32.74 % higher as compared to GE composite. Decrease in strength and modulus was observed for short time span of liquid nitrogen conditioning i.e. 0.25 hr. Long cryogenic conditioning resulted in increment in strength. To understand the failure mechanisms, post failure analysis was carried out using Scanning Electron Microscope (SEM). The design parameters are calculated using Weibull distribution model.

Keywords: Fiber Reinforced Polymer (FRP) composites, liquid nitrogen conditioning, mechanical properties, Carbon nanotubes (CNTs).

1. Introduction

Fiber Reinforced Polymer (FRP) composites are one of the most promising and reliable materials in today’s world. Their unparalleled properties like high strength to weight ratio, low density, high corrosion resistance, high specific strength and modulus, etc. earn the responsibility for making FRP composite a word of every mouth. FRP composites find application in aerospace industry, marine vehicles, sports goods, structural applications, cryogenic fuel tanks, hydrogen storage tanks, pressure vessels, satellite solar panels, superconducting devices, thermal insulators, etc. FRP composites have been proved to be a better choice of material in replacing metallic materials in various cryo applications mostly cryo fuel tanks for its efficient storage and transportation. This is due to their desirable properties like high resistance to corrosion, high specific strength, specific stiffness, etc. (1). Due to this increasing demand of FRP composites for its application in cryo tanks for storing liquid oxygen, liquefied natural gas (LNG) and liquid hydrogen (2) it is necessary to identify and avoid potential failure modes like cracks, etc. for avoiding leakages (causing undesirable pressure drop). Hence improvement in these composites can be brought by fabricating nano-engineered FRP composites. The performance of these materials is adversely affected when subjected to various environmental conditions like humidity (3,4), elevated temperatures (5), low and cryogenic temperatures (6), etc. Every field of science is taking advantage of nano-technology due to its out throwing properties and so do the

FRP composites (7). Modifying the matrix using nano-fillers is a recent trend observed in fabrication of FRP composites to enhance the matrix dominated properties (8). Carbon nanotube (CNT) as a nano-filler has been the first choice of many materials researchers (9,10). Allaoui et al. (11) studied the mechanical and electrical properties of MWCNT/epoxy nano-composite and stated that addition of 1 wt. % CNT in epoxy has increased the young's modulus and yield strength by 100 and 200 % respectively. Wang et al. (12) suggested that the addition of CNT in phenolic resin enhances its high temperature ablation resistance. Lu et al. (13) reported enhanced glass transition temperature and mechanical strength on addition of 0.25 wt.% of 3-glycidoxypropyltri-methoxysilane functionalized multi-walled carbon nanotubes. Further, few studies suggested the cryogenic property enhancement on adding CNTs to polymer resin. Chen et al. (14) evaluated the cryogenic properties of CNT modified epoxy resin and noted improvement in strength as compared to that of neat matrix. Wei et al. (15) studied the flexural fatigue performance of CNT based polymer composites and found that as the temperature was reduced from room temperature to 77 K, there was increment in its fatigue resistance and fatigue performance was affected by the content of CNT used. Takeda et al. (16) investigated cryogenic mechanical properties of CNT modified woven glass/epoxy composites under tensile and fatigue loading. They observed that there was hardly any improvement in young's modulus and ultimate tensile strength on addition of CNT, but noticed improvement in fatigue resistance. Thus they demonstrated that addition of CNT has a potential to increase matrix dominated properties of composite materials at cryogenic temperatures. To the best of our knowledge, very little literature is available on cryogenic treatment of nano-engineered FRP composite for short and long term exposure duration. Hence there arrives a need to combine all these above stated positive effects by fabricating a composite whose matrix is modified with CNT and thus evaluate its flexural performance after liquid nitrogen treatment for certain durations of time.

For effective design and analysis, handsome amount of experimental data has to be collected on effect of thermal and mechanical environment on these composites. Hence this paper intends to provide an initial set of data and fabrication technique for improving FRP composite for cryogenic application.

Objective of present investigation is to study the flexural behaviour of glass/epoxy (GE) composite and MWCNT modified GE (CNT-GE) composites after exposing them in a cryogenic environment (liquid nitrogen) for various time durations. The study involves varying the amount of CNT in GE composites and evaluating their response for different cryogenic exposure time. Further their damage constitutive model using Weibull probability function is adopted to obtain shape parameter and scale parameter.

2. Experimental

2.1 Materials

Diglycidyl ether of Bisphenol A (DGEBA) type epoxy resin was used as matrix and Triethylene tetra amine (TETA) as hardener. Both were supplied by Atul Industries Ltd, India under the trade name of Lapox L-12 and K-6 respectively. The 3K plain weave glass fiber having filament diameter of 15 µm was supplied by Saint Gobain, India. It served the need for reinforcement. MWCNTs having an outer diameter of 6-9 nm and 5 µm length were supplied by Sigma-Aldrich. Some salient properties of the constituents of laminated composite are provided in Table-1.

Table-1Salient properties of epoxy and glass fiber

Property	**Epoxy**	**Glass fibres**
Density (g/cm^3)	1.162	2.58
Tensile modulus (GPa)	4.1	72.3
Tensile strength (GPa)	0.11	3.4
Strain at failure (%)	4.6	4.8
Poisson’s ratio	0.3	0.2

Specifications of glass woven fabric-
Warp and weft density: 16 and 14 yarns/inch,
Fabric weight: 360 gsm.

2.2 Fabrication process

2.2.1 Dispersion of MWCNT into epoxy resin

Before commencing the hand lay-up method, in order to fabricate MWCNT reinforced glass/epoxy (CNT-GE) composite, the epoxy resin was modified by incorporating MWCNT to it. The amount of CNT in CNT-GE composite was varied as 0.1 wt. %, 0.3 wt. % and 0.5 wt. % of epoxy. Three different laminates were fabricated using these different compositions. Required amount of CNT was dispersed in 150 mL of acetone. This suspension was stirred for 30 minutes at 1000 rpm using magnetic stirrer. Sonication of this suspension was done for 30 minutes. The purpose of stirring and sonication is to deagglomerate the existing agglomerates of CNTs. This CNT/epoxy mixture was added to pre-weighed epoxy, which was brought to sufficient fluidity by heating. Further, stirring of epoxy/CNT/acetone mixture was done at 1000 rpm for 2 hr at 70 °C. Sonication was again carried out at 70 °C for 1 hr. At this stage, evaporation of all acetone was assured. The left epoxy/CNT suspension was vacuum degassed for 12 hr to facilitate removal of air bubbles entrapped in the suspension during earlier stages.

2.2.2 Fabrication of Fiber reinforced nano-composites

Specified amount of hardener (10 wt. % of epoxy) was blended and stirred properly in the epoxy/CNT suspension. Matrix and fibers were approximately in 1:1 proportion by weight. The laminates were prepared by hand lay-up process using 14 layers of woven fabric E-glass fibres. Curing was done at 60 °C temperature and 10 kg/cm^2 pressure in a hot press for 20 minutes. Likewise, using the same parameters as used in CNT-GE composite, the control glass fiber/epoxy (GE) composite laminate was fabricated using neat epoxy and 14 layers of glass fiber by hand lay-up method followed by hot pressing. The laminates were allowed to keep at room temperature for 24 hrs. Flexural (as per ASTM D7264) samples were cut from the prepared laminates with a diamond cutter. The samples were then post-cured at 140 °C for 6 hr (17).

2.3 Material characterization

2.3.1 Mechanical characterization

All the samples of different compositions were dipped in liquid nitrogen for different conditioning time of 0 hr (no conditioning), 0.25 hr, 1 hr, 4 hr and 8 hr. The samples were drained out and instantaneously tested at room temperature for flexural test using 3 point bending fixture of Universal Testing Machine (Instron 5967) as seen from Figure 1 (a) following the ASTM D7264 standard. The loading rate was kept at 1 mm/min.

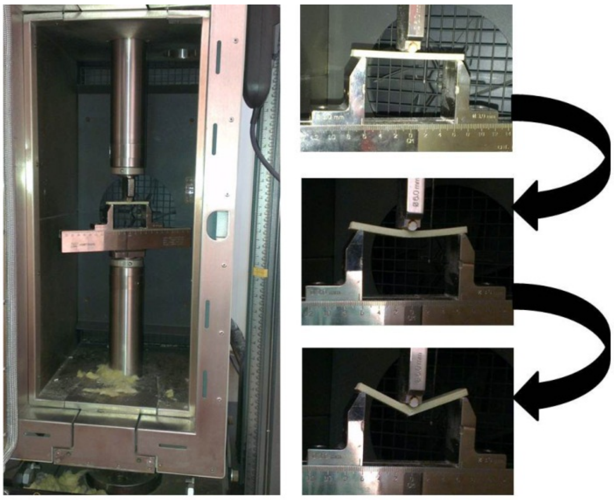

Figure 1. *Experimental Setup for Flexural test.*

2.3.2 Fractographic analysis

To analyse the failure modes of composites, their fracture surfaces were observed under Scanning Electron Microscope (SEM) with JEOL-JSM 6480 LVSEM operated at 20KV. The dispersion of MWCNTs in epoxy in case of CNT-GE composite is observed under Field Emission SEM. The surfaces of fractured samples were coated with a thin film of gold for increasing its electrical conductivity.

3. Results and Discussion

3.1 Flexural performance after cryogenic treatment

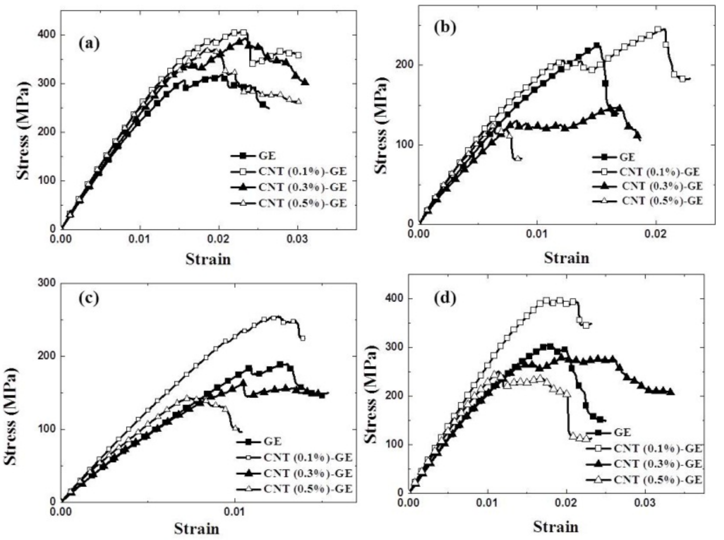

Figure 2. *Flexural stress-strain curves for GE and CNT-GE composites (a) in as fabricated condition and after conditioning in liquid nitrogen for (b) 0.25 hr, (c) 1 hr and (d) 4 hr.*

Figure 2 shows flexural stress vs. strain curves for GE and CNT-GE (all the compositions) samples conditioned in liquid nitrogen for 0 hr, 0.25 hr, 1 hr and 4 hr. The flexural properties i.e. flexural strength and flexural modulus are plotted against conditioning time at various amount of CNT content as shown in Figure 3 and Figure 4.

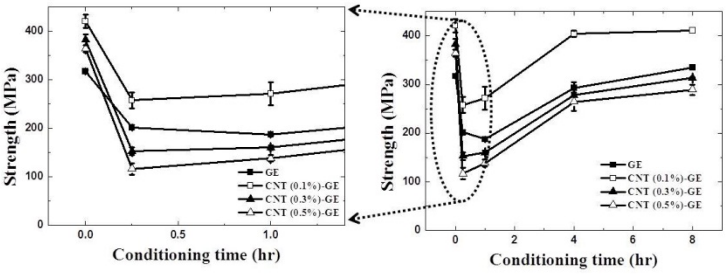

Figure 3. *Variation in flexural strength for GE and CNT-GE composites with conditioning time in liquid nitrogen.*

It is inferred from Figure 3 that on addition of CNT, the flexural strength increases in its as fabricated condition. The maximum increase in strength obtained among all the four compositions without conditioning is 32.7 % in case of CNT (0.1 %)-GE composite as compared to GE composite as shown in Figure 6. The reinforcement efficiency (ratio of increase in strength to amount of CNT content) obtained here is 1038 MPa per % CNT which is quite close to the reinforcing efficiency of 1000 MPa per % CNT obtained by Rahman et al. (18) on fabricating amino functionalized-CNT reinforced glass/epoxy composites using calendaring technique with same CNT content. This may be due to availability of enormously high CNT/epoxy interfacial area due to high surface area of

CNTs and hence more stress/load transfer from the soft polymer to the strong CNTs across the interface takes place. Thus results in more stress required to break the sample. On the contrary, the strength of CNT (0.3 %)-GE and CNT (0.5 %)-GE composite decreases at room temperature as compared to CNT (0.1 %)-GE composite due to agglomeration of CNTs at certain sites to reduce high surface energy. These agglomerated CNTs will not be able to contribute at a nano-scale and micro-level properties will be obtained i.e. low CNT/epoxy interfacial area will result. Hence this will lead to decrement in strength.

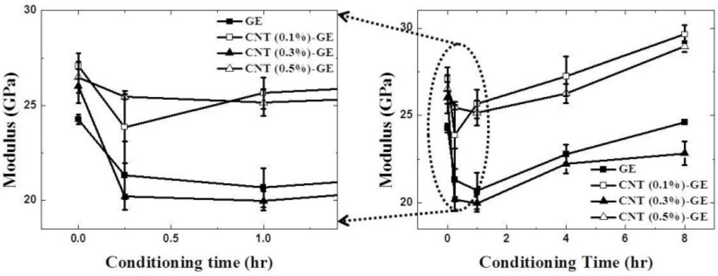

Figure 4. *Variation in modulus for GE and CNT-GE composites with conditioning time in liquid nitrogen.*

From Figure 4 it can also be seen that modulus also follows a similar trend as that of strength for all the composite systems.

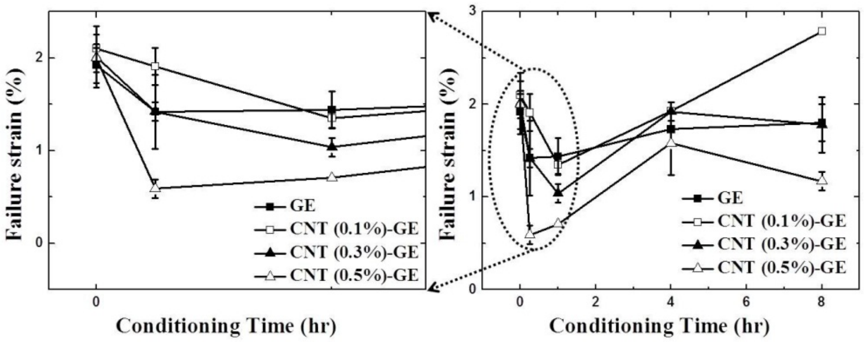

Figure 5. *Variation in failure strain for GE and CNT-GE composites with conditioning time in liquid nitrogen.*

Figure 5 shows the variation in failure strain with liquid nitrogen conditioning time for various MWCNT content in the GE composites. This suggests that the ductility of all the composites drops significantly due to sudden cryogenic exposure. 0.25 hr shock conditioning makes polymeric chains frozen and results in drastic matrix embrittlement. After 1 hr of conditioning a noticeable enhancement in the ductility was ensured for all composite systems upto 4 hrs of conditioning. A further longer conditioning time brings no change in ductility for GE composites with 0 and 0.3 wt% of CNT, whereas the trend still continues to go up even till 8 hrs in case of GE composite with 0.1 wt% CNT.

To visualize the effect of CNT addition into GE composites, the enhancement in mechanical properties are shown in figure 6. This clearly shows that out of these 4 compositions, GE composite with 0.1 wt% CNT shows the best combination of strength with stiffness and ductility.

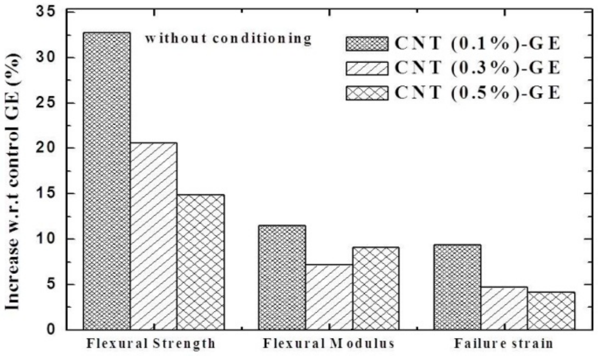

Figure 6. *Improvement in flexural strength, flexural modulus and failure strain after incorporating MWCNT in GE composite*

Exposing the samples for 0.25 hr in liquid nitrogen caused a drastic reduction in strength and modulus for all the composites. The samples may have experienced thermal shock when suddenly brought to -196°C from room temperature (20°C). In case of GE composites, this sudden exposure to liquid nitrogen may have generated localised tensile residual stress at the glass/epoxy interface due to differential thermal contraction between E-glass fibers (5-12 × 10^{-6} K^{-1}) (19) and epoxy (6.2 × 10^{-5} K^{-1}) (20). This might have resulted in generation of micro-cracks, voids at the interface. In addition to this interface, CNT/epoxy interface also exists in case of CNT-GE composites and there is a significant probability of interfacial debonding at CNT/epoxy interface again due to differential thermal coefficient of expansion between CNT (0.73–1.49 × 10^{-5} K^{-1})

(14) and epoxy (6.2×10^{-5} K^{-1}) (20). Hence the rate of relative degradation in strength and modulus is expected to be higher for CNT-GE composites than GE composites due to short liquid nitrogen exposure. The strength dropped by 36.3 %, 38.8 %, 60.1 % and 68 % in case of GE composite, CNT (0.1%)-GE composite, CNT (0.3%)-GE composite and CNT (0.5 %)-GE composite respectively after 0.25 hr conditioning in liquid nitrogen.

On testing the samples exposed for 1 hr, GE composite showed further decrement in flexural strength because of generation of more micro-voids and micro-cracks at the interface and insufficient matrix hardening. On the contrary, the CNT-GE (all the compositions) composite showed relative increment in strength as compared to strength obtained after 0.25 hr conditioning. The addition of CNTs may have caused hindrance in the path of propagation of these micro-cracks and resulted in crack bridging and thus delayed the failure of the composite.

On further exposing the samples for 4 hr, increment in both strength and modulus was observed for GE composite and CNT-GE composites (all compositions). This can be attributed to the high amount of matrix hardening due to enough time available for the polymer chains to freeze (low mobility of polymer molecules). In addition to this, in CNT-GE composites the clamping force between CNT/epoxy increases and also stiffening of CNTs take place after this sufficient amount of time. Thus, this increased the strength of GE, CNT (0.1%)-GE, CNT (0.3%)-GE and CNT (0.5%)-GE, composite by 45.11 %, 56.83 %, 74 % and 127.4% respectively as compared to strength after 0.25 hr conditioning.

Finally the samples exposed for 8 hr showed very little increment in strength as compared to the strength after 4 hr conditioning. The reason may be ascribed to decrease in free volume, restricted mobility and freezing of polymer chains saturated after such long time. Also the density of micro-cracks remained same and hence not much significant increase in strength was observed.

From the above discussion it can be inferred that the mechanical response of GE and CNT modified GE composite is strongly dependent on the duration of liquid nitrogen conditioning. On exposing the composites for short duration, GE composites experienced a drastic loss in mechanical properties because of thermal shock, which is even higher in CNT-GE composites. But when they were exposed for 8 hr, recovery of properties was observed.

3.2 Damage constitutive failure model

For fabricating a composite for its durable and safe application, it is necessary to design the critical parameters properly. Failure modes such as matrix cracking, fiber pull out, fiber fracture, local fiber fragmentation, etc. are combinedly responsible for the overall failure of composite. These modes originate from the micro-mechanisms generated in the composite. Modelling of stress strain relationship can be undertaken with the help of

Weibull probability function. Critical parameters needed for designing a composite can be ascertained from this distribution model. The simulated stress can be obtained with the help of equation 1 (21,22).

$$\sigma = E\varepsilon \exp\left[-\left(\frac{E\varepsilon}{\sigma_o}\right)^{\beta}\right] \qquad \ldots 1$$

Where, E is the flexural modulus, ε is the strain, σ_o is the Weibull scale parameter, and β is the shape parameter. The scale parameter σ_o is a representative of the average/nominal strength of the composite. The shape parameter β will give idea about the scattering in strength. Increase in β suggests low scatter in strength. Equation 1 can also be written as:

$$\ln\left[\ln\left(\frac{E\varepsilon}{\sigma}\right)\right] = \beta\ \ln(E\varepsilon) - \beta\ln(\sigma_o) \qquad \ldots 2$$

Equation 2 suggests a linear relationship between $\ln\left[\ln\left(\frac{E\varepsilon}{\sigma}\right)\right]$ and $\ln(E\varepsilon)$ with slope β and intercept $-\beta\ln(\sigma_o)$. From this straight line equation, the slope will be the magnitude of β and the intercept will help in calculating the value of σ_o.

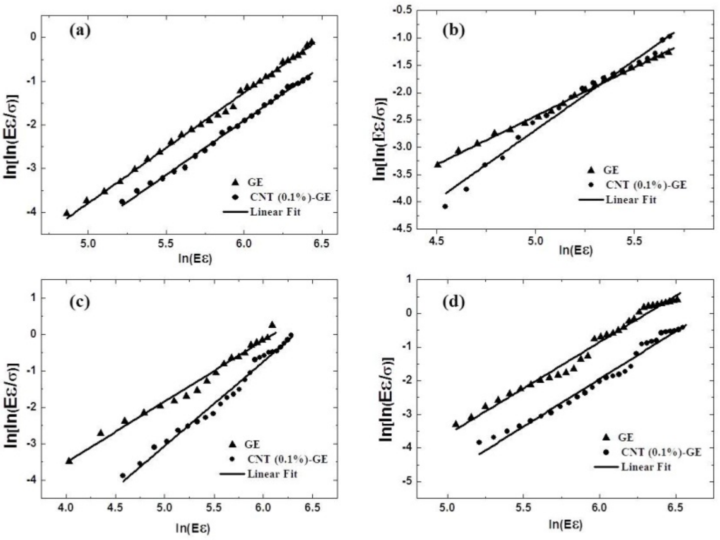

Figure 7. *Weibull fitting for experimental data of GE and CNT (0.1%)-GE composite (a) in as fabricated condition and after conditioning in liquid nitrogen for (b) 0.25 hr, (c) 1 hr and (d) 4 hr.*

Figure 7 shows the Weibull fitting plots of GE and CNT (0.1%) samples conditioned for 0 hr, 0.25 hr, 1 hr and 4 hr and the derived β and σ_o values are plotted in figure 8. From Figure 5, it can be seen that the value of σ_o decreases after conditioning GE and CNT-GE (all compositions) composites in liquid nitrogen for 0.25 hr and further increases (except GE composite) as the conditioning time increases to 1 hr. This varying trend in σ_o can be considered quite similar to the trend obtained for flexural strength variation with conditioning time as shown in Figure 3. It can be seen from Figure 8 that the value of β is unaffected for varying amount of CNT without any conditioning. It is further significantly affected after 0.25 hr liquid nitrogen conditioning as compared to that of without conditioning. After this conditioning, the value of β is maximum for CNT

(0.1%)-GE composite showing minimum scatter. Further exposure of 1 hr and 4 hr resulted in decreased variation in β. Overall results showed that the strength of all these composites obey the Weibull distribution model.

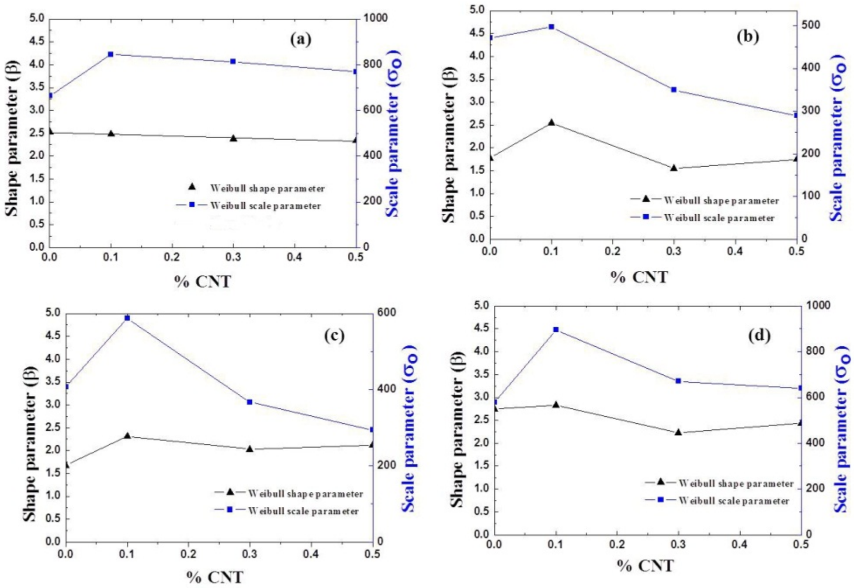

Figure 8. *Variation in Weibull shape parameter and scale parameter with varying amount of CNT (a) in as fabricated condition and after conditioning in liquid nitrogen for (b) 0.25 hr, (c) 1 hr and (d) 4 hr.*

Using the parameters derived from the Weibull model, the experimental and simulated stress vs. strain curves were plotted for GE and CNT (0.1% and 0.5%)-GE composite for the conditioning time of 0 hr, 0.25 hr, 1 hr and 4 hr as shown in Figure 9. This shows that the experimental stress vs. strain curves are in close agreement with the simulated stress vs. strain curves for 0, 1hr and 4hrs whereas in case of short term exposure (0.25 hr) the experimental and simulated stress-strain curves are not matching to that extent. Hence this implies that the materials mechanical response become less uniform and less predictable under short term liquid nitrogen exposure or thermal shock.

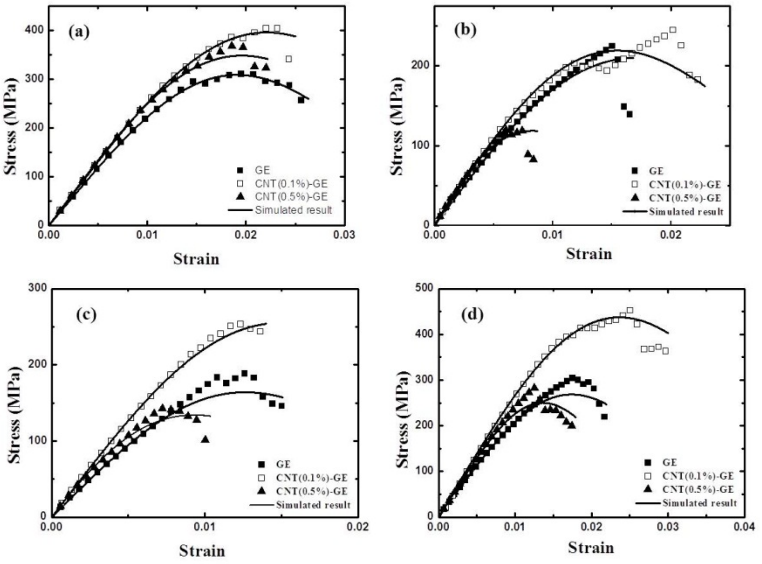

Figure 9. *Comparison between experimental and simulated flexural stress-strain curves for GE and CNT-GE composites (a) in as fabricated condition and after conditioning in liquid nitrogen for (b) 0.25 hr, (c) 1 hr and (d) 4 hr.*

3.3 Fractography

In order to identify the failure mechanisms responsible for the overall failure of the GE and the CNT-GE composite, Scanning Electron Microscopy (SEM) technique was adopted. Figure 10 shows the SEM images of fractured surfaces of GE composite and CNT (0.1%, 0.3%, 0.5%)-GE composite after conditioning them in liquid nitrogen for 0.25 hr.

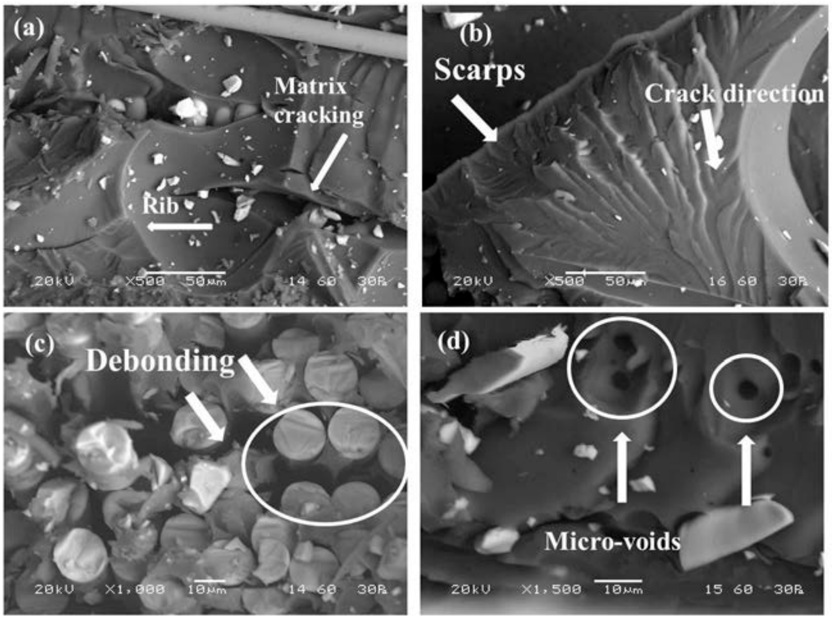

Figure 10. *SEM micrographs of fractured samples of (a) GE, (b) CNT (0.1%)-GE, (c) CNT (0.3%)-GE and (d) CNT (0.5%)-GE composites conditioned in liquid nitrogen for 0.25 hr.*

It was noted earlier that the strength of all the composites decreased drastically after 0.25 hr conditioning. It is evident from Figure 10 (a) that in case of GE composite, the major failure modes are matrix cracking, due to differential thermal contractions and formation of ribs. Figure 10 (b) shows the fractograph for CNT (0.1%)-GE composite and indicates that generation of scarps due to multiple micro-cracks lowers the strength of this composite. The convergence of cracks at some point is also an indicative of direction of propagation of cracks (23). These micro-cracks are generated due to coalesce of many micro-voids formed in the matrix. This can be seen from Figure 10 (d) of CNT (0.5%)-GE composite. Also, fiber/matrix debonding is observed for CNT (0.3%)-GE composite as shown in Figure 10 (c). Debonding results due to large interfacial shear stress generated between fiber and matrix at the time of loading.

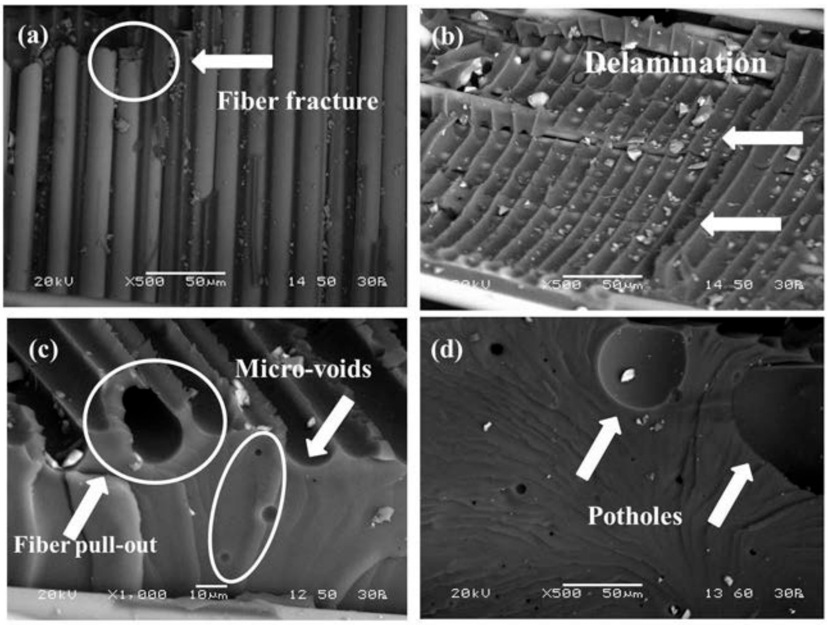

Figure 11. *SEM micrographs of fractured samples of (a) GE, (b) CNT (0.1%)-GE, (c) CNT (0.3%)-GE and (d) CNT (0.5%)-GE composites conditioned in liquid nitrogen for 4 hr.*

Figure 11 shows the SEM images of fractured surfaces of GE composite and CNT (0.1%, 0.3%, 0.5%)-GE composite after conditioning them in liquid nitrogen for 4 hr. It can be seen from Figure 11 (a) that fiber fracture is the mechanism accountable for failure of GE composite after 4 hr. Also, as fiber fracture is more energy absorbing failure mode, the strength obtained after 4 hr is higher than that obtained after 0.25 hr, as confirmed from the Figure 3. As the loading continues, the fracture will proceed and the broken fibers will ultimately be pulled out of the matrix as shown in Figure 11 (c). This increased strength after 4 hr conditioning is because of matrix hardening due to freezing of polymer chains. Thus debries are formed as shown in Figure 11 (b) due to abrasion between hardened matrix and fiber. Delamination of fibers from matrix is also observed for CNT (0.1%)-GE composite. Extensive river line markings and potholes, in addition to voids are observed in case of CNT (0.5%)-GE composite as evident from Figure 11 (d).

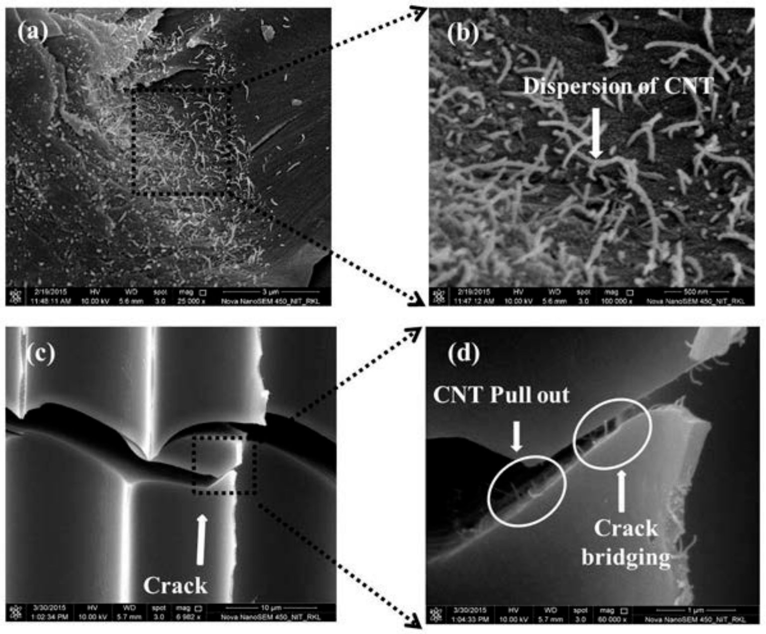

Figure 12. *SEM micrographs of fractured sample of CNT (0.1%)-GE composite tested at room temperature showing (a, b), dispersion of CNTs in the polymer and (c, d) crack bridging and crack pull-out by CNTs.*

Figure 12 shows the fractured surface of non-conditioned CNT (0.1%)-GE composite tested at room temperature. The figure is a clear indicative of effect of adding optimal amount of CNT in epoxy resin. The dispersion of CNTs in epoxy is clearly visible in Figure 12 (a), (b). It can be clearly noticed that on application of load, crack propagation is delayed by crack bridging mechanism taking place in the composite due to bridging of CNTs (Figure 12 (d)) between two surfaces of a crack. This suggests that CNTs may provide a tortuous path for propagation of cracks. In addition to this, further load application results in either CNT fracture or CNT/epoxy debonding and then CNT pull-out, which in turn, maximizes the fracture surface area. Thus, failure of the CNT-GE composite occurs after absorbing more energy. This crack bridging may also hold the key to increased strength for liquid nitrogen conditioned CNT (0.1%)-GE composite. Hence, resulting in higher strength compared to GE composite.

4. Conclusion

Evaluation of cryogenic treatment on the flexural performance of GE and CNT-GE composites was done for various conditioning time. Present study suggested that liquid nitrogen conditioning time has a strong impact on the mechanical behaviour of conventional composites as well as the nano-filler engineered composite. In the as fabricated condition, CNT (0.1%)-GE composite showed the highest strength among all the other composites fabricated i.e. the reinforcement efficiency (relative change in modulus in comparison to GE composite) was 28 %. This further dropped down to 25.1 % when cryogenically treated for short duration i.e. 0.25 hr. This decrease is due to sudden thermal shock experienced by the sample and generation of thermal residual stresses at the CNT/epoxy interface providing high probability for generation of interfacial voids, cracks, etc. On the other hand, a longer conditioning time enhances the strength of CNT-GE composite more effectively than GE composite due to stiffening of CNTs and generation of clamping stress at CNT/epoxy interface. Increment in σ_o was observed in CNT (0.1%)-GE composite for all the conditioning time. Non-conditioned samples showed approximately same β value irrespective of CNT content. Further conditioning time altered the β value and made it CNT content dependent**.** Thus, this study suggests that the flexural behaviour of GE and CNT-GE composite is strongly affected by liquid nitrogen conditioning time. This paper also suggests that further high strength can be expected by cryogenic conditioning of CNT-GE composite and GE composite when the rate of cooling is significantly low to avoid thermal shock. This paper unearths the dependency between flexural properties and liquid nitrogen conditioning time and widens the possibility for material researchers to tailor FRP composites as required for its safe and beneficial cryogenic application.

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