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FABRICATION OF ADVANCED FIBER REINFORCED COMPOSITE MATERIALS AND THEIR CRYOGENIC BEHAVIOR ASSESSMENT

D.S.Kumar[1], K.Prema Kumar[2], K.Dileep Kumar[3], B.C.Ray[4]

[1,2,3]*Department of Mechanical Eng., Sri Sivani College of Engineering, Srikakulam, A.P, (India)*

[4]*Department of Metallurgical and Materials Eng., National Institute of Technology, Rourkela, (India)*

ABSTRACT

Composite materials can be defined as a combination of two or more materials that results in better properties than those of the individual components used alone. These materials have high strength and stiffness combined with low density compared to the conventional metals. Polymers are structurally more complex than metals. Polymer – matrix composites are viscoelastic materials and their mechanical properties are significantly influenced by temperature. Carbon fibers are strong, stiff and low density but highly expensive, while glass fibers are relatively cheap and have better fracture strain and fracture stress. By combining the two fibers with in the same resin matrix it is possible to achieve balance between the properties of all GFRP and all CFRP. In this studyinter-laminar shear strength (ILSS) was carried out to quantitatively evaluate the mechanical behavior of glass/epoxy and lass/carbon/epoxy hybrid composite, glass/Kevlar hybrid and carbon nano tube reinforced in glass epoxy composite at ambient and liquid nitrogen environment. The glass/epoxy composite consist 60wt% of E-glass and 40 wt. % of epoxy. In case of glass/carbon /epoxy hybrid composite 30wt % of E-glass, 30 wt. % of carbon and 40wt % of epoxy and 0.1% CNT , 60wt % glass fiber and 40wt% epoxy. This work mainly focused on response of ILSS for GFRP, glass/carbon, glass / Kevlar and MWCNT reinforced in glass / epoxy hybrid composites at cryogenic environment.Hence this results in choosing G/C hybrid is a better choice of material in low temperature environmental applications.

Keywords: Carbon nanotube (CNT), cryogenic temperature, hybrid laminate, GFRP, kevlar fiber, Polymer composites.

I. INTRODUCTION

In recent years material engineers focused on characterization and mechanical behavior of polymer composites at cryogenic temperature. They are used mostly in areas from aerospace, automobiles and boats to cryogenic equipment such as cryogenic fuel tanks, cryogenic fuel delivery lines, cryogenic wind tunnels and parts of the cryogenic side of turbo-pumps because of their ease of handling, low fabrication cost and excellent mechanical properties. Nano-particle reinforced polymer composites have been widely studied and some researchers already studied the improvement of the fracture toughness of polymers. Carbon nanotubes (CNT) have shown a high potential to improve the mechanical properties of polymers as well as electrical properties [1]. Due to their excellent physical and mechanical properties [2],carbon nanotubes (CNTs) have played an important role in variousfields of engineering material researches since their discovery byIijima and Ichihashi [3]. In recent years, CNTs have been used as fillerin order to enhance some physical, electrical and mechanicalproperties of polymers. Their mechanical properties [4], a high aspect ratio and a high Young's modulus and tensile strength, in combination with an electrical and thermal conductivity make them interesting materials for the use as nano-fillers in polymers and open up new perspectives for multi-functional materials, e.g. conductive polymers with improved mechanical performance. The properties of polymer nano composites depend on the dispersion of nanoparticles and the interaction between nanoparticles and the polymer matrix. Homogeneous dispersion and good interfacial adhesion can transfer mechanical loads effectively to carbon nanotubes (CNT) and remarkably improve bulk properties. However, because of the high inertness of CNT, efforts to enhance the mechanical properties of CNT/polymer nano composites have led to only moderate success.Gojny et al. [5]reported that DWCNT (double walled CNT) could increase both tensile strength and fracture toughness. The linear coefficient in thermal expansion of the kevlar fiber material has been studied [6].it has been absorbed that the axial coefficient of these fibers were not linear, the longitudinal expansivity of these components were abstained by the Schapery's equation and is modified to get the transverse expansivity.The meet et.al [7] said that the Liquid nitrogen conditioning for 8 hrs showed decrease in ILSS of GFRP and G/C hybrid by 26.2 % and 16.2 % respectively.It can be noted that at low temperature the performance of hybrid is better than GFRP.

II. EXPERIMENTAL DETAILS

2.1Materials

1. Woven fabric E-Glass Fibers (FGP, RP-10)
2. Woven fabric Carbon Fibers (TC-33)
3. Epoxy Resin (Lapox, L-12) based on Bisphenol A
4. Hardener (K 6, primary amine)
5. Woven fabric Kevlar fibers
6. Multi-walled Carbon nanotubes.

Table 1: Properties of epoxy resin, glass fiber and carbon fiber:

Property	Epoxy resin	Glass fibre	Carbon fibre	Kevlar 29	CNT
Tensile strength(GPa)	0.11	3.4	4	2.6	11-63
Tensile modulus(GPa)	4.1	72.3	240	58.9	250-970
Density(g/cm^3)	1.162	2.58	1.8	1.4	0.037

III. EXPERIMENTAL METHODS

3.1 Sample Preparation

Woven fabric E- glass fiber were cut into the size of 25 cm X 25 cm to form 14 layer sheets (laminates) and weighed by using electronic weighing machine. LY-556 epoxy resins based on Bisphenol A is weighed to be 40% of the total weight of the fiber and epoxy resin. For fabrication of glass/epoxy (weight fraction of glass fiber is 60%) and of glass/carbon/epoxy weight fraction of glass fiber is 30% and carbon weight fraction 30%.ILSS test for G/C hybrid 5 layers glass fiber and 4 layers carbon fiber, for G/K hybrid where Kevlar layers occupied the positions as (1,14). Then, Hardener HY 951 (aliphatic primary amine) at the ratio of 10% by weight of epoxy resin was used. Glass fiber/epoxy,glass/carbon fiber/epoxy hybrid, glass/kevlar hybrid composite laminate and glass/epoxy with MWCNTs have been prepared by hand lay-up method and cured in a hydraulic press by compression moulding method at 60° C temperature and 10 kg/cm^2 pressure for 20 minutes.

3.2 Dispersion of MWCNT into Epoxy Resin

Fabricate the MWCNT reinforced glass/epoxy (CNT/GE) composite, the epoxy resin was modified by incorporating MWCNTs in to it. The amount of CNT in CNT-GE composite was 0.3wt.%of epoxy for low temperature conditioning and 0.1 wt. %, of epoxy for cryogenic treatment. Pre-calculated CNT was slowly poured into 150 mL of acetone. By using magnetic stirring the suspension was stirred at room temperature for 30 min at 1000 rpm. Followed by the sonication for 30 min. Because of stirring and sonication, the CNTs get distributed throughout suspension. After sonication the suspension was mixed with pre-calculated epoxy. Magnetic stirring of epoxy/CNT/acetone mixture was done at 1000 rpm for 1 hr at 70 °C. Sonication was again carried out at 70 °C upto evaporate entire acetone. During process might be air bubbles entrapped into the suspension. To remove these air bubbles, suspension was vacuum degassed for 18 hrs. The figure shows the dispersion of CNTs in composite and fabricates the fibre reinforced nanocomposite.

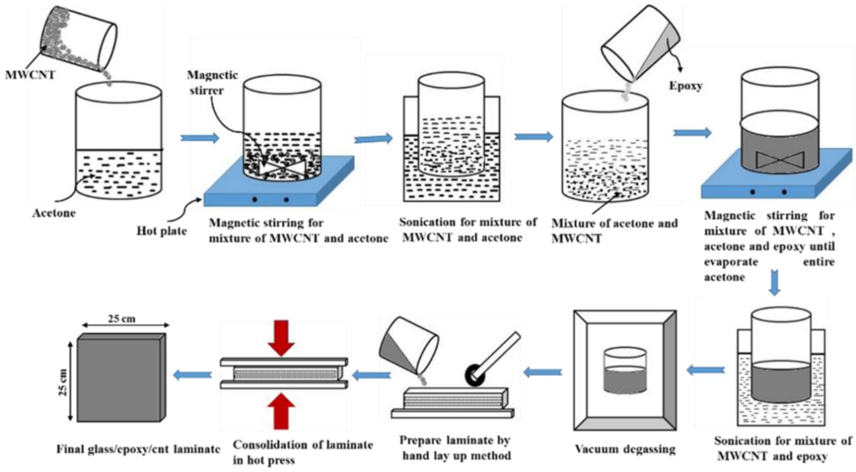

Fig.1 Dispersion of CNT in epoxy and further fabrication of laminated composite

IV. TESTING OF COMPOSITES

4.1 Inter Laminar Shear Strength (Ilss)

Inter-laminar shear stresses are the source of failure, unique in composite structures. The presence of inter-laminar shear stress at the interface leads to de-lamination, if it exceeds beyond its strength and hence reduces the strength of structural component. Inter-laminar shear stress arises due to various reasons. All layers may have different properties depending upon the selection of fibers in lamina as well as its orientation. Inter-laminar shear stress is the out of plane stress σ_z, τ_{xz} and τ_{yz} at the interface between layers in laminated composite structures. The specimens for the evaluation of ILSS were prepared as per the ASTM standard ASTM- D2344 using diamond cutter. Before testing the specimens were kept in oven at 50^{0}C for 3 hours to remove moisture and volatile substances. The sample dimensions are 32mm×9mm×4mm of glass/epoxy and for glass/carbon/epoxy hybrid composite 24mm×7mm×3mm. Ex-situ short beam shear (SBS) tests of glass/epoxy, glass/carbon/epoxy, G/K and CNT reinforced specimens were carried out with the help of INSTRON 5967 (Servo hydraulic machine with 30 KN load cell) at ambient temperature without post cured and post cured.

$ILSS = \frac{0.75F}{bt}$ Where b=width of specimen.

t=thickness of specimen.

F=maximum load.

4.2 Ilss Test For Liquid Nitrogen Condition For 1 Hr, 36hrs And 48 Hrs

The test have been performed by keeping samples inliquid nitrogen (-196^{o}C) for 36 hrs and 48 hrs. For each point of testing 3-4 specimens were tested and average value was taken. The ILSS value is calculated from the above equation.

Table 2: ILSS at different temperature conditions:

Material	Ambient	LN_2 for 1 hrs	LN_2 for 36 hrs	LN_2 for 48 hrs
GE	22.20± 0.38	15.75± 0.12	24.23 ± 0.61	25.12 ± 1.17
(G/C)	37.42 ± 0.81	35.62 ±0.21	40.05 ± 1.61	41.36 ± 0.86
G/K(1,14)	25.33 ± 0.29	21.29±0.43	29.25 ± 1.07	29.78 ± 0.97
CNT (0.1%)-GE	27.31±0.53	22.36 ±1.2	32.34 ± 1.76	33.02 ± 0.94

V. RESULTS & DISCUSSION

5.1 Interlaminar Shear Strength of Liquid Nitrogen Temperature

From fig 2 it can be seen that ILSS of GE conditioned at -196°C for 1 hr. decreases by 29.05 % as compared to ILSS at ambient. Similarly G/K (1, 14) showed decrease in ILSS by 15.94 % at liquid nitrogen temperature. Glass/carbon hybrid showed decrease in ILSS by 4.8 % over ambient. Finally CNT (0.1 %)-GEshowed decrease in ILSS by 18.13 % as compared to ILSS obtained at ambient conditions. The reason may be attributed to micro crack density formed due to thermal stresses. When the samples were exposed to liquid nitrogen, micro cracks were formed immediately in the material.From fig 2 it can be seen that ILSS of GE conditioned at -196°C for 36hrs increases by 9.14 % as compared to ILSS at ambient. Similarly G/K (1, 14) showed increase in ILSS by 15.48 % at liquid nitrogen temperature. Glass/carbon hybrid showed increase in ILSS by 7.03 % over ambient. Finally CNT (0.1 %)-GE showed increase in ILSS by 6.88 % as compared to ILSS obtained at ambient conditions.

From fig 2 it can be seen that ILSS of GE conditioned at -196°C for 48hrs increases by 13.15 % as compared to ILSS at ambient. Similarly G/K (1, 14)showed increase in ILSS by 17.50 % at liquid nitrogen temperature. Glass/carbon hybrid showed increasein ILSS by 10.53 % over ambient. Finally CNT (0.1 %)-GE showed increase in ILSS by 20.98 % as compared to ILSS obtained at ambient conditions. From fig.2 inferred that the ILSS for G/C increases by 10.53% over ambient for long time conditioning in liquid nitrogen. Whenever

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samples conditioned in liquid nitrogen for long time increases the ILSS for all composites, but G/C hybrid shows maximum ILSS. The reason may be attributed to strong bond between carbon fibre and epoxy. Carbon fibers are having good adhesion property than other reinforcements.

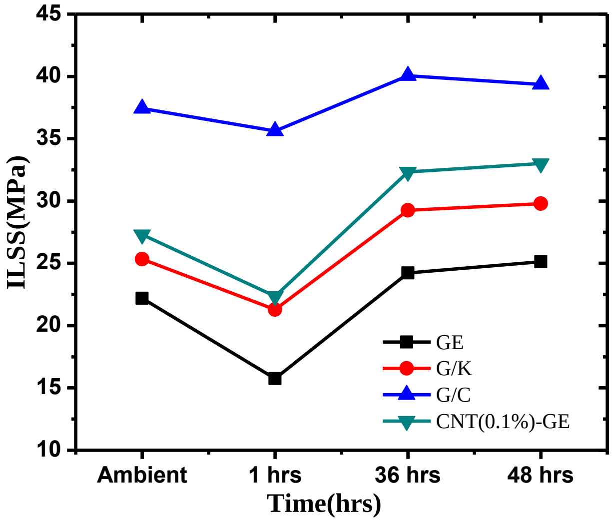

Fig.2 Variation in ILSS for GE,G/C,G/K and CNT(0.1%)-GE composites with conditioning time in liquid nitrogen.

After 1 hr of conditioning a noticeable enhancement in the ductility was ensured for all composite systems probably due to stress relaxation and this enhancement continues up to 36 hrs of conditioning. A further longer conditioning time brings no change in ductility for all composite systems. Exposing the samples for 1 hr in liquid nitrogen caused a gradual reduction in ILSS for all the composite systems. The samples may have experienced thermal shock when suddenly brought to -196°C to room temperature (20°C).Finally, the samples exposed for 48 hrs showed very little increment in strength as compared to the strength after 1 h conditioning. The reason may be ascribed to decrease in free volume, restricted mobility and freezing of polymer chains saturated after such long time.From the above discussion it can be inferred that the mechanical response of different composite systems were strongly dependent on the duration of liquid nitrogen conditioning. On exposing the composites for short duration, all composites experienced a gradual loss in mechanical properties because of thermal shock.

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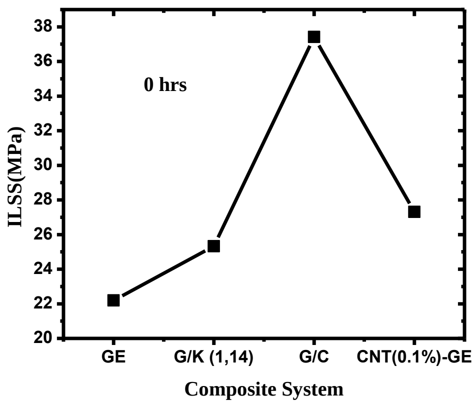


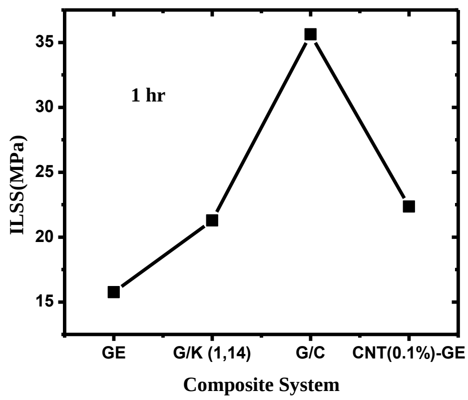
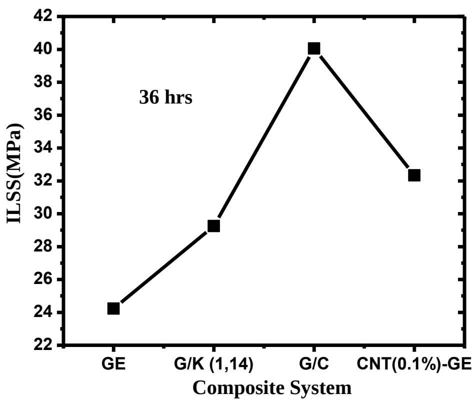


Fig 3: Effect of liquid nitrogen temperature on ILSS for 0 hrs Fig 4: Effect of liquid nitrogen temperature on ILSS for 1 hrs.



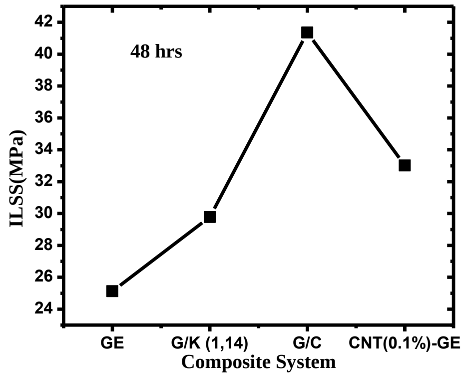


Fig 5: Effect of liquid nitrogen temperature on ILSS for 36 hrs Fig 6: Effect of liquid nitrogen temperature on ILSS for 48 hrs.

From the figures 3, 4, 5 and 6 represents that effect of liquid nitrogen temperature on ILSS for different time durations. All figures show same trend, in that G/C hybrid shows maximum ILSS than remaining.

VI. CONCLUSION

Evaluation of cryogenic treatment on the Interlaminar shear stress performance of different composite systems such as GE, G/C,G/K(1.14) and CNT(0.1%)-GE was done for various conditioning time. Present study suggested that liquid nitrogen conditioning time has a strong impact on the ILSS of different composite system. The conventional materials have high density, cost and less fatigue resistance than polymer reinforced composites. The composites tanks will be enable the next generation of rocket and spacecraft needed for space applications. Conventional laminated composites have poor through thickness and poor interlaminar properties. Further at low and cryogenic temperatures the tendency of micro crack generation is higher. Addition of reinforcement such as (kevlar, carbon) to the polymer can significantly enhance their matrix dominated properties.

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