Digest Journal of Nanomaterials and Biostructures Vol. 14, No. 3, July - September 2019, p. 641 - 653

REINFORCEMENT EFFECT OF GRAPHENE ENHANCED GLASS FIBRE REINFORCED POLYMERS: A PROMINENCE ON GRAPHENE CONTENT

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Graphene has shown exceptional mechanical properties and multifunctional characteristics. The aim of this study is to explore the scope of graphene enhanced composites for industrial and engineering applications. G[1]raphene, an allotrope of carbon, has many exclusive properties and it is the strongest material ever tested. Glass fibre reinforced composites were fabricated with graphene fractions ranging from 2% to 10% using solvent assisted dispersion and ultrasonic mixing. The fabricated composites were submitted to tensile, impact and flexural/three point bending tests after curing. Micro structural features and morphology of epoxy nanocomposites were investigated using Scanning Electron Microscopy and X-ray Diffraction (XRD). This investigation reveals that due to incorporation of graphene nano fillers in epoxy composites to some extent increases tensile modulus and mechanical properties. It also shows that Graphene/epoxy composites reinforced with glass fibreas giving higher mechanical performance than conventional glass fibre reinforced epoxy composites. The driving force behind the fabrication of graphene enhanced nano composites is to achieve high functional mechanical properties for high end industrial applications.

(Received February 25, 2019; Accepted August 6, 2019)

Keywords: Graphene, Glass fiber,Composites, Epoxy resin

1. Introduction

Glass Fibre Reinforced Polymers (GFRP) has become a staple in the industry due to its high strength to weight ratio. GFRP is a variant of FRP, and compared with the traditional reinforcement material, the weight of GFRP is a fourth of the weight of steel with 2X the tensile strength of steel. It is also cost effective compared with other material and GFRPs have excellent properties like tensile flexibility, stiffness and resistance to harm arising from the use of chemicals. GFRP is an excellent choice for the construction of chemical storage tanks, apparatus, piping systems and other types of industrial process equipments. The GFRP material properties beat many conventional materials such as steel when it comes to chemical and corrosion resistance. GFRP materials consist of thermosetting resins and Fibre glass. The combination of resin and glass fibres, makes GFRP the main ingredient of FRP products.

The resin provides environmental and chemical resistance to the product. It is the binder for the glass fibre in the laminate. A resin, also called thermosetting resin, is the binder of the glass fibre in the laminate. Making a GFRP laminate with resins involves a chemical reaction in which molecules are formed and warmth is released. The existence of new network of molecules helps creation of the final shape. In general resins are of three types namely Polyester, Vinyl ester and Epoxy resin. Polyester resins are viscous, pale colored liquids consisting of a solution of polyester which is easy to use, fast curing and tolerant of temperature and catalyst extremes. They are usually referredto as Unsaturated Polyester resins. Epoxy resin is used by manufacturers of composite parts that demand ultimate strength. In addition, the choice of epoxy resin is due to its dimensional stability and increased bonding with other materials. The term ‘epoxy’ refers to a

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chemical group consisting of an oxygen atom bonded to carbon atoms that are already bonded in some way. Among resins, epoxy is considered to be the most advanced in the market and usually identifiable by its characteristics and a number of useful properties. Vinyl ester resin is considered to be a hybrid of Polyester and epoxy resins.

Nano fillers are basically additives in solid form, which differ from the polymer matrix in terms of their composition and structures. The activity of fillers arises due to a variety of causes, such as formation of bond or filling of a certain volume and disruption of the conformational position of a polymer matrix and also the immobilization of adjacent molecule groups and possible orientation of the polymer material. Earlier nano fillers such as nano clay, single walled carbon nano tubes, Multi walled carbon nano tubes were used in the fabrication of laminates. This paper deals with the use of Graphene nano filler for cross linking the carbon black in elastomers. Graphene is an allotrope of carbon consisting of a layer of carbon atoms arranged in a lattice that has many exclusive properties ever tested.

2. Literature survey

Ian A et al. [1]reviewed composites manufactured and reinforced with carbon nano tubes and graphene. In the paper studies about composites reinforced with single walled nano tubes, multi walled nano tubes and graphene were discussed.

Z Li et al.[2] studied the effect of orientation of graphene based nano platelets upon the young’s modulus of composites on varying the orientation of fibres and young’s modulus is found to be varied.

C.M.Meenakshi, Krishnamoorthy A et al. [3] Prepared flax and glass fibre reinforced polyester hybrid composite laminate by hand lay up method and tested for its tensile, flexural and impact strength of laminates. The author had concluded that hybrid composte laminate also have equal mechanical strength compared to glass fibre reinforced polymers.

I Infantamarypriya et al.[4] did experiments with graphene platelet nano powder dispersed in epoxy resin and fabricated bi-axial GFRP. The addition of graphene platelet nano powder with the parent material showed a big increase in the strength of the composite.

Arun G K et al.[5]have investigated the mechanical properties of graphene oxide reinforced with GFRP. Graphene was added in proportions of weight ranging from 1% to 3% wt. The specimens were fabricated using the hand layup process and subject to experiments to predict their strength, hardness and flexural strength. The composite material with 2.5% wt fraction of graphene was seen having superior properties.

Pradhan Aiyappa M R et al.[6] used graphene as a nano filler and found a considerable improvement in the mechanical properties with increase in percentage of graphene weight.

Rathinasabapathi.G, Krishnamoorthy A et al.[7] experimented MWCNT with varying percentage weights dispersed in epoxy resin and fabricated GFRP. The addition of MWCNT with the GFRP caused an increase in the strength of the composite at 4%MWCNT weight. There was deterioration in mechanical properties with further addition of MWCNTs.

C Juan et al. [8]fabricated glass fibre with graphene oxide and modified the glass fibre reinforced plastics by modifying its performance to a highest level.

Balamugundan B et al.[9] studied the feasibility of fabricating HDPE composites by adding nano particles. The author had tried using nano particles such as multi walled carbon nano tubes and graphene. The results were predicted by X-ray diffraction method.

Dongyan Liu et al.[10]synthesized graphene to fabricate epoxy resin sandwiched composite and investigated the varying properties. Composites were prepared using the dip coating method with the ultrasonication method used forbetter dispersion of graphene whiskers into epoxy resin. XRD and SEM were used for a study of the morphology of composites. XRD showed the presence of nano filler and SEM images led to observation of a better bonding in graphene with resin and reinforcement material.

Bindusharmila T K et al.[11] investigated the mechanical properties of epoxy hybrid composites and found a considerable increase in mechanical properties such as tensile, impact strength and fracture toughness of composites. This is due to better nano filler adhesion.

Y Li et al.[12] used graphene platelet that is ultrasonicated with epoxy resin to fabricate carbon fibre reinforced plastics and also found that the mechanical properties enhanced when percentage weight of grahene platelet were increased.

X Zhang et al.[13]designed and developed GFRP reinforced with CNT using it potentially for sports instruments.

SridharanVeerapuram et al.[14] investigated graphene filled Jute fibre reinforced plastics and studied the drilling characteristics. The author concluded that surface roughness characteristic have improved upto 64.7% and addition of graphene played an effective role in improving the mechanical properties.

Umer R et al. [15] studied the effect of graphene particles that is infused with epoxy resin that is processed to manufacture glass fibrecomposiotes.

R Jyothilakshmi et al. [16] characterized mechanical properties of graphene reinforced GFRP laminates and predicted that the properties of graphene influences GFRP until it is added upto 5% weight.

The authors experimented the graphene powder till 5% weight fractions and found an increase in mechanical properties. The current work has identified this gap and adoptedaddition of graphene nano filler till 10% weight fraction. Graphene nano filler was added to epoxy resins in percentage of 2%, 4%, 6%, 8% and 10% wt. fractions. Ultrasonicator was used for dispersion of the graphene powder effectively with epoxy resin. Addition of more than 6% graphene powder showed deterioration in the mechanical properties.

3. Experimentation

3.1. Materials used for fabrication

The material used for the study was Glass Fibre oriented in a bi-axial manner, supplied by SakthiFibreTraders, Chennai. Composite Laminates were fabricated using the hand lay upprocess and employing an ultrasonicator for dispersion of nano filler with epoxy resin. Epoxy resin (LY5561) and hardener (HY9511) were mixed up to a ratio of 10:1 by weight for the preparation of composite laminates. Epoxy resin and hardener were purchased from MahalakshmiChemicals, Chennai. Bidirectional woven glass fiber was utilized for fabricating the composite laminate.

Graphene, an allotrope of carbon, is a black powder with a minimum of 99.5% carbon. The properties of epoxy resin, hardener and graphene nano filler are listed in Table 1 and Table 2.

Table 1. Properties for epoxy resin LY5561 and hardener HY9511.

Properties for epoxy resin LY5561	
Tensile Strength (N/mm^2)	85 - 92
Compressive Strength (N/mm^2)	128 - 146
Elongation of Break (%)	7 – 9.5
Elasticity Modulus (kN/ mm^2)	3.2 – 3.5
Density (g/ cm^3)	1.11 – 1.32

Table 2. Properties of Graphene Nano Filler.

Properties of Graphene Nano Platelet Powder	
Carbon (%)	99.5
APS (Micron)	15
Thickness (Nm)	6 - 8
Surface Area (m^2/g)	150

The SEM image of graphene with intensity of image ranging from 1μm to 50μm is shown in Fig. 1.

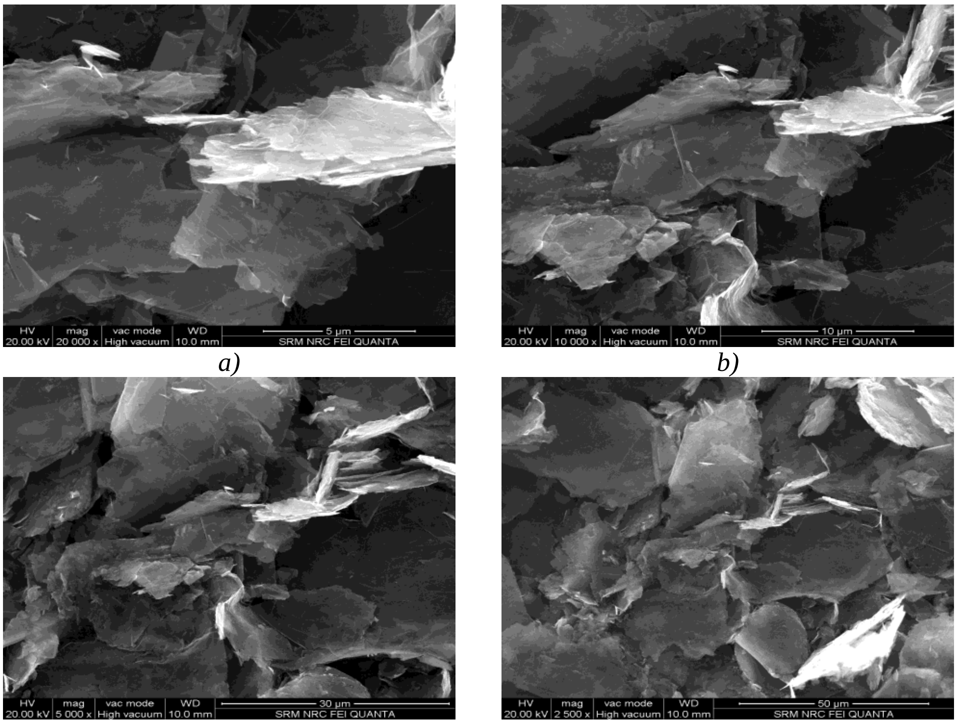


Fig. 1 SEM image of Graphene powder at a) 5 μm, b) 10 μm, c) 30 μm, d) 50 μm

3.2. Fabrication of graphene epoxy GFRP composite

Two types of composites were manufactured, of which one was with neat epoxy and other with epoxy/glass fibre composite enhanced with graphene of different weight proportions.

Graphene in proportions of different specimens (2% wt, 4% wt, 4% wt, 8% wt, 10% wt) was added slowly to epoxy resin and subjected to stirring with an ultrasonicator for about 30 minutes. A hardener is added for increasing the strength of the laminate and epoxy/graphene solution. The stirring was continued for 15 min using anultrasonicator with 1000 rpm.

Then the glass fibre with graphene was fabricated using the hand layup process followed by curing at room temperature for 30 hours. High precision water jet machingwas used for cutting fabricated laminates for mechanical testing according to the desired dimension from the ASTM standards.

4. Characterization of graphene epoxy GFRP composite

4.1. Tensile test

Tensile test was performed on the prepared specimens of composite to determine the elastic properties of the composite. The most commonly used specimen geometries were the dog-bone specimen and straight-sided specimen with end tabs. Tension test was performed on all the five samples as per ASTM D - 638 [20] test standards using a computer controlled Universal Testing Machine (UTM) with 50kN load cell pulled at the recommended cross head speed of 4mm/min which corresponds to a strain rate of 0.2%/sec, . The tests were closely monitored and conducted at room temperature.

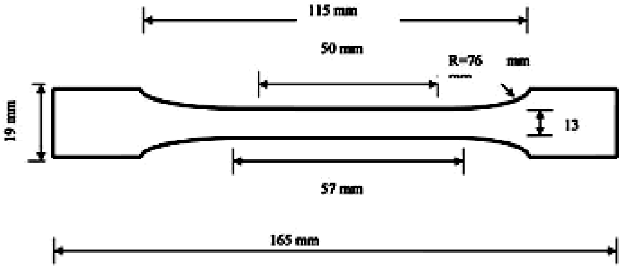


Fig.2. ASTM D – 638 (20) Standard size for Testing.

The specimens were cut as per ASTM standards D-638 for carrying out Tensile testing using a universal testing machine. Tests were conducted on MWCNTs reinforced epoxy GFRP samples. For each sequence, 3 identical specimens were tested and the average result wascomputed. Fracture Toughness is calculated by the equation

$$\text{Fracture Toughness} = \frac{\sigma^2 \Pi a}{E}$$

Where σ is the stress (N/mm^2), a is the crack length and E, the Young's modulus.

4.2. Flexural test

The determination of flexural strength is an important characterization of any structural composite. It is the ability of a material to withstand the bending before reaching the breaking point. Conventionally a three point loading was conducted for finding out the flexural properties of the composites using a computer controlled Universal Testing Machine (UTM). A span of 80 mm and thickness of 4 mm and cross head speed were maintained at 4 mm/min under room temperature. The strength of a material in bending is expressed as the stress on the outermost fibers of a bent test specimen, at the instant of failure.

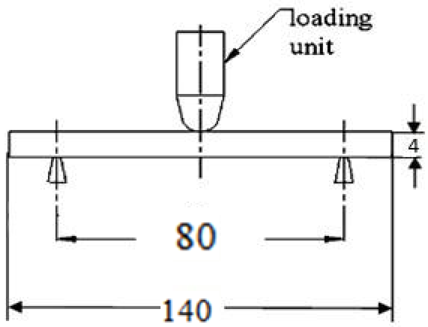


Fig.3. ASTM D – 790 Standard size for Flexural Testing.

Flexural test was performed for the evaluation of the stiffness of the material. Flexural tests were conducted using a universal testing machine in accordance with ASTM standard ASTM D-790. Specimens of 120 mm length and 15 mm wide with 4 mm thickness were cut and tested. Flexural strength was calculated using the equation

$$\text{Flexural Strength} = \frac{3PL}{2bx^2}$$

where P is the break load, L is the support span, b is the specimen width and x the specimen thickness respectively.

4.3. Impact test

Impact test finds the ability of the material to withstand sudden shock loads. This test is conducted using theIzod method of impact testing. The specimen made as per the specification was kept in the machine and the load was released. The absorbed energy was indicated in the dial.

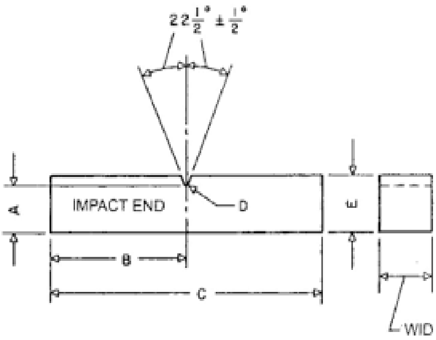


Fig.4. ASTM D – 256 Standard size for Impact Testing.

Impact test was conducted as per ASTM D-256 standard. The standard specimen used for the test was 70mm x15mm x3 mm.

5. Results and discussions

5.1. Tensile Behaviour of Graphene reinforced glass fiber reinforced polymeric Composite

Tensile test was performed by application of an increasing load to a test sample up to the point of failure. A stress/strain curve showing the property of the material throughout the tensile test is shown in figure. The following quantitative measurements obtained by the data generated during tensile test provided the ability to determine mechanical properties. Tension test was performed on all the six samples as per ASTM D638.

Six composite specimens with composition of Graphene were tested and the results of various mechanical properties are listed below.

Table 3 . Mechanical properties of Graphene reinforced GFRP.

Composition of Graphene by weight	Ultimate Tensile Strength (MPa)	Peak load(kN)	Percentage Elongation(%)	Fracture Toughness (N/mm)
Neat GFRPwithout Graphene	100.82	4.01	3.98	189.00
GFRP with 2% wt. Graphene	115.77	4.32	5.02	273.73
GFRP with 4% wt. Graphene	121.12	4.45	5.09	290.37
GFRP with 6% wt. Graphene	215.88	5.26	5.95	604.99
GFRP with 8% wt. Graphene	165.53	4.20	4.06	316.54
GFRP with 10% wt. Graphene	166.58	4.33	4.38	343.65

Increases in tensile strength and young's modulus were seen following an increase in the Graphene content up to 6% wt. Further, an addition of Graphene caused a decrease in strength value. The table clearly shows the strong dependence of tensile strength and tensile modulus on the Graphene content for GFRP Composite only to a certain limit.

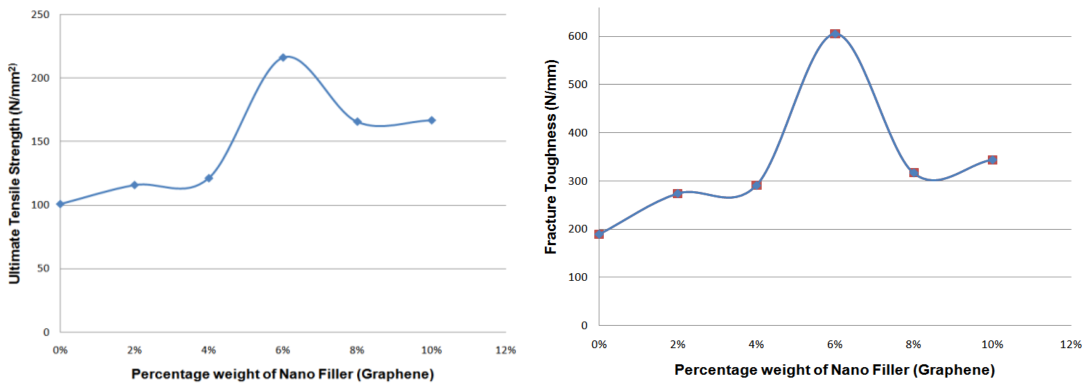


Fig.5. Curves of Fracture Toughness (Left) and Ultimate tensile strength (Right) for GFRP Composites at various Graphene contents.

The table above shows the composite laminate with 6% wt. of Graphene as exhibiting a higher tensile strength. The composite laminate with 6% wt. of graphene is also found to be more ductile compared to other specimens of different wt. percentage of graphene. The above Table and curves show an increase in the tensile strength with increase in the percentage of Graphene above 6%wt., with the occurrence of a decrease of fracture toughness below on a further increase in the percentage of Graphene.

This happens due to the agglomeration and nano voids formed into the reinforced epoxy composite. This was also due to the uneven distribution of graphene powder in the resin. This occurs as a result of the increase in percentage of graphene weight that caused poor mixing with the resin due to its viscosity.

5.2. Stress strain curves

A stress-strain curve is a graphical representation of the behavior of a material when it is subjected to a load or force. The two characteristics that are plotted are the stress on the y-axis and the strain on the x-axis. Stress is the ratio of the load or force to the cross-sectional area of the material to which the load is applied.

Strain, on the other hand, is a measure of the deformation of the material as a result of the force applied. It is a change in the shape or form of the material. The values of Young's modulus and strain with respect to each composition are listed in the table.

Table 4. Young's Modulus of Graphene reinforced GFRP.

Composition of Graphene by weight	**Young's Modulus, E**
Neat GFRP without Graphene	2533.17
GFRP with 2% wt. Graphene	2306.17
GFRP with 4% wt. Graphene	2379.56
GFRP with 6% wt. Graphene	3628.23
GFRP with 8% wt. Graphene	4077.09
GFRP with 10% wt. Graphene	3803.19

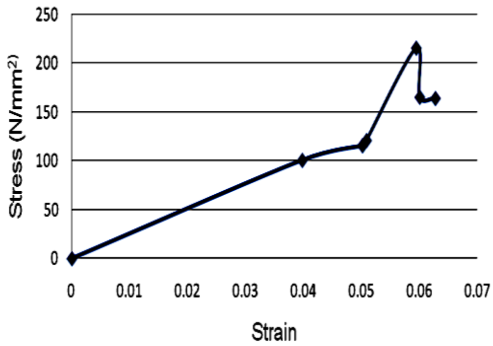


Fig.6 Stress-strain curves for GFRP composites at various Graphene contents.

It is inferred from the stress strain curve that highest ultimate stress occurs at a point with weight of graphene composition at 6%.

5.3. Flexural test results

Flexural test was conducted for the evaluation of the stiffness of the material. It is the ability of a material to withstand bending before reaching the breaking point. The test was performed as per the ASTM D790, where a three point bend test was carried out for obtaining the flexural strength of GFRP Composite. These tests were carried out till failure of composite specimens was noted.

After the test, a linear increase in the flexural strength was noted. A comparison was made on the basis of the percentage increase in graphene weight. There was a linear increase in flexural strength from 2% to 6% weight of Graphene Nano fille. The specimen with 6% graphene wt. was found to have the highest flexural strength among these.

The Table and the curve below show 6% Graphene content having better flexural strength and 8% wt. & 10% wt. Graphene content has a lower flexural strength due to increase in the agglomerates, inhomogeneities and nano voids with increasing amounts of the Graphene.

Table 5. Mechanical properties of Graphene reinforced GFRP.

Composition of Graphene by weight	Ultimate/ Break load(kN)	Maximum Displacement (mm)	Displacement At Fmax(mm)	Ultimate Stress(N/mm^2)	Flexural Strength (MPa)
Neat GFRP without Graphene	0.21	5.8	4.7	6	326.67
GFRP with 2% wt. Graphene	0.24	6.5	4.8	6	373.33
GFRP with 4% wt. Graphene	0.32	8.9	8.4	8	497.78
GFRP with 6% wt. Graphene	0.38	9.7	8.9	10	591.11
GFRP with 8% wt. Graphene	0.311	7.3	6.5	7	483.78
GFRP with 10% wt. Graphene	0.310	7.0	6.4	7	482.22

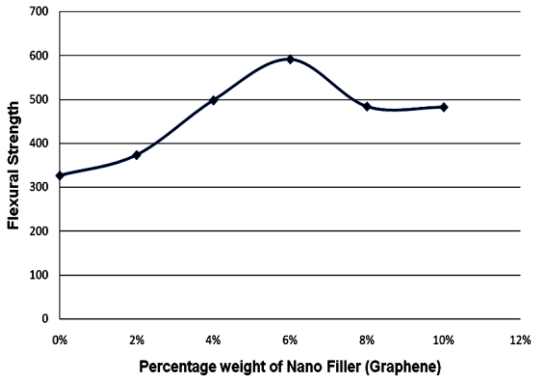


Fig .7.Variations in Flexural Strength with Graphene content.

5.4. Impact test results

A pivoting arm was raised to a specific height (constant potential energy) and then released. The arm did a swinging down hitting the sample and breaking the specimen. The energy absorbed by the sample was calculated from the height the arm that did swinging after hitting the sample. The ASTM International standard for Izod Impact testing of plastics is ASTM D256. Impact test reflects the ability of material absorbing energy at fracture, when exposed to sudden impact. The dimensions of test specimens were 70 mm length, 15 mm width and 3 mm thickness respectively.

Table 6. Impact Values of GFRP with various composition of Graphene

Composition of Graphene by weight	**Impact Values in Joules**
Neat GFRPwithout Graphene	2.9
GFRP with 2% wt. Graphene	3.0
GFRP with 4% wt. Graphene	3.8
GFRP with 6% wt. Graphene	6.5
GFRP with 8% wt. Graphene	5.2
GFRP with 8% wt. Graphene	5.2

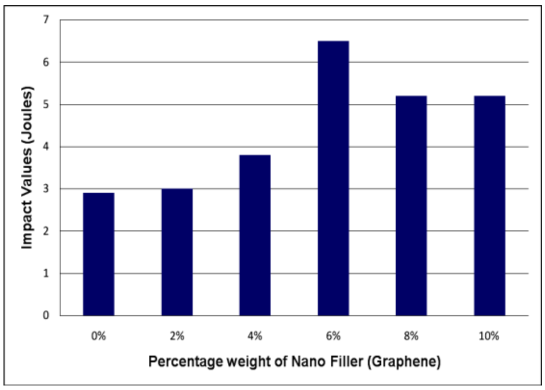


Fig.8 .Variation of Impact Strength in Joules.

5.5. Morphological Structural Analysis by SEM

The fabricated polymer composite showed uniform microstructures for additions of Graphene amounts up to 6 wt%. The Graphene platelet powder in the epoxy matrix was well dispersed despite the presence of some small agglomerates (Fig. 9 (b) SEM image of GFRP + 4%

Wt. Graphene). The frequency and the size of the agglomerates increased with increase in Graphene. Fig. 9 (c) SEM image of GFRP + 6% Wt. Graphene shows good interfacial bonding at 6% wt of MWCNT.

There was a significant increase in porosity for Graphene contents above 6% by wt.. In fact, these samples showed a high viscosity during resin processing, which eliminated air during the mixing and resulted in the presence of extensive voids in the 8% by wt Graphene samples (Fig. 9 (d) SEM image of GFRP + 8% Wt. Graphene). Also the Graphene /epoxy interfacial adhesion appeared to be weak and hence flexural strength was reduced.

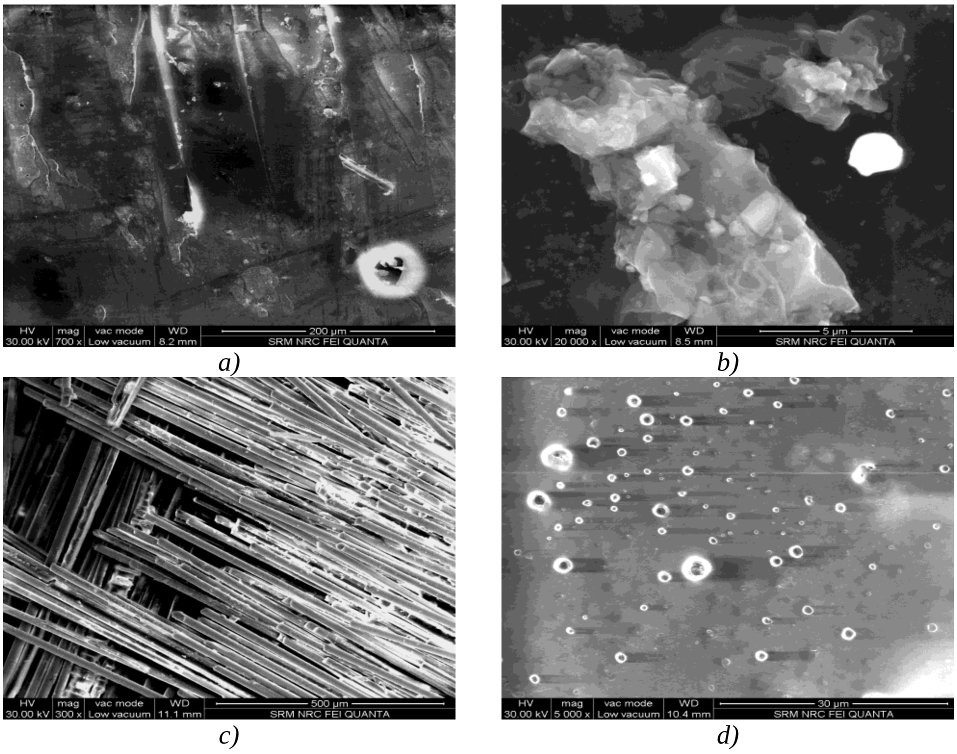
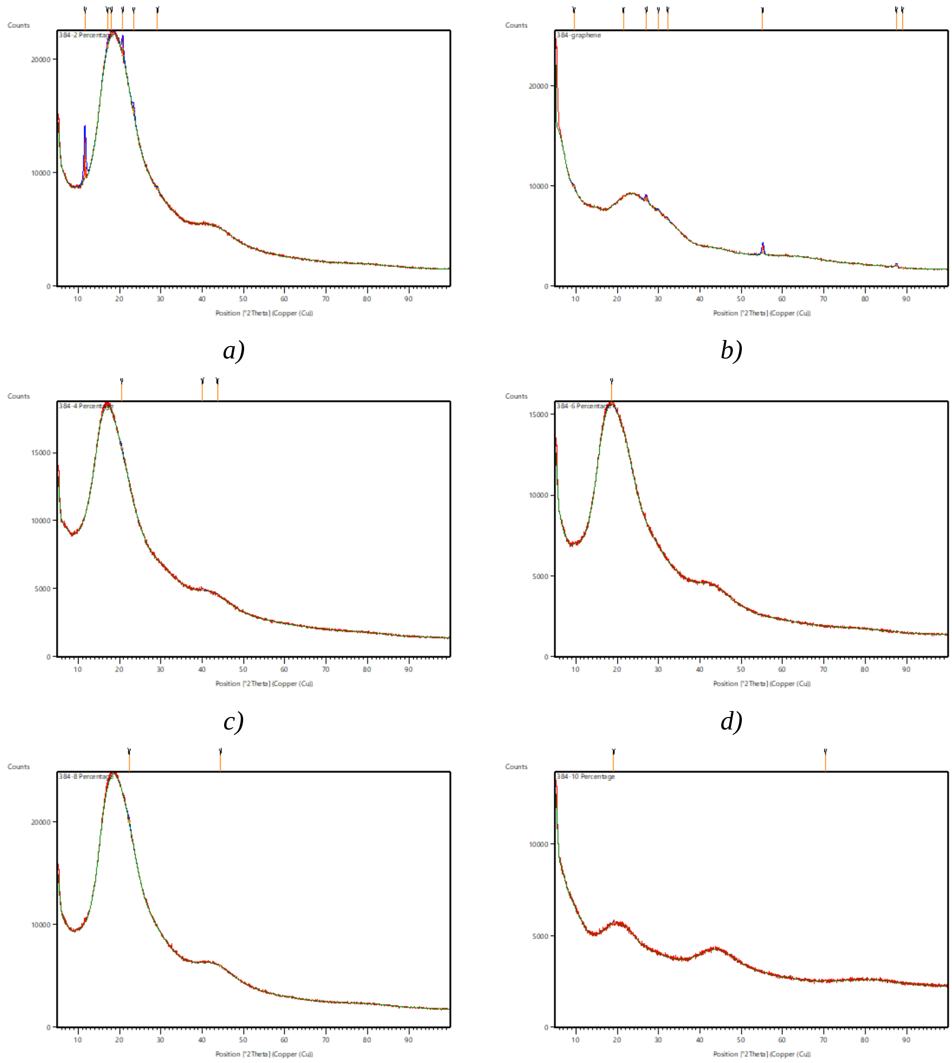


Fig. 1 SEM image of GFRP
a) + 2% Wt. Graphene, b) + 4% Wt. Graphene,
c) + 8% Wt Graphene., d) + 10% Wt Graphene.

5.6. Identification of Crystalline material (Graphene) by XRD

X-ray diffraction analysis was made for predicting the intensity of the crystalline material in each specimen. XRD analysis was performed on samples of graphene reinforced composites to identify important solid state structural information such as the degree of cyrstallinity. The presence and the relative quantity of crystalline material (Graphene) depend on how the polymer was formulated and processed and this in turn is known to affect various mechanical properties. Consequently the degree of crystallinity is an important property to enable determination with accuracy. XRD aids in the identification of crystalline polymers in polymer samples using the position and relative intensities of crystalline phases of the polymer composite. Polymers are processed into fibres and composite films with crystalline material while the orientation of molecules is predicted using diffraction.



a) b) c) d) e) f)

Fig. 10. XRD image of a) Graphene Powder, (b) GFRP + 2% Wt. Graphene c) GFRP+4% Wt. Graphene, d) GFRP+6% Wt. Graphene, e) GFRP+8% Wt. Graphene, (f) GFRP+10% Wt. Graphene

XRD pattern of pure Graphene powder was recorded and used as a reference for compositions of different composite.

X ray diffraction patterns GFRP/ Graphene (2%, 4%, 6%, 8 % and 10 %) composite system are shown in Figure. The XRD pattern of the pure Graphene indicated the presence of crystalline behaviour. Addition of Graphene made to various compositions in GFRP to form the different compositions of the composite showed the amorphous nature of composites while increased with increase in the concentration of the added Graphene, because the crystalline structure of the Polymer vanishes by increasing the Weight percentage of Graphene. The absence of peak in the intensity versus 2θ curve represents complete amorphous state of the composites. Indication of peak to peak in curve led to the inference of the formation of phase or phases in the composite during polymerization process. All the spectra for 2, 4, 6, 8 and 10% wt. Graphene reveal an amorphous halo in the low 2θ region. This is due to short-range order and indicating these composite films as amorphous. XRD pattern showed the semi-crystalline nature of composites.

6. Conclusions

In general, GFRP reinforced with Graphene platelet powder provides a better performance than from the commercially available normal GFRP. Experiments conducted with Glass fiber reinforced polymer composites with different variations in epoxy resin reinforcement along with Nano fillers of 2%, 4%, 6%, 8%, 10% of Graphene were successful and urbanized with a minimum percentage of voids using the ultrasonication method of fabrication at room temperature.

The tests showed the accumulation of Nano fillers causing a reduction in the brittleness of composite laminates and augmentation of the tensile and flexural strength.Based on the tests carried out on the Graphene reinforced composites, the following conclusions have been drawn:

Incorporation of Nano fillers, mechanical properties such as flexural strength and fracture toughness caused an increase of heartening results but, these properties get depleted after toting up of additives of a certain amount. The mechanical properties were appreciable and better only till 6% and the properties got deteriorated beyond this.

Increase in the percentage of filler content caused increase in the void content in reinforced epoxy composites and some agglomerates were generated. This gave rise to defects in higher value of Graphene contents.

The addition of Nano filler material with bidirectional GFRP composite increases the tensile strength by 46.5% of 3 mm thickness specimen (GFRP with 6% Wt. Graphene) respectively when compared to Neat GFRP. The Young's modulus of the composite is increased on addition of Graphene weight at 6% and get reduced on further addition of Nano filler.

The addition of Graphene in the epoxy resin leads to better ductility in GFRP composites when compared to neat GFRP.

The GFRP composite of 6% weight of graphene was found to exhibit superior properties in contrast with other GFRP composites of different Nano filler weight percentage.

The conclusion is that the tensile and flexural strength of the graphene reinforced GFRP increases with the addition of increasing percentage of graphene till 6% wt. But there is a depletion of the properties with further addition of nano fillers.

Scope of future work

The present research work experimented and investigated the addition of graphene to the GFRP Composite. The GFRP composite with 6% graphene weight showed noteworthy improvement in tensile and flexural strength. The following can be looked into and explored in depth for the scope of future work.Investigation of the mechanical properties using reinforcement of Graphene into carbon fibre reinforced polymers (CFRP) and BassaltFibre reinforced Polymers (BFRP).The effect of Graphene Nano filler on Dynamic Behaviour of FRP can be studied and reviewed.

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