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Effects of multi walled carbon nanotubes and graphene on the mechanical properties of hybrid polymer composites

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ABSTRACT

A small amount of Graphene and Multi walled carbon nanotubes (MWCNT) by direct mixing were employed to disperse these nanoparticles into a mono-component epoxy system and used as matrix for advanced composites with woven Glass and Carbon fiber reinforcements. These nanoparticles were added directly into the hosting system and dispersion was carried out by using mechanical stirring. In this study the hybrid polymer composite with Glass fiber, Carbon fiber and epoxy polymer in the ratio of 9:12.5:78.5, 13.5:18.75:67.75 and 18:25:57 percent of volume with addition of fillers, Graphene (0.2 wt%) and MWCNT (0.2wt%) have been developed. The mechanical characterization results confirm that the composite developed by using graphene nanoparticles represents a fundamental feature in enhancing the tensile elastic modulus and hardness behavior of the composite system, where as MWCNT has significant effect on the bending modulus and impact behavior. The optical microscopic study for the fractured samples reveals a significant increase in the fiber-matrix interface adhesion where as decrease in fiber breakage, fiber pullout and debonding. Copyright © 2013 VBRI press.

Keywords: Multi walled carbon nanotubes (MWCNT); graphene fillers; hybrid polymer composites.



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Introduction

In recent years, composite materials have found increasing applications in construction, aerospace and automotive industries due to their good characteristics of light weight, improved strength, corrosion resistance, controlled anisotropic properties, reduced manufacturing and maintenance costs. However, there is a growing demand to improve on composite materials with reduction in the cost of construction [1]. Hybrid Polymer Composites (HPC) are one of the recent developments to reduce the cost of expensive composites containing reinforcements like carbon fiber by incorporating a proportion of cheaper, lowquality fibers such as glass, textile, natural fibers etc. and fillers like Multi walled carbon nanotubes (MWCNT) and graphene, without reducing the mechanical properties of the original composite.

The properties of a hybrid composite mainly depend upon the fiber content, length of individual fiber, orientation, extent of intermingling of fibers, fiber to matrix bonding and arrangement of both the fibers. The strength of the hybrid composite is also dependent on the failure strain of individual fibers. Maximum hybrid results were obtained when the fibers are highly strain compatible [2].

Since the discovery of CNTs, they have been the focus of frontier research. It has opened vast areas of research which also includes nanoscale reinforcements in composites in order to improve their mechanical, thermal and even electrical properties. Although the focus of the research in nanotubes based composites has mostly been on polymer based composites, the unique properties of carbon nanotubes can also be exploited in hybrid polymer matrix composites. Understanding the basic science and technology of carbon nanotubes [3] can be very useful for better insight into the vactual proceedings of the work of research. Starting with the fundamentals, the stunning discovery of fullerenes by Nobel laureates namely Dr. Richard Smalley, Dr. Harold Kroto and Dr. Robert Curl in the year 1985 opened a new area of carbon chemistry. Carbon nanotubes were first observed under an electron microscope by Sumio Iijima and his co-workers. It has been the object of intense scientific research ever attempted in this area. The multi-walled carbon nanotubes with a diametrical range of 5-40 nm are known for their exceptional mechanical properties. MWCNT whose modulus is comparable to that of diamond (1.2 TPa), are reported to have strengths 10- 100 times higher than the strongest steel at a fraction of the weight [4]. This, coupled with approximately 500 times more surface area per gram (based on equivalent volume fraction of typical carbon fiber) and aspect ratios of around 1000, has created a great deal of interest in using CNT as a reinforcing phase for polymer matrices.

Multiple reports showed the positive effect of carbon nanotube filling on the crack propagation resistance of polymer resins [5, 6]. Gojny F..H. and others [7] have tested a standard epoxy resin mainly used for resin infusion filled with functionalized and unfunctionalized nanotubes. According to their tests a 43% increase in the mechanical properties of the resin could be observed at a 0,5% loading of amine functionalized DWCNTs (double walled carbon nanotubes). Ganguli and others [8] examined the effect of MWCNT filling on the fracture toughness of a tetrafunctional epoxy resin through single edge notch threepoint bending tests. They have measured a threshold increase in the stress intensity factor at 1% weight MWCNT loading. Delamination occurs in the resin filled interlayer between the reinforcement material layers in composite structures. So any toughening of the matrix material can improve delamination resistance. Some promising theoretical results [9] and improvements in other interlaminar properties like shear strength [10, 11] have been reported.

Only limited work could be found in the literature regarding the mechanical characterization of hybrid polymer composite using Glass fiber, carbon fiber and epoxy polymer with the addition of filler like Graphene. Studying effect of graphene is expected to provide a fundamental insight into all carbon materials. In comparison with carbon nanotubes, graphene exhibits potential advantage of low cost, high surface area, ease of processing and safety [12], excellent thermal conductivity [13] and strong mechanical strength [14]. Another mass production method is chemical [15] or thermal reduction [16] of graphite oxide. Various attempts have been made in mechanical characterization of the composites, but they

have been concentrated only on a few fibers, the vast area in composites remains unexplored. In the present investigation, the studies are carried out and interpreted the mechanical morphological properties of glass-carbon reinforced epoxy based hybrid polymer composite. In this work the experiments have been conducted in three variations *viz*, glass-carbon epoxy resin with and without the fillers, Graphene and MWCNT. The comparison mentioned in theses variations shows the properties of each composite system. Hence it is of primary importance to develop an alternative for carbon nanotubes in the hybrid polymer composites. The experimental results which have been derived out of this investigation will reveal the different mechanical properties using fillers like MWCNT and graphene.

The main novelty of this work is related to the explanation of the major benefits by replacing MWCNT to graphene in order to reduce the cost of the hybrid composite system for the structural applications like aerospace, military and space settles etc, without seriously reducing its mechanical and morphological properties. graphene based hybrid polymer composites may have wide potential applications due to their outstanding properties and the availability of graphene in a large quantity at low cost.

Experimental

Materials

The present investigation has been carried out with reinforcements like Glass, Carbon woven fibers with epoxy resin (LAPOX L-12) at a room temperature with a curing hardener (K-6). All these polymer products were supplied by Atul Limited, Polymer Division (Gujarat, India). Fillers like multi walled carbon nanotubes (MWCNT) with carbon purity more than 90%, carbon nanotubes of diameter 20-40nm and surface area 500m²/gm supplied by Applied Science Innovations Private Limited (Maharashtra, India). Graphene was synthesized from natural flake graphite with carbon purity more than 85% by the modified Hummer's method with pre-oxidation treatment [17]. The matrix material was of medium viscosity epoxy resin of moderate cost. This requires minimal setup costs and the physical properties can be tailored to specific applications. The plain-woven glass having 0.19mm thickness and carbon fiber mats having 0.37mm thickness were the reinforcements and all the fibers in the fabric have diameters less than 30 µm. These are manufactured by Interglas Technologies (Benzstrasse, Germany).

Development and processing of hybrid polymer composite

The aim of this is to determine the mechanical characterization of Glass-Carbon-Epoxy with and without fillers thermosetting composite material. To fabricate the specimens by hand layup method **[18, 19]**, which is cost effective and easy process of manufacturing, the layers of cross-ply glass and carbon laminates each of 0.18 and 0.25mm thickness alternatively placed and having 350 mm length and 350 mm width was put together to form a block with dimension of 350*350*4 mm for mechanical characterization. Different weight percentages of fibers, fillers and matrix materials used is shown in **Table 1**, which

can be combined to further enhance the overall performance of the laminated composite material. Resins are impregnated by hand into fibers, which are in the form of woven, knitted, stitched or bonded fabrics. This is usually accomplished by rollers or brushes, with an increasing use of nip-roller type impregnators, for forcing resin into the fabrics by means of rotating rollers and a bath of resin. Laminates are left to cure under standard atmospheric conditions.

 Table 1. Material Composition for development and processing of composites.

Samples		W2 Serie	es		G2	Series			C2	Series	
Fibers, Matrix & Fillers (%weight)	Glass	Carbon	Epoxy	Glass	Carbon	Epoxy	Graphene	Glass	Carbon	Epoxy	MWNCT
Sample1	9	12.5	78.5	9	12.5	78.5	0.2	9	12.5	78.5	0.2
		W3 Serie	es		G3	Series			C3	Series	
Sample 2	13.5	18.75	67.75	13.5	18.75	67.75	0.2	13.5	18.75	67.75	0.2
		W4 Serie	es		G4	Series			C4	Series	
Sample 3	18	25	57	18	25	57	02	-		-	-

Tensile test

The tensile test is performed on the prepared specimens of composite to determine the elastic properties of the composite. The most commonly used specimen geometries are the dog-bone specimen and straight-sided specimen with end tabs. The tension test is performed on all the five samples as per ASTM D638 [20] test standards using computer controlled Universal Testing Machine (UTM) with 50kN load cell. This is subjected to monotonic uniaxial tension at a displacement rate of 4 mm/min on 4 mm thick strips, with a gauge length of 25 mm under low strain rates of $6.5 \times 10^{-4} \text{ S}^{-1}$. The tests are closely monitored and conducted at room temperature. The load extension values are recorded to generate the stress strain curves.

Flexural test

The determination of flexural strength is an important characterization of any structural composites. It is the ability of a material to withstand the bending before reaching the breaking point. Conventionally a three point loading has been conducted for finding out the flexural properties of the composites using computer controlled Universal Testing Machine (UTM). A span of 80 mm and thickness of 4mm under low strain rates of $6 \times 10^{-4} \text{ S}^{-1}$ was taken and cross head speed was 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. The test was performed as per the ASTM D790 [21].

Pendulum impact test

The impact test is carried out as per the ASTM D 256 [22]. The pendulum impact testing machine ascertains the notch impact strength of the material by shattering the specimen with a pendulum hammer, measuring the impact energy, and relating it to the cross section of the specimen. The standard specimen used for test is $60 \times 13 \times 4$ mm. The specimen's dimensions with respect to the ASTM standards are loaded on an impact tester (which is used for polymer

Hardness test

Hardness test measurement is carried out using Durometer hardness tester as per ASTM D2240 **[23]**. A ball indenter of the tester is pressed into the material under hand pressure on the knob which is at the top of the instrument under room temperature. The hardness of the specimen tested is indicated directly on the dial gauge of the instrument.

Factrography

The surfaces of the composite specimens are examined directly by optical microscopic images. The samples are placed in the microscope; the required magnification is done using the fine tuning knobs of the instrument. The images are taken with the help of the camera for the purpose of study.

Results and discussion

Experiments have been carried out to characterize the candidate composite material under different loading conditions and with various specimen configurations. The analysis of the results and the influence of various parameters on the properties are summarized in the following sections.

Tensile behavior of composite

Five specimens from each variation of the composite were tested. The dimensions of the test coupons were 165 mm in length, 12 mm in width, and, 4 mm in thickness respectively. The 40 sample specimens were tested at a cross-head speed of 4 mm / min. Stress versus strain responses were plotted and are shown in **Fig. 1** to **8**.

The average modulus for the glass-carbon epoxy composite with and without fillers was found to be 2.44 MPa, 3.03 and 2.16 MPa respectively. Fig. 1, 2 and 3 indicate the insight tensile behavior of W2, G2 and C2 series hybrid polymer composite samples.



Fig. 1. Stress strain graph (W2 Series).



Fig. 2. Stress strain graph (G2 Series).



Fig. 3. Stress strain graph (C2 Series).



Fig. 4. Stress strain graph (W3 Series).

 Table 2. Elastic properties for W2 G2 C2 series specimens.

SI. No.	Young's Modulus (N/mm ²) W2 Series	Young's Modulus (N/mm ²) G2 Series	Young's Modulus (N/mm ²) C2 Series
1.	3.33	3.00	2.00
2.	2.20	3.00	2.50
3.	2.20	2.86	2.00
4.	2.50	3.33	2.00
5.	2.00	3.00	2.00
Average	2.44	3.03	2.16

From these results, it is concluded that the average modulus of elasticity will be increased by 19.44% between the hybrid composite samples made from without Graphene and MWCNT and with Graphene. Similarly it is observed that the modulus of elasticity or young's modulus increased by 28% in case of hybrid composite samples made with Graphene filler as compared with the composite made of MWCNT cited in **Table 2**.



Fig. 5 Stress strain graph (G3 Series).



Fig. 6. Stress strain graph (C3 Series).

Table 3. Tensile test results for W3, G3 and C3 series specimens.

SI. No.	Young's Modulus (N/mm ²) W3 Series	Young's Modulus (N/mm ²) G3 Series	Young's Modulus (N/mm ²) C3 Series
1.	3.25	3.55	2.20
2.	3.75	3.55	1.30
3.	3.00	3.60	1.60
4.	3.75	3.40	1.60
5.	3.00	3.95	1.60
Avg. Value	3.35	3.61	1.70

Fig. 4, 5 and 6 give the insight about tensile behavior of the W3, G3 and C3 series hybrid polymer composite samples. From the above results, it is observed that the average modulus of elasticity will be increased by 7% between the hybrid composite samples made from without graphene and MWCN and with graphene. Similarly it is observed that the modulus of elasticity or young's modulus will be increased by 52% in case of hybrid composite samples made with Graphene filler as compared with the composite made of MWCNT cited in the **Table 3**.



Fig. 7. Stress strain graph (W4 Series).



Fig. 8. Stress strain graph (G4 Series).

Table 4. Results of tensile test for W3, and G3 series specimens.

Samples	Young's Modulus (N/mm ²) W4 Series	Young's Modulus (N/mm ²) G4 Series
1	6.66	7.15
2	6.66	7.20
3	6.66	7.50
4	6.66	7.25
5	6.66	7.30
Avg. Value	6.66	7.28

Fig. 10 and 11 give the insight of tensile behavior of the W4 and G4 series hybrid polymer composite samples. From these results, it is observed that the average modulus of elasticity will be increased by 8.51% (cited in the **Table 4**) between the hybrid composite specimens made with and without Graphene fillers. These results are in good agreement with result showed by J-H. Du, J. Bai, and H-M. Cheng [24].

Flexural behaviour of composite

Five specimens from the variations of the composite were tested. The dimensions of the test coupons were of 127 mm in length, 13 mm in width, and, 4 mm in thickness respectively. The 10 sample specimens were tested at a cross-head speed of 4 mm / min. Stress versus strain responses were plotted and are shown in **Fig. 9** and **10**.



Fig. 9 Stress strain graph (W4 Series).

Fig. 9 and 10 give the insight into flexural behavior of the W4 and G4 series hybrid polymer composite samples. From these results it is observed that the significant effect of average ultimate bending stress will be increased by 35.4% (cited in the **Table 5**) in case of composite specimens made without Graphene fillers. These results are in good with the results showed by Buong Woei Chieng and Nor Azowa Ibrahim [25].



Fig. 10. Stress strain graph (G4 Series).

Table 5. Flexural test results for W4 and G4 series specimens.

Samples	Ultimate bending Stress (N/mm ²) W4 Series	Ultimate bending Stress (N/mm ²) G4 Series
1.	019	010
2.	018	012
3.	017	011
4.	017	011
5.	017	013

Impact strength behaviour of composite

Impact test reflects the ability of material absorbing energy at fracture, when exposed to sudden impact. The dimensions of the test coupons were 60 mm in length, 13 mm in width, and, 4 mm in thickness respectively. The 40 sample specimens were tested with a pendulum of 1 Kg in weight. Results are shown in **Fig. 11** to **13**.

Fig. 11 gives the insight into the impact strength behavior of the W2, G2 and C2 series hybrid polymer

composite samples. From the above results it is observed that the average impact strength will be increased by 48% (cited in the **Table 6**) between the hybrid composite samples made with and without Graphene fillers.

	Impact Strength in J/m		
Samples	W2	G2	C2
1	48.78	48.78	85.36
2	51.16	97.56	140.00
3	35.71	59.52	83.33
4	73.17	47.05	85.36
5	47.61	47.61	85.36
Average	31.30	60.10	95.88

Table 6. Impact strength results of for W2, G2 & C2 series specimens.



Fig. 11. Impact strength v/s specimen variation (W2, G2 and C2 series).



Fig. 12. Impact strength v/s specimen variation (W3, G3, C3 series).

 Table 7. Impact strength results for W3,G3 and C3 series specimens.

	Impact Strength in J/m			
Samples	W3 Series	G3 Series	C3 Series	
1	49.38	58.82	116.27	
2	59.52	104.65	96.38	
3	58.52	48.19	178.57	
4	68.29	48.78	178.57	
5	58.13	71.42	96.38	
Average	58.76	66.32	133.23	

Fig. 12 gives the insight into the impact strength behavior of the W3, G3, and C3 series hybrid polymer

composite samples. From these results it is observed that the average impact strength is increased by 11.39% (cited in the **Table 7**) between the hybrid composite samples made from without Graphene (W3 series) and with Graphene (G3 series).

Table 8. Impact strength results of for W4 & G 4series specimens.

	Impact Strength in J/m			
SINo.	W4 Series	G4 Series		
1	72.81	60.24		
2	59.52	83.33		
3	91.95	97.56		
4	58.82	73.17		
5	71.42	70.58		
Average	70.90	76.97		



Fig. 13. Impact strength v/s specimen variation (W4, G4, series).

Fig. 13 throws light on impact strength behavior of the W4 and G4, series hybrid polymer composite samples. From the results, it is observed that the average impact strength will be increased by 8% (cited in the **Table 8**) in case of hybrid composite samples made with 0.2wt% of Graphene and MWCN. The results cited in the above tables 5, 6 and 7 are in good agreement with results showed by Buong Woei Chieng and Nor Azowa Ibrahim **[25]**.

Hardness of composite

Five specimens from the different variations of the composite were tested. The 40 sample specimens tested with a durometer are shown in **Fig. 14 - 16**.

The measured hardness values of all the three variations of the composites are presented in **Fig. 14-16**. The hardness properties of the composites are studied by applying indentation load normal to fibers diameter and normal to surface area of the specimen. The effect of fiber loading and post curing time on shore-D hardness is illustrated in **Fig. 14 -16**. It can be seen that the hardness values are increases by 0.05% (cited in the **Table 9-11**) in case of G4 series composites samples made with fillers like Graphene as compared with C2, C3 and W2,W3 series samples made with and without MWCNT. The results cited in the tables

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9, 10 and 11 are in good agreement with the results showed by Jonathan N. Coleman, Umar Khan [26].

Table 9. Hardness values for W2, G2 &C2 specimens.

Load points	Hardness Shore D W2 Series	Hardness Shore D G2 Series	Hardness Shore D C2 Series
1	70	75	76
2	72	75	75
3	71	74	75
4	72	73	74
5	69	75	75



Fig. 14. Hardness number v/s specimen variation (W2, G2, C2 series)

Table 10. Shore D hardness values for W3, G3 and C3 series specimens.



Fig. 15. Hardness number v/s specimen variation (W3, G3, C3 series)

Surface morphology of composite

Investigations on fractured surfaces by optical microscopic examination is determine the failure criteria and further to identify the mechanism involved in improving the strength by observing the phase morphology in glass carbon epoxy composite. Brittle fracture was characterized by a smooth corrugated surface, which could also be the result of fiber de-bonding in the interface or at cleavage. However, the weak interface does not necessarily refer to less strength. Interfacial failures somehow cause fiber bridging that enhances the de-lamination growth and combined with fiber pull-out will dissipate additional energy.

Table 11. Shore D hardness values for W4 and G4 series specimen.

SI. No.	Hardness Shore D W4 Series	Hardness Shore D G4 Series
1	75	80
2	70	82
3	73	80
4	72	80
5	70	81



Fig. 16. Hardness number v/s specimen variation (W4, G4, series).

Morphology of fractured specimens in tensile test

The investigations carried on the variations show that there is fiber pullout of glass fibers observed for all variations of composites rather than the carbon fibers. The delamination is also seen in all variations except W2, G2, C2 series of specimens. The resulting microscopic images are shown in Fig. 17 & 18. The fracture of the test coupons, shown in the Figure 20a and b, began with delamination. At these points an accumulation of stresses occurred and this is shown by the crazing (white area) on the coupon. Thereafter there was catastrophic failure along the width of the specimen as the load bearing area decreased. The test coupons exhibit a fracture path that is angled across the thickness of the sample. This angle was determined to be approximately 15° to the vertical in Fig. 17a, b. and 18a, b. The delamination and fiber pullout are significantly lower in case of composite samples processed using without graphene filler and with MWCN as witnessed by the microscope images shown in the Fig. 17a-b.



Fig. 17. (a) Images of tensile fracture W3T (b) Images of tensile fracture C3T.



Fig. 18 (a) Images of tensile fracture G4T (b) Images of tensile fracture W4T.

Morphology of fractured specimens in Flexural test

The microscopic images shown in **Fig. 19 a, b** and **20a, b** taken during the microscope examination give the details like disintegration of the matrix (particles) from the fiber. This has happened on the opposite surface where the load is applied. The delamination has occurred just after the matrix breakage. This can be clearly observed from the above **Fig. 19** and **20.** The delamination, fiber pullout and matrix breakage is significantly lower in case of composites processed and developed using fillers like graphene and

MWCNT. This can be witnessed by the images shown in **Fig 20**a-b.



Fig 19 (a) Images of bending fracture G2B (b) Images of bending fracture W3B.



Fig 20 (a) Images of bending fracture C3B (b) Images of bending fracture G4B.

Morphology of fractured specimens in impact test

Micrographs (Fig. 21a, b, c) confirm the brittle nature of the composite with deeper micro-cracks. During normal impact the largest part of the initial energy is converted into heat and hence matrix is softened which resulted in embedment of cured resin particles (Fig 21). The embedded cured resin particles control the further fracture of the target surface. Fig. 21 b, c shows micrographs of fractured surfaces of 0.2wt% of MWNCT and Graphene glass-carbon fiber reinforced epoxy composite. At fracture surface, micrographs (Fig. 21a, b) show matrix that plastically deformed and amount of deformation is proportional to impact velocity of particles. Further the resin was clearly adhered to the fibers. Hence the removal of matrix along the length of the fiber and subsequently exposed fiber getting removed can be seen from the micrograph (**Fig. 21 b, c**).



Fig. 21. Micrographs at fracture surface (a) Without MWCN and Graphene (b) 0.2% wt MWNC and (c) 0.2% wt Graphene.

Conclusion

The laminated hybrid polymer composites with different variations of glass carbon epoxy resin reinforcement along with fillers of 0.2 wt% of Graphene and MWCNT was successfully processed and developed with minimum percentage of voids using hand lay- up method of fabrication at room temperature. The experiments on tensile tests conducted with the chosen variations of glass, carbon reinforcements with 0.2 wt% Graphene and MWCNT, show that there will be an increase in modulus of elasticity by 10 to 15% and also sustain greater loads. Similarly the flexural behavior indicates that the average maximum bending strength and average ultimate stress will be increased by 35.4% without using 0.2 wt% Graphene fillers. Hence it can be concluded that the flexural strength decreases by adding graphene fillers. Further, it is concluded that the impact strength increases by 48% without use of fillers and 37% in case of composite made

with Graphene and MWCNT respectively. Also it can be seen that the hardness of the hybrid polymer composite increases by 12.34% by adding 0.2 wt% of Graphene and 8.5% by adding 0.2 wt% MWCNT. Hence it can be concluded that the composite made with graphene fillers have higher hardness than that of MWNCT. The effect of Graphene and MWCNT on delamination, fiber pullout, fiber breakage and voids are studied by using optical microscope, in which it is observed that the delamination and fiber breakage are minimal by adding Graphene and MWCNT fillers. The fiber pullout is seen in tensile, flexural loadings, but with impact load there is less fiber pullout.

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