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Effects of Multi Walled Carbon Nanotubes and Alumina Filler on the Mechanical Properties of Polymer Composites

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Abstract : The focus of this paper is to explore the effects of MWCNTs/Alumina as filler on mechanical properties of polymer composite. Aluminum Oxide (Al_2O_3) and Aluminum Nitrate (Al $(NO_3)_3 \cdot 9H_2O$) were used as filler materials in MWCNTs-glass fiber reinforced epoxy composite. Scanning Electron Microscopy (SEM) results showed homogenous dispersion of the Alumina fillers in MWCNTs. However, Aluminum Nitrate/MWCNTs based sample suffered from aggregation problem during the fabrication of composite. In addition, the uniaxial tensile tests, flexural and impact tests were employed to characterize mechanical properties of MWCNTs-based glass composite.The addition of 0.5 wt. % MWCNTs/Alumina fillers showed a significant improvement in tensile strength and flexural modulus of composite as compared to baseline virgin glass composite sample. The Aluminum Nitrate/MWCNTs-based composite showed higher tensile strength than Aluminum Oxide/MWCNTs-based composite. The flexural strength and modulus of Aluminum Oxide/MWCNTs-based composite was enhanced by 23% higher than other fillers composite. In particular, Aluminum Oxide addition seems to increase the impact strength approximately by 7% compare to non-heat treated and other fillers.
Keywords: Glass Fiber, MWCNTs-Alumina Fillers, Tensile Strength, Mechanical Properties.

Introduction

Glass fiber reinforced polymer (GFRP) composites have shown excellent performance combined with high strength, high stiffness and low moisture absorption. GFRP composite have widely used for structural and functional applications[1]. In addition, with the development of many industries such as aerospace systems, superconducting magnet and cryogenic equipment, the properties in terms of mechanical strength and thermal conductivity were proposed higher requirement GFRP based composite has been widely used[2,3]. Multiple reports showed the positive effect of carbon nanotube addition on the crack propagation resistance of polymer resins[4,5]. Gojny have tested a standard epoxy resin mainly used for resin infusion filled with functionalized and un-functionalized nanotubes. With addition of 0.5 % functionalized CNTs the 43 % increment in the mechanical properties of the resin was reported by the authors[6,7]. One of the most significant parameter in fabricating CNTs reinforced composites is the dispersion of CNTs itself. Since the Van der Wasls forces and nanoparticles high surface area causes aggregation in large clusters. Kim has reported that the degree of CNTs dispersion into epoxy strongly affected the matrix-dominated mechanical properties[8]. In the literatures various methods were reported to disperse the nanotubes in polymer resins, such as stirring, sonication and high shear mixing[9,10,11]. Brigatti reported that the addition of halloysite nanotubes in the matrix woven fiber epoxy nanocomposites significantly increased the flexural strength about 14% and the interlaminar shear strength approximately 25%[12]. Liao used alumina nanoparticles (25 nm) in carbon fiber epoxy nanocomposites and the flexural toughness and interlaminar shear strength were improved significantly[9]. However, with addition of 48 µm Al particles in the epoxy matrix of a woven continuous glass fiber composite resulted in 78% increase in flexural modulus and 33% increase in flexural strength[13]. With 20-30 nm silica particles in the epoxy matrix of a glass fiber fabric composite the interlaminar fracture toughness and impact resistance considerably increased[14]. Electrophoretic deposition (EPD) is one of the effective methods to manufacture GFRP composites at a large loading of CNTs. During an EPD process, surface charged CNTs are uniformly dispersed into a liquid medium and deposited onto woven layers with an electric field. Bekyarova[15] deposited CNTs on to woven carbon fabric and manufactured composites. Some researchers proposed the multi-scale hybridization of CNTs with various types of micro particles, where the CNT structure and hybrid organization can be tailored by adjusting synthesis parameters[16-20]. With the addition of hybrids into polymer matrix, the uniform CNT dispersion and improved interfacial properties were achieved[21,22,23]. It is more recently that the CNT–graphene nanoplatelet (GnP) and CNT–silicon carbide (SiC) hybrids were used as high performance reinforcements in the composites[24-26].Combining ceramic micro particles with CNTs may be helpful with the dispersion of CNTs in the polymer matrix. Hence, with the help of the ceramic micro-beads ‘vehicles’, it may be much easier to disperse CNTs by conventional methods[27]. In the present paper, the glass fiber/epoxy composites were prepared by using plain-woven glass fiber into the epoxy matrix modified with MWCNT–Al hybrids. The morphologies of composite were evaluated by scanning electron microscopy (SEM) to enlighten the reinforcing mechanism. The mechanical properties were tested by tensile, flexural and impact tests. The latent of MWCNT–Al hybrids as high performance reinforcements in fibrous composites was investigated and conferred.

Experiment

Materials and Methods

MWCNT were procured from Janaki SciTech Chemicals Co. Ltd., with size of 10-20 nm in external diameter and 10-30 µm in length (95% purity) and Alumina micro particles with 5-8 µm in length (Sigma Aldrich Co Ltd) were used. Commercial High strength Bi-directional E-Glass fibre was used in current study. Epoxy resin was used to form matrix material. The MWCNT-alumina micro particles were synthesized by Sol-gel process. Aluminum oxide (Al_2O_3) and Aluminum nitrate ($Al(NO_3)_3 \cdot 9H_2O$) was used as a precursor of alumina for preparing two different alumina fillers. Alumina sol was synthesized by Yolda’s process, which consists of hydrolysis and peptization of alumina hydroxide[28]. The MWCNTs dispersed in the form of suspension within ethanol were added to alumina sol during the gelation process. The MWCNT-alumina gel was dried at 350 °C for 6 hours. For the heat treated samples the MWCNT-alumina hybrid nano fillers were by calcinations at 900 °C for 1 hour in vacuum of 1Pa[29].

Composite Fabrication

Bi-directional E-Glass fiber mats were reinforced into the epoxy resin with addition to various kinds of filler, namely, the MWCNT, MWCNT- Aluminum Nitrate, and MWCNT- Aluminum Oxide hybrids. The preparation of Epoxy/(0.3 wt%) MWCNT-(0.2 wt%) Alumina hybrid filler mixture was sonicated (Sonics Vibra Cell ultrasonic processor) for 25 min for homogenous distribution. The composite were prepared using hand layup method, the fibers were stacked into plies without changing the fiber orientations. The glass fibre /neat epoxy composites were also prepared using neat epoxy to serve as reference samples.

Characterization

The morphological features of the MWCNT reinforced in Alumina composite powder and their dispersion in the glass fiber/epoxy composites were characterized by scanning electron microscopy. The tensile tests were performed to measure the elastic properties of the composite. The tensile tests were performed on samples as per ASTM D638 standard using computer controlled Universal Testing Machine. These tests were carried out on rectangular specimens (250 mm x 25 mm x 3 mm) at room temperature. The tests speed was 5 mm/min; an extensometer/strain gauge was used to determine the elongation and tensile modulus. Flexural strength is the ability of the material to resist bending under the application of load. The flexural strength and modulus of the composites was determined using the three point bending test method according to ASTM D790 using computer controlled Universal Testing Machine. Samples were cut into rectangular sections of 120 mm x 12 mm x 3 mm and the load was applied midway between the supports. The Charpy’s impact tests were a standardized high strain-rate tests which determines the amount of energy absorbed by a material during fracture. Test specimens were cut from the composite sheets having dimensions 60 mm x 13 mm x 3 mm, according to the ASTM D256. The specimen was held as vertical cantilever beam and was impacted by a single swing of the pendulum.

Results and Discussion

Morphology Characterization

Fig.1 (a-b) shows the SEM micrographs of Al nitrate-MWCNT hybrids reinforced in glass fiber/epoxy samples. Significantly, homogenous dispersion of hybrid filler in matrix offer higher possibility of polymer filler interactions. It is observed that the hybrids filler dispersion is randomly distributed on the surface of the glass-epoxy composites. Al_2O_3-MWCNT hybrids reinforced GFRP composites; Fig.1(c-d) shows a large area of matrix containing Al_2O_3-MWCNT hybrids were adhered to the surface of glass fibre, indicating strong interfacial bonding between glass fibre and matrix[30]. The Al_2O_3-MWCNT hybrids dispersed between adjacent Glass-fibres serve as bridges interconnecting the neighbouring fibres to each other. Subsequently, the growth of micro cracks can be slow down by this bridging effect[31]. As a result, the addition of Al_2O_3-MWCNT increases strength of the interfacial regions surrounding the fibres by bridging the micro cracks. Also the Al_2O_3-MWCNT hybrids shows better fiber matrix interaction which is observed in Fig.1 (e)

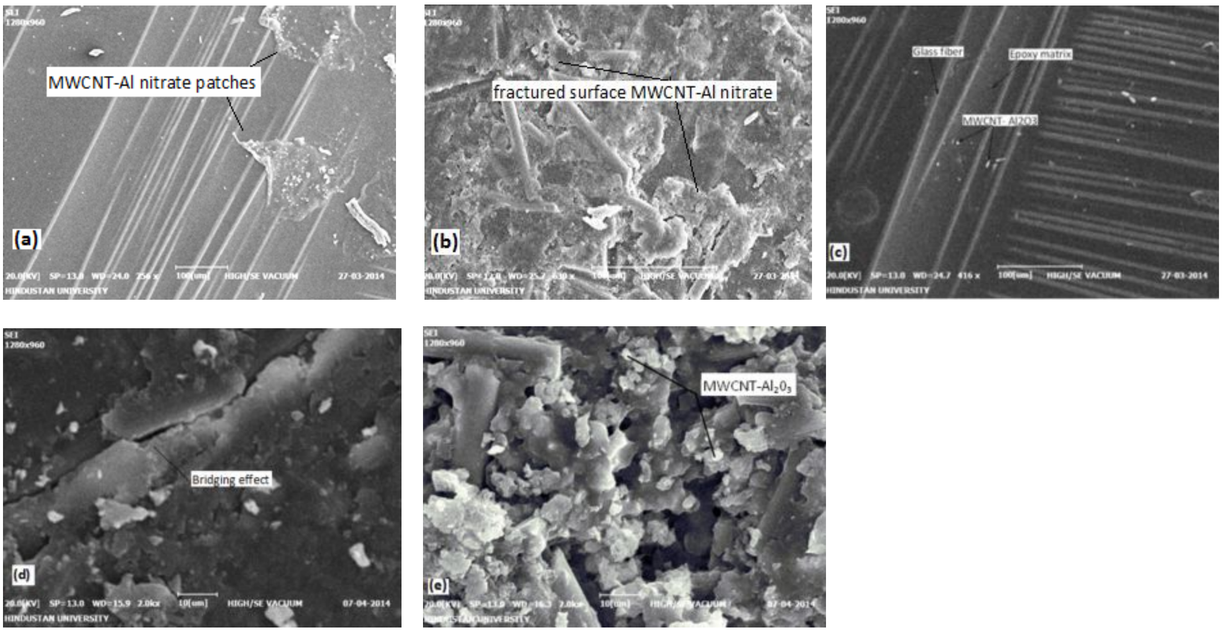

Figure 1. SEM Micrographs of (a) randomly distributed MWCNT-Al nitrate patches on the glass fiber/epoxy composite (b)fractured surface MWCNT-Al nitrate glass fiber/epoxy composite (c) MWCNT-Al_2O_3 glass fiber/epoxy composite (d)Magnification image of MWCNT-Al_20_3 Glass fiber/epoxy composite(shows Bridging effect) (e)Magnification image of MWCNT-Al_2O_3 glass fiber/epoxy composite(shows better fiber matrix interaction).

Tensile properties

Fig.2 shows the load–displacement curves of the glass fibre reinforced with neat and modified epoxy matrix under the uniaxial tensile testing. One can observe from Table.1 that the tensile strength value increased from 182 MPa to 216 MPa. It clearly indicates that heat-treated specimen has higher tensile strength compared non heat-treated specimen. Composites filled by Aluminium Nitrate (Al $(NO_3)_3 \cdot 9H_2O$) exhibited better ultimate strength when one compared with other fillers. This may be attributed to good particle dispersion and better polymer/filler interface adhesion for effective stress transfer[32].

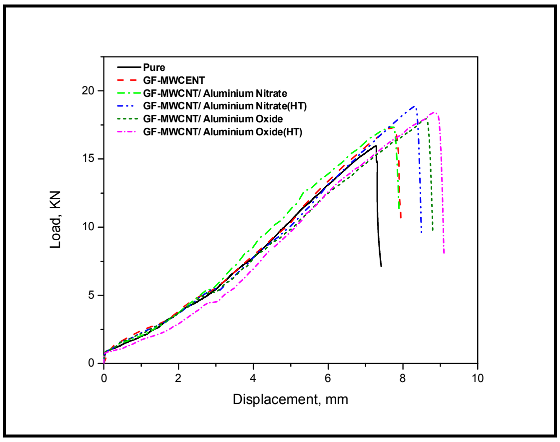

Figure 2. Typical Load Vs Displacement curves obtained from tensile tests of Glass-Epoxy based composites

Table 1 Mechanical Properties of Glass-Epoxy based composites

Sample Formulation	Tensile Strength, (MPa)	Elastic Modulus, (MPa)	Flexural Strength, (M Pa)	Flexural Modulus, (MPa)	Impact Strength, (J)
GF	182.7±3.2	4539.6±23.2	243.33±3.2	1729.6±17.2	1.1±0.08
GF-MWCNT	197.9±2.3	5042.0±21.9	267.88±2.2	1966.5±23.2	2.3±0.1
GF-MWCNT/ Aluminium Nitrate	199.9±4.9	3866.3±17.8	298.82±4.2	2586.2±14.2	4.7±0.3
GF-MWCNT/ Aluminium Nitrate (HT)	216.2±3.8	4307.9±26.5	338.38±3.6	2876.4±18.5	6.6±0.1
GF-MWCNT/ Aluminium Oxide	205.3±2.2	3785.7±20.6	380.43±2.9	3168.2±16.3	6.2±0.3
GF-MWCNT/ Aluminium Oxide(HT)	210.4±3.5	4287.5±18.6	402.70±4.6	3938.8±18.3	7.8±0.2

Flexural properties

Typical load–displacement curve obtained from three point bending tests are presented in the Fig. 3. Compared with the reference samples significant improvement on the flexural strength and modulus can be observed. The result shows that of flexural strength and modulus of Aluminum Oxide (Al_2O_3) filled composites are increased by 16% and 23% respectively. It showed that the filler provide reinforcing mechanism to strengthening the matrix, which is more obvious in the case of heat-treated Aluminum Oxide.

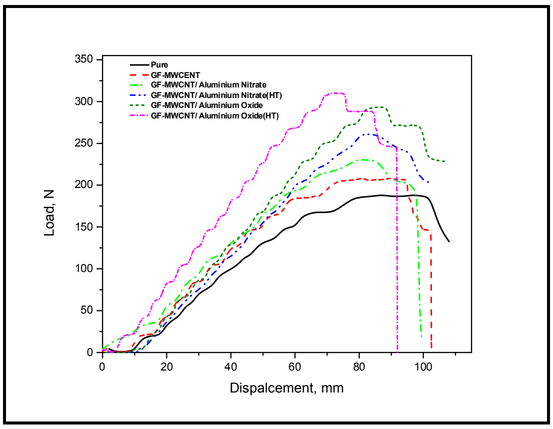

Figure 3. Typical Load Vs Displacement curves obtained from Flexural tests of Glass-Epoxy based composites

Impact properties

The absorbed impact energies of composite samples are shown in Fig 4. These averaged values suggest that the addition of MWCNT-Aluminum Oxide (Al_2O_3) filled GFRP composites enhances the impact energy absorption capacity of the composites almost 7 %. This enhanced impact resistance can be attributed to the increased interlaminar fracture toughness of the fibres. In addition, hybrid filled composite, stiffened matrix laterally supports the slender fibres and increases the impact absorption.

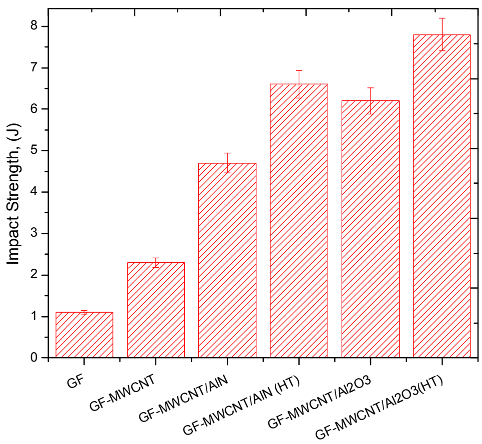

Figure 4. Impact Strength of Glass-Epoxy based composites

Conclusion

In this study, hybrid nano filler were incorporated in glass/epoxy composites to enhance the mechanical properties. Based on the experimental and micrographic results, the following conclusions are drawn:

- Morphological studies revealed that a large number of matrix containing Al-MWCNT hybrids were adhered to the surface of glass fiber, indicating strong interfacial bonding between glass fiber and matrix.
- Composites with Aluminum Nitrate (Al $(NO_3)_3 \cdot 9H_2O$) exhibited better ultimate strength when compared with other fillers.
- The addition of Al- MWCNT heat-treated to 900 °C shows improved flexural strength and modulus of the fibrous composite by 16 % and 23 % respectively.
- It is found that (0.5 %wt) heat treated (Al_2O_3)-MWCNT impregnated GFRP composites yields better impact energy of 7% compare to non-heat treated and other fillers.

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