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EXPERIMENTAL INVESTIGATION ON MECHANICAL BEHAVIOR OF CARBON NANOTUBES –ALUMINA HYBRID EPOXY NANOCOMPOSITES

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The multi scale hybridization of multiwall carbon nanotubes (MWCNTs) with alumina (Al_2O_3) in epoxy offers a new opportunity to fabricate high performance multifunctional composites. In this study the multifacial hybrid composites consists of MWCNTs and alumina particles were incorporated into pure epoxy with the various weight fraction of 0.5,1.0,1.5,2.0,2.5 Wt% of hybrid nano fillers. The hybrid weight ratio of MWCNTs-Al_2O_3 is mixed in the ratio of 1:4. In this study the promising improvements in the tensile strength, micro hardness and impact strength of these composites presence were compared with the pure epoxy. The test specimens of the hybrid composites were fabricated by moulding method at the room temperature. The hierarchical hybrid was observed to exhibit significant improvement of 518MPa of tensile strength, 165H_v of micro hardness and 80% of impact strength respectively. These hybrid reinforcements are mainly attributed to the improvements of epoxy property resulted from the good dispersion of hybrid fillers. The experimental investigation reveals that the mechanical properties of these new class multifunctional composites are improving with hybrid fillers addition in considerable quantity. The field emission scanning electron microscopy (FESEM) was used to study the possible fracture surface morphology.

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*Keywords:*Epoxy, MWCNTs, Alumina, Hybrid nanocomposites, FESEM

1. Introduction

The recent development and advances in nano - scale science and engineering have provided new prospects to develop the composite materials with superior performances. The multi-scale hybridization of carbon nanotubes (CNTs) with micro-particles in polymers offers new opportunity to develop high performance multifunctional composites. Hybrid composites are used as structural materials due to their high strength, low weight ratio and specific modulus [1]. The CNTs play an important role in the load carrying due to their excellent mechanical properties, exceptionally high stiffness, strength, resilience, as well as superior electrical and thermal properties and moreover are attractive candidates for the reinforcement materials [2-4]. The Young's modulus and the yield strength have been doubled and quadrupled for composites with respectively 1 and 4 wt. % nanotubes, compared to the pure resin matrix [5]. Alumina exhibiting high specific stiffness, superior high temperature, mechanical properties and excellent oxidation resistance is widely used in structural applications [6-7]. Rahmanian et al [8] stated that the multiscale composites revealed significant improvement in elastic and storage modulus, strength as well as impact resistance in comparison to CNT–epoxy composites. McGrath et al [9] reveal that the epoxy-alumina composites are a robust materials system and fairly large changes in particle shape, size, and size distribution can be tolerated without causing a substantial change in the thermal and mechanical properties or fracture toughness of the system. This is likely due to a weak epoxy-alumina interaction. Traditional wisdom is supported. When this data are compared

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with existing literature, it is clear that the incorporation of filler into an epoxy matrix is more effective at improving the relative fracture toughness in composites with resins that exhibit a lower initial unfilled matrix toughness. Li et al [10] reported that the embedding of CNT–GNP hybrids into pristine epoxy endows optimum dispersion of CNTs and GNPs as well as better interfacial adhesion between the carbon fillers and matrix, which results in a significant improvement in load transfer effectiveness and the resulting the tensile strength was enhanced by significantly with respect to the neat epoxy. The chemical hybrid filler of CNT–Al_2O_3 produced via CVD performs better than the physically mixed hybrid filler of CNT– Al_2O_3 in terms of tensile and thermal properties for given filler loading. The increase in tensile performance of the chemical hybrid CNT– Al_2O_3 is associated with improved stress transfer between filler and matrix with the presence of the CNTs on the surface of Al_2O_3 particle [11]. Khashaba [12] described that the well dispersion of hard alumina-nanoparticles in epoxy resin played a key role in improving the alumina/matrix interfacial bond strength. Therefore, the applied stress was effectively transferred to the particles from the matrix and accordingly, the flexural strength, modulus, and fracture toughness of alumina nanocomposites are enhanced considerably. Lee et al [13] concluded that the improved tensile properties of the silanized carbon/CNT/epoxy three-phase composite were due to the increased dispersibility and interfacial interactions between the silane-functionalized CNTs and epoxy in the carbon/epoxy composite. The well dispersed alumina-nanoparticles in epoxy resin significantly improved the mechanical properties of alumina-nanocomposites. Tian and He [14] concluded that Fracture surface showed homogenous dispersion of nanotubes and Al_2O_3 in the HDPE matrix and presence of interlocking like phenomena between hybrid and HDPE matrix, which might contribute to the effective reinforcement of the HDPE composites. Zhang and Jiang [15] reported that chemically functionalized MWCNTs, chemically interconnected MWCNTs improved the fracture strain and therefore the toughness of the composites significantly improved. Mishnaevky Jr and Dai [16] concluded that Glass/carbon fibers hybrid UD composites clearly demonstrate higher stiffness and lower weight with increasing the carbon content; however, they can show lower strength and elongation to failure as compared with usual glass fiber polymer composites. The strength (critical stress) of hybrid composites can be lower than that for both pure glass and pure carbon composites, especially under uniform displacement loading. The critical elongation of the hybrid composites decreases with increasing fraction of carbon fibers in the hybrid.

Bearing in mind the above discussion of existing techniques and various new findings, the present study aims to develop and observe the influence of MWCNTs– Al_2O_3 addition on the mechanical properties such as tensile strength, micro hardness and impact energy of traditional epoxy reinforced composites. As a major ceramic material commonly used for structural applications due to its high specific stiffness, Al_2O_3 was selected as the binder for CNTs. MWCNTs– Al_2O_3 hybrids comprised of well-aligned MWCNTs were synthesized by chemical vapour deposition process (CVD). Hybriding Al_2O_3 with MWCNTs may be helpful with the dispersion of CNTs in the host matrix.

2. Materials and methods

2.1Materials

The MWCNTs used for the sample preparation was obtained from bottom up technologies, India with the specification of length 15µm, number of walls 7-8,specific area of 180-200 m^2 g^{-1} and an average outer diameter of about 20-30 nm .The alumina nano filler used for this experimentation was obtained with the specifications of specific area of 350-420 m^2 g^{-1} ,melting point 2100 ^{0} C, gamma phase and particle size of < 100 nm. Diglycidyl Ether of Bisphenol A epoxy resin was used as a matrix that is available commercially. Chemical vapour deposition (CVD) was used to synthesize the carbon nanotubes and functionalized with COOH to enhance the wall surface.

2.2 Preparation of MWCNTs – Al_2O_3 hybrid powder

By using sonication process the required amount of MWCNTs were dispersed in sodium dodecyl sulphate containing acetone solution. This sonication process continued for 1.5 hours by using power of about 450-500W and frequency range of 10-150 KHz. Later the alumina nano powder was added after and the processes continued for another 30 minutes. By using ball milling machine the liquid suspension was milled for 12 hours at 200 rpm. The mixture was dried at 100^0 C for 8 hours in an oven for removal of dispersion. The dried mixture was spread in a vacuum chamber plate and drying continued at the same temperature for another 24 hours. The MWCNTs - Al_2O_3 was mixed in a weight ratio of 1:4.

2.3 Preparation of MWCNTs – Al_2O_3 epoxy composites

MWCNTs – Al_2O_3 hybrid powder were dispersed with Diglycidyl Ether of Bisphenol A epoxy resin in the ratio of 0.5, 1.0, 1.5, 2.0 and 2.5wt% by using ultrasonic mixture at frequency of 25 KHZ for 30 min. The suspension was maintained at a temperature between 50^0 C to 60^0 C. The obtained mixture was kept in vacuum chamber for 30 min to remove the air bubbles. Araldite HY951 was added in the volume ratio of 10:1 as a curing agent. The mixed suspension was transferred into the acrylic dog shaped plastic mould which was made by using laser cutting machine with the length of 165 mm,width 19mm,gauge length 57.15 mm and thickness of about 7mm respectively. The mould was kept in an oven at 100^0 C for 12 hours for curing. Knurling was done on both ends of the sample for purpose of gripping.

3. Tensile strength

The schematic diagram of dog bone shape tensile specimen is shown in fig.1. The flat type specimen was generally used for performing tensile strength. The specimens with both ends knurled are commonly used for determining tensile strength. ASTM D 638 standard test method is employed for hybrid composites for tensile test. The dimensions of the sample were 165 x 19 x 7 and gauge radius was 76 mm (Fig.2). The tensile test was performed in the universal testing machine at a cross head speed of 10 mm/min. six number of specimens with same composition was used to get the average value of the tensile strength.

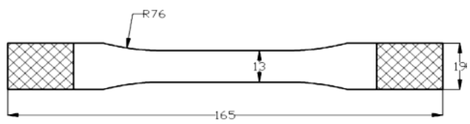

Fig.1: Schematic diagram of dog bone shape tensile specimen (ASTM D 638)

Fig.2: Dog bone shape pure epoxy and MWCNTs/alumina hybrid nanocomposites specimen after polishing

4. Micro hardness

The measurement of micro hardness was carried out using HM113 Vickers hardness tester -mitutoyo. The right angle pyramid with a square base diamond indentor angle of 136^0 between opposites faces compressed the composite specimen under a load of F=20N for a loading time of 15 seconds. Six number of indentation was made on each sample to get the mean value of the hardness. Vickers hardness number was calculated by the following equation $H_v = 0.1889F/L^2$.Diagonal of the square impression *(L) =X+Y/2*. Where X and Yare horizontal and vertical length in mm.

5. Impact strength

Impact strength are done as per ASTM D256 using a low density instrumented impact tester. The standard dimension of the specimen is 64 x 12.7 x 4.5 mm^3 and the notch depth is 10.2 mm. the notch impact strength is measured by pendulum impact testing machine by striking the v-notch specimen with a pendulum hammer. The specimen is clamped in a square support and is struck at their central point by a hemispherical bolt with diameter of 5 mm. Different specimen values of the impact energy are recorded from the dial indicator. The mean value of the impact strength was obtained as an average value of six tests concluded.

6. Results and discussion

6.1 Tensile strength

The tensile strength of epoxy filled with MWCNTs-Al_20_3 hybrid nanocomposites as shown in Fig.3. Compared with the pure epoxy matrix the tensile strength of the hybrid nanocomposites significantly is found to increase by the addition of MWCNTs-Al_20_3.From this experimental investigation it is found that there is a gradual drop in tensile strength with further increase in hybrid filler content. The pure epoxy has the strength of 90MPa in tension and this value decreased to 475 MPa and 101 MPa with the MWCNTs-Al_20_3 addition of 2.0wt% and 2.5wt%, respectively. This value was found to increase to 102 MPa, 137 MPa and 518 MPa with MWCNTs- Al_20_3 hybrid nanofillers addition of 0.5 wt%, 1.0wt% and 1.5wt%, respectively. Furthermore addition of hybrid nanofillers in the pure epoxy decreased the value of tension to 475 MPa and 101 MPa of 2.0wt% and 2.5wt%, respectively. It is believed that excessive addition of hybrid nanofillers adversely affect the homogeneous dispersion in the pure epoxy matrix. It is found that the excessive content of hybrid nanofillers form scattered bundles in the casted specimen. Based on the investigation and wide variation in the tensile results of pure epoxy and hybrid nanofillers for various compositions it is proved that epoxy with 1.5wt% of MWCNTs-Al_20_3 hybrid nanocomposites showed considerable predominant tensile strength. The morphology of the fractured surface of the casted specimen was analyzed by Field Emission Scanning Electron Microscope (FESEM). The fractured surface of pure epoxy is shown in fig. 4(a) it can be seen that the smooth irregular surface with small pinch hole was exhibited in pure epoxy relatively poor toughness and ductile in nature. However the hybrid nanocomposites show rougher surface morphology with small crack and massive plastic deformation Fig.4 (b), indicating that a reinforcement behavior of MWCNTs/alumina hybrid composites. Pure epoxy with 1.5wt. % of hybrid nanofillers shows a more rough morphology, which indicates its relatively higher tensile properties. Further investigation of morphology with increased addition of hybrid nanofillers indicate the presence of larger smooth surface with bundles as shown in fig. 4(c).

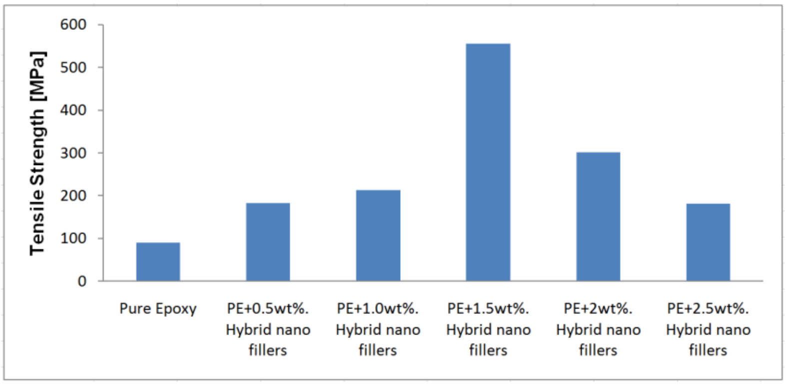

Fig.3: Tensile strength of pure epoxy and hybrid nanofillers

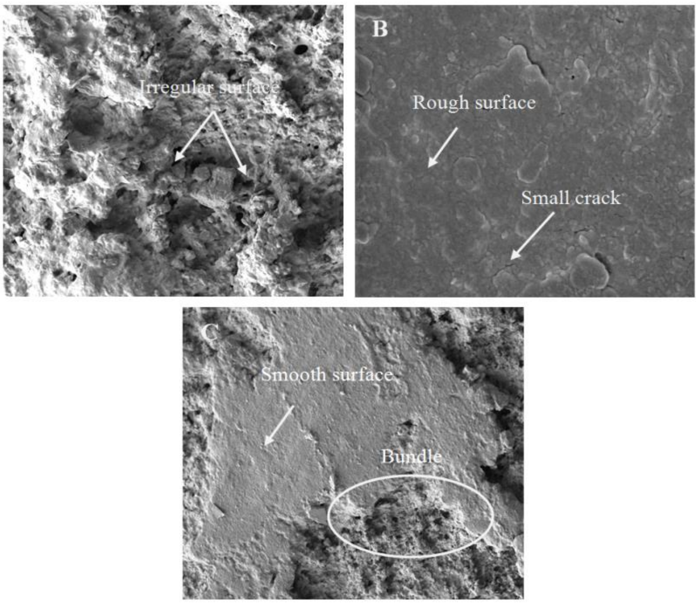

Fig.4.SEM images of tensile fracture surface of (a) pure epoxy (b) 1.5wt. % of MWCNTs/alumina (c) 2.5wt. % of MWCNTs/alumina nanocomposites

6.2 Micro-hardness

The experimental measured values of pure epoxy 0.5, 1.0, 1.5, 2.0 and 2.5wt%, respectively of hybrid nanocomposites are presented in the fig.5. It is found that the hardness is affected significantly with the addition of hybrid nanofillers. It is observed that micro hardness evaluation is considered to be one of the important factors in composites for determination of mechanical characteristics and tribological characteristics with the increase in MWCNTs-Al_20_3 nanofillers content from 0.5 to 1.5 wt% .The hardness is found to be increases from 75 H_v to 156 H_v .This implies an increment of 16 % in the hardness of the pure epoxy. The improvement in hardness in the hybrid nanocomposites can be explained as follows. The process of hardness tests a compressive force in the action. So the matrix face and hybrid nanofillers face would be compressed together and touch each other more tight. Thus the homogeneous interface can transmit pressure more effectively. This results in enhancement of hardness and similarly further addition of hybrid nanofillers to form a scattered bundle .Due to this reason the compressive pressure is not effectively transferred to interfacial bond resulting in local failures on the surface and crack initiative.

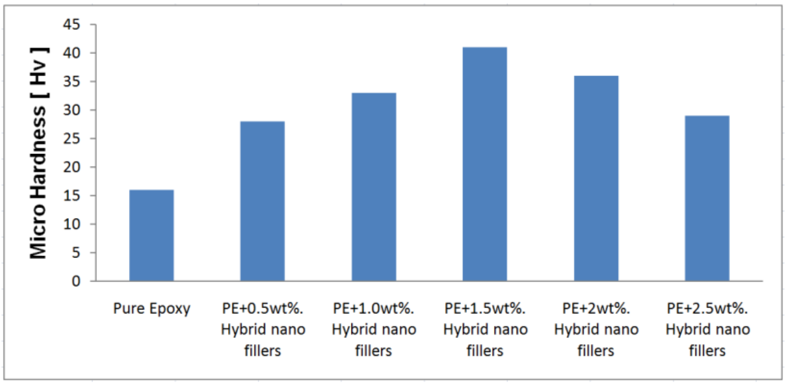

Fig.5: Micro-hardness of pure epoxy and MWCNTs/alumina nanofillers

6.3 Impact strength

The impact energy values of pure epoxy and MWCNTs-Al_20_3 hybrid nanofillers during the impact test are recorded and presented in the (fig.6). Its result shows that the impact resistance to loading of hybrid nanocomposites increases with considerable addition of hybrid nanofillers. It is seen that nanocomposites increases by 56 %, 65 % and 80 % with the addition of 0.5, 1.0 and 1.5wt%, respectively when compared with the pure epoxy. A literature review revealed that the mobility of polymer chain is restricted by the filler content which reduces their deformability to make the material brittle in nature .Therefore the capability of energy absorbing capacity of the hybrid composites reduces with addition of hybrid fillers. However the homogeneous dispersion of hybrid nanofillers (0.5.1.0 and 1.5wt %) in the host matrix it is noticed which indicates a significant improvement of impact strength. The SEM images reveal that, the pure epoxy deformation is brittle in nature (7a), However the sample exhibits (7 (b-d)) layered, soft and dense peeled off surface. With addition of 0.5wt% hybrid nanofillers react with polymer chain and absorb the energy and restrict the deformation. The force exceeds the restriction limit, the maximum energy absorbed by the hybrid filler tends to deform and form a layer over the surface and tear off. The addition of 1.0wt% and 1.5wt% hybrid nanofillers forms a soft and hard layer to absorb the impact energy and tends to maintain the material in brittle in nature. Similarly further addition of 2.0 and 2.5 wt %. Hybrid nanofillers form a crimped and hard peeled (7 (e-f)) off surface due to non homogeneous distribution of hybrid nanofillers. Therefore further improvement is to be decided judiciously before any specific application keeping strength in mind.

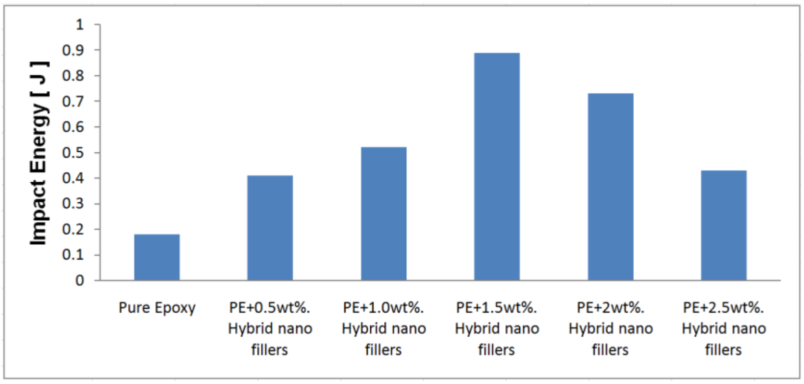

Fig.6: Impact energy value of pure epoxy and MWCNTs/alumina nanofillers

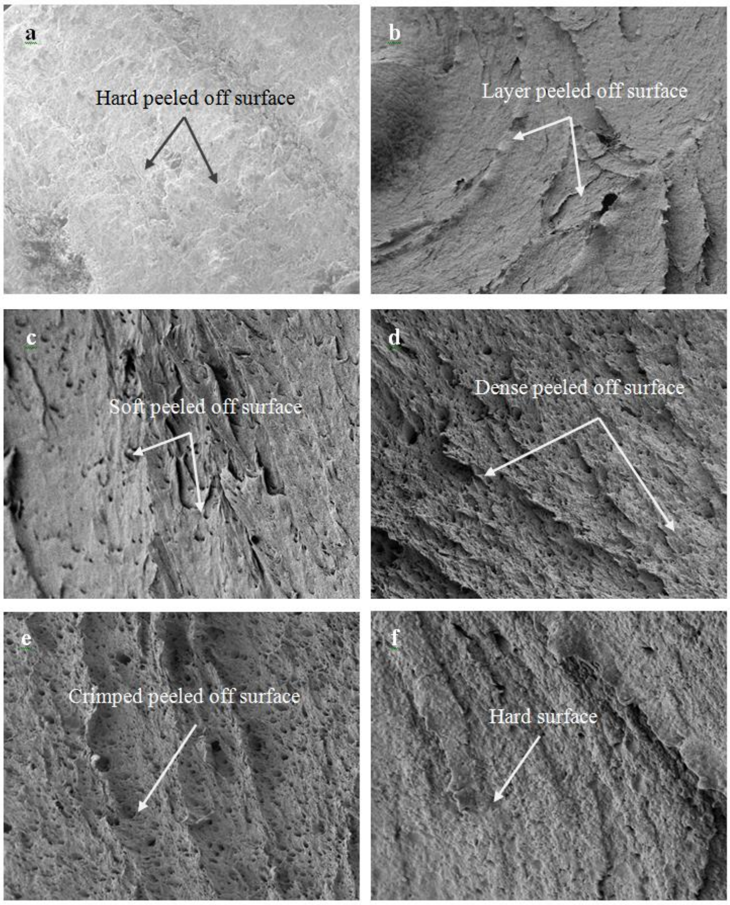

Fig.7 (a-f):SEM images of impact fractured surface of (a) pure epoxy (b) 0.5wt.% (c) 1.0wt.% (d) 1.5wt.% (e) 2.0wt.% and (f) 2.5wt.% of MWCNTs/alumina hybrid nanocomposites.

7. Conclusion

In this study, investigation was made to study the effect of adding MWCNTs-Al_20_3 hybrid nanocomposites on the tensile strength, microhardness and impact strength of epoxy 0.5, 1.0, 1.5, 2.0 and 2.5wt% respectively. The composite specimens were fabricated by casted moulding method. Based on the experimental results, the conclusions are summarized as follows.

1. The tensile strength, hardness and impact energy of these new class epoxy / MWCNTs-Al_20_3 hybrid nanocomposites are improving with considerable addition of hybrid nanofillers.

2. Epoxy with 1.5 wt% of MWCNTs-Al_20_3 hybrid nanofillers significantly gives good result compared with pure epoxy. 0.5, 1.0, 1.5 wt% of MWCNTs-Al_20_3 hybrid nanocomposites exhibited considerable improvement in tensile strength, hardness and impact energy. However 1.5wt% hybrid nano composites significantly exhibit good result than pure epoxy.

3. Furthermore addition of 2.0% and 2.5 wt% of MWCNTs-Al_20_3 hybrid nanocomposites do not exhibit any considerable improvement in the mechanical properties of the test specimen.

4. The excessive addition of hybrid nanofillers forms a localized bundle and are scattered resulting in poor performance than the pure epoxy. The enhancements in the mechanical properties will depend on the homogeneous dispersion of the host matrix.

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