Characterization of Biaxial Carbon Braided Nanocomposites

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Abstract—**Biaxial carbon braided nanocomposites were fabricated using biaxial braided carbon sleeves and epoxy resin mixed with three different nanoparticles such as Titanium (IV) oxide, nano clay and carbon nanofiber. Magnetic hot plate stirring method was used to mix nanoparticles with epoxy resin. The braided nanocomposites were manufactured through hand-layup technique. In this paper, the effects of biaxial braided nanocomposites with neat epoxy, Titanium (IV) oxide (1, 3 and 5 wt %), nanoclay (1, 3 and 5 wt %) and carbon nanofiber (0.2, 0.5 and 1 wt %) as reinforcing agent were considered to study the mechanical properties. A small amount of carbon nanofiber (0.5wt%) added into the epoxy of braided nanocomposites can enhance the tensile strength by 62%, flexural strength by 79% and double shear strength by 60% than neat epoxy biaxial carbon braided nanocomposites. Morphologies of the biaxial braided nanocomposites were analyzed by Scanning Electron Microscopy. Microscopy study revealed better dispersion of 3wt% of Titanium (IV) oxide, 3wt% of Nano clay and 0.5wt% of carbon nanofiber in biaxial braided nanocomposites.**

Keywords: Biaxial braided fiber, Mechanical properties, Nanoparticles, SEM image,

I. INTRODUCTION

Now a day, woven, knitted and braided fabrics for an advanced composite has gained extensive deliberation in light weight materials for various applications such as aeronautical, marine, sports and automobile industries. Among them, braided fibers can give continuous arrangement of fibers in different directions, tolerate axial loads. Thus, the braided fiber has been predicted to be good preforms for the reinforcement of composite materials.

In recent years, many researchers have concentrated in developing a new material with braided fibers. Yan et al. [1] investigated the transverse impact behavior for four-step 3D carbon/epoxy braided composites in experimental and finite element analysis. It was observed that the load-displacement curve, energy absorption and impact damage had compared between finite element simulation and experimental. They found good agreements during the comparison. Microscopic damage development is decreased in the fatigue lives at elevated temperatures. And also they yielded an improved perceptive of microscopic damage mechanisms and local deformation behavior for an advanced composite material, which is valuable for designers. Benjamin et al. [2] manufactured the braided and woven textile composite beams and determined their mechanical properties (tensile and flexural). It was observed that the woven performs exhibited linear stress-strain relationship while braided performs showed nonlinear stress-strain relation. It was shown that the flexural tangent moduli of the braided composites were greater than the corresponding tensile moduli.

Epoxies are used as matrices for FRP composites. The epoxy yielded many properties, such as high strength and high elastic modulus. But, most epoxies are brittle. This drawback has limited its applications. One of the effective techniques is to improve the epoxy matrix ductility by adding fillers, such as TiO_2, nano clay and carbon nanofiber. Sajjad et al. [3] studied the mechanical properties of ternary nanocomposites (polypropylene/linear low-density polyethylene/ nano-titanium dioxide. It is showed that adding a small amount of TiO_2 nanoparticles (up to 2 wt %) improved the mechanical properties of nano-composites. Abdolhossein et al. [4] investigated the mechanical properties of hybrid graphene/TiO_2 nanocomposites and hybrid hydroxylated graphene/TiO_2 nanocomposites. It has been proved that Young's modulus and shear modulus of TiO_2 increase about 14% and 12% as a result of interaction with graphene layer. Tijana et al. [5] investigated pure epoxy and epoxy/TiO_2 nanocomposites. It is observed that glass transition temperature and anticorrosive properties were improved by adding TiO_2 with epoxy resin. Vu et al. [6] studied the effect of titanium dioxide on the properties of polyethylene/TiO_2 nanocomposites. They obtained better mechanical properties such as tensile properties and dynamic storage modulus for polyethylene/ TiO_2 nanocomposites. Parimala and Jabaraj [7, 8] studied the mechanical properties of carbon braided biaxial fiber composite with TiO_2. It was shown that 3wt% of TiO_2 composites showed better mechanical properties when compared to 1wt% and 5wt% of TiO_2.

In recent researchers, nano clay has been used effectively as reinforcements for nanocomposites, providing outstanding mechanical properties. Mohammed et al. [9] studied mechanical and thermal properties of carbon fiber/polypropylene composite filled with nano-clay. Their results revealed that at filler content 3% of nano clay, initiation and propagation interlaminar fracture toughness in Mode I was improved significantly by 64% and 67% respectively. Md Ekramul et al. [10] studied the characterization of carbon fiber reinforced epoxy composites modified with Nano clay and Carbon nanotubes. It has been shown that nano clay composites showed improvement in flexural strength, modulus and absorbed a higher amount of energy in low impact test. Vahdat et al. [11] studied the effects of processing parameters such as mixing system, curing system and impact behavior of epoxy nanocomposites reinforced with halloysite nanocomposites. Their study showed that the properties of epoxy/HNTs nanocomposites can be controlled through mixing method, compound formulation, and HNTs type, which makes the materials selection and manufacturing processes important for achieving the most optimum structure–property relationship and improving the overall properties of the epoxy nanocomposites. Qi et al. [12] investigated the mechanical properties of DGEBA-based epoxy resin with nano clay additives. They observed that the addition of nano clay increased the elastic modulus and fracture toughness of epoxy resin; it also reduced the failure strength and failure strain with increasing nano clay level.

Researchers used a different type of approaches to improve the performance of epoxy resins including, the addition of TiO_2, nano clay, rubber agents, diluents etc. Recently, Carbon nanofiber fillers have drawn increasing interest as it is possible to improve the mechanical properties of the epoxy resin. Jin et al. [13] studied the effect of carbon nanofibers on self-healing epoxy/poly blends. They significantly enhanced mechanical performance by the low content of CNFs enables the development of epoxies and advanced polymer composites with longer service life and less maintenance. Sohel et al. [14] was dispersed CNFs in the matrix of carbon fabric reinforced epoxy composites in order to develop carbon/epoxy/CNF three-phase composites. They proved that dispersion of 0.5wt%CNF in the matrix using the optimized dispersion route resulted in improvement in the mechanical, thermal and electrical properties of three phase composites. Smrutisikha [15] studied about epoxy nanocomposites of different content of carbon nanofibers up to 1wt% under room temperature and refrigerated curing conditions. It has been shown that the addition of a very low amount of CNFs brought improvement in mechanical and electrical properties. Parimala and Jabaraj [16] were shown that the mechanical properties were improved by adding 0.5wt% carbon nanofiber particles in biaxially carbon braided fiber/epoxy composites. Therefore, TiO_2, nano clay (NC) and carbon nanofibers (CNF) are the potential candidates for mixing in epoxy resin.

The objective of this paper is to fabricate the biaxially carbon braided fiber nanocomposites using biaxial braided carbon sleeves and epoxy resin mixed with three different nanoparticles such as Titanium (IV) oxide (TiO_2), nano clay (NC) and carbon nanofiber (CNF). The effects of biaxial braided nanocomposites with neat epoxy, TiO_2 (1, 3 and 5 wt %), NC (1, 3 and 5 wt %) and CNF (0.2, 0.5 and 1 wt %) as reinforcing agent were considered to study the mechanical properties. The mechanical behaviors such as tensile, flexural and shear were studied and compared for the biaxially carbon braided fiber nanocomposites. Scanning Electron Microscopy (SEM image) is employed to track agglomerations and damage development.

II. MATERIALS AND METHODS

A. Materials

The fiber used was high firmness biaxially carbon braided fiber sleeves with 3k tow manufactured by ACP composites. The fiber orientation, diameter, yard, longitudinal modulus and shear modulus are 45^o, 4", 15.1 oz/square yard, 17GPa and 33GPa respectively. The epoxy resin used was LY 556 Bisphenol-A-Diglycidyl-Ether (DGEBA), which is insoluble in water and has a flash point of over 200^oC. The curing agent used was HY951, which has a density of 0.97 to 0.98 g/cm^3 and the flash point of 110^oC. The epoxy resin and curing agent were mixed in the ratio of 10:1. TiO_2 (Titanium (IV) oxide, <25nm particle size, anatase nanopowder, 99.7% trace metal basis), Montmorillonite nano clay (NC, Nanomer 1.28E**)** and carbon nanofiber (pyrolytically stripped platelets ((conical). >98% carbon basis, D x L 100nm x 20-200 µm) nanoparticles from Sigma-Aldrich were used separately as filler materials to modify the epoxy resin matrix.

B. Dispersion methods of filler materials

The epoxy resin was heated (75^oc) to lower the viscosity. For the preparation of neat epoxy sample (0 wt%), the hardener HY 951 was added to the heated epoxy resin and mixed manually for 10 sec to avoid the formation of bubbles. For the preparation of TiO_2 samples (1wt%, 3wt% and 5wt%), the magnetic stirrer is used to mix TiO_2 with epoxy resin for 4h at 50^oC. The hardener HY 951 was then added to the mixture and mixed manually for 10sec to avoid the formation of bubbles. For the preparation of nano clay samples (1wt%, 3wt% and 5wt%), the nano clay was heated up to 100^oC for 1 h in order to remove moisture. The above mixing method for TiO_2 was followed by mixing heated nano clay with epoxy resin and hardener [17]. For the preparation of carbon nanofiber samples (0.2wt%, 0.5wt% and 1wt %), the magnetic stirrer is used to mix carbon nanofiber with heated epoxy resin for 4h at 75^oC. The mixture was subjected to sonication using an ultrasonicator at an ultrahigh frequency for 3h to further disperse the CNF while maintaining the resin temperature at 75^o C using a hot water bath. The mixture was followed by addition of HY951 and the mixture was stirred well to avoid the formation of bubbles [18].

C. Fabrication of laminates

The fabrication was done using hand layup technique to manufacture biaxial carbon braided nanocomposites as shown in Fig. 1. Biaxial carbon braided fiber sleeves were impregnated with the mixed nanoparticles epoxy resin system using brush and roller. It is followed by a press machine for 24 h at room temperature. The composite samples were put in an oven with a post cure treatment for 1h at 100^{o}C. 8 layers of biaxial fiber were used to get a 3mm thickness of the composite material. Biaxial braided nanocomposites prepared using different nanoparticles are listed in Table 1. Three samples for each test specimen with ASTM standard dimensions were cut from the composites panels, using an abrasive water jet cutting technique. The fiber volume fraction is 45±0.6%.

Fig. 1. Fabrication of Biaxially carbon braided nanocomposites

Table I. Biaxially carbon braided nanocomposites and their sample codes

Sample Codes	Nanoparticles	wt%	Descriptions
NE	-	0	Biaxially carbon braided fiber sleeve/ epoxy composite
1wt%TiO_2	Titanium (IV) Oxide	1	Biaxially carbon braided fiber sleeve/1wt% TiO_2 /epoxy composite
3wt%TiO_2	Titanium (IV) Oxide	3	Biaxially carbon braided fiber sleeve/3wt% TiO_2 /epoxy composite
5wt%TiO_2	Titanium (IV) Oxide	5	Biaxially carbon braided fiber sleeve/1wt% TiO_2 /epoxy composite
1wt%NC	Nano clay	1	Biaxially carbon braided fiber sleeve/1wt%NC /epoxy composite
3wt%NC	Nano clay	3	Biaxially carbon braided fiber sleeve/3wt%NC /epoxy composite
5wt%NC	Nano clay	5	Biaxially carbon braided fiber sleeve/5wt%NC /epoxy composite
0.2wt%CNF	Carbon nano fiber	0.2	Biaxially carbon braided fibers sleeve/0.2wt%CNF/epoxy composite
0.5wt%CNF	Carbon nano fiber	0.5	Biaxially carbon braided fibers sleeve/0.5wt%CNF/epoxy composite
1wt%CNF	Carbon nano fiber	1	Biaxially carbon braided fibers sleeve/1wt%CNF/epoxy composite

III. MECHANICAL TESTS

A. Tensile test

Dog bone shaped samples of 165mm x 12.7mmx 3mm (length x width x thickness) unfilled (0wt%), TiO_2 (1wt%, 3wt% & 5wt%), nano clay (1wt%, 3wt% & 5wt%) and carbon nanofiber (0.2wt%, 0.5wt% & 1wt%) biaxially braided nanocomposite plates were characterized through tensile test in accordance with ASTM D638 standards. The samples with gauge length 57mm and radius 25mm were tested using a Universal Testing Machine, model744, with hydraulic grip and MTS 632 12C-20 extensometer, at the rate of 5.00000mm/min, at room temperature of 23^{o}C.

B. Flexural test

Flexural strength and flexural modulus were determined by 3-point bend test. The samples of 127mm x 12.7mm x 3mm (length x width x thickness) were tested according to the ASTM D760 standards.

C. Double shear test

Shear strength is the utmost load applied normal to a fastener's axis that can be supported previous to break. Double shear is the instantaneous shear across two usually parallel planes and cuts into three pieces. The composite samples of 45x 10x 3 mm were tested according to the ASTM D 5961M for fiber resin composites.

D. Scanning electron microscopy (SEM)

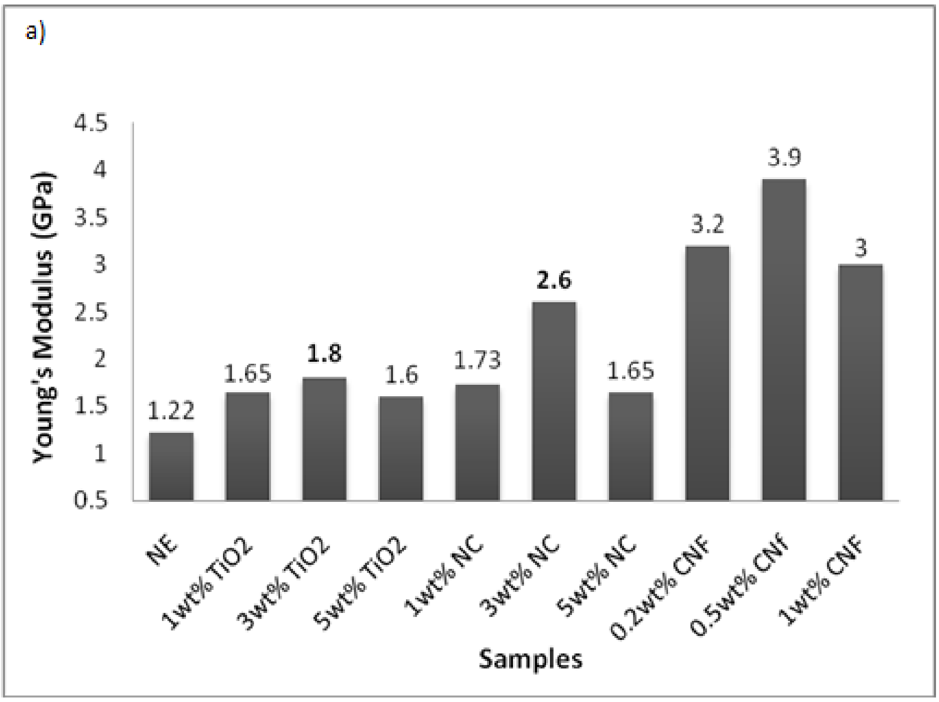
The tensile test coupons, 3-point bending test coupons, shear test coupons and impact test coupons were examined with a voltage of 10kV in secondary electron mode using a Phenom world pro scanning electron microscope.

IV. RESULTS AND DISCUSSIONS

A. Tensile Properties

The average value of three samples of each specimen's Young's modulus and tensile strength plot of biaxial braided nanocomposites were compared by using histograms as presented in Fig. 2a and b. NE has the Young's modulus of 1.22 GPa and tensile strength of 0.1 GPa. It can be seen from the Young's modulus and tensile strength plot that all biaxial braided nanocomposites have greater Young's modulus and tensile strength than neat epoxy (NE). It has been noted that the samples namely, 3wt%TiO_2, 3wt%NC and 0.5wt%CNF shows the greatest tensile strength of 0.22 GPa, 0.24 GPa and 0.26 GPa respectively in comparison with remaining samples. But the the Young's modulus and tensile strength were shown greater improvement in the addition of CNF nanoparticles when compared to nano clay and TiO_2. This is due to high modulus and strength of CNF than nano clay and TiO_2 nanoparticles. The highest Young's modulus and tensile strength are achieved when the CNF particle loading is 0.5wt%, which is about 68.8% and 62% the NE. Further addition of CNF (1wt %) decreases the Young's modulus and tensile strength. However, the Young's modulus of the composites can be improved slightly when the TiO_2 nanoparticles are added. When TiO_2 nanoparticles vary from 1 to 5 wt% the highest Young's modulus and tensile strength value are only 32.3% and 54.6% the NE. When nano clay nanoparticle varies from 1 to 5 wt% the highest Young's modulus and tensile strength value are 53.1% and 58.4% the NE.

The Young's modulus and tensile strength of the composites were decreased by the addition of the higher percentage of nanoparticles such as 5wt% TiO_2, 5wt% NC and 1wt%CNF. The reason seems that higher percentage of nanoparticles were not enough mixed with epoxy resin and forms agglomeration. Due to the above reason, loads were not successfully transferred to the composite material during loading and each ply was fractured individually. The fracture surface of the tensile test specimens indicates the brittle fracture mechanism of neat epoxy resin**.** Another composite specimen indicates ductile fracture mechanism and improved tensile properties up to 3wt% TiO_2, 3wt% NC and 0.5wt%CNF. The reduction in strength and modulus is linked with a poor dispersion and void formation of the high content level of nanoparticles in the resin.



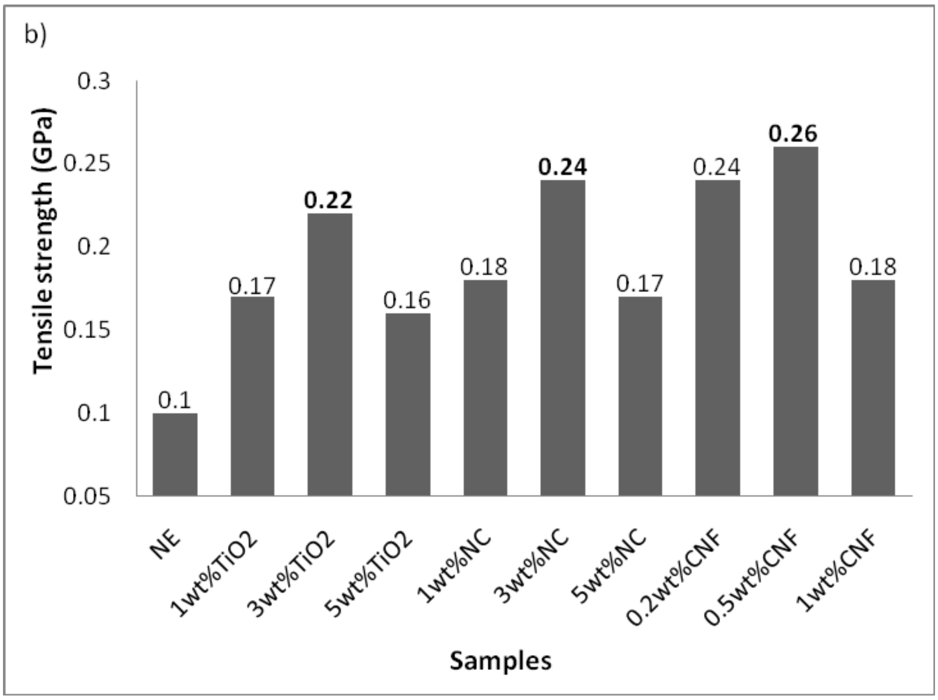


Fig. 2. Tensile properties of biaxially carbon braided fiber nanocomposites: a) Young's Modulus and b) Tensile Strength

The fracture surfaces of the tensile failed specimens were studied by SEM and shown in Fig. 3**.** Fiber fracture, interface debonding and delamination can be experiential in this SEM image. The micrograph of neat epoxy in Fig. 3a indicated a typical feature of brittle fracture behavior, thus secretarial for the low fracture toughness of the neat epoxy. Generally, a large amount of rough surface was seen upon adding nanoparticles into the composites. The increased surface roughness indicated that the path of the crack is vague because of the nanoparticles, creating crack propagation more complicated with lots of ridges and wavy lines. This type of rough crack morphology indicated ductile fracture mechanism. Fig. 3b, 3c, 3e, 3f, 3h and 3i also clearly represented that in 1wt%TiO_2, 3wt%TiO_2, 1wt%NC, 3wt%NC, 0.2wt%CNF and 0.5wt%CNF were well spread and uniformly surrounded in the composite materials. When the nanoparticles content increased to 5wt%TiO_2, 5wt%NC and 1wt%CNF, the failure of composite materials were initiated at a large particle that appeared to be an agglomeration of several TiO_2, NC and CNF (Fig. 3d, 3g and 3j)**.** Due to agglomeration, the fracture initiations were caused by the stress concentration.

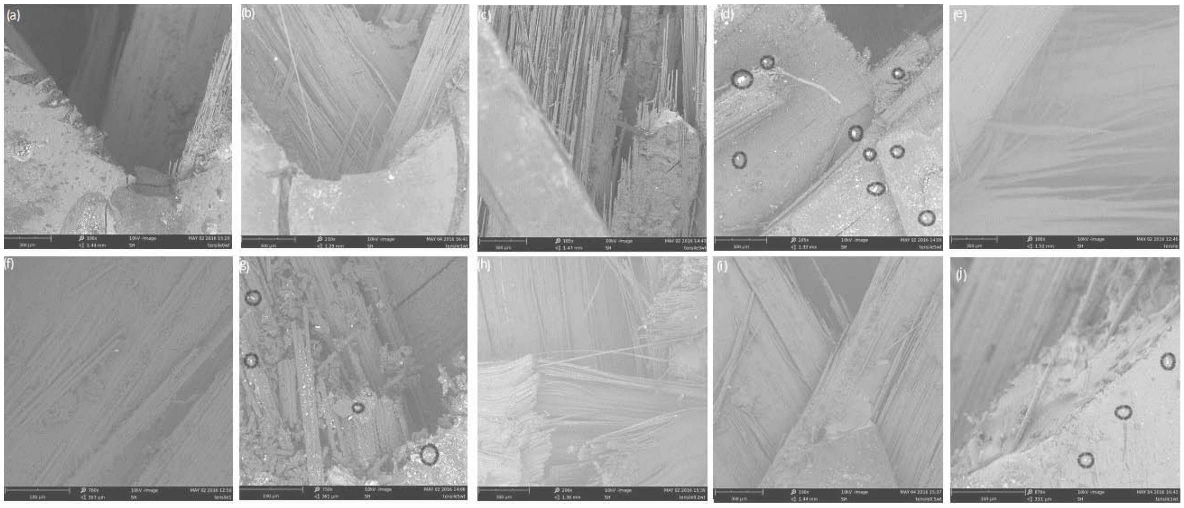


Fig. 3. SEM Micrographs of Tensile broken Specimens of biaxially carbon braided fiber nanocomposites: (a) NE; (b)1wt%TiO_2; (c) 3wt% TiO_2; (d) 5wt%TiO_2; (e) 1wt%NC; (f) 3wt%NC; (g) 5wt%NC; (h) 0.2wt%CNF; (i) 0.5wt%CNF; (j) 1wt%CNF

B. Flexural Properties

The average value of three samples of each specimen's flexural modulus and strength as a function of biaxially carbon braided composite were displayed in Fig. 4a and 4b. The flexural modulus and flexural strength of the nanocomposites increased with less percentage of nanoparticles and reach the maximum value at 3wt%TiO_2, 3wt%NC and 0.5wt%CNF and then decreased. The flexural strength of o.5wt%CNF composite showed a maximum of 0.76 GPa which was 16% and 68% higher than that of 3wt%NC and 3wt%TiO_2 respectively. This is due to the high flexibility of carbon braided fiber and high strength of carbon nanofibers in the composites improved the stress level at fracture. The biaxial carbon braided composites did not fail entirely and had high deflection against the flexural load. It could be also noted that though an improved epoxy resin matrix has increased the flexural properties of the biaxial carbon braided composites, the architecture of braid and its braid angle also had a significant effect on the composite's strength and modulus [17]. The observed lower flexural properties at 5wt%TiO_2, 5wt%NC and 1wt%CNF were probably due to fiber contacts as uniform nanoparticles distribution was not easy at higher nanoparticles loading during the preparation of composite materials.

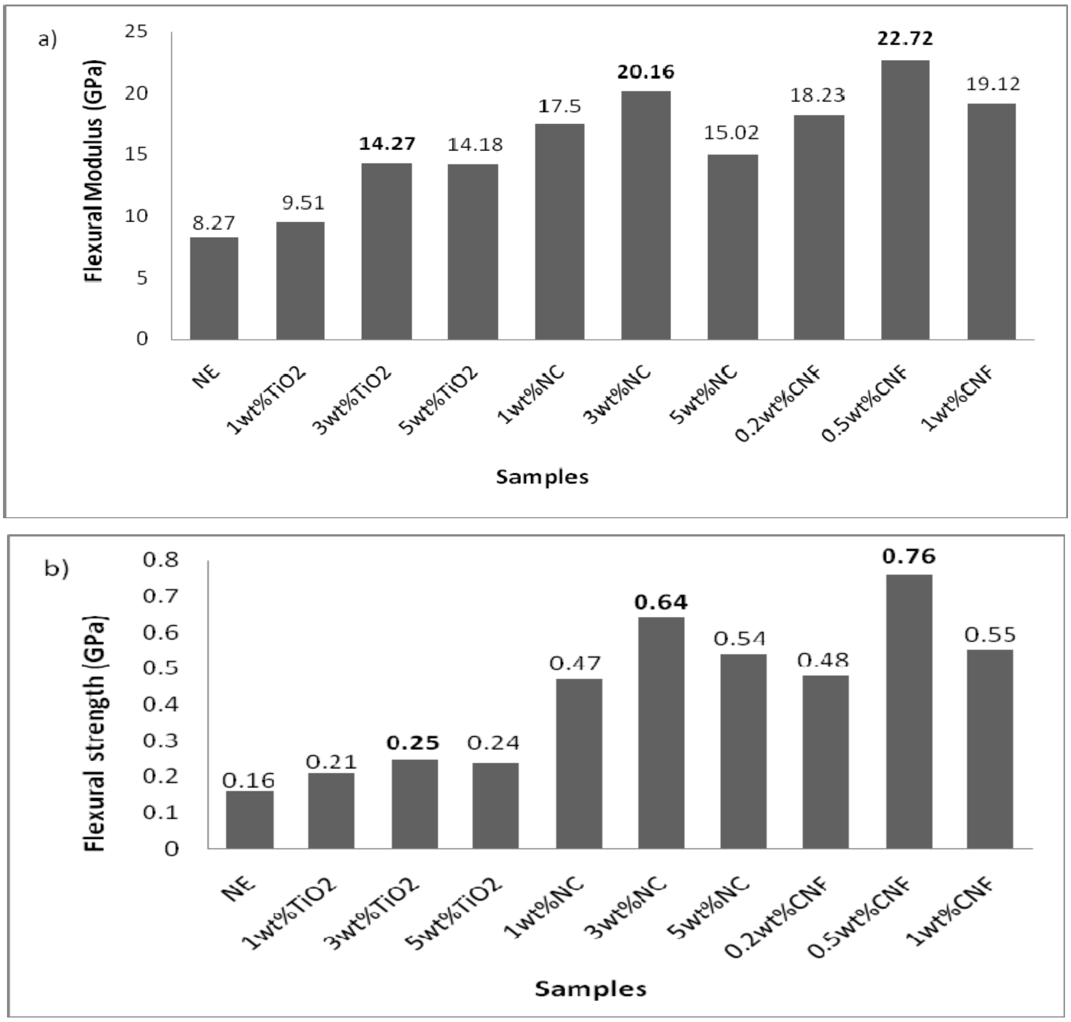


Fig. 4. Flexural properties of biaxially carbon braided fiber nanocomposites: a) Flexural Modulus and b) Flexural Strength

Scanning Electron Microscopy (SEM Image) Studies were carried out on the specimens subjected to flexural testing Fig. 5 showed the different wt% of nanoparticles loading samples of biaxial braided carbon fiber composites. Due to the high flexibility of biaxial carbon braided sleeves, most of the specimens had not failed. It can be seen the NE sample did not exhibit good bonding between the epoxy resins and biaxial carbon braided fiber. Tow fracture followed by delamination was clear from the Fig. 5a. The 3wt%TiO_2, 3wt%NC and 0.5wt%CNF showed that break propagated from loading point to tensile side and also exhibit good bonding strength between plies, which is observed in Fig. 5c, 5f and 5i. Delamination failure and broken tows were observed in Fig. 5b, 5e and 5h. For the specimen 5wt%TiO_2, 5wt% NC and 1wt%CNF braid tow was broken on the compression side, which is shown in Fig. 5d, 5g and 5j.

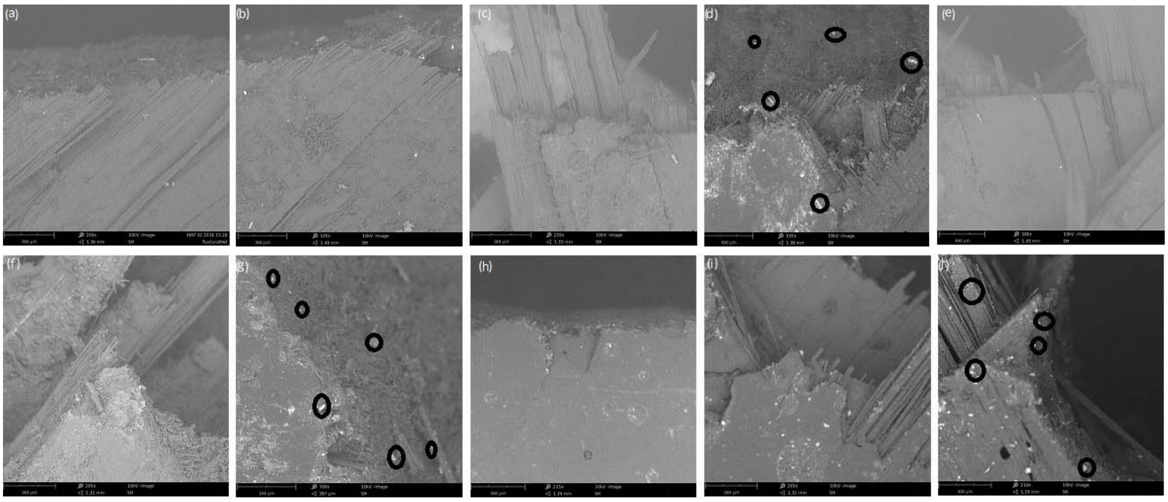
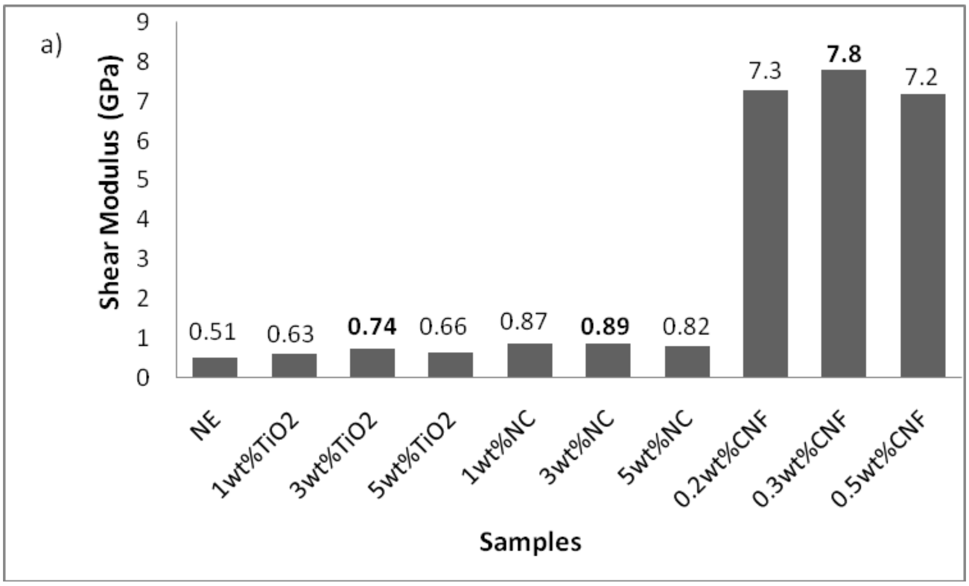


Fig. 5. SEM Micrographs of Flexural Specimens of biaxially carbon braided fiber nanocomposites: (a) NE; (b)1wt%TiO_2; (c) 3wt% TiO_2; (d) 5wt%TiO_2; (e) 1wt%NC; (f) 3wt%NC; (g) 5wt%NC; (h) 0.2wt%CNF; (i) 0.5wt%CNF; (j) 1wt%CNF

C. Shear Properties

Fig. 6a and 6b showed the dependence of the average value of three samples of each specimen's shear modulus and shear strength of the biaxial carbon braided composites on the relative weight percentage of nanoparticles respectively. The shear modulus and shear strength increased initially with nanoparticles content and then showed a reduction. Sample 0.5wt%CNF, showing the highest tensile strength and flexural strength, also exhibited the greatest shear strength among all specimens. It is known that CNF with biaxially carbon braided fiber act as reinforcement to the matrix rich edge and endorse shear properties by contributing resistance to cracking [19]. The shear modulus of 3wt%TIO2 is 0.74GPa and 3wt%NC is 0.89GPa, which are much smaller as it's compared with 7.8 GPa of the sample 0.5wt%CNF. It is clear that the biaxially braided carbon fiber composites containing CNF from 0.2wt% to 1 wt% were more efficient for improving the shear properties of the composite material than the other samples.



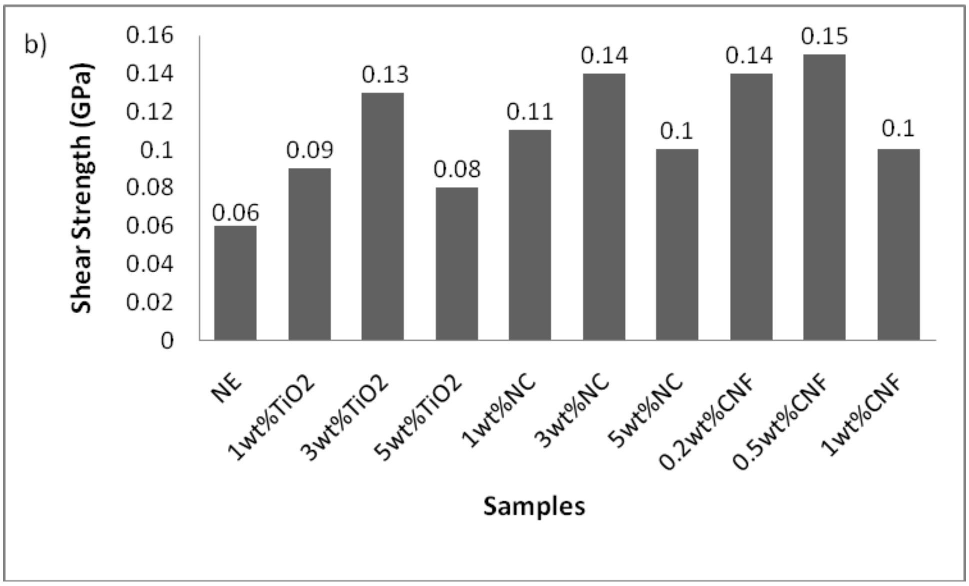


Fig. 6. Shear properties of biaxially carbon braided fiber nanocomposites: a) Shear Modulus and b) Shear Strength

To identify the mechanisms responsible for the enhancement of shear properties, the fracture surface of the specimens were examined in Fig. 7. It can be found that the fracture surface of the NE sample is relatively smooth in Fig. 7a, but the cross sections of the nanoparticle samples are rougher and more fractured, which means that more energy is needed to break the nanoparticle samples in Fig. 7b to 7j. It can be seen that nanoparticles dispersed in between the fibers, which is helpful for the strengthening of the composite materials. But few of nanoparticles exist in 5wt%TiO_2, 5wt%NC and 1wt%CNF and there is an inhomogeneous dispersion for the samples shown in Fig. 7d, 7g and 7j. So the increase of shear properties are mainly due to the addition of nanoparticles up to 3wt%TiO_2, 3wt%NC and 0.5wt%CNF as shown in Fig. 7c, 7e and 7i have the good dispersion compared with 5wt%TiO_2, 5wt%NC and 1wt%.

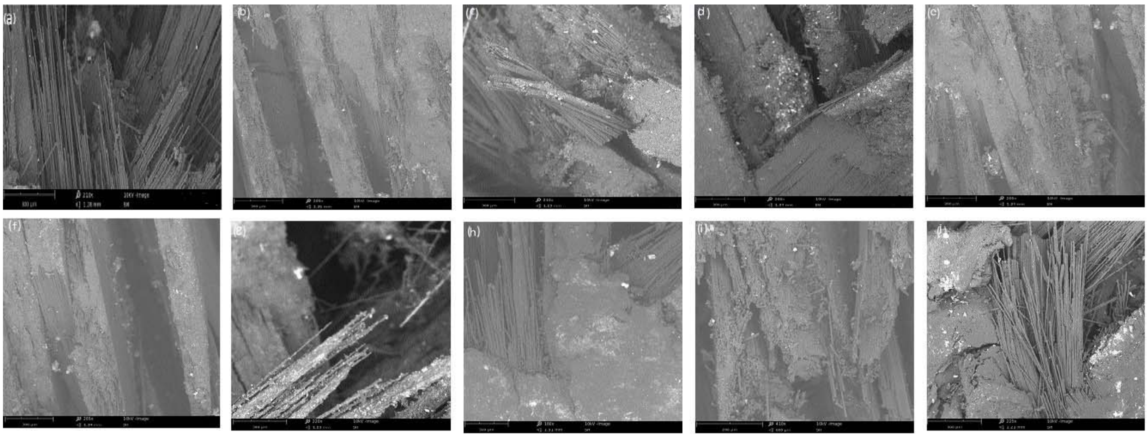


Fig. 7. SEM Micrographs of Shear Specimens of biaxially carbon braided fiber nanocomposites: (a) NE; (b)1wt%TiO_2; (c) 3wt% TiO_2; (d) 5wt%TiO_2; (e) 1wt%NC; (f) 3wt%NC; (g) 5wt%NC; (h) 0.2wt%CNF; (i) 0.5wt%CNF; (j) 1wt%CNF.

V. CONCLUSIONS

The demand for a high quality of lightweight materials is, for scientific and technical interests, increasingly becoming the focus of developing new materials. The effect of different nanoparticles on biaxial carbon braided composites was compared and characterized. The biaxial carbon braided composites utilizing CNF nanoparticles have unique mechanical properties not seen in any dispersed TiO_2 and NC nanoparticle samples.

In all measures, 0.5wt%CNF has the highest value in term of the tensile, flexural, shear, properties and also in analysis. From the study, it was seen that 3wt%TiO_2 and 3wt%NC loading also offered promising outcome in most cases. However, the secret of success might be with the selection, mixing and quantity of the nanoparticles. As long as the nanoparticles are well dispersed, it is possible to get good improvement in the properties. Heavy quantity and poor dispersion of nanoparticles lead to agglomerations. These lead to stress concentration locations from where the crack will commence and grow. .

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