FLEXURAL FATIGUE ENHANCEMENT OF CARBON/EPOXY COMPOSITES THROUGH NANOCLAY/GRAPHENE NANOPARTICLES

Mahesh Hosur[1*], Md. Sarower Tareq[2], Shaik Zainuddin and Shaik Jeelani[3]

[1] Mechanical and Industrial Engineering, Texas A&M University-Kingsville, Kingsville, TX, USA, mahesh.hosur@tamuk.edu
[2] Mechanical Engineering, Michigan State University, Lansing, MI, USA, tareqsarower30@gmail.com
[3] Materials Science and Engineering, Tuskegee University, Tuskegee, AL, USA, szainuddin@tuskegee.edu, sjeelani@tuskegee.edu

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ABSTRACT

In this work, the effect of two different nanoparticles on the flexural static and fatigue properties of carbon fiber reinforced polymer (CFRP) composites was investigated. Montmorillonite nanoclay (MMT) and graphene nanoplatelet (GnP) were dispersed in the SC-15 epoxy resin using combinations of ultrasonication, magnetic stirrer and 3-roll shear mixture. The CFRP composites were fabricated by hand layup and compression molding process. 3-point static flexural test result showed that nanoclay is more effective in achieving higher flexural stiffness and GnPs are more effective in increasing the flexural strength. However, addition of nanoclay and GnPs at a time (binary) did not show significant changes in the flexural properties. Load control 3-point flexural fatigue test was conducted at different stress levels (0.9, 0.8, 0.75 and 0.7). It was found that addition of nanoparticles significantly improved the flexural fatigue performance of the CFRP composites. These samples have also showed higher residual fatigue properties throughout the fatigue life. Optical microscopic (OM) and scanning electron microscopic (SEM) analyses of the fracture images indicated that addition of nanoparticles has improved fiber-matrix interfacial bonding of the CFRP composites resulting in reduced delamination and matrix cracking during the failure.

1 INTRODUCTION

Fiber reinforced polymer (FRP) composites are widely used in various high-performance applications such as aerospace, automotive, offshore, wind turbine blades, and sports, replacing conventional metal-based materials, due to their high specific strength and stiffness, combined with design flexibility ([1, 2]). The behavior of FRPs are highly dependent and controlled by the properties of their constituents, i.e., fiber, matrix, filler and the interactions between them ([3]). Among various reinforcing fibers, carbon fibers (CFs) provide the best specific properties ([4, 5]) Assessing the performance of FRPs under various loading conditions (i.e. axial, transverse, impact, torsional) is crucial in ensuring the durability and the desired design life of the structural components. Use of conventional carbon fiber reinforced polymer (CFRP) composites in some high-end applications is still restricted because of their low transverse load bearing capacity, poor resistance to crack propagation and delamination ([6, 7]). Specifically, when subject to transverse fatigue loading, composites faces fluctuating load with time resulting in initiation and growth of crack leading to fiber-matrix debonding and delamination. Therefore, it is important to find ways to improve the fatigue performance of CFRP composites in order to obtained the desired design life. One of the ways to achieve higher transverse properties is to increase the toughness of resin matrix. It can be done by changing the chemistry of the polymer matrix or by adding fillers that can achieve the objective.

In recent years, addition of nanoparticles to the polymer matrix has seen to enhance not only the properties of the polymers but also that of the composites by improving the interfacial bonding between the fibers and the matrices ([8-10]). Incorporation of modified nanoparticles in multiscale composite have been reported to significantly increase fiber-matrix interfacial bonding due to their outstanding specific strength and modulus, along with high surface to volume ratio that allows them to provide good interactions between fiber and matrix. As a result, these composites show improved flexural and interlaminar shear properties which are dominated by matrix rather than fiber ([11, 12]). Among all potential nanofillers montmorillonites nanoclay (MMT) is most widely studied because of the low cost and ease of dispersion in the resin. Among the carbonaceous nanoparticles, graphene nanoplatelets is the well-known 1-D nanoparticle that provide reasonable base to investigate and compare to the another 1-D nanoparticle nanoclay.

Apart from using one type of nanofiller, in recent years, addition of binary nanofillers attracted researchers' interest to explore the synergistic effect to further increase the interlocking effect and enhance fiber matrix interface and overall properties of FRP composites. Effect of binary nanofillers on epoxy composite have been studied by number of researchers with different mixture i.e., multiwalled carbon nanotubes (MWCNT) with graphene nanoplatelets GnP ([13]), carbon nanotubes (CNT) with carbon black ([14]) and graphene oxide with CNT ([15]).

This research work was focusing on investigating the static and fatigue behavior of CFRCs composites by incorporating two different kinds of nanoparticles. The flexural fatigue behavior of CFRCs in respect to stiffness degradation, fatigue life prediction and 3D damage mode with addition of graphene and nanoclay as matrix filler was investigated. Three-point flexural static tests were conducted to measure the elastic modulus and failure strength/deflection of the composites. Load-controlled flexural fatigue tests in the three-point bending mode was performed at different stress levels to determine the fatigue life and stiffness degradation, respectively. Finally, to investigate the fatigue failure mode optical microscopy (OM) and scanning electron microscopic (SEM) analysis was performed.

2 EXPERIMENTAL

In this study, 8 harness satin weave (8'' HS) with tow size 3k and thickness of 0.4572 mm carbon fiber supplied by US Composites Inc was used for reinforcement. The polymer resin used was SC-15 epoxy manufactured by Applied Poleramic, Inc., California, USA, which consists of two parts; part A (a mixture of 60–70% Diglycidylether of Bisphenol A and 10–20% aliphatic diglycidylether) and part B as hardener (a mixture of 70–90% cycloaliphatic amine and 10–30% polyoxylalkylamine). This is a room temperature cure epoxy resin with low viscosity of 300 cps and pot life of 6 h. The mixture ratio for the resin is 10:3 for part A and part B, respectively. Montmorillonite nanoclay (MMT) sold under the trade name Nanomer I.30E was supplied by Sigma Aldrich, USA. Surface of MMT has been modified by proprietary functional groups to make it compatible with the epoxy resin system.

For nanoclay dispersion, at first 2 wt% was measured and dried at 100 °C for 2 hours to remove moisture and avoid lump formation. Dried nanoclay was then mixed with part A of SC-15 epoxy resin by means of magnetic stirrer at 500 rpm for 3 hours at 40 °C. Three dispersion steps were followed to disperse graphene nanoplatelets (GnPs) in epoxy resin i.e., ultrasonication, magnetic stirrer and three roll calendaring mixing. At first 0.1 wt% GnPs was measured and mixed with part-A by means of ultrasonic cavitation technique (Hetofrig, Denmark) for 1 hour at 40 °C. To control the temperature of mixture a pulsar cycle (turning on and off-time ratio of 2:3) and water bath was used. The sonicated GnP-resin mixture was then put for magnetic stirring mixture for 3 hours at 500 rpm at 40 °C. At last three roll high shear mixture (Exakt 80E/ 0224, Germany) was used to disperse the platelet thoroughly and uniformly maintaining three consecutive gap between the roller of 15 µm, 10 µm and 5 µm were used at 120 rpm.

To disperse binary nanofillers (nanoclay and GnP), at first nanocaly was dried at 100 °C for 2 hours; and on the other side GnP was mixed with resin part-A by means of ultrasonication (as same condition as done for GnP alone). The dried nanoclay was then mixed with sonicated GnP-resin mixture manually, followed by magnetic stirrer and three roll high shear mixture (as same condition done for GnP alone).

To fabricate nanophased CFRP composites, unmodified (for control sample) and modified (containing nanofiller) part-A of the SC-15 resin system was mixed with part-B (hardener) at 10:3 stoichiometric ratio using a high-speed mechanical stirrer at 400 rpm for 10 minutes. The laminates were then fabricated by hand-lay-up process followed by compression molding technique. A total of ten layers of woven carbon fiber sheet were stacked one after another placing epoxy resin in between them. The laid-up laminate was packaged by porous Teflon, bleeder cloth and non-porous Teflon, and put on the platen of Wabash hot compression molding chamber for 4 hours at 60 °C and 1-ton pressure for curing. The cured laminate was cooled by annealing to avoid thermal stress and was collected after 12 hours. Finally, to eliminate the built-in stress, all laminates were post-cured at 100 °C for 3 hours. The fabricated composite laminates were cut using a tile saw cutter to make the samples for flexure test. The average thickness of the composite laminate was 3.5, 3.65, 3.55 and 4.05 mm for the control, nanoclay added, GnP added and binary nanoparticles added samples, respectively. The width and length of all the samples were maintained at 12 mm and 85 mm respectively.

Three-point flexural test was conducted on MTS 312.21 uniaxial testing machine (using 5 KN load cell) according to ASTM D790-03 ([16]). The test was conducted in displacement control mode at room temperature and at a crosshead speed of 1.2 mm/min. At least five specimens of each type were tested, and the properties were compared with neat composites. Three-point flexural fatigue test was performed in the same machine (MTS 810 and 5 KN load cell) according to the specifications of ASTM D7774-17 ([17]). The test was conducted in constant amplitude sinusoidal load control mode at a stress ratio of 0.1 and frequency of 5 Hz. The samples were tested in four stress levels, S = 0.9, 0.8, 0.75 and 0.7 respectively, where S is the ratio of applied stress to the ultimate flexural stress obtained from the static test. At least 5 specimens were tested for each stress level up to 1 million (1 x 10^6) cycles that is generally defined as "run-out" fatigue criteria. The test termination criteria were defined as the 25% drop of the responded load or the tests were manually stopped as the samples were found to exceed the "run-out" criteria. To examine the residual fatigue properties, static flexural test was performed by terminating the fatigue test after a certain number of cycles.

Optical Microscopy (OM) and Scanning Electron Microscopy (SEM) were conducted on flexure fractured samples using Olympus SZX16 and JSM-7200F FESEM, respectively. Prior to SEM, the samples surfaces were sputtered by Au-Pd particle in a Hummer ® 6.2 sputtering system. In the purpose of analysis and comparison several SEM micrographs of both static and fatigue fractured samples were obtained from each sample at various magnifications.

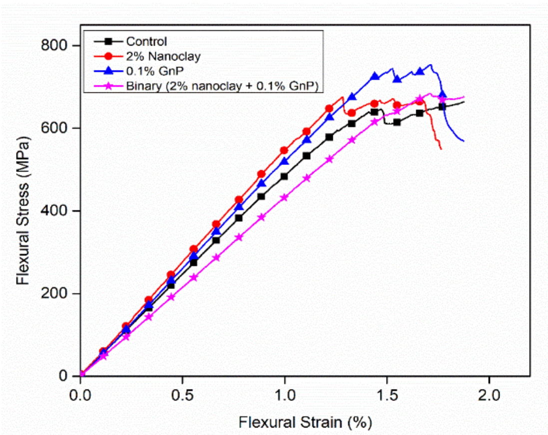

Figure 1: Flexural Stress-Strain response of as received control and nanoparticles added CFRP composites.

3 RESULTS AND DISCUSSIONS

3.1 Static Flexure Test

Figure 1 represents the typical flexural stress-strain response of CFRP composites tested in 3-point loading. All of the samples showed linear pattern with a steep rise in stress up to yield, followed by nonlinear response with decreasing slope up to the maximum flexural stress. The reason for nonlinear portion is the initiation of microcracks at the fiber-matrix interface that make the load bearing capacity of the composite to fluctuate. However, the interfacial bonding and overall internal structure is still strong enough to absorb load up to a maximum point. After the maximum stress point, a relative sudden fall in stress-strain curve was observed in individual nanoclay and GnPs added samples compared to the control counterpart. This indicates that after maximum stress point, fiber-breakage are more dominating in nanoparticle added CFRP composites that causes abrupt fall in strength; whereas, for control sample, cracking and delamination are more dominating which cause slow fall down. This is because of the improved interfacial bonding achieved by the addition of nanoparticles resulting fiber-matrix debonding and delamination more difficult, and hence, the ultimate failure in these samples occurred due to fiber-breakage.

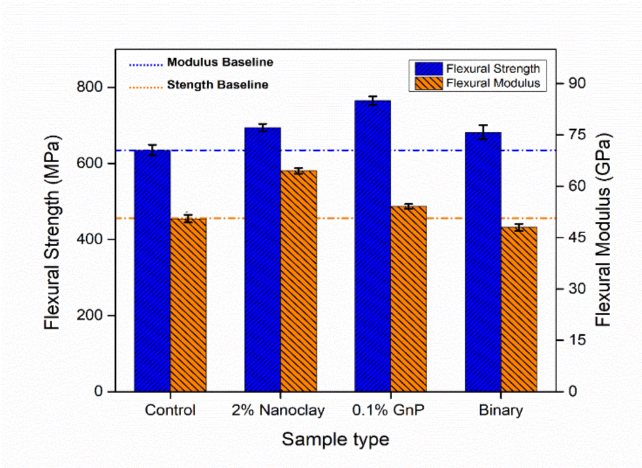

Figure 2: Changes in flexural strength and modulus upon addition of nanoparticles in the CFRP composites.

It can be seen from figure 2 that individual nanoclay and GnPs added CFRP composites showed increased flexural strength and modulus compared to the control samples. Nanoclay added CFCs exhibited 28% higher flexural modulus (64.5 GPa) than the control samples (50.5 GPa), that is the highest improvement in flexural modulus among the all types. In contrast, though GnPs added samples showed little improvement (7% than the control samples) in flexural modulus, these samples showed highest flexural strength of 765 MPa with a 21% increase over control samples (635 MPa). These results indicated that nanoclay are more effective in increasing the stiffness of the CFRP composites and GnPs are comparatively more effective to achieve higher strength of the CFRP composites. However, the change in flexural strength in binary nanoparticles added CFRP composites was not considerable significant. In contrast to the individual nanoparticles, binary nanoparticles added samples showed reduced stiffness by 5%, and thus, exhibited maximum strain to failure of 17%, whereas, control samples showed 12% strain to failure. Therefore, by adding both nanoclay and GnPs together the CFRP composites was found flexible and exhibited higher deformation before failure.

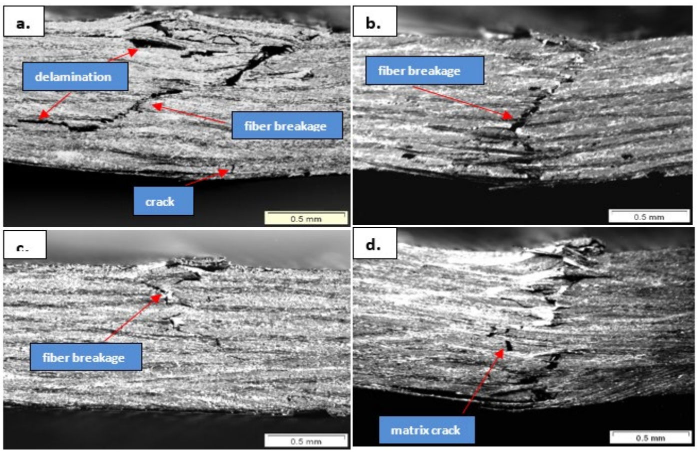

Figure 3: Optical microscopic images of fractured specimen. a) control, b) nanoclay added, c) GnP added, d) binary nanoparticles (nanoclay and GnP) added.

OM and SEM images of flexure fractured samples are shown in figures 3-5. OM image of control sample (Figure 3) showed large delaminated area at both compressive and tensile sides of the samples. Matrix cracking and fiber breakage were also observed in these specimens. On contrary, fractured specimens of nanoparticle added CFRP composites (Figs. 3 b-d) showed less delamination, specially, individual nanoparticle added CFRP composites (Figs. 3 b-c) showed no considerable delamination. The large delaminated area and severe matrix cracking in control fractured specimens could be attributed to poor fiber-matrix interfacial bonding, and relatively brittle and weaker nature of matrix.

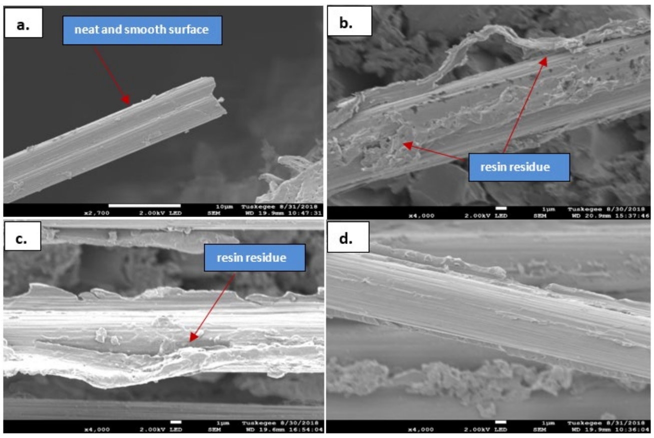

Figure 4: SEM images showing single fiber surfaces of fractured specimens in flexure test of a) control, b) nanoclay added, c) GnP added, d) binary nanoparticles (nanoclay and GnP) added CFRP Composites.

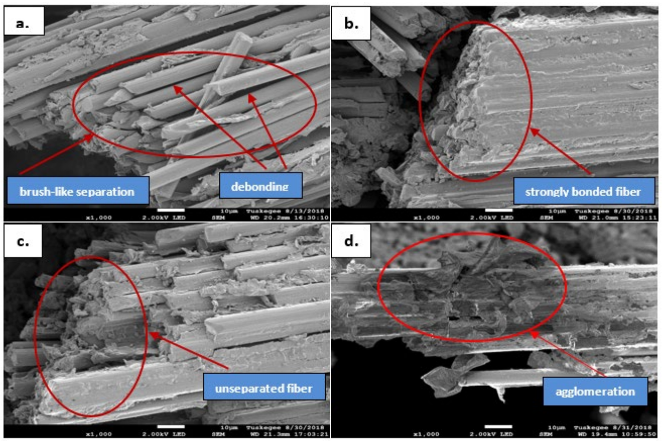

Figure 5: SEM images of fractured fiber bundle in flexure test of a) control, b) nanoclay added, c) GnPs added and d) binary nanoparticles (nanoclay and GnP) added CFRP composites.

On the other hand, less delaminated areas and matrix cracking in nanoclay and GnPs added CFRP composites indicated enhanced interfacial bonding that may have restricted the fiber-matrix debonding, fiber pull-out and ultimate delamination evident from SEM images of ‘protruded single fiber’ and ‘broken fiber bundle’ of fractured specimens, respectively (Figs. 4 and 5). From Figure 4, it can be seen that single fiber surface of control specimen (Fig. 4 a) is smooth with no resin residue, whereas, in contrast, nanoclay and GnPs added CFRP composites (Fig. 4 b-d) were found to show a considerable amount of resin residue on surface, even after separation. SEM images of broken fiber bundle (Fig. 5) of fractured specimen indicated that the fibers were remained intact with almost no separation or debonding of fibers (Figure 5 b-c), whereas, the control CFRP composites samples showed brush-like separated and unbonded smooth fibers (Fig. 5 a). The reason behind these improvement in interfacial bonding can be attributed to the presence of surface modified nanoparticles in epoxy matrix that may have facilitated the fiber matrix interactions due to their very high aspect ratio and ensured good fiber-matrix bonding because of active functional groups on nanoparticles' surface ([18]). In addition, nanoparticles in epoxy matrix may have obstruct the polymer chain mobility, and increased shear strength that that allows stress to transfer through friction and increase interfacial bonding in fiber composites. This effect of strong interfacial bonding is found to be more significant for individual GnPs added samples, as they showed highest flexural strength and almost no delamination during fracture (Figure 5 c). This could be attributed to the inherent strongest mechanical properties of GnPs and the presence of active NH_2 functional groups on GnPs' surface that acted effectively to increase interaction. These changes in fiber-matrix bonding and morphology ultimately increased transverse (out-of-plane) properties of the CFRP composites with good flexural strength and modulus.

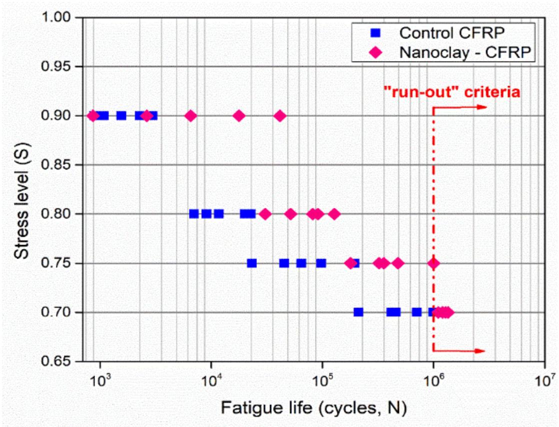

Figure 6: Fatigue life for the control and nanoclay added CFRP composites at four stress levels.

3.2 Flexural Fatigue Test

Figure 6 illustrates the tested fatigue life in respect to the four stress levels of the control and nanoclay added CFRPs. It is seen that irrespective of the stress level, nanoclay added samples exhibited significantly longer fatigue life than control samples. At stress level of 0.9, 0.8 and 0.75, mean fatigue life of the nanoclay added CFRPs were found to be 687%, 327% and 384% higher than the control CFRP composites respectively. At 0.7 stress level, all of the nanoclay added samples demonstrated infinite fatigue life as they exceeded “run-out” fatigue criteria (10^6 cycles), whereas, majority (80% among tested) of the control samples at 0.7 stress level failed at lower fatigue cycles than the “run-out” criteria. Fatigue data for nanoclay added samples at stress level 0.7 are seen to be clustered at a small region, since the tests were manually stopped when the number of fatigue cycles for these specimens were found exceeded the “run-out” criteria.

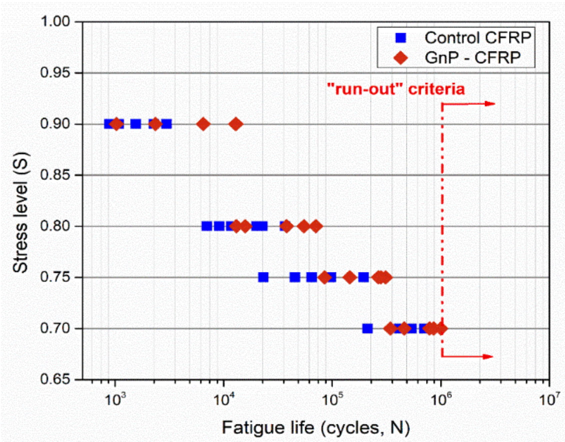

Figure 7: Fatigue life for the control and GnP added CFRP composites at four stress levels.

Figure 7 is showing the tested fatigue life data of the graphene nanoplatelet (GnP) added CFRP composite composites in compare to the control CFRP composite samples. The fatigue life data have been presented in respect to the different stress level. It is seen that GnP added added CFRP composite has demonstrated longer flexural fatigue life than the control CFRP composite samples, irrespective of the stress level. However, though some GNP added samples showed fatigue life in between the control samples at a definite stress level, majority of the GnP added samples showed significantly higher fatigue life in comparison. It is seen that addition of GnP improved the flexural fatigue life of the CFRP composites by 60% to 155% in different stress level. Maximum improvement was obtained at the stress level, S = 0.75.

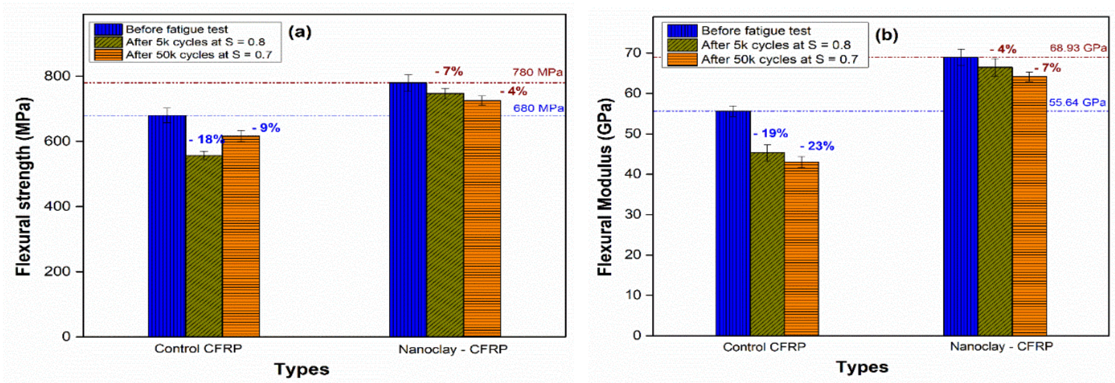

Figure 8: Comparison of the (a) residual flexural strength and (b) modulus of control and nanoclay added CFRP composites after 5K and 50K cycles at 0.8 and 0.7 stress level, respectively.

3.3 Residual Fatigue Properties

To investigate the residual properties, static flexure test was performed by terminating the fatigue test after a certain number of cycles for CFRP composites reinforced with nanoclay. Figure 8 is showing the comparison of residual flexural properties after the fatigue cycles of 5k and 50k at stress level of 0.8 and 0.7, respectively. From Figure 8a, it is seen that loss of flexural strength after the mentioned fatigue cycles was more than twice (in percentage) for the control samples than the respective nanoclay added samples. For example, after 5k cycles at 0.8 stress level, control samples lost about 18%, whereas nanoclay added samples lost about 7% of the respective initial flexural strength. Loss of flexural modulus after the mentioned cycles was also higher for the control samples than the respective nanoclay added samples. Hence, it is reasonable to mention that nanoclay added CFRP composites exhibits higher residual strength and stiffness than the control CFRP composites throughout the fatigue life.

6 CONCLUSIONS

In this work, a comparative study of the effect of two different types of nanoparticles (nanoclay/ GnP) on the static and fatigue properties of CFRP composites under flexural loading has been carried out. Nanoclay and GnPs when added individually improved both flexural strength and modulus of the CFRPs. Maximum improvement in flexural strength and flexural modulus were obtained for GnPs (14.3%) and nanoclay (19%), respectively. Microstructural analysis indicated that nanoclay and GnPs significantly improved interfacial bonding of CFRP composites. Delamination and matrix cracking were found to be significantly less. In contrast, fiber breakage was found as the main failure mode in nanoparticle added samples. Incorporation of nanoclay significantly improved the flexural fatigue performance of the CFRP composites. GnP added CFRP composites also demonstrated better fatigue performance than the control CFRP composites though the improvement is lower than the nanoclay added CFRP composites.

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