EXPERIMENTAL AND NUMERICAL STUDIES OF FATIGUE PROPERTIES OF CARBON/GLASS FIBER/EPOXY HYBRID COMPOSITES ENHANCED WITH NANO TiO$_2$ POWDER

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Abstract

The present work deals with the fatigue behavior of hybrid nanocomposites consisting epoxy strengthen by unidirectional carbon fibres, and/or woven roving glass fiber and TiO$_2$ nanofiliders. For this purpose, nanocomposite material was manufactured by mixing TiO$_2$ nanoparticles with the epoxy using an ultrasonic mixer to insure complete dispersion of such particles in the base material. Different particle concentrations (1, 3, and 5) % wt. of TiO$_2$ nanoparticles have been added to the epoxy. Different types of hybrid nano composite materials were manufactured by adding three layers of carbon fibers and/or woven roving glass fiber to the prepared epoxy nanocomposite materials with a constant weight fraction of 30%. The laminated hybrid nanocomposite materials were then prepared using hand lay-up technique using a vacuum device. For experimental purposes tensile and fatigue test specimens have been manufactured according to ASTM-D3039 and ASTM D 3479/D 3479M–96, respectively, while ANSYS19 program was used to analyze the fatigue behavior of such materials numerically. Tensile tests were carried out at room temperature while fatigue tests has been carried out at constant stress ratio (R=0.1). Scanning electron microscope (SEM) was used to identify the underlying mechanisms for fatigue failure and the progressive of damage growth. For each test, three specimens were tested and the average magnitude for each property was taken. The results obtained indicated that the hybrid nanocomposite (EP+C/C/C+3% TiO$_2$) has the highest fatigue limit and tensile strength in comparison with the other tested material, while the SEM showed that the composite failed by a brittle way. It has been also generally observed that the addition of (TiO$_2$) nanoparticles has a positive effect on the fatigue behaviour of the such materials.

Keywords: Hybrid Nano-composite materials, Fatigue, Mechanical properties.

1. INTRODUCTION

Structures are normally subjected to vibrations and other changing loads which degrade stressful materials. Fatigue is a major cause of the failure of structural components. Mechanical behavior of composites under fatigue loading seems to be affected by fiber breakage, matrix cracking, inter-fiber fracture, and delamination between the layers. Different types of polymers such as epoxy, polyester, and vinylesters were used as a base for the composite materials [1][2]. Many studies have confirmed that the incorporation of the small volume fraction of nanofibers as a filler in the polymer led to improve several fatigue properties. Grimmer and Dharan [3] show that the addition of small volume fractions of multi-walled carbon nanotubes [MWCNTs] to the epoxy matrix /glass fibers, resulted in a significant increase in high cycle fatigue life. Khan, et al [4] stated that combination of nano clay into CFRP composites can be improve the composite's mechanical properties in static loading as well as the fatigue life for a cyclic load and the mechanical residual properties after a given cyclic fatigue period. Pinto et al [5] displayed that the fatigue crack propagation resistance of the epoxy resin can be improved by the addition of TiO$_2$ nanoparticles. Wetzel et al [6] exhibit that adding Al$_2$O$_3$ nanoparticles to the epoxy improves its fatigue crack propagation resistance. It has been shown that the crack in dynamically loaded nanocomposites propagated at lower rates than in neat epoxy. Ajaj et al [7] observed that the existence of silica nanoparticles in the epoxy polymer decreases the brittleness of epoxy resin and improving the maximum fatigue stress, fatigue life, and surface roughness of the composite. Khan, et al [8] investigated the influence of nano clay addition on fatigue behavior of carbon fiber-reinforced polymers (CFRP). The obtained results showed that combination of nano clay into CFRP composites not only increases the composite's mechanical properties in static loading as well as the fatigue behaviour for a given cyclic load level and the mechanical properties after a given cyclic fatigue period. Borrego, et al [9] studied the fatigue behavior of nano clay and multiwalled carbon nanotubes composites. It has been observed that the
fatigue ratio in tension–tension loading rises with the addition of nano clay and multi-walled carbon nanotubes, proposing that both types of nanoparticles can act as retard to the fatigue crack propagation. Ansari, et al [10] studied the effects of fiber volume fraction, fiber orientation, fiber type, etc. on the fatigue life behavior of fiber-reinforced polymer composites. It has been concluded that the frequency of fatigue increases and then decreases with the increase of the fraction of the fiber volume to a certain amount. C. Capela et al. [11], investigated the fatigue life of Epoxy-based composite strengthen with carbon fiber with various volume fractions within 5-20 % and 500 μm length. The results indicated an enhancement in the fatigue, tensile strength, and stiffness attendant to the fiber volume fraction growing up to 17.5% by 140%, 52%, and 400%, respectively but then, the mechanical properties were slightly decreased. Amore and Grassia [12] studied the response of carbon/epoxy composite when exposed to constant amplitude cyclic loadings to determine static strength, fatigue limit, and residual strength. It has been noticed that the model captures the essential features of the composite’s response, including the hierarchical damage into particular insights development. Lee et al. [13] studied the effect of the fiber direction with three various angles (0°, 45°, and 90°) on the high cycle fatigue behavior of glass fiber with 30 wt.% implanted in Polyamide 6,6. The results exposed that the glass fiber with 0° direction has the highest fatigue life strength for all the testing conditions, and the failure has occurred due to microcracks generation, debonding, and voids at the fiber ends. Bondy et al. [14] studied the influence of the direction of the fiber on the fatigue life of nylon 6,6 reinforced with 40 percent long carbon fiber directed at three different flow angles (0°, 45°, and 90°). The results showed that the rise in the fiber flow angle affected a decrease in the degree of fatigue stress. Mustafa B. Hunain et al. [15] investigated experimentally the effects of activated carbon powder, with various weight fraction ratios of (0, 5, 10, 15, 20, 25, 30, 35 and 40) % wt., with epoxy resin on tensile properties. The results showed that the tensile strength magnitude increased with increasing AC content up to 15 % wt., then it decreased at 40 % wt. Basim A. Abass et al [16] investigated the mechanical properties of a hybrid epoxy composite strengthened by TiO₂ nano powder, glass fibers and unidirectional carbon fiber. Different weight percentages of TiO₂ nano-powder was dispersed in epoxy using sonication. The results show that the addition of TiO₂ nanoparticles at up to 3%wt enhances the mechanical strength of such material. Ali S. Al-Turaihi et al [17] deals with the effect of volume fractions (0, 10, and 20%) on the fatigue life of unsaturated polyester matrix reinforced with E-glass chopped fiber experimentally and numerically. The results show that the tensile strength and elastic modulus of such composite is increased when the volume fraction increased from 0% to 20%, while the fatigue strength of composite reinforced with 20% of volume fraction increased by about 48% compared with that pure polyester. The numerical part using ANSYS/16 workbench software shows good agreement with the experimental part with maximum max difference of 7%.

Mustafa B. Hunain et al [18] examined the tensile strength of laminated polymer matrix carbon fiber composites for different stacking sequence. The results showed that the sequence of the fibre layer is very important in case of use unidirectional fibres.

It is clear from the above literatures that the tensile and fatigue and behaviour of laminated composite material consists of epoxy resin strengthened with unidirectional carbon and/or woven roving glass fibres with and without nano titanium dioxide have been a little studied, which is the essential purpose of the present work.

In this work, the effect of adding TiO₂ nanoparticles with various weight percentages (1, 3, and 5), on tensile and fatigue properties of epoxy reinforced with three laminates of carbon and/or woven roving glass fibres with 30%wt. was examined and discussed in detail experimentally and numerically.

2. EXPERIMENTAL WORK

2.1. Materials Used

Materials used for experiments of the present work include the epoxy resin used as a matrix of the nano hybrid composite materials. Epoxy type Quickmast 105 produced by “Don Construction Products (DCP) company”, with suitable hardener was used in the present work. Mixing ratio is 1:47:4, solidifying time 40 minutes at 35°C, density 1.1 g/cm³, and mixing viscosity 1.0 poise at 35°C [19]. TiO₂ nanoparticles produced by “MTI Corporation” with surface area per unit mass 210 ± 10 m²/g, average particle size of 50 nm, a density of 0.25 g/cm³, and purity greater or equal to 99.9%. Unidirectional carbon fiber fabric delivered by “Sika Wrap-300 Company, Switzerland” with density 1.81 gm/cm³, Young modulus of elasticity 230GPa and Poisson’s ratio of 0.21 [20]. Woven Roving glass fibers (GF) in the form of bidirectional fabric delivered by interweaving direct roving Fabrication Process “china” having a density of 2 gm/cm³, Young’s modulus of elasticity 72.53 GPa and Poisson’s ratio of 0.22 [21].

2.2. Nanocomposite materials preparation

The nanocomposite material was manufactured by adding different amount of TiO₂ nanoparticles (1, 3, and 5 % wt.) to the epoxy resin. The epoxy resin was firstly diluted by using 2 parts of acetone solvent to 10 parts of epoxy and stirred manually for 5 min [22]. The TiO₂ nanoparticles with specified particle concentration was then added to the epoxy and mixed using homogenizer vibrator.
“Ultrasonic Homogenizer FS-1200N”. The mixture was subjected to high speed for 10 minutes in distinct designs of 20 s on and 10 s off; in order to break down the agglomerating bodies and create they fully dispersed in the liquid medium. Then, the mixture was mixed under ambient conditions using a magnetic hotplate stirrer “Labtech Co. LTD” at a high power for duration 30 min. to confirm the evaporation of acetone. The hardener with recommended ratio (1.47:4) was then added to the mixture and all components were mixed at room temperature (25°C). Reinforcing fibres and liquid matrix resin layers are added sequentially to fabricate laminated sheets using hand lay-up technique using suitable glass mould that has dimensions of (250× 250× 4) mm$^3$ [23]. Finally, the manufactured lamina subjected to low vacuum at -30 psi for 24 hours in order to eliminate the trapped air formed as a consequence of the reaction between the components. The laminated composite was left to dry at ambient temperature for 48 hours and then the samples were left for 3 hours in an oven at 70 °C in order to achieve a sufficient curing. This procedure is essential for finishing polymerization, releasing stresses convinced during the manufacturing process, and completing sheets hardening [20].

2.3. Fabrication of the samples
The sheets for each hybrid nanocomposite material were then used to prepare the fatigue and tensile testing samples according to the standard of each test using CNC lathe machine “Suda ST1212 CNC router”.

2.4. Experimental Testing Procedure
2.4.1. Tensile test
Tensile tests for “EP+C/C/C”, “EP+C/G/C”, “EP+G/C/G”, and “EP+G/G/G” as well as that hybridized with 3%TiO$_2$ have been performed according to the ASTM-D3039 standard [24]. A universal tensile test machine with maximum load of (5KN) “LARYEE-50 KN” was used to carry out such tests. The load was applied with speed of (2 mm/min) at ambient temperature until the specimen’s failure [25]. Young’s modulus (E), ultimate tensile strength, and percentage of elongation at the break point are the main results of such test. Each test was duplicated three times for each specimen and the average magnitude were adopted.

Fig. 1. Dimensions of tensile specimen according to the (ASTM D3039).

2.4.2. Fatigue test
Fatigue test specimens were fabricated according to “ASTM D 3479/D 3479M-96”, standard test method for the fatigue of epoxy composite materials [26]. A flat plate test specimens to satisfy the machine test section with the dimensions shown in figure 1 [27] have been prepared. Fatigue tests were performed using “avery fatigue testing machine type 7305” which allow the application of cyclic reversed bending loading with cycling rate of 1400 rpm with or without an initial static load.

Grips are given for the bend test where the load is applied at one end of the specimen by an fluctuating spindle driven by means of a double eccentric attachment, crank, and connecting rod. The eccentric attachment is adaptable to give the essential range of bending angle. The applied stress is calculated from the deflection angle and the applied moment. The cyclic bending stresses were tension-compression stresses, so the machine was adjusted at a stress ratio R = -1. Some of the tested fatigue specimens can be shown in figure 3.

Fig. 2. Fatigue test specimen with dimensions in mm [26].

Fig. 3. Fatigue test specimen after testing.

2.4.3. Scanning electron microscope test (SEM)
Test specimens were cut and examined at the fracture surface using the Scanning Electron Microscope (SEM) model “VEGA3-TESCAN” shown in figure 3. The SEM was employed to determine the morphology of composite polymer specimens fabricated in this work. Specimen of small sizes (10 mm x 5 mm) was employed for
analysis at fracture plane. To get strong electrical conductivity all specimens were coated with gold by sputtering and the secondary electron images were recorded [28].

3. NUMERICAL ANALYSIS

A mathematical model for the fatigue of hybrid nanocomposite material was created using ANSYS 19 program. The fatigue specimen described previously was created using solid work program. Then the model was discretized using solid 185 structural element with total number of elements and nodes 6768 and 35579 respectively as can be shown in figure 5 (a). The main inputs to the program are Young’s modulus of elasticity, poisons ratio and density while the main output is the fatigue strength of the material. The main boundary conditions are fixed support was applied on one side while the applied load (displacement) is applied on the other side as shown in figure 5 (b).

4. RESULTS AND DISCUSSION

4.1. Tensile modulus.

The tensile modulus of different materials studied in the present work has been obtained from the stress strain curves shown in figure 5 (a-d).

(a) EP+CCC fibers + wt.% of TiO$_2$ nanoparticles.

(b) EP+CGC + wt.% of TiO$_2$ nanoparticles.

(c) EP+GCC + wt.% of TiO$_2$ nanoparticles.

(d) EP+GGG + wt.% of TiO$_2$ nanoparticles.

Fig. 4. Scanning electron microscope (SEM).

Fig. 5. Stress-strain curves of different hybrid nanomaterials.
The variation of tensile modulus for the composite and hybrid nanocomposite material samples with various weight fractions of TiO$_2$ and different stacking arrangement of the fibers are presented in figure 6. It can be noticed from this figure that the hybrid nanocomposite material with 3% TiO$_2$ has the highest tensile modulus comparison with the composite and other tested hybrid nanocomposite materials. The percentage increase in tensile modulus hybrid nanocomposite materials with 3% wt TiO$_2$ in comparison with other studied composite materials can be shown in table 1.

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**4.2. Fatigue test results.**

**4.2.1. Experimental results**

Fatigue test was carried out for all the types of the prepared laminated composite materials. Nine specimens were tested for each type of hybrid nanocomposite materials under different applied moments. The results have been graphically presented in the form of S-N curves. The S-N curves for different hybrid nanocomposite materials with different TiO$_2$ nanoparticles addition can be shown in figures 8-11. The results presented in these figures and that summarized in table 2 for the maximum stress to failure and the total number cycles to failure show that the laminated composite material with three layup of GGG is the more affected material by the addition of 3% TiO$_2$ than the other tested in the present work. The maximum percentage increase in stress to failure for GGG+3% wt TiO$_2$ has been calculated and found to be 30%. In general, the addition of TiO$_2$ to the composite material with percentage up to 3% wt enhances the fatigue strength of the hybrid nanocomposite material. This can be attributed to the greater surface area of the nanoparticles which allow good adhesion between the material components and higher resistance to the crack propagation. The addition of the nanoparticles by weight percentage greater than 3% may lead to the agglomeration of such particles and decreases the fatigue strength of the material in this case. These findings supported by that obtained in references [5] and [29].
The data presented in figures 8-11 have been correlated using Basquin’s equation of the power-law to give the equations presented in table 3. It can be observed from this table that the obtained data are well correlated since the coefficients of determination $R^2$ are high enough to ensure accurate fitness of the data. This result is in agreement with the reference [30].

The effect of TiO$_2$ addition by (1%, 3% and 5%) to the each of three lamina of different composite materials can be shown in figures 12-15.
4.2.1. Numerical Results

Numerical model was built using ANSYS 19.0 software. This model was validated by comparing the S-N curve for [EP+C/C/C+3%TiO$_2$] obtained experimentally with that obtained numerically as can be shown in figure 16. The maximum percentage error between results was calculated and found to be 5% as can be shown in table 4. This confirms the good agreement between the numerical and experimental results.

4.3. Results of Scanning Electronic Microscope (SEM)

The scanning electronic microscope was used to study the morphological properties of the failed specimens under fully reversed fluctuating bending fatigue for hybrid nanocomposite material reinforced with three layers of carbon fiber and TiO$_2$ nanoparticles with different particle concentrations as can be shown in figure 17 (a-h). This figure shows the main modes of fatigue fracture for different types of hybrid nanocomposite materials. It is clear that the main modes of failure for this material is the fiber fracture, delamination and fiber pullout. Formation of transverse matrix cracks can be also seen from these figures. This can be attributed to the presence of higher stress concentration followed by de-bonding of the fiber matrix and induced localized ply delamination. The matrix cracks and plies delamination expand and trigger bundles as the fatigue cycling continues (at 900 to loading direction) to split and fracture, setting the stage for final fracture.

5. CONCLUSIONS

Numerical and experimental investigations of the fatigue properties of epoxy resin strengthened by unidirectional carbon fibers, woven roving glass and Titanium dioxide with different weight fractions have been implemented in the present work. The following are the main conclusion remarks:

1. Increasing the weight fraction of TiO$_2$ nanoparticles up to 3% enhances the mechanical
and fatigue properties of the manufactured composite and hybrid nanocomposite.
materials was validated successfully by comparing the obtained results with that obtained from the experimental work with only 5% deviation in the results.

6. RECOMMENDATIONS FOR FUTURE WORK

1. Investigate the influence of varying volume fraction on the mechanical properties, and fatigue behavior, of such laminated composite materials.
2. Study the effect of particles addition by varying particle type (like carbon nano tube), particle size, and particle volume fraction.
3. Evaluate the fatigue damage of the hybrid nanocomposite materials under combined loading.

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