

Fatigue Analysis of Hybrid Glass Carbon Epoxy Composite

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Abstract- Two types of glass fiber reinforced plastic (GFRP) composites were fabricated viz., GFRP with neat epoxy matrix (GFRP-neat) and GFRP with hybrid modified epoxy matrix (GFRP-hybrid) containing 9 wt. % of rubber microparticles and 10 wt. % of silica nanoparticles. Fatigue tests were conducted on both the composites under the WISPERX load sequence. The fatigue life of the GFRP-hybrid composite was about 4-5 times higher than that of the GFRP-neat composite. The underlying mechanisms for improved fatigue performance are discussed. A reasonably good correlation was observed between the experimental fatigue life and the fatigue life predicted under the spectrum loads.

Index Terms- glass fiber composite, silica nanoparticle, rubber particle, spectrum fatigue.

I. INTRODUCTION

Due mainly to their high specific strength and stiffness, continuous fiber reinforced plastic (FRP) composites are widely used in various structural applications such as airframes, wind turbines, ship hulls, etc. Such composite structural components experience variable amplitude or spectrum fatigue loads in service. Hence, the fatigue-durability of the composite materials under spectrum loads is an important requirement in these applications.

Engineering polymer matrix composite materials generally consist of continuous glass or carbon fibers embedded in a thermosetting epoxy polymer. The epoxy polymer, being an amorphous and highly cross-linked material, is relatively brittle and exhibits a relatively poor resistance to crack initiation and growth, thus affecting the overall mechanical properties, including the fatigue and fracture behavior of FRP composites. One of the ways to improve the mechanical properties of FRPs is to add a second phase of fillers into the epoxy matrix.

Incorporation of various types of micro- and nano-sized spherical, fibrous and layered fillers into the epoxy has been shown to improve the mechanical properties of composites [1-3]. Considerable improvements in the strength and stiffness [4], and dramatic improvements in the fracture toughness [3-5] of polymer composites by addition of particles such as nanosilica have been observed. The presence of minute amounts (0.3 wt. %) of double walled carbon nanotubes (DWCNT) increases the matrix-dominated interlaminar shear strength (ILSS) of GFRP by 20% [6]. A recent review on CNT-polymer composites by Spitalsky et al. [7] clearly shows the improvement in tensile strength and toughness of epoxies due to presence of carbon nanotubes. Chisholm et al [8] observed that the addition of 1.5 wt. % of nanosized SiC particles in an epoxy led to a 20–30% increase in the tensile properties. The incorporation of 2 wt. % of carbon Nano fibers in a SC-15 epoxy/carbon fiber composite improves the tensile and flexural strengths by 11 and 22% respectively [9]. The use of 5 wt. % of nanoclay in polypropylene improves both the modulus and yield strength, by 90 and 5% respectively [10].

Several investigators have studied the fatigue behavior of bulk epoxies modified with various types of fillers. A significant enhancement in fatigue life [11] and considerable reduction in the fatigue crack growth rate (FCGR) of epoxies containing rubber particles have been well established [12-14]. The energy absorption mechanisms of rubber particle cavitation and the associated plastic deformation of the surrounding epoxy have been attributed to such improved fatigue performance. Similarly, epoxy containing silica nanoparticles has been shown to exhibit improved fatigue life [15, 16] and reduced FCGR [17]. The creation of nanovoids by the debonding of hard silica particles and the subsequent

void growth has been shown to absorb energy and thereby increase the fatigue life. The influence of carbon nanotubes and carbon nanofibers on the fatigue behavior of epoxies has been investigated by several authors [18, 19]. Once again, large increase in the fatigue life and significant reduction in fatigue crack growth rate have been observed and attributed to energy absorbing mechanisms such as nanotube/nanofiber pull out and crack bridging.

The above studies mentioned pertain to the study of fatigue behavior of modified bulk epoxies. Recent investigations have confirmed that use of such modified epoxies in FRPs give improved fatigue properties of the FRPs as well. Grimmer and Dharan [20] observed that the addition of 1wt.% of multi walled carbon nanotubes (MWCNTs) to the polymer matrix of a GFRP composite laminate improved the high-cycle fatigue strength by 60–250%. Bortz et al. [21] observed that carbon nanofiber (CNF) reinforced composites collectively possess improved fatigue properties over their unmodified counterparts. Fatigue life improvements of 150–670% were observed in fully compressive, tensile and tensile-dominated loadings. The addition of silica nanoparticles into an epoxy matrix has been shown to enhance the fatigue properties of GFRP composites [15, 22]. Incorporation of nanoclay into an epoxy matrix has also been shown to improve the fatigue behavior of FRP composites [23].

Recently, the authors have observed that the addition of either 9 wt.% rubber micro particles [11] or 10 wt.% silica nano particles [15] to an epoxy matrix enhances the fatigue life of a GFRP composite by about three to four times. Further, we have studied [24] the fatigue behavior of a hybrid GFRP composite containing both 9 wt. % of micron-rubber and 10 wt. % of nano-silica particles in the epoxy matrix. It was clearly observed that the fatigue life of GFRP composite is enhanced dramatically, by about eight to ten times.

Most of the fatigue studies on modified epoxies and FRPs with such modified epoxies, including the one referred to above, have been limited to constant amplitude fatigue behavior. As an intermediate step between constant and variable amplitude fatigue studies, the authors [25] have investigated the three-step block load fatigue behavior of the GFRP hybrid composite. It was observed that under both an increasing and a decreasing block load sequence, the

fatigue life of GFRP composite is enhanced by incorporation of the micron-rubber and nano-silica particles into the epoxy matrix.

Composites in engineering structures, in general, are subjected to spectrum fatigue loads. Hence, fatigue studies under spectrum or service load sequence are more appropriate in development of new materials. In continuation of constant amplitude and block load fatigue studies [24,25], the main aim of this investigation was to study the experimental fatigue behavior of a GFRP hybrid composite under a standard spectrum load sequence. An attempt was also made to predict the fatigue life of the GFRP hybrid composite under the same spectrum load sequence by constructing a constant life diagram and using a simple linear damage accumulation model to compare with the experimental results.

II. LITERATURER SURVEY

The materials used and the processing employed to manufacture GFRP composites are briefly explained in this section. However, a detailed description of the materials and processing can be found in [24]. The epoxy resin used was LY556 from Huntsman, which is a diglycidyl ether of bisphenol A (DGEBA) resin. The silica (SiO₂) nanoparticles were obtained as a colloidal silica sol with a concentration of 40 wt.% in LY556 from Nanoresins. The reactive liquid rubber was a carboxyl-terminated butadiene-acrylonitrile (CTBN) rubber, obtained from Nanoresins as a 40 wt.% CTBN-LY556 epoxy adduct. The curing agent was an accelerated methylhexahydrophthalic acid anhydride, HE600 from Nanoresins. The E-glass fiber cloth was a non-crimp-fabric (NCF) with an areal weight of 450 g/m².

The required quantity of the neat epoxy resin, the calculated quantities of silica nanoparticle-epoxy resin and CTBN-epoxy adduct, to give 10 wt.% of nano-silica and 9 wt.% of CTBN rubber in the final resin, were all individually weighed and degassed at 50°C and 0.1 atm. The resins were mixed together and a stoichiometric amount of curing agent was added, stirred and degassed again.

The resin mixture was used to prepare GFRP composite laminates by the ‘Resin Infusion under Flexible Tooling’ (RIFT) technique [26]. Glass fiber fabric pieces, about 330 mm square, were cut and laid up in a quasi-isotropic sequence [(+45/-45/0/90)_s]₂

with a fluid distribution mesh. The resin mixture was infused into the glass-cloth lay-up at 50°C and 0.1 atm., then cured at 100°C for 2 hours and post-cured at 150°C for 10 hours, maintaining the vacuum throughout the curing cycle. The resulting 2.5-2.7 mm thick GFRP composite laminates had a fiber volume fraction of about 57%. Two types of composites were prepared following the above procedure viz. GFRP with neat epoxy matrix (GFRP-neat) and GFRP with hybrid epoxy matrix (GFRP-hybrid) containing 9 wt.% of rubber microparticles and 10 wt.% of silica nanoparticles.

III. EXPERIMENTAL PROCEDURES

An atomic force microscope (AFM) was used to characterize the microstructure of the epoxy polymers, by scanning an ultramicrotomed surface. The AFM phase images of the hybrid modified bulk epoxy polymer are shown in [24]. The rubber particles were evenly distributed and had an average diameter of about 0.5 to 1 μm . The silica particles of about 20 nm in diameter were somewhat agglomerated to give a 'necklace-type' structure with an average width of about 1 μm .

Both the tensile and compressive properties of the composites were determined. Five replicate tests were conducted for both materials for each type of test and the average values were obtained. The tensile properties were determined according to ASTM D3039 test standard [27] specifications. Specimens about 250 mm long with a constant rectangular cross section (25 mm x 2.7 mm) were cut from the laminate, and end-tabs attached. The tensile tests were performed using a 100 kN computer controlled screw-driven test machine with a constant crosshead speed of 1 mm/min. The tensile properties determined are shown in Table 1. The GFRP-hybrid composite was observed to exhibit a higher tensile strength, by about 6%, but a lower tensile modulus, by about 9%, when compared to the GFRP-neat composite.

The compression tests were performed as per the ASTM D3410 test standard [28] specifications. Specimens about 12.5 mm wide and 140 mm long were cut from the laminate and prepared (without end tabs). Compression tests were conducted using a 50 kN servo-hydraulic test machine using an IITRI test fixture. Back-to-back strain gages were bonded to the

specimen surface in the loading direction, and the average modulus was obtained from data of both gages. The compressive properties of both GFRP composites determined are shown in Table 1. Unlike the tensile properties, the compressive properties of the composite were observed to be almost unaffected by the addition of the rubber and silica particles to the epoxy matrix. These tensile and compressive properties of the composites were employed to construct constant life diagrams (CLD) for fatigue life prediction under spectrum loads.

Fatigue Testing

Spectrum fatigue

Fatigue tests on both the GFRP-neat and GFRP-hybrid composites were conducted under spectrum loads. The spectrum load sequence used was a wind turbine load sequence, WISPERX [29, 30], as shown in. This is a modified version of the WISPER load spectrum, which is a standardized variable-amplitude test load sequence developed for the fatigue analysis of materials for wind turbine blades. This particular load sequence was considered in the present investigation since GFRP composites are used in the construction of large wind turbine blades. In, the normalized stress is plotted against the peak/trough points of the load sequence. One block of this load sequence consists of 25,663 reversals at 64 different stress levels. The stress sequence for experiments and fatigue life prediction was obtained by multiplying all the peak/trough points in the entire block with a constant reference stress, σ_{ref} .

Spectrum fatigue tests with various reference stress levels were conducted on both GFRP composites. The geometry and dimensions of the test specimens employed for the spectrum fatigue tests are shown in. Tests were conducted in a computer controlled 50 kN servo-hydraulic test machine. For any given reference stress, the number of load blocks required to fail the test specimen, $N_{\text{b-expt}}$, was determined. Whenever a specimen failed in-between a full block, it was rounded-off to the nearest complete block number.

The stiffness variation of the specimens subjected to spectrum fatigue loads was determined during the tests as a function of the number of applied load blocks. One additional load cycle with $\sigma_{\text{max}} = 0.5 \sigma_{\text{ref}}$ and stress ratio $R = \sigma_{\text{min}} / \sigma_{\text{max}} = 0$ was introduced at the beginning of the load block sequence, and the load versus displacement data for

this one complete load cycle was obtained and analyzed. For the purpose of comparison, the normalized stiffness of the specimen was defined as the ratio of measured stiffness at the end of any given load block to the initial stiffness (obtained before application of the first spectrum load block). For one particular test with $\sigma_{ref} = 225$ MPa, the specimens were dismantled at the end of application of 3 blocks of loading and photographs showing matrix cracks were obtained, as explained in [24].

Constant amplitude (CA) fatigue

In order to predict the fatigue life under the WISPERX spectrum load sequence, constant amplitude fatigue data was generated at various stress ratios. The fatigue test specimens, as shown in, were prepared from the GFRP composite laminates. The CA fatigue tests were performed as per the ASTM D3479M-96 test standard specifications [31], using a 25 kN / 50 kN computer-controlled servo-hydraulic test machine. The tests were conducted with a sinusoidal waveform at a frequency, $\nu = 1$ to 3 Hz. The test frequency was kept below 3 Hz to prevent thermal effects leading to reduced fatigue lives [32-34]. Tests were performed at various stress ratios to include all three regions of loading cases, viz., tension-tension (T-T) fatigue at $R = 0.1, 0.3, 0.5$ and 0.7 , tension-compression (T-C) fatigue at $R = -1$ and -4 and compression-compression (C-C) fatigue at $R = 10$.

IV. RESULTS AND DISCUSSIONS

The experimental spectrum fatigue lives determined for both the GFRP-neat and GFRP-hybrid composites under the WISPERX load sequence at various reference stresses is shown in Fig. 4. The fatigue life was increased with reduced reference stress in both GFRP composites, a similar trend observed by Philippidis and Vassilopoulos [35] in GFRP under MWISPERX load spectra. However, it may be clearly seen that, for a given reference stress, the GFRP-hybrid composite exhibits an enhanced fatigue life compared to the GFRP-neat composite. The fatigue life was observed to increase by about four to five times over entire range of reference stress levels investigated.

The variation of the normalized stiffness with the number of spectrum load blocks, evaluated for the fatigue test with $\sigma_{ref} = 225$ MPa, for both GFRP

composites is shown in Fig. 5. In general, both the GFRP-neat and GFRP-hybrid composites exhibit a typical stiffness reduction trend as observed in FRP composites [36-39]. The three regions of the stiffness reduction curve are clearly identifiable. It may be noted that the stiffness reductions in region I and region II are quite steep and significant in the GFRP-neat composite when compared to the GFRP-hybrid composite.

Photographs of the matrix cracks observed on the surface of the top $+45^\circ$ layers of the composites subjected to three complete load blocks of the WISPERX spectrum load sequence with $\sigma_{ref} = 225$ MPa are shown in Fig. 6. Gagel et al. [40, 41] have also observed the initiation and growth of such matrix cracks under cyclic fatigue loads in a GFRP composite. As observed in our earlier constant amplitude fatigue studies [24], the GFRP-neat composite exhibited more severe cracking than the GFRP-hybrid composite. The crack density, expressed as the number of cracks per unit length [24] was about 1.15/mm in the GFRP-neat and 0.58/mm in the GFRP-hybrid composite. Thus, suppressed matrix cracking was clearly observed in GFRP-hybrid composite under the WISPERX load sequence.

The progressive fatigue damage accumulation leading to final failure, under cyclic loads in polymer composites has been well documented [36, 38, 39]. The complete damage progress has been observed, involving (i) initiation and growth of matrix cracks, (ii) initiation of disbonds and delaminations due to coalescence of primary and secondary matrix cracks, and (iii) subsequent growth of cracks / delaminations to lead to final failure. In an earlier investigation [42] we have observed that the fatigue crack growth rate of the hybrid bulk epoxy (containing both micron-rubber and nano-silica particles) is over an order of magnitude lower than that of the neat epoxy. Further, we have observed that the use of such a hybrid matrix in GFRP enhances the constant amplitude fatigue life due to suppressed matrix cracking, delayed initiation of delamination and reduced crack / delamination growth rate [24]. Similar mechanisms being operative and contributing to improved fatigue life under block loads have also been observed by the authors [25].

The stiffness loss behavior shown in Fig. 5 is an indication of the underlying mechanisms being operative in the composite material [24, 25]. The

stiffness loss in stage I and stage II results primarily from matrix cracking [24, 36, 39, 40]. Once the matrix crack density saturates and attains the characteristic damage state (CDS) [24, 36, 39], the disbonds and delaminations created due to the coalescence of primary and secondary matrix cracks grow, and this leads to a further loss in stiffness. The present results show that when both composites are subjected to same number of spectrum load blocks, the crack density is lower in the GFRP-hybrid compared to the GFRP-neat composite (see Fig. 6). Thus, the stiffness loss curves shown in Fig. 5 clearly indicate the underlying mechanisms, i.e. suppressed matrix cracking, delayed initiation of delamination, and reduced crack / delamination growth rate [42], for improved spectrum fatigue behavior in the GFRP-hybrid composite.

It may be noted that the fatigue life enhancement is about eight to ten times under constant amplitude loads [24] whereas, it is reduced to about four to five times under WISPERX load sequence as observed in the present investigation. Load interaction effects in composites could alter the fatigue lives of composites significantly. Further detailed investigations with respect to load interaction effects in hybrid composite may provide some insight into such modifications in fatigue life enhancement factor.

Fatigue Life Prediction

The fatigue life of the GFRP-neat and GFRP-hybrid composites under the WISPERX load sequence was predicted and compared with the experimental results. Post et al. [43] have recently reviewed the modelling and prediction of fatigue life under spectrum loads in composites. The general fatigue life prediction procedure involves [43, 44] (i) rainflow counting of fatigue cycles in the spectrum load sequence [45] (ii) determination of cycles to failure, N_f , for each of the counted load cycles using a constant life diagram (CLD) constructed from the stress versus number of cycles (S-N) data, (iii) calculation of the damage fraction for each of the counted load cycles, and (iv) determination of the total fatigue damage per load block by summation of the damage fraction with or without load interaction effects [43]. The material is assumed to fail when the total damage fraction reaches 1.0. A flow chart showing the procedure employed in this investigation

for fatigue life estimation under the WISPERX spectrum load sequence is shown in Fig. 7.

As a pre-requisite for fatigue life prediction under spectrum loads, the constant amplitude fatigue behavior (S-N curves) of the GFRP composites at various stress ratios was determined, to obtain the fatigue properties required for construction of the CLD. The stress-controlled, constant-amplitude, T-T, T-C and C-C fatigue test results at various stress ratios, obtained for both the GFRP-neat and GFRP-hybrid composites are shown in Fig. 8(a) and 8(b) respectively. For the sake of comparison, typical S-N curves from each region at the same stress ratio are shown in Fig. 8(c). It may be seen that, as observed earlier [24], for any given stress ratio, over the entire range of stress levels investigated, the addition of particles into the epoxy matrix enhances the fatigue life of the GFRP composite significantly.

The predictions also suggest an improvement in fatigue life of GFRP-hybrid composite by about three times. The predicted enhancement factor is lower than the experimental observation of about four to five times. This under-prediction may be due to use of the simple linear damage accumulation model. Consideration of load interaction effects [43] in the damage accumulation model may improve further the accuracy of the predictions.

Load interaction effects in fatigue of composites have been studied by several authors. Gamstedt et al. [51] showed that in a high-low load sequence, initial high load creates matrix cracks and subsequently it is easy to initiate delamination growth which leads to lesser life when compared to low-high load sequence. Paepgen et al. [52] showed that the transition from low to high are most damaging and that the number of transition determine which block loading is most devastating. In the present investigation, it is clearly observed in the experiments that the suppressed matrix cracking due to addition of micron-rubber and nano-silica particles lead to improved fatigue life under spectrum loads. Since matrix cracking is one of the major phenomena influencing load interaction effects [51], we believe that further studies are necessary to investigate such effects which lead to alter the enhancement factors between constant amplitude and spectrum fatigue lives. However, the present investigation clearly shows that the fatigue life of a GFRP composite under the WISPERX load sequence is enhanced by four to five times due to the

incorporation of micron-rubber and nano-silica particles in the epoxy matrix. Also, the simple linear damage summation model appears to provide a reasonably good correlation between the experimental and the predicted lives.

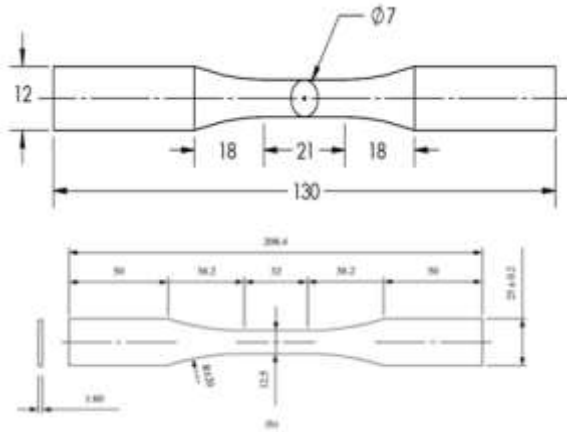
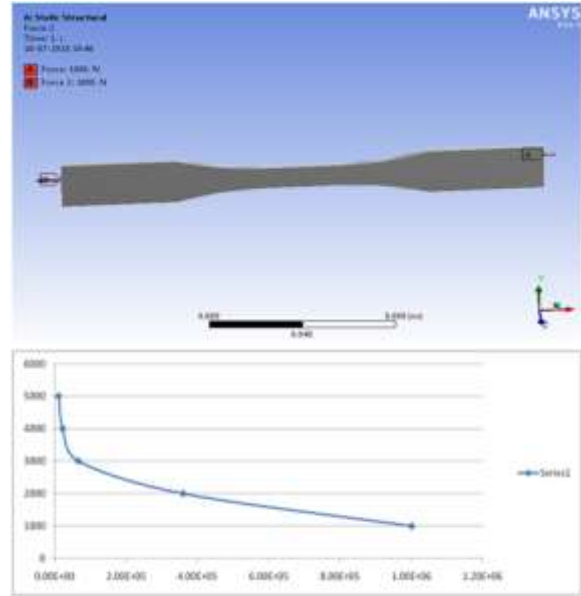
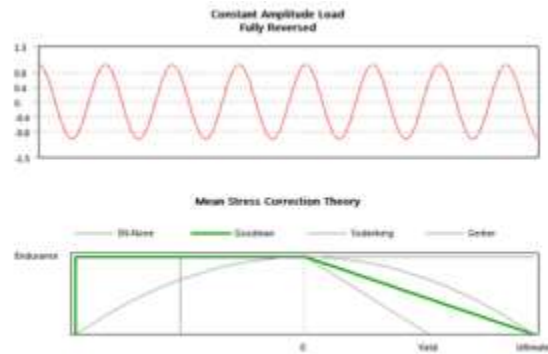
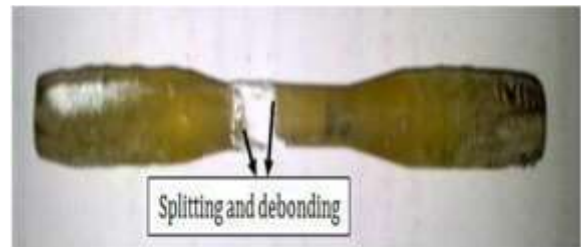


Figure 1. Size and geometry of the (a) tensile and (b) fatigue specimens. The dimensions are in mm.



Test Procedure

Density. The theoretical density (pct) of composite materials in terms of weight fractions of different constituents can easily be obtained using the following equation .



where W and ρ represent the weight fraction and density respectively. The suffixes f and m stand for the fiber and matrix respectively. Since the composites under this investigation consist of three components namely matrix, fiber and particulate filler, the expression for the density is modified as where the suffix p stands for the particulate filler. The actual density (pca) of the composite is however,

determined experimentally by simple water immersion technique with the use of the sample of 30 mm× 12 mm× 6 mm dimensions. Four samples of same composite are tested and average value is presented. The volume fraction of voids (Vv) in the composites is calculated using the following equation:

Micro-hardness. Hardness is the resistance of a material to deformation, indentation or scratching. The basic goal of hardness testing is to quantify the resistance of a material to plastic deformation. The indentation value has high importance for technical applications which reflects the resistance to deformation which is a complex property and related to modulus, strength, elasticity, plasticity and dimensional stability of a material. Hardness is generally classified into three different categories with respect to the depth of indentation (d). When the depth of indentation ranges between 1-50 µm, it is termed as micro hardness. The test methods commonly used for expressing the relationship between hardness and the size of impression are Brinell, Vicker's and Rockwell hardness tests.

Measurement of hardness. Hardness values offer a comparative measurement of a material's resistance to plastic deformation from a standard source, as different hardness techniques have different scales. The Vickers hardness test is very popular among researchers since it is easier to perform compared to other hardness tests and also the hardness calculations are independent of the size of the indenter and load applied. In this study, Vicker's hardness test setup is used to find out the microhardness values of different composites. Microhardness testing is carried out in a UHL micro hardness tester (Model - VMHT MOT, Sl. No. 1002001, Technische Mikroskopie) with a Vickers diamond indenter. The specimen used for the test is of the dimension of 16 mm× 12 mm× 6 mm. Fig. 3 shows the Vicker's microhardness test setup. The dwell time is kept at 10s while the speed of indentation is set at 50µm/s and indentation load of 1000 kgf. The two diagonals (d1 and d2) of the indentation left in the surface of the material after removal of the load are measured using a microscope and their average (d) calculated.

VI. CONCLUSION

The following conclusions may be drawn based on the results obtained in this investigation: Under the WISPERX spectrum load sequence, the fatigue life of the GFRP composite with the hybrid epoxy matrix containing 9 wt. % CTBN rubber microparticles and 10 wt. % silica nanoparticles is about four to five times higher than that of the GFRP composite with the neat epoxy matrix. The suppressed matrix cracking and reduced crack / delamination growth rate contribute to the enhanced fatigue life. The constant amplitude fatigue life of the GFRP-hybrid composite is always higher than that of the GFRP-neat composite at all stress ratios investigated in the tension-tension, tension-compression and compression-compression regions. The predicted fatigue lives under the WISPERX spectrum load sequence correlate reasonably well with the experimental observations for both GFRP-neat and GFRP-hybrid composites. Predictions suggest an improvement in the fatigue life of about three times in the GFRP-hybrid composite. Consideration of load interaction effects in the damage accumulation model may improve the prediction accuracy further.

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