Improved variable-amplitude fatigue behavior of a glass-fiber reinforced hybrid-toughened epoxy composite

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ABSTRACT

A thermosetting epoxy polymer was hybrid-modified by the addition of 9 wt.% of rubber micro-particles and 10 wt.% of silica nano-particles. Glass-fiber reinforced plastic (GFRP) composite laminates employing the unmodified epoxy matrix (GFRP-neat), and the hybrid epoxy matrix (GFRP-hybrid), were produced by a resin infusion technique. The experimental fatigue lives of both GFRP composites under three different variable amplitude load sequences, namely (i) a three-step increasing block, (ii) a three-step decreasing block and, (iii) a random block load sequence derived from a three-step load block, were determined. The fatigue life of the GFRP-hybrid composite was higher than that of the GFRP-neat composite under all the three load sequence blocks investigated, by about x2.6 to x4.0 times. The saturated matrix crack density and the stiffness reduction rate were both lower in the GFRP-hybrid composite compared to the GFRP-neat composite material. The suppressed matrix cracking and reduced delamination growth rates measured in the hybrid-modified epoxy matrix enhanced the fatigue life of the corresponding GFRP-hybrid composite. Using the constant amplitude fatigue data generated at various stress ratios, the fatigue lives under these variable amplitude load sequence blocks were predicted using empirical models. The predicted fatigue lives, although non-conservative, were in reasonably agreement with the experimental results.

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INTRODUCTION

Fiber reinforced plastic (FRP) composites, due to their high specific strength and stiffness, are widely used in various structural applications such as ship hulls, airframes, wind turbines etc. Such structural components experience various types of constant and variable amplitude fatigue loads in service. Hence, composite materials, in addition to their good static properties, need to possess high fatigue-resistance for these applications.

The commonly used FRPs in structural applications consist of continuous fibers of carbon or glass, reinforced in a thermosetting epoxy polymer matrix, frequently with an epoxy resin being used as the matrix material. However, whilst possessing many very good mechanical and thermal properties, epoxy polymers are relatively brittle and exhibit poor resistance to crack initiation and growth. This in turn, adversely affects the overall performance of the FRPs under static and fatigue loads. Thus, efforts have been made to improve the mechanical properties of composites through the incorporation of second-phase fillers in the epoxy polymeric matrix. The addition of dispersed rubber micro-particles has been shown to improve the fracture toughness and fatigue behavior of carbon and glass fiber composites significantly [1,2]. More recently, polymeric nanocomposites have been studied, where at least one of the dimensions of the filler is of the order of nanometers. These have been used as the matrices to manufacture a new class of FRPs, which have shown some remarkable improvements in mechanical properties. Various types of nano fillers such as carbon nanotubes and nanofibers, nanoclay, and SiO_2 and SiC nanoparticles [3-9] have been employed in the epoxy resin matrix to improve the fatigue and fracture properties of the epoxy polymeric matrices and the resulting fiber composite materials.

Most recently, hybrid composites which contain two different types of fillers in the epoxy have been shown to further improve the mechanical properties [10,11]. Recently, the authors have shown that the addition of both rubber micro-particles and silica nanoparticles enhance the constant amplitude fatigue life of a thermosetting epoxy and also glass-fiber reinforced plastic composites (GFRPs) employing such a hybrid-modified epoxy matrix [12]. The main aim of the present study was to investigate further the fatigue behavior of a GFRP composite using such a hybrid-toughened epoxy matrix when subjected to a variable amplitude load sequence, and to predict the fatigue life using empirical models.

EXPERIMENTAL

Materials and Processing

The materials were based upon a single-component hot-cured epoxy formulation. The epoxy resin was a standard diglycidyl ether of bis-phenol A (DGEBA), LY556. The silica nano-particles were obtained at a concentration of 40 wt.% in a DGEBA epoxy resin. The reactive liquid carboxyl-terminated butadiene-acrylonitrile (CTBN) rubber (which give rises to micrometer-sized particles upon curing) was obtained as CTBN-epoxy adduct with a rubber concentration of 40 wt.% in DGEBA epoxy resin. The curing agent was an accelerated methylhexahydrophthalic acid anhydride, Albidur HE 600. The E-glass fiber was a two-layered, non-crimp-fabric arranged in $\pm 45^\circ$ orientation.

The required quantity of the neat DGEBA epoxy resin was weighed and degassed at -1 atm. and 50 °C. The calculated quantities of CTBN-epoxy adduct and silica nanoparticle epoxy-resin mix, to give the required level of 9 wt.% of CTBN rubber and 10 wt.% added silica respectively in the final resin, were also individually weighed and degassed. All the resins were then mixed together, a stoichiometric amount of curing agent added, stirred and degassed once again.

The GFRP composite panels were manufactured by a resin infusion under flexible tooling (RIFT) technique [13]. Glass fiber cloth pieces of about 330 mm square, were cut and laid up in a quasi-isotropic (QI) sequence $[(+45/-45/0/90)_s]_2$. The prepared resin mixture was then infused into the glass-cloth lay-up at a temperature of 50 °C and -1 atm. Once infusion was complete, the composite laminate was cured at 100 °C for 2 hours, and then post-cured at 150 °C for 10 hours. In this way, two different laminates were produced: (i) GFRP with the neat (i.e. unmodified) epoxy matrix (denoted as GFRP-neat), and (ii) GFRP with the hybrid-modified epoxy matrix (denoted as GFRP-hybrid). The fiber volume fraction in the cured composites was about 0.57.

The atomic force microscopic (AFM) phase image of the hybrid epoxy polymer [12] is shown in Fig. 1. The rubber particles of about 0.5 to 1 μm diameter are evenly distributed. However, 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 . The tensile properties [12] of the GFRP composites are shown in Table 1. The ultimate tensile strength was observed to increase slightly, by about 4 %, and tensile modulus decreased by about 9 % due to the hybrid epoxy matrix used in the GFRP composite.

Fatigue Testing

The constant rectangular cross-sectioned fatigue test specimens, shown schematically in Fig. 2, were machined and prepared from the GFRP composite laminates. The fatigue tests were conducted using 25 kN / 50 kN computer-controlled servo-hydraulic test machines. Sinusoidal waveform at a low frequency□ of 1 to 3 Hz was used to avoid thermal effects during fatigue test [14,15]. Constant amplitude fatigue tests were conducted as per ASTM D3479 [16] test standard specifications.

Three different types of variable amplitude load sequences were employed in this investigation, namely: (i) a three-step increasing block (IB), (ii) a

three-step decreasing block (DB) and, (iii) a random block (RB) load sequence which was derived from the IB block load sequence. A schematic diagram and further details of all these load sequences are shown in Fig. 3 and Table 2, respectively. In Fig. 3, the stress is expressed as a normalized stress so that, for any given reference stress, the load block can be converted to a stress block by multiplying all the peak-trough points by the reference stress value.

The IB load sequence consists of three steps of constant amplitude loads arranged in an increasing amplitude order. Each of these steps consists of different number of load cycles at different stress ratios but having same mean stress (Fig. 3 (a) and Table 2). Similar types of three-step block load sequences at various stress ratios have been designed and employed earlier for testing and analysis of GFRP composites [17,18]. The DB load sequence (Fig. 3 (b)) is similar to IB load sequence in all respects, except that the load sequence order has been reversed; i.e. the loads are arranged in decreasing amplitude order. A random load sequence block was derived from the IB load block by randomly arranging all the peaks and troughs alternatively to yield the RB load sequence, as shown in Fig. 3 (c).

The variable amplitude load sequence block was repeatedly applied to the GFRP composite test specimens until failure of the test specimen occurred. For the ease of experimental investigation, a reference stress of 225 MPa was employed to obtain the stress sequence of these blocks. Five repeat tests were conducted for both the GFRP-neat and GFRP-hybrid composites and the average number of load blocks required to fail, N_{b-expt} , was obtained under each of these variable amplitude load sequences. The variable amplitude fatigue lives determined either by experiment, or by prediction, was rounded to the nearest lower integer.

During one of the fatigue tests conducted under each of the variable amplitude tests, the stiffness of the specimen was monitored until failure occurred. The stiffness was determined from the load-displacement data

obtained at regular intervals during the fatigue test. The matrix crack density in the test specimen was determined from the photographed image of the test specimen before final failure. The detailed procedure for the determination of stiffness and further details on measurement of matrix crack density can be found elsewhere [12].

RESULTS AND DISCUSSION

Variable Amplitude Fatigue Behavior

The variable amplitude fatigue life are shown in Fig. 4, expressed as the average number of load sequence blocks required to fail a test specimen, determined under IB, DB and RB load sequences for both the GFRP-neat and GFRP-hybrid composites. As was observed in earlier studies [12] for the constant amplitude fatigue behavior, the fatigue life of the GFRP-hybrid composite was higher than that of the GFRP-neat composite under all the load sequence blocks investigated. The fatigue life of the GFRP-hybrid composite was enhanced by a factor of about x3.9, x2.6 and x4.0 under IB, DB and RB load block sequences, respectively, as is shown in Table 3.

The variation of normalized stiffness (i.e. the ratio of the stiffness determined at any given load block to the stiffness measured during the first load block) with applied load block, evaluated for the GFRP-neat and GFRP-hybrid composites under IB, DB and RB load blocks, is shown in Fig. 5. In general, both the GFRP composites exhibit a stiffness reduction trend as observed in FRP composites [19-21]. Indeed, 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 were quite steep and significant in the GFRP-neat when compared to GFRP-hybrid composite, under all three types of variable amplitude load sequence blocks.

Because of the translucent nature of the GFRP test specimens, the matrix cracks were readily visible in the test specimen during fatigue testing. Indeed, matrix cracks were observed to initiate and grow. A typical matrix cracking pattern in the off-axis plies observed in the test specimen before failure, under all the variable amplitude load sequences is shown in Fig. 6, for both the GFRP-neat and the GFRP-hybrid composites. The measured average matrix crack density (i.e. the number of cracks per unit length) is shown in Fig. 7. It is clear from Fig. 6 and Fig. 7 that the GFRP-neat composite is subject to the initiation and growth of matrix cracks to a far greater extent than the GFRP-hybrid composite under all the load sequences investigated.

The initiation and growth of interlaminar delaminations, particularly from the free edges of the test specimens were observed in the test specimens during the later stages of fatigue cycling. Such free edge delaminations have previously been observed in composite fatigue tests [22]. The further growth of such delaminations under fatigue loading leads to final failure of the specimen.

The sequence of fatigue damage development leading to final failure and hence defining the fatigue life in a quasi-isotropic (QI) lay-up GFRP composite has been studied in detail [12,23-26]. Initially, matrix cracks develop in the off-axis plies due to the applied cyclic-fatigue loads. The density of these matrix cracks increase and the cracks propagate with further continued application of load cycles, resulting in a continuous decrease in the global stiffness of the composite. The matrix cracking process continues until it attains a characteristic damage state (CDS), from which point the formation of secondary cracks in the epoxy matrix leads to the initiation of interlaminar delaminations. The further growth of such delaminations under fatigue leads to final failure of the test specimen.

The results obtained in the present investigation suggest a similar trend in fatigue damage development leading to final failure in both the GFRP-neat and GFRP-hybrid composites. However, in the hybrid-modified matrix, the degree of

matrix cracking is suppressed and the crack density is lower in the GFRP-hybrid matrix. These factors result in a lower damage rate, as observed in the reduced stiffness loss in Fig. 5, for the GFRP-hybrid compared to the GFRP-neat composite.

It has been shown that the fatigue crack growth rate is significantly lower in the hybrid-modified epoxy polymers [27-29]. This is undoubtedly responsible for the stiffness reduction rate being far lower in the GFRP-hybrid composite in region II, compared to the GFRP-neat composite, see Fig. 5. The accumulation and growth of all the matrix cracking, followed by the delamination, damage leads to final fatigue failure of the composites. However, of course, the GFRP-hybrid composite exhibits an improved fatigue life compared to that of the GFRP-neat composite, for the reasons discussed above.

It may also be observed from Fig. 4 and Table 3 that there is a significant load sequence effect in both the GFRP-neat and the GFRP-hybrid composites. In the GFRP-neat composite, the fatigue life under IB load sequence is approximately 119 blocks and reversing the load sequence (i.e. going from the IB to the DB load sequence) appears to increase the fatigue life to 202 blocks. Under the RB load sequence, which was derived from the IB sequence and contains the same number of peak-trough load points but arranged randomly, then the GFRP-neat composite exhibits a fatigue life of 181 blocks, which is not significantly different to the result from the DB load sequence. A load sequence effect is also observed in the GFRP-hybrid composite, where the fatigue life is in the order $IB < DB < RB$, as is shown in Table 3.

It is of interest to note that, load sequence effects on the fatigue life of composites have been investigated by several authors [30-37]. Many studies have shown that a high-low sequence (DB type) lead to a lower fatigue life compared to a low-high (IB type) sequence [30]. In contrast, other investigations have revealed the opposite trend [31,32,36]. Further, several studies have also suggested that no load sequence effects exist in composites [33,34]. Clearly, a

further detailed investigation on the mechanisms of load interaction effects would assist in understanding the load sequence effects in GFRP-neat and GFRP-hybrid composites.

Fatigue Life Prediction

Numerous empirical and phenomenological models have been introduced over the last four decades for predicting the fatigue life of composite materials [37]. In the present investigation, three different empirical models were employed to predict the fatigue life of both the GFRP-neat and the GFRP-hybrid composites under all the variable amplitude load sequence blocks studied in the present work.

The Palmgren-Miner (PM) rule [38], which is a linear damage accumulation law, states that the cumulative fatigue damage is given by:

$$D = \sum_{i=1}^k \frac{n_i}{N_i} = 1 \quad (1)$$

where, D = total fatigue damage, n_i = number of load cycles in the i^{th} step and the N_i is the number of load cycles to failure at the i^{th} step stress level.

The Hashin-Rotem (HR) model [39] is a non-linear damage accumulation law which states that:

$$D_i = D^{\frac{\log \sigma_{p,i}}{\log \sigma_{p,i-1}}} + \frac{n_i}{N_i} \quad (2)$$

where, $\sigma_{p,i}$ = maximum stress in the i^{th} load step, n_i = number of load cycles in the i^{th} step and, N_i = number of load cycles to failure at the i^{th} step stress level.

The Broutman-Sahu (BS) rule [40] is a residual strength based model and states that:

$$S_r = S_u - \sum_i (S_u - \sigma_{p,i}) \frac{n_i}{N_i} \quad (3)$$

where, S_r = residual strength, S_u = ultimate strength $\sigma_{p,i}$ = maximum stress in the i^{th} load step.

For predicting the fatigue life using all the above models, it is necessary to estimate the number of cycles required to fail, N_i , for a given load amplitude and the stress ratio combinations implicit in the IB, DB and RB load blocks. Hence, constant amplitude fatigue tests were conducted at various stress ratios for both GFRP composites. The stress-life curves determined for GFRP composites at stress ratios ranging from $R=0.1$ to $R=0.7$ are shown in Fig. 8. As observed in previous investigations [14,41], the fatigue life was observed to increase with an increasing stress ratio, R . This was the case for both the GFRP-neat and the GFRP-hybrid composites. However, for any given value of R , the GFRP-hybrid composite exhibited a higher fatigue life compared to that of the GFRP-neat composite. This enhanced fatigue life in the GFRP-hybrid composite was observed over the entire range of stress levels investigated. This is in agreement with previous work where fiber composites with modified matrices have been shown to exhibit higher constant amplitude fatigue lives when compared to their respective neat-matrix composites [2-4,12,42].

The experimental constant amplitude fatigue data of GFRP composites shown in Fig. 8 were fitted to Basquin's law [43]:

$$\sigma_{\max} = \sigma_f' (N_f)^b \quad (4)$$

where, σ_f' and b are the fatigue strength coefficient (FSC) and fatigue strength exponent (FSE) respectively. The fatigue parameters FSC and FSE were determined for the GFRP composites at various stress ratios, and are shown in Table 4. These fatigue parameters were then employed in the prediction of the fatigue life when the fiber composites were subjected to the variable amplitude load blocks.

For the estimation of the fatigue life under IB and DB load sequences, the value of N_i for any given load amplitude was obtained from eqn. (4). The cumulative fatigue damage was determined from equations (1) and (2) respectively. The specimen was assumed to fail when the damage fraction, $D = 1.0$.

The fatigue life estimation under the RB load sequence was estimated using eqn (3). However, the estimation of the value of N_i for a given load amplitude in this case requires an interpolation procedure. The general procedure for life prediction under spectrum load sequence can be found elsewhere [37]. It involves the (i) 'rainflow counting' of fatigue cycles in the spectrum, (ii) estimation of N_i and fatigue damage fraction for each of the counted load cycles, and (iii) summation of the damage fraction as per an empirical law (i.e. eqn. (3)). Since, each and every load cycle in Fig. 3 (c) is of different amplitude and stress ratio, the estimation of N_i for any rainflow counted cycle requires the use of a constant life diagram (CLD) [37]. The fatigue properties from Table 4 were employed to construct the CLD for both GFRP composites. The value of N_i was estimated from this CLD using a piece-wise linear interpolation technique [37].

The predicted fatigue life, N_{b-pred} (i.e. the number of load blocks required to fail the test specimen) under all the variable amplitude load blocks using three models described above, is shown in Table 3. It may be observed that the fatigue lives predicted by these models, although non-conservative, are in reasonable agreement with the experimental results. The ratios of the predicted to experimental fatigue lives, $N_{b-pred} / N_{b-expt.}$, vary from 0.56 to 0.96.

Now, several investigators have attempted to predict the fatigue life of GFRP composites under different types of variable amplitude fatigue loads [e.g. 18,37,44]. They have all observed a far wide difference in the predicted and experimental results than recorded in the present study. For example, Epaarachchi [18] observed significant values from unity in the N_{b-pred} / N_{b-expt} ratio

for GFRP composites under various block loads, i.e. values of 0.5 to 66.6 were recorded for a two step block load sequence, and 3.7 to 14.5 for a three-step block load sequence. Considering the reported wide scatter in predicted fatigue lives under block loads, then in the present investigation there is a relatively good agreement between the predicted and experimental results from all three models, for both the GFRP-neat and the GFRP-hybrid composites,.

CONCLUSIONS

The following conclusions may be drawn based on the results obtained in the present investigation:

- (i). The fatigue life of the GFRP-hybrid composites is significantly higher than that of the GFRP-neat composites under all the IB, DB and RB variable amplitude load sequence blocks employed. Indeed, the fatigue lives of the GFRP-hybrid composites are enhanced by a factor of x2.6 to x4.0. Suppressed matrix cracking and reduced delamination growth rate in the hybrid-modified epoxy matrix appear to be responsible for this increase in the fatigue lives of the GFRP-hybrid composites.
- (ii). The fatigue life under IB, DB and RB load sequence blocks has been predicted by three empirical models. Namely, the (i) Palmgren-Miner (PM), (ii) Hashin-Rotem (HR), and (iii) Broutman-Sahu (BS) models have been used. Although non-conservative, the results from these models are in good agreement with the experimental results, with the ratio of predicted to experimental fatigue life, $N_{b-pred.} / N_{b-expt.}$, varying from 0.56 to 0.96.

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Figure 1. *Tapping mode atomic force microscopic (AFM) phase image of the hybrid-modified bulk epoxy polymer [12]*

Figure 2. A schematic diagram showing the dimensions of the fatigue test specimen.

(a) three-step increasing block (IB)

(b) three-step decreasing block (DB)

(c) random block (RB) load sequence

Figure 3. A schematic of variable amplitude load sequence blocks

Figure 4. *Experimental fatigue lives determined for GFRP composites under variable amplitude fatigue load sequence blocks*

Figure 5. The variation of normalized stiffness with load blocks in GFRP composites

IB load sequence (a) GFRP-neat and, (b) GFRP-hybrid

DB load sequence (c) GFRP-neat and, (d) GFRP-hybrid

RB load sequence (e) GFRP-neat and, (f) GFRP-hybrid

Figure 6. The optical photographs showing the matrix cracks in the test specimens

Figure 7. Measured matrix crack densities in the composite test specimens

Figure 8. The constant-amplitude stress-life (S-N) curves determined at various stress ratios for GFRP composites.

Figure 9. Constant life diagram of GFRP composites

Table 1. Tensile properties of the composite materials investigated [12]

Material	Tensile properties	
	σ_{UTS} (MPa)	Modulus, E (GPa)
GFRP-neat	365 ± 13	17.5 ± 0.6
GFRP-hybrid	380 ± 11	15.9 ± 0.9

Table 2. Details of variable amplitude load sequences shown in Figure 3.

Step in the load block	Normalised stress ($\sigma_{app} / \sigma_{ref}$) [*]		Stress range ($\Delta\sigma$)	Mean stress (σ_{mean})	Stress ratio (R)	No. of load cycles (n_i)
	σ_{max}	σ_{min}	($\sigma_{max} - \sigma_{min}$)	($\sigma_{max} + \sigma_{min}$)/2	($\sigma_{min} / \sigma_{max}$)	
S1	1.000	0.100	0.900	0.55	0.1	5
S2	0.846	0.253	0.593	0.55	0.3	15
S3	0.733	0.366	0.367	0.55	0.5	25

^{*} σ_{app} = applied stress, σ_{ref} = reference stress

Table 3. *Experimental and predicted variable amplitude fatigue life of GFRP composites investigated*

Type of load block	Material	Expt. Fatigue life (N_{b-expt})	Enhancement factor N_{hybrid}/N_{neat}	Predicted fatigue life (N_{b-pred})					
				Palmgren-Miner		Hashin-Rotem		Broutman-Sahu	
				N_{pred}	N_{pred}/N_{expt}	N_{pred}	N_{pred}/N_{expt}	N_{pred}	N_{pred}/N_{expt}
IB	GFRP-neat	119 $\pm$ 14	-	115	0.96	115	0.96	114	0.96
	GFRP-hybrid	467 $\pm$ 33	3.9	423	0.90	418	0.89	402	0.86
DB	GFRP-neat	202 $\pm$ 19	-	115	0.56	114	0.56	113	0.56
	GFRP-hybrid	535 $\pm$ 45	2.6	423	0.79	418	0.78	402	0.75
RB	GFRP-neat	181 $\pm$ 16	-	151	0.83	149	0.82	145	0.80
	GFRP-hybrid	732 $\pm$ 34	4.0	532	0.72	527	0.72	503	0.69

Table 4. *Constant amplitude fatigue parameters determined at various stress ratios for GFRP composites investigated*

Stress ratio, R	Fatigue properties			
	GFRP-neat		GFRP-hybrid	
	FSC* (MPa)	FSE*	FSC* (MPa)	FSE*
0.1	469.68	-0.1135	532.23	-0.1092
0.3	573.98	-0.1114	548.78	-0.1013
0.5	580.76	-0.1021	549.16	-0.0916
0.7	547.77	-0.0856	556.67	-0.0811

* FSC = fatigue strength coefficient, FSE = fatigue strength exponent