TECHNICAL PAPER



Micromechanics Modeling and Prediction of Stiffness Degradation Behavior of a Fiber Reinforced Polymer Nanocomposite Under Block Amplitude Fatigue Loads

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Received: 15 October 2015/Accepted: 7 December 2015/Published online: 4 January 2016 © The Indian Institute of Metals - IIM 2016

Abstract The micro-mechanisms of fatigue damage initiation and growth in polymer composites lead to observable progressive degradation in global properties such as strength and stiffness. Thus monitoring stiffness degradation behavior of a composite will assist in evaluating the residual strength, stiffness and remaining fatigue life of the material. In the present investigation, the stiffness degradation behavior of a glass-fiber epoxy silica-nano-particle composite (GFRP nanocomposite) under a two step block load sequence was predicted from micro-mechanics based models. The stiffness of nanocomposite was determined from the properties of the constituent materials. To compare the predicted results, experiments were conducted on a GFRP nanocomposite. The stiffness of the specimen was monitored at regular intervals during the fatigue tests. The predicted stiffness degradation behavior of the nanocomposite under variable amplitude fatigue loads was observed to compare quite well with experiments.

Keywords Polymer composite · Fatigue · Stiffness degradation · Micro-mechanics modeling

1 Introduction

Fiber reinforced plastic (FRP) composites are widely used in various structural applications. FRP composites consist of continuous fibers reinforced in an epoxy polymer matrix

Ramesh Bojja rameshyld@nal.res.in material. Epoxy polymer is, in general, brittle and has lower strength than fibers. Hence, mechanical behaviour of the FRP composite is sensitive to the epoxy employed. Efforts have been made in recent times to improve the mechanical properties of FRP composites by incorporation of second phase fillers in the epoxy matrix. Addition of various types of micro and nano sized fillers such as silica particles, carbon nano tubes and various types of clays have all been shown to improve several specific properties of epoxies and FRPs [1, 2]. Recently, addition of rubber and silica nano particles to epoxy has been shown to improve the fatigue and fracture properties of FRPs significantly [3-6]. In addition, presence of silica nano particles has been shown to improve the tensile properties and does not affect the glass transition temperature of the composite [5, 6].

Structural composites experience fatigue loads in service which are of constant amplitude or variable-amplitude in nature. Application of fatigue load leads to progressive damage development and growth resulting in fatigue failure. The damage mechanism behind fatigue failure was observed to be an individual or combined effect of matrix cracks, disbonds, and delaminations etc., formed during loading [7]. Fatigue damage causes degradation of stiffness and strength of the material towards final failure. Prediction of fatigue life of composite is important in the design and safety of the structure.

A review of the various fatigue life prediction models was published by several authors [8, 9]. Of all the types of life prediction models, phenomenological models such as stiffness degradation and strength degradation models have attracted the interest of the engineering community since it is amenable for industrial structural health monitoring. Since stiffness is a non-destructive measurable quantity, stiffness based models are widely used for fatigue life

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estimations of FRPs [9]. However, limited studies are available on the application of such models to polymer nanocomposites [10]. Also, they are limited to constant amplitude fatigue loading and to a particular layup sequence. To the best knowledge of authors, no studies are available in the literature for the extension of stiffness degradation based life prediction models to variable or block amplitude loading conditions that simulate real service loads. In this study, the stiffness degradation behaviour of GFRP nanocomposite containing silica nano particles in the epoxy matrix under a two step block load sequence was predicted from micro-mechanics based models and compared with experimental observations.

2 Micromechanics Modeling

2.1 Modeling Procedure

A flowchart for micromechanics modeling procedure is shown in Fig. 1. It involves: (1) estimation of stiffness of the bulk epoxy modified with silica nano particles from the properties of neat epoxy and silica nano particles using Halpin–Tsai model [11, 12], (2) prediction of stiffness of GFRP unidirectional nanocomposite using rule of mixtures [13], (3) estimation of stiffness of GFRP nanocomposite laminate for the given layup sequence using classical laminate analysis approach [13], (4) generation of stress-life (*S*-*N*) data for GFRP nanocomposite at given stress ratios experimentally, (5) prediction of stiffness degradation under constant amplitude fatigue load from static stiffness and *S-N* data using Shokrieh's model [14], (6) rainflow counting of the fatigue cycles in the variable-amplitude sequence, (7) estimation of stiffness degradation for each cycle from constant-amplitude data, (8) check for the critical stiffness and determination of number of blocks to failure.

2.2 Stiffness Prediction

The modulus of an epoxy/particle composite can be estimated from Halpin–Tsai model [11, 12]. Halpin–Tsai equation for modulus estimation is expressed as a function of modulus of the matrix material and modulus of particle material. The equation is given by

$$E_c = \frac{1 + \xi \eta V_f}{1 - \eta V_f} E_m \tag{1}$$

where,

$$\eta = \frac{\left(\frac{E_f}{E_m} - 1\right)}{\left(\frac{E_f}{E_m} + \zeta\right)} \text{ and } \xi = 2w/t$$
(2)

and $E_{\rm m}$ is modulus of epoxy material, $E_{\rm f}$ is modulus of particle material, $V_{\rm f}$ is volume fraction of particles in the epoxy. ξ represents shape factor, for spherical particle geometry, ξ is 1. For a fiber reinforced composite with modified epoxy matrix, the stiffness of the composite based on rule of mixture [13] is given by



Fig. 1 Flow-chart for prediction of stiffness degradation behavior

$$E_{c,frp} = V_f E_f + V_m E_m \tag{3}$$

where, $E_{\rm m}$ is modulus of modified epoxy estimated from Halpin–Tsai equation, $E_{\rm f}$ is modulus of fibre, $V_{\rm f}$ is volume fraction of fibres and $V_{\rm m}$ is volume fraction of the modified epoxy. The stiffness of a multi directional FRP nanocomposite for any given layup sequence can be estimated from classical laminate analysis [13]. In this method, the stiffness of a composite laminate is estimated from the extensional stiffness matrix.

2.3 Stiffness Degradation

Shokrieh and Lessard [14] developed a semi empirical stiffness degradation model for uni-directional composite material for multi-axial loading conditions. For an arbitrary stress ratio, the residual stiffness is expressed as a function of number of cycles, static stiffness in the normalized form and is given by

$$E(n) = \left[1 - \left(\frac{\log(n) - \log(0.25)}{\log(N_f) - \log(0.25)}\right)^{\lambda}\right]^{\frac{1}{\gamma}} \left(E_s - \frac{\sigma}{\varepsilon_f}\right) + \frac{\sigma}{\varepsilon_f}$$
(4)

where, *n* is number of cycles, $N_{\rm f}$ is fatigue life at applied stress σ , $E_{\rm s}$ static stiffness, $\varepsilon_{\rm f}$ is static failure strain and γ , λ are curve fitting parameters. Since the equation is in normalized form, for a particular stress ratio (*R*), the curve fitting parameters remains constant for any applied stress level.

3 Experimental

3.1 Material

The epoxy resin was standard diglycidyl ether of bisphenol A (DGEBA), LY556. The silica (SiO₂) nano particles were obtained as a colloidal silica sol. with a concentration of 40 wt% in DGEBA epoxy resin. The curing agent was an accelerated methylhexahydrophthalic acid anhydride. The resins and the curing agent were individually weighed, degassed, and mixed together to get resin mixture containing 10 wt% silica nano particles. The silica particles are about 20 nm in diameter and are homogeneously distributed throughout the epoxy. Using non-crimp-fabric type E-glass fiber cloth, the GFRP composite laminates ($[(+45/-45/0/90)_s]_2$) were manufactured by resin-infusion technique. The detailed manufacturing procedure can be found in references [3-5]. The tensile properties of the composite laminates were as follows [5]: ultimate strength is about 382 MPa and elastic modulus is about 18.8 GPa.

3.2 Fatigue Testing

Constant amplitude fatigue data was generated at stress ratios, R = 0.1 and R = -1 under various stress levels. The load-displacement data was recorded at frequent cycle intervals and stiffness was determined. The two-step block load sequence employed in the present study is schematically shown in Fig. 2. The normalized stress is plotted against peak-trough points. One block of load sequence contains 400 cycles: 200 constant amplitude cycles at R = 0.1 and 200 cycles at R = -1. The load block can be converted to a stress block by multiplying all the peak-trough points by a reference stress value.

The block load sequence was repeatedly applied on to the GFRP nanocomposite test specimens, until failure. In the current experimental investigation, a reference stress of 180 MPa was employed to obtain the stress sequence of these blocks. Three repeat tests were conducted and the average number of load blocks required to fail was obtained. The stiffness of the specimen was monitored at the end of every block until failure occurred. The geometry and dimensions of the test specimen employed for fatigue tests is shown in Fig. 3. The gauge length (*GL*) and width (*W*) of the fatigue specimen varies as the type of loading varies. For loading at R = 0.1, GL = 50 mm and W = 25 mm. For loading at R = -1, GL = 10 mm and W = 12.5 mm.



Fig. 2 A schematic of variable-amplitude block load sequence



Fig. 3 Test specimen geometry used for fatigue testing

Material	Epoxy	Silica	Glass fiber	
Elastic modulus (GPa)	2.62	70	70	
Shear modulus (GPa)	0.97	30	28.7	
Poisson's ratio	0.35	0.17	0.22	
Density (g/cc)	1.20	2.65	2.50	

Table 1 Properties of constituent materials

Table 2 Predicted modulus (E₀)

Material	Modulus (GPa)		
	Estimated	Expt. [5]	
Epoxy/silica composite	2.97	3.07 ± 0.03	
GFRP nanocomposite-UD	41.2	_	
GFRP nanocomposite-MD	19.3	18.8 ± 0.7	

4 Results and Discussion

The mechanical and physical properties of the constituent materials i.e., neat epoxy, silica particles and glass fibers [6, 15] used in the micromechanics modeling are given in Table 1. The tensile modulus of GFRP nanocomposite estimated from its constituent properties is shown in Table 2 along with the experimental values taken from the literature [5]. As can be seen in Table 2, the predicted and experimental modulus correlates quite well. The stress-life (*S-N*) data generated for GFRP nanocomposite at two stress ratios R = 0.1 and -1 is shown in Fig. 4a. It was observed that the number of cycles to failure ($N_{\rm f}$) for an arbitrary stress level is different for two R ratios. For the case of R = 0.1, the experimentally obtained stiffness degradation curves at various stress levels are shown in Fig. 4b. The stiffness (*E*) is normalized with the initial stiffness

(modulus) of the material (E_0). It was observed that, as the applied stress level increases, the stiffness degradation of the material becomes steeper and results in lower N_f values.

When the normalized stiffness (E/E_0) is plotted as a function of normalized number of cycles to failure (n/Nf), for a given R ratio, all the curves corresponding to different stress levels follow a single curve as shown in Fig. 4c. This indicates that the stiffness degradation behavior for any R ratio can be represented by a single curve. Figure 5a shows the stiffness degradation predicted for GFRP nanocomposite using Shokrie's model under constant amplitude loading at a stress level of 150 MPa for two R ratios 0.1 and -1 along with experimental data. It was observed that the predicted stiffness behavior closely represents the experimental behavior. The predicted stiffness degradation of GFRP nanocomposite under two step block load sequence is shown in Fig. 5b. Residual stiffness of the material at any given number of blocks was estimated from the stiffness loss curves of the material at R = 0.1 and R = -1 stress ratios. Based on the experimental observations, a critical stiffness ratio of 0.8 was assumed as the failure criterion. It was observed that the predicted stiffness data lies within the experimental data scatter. The estimated fatigue life is 23 blocks whereas the life obtained from experiments is about 40 blocks.

5 Conclusions

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

• The stiffness of GFRP nanocomposite estimated using the micromechanics models was observed to correlate quite well with experimental values.



Fig. 4 a Stress-life data of GFRP nanocomposite at stress ratios R = 0.1 and -1, b stiffness degradation curves for various stress levels under constant amplitude loads at R = 0.1, c normalized stiffness degradation curves for various stress levels under constant amplitude loads at R = 0.1



Fig. 5 a Stiffness degradation behaviour of GFRP nanocomposite at stress ratios R = 0.1 and -1 at $\sigma_{max} = 150$ MPa, b stiffness degradation behaviour of GFRP nanocomposite under variable-amplitude block load sequence at reference stress =180 MPa

- Stiffness degradation behaviour of the material under constant amplitude fatigue load was predicted using Shokrieh's model. The predicted stiffness loss curves closely represent experimental behaviour.
- The stiffness degradation behaviour of GFRP nanocomposite under a two-step variable amplitude block load sequence was predicted using constant amplitude stiffness degradation data. The predictions closely match with the experiment.

Acknowledgments Authors wish to thank Mr. Shyam Chetty, Director, Dr. Satish Chandra, Head, STTD, CSIR-NAL for their constant support. We acknowledge the assistance provided by Prof. Kinloch and Dr. Taylor, Imperial College, London, UK. Thanks are also due to the technical support staff members of FSIG-STTD for their assistance in experimental work.

References

 Thostenson E T, Li C, and Chou T W, Compos Sci Technol 65 (2005) 491.

- Hussain F, Hojjati M, Okamoto M, and Gorga R E, J Compos Mater 40 (2006) 1511.
- Manjunatha C M, Bojja R, and Jagannathan N, Mater Perform Charact 3 (2014) 327.
- 4. Manjunatha C M, Bojja R, Jagannathan N, Kinloch A J, and Taylor A C, *Int J Fatigue* **54** (2013) 25.
- Manjunatha C M, Taylor A C, Kinloch A J, and Sprenger S, Compos Sci Technol 70 (2010) 193.
- Kinloch A J, Masania K, Taylor A C, Sprenger S, and Egan D, J Mater Sci 43 (2008) 1151.
- 7. Reifsnider K, Int J Fract 16 (1980) 563.
- 8. Post N L, Case S W, and Lesko J J, Int J Fatigue 30 (2008) 2064.
- 9. Degreck J, and Van Paepegem W, Appl Mech Rev 54 (2001) 279.
- 10. Shokrieh M M, and Esmkhani M, J Mater Sci 48 (2013) 1027.
- 11. Halpin J C, J Compos Mater 3 (1969) 732.
- 12. Halpin J C, and Kardos J L, Polym Eng Sci 16 (1976) 344.
- Kaw A K, Mechanics of composite materials (2nd ed.), CRC Press, Boca Raton (2005).
- 14. Shokrieh M M, and Lessard L B, J Compos Mater 34 (2000) 1056.
- Swaminathan G, Hossain M, and Shivakumar K N, Proceedings of 51 st AIAA Structures, Structural Dynamics and Materials Conference, Florida (2010).