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Load-increasing fatigue test to characterize the interface of composites under fatigue loadings

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The effect of glass-fiber epoxy interface in cross-ply reinforced composites on the fatigue behavior is studied by using load-increasing fatigue test. The damage as measured by stiffness reduction is more significant for the composites with poor bonded fibers as was found for EP sized ones, dependent on test conditions. Energy loss is shown to be a sensitive tool to characterize the nature of fiber matrix adhesion. The energy loss for composites with poor adhesion between fiber and matrix results in significantly higher amounts of consumed energy during a single stress-strain loop than those composites containing well-bonded fibers. © *2003 Kluwer Academic Publishers*

1. Introduction

The fatigue behavior of composite materials has been a subject of active research in recent years. The damage process in laminated composites subjected to fatigue loadings is significantly different from that observed in non-fibrous materials. Four main damage modes have been observed in laminate composites under fatigue loadings: matrix cracking, fiber-matrix debonding, delaminations, and fiber fracture. Typically, matrix cracking and delamination occur early in the life, while fiber-matrix debonds and fiber fractures initiate during the beginning of the life and accumulate rapidly towards the end, leading to final failure. It has been observed that the stiffness of the laminate reduces during the process of damage accumulation in laminated composites by using stiffness change as non-destructive fatigue damage parameter [1].

Crack propagation (crack bridging or debonding) controlled by the interfacial strength plays an essential role in fatigue behavior of composites based on brittle matrices such as epoxies. When interface bonding is relatively weak, debonding and frictional sliding occur readily upon crack extension, allowing fibers to remain intact and bridge the crack. When the fibers are frictionally bonded to the matrix, for instance in CCMs, the interfacial sliding can be fully characterized by the interfacial sliding shear stress, τ, found to be not constant. Changes in τ have been attributed to fiber surface abrasion, asperity wear, and matrix plasticity. A strong interface would inhibit interface sliding and lead to fiber fracture instead of crack bridging by intact fibers [2–4].

Other PMCs also show brittle characteristics as plastic deformation is suppressed at the microstructural level as much as the load is carried by the fibers. Microcracking in toughened epoxies and in thermoplastic matrices are also dominant dissipation mechanisms precluding plastic deformation that occurs readily in bulk matrices [5].

Further, damage initiation and growth rate depend on test conditions such as cyclic stress range, load ratio and test frequency [6–10]. For instance, it was found for unidirectional phenolic composites [7] that fatigue strength decreases with an increase of stress ratio from 0 to 0.4.

Mandell *et al.* [8] found for [0°, 90°] glass-fiber epoxy composites that specimen tested at higher frequencies have longer life times. Initial strength and the rate of loss of initial strength per decade of cycles was found to be greater for waveforms with less time at maximum strength.

Ellyin and co-workers [9, 10] discussed the fact that fiber-dominated failure in unidirectional composites is essentially independent of the rate/frequency of loading, while matrix dominated failure mode is a rate/frequency-dependent phenomenon due to the viscous matrix behavior.

2. Load-increasing fatigue test

Wöhler or S-N method is usually adopted for determining the behavior of materials exposed to fatigue loadings. Generally, new engineering materials must be tested under these conditions before they can be utilized for construction components because of the fact that roughly 80% of all structures fail because of fatigue. Taking into account the maximum frequencies for tests

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without frequency induced heatings of the specimen with 10 Hz for brittle and 2 Hz for ductile composites and the dependency of the fatigue behavior from further test conditions such as stress ratio, one can see that a screening method can be very helpful for R&D from both the financial and the timesaving viewpoint.

Load-increasing fatigue tests and an additional online measuring of characteristic values like energy loss and stiffness seem to be one of these possible screening tests. In a load-increasing test, the initial applied maximum load is chosen in a damage-free load level with increasing the load by a defined rate until final failure.

Ehrenstein and his co-workers [11–14], for instance, used this method for discussing structural effects of SMC and reinforced PBTP and SAN taking into account fiber content, fiber length and interfacial strength with significant differences in damping vs. max. stress curves. The authors [15–19] used this test procedure to characterize developments on fibers [16, 18], fiber-matrix adhesion [15–17, 19] as well as environmental test conditions [15, 16, 19] on the fatigue behavior of natural-fiber reinforced plastics with significant differences in critical load for damage initiation, load of final failure and energy loss and dynamic modulus vs. max. stress curves.

It is known from load sequence test [33] that, if the material memory effect is negligible, the low to high load sequence is more damaging than the high-low sequence as far as the fatigue life is concerned.

This paper is dealing with the influence of test frequency and stress ratio on the fatigue behavior of cross-ply glass-fiber epoxy composites with differences in interfacial strength. The influence of the interfacial strength on the S-N-curves for these materials was discussed previously [20].

3. Experimental

3.1. Materials

In this study continuous E-glass fibers with a specially developed epoxy compatible sizing (EP-sizing) for a strong fiber matrix adhesion and with a polyethylene sizing to generate a weak fiber-matrix interaction (PE-sizing) were used and embedded in a brittle (LY556/HY917/DY070 from Ciba Geigy GmbH) epoxy resin (tensile strain at failure = 3.3% for pure resin). The EP sizing bases on uncured bisphenol A epoxy binder which contains gamma-aminopropyltriethoxy silane, while the PE sizing is pure high molecular weight polyethylene (Hordamer PE 03 from Hoechst AG). The laminates were manufactured by using a two step process: unidirectional prepreg tapes were produced by filament winding technology and hot pressed under vacuum and 80°C to cross-ply composites.

Finally, the composites were additionally cured by 1 h at 100°C and 8 h at 140°C. The fiber volume fraction in each of the cured panels was determined using DIN EN 60. The average fiber volume fraction in the 'EP-sized' and 'PE-sized' panels was 0.4. The 150 mm long, 16 mm wide, and 2 mm thick specimen were cut from the cross-ply laminates. Cross-ply end tapes were bonded to the coupons.

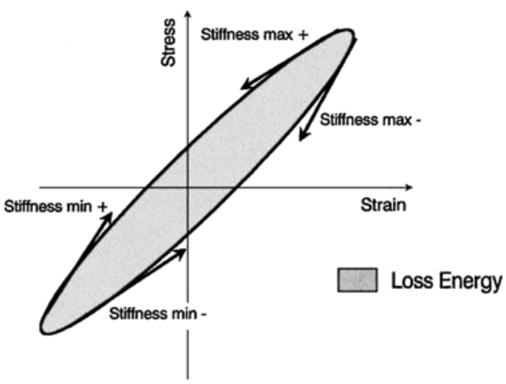


Figure 1 Dynamic stress-strain curve and definition of the characteristic values used.

3.2. Test procedure

All of the load-increasing fatigue tests were performed on a servo-hydraulic MTS test machine under load controlled mode. A 25 mm extensometer was used to monitor strain continuously during the fatigue test. Tension-tension fatigue tests in load-increasing mode with different stress-ratios 'R' and frequencies 'f' were done for the different material systems. Damage was monitored by recording the dynamic stress-strain curves continuously during the test. Further, different stiffness characteristics and the energy loss, both defined according to Fig. 1, were calculated.

4. Results and discussion

4.1. Influence of interfacial strength

Transverse-ply cracking is a principal cause of stiffness reduction in laminated composites loaded either quasi-statically or in fatigue [21–23]. The stiffness reduction due to other damage mechanisms (such as delamination and fiber breakage and splitting of the 0° plies) is very much less than that arising from matrix cracking [22]. By using this idea as a parameter to characterize the effect of the fiber-matrix interfacial phenomena, Subramanian *et al.* [1, 24] found differences in stiffness reduction at 80% load level for carbon fiber cross-ply epoxy composites in the range from 0.88 to 0.96 as a result of the differences in transverse crack density. The modulus reduction at 60% and 67% load level, respectively, of untreated and MA-PP treated glass-fiber polypropylene composites for 10° off-axis laminates at CDS was found to be very similar for both [25], because of the low crack sensitivity of PP in comparison to a brittle epoxy resin as used by Subramanian *et al.* [1, 24] and throughout this study. Aveston's (ACK) classical 1971 paper(used and discussed by [26]) treats the problem of matrix cracking process in unidirectional CMCs by an overall energy balance, illustrates that matrix cracking strain $\varepsilon_{\mathrm{mu}} \propto \tau^{1/3}$ with τ as interfacial shear strength.

Dynamic modulus vs. load cycles and applied max. load, respectively, for both types of composites is shown in Fig. 2. One can see that the dynamic modulus is in general lower and CDS is reached at lower max. applied loads in the case of composites containing PE-sized fibers. Furthermore, because of damages, hysteresis stress-strain loops are characterized by changing area and decreasing main slope. A theoretical loop is

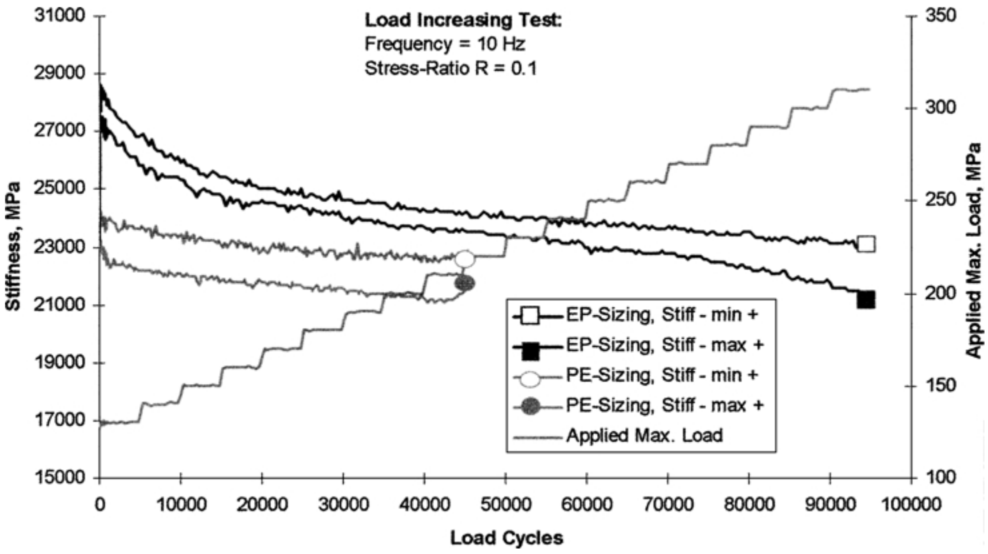


Figure 2 Influence of fiber-matrix adhesion on the stiffness vs. load cycles of glass-fiber epoxy composites containing 'PE-sized' and 'EP-sized' fibers.

given in Fig. 1, careful stress and strain measurements allow to determine the changes in some stiffness characteristics (tangent modulus) of the loop, viz. 'Stiffness min +' at the start of loading and 'Stiffness max +' at the end of loading with typical changes in these stiffnesses for composites with PE- and EP-sized fibers. Both composites tested possess a strong non-linearity in the loading part of the stress-strain hysteresis loop (ratio 'stiffness max +' to 'stiffness min +') which is more significant for the PE-sized composites. Reasons for this non-linearity could be a result of fiber matrix sliding (dependent on interfacial adhesion and applied load) in the 0° plies as well as the possibility of crack closure arising from frictional effects. Consequently, these cracks will not open unless a certain stress is applied [26]. A similar discussion is published by Walls *et al.* [27] for MMC's, where the reduction in modulus is connected with matrix cracking, while the non-linearity in the stress-strain response was more a result of the interface sliding. Both of these structural mechanisms lead to a more significant non-linearity of the PE-sized composites during the whole test and are independent from applied max. load.

As a result of the changes in fracture mechanisms, significant differences in dynamic failure load for the 'PE-sized' and 'EP-sized' brittle epoxy composites with 220 MPa and 310 MPa, respectively, were observed.

It is obvious that in fibrous composite materials energy is dissipated during crack initiation and propagation by a multiplicity of microfracture events occurring at the crack tip including fiber fracture, matrix cracking, interfacial breakdown, fiber 'relaxation', and fiber pull-out [28, 29]. Because of the fact that each of these different microfracture events is related to a defined amount of dissipated energy [28, 29], the energy loss, measured with load-increasing load, seems to be an effective and sensitive tool for measuring damages directly in materials as was shown and discussed previously [16, 20].

With regard to the influence of the interfacial strength, Beaumont *et al.* [28, 29] showed for static loadings that the energy for debonding, post debonding friction and pull-out energy is dependent on interface properties. Further, because of the fact that initial matrix cracking load 'σ_c' (Equation 1—developed for static loadings [31]) increases with increasing interfacial shear strength 'τ', differences in critical load for damage initiation has to be found in load-increasing fatigue tests.

$$\sigma_c = \left[\frac{6E_f f^2 \tau E^2 \Gamma_m}{E_m^2(1-f)R}\right]^{1/3} \tag{1}$$

where 'E_f' is the fiber Young's modulus, 'f' the fiber volume fraction, 'E' the composite Young's modulus, 'Γ_m' is the matrix fracture energy, 'E_m' is the matrix Young's modulus, and 'R' the fiber radius.

Both facts could be observed in Fig. 3 where the influence of the interfacial strength on the energy loss-applied max. load curves are illustrated. First, one can see that the critical load for damage initiation/propagation is lower for the 'PE-sized' than that for the 'EP-sized' glass-fiber reinforced epoxies as expected because of Equation 1. Because of higher energy loss with increasing debonding length and decreasing interfacial strength, a higher energy loss at a given applied max. load has to be found for the 'PE-sized' composites and is shown in Fig. 3. Furthermore, a more significant period of continuous damage propagation is shown for the 'EP-sized' composites which is not in agreement with the results on 10° off-axis tension-tension fatigue tests (S-N method) of glass-polypropylene composites published by van den Oever *et al.* [25]. The authors found a more continuous modulus reduction for composites with a poor adhesion (global damage), while the composites with a good adhesion showed a local damage (catastrophic failure).

4.2. Influence of test conditions

4.2.1. Test frequency

The measurements about the influence of test frequency were carried out with a stress-ratio of $R = 0.1$. The frequencies used were 5 Hz and 10 Hz.

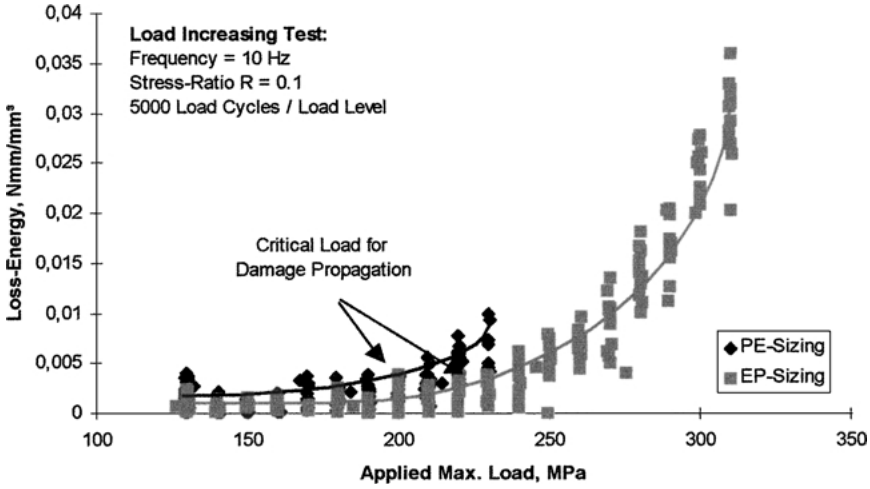


Figure 3 Influence of fiber-matrix adhesion on energy loss vs. applied max. load of glass-fiber epoxy composites.

Richardson *et al.* [7] concluded for glass-fiber phenolic composites by doing S-N tests that frequencies between 1.5 Hz to 25 Hz have only a minor but increasing influence on the fatigue strength. mandell *et al.* [8] showed with S-N curves by square wave fatigue and cross-ply glass-fiber epoxy composites that higher test frequencies result in longer life-times. Stinchcomb *et al.* [34] measured a significant reduction in dynamic compliance during strain-controlled fatigue test with an increase in cyclic frequency for boron-aluminum and boron-epoxy composites. Phenomenologically, Masters *et al.* [35] concluded for $[0/\pm45/90]_s$ and $[0/90/\pm45]_s$ graphit-epoxy composites that saturation crack spacing in the 90° plies is insensitive to cyclic load frequency.

The load-increasing tests done throughout this study on 'EP-sized' composites showed a similar tendency as for fatigue strength with a marginal increase in load at failure for higher test frequency (Fig. 4). The other important characteristic value for fatigue loadings, the stiffness, increases for higher test frequencies at damage free applied max. loads because of its visco-elasic nature. On the other hand a higher frequency leads to higher load cycles and applied max. load for reaching the characteristic damage stage (CDS), defined analogous to that for S-N tests by Reifsnider and co-workers [1, 23, 24], while non-linearity of the stress-strain response (ratio 'stiffness max +' to 'stiffness min +') is higher for lower frequencies. Taking into account the fact that the non-linearity in the loading part of the stress-strain hysteresis loop is also a result of the interface, one can conclude that this observation should be a result of interfacial relaxation processes.

4.2.2. Stress-ratio

It has long been recognized that the stress ratio, i.e. the ratio of the minimum to the maximum applied load in fatigue, has a strong influence on fatigue response [7–10, 31, 32]. For instance, Richardson *et al.* [7] showed for unidirectional glass-fiber phenolic composites for tension loadings that fatigue strength decreases with an increase in stress ratio from 0 to 0.4. Mandell *et al.* [8] concluded for glass-fiber epoxy composites a continuous increase in lifetime as the minimum stress is increased to approach the maximum stress, with the exception of cases very close to the static fatigue conditions.

The load at failure measured (Fig. 5) by load-increasing test increases with increasing stress ratio from $R = 0.01$ to 0.1 and a frequency of 10 Hz for both the 'EP-sized' and 'PE-sized' based composites and is in contrast to the observation on unidirectional

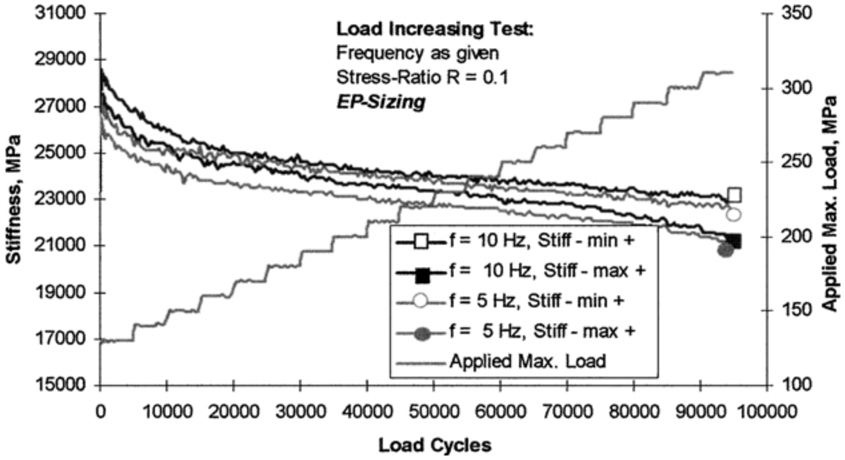
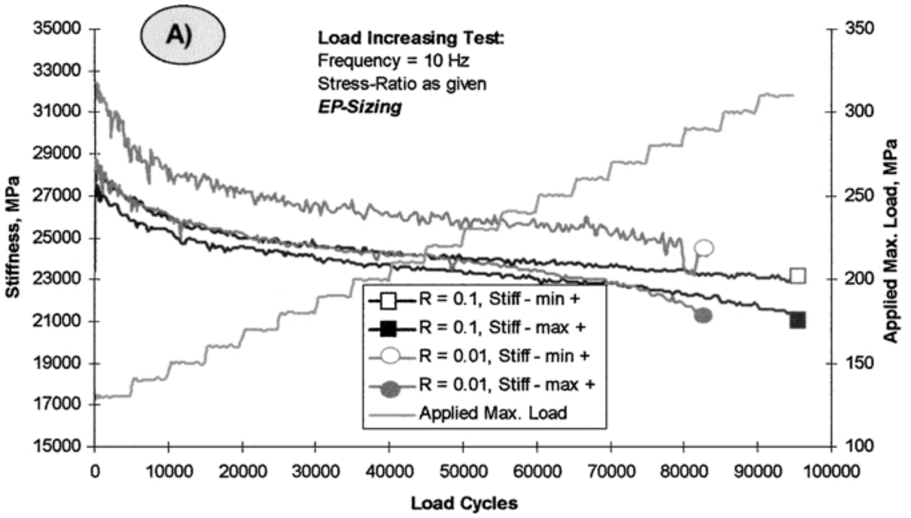


Figure 4 Influence of test frequency on stiffness of 'EP-sized' glass-fiber reinforced epoxy composites (stress-ratio = 0.1).



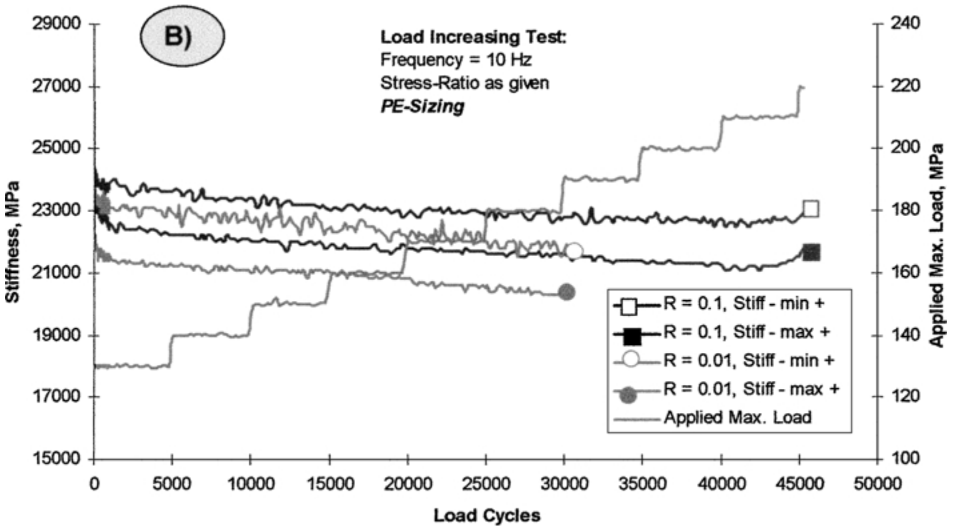


Figure 5 Influence of stress-ratio (frequency = 10 Hz) on stiffness of (A) 'EP-sizes' epoxy composites and (B) 'PE-sizes' epoxy composites.

glass-fiber phenolic composites published by [7]. Further it can be seen that loss in stiffness at CDS and because of the correlation to crack density in the 90 deg plies, both are not sensitive to tension-tension stress ratio. Independent from interfacial strength, CDS is reached at similar load cycles and applied max. loads for both ratios, while the period till CDS is more marked for the composites with a strong interface as mentioned in Section 4.1. More significant is the fact that the degree of non-linearity of the stress-strain response (ratio 'stiffness max +' to 'stiffness min +') is higher for lower stress-ratios. The reason for this could be a mechanism published by Pryce *et al.* [26] with a critical load for crack opening. A lower stress ratio leads to lower applied min. loads, with higher modulus at the start of the loading (stiffness min +) as a result, and can be seen in the significant differences in 'stiffness min +' between both stress-ratios for the 'EP-sized' composites (Fig. 5A). In contrast 'stiffness max +' is quite similar, the differences measured are more related to differences of strain rates. Because of the fact that the above-mentioned critical load for crack opening is strongly affected by the interfacial strength [26], the changes in non-linearity have to be much smaller for composites with a weak interface as can be seen in Fig. 5B for the 'PE-sized' composites.

5. Conclusion

The effect of glass-fiber epoxy interface in cross-ply reinforced composites on the fatigue behavior was studied by using differently sized glass-fibers and the load-increasing fatigue test. The composites only differed in the interface, all other conditions being kept constant. To generate a weak interface a 'PE' sizing was used, while a 'EP' sizing led to a strong adhesion. The damage as measured by stiffness reduction in the laminates was more significant for the composites with 'PE-sized' fibers than for the 'EP-sized' ones and more or less independent from tension-tension stress-ratio used. The degree of non-linearity of the stress-strain hysteresis loop (defined as ratio 'Stiffness max +' to 'Stiffness min +') was measured to be more significant for the 'PE-sized' composites and affected by stress-ratio and frequency. The energy loss was shown to be a sensitive parameter to characterize the nature of fiber matrix adhesion with lower values for an increase in interfacial strength.

It can further be concluded that the load-increasing fatigue test and its on-line measured characteristics can be successfully used to characterize interfacial behavior of composites under dynamic loadings.

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