Low-cycle Fatigue of Thermally-Cycled Carbon-Epoxy Composite

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Abstract

Investigation of low-cycle fatigue of composites is of vital importance for the design of structural components. In this study, the effects of thermal cycling on low-cycle fatigue behavior of carbon-epoxy laminate are investigated in a purely bending mode at strain ratios of -1 and 0.1. It is found that a very small increase in the plastic strain amplitude catastrophically shortens the fatigue life N of the composite determined as a number of cycles corresponding to 0.9 of the initial force. The preliminary thermal cycling of the composite at the temperature varied from 180°C to −195.8°C shortens N values in comparison to the reference at small strains.

Keywords : Carbon fiber laminate, Fatigue, Thermal cycling.

Introduction

The increasing application of fiber-reinforced composites, especially in aircraft and automotive industries, requires the knowledge of their fatigue behavior. Most components may appear to have nominally cyclic elastic stresses, but microcracks, notches, welds or any other stress concentrators may result in local cyclic plastic deformation. In these areas, plastic deformations may be very significant, while the load applied to a component is much less than the yield point of the material. Under these conditions, another approach was developed in 1950s, which uses the local cyclic strain as an independent and governing fatigue parameter (the local strain - life method) related to low-cycle fatigue (LCF) region.[1, 2]

Fatigue experiments in pure tension and compression are most often used in fatigue research in general and in LCF, in particular. [3,4] However, in real applications, loading modes are much more complicated. Bending fatigue tests realized in glass- or carbon fiber reinforced polymers include three-point, four - point, and cantilever bending loading modes.[5-10] One of drawbacks of these methods is the local stress concentration due to the use of pins or rollers in contact with the sample.

Methods of cyclic bending of composites are known without any physical contact between the sample and any sort of guide like pin or roller.[8-11] It was determined that the fatigue damage in fully reversed bending load manifested as a loss of bending resistance, occurred at high strains through fiber breakage, matrix cracking, and interfacial shear failure. But as the number of bending cycles increased, crack growth occurred in the matrix along fiber interfaces.[8] Post - buckling bending test without stress concentration under the loading nose of the device was performed using in situ detection of damage in unidirectional carbon fiber reinforced polymer (CFRP) by means of electric resistance measurements.[11] When the applied load reaches the Euler critical load, the specimen buckling induces a flexural moment. But in addition to this moment, axial compressive load application to the sample also causes shear and compressive stresses. Besides, this method requires a carefully performed electroplating of the composite.

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For polymeric composites used in aircraft industry, the material will be inevitably exposed to cyclic thermal loading. Such loading is defined as "thermal fatigue", which is considered as low-cycle fatigue because thermal fatigue cracks usually start after less than 50,000 cycles.[12] It is known that relatively brittle CFRPs are very sensitive to thermal cycling, and transverse cracking was found to be a dominant damage mechanism of graphite-epoxy specimens at thermal cycling at the temperature varied from −121°C to +121°C.[13] Using thermal cycling or simulated low earth orbit (LEO) environmental conditions including thermal cycling, ultraviolet radiation and high vacuum, a deterioration of mechanical properties of CFRP was found. Thermal cycling slightly reduces the tensile strength and modulus of the composite, but has a more serious effect on matrix-dominated mechanical properties of the composite, such as flexure, compression and interlaminar shear.[14-17] For instance, transverse flexural stiffness and strength decreased by 25% and 34%, respectively, after 80 cycles in simulated LEO environment with the temperature change from -70°C to 100°C.[15]

Polymer composites subjected to previous thermal cycling have a greatly shortened fatigue life in comparison with reference material, as it was found, e.g., for carbon/PEEK laminate exposed to 750 cycles at the temperature varied from 60°C to -60°C and then tested under tension–tension fatigue load with a stress ratio of 0.1. X-ray inspections indicated that no cracks were generated due to cyclic thermal exposure.[18]

In spite of a relatively large amount of data on flexural fatigue of carbon-epoxy laminates, their fatigue behavior in a purely bending mode is not studied in-depth. Therefore, an investigation of low-cycle fatigue of carbon-epoxy laminate and the effect of thermal cycling on LCF is of vital importance for the design of structural components. In this study, the effects of cyclic pre-loading and thermal cycling on low-cycle fatigue behavior of carbon-epoxy laminate are investigated.

Materials and Experimental Procedures

The carbon fiber-epoxy laminate used in this study was consolidated from prepreg and structural epoxy resin supplied by Hexcel Corporation of the USA. The prepreg HexPly® 8552-type is an amine-cured, toughened epoxy resin system supplied with woven carbon fibers operating in environments up to 121ºC. Typical mechanical and physical properties of components of the carbon-epoxy laminate are given in Tables 1, 2.[19]

Table 1: Mechanical properties of components of carbon-epoxy laminate based on HexPly® 8552-type prepreg (dry conditions, room temperature).

Material	0^oTensile strength, MPa	Tensile strain, %	90^oTensile strength, MPa	0^oTensile modulus, GPa	90^oTensile modulus, GPa	0^oILSS (short beam shear), MPa
Fiber	5310	1.80	-	276		-
Matrix[*]	121	-	121	4.7	4.7	-
Prepreg	1090	-	945	85	80	88
Laminate[**]	800	1.52	800	82	82	70

[*] Toughened epoxy resin with glass transition temperature T_g (dry/wet) of 200°C/154°C.[19]
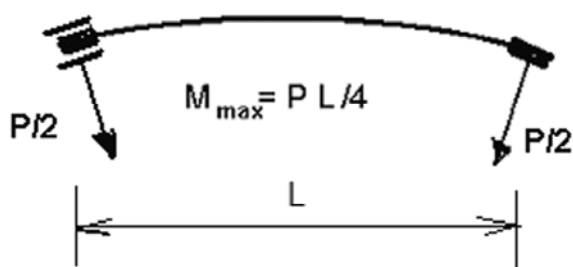[**] The $\pm 0_n/90_m$ angle-ply configuration.

Table 2: Physical properties (dry conditions, room temperature).

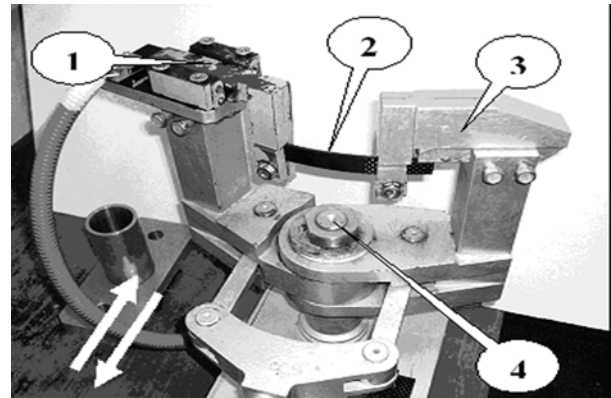| Fiber type	Density, g/cc	Mass, g/m^2	Cured ply thickness, mm	Nominal fiber volume, %
IM7 6K-type fiber (SPG 196-P)	1.77	-	-	-
Matrix	1.30	-	-	-
Prepreg	1.56	196	0.199	55.57

The carbon-epoxy specimens had the angle-ply configuration of $\pm 0_n/90_m$ with the thickness of, mainly, 3 mm; width of 16 mm; length of about 90 mm and the load span of 50 mm. Some samples had a thickness of 2 mm and 3.3 mm. The low-cycle fatigue tests were tested on a Model IP-2 pure bending fatigue machine with the capacity of around 50 Nm in a strain-controlled loading mode under reversible or symmetric (R = -1) and asymmetric (R = 0.1) load, where R = $\varepsilon_{min}/\varepsilon_{max}$ is the strain ratio; ε_{min} and ε_{max} are minimum and maximum values of total strain, respectively.

The scheme of the LCF test and a view of the sample deflection measurement are given in Figure 1. The sample 2 fixed in grips 1 and 3 was free to move in the longitudinal direction under a flexural load due to a possibility for reciprocal motion together with grip 1. The sine-wave input form with the frequency of 1.2 Hz was used. The force response of the samples during the LCF tests was monitored by the Model 614 Tedea load cell with loading range of 0 - 2,000 N and a special USB 9237 card (National Instruments Co.), and recorded using Labview program. The testing environment was air at 25°C ± 2°C.

(B)

Figure 1: Scheme (a) and view (b) of the sample grip of IP - 2 pure bending machine. 1 – first twist grip with a possibility for reciprocal motion, 2 – sample, 3 – second twist grip, 4 – pivot pin.

Three sets of samples were prepared: (1) reference, (2) fatigued for the short-beam shear test (SBS) and (3) thermally-cycled (TC) ones. The samples for SBS test were preliminary fatigued (without fracture) at the total strain amplitudes of 0.006, 0.008 and 0.009 and the strain ratio of -1. The number of cycles ranged from 200 to 5,000 for each strain. TC samples were prepared at the maximum and minimum operating temperatures of 180°C and −195.8°C (liquid nitrogen), respectively. Temperature increases at the rate of approximately 10°C/min and decreases approximately at 7°C/min; the duration of each of 10 thermal cycles was about 50 minutes.

(A)

The total strain was calculated using the measurement of the deflection amplitude *A*. The last was measured by a displacement indicator to ±0.005 mm. In accordance with the simple-beam theory,[20] true bending strain and engineering bending strain of a material ε_x near its external surface in the direction of the longitudinal axis X can be calculated using Eqs. (1) and (2), respectively:

$$\varepsilon_x = \ln (1 - y / R_n) \qquad (1)$$

$$\varepsilon_x = - y / R_n \qquad (2)$$

where the maximum strain amplitude corresponds to *y* value amounting to a half of the beam (sample) thickness; R_n is the neutral axis radius. For $\varepsilon_x < 0.1$, the difference between these two strains is less than 5%. Eq. (2) was used for the total strain amplitude measurement. The external radius R_o and radius of the neutral axis R_n were calculated for a sample with the thickness *d* and span *2a* using the following equations: $R_o = (A^2 + a^2)/2A$ and $R_n = R_o - d/2$. The plastic strain amplitude $\Delta\varepsilon_p$ was calculated as a difference between the maximum total strain $\Delta\varepsilon_{tot}$ and maximum elastic strain $\Delta\varepsilon_e = TYS/E$, where *TYS* and *E* are tensile yield stress and elastic modulus, respectively.

Tensile and interlaminar shear stresses were determined using the standards ASTM D3039[21] and D2344, [22] respectively. Small samples (12 x 9 x 2 mm³) for the short-beam shear tests (SBS) were cut out from the center of a fatigued sample (80 x 12 x 2 mm³) with the span L of 8 mm and tested on the Zwick-1445 testing machine using a cross-head speed of 1.3 mm/min Figure 2. The failure load was interpreted as the first maximum load attained before failure of the outer layer.

This load was taken as an apparent shear strength of the material.
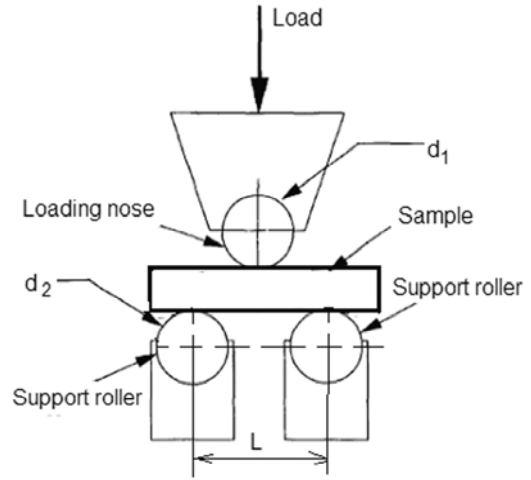

Figure 2: Scheme of SBS test setup.

Figure 3 shows the strain–stress diagram for a laminate sample in the two-point bending test performed on the fatigue machine. The strain was calculated in accordance with Eq. 2 using deflection values measured by an indicator. It is shown that the laminate is very brittle, with strain to failure of about 0.015. Using Eq. 3, the ultimate flexural strength (*UFS*) amounting to 885 MPa was found:
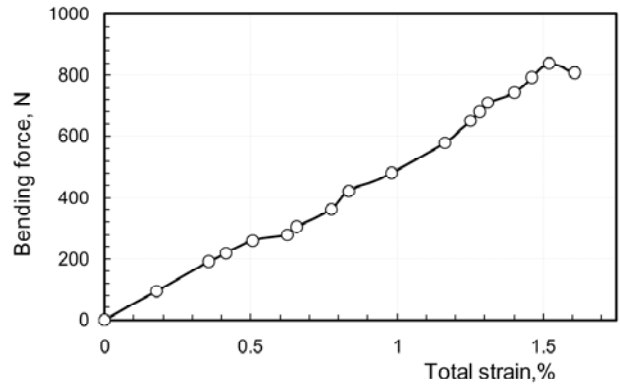

Figure 3: Example of stress - strain diagram for 3-mm-thick composite sample (two-point bending test performed on a fatigue machine).

$$\sigma_{UFS} = M_b/W_x = 3 F_{max} L /bh^2; \qquad (3)$$

where $M_b = F_{max} L/2$ and $W_x = bh^2/6$ are the bending moment and moment of inertia, respectively; $F_{max} = 850$ N is ultimate flexural force; L= 50 mm, b = 16 mm and h = 3 mm

are length (span), width and thickness of the sample, respectively. The reference and thermally-cycled 3 mm-thick samples had approximately the same tensile properties given in Table 1 for the laminate.

The microstructure was studied using an optical microscope 'Nicon' and a scanning electron microscope JEOL JSM-5600 with 'NORON' energy-dispersive analysis system.

Results and Discussion

Short-Beam Shear Test

An apparent shear strength of the material measured in accordance to Ref.[22] has relatively large scatter of results Figure 4. However, in the first approximation, we can conclude that shear stress (σ_s) for samples pre-fatigued, e.g., at the lowest total strain amplitude ($\Delta_{\varepsilon tot}$ = 0.006) is practically independent on the number of cycles varying from 0 (reference samples) to 5,000. At this total strain, cyclic deformation is practically elastic, namely, the plastic strain portion amounts to a very small value of about 0.0004. However, the average shear stress at increasing strain amplitudes of 0.008 and 0.009 decreased comparing to the reference one from 65 MPa to 60 MPa and from 65 MPa to 53 MPa, or by about 7 % and 18 %, respectively, after 2,000 cycles Figure 4.
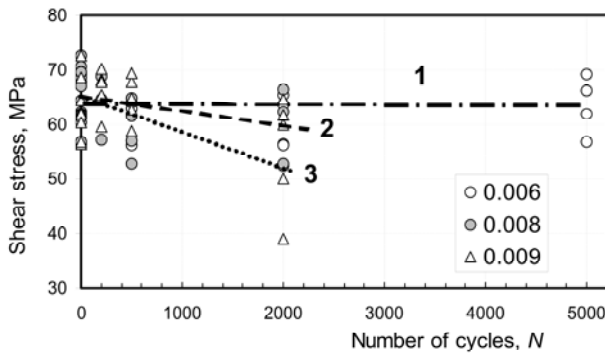

Figure 4: Shear stress measured in the short-beam test on fatigued graphite-epoxy samples as a function of the number of applied cycles at the maximum total strain of 0.006 (1), 0.008 (2) and 0.009 (3).

Low Cycle Fatigue Test

Carbon-epoxy composite belongs to materials showing strain softening behavior during cyclic loading with monotonous decreasing resistance force Figure 5. As a criterion of fatigue failure of composite (lifetime N_f), the number of cycles $N_{10} = N_f$ corresponding to a 10%-decrease in the cyclic force amplitude F_{max} was selected. For example, at the maximum total strain of 0.011, the lifetime of a 2-mm-thick laminate corresponding to 10%-decrease in the force (from 500 N to 450 N) amounted to 100 and 800 cycles at strain ratios of -1 and 0.1, respectively (Figure 5, curves 2, 3). However, the sample tested at strain amplitude of 0.007 and under an initial reversible force of 350 N did not fail in the range of LCF Figure 5.
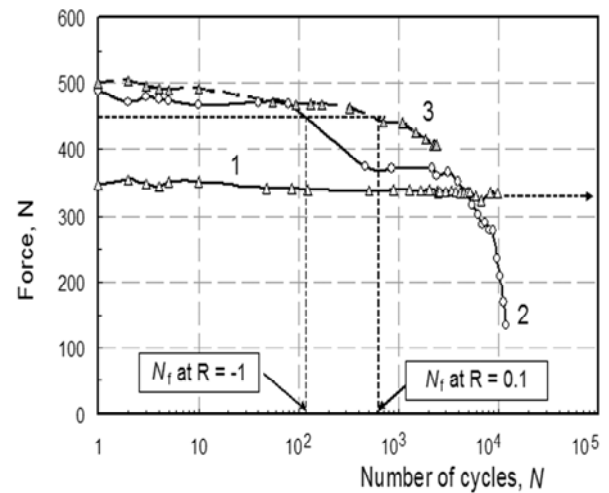

Figure 5. Force history for 2 mm - thick samples tested in reversible (1; 2) and asymmetrical(3)loading modes at strain values of 0.011 (2,3) and 0.007 (1).
Number of cycles to failure N_f corresponds to the force amounting to 0.9 of the initial force.

A typical presentation of low-cycle fatigue test results is satisfactorily described by Coffin-Manson's relation[1-2] :

$$\Delta\varepsilon_p = \varepsilon_f \ N^{-c} \qquad (4)$$

where $\Delta\varepsilon_p$ is the plastic strain amplitude, ε_f is approximately equal to the true fracture strain and is called fatigue ductility coefficient, N is the number of cycles to failure, c is so - called fatigue ductility exponent. The latter, as a rule, varies in a relatively narrow interval for metals $(0.4 – 0.6^{(1,2)})$, but these variations can significantly affect the values of fatigue life – larger values of c result in smaller values of the fatigue life.

It is known that c can increase with temperature increase and loading frequency decrease, i.e. external actions increasing ductility lead to higher values of c. Here c reflects the ductility and hardening of the material under cyclic strain, and there is a definite correlation between c and cyclic strain-hardening exponent n' found from the cyclic stress-plastic strain curve represented by a single straight line in log stress –log $(\Delta\varepsilon/2)$ plot.[23] Materials with high monotonic strain-hardening exponents undergo cyclic hardening, while in the opposite case they undergo cyclic softening, e.g., carbon-epoxy laminate shows cyclic softening Figure 5.

A very significant effect of plastic strain amplitude on the fatigue life of the composite was revealed (Figure 6, Table 3). A reversible loading mode significantly shortens the lifetime of the laminate in comparison with asymmetrical mode with the strain ratio of 0.1. For instance, the lifetime of 3 mm-thick laminate increases from 70 cycles for R = - 1 to 7×10^4 cycles for R = 0.1 or in two orders of magnitude at the same plastic strain of 0.0055 Figure 6. The similar trend was observed, e.g., for a ±45°- angle-ply carbon/epoxy laminate: under the maximum stress of 120 MPa, the lifetime of the composite increased from 10^2 to 10^5 cycles at stress ratios of - 1 and 0.1, respectively.[24]
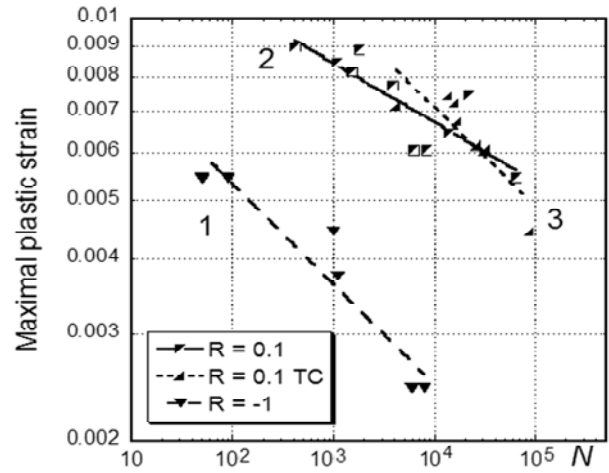

Figure 6: Diagram $\Delta\varepsilon_{pl} – N$ for 3-mm-thick graphite – epoxy reference (1, 2) and thermally – cycled (3,TC) samples tested at strain ratios R of -1 (1) and 0.1 (2, 3).

Table 3: Coefficients of fitting equation $\Delta\varepsilon_{pl} = \varepsilon_f N^{-c}$ [a].

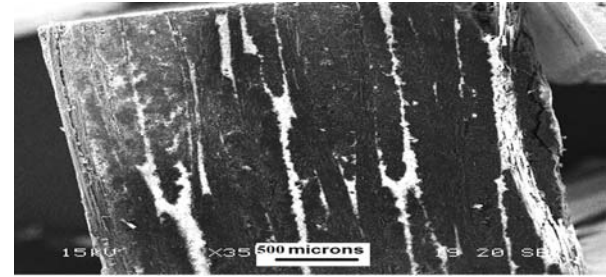| Samples	Strain ratio	ε_f	c	Correlation coefficient r
Reference	0.1	0.0171	-0.1023	0.88
Thermally cycled	0.1	0.0367	-0.1747	0.94
Reference	-1	0.0114	-0.1673	0.97

[a] Here ε_f and c are called fatigue ductility coefficient and fatigue ductility exponent, respectively.

The Coffin - Manson relationship (Eq.4) is valid both for reference and for thermally-cycled samples with fatigue ductility exponent c varying in a relatively narrow range from -0.102 to -0.167 (Table 3). There is an inversion point in log $\Delta\varepsilon_p$ - log N plot that describes the LCF behavior of reference and thermally-cycled laminates at N
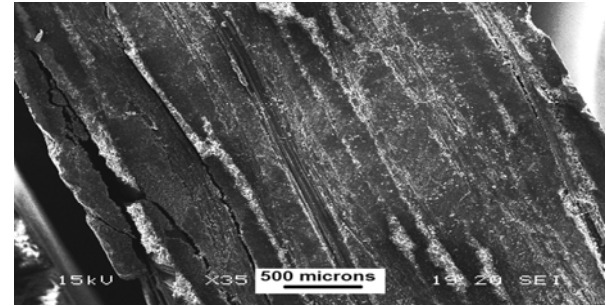
values of about 20,000 cycles at the strain ratio of 0.1 Figure 6. At plastic strain amplitudes exceeding 0.006, the lifetime of both the reference and thermally-cycled laminates was less than 20,000; however, thermally-cycled laminate showed a slightly longer lifetime in comparison with the reference one. On the contrary, close to high-cycle fatigue region at N higher than 20,000 cycles thermal cycling of the composite slightly shortens its lifetime (curves 2 and 3, Figure 6). A similar behavior was found for carbon/PEEK laminate in fatigue tests performed in a 'push-pull' mode: the fatigue life under the stress of 0.75 of *UTS* decreased from around 158,000 cycles for reference samples to about 40,000 cycles for thermally-cycled samples.[18]

It is reported that the bending strength of a carbon-epoxy composite thermally-cycled less than 40 times increases comparing to the reference one due to some increase in the cross-linking density in the epoxy resin layers.[17] Probably, in our case, at plastic strain exceeding 0.006 corresponding to the short N values (less than 2×10^4), increasing bending strength of TC samples plays a dominant role in a higher fatigue life of the composite. It can be suggested that the dominant damage mechanism of the thermally-cycled composite at lower plastic strains and the longer fatigue test duration is formation of small transverse cracks resulting from thermal loading as it was reported for flat and tube carbon-fiber composites. [13]
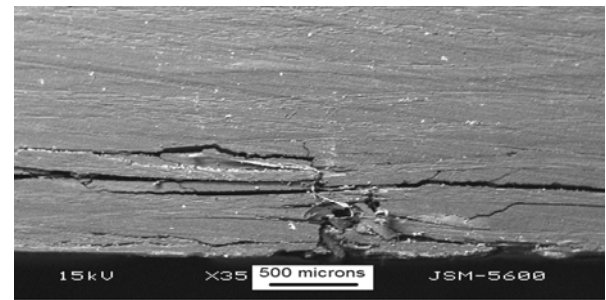
Fatigue ductility coefficient increases from 0.0171 for reference samples to 0.0367 for thermally-cycled laminate. It points to increasing plasticity of the laminate after thermal cycling, which leads to increasing fatigue life at relatively large strains. Some damage patterns on the cross-section of a thermally-cycled sample were found with comparison to the reference one (Figs.7a, 7b).
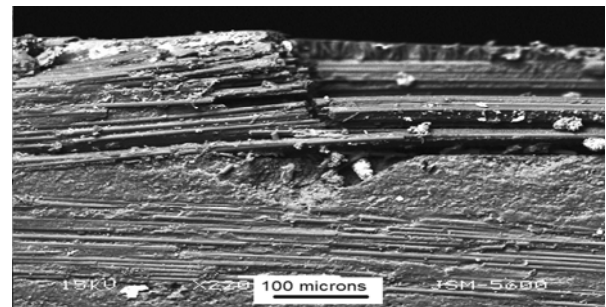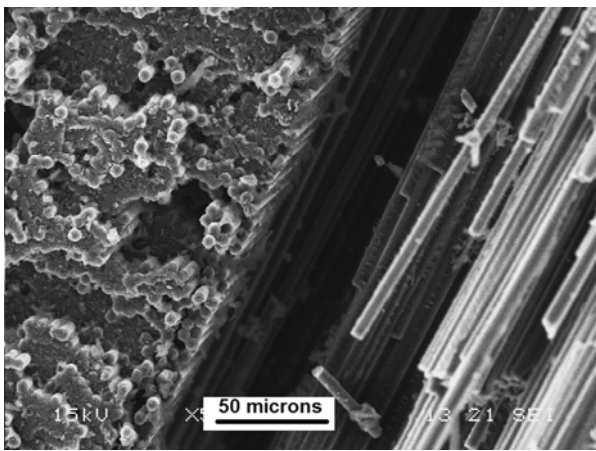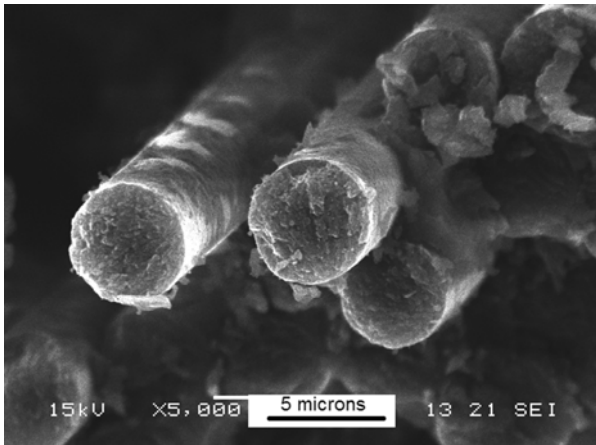

(A)

(B)

(C)

(D)

Figure 7: Fracture patterns in a reference (a, c, d) and thermally - cycled (b) composite on compression (c) and tension (d) sides after LCF tests at maximum strain amplitude $\Delta \varepsilon_{tot}$ of 0.013 (R = 0.1).

In an asymmetric mode, the sample side that experienced tension exhibited, mainly, matrix and fiber cracking, while the side that experienced compression, underwent buckling, which caused delamination and cracking Figure 7d. Of course, during reversible fatigue mode, both sides of the sample underwent tension and compression and therefore, experienced matrix and fiber cracking. Failure of the composite in a tensile test is characterized by fiber and matrix crashing without delamination in the sample Figure 8.

(A)

(B)

Figure 8: SEM micrographs of the composite fracture area (a) and several broken fibers (b) after a tensile test.

Energy released at the time of the process of damage accumulation leads to the sample heating. The sample temperature did not seem to change noticeably prior to failure of the first ply characterized by a drastic decrease in force, as observed for 3.3-mm thick sample in an asymmetric loading mode after about 200 cycles Figure 9. A weak initial reduction of the force and a small increase in the temperature due to damage accumulation can be explained by the appearance of several transverse cracks before the crack density saturation.[13,25,26] Then, several damaging modes can develop simultaneously (cracking and delamination) with a final catastrophic failure of the whole sample accompanied by a marked increase in the temperature from 23.9°C to 29.0°C (Figs.7 and 9).
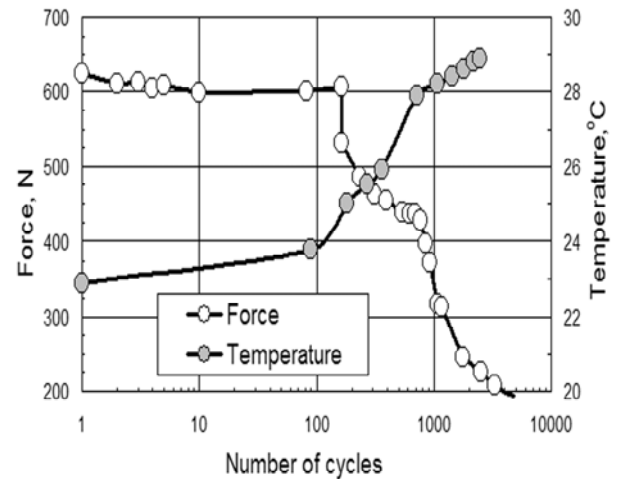
Figure 9. Force- and temperature history for 3.3 mm - thick - sample (R = 0.1, $\Delta\varepsilon_{tot}$ = 0.013).

Conclusions

In the short-beam shear test, it was found a decrease in the average values of shear stress of preliminary fatigued laminate after 2,000 cycles by about 7 % and 18 % at the total strain amplitude of 0.008 and 0.009, respectively.

It was also found that a very small increase in the plastic strain amplitude $\Delta\varepsilon_p$ catastrophically shortens the fatigue life of the composite. For example, in case of

reversible loading, an increase in $\Delta\varepsilon_p$ from 0.002 to 0.003 corresponds to lifetime decrease by an order of magnitude (from 10^4 to 10^3 cycles). The Coffin-Manson relationship for plastic strain amplitude $\Delta\varepsilon_p = A\,N^{-c}$ is valid both for reference and for thermo-cycled samples with the same fatigue ductility exponent c varying in a relatively narrow range from -0.102 to -0.167.

An inversion point in a log $\Delta\varepsilon_p$ - log N plot was found for reference and thermally cycled composite at strain ratio of 0.1. At maximum plastic strain more than 0.006 corresponding to $N \leq 20,000$, thermally-cycled laminate shows slightly longer fatigue life with comparison to that for the reference one. On the contrary, at smaller strains corresponded to the high-cycle fatigue region, thermal cycling of the composite shortens its fatigue life.

Acknowledgments

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