

Fatigue Performance of Kevlar/Epoxy Composites with Filled Matrix by Cork Powder

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Abstract: The objective of this study is to characterize the fatigue strength of a Kevlar/epoxy laminate composite as well as the benefits obtained by using an epoxy matrix filled by cork powder. Twelve ply laminates, all in the same direction, of woven bi-directional Kevlar 292, were prepared by hand lay-up, using an SR 1500 epoxy resin. The composite sheets were produced by a vacuum moulding process. The addition of cork powder reduces the static strength, however, in terms of fatigue strength a similar behavior was found for both laminates.

Keywords: Composite laminates, Aramid fiber, Fillers, Fatigue, Mechanical testing

Instruction

Composite materials have been applied to various engineering fields such as aircraft, space, automotive, sport and marine industries but, particularly, in military applications. They offer an attractive potential for reducing the weight as consequence of their high specific strength and stiffness.

For military applications aramid fibers are a very important reinforcement for advanced composites as consequence of their high degree of toughness and damage tolerance. In fact these fibers do not present brittle behavior, like glass or carbon fibers, but fail by a series of small fibril failures. These many small failures absorb much energy and, therefore, result in very high toughness.

In this context the literature shows a really interest in these fibers and several studies can be found. To improve the interfacial adhesion, as consequence of the low surface energy and chemically inert surface of the Kevlar fiber, there are extensive works [1-9]. On the other hand to improve the impact strength several techniques are proposed [10-19], and, more recently the inclusion of fillers was also successful used [20-24].

For the industries mentioned above, structural components are usually subjected to complex fatigue load histories. In this case the performance of the composite materials is strongly dependent of the fibers behavior. In case of aramid fibers shows time-dependent mechanical behavior, indicating that their viscoelasticity may play an important role on the fracture behavior of the composites [25]. Tanaka *et al.* [26] investigated the influences of stress waveform and wet environment on the fatigue fracture behavior of aramid single fiber (Kevlar 49). They observed that, in air, the fatigue strength under negative pulse waveform was higher than those under sinusoidal and positive pulse waveforms.

The fatigue strength in wet air was lower than that in laboratory air and the strength degradation under cyclic loading by wet environment was much larger than that under quasistatic tensile condition. The fatigue fractured fibers under the sinusoidal and the positive pulse waveform in air also broke with fiber splitting, similar to the case of the tensile test. Under the negative pulse waveform, the specimens that fractured at 10^2 - 10^4 stress cycles broke with fiber splitting and for fatigue lives higher than 10^5 stress cycles transverse crack to the fiber axis occur. Bunsell [27] subjected individual Kevlar 49 fibers to sinusoidal tensile load and found that the minimum load required to develop fatigue failure of individual Kevlar 49 fibers was approximately 80 % of the UTS. Similar tests were developed by LaFitte and Bunsell in Kevlar 29 fibers [28]. Maximum stress levels between 56-90 % of the fibers' UTS were analyzed. Their results indicate that, under cyclic loading conditions where the maximum load is below about 75 % of the UTS, individual Kevlar 29 fibers exhibit little or no reduction of lifetime compared to the creep lifetime under constant stress equal to the maximum level used in the cyclic testing. At higher stress levels if the cyclic amplitude is small enough, such that the minimum stress applied during fatigue remains above about 25 % of UTS, the creep behavior dominates and lifetime is enhanced due to the intermittent exposure to high stress. On the other hand, when the cyclic amplitude is great enough to cause the minimum stress to fall below about 25 % of UTS, fatigue damage ensues and lifetime is reduced. However for bending fatigue the main mechanisms that promoted strength degradation are: compressive fiber buckling, kinking and fiber-to-fiber surface abrasion [29,30]. These mechanisms result from the high tensile modulus of the fiber coupled with its low compressive and transverse strengths [31,32].

The fatigue behaviour of Kevlar fibre reinforced composites is a subject requiring a better understanding, particularly the

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fatigue behavior of the Kevlar fiber reinforced composites with filled matrix. In this context the aim of this work is study the bending fatigue response of a Kevlar/epoxy composite with filled epoxy matrix by cork powder. Cork presents an alveolar structure and it is characterized by high specific strength and stiffness, near zero Poisson coefficient, high damage tolerance to impact loads, impervious to liquid and gases, resistance to reactive agents and microorganisms, resistance to wear and fire, very low thermal conductivity, good acoustic insulation capacity and excellent damping characteristics [33-36].

Material and Experimental

Twelve ply laminates, all in the same direction, of woven bi-directional Kevlar 170-1000P (170 g/m²), were prepared by hand lay-up and the overall dimensions of the plates were 330×330×3 mm and the fiber weight fraction was 66 %. SR 1500 epoxy resin and a SD 2503 hardener, supplied by Sicomin, were used. The system was placed inside a vacuum bag and a load of 2.5 kN was applied for 24 hours in order to maintain a constant fiber volume fraction and uniform laminate thickness. During the first 10 hours the bag remained attached to a vacuum pump to eliminate any air bubbles existing in the composite. The post-cure was followed according to manufacturer datasheet (epoxy resin) in an oven at 40 °C for 24 hours.

With the same manufacturing process were produced composite laminates with filled epoxy matrix by cork powder. The bulk density of the cork powder is 0.1095 gcm⁻³ and the particles' size, in terms of percentile, is: d(0.1)=18.6 μm, d(0.5)=78.9 μm and d(0.9)=208.3 μm. More details about the powder characterization can be found in [37]. Before being added into the resin, the cork powder was dried in an oven-dried (Heraus, model UT 6060) at about 120 °C for 2 h. When dried it was placed in a desiccators, cooled to room temperature, and stored until use.

The mixture, epoxy resin and the cork powder, was conducted at 900 rpm for 2 hours and, at same time, subjected to ultrasonic bath sonicator. The filler content employed was 3 wt.% of the epoxy resin-hardener mixture. The mixture was degassed in a vacuum oven followed by addition of hardener agent. Special care occurs in this process to avoid the formation of bubbles.

The specimens used in the static and fatigue three point bending tests, were cut from these plates, with dimensions of 60×10×3 mm and tested with a span of 45 mm. The static tests were performed according the recommendations of ASTM D 2344 Standard in an electromechanical Shimadzu AG-10 universal testing machine, equipped with a 5 kN load cell and TRAPEZIUM software. Four specimens were tested for each material. The fatigue tests were carried out in constant displacement control, at room temperature in an electromechanical machine where the stress ratio can be

changed. A load cell was used to monitor the load. The load wave was sinusoidal, having a stress ratio of $R=0.1$ and a frequency of 25 Hz.

The nominal bending stress (σ) was calculated using:

$$\sigma = \frac{3PL}{2bh^2} \quad (1)$$

being P the load, L the span length, b the width and h the thickness of the specimen.

The water effect on the flexural properties of the laminates was also analyzed. In order to obtain the water absorption it was used the following procedure, in accordance with BS EN ISO 62:1999. The samples were placed in an oven at 80 °C for 2 hours, then cooled and weighed in order to obtain the dry weight (DW). Afterwards, a series of samples were immersed in water and periodically weighted to obtain the current wet weight (CWW). The water absorption in weight percentage (W%) was calculated from equation (2):

$$W\% = \frac{CWW - DW}{DW} \times 100 \quad (2)$$

Results and Discussion

The flexural properties of the Kevlar/epoxy composites with pure resin and filled by cork powder were obtained by 3PB static tests. Typical load-displacement curves for both materials are plotted in Figure 1. Both materials show a linear elastic behavior, in an early stage, with a non-linear region that starts around 150 MPa. After peak stress the load decreases, however, the drop is more significant for filled laminates. The soft decreasing of the load-displacement

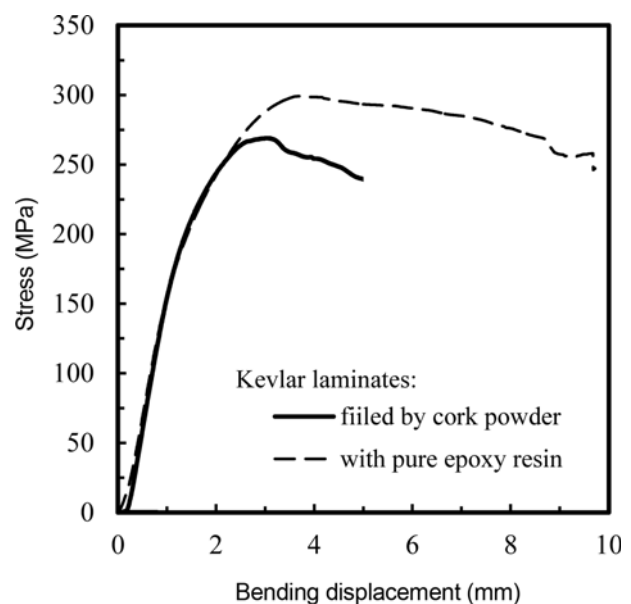


Figure 1. Typical load-displacement curves for Kevlar laminates with pure epoxy resin and filled by cork powder.

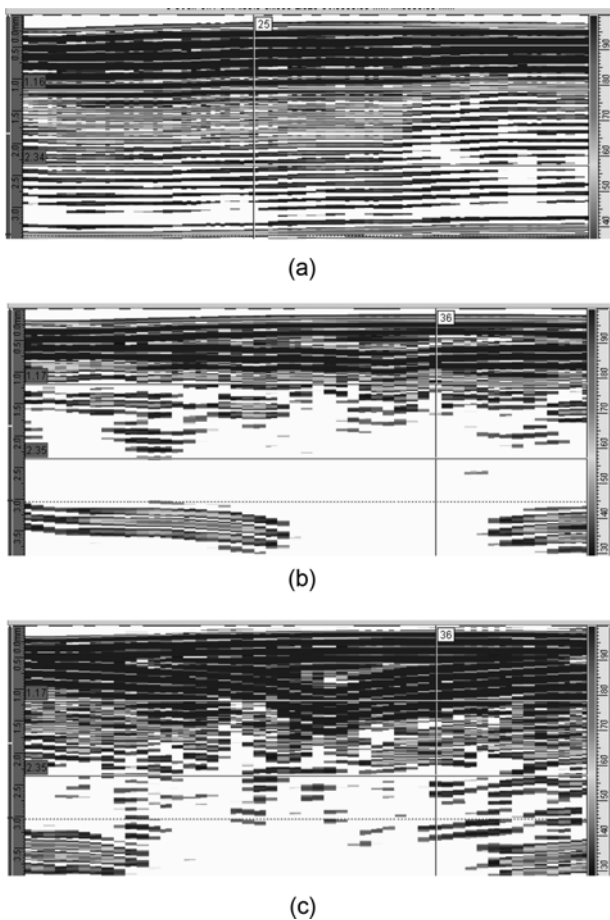


Figure 2. S-scan observations: (a) specimens before tested, (b) laminates with pure resin after tested, and (c) laminates filled by cork powder after tested.

curve, for Kevlar with pure resin laminates at maximum load, denotes a more stable grow of the damage mechanisms than the one observed for filled composites. In this case it is possible to conclude that the fillers change the mechanical behavior of the matrix and agrees with the study developed by Reis *et al.* [37], where was found that the introduction of cork powder in a polyester matrix decreases the flexural strength.

Figure 2 presents the damage obtained by S-Scan, using an Olympus phased array instrument, Omni-K-PA1664MW10. In detail, it is possible to observe that Figure 2(a) presents the material without damages of a specimen before testing and, by contrast, the specimen observed in Figure 2(b) and 2(c) shows significant delaminations (by absence of wave transmission) for laminates with pure resin and laminates filled by cork, respectively. In fact delaminations, for Kevlar composites, are the main energy absorption mechanism [38]. At same time the aramid fibers do not fail by brittle cracking, as do glass or carbon fibers, but they fail essentially by a series of small fibril failures. All these damage mechanisms

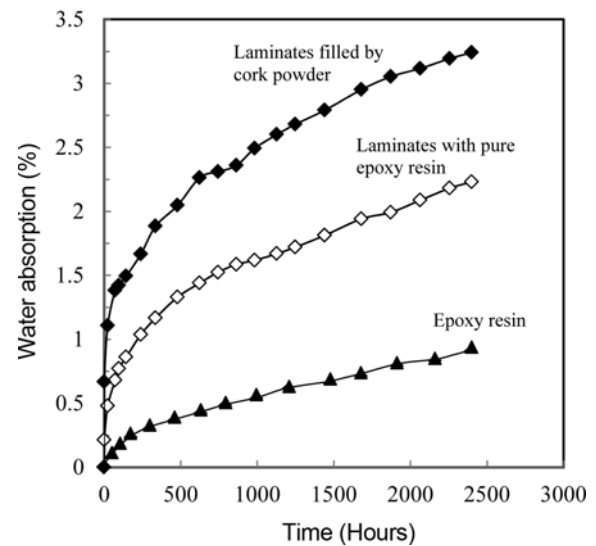


Figure 3. Water absorption curves.

are more pronounced as consequence of the high compressive stress concentration in the pin load contact region [39].

Figure 3 shows the water absorption curves for two material configurations. All these configurations show water absorption up to 100 days (2400 hours) and the same trend is expected after this value. According with the open literature, for Kevlar-reinforced composites the hygroscopic nature of the resin and the fiber must be considered [40]. For epoxy resins the water absorption varies substantially, depending on the resin type and the curing system. Eckstein [41], for example, shows that the water absorption may differ by a factor of ten between different epoxy resins, and to a factor of up to three for the same resin with a different curing agent. On the other hand, aramid fibers are strongly hygroscopic and often with values higher than the matrices. A maximum moisture content of 6% was reported by the manufacturers for Kevlar-49 at room temperature 96% RH [40]. In this context, the water absorption was studied for the system SR 1500 epoxy resin and SD 2503 hardener, and it is presented in Figure 3 also, in order to understand better the results for laminates.

After 100 days, the epoxy system used present around 0.9% of moisture content, value much lower than observed for the laminates. In fact when the Kevlar fibers were added to the resin it was observed an increase of 2.5 times of water uptake, as consequence of their hygroscopic behavior [40], and around 3.6 times for laminates with fillers. Similar tendency was obtained by Reis *et al.* [37] when similar fillers were added to polyester resin.

Finally the UV effect on the flexural properties of the laminates was also studied. The samples were exposed around 450 hours in a UV-Irradiation chamber BS-02 supplied by Dr. Gröbel UV-Elektronik GmbH. It was used four lamps, type UVB, with intensity of 2.3 mW/cm². According with

Woo *et al.* [42], photo-degradation of polymers due to UV exposure occurs by activation of polymer macromolecules arising from the absorption of photons. This process generates free radicals in the presence of air or oxygen, and induces dissociation of chemical bonds on the surface as well as inside the material by diffusion. Physical changes consist mainly of discoloration and micro-cracking phenomenon. However for relatively short periods of exposure only some changes in surface morphology are observed, but, in extended exposure to UV radiation, the elongation and rupture of polymers shows to be very sensitive to irradiation [42]. On the other hand the UV stability of Kevlar has been described in the literature [43,44] and it is possible to conclude that its light stability depends on the thickness of the exposed item. Very thin Kevlar 49 fabric when exposed directly to very high intensity sunlight for an extended period will lose about half its tensile strength within few days. In thicker items, such as a half-inch diameter rope, the outer layer protects the majority of the rope and strength loss is minimized [44].

Table 1 presents all results obtained in 3PB static tests, in terms of the average values and standard deviation. Ultimate stresses, σ_{UTS} , were obtained using equation (1) with the peak load values. It is possible to observe that the average value of the ultimate stress, at room temperature, is higher for laminates with pure resin, around 10.4 %, in comparison with laminates filled by cork powder. According with previous studies developed by the authors [37], it was also observed a decrease of the flexural strength with the filler content. This tendency was well fitted by the Nicolais and Nikodemo model, which is specially used when poor adhesion occurs between matrix and filler. The immersion in water promotes decrease in terms of ultimate stress for both laminates, relatively to dry ones. For laminates with pure epoxy resin the ultimate strength decreased around 23.1 % while for laminates filled by cork powder this value was around 31.5 %. This can be explained by the fact that the laminates filled by cork powder absorb more moisture. For epoxy resins, according with Akay *et al.* [40], a drop in the glass-transition temperature (T_g) of 15-20 °C per 1 % moisture pickup have been widely reported and, consequently, matrix plasticization and matrix/fiber interface degradation have been shown to influence the mechanical properties of

aramid-fiber/epoxy-resin composites. An approximately 35 % loss in the room-temperature flexural strength was observed in Kevlar-49/epoxy resin laminates at 5 % moisture uptake, apparently as a result of the ease of fiber buckling in a plasticized matrix [40]. Finally the UV effect on flexural properties is negligible for the time of exposure tested. According with Woo *et al.* [42] it is possible to say that the period of exposure was relatively short and insignificant changes in surface morphology (micro-cracks) and/or chemical bonds occurred. Consequently the flexural properties remained unchanged.

The results of the fatigue tests are plotted in Figure 4 in terms of maximum stress, at the beginning of the test, versus the number of cycles to failure (N). It was observed that fatigue strength depends significantly on the defined failure criterion, so the criterion of failure was defined as the moment when the loss of maximum stress reaches 25 % of the initial value. In terms of Kevlar fibers, its fatigue lifetime is dependent on the amplitude of the applied oscillatory load as well as the maximum load to which the fiber is cycled [27]. On the other hand these fibers show time-dependent mechanical behavior, indicating that their viscoelasticity may play an important role on the fracture behavior of the composites [25]. At same time the aramid fibers fail by a series of small fibril failures, which can explain also the fatigue performance for long fatigue life. The fatigue data were analyzed according with the ASTM 719 and respective parameters are plotted in Figure 4. Comparing the results shown in Figure 4 it is possible to observe a similar behavior for both laminates: medium and standard deviations of both parameters (A and B) of the two S-N curves have very similar values.

Figure 5 shows the normalized stress range ($\Delta\sigma/\Delta\sigma_0$) versus N/N_f curves for filled and unfilled laminates. $\Delta\sigma$ is the current stress range of the load cycle, $\Delta\sigma_0$ is the initial value of $\Delta\sigma$, N the current number of cycles and N_f is defined by the number of cycles from which a stabilization of $\Delta\sigma$

Table 1. Three point bending properties for several environmental conditions

| Kevlar laminates | Conditions | Average σ_{UTS} (MPa) | Std. dev. (MPa) |
|-----------------------------|-------------------|------------------------------|-----------------|
| With pure resin | Room temperature | 298.96 | 2.87 |
| | Immersed in water | 229.79 | 7.07 |
| | UV degradation | 296.7 | 3.94 |
| Resin filled by cork powder | Room temperature | 267.96 | 6.16 |
| | Immersed in water | 183.49 | 8.24 |
| | UV degradation | 265.97 | 8.47 |

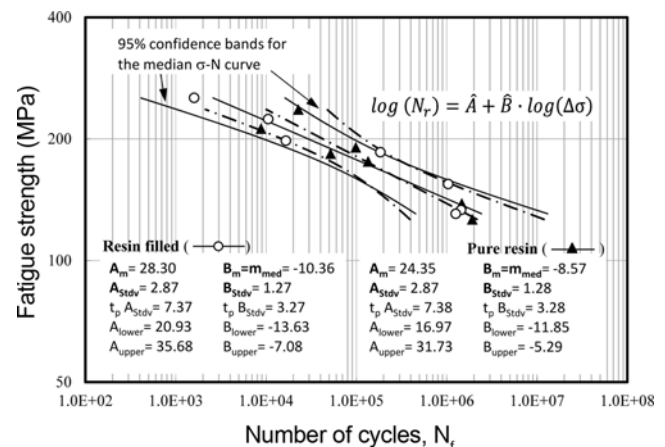


Figure 4. Fatigue strength, S-N curves.

occurs. These figures show an important dependency of the initial stress range. When the cyclic displacement amplitude

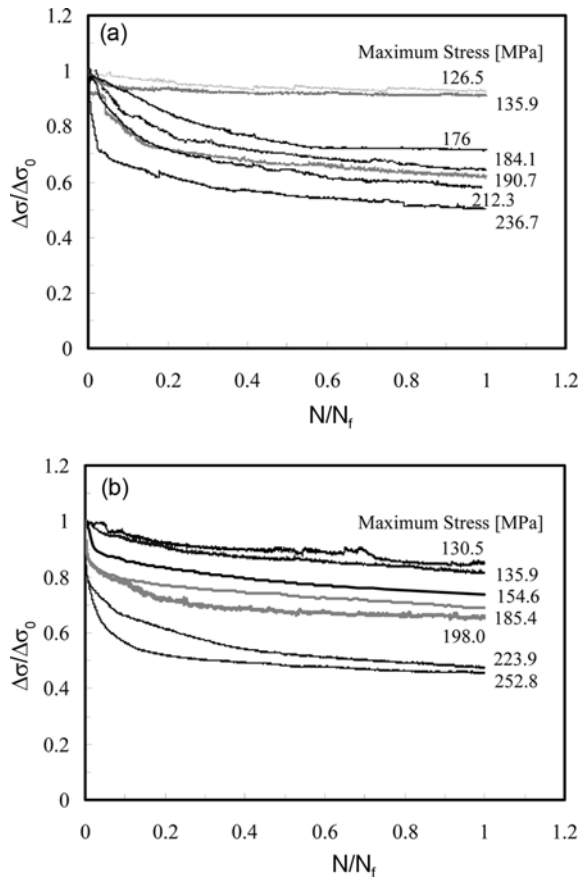


Figure 5. Typical $\Delta\sigma/\Delta\sigma_0 - N/N_f$ curves for: (a) Kevlar/epoxy laminates and (b) Kevlar with epoxy filled by cork powder laminates.

increases, the damage, quantified by the stress range decreasing, is faster. In all cases significant decreases of stress range were observed, since the first cycles of fatigue life, caused not only by matrix creep and microcracking, but essentially by significant delaminations between laminate layers. In fact delaminations, for Kevlar composites, are the main energy absorption mechanism [38]. The curves presented in Figure 5 were fitted by a curve with following mathematical equation:

$$\frac{\Delta\sigma}{\Delta\sigma_0} = a \cdot \left(\frac{N}{N_f} + c\right)^b \tag{3}$$

with

$$c = \left(\frac{1}{a}\right)^{1/b} \tag{4}$$

The values of “a” and “b” are constants obtained by a power trend line that fits the curves represented in Figure 4. Parameter “c”, obtained by the equation (5), was found to force the curve to start at 1. All constants are presented in Table 2.

Figure 6 shows a direct comparison of the normalized stress range, for filled and unfilled composites, relatively to the tests carried out at initial $\sigma_{max}=135.9$ MPa. It is possible to observe that the curve for laminates with pure resin is above of the curve for laminates with resin filled by cork powder. This suggests that occurs a significant stress realize and, consequently, a drop of the stiffness.

Figure 7 shows the UV effect on fatigue strength. Laminates with epoxy resin filled by cork powder were exposed by 450 hours and compared with other ones without any exposition time (control samples). The criterion of failure was defined, again, as the moment when the loss of maximum stress reaches 25 % of the initial value. It is possible to observe that both laminates present similar performance, which is

Table 2. Constants of the equations that fitting the different curves

| Material | Curves | Maximum stress (MPa) | Constants | | | Correlation coefficient |
|--------------------------------------------|---------------------------------------|----------------------|-----------|--------|--------|-------------------------|
| | | | a | b | c | |
| Kevlar + Epoxy resin | $\Delta\sigma/\Delta\sigma_0 - N/N_f$ | 126.5 | 0.929 | -0.017 | 0.0131 | 0.940 |
| | | 135.9 | 0.913 | -0.012 | 0.0005 | 0.970 |
| | | 176.0 | 0.705 | -0.099 | 0.0293 | 0.966 |
| | | 184.1 | 0.644 | -0.116 | 0.0225 | 0.997 |
| | | 190.7 | 0.622 | -0.095 | 0.0068 | 0.986 |
| | | 212.3 | 0.584 | -0.125 | 0.0135 | 0.992 |
| | | 236.7 | 0.511 | -0.107 | 0.0019 | 0.992 |
| Kevlar + Epoxy resin filled by cork powder | $\Delta\sigma/\Delta\sigma_0 - N/N_f$ | 130.5 | 0.869 | -0.030 | 0.0093 | 0.863 |
| | | 135.9 | 0.830 | -0.051 | 0.0259 | 0.974 |
| | | 154.6 | 0.775 | -0.035 | 0.0007 | 0.893 |
| | | 185.4 | 0.710 | -0.042 | 0.0003 | 0.987 |
| | | 198.0 | 0.650 | -0.066 | 0.0015 | 0.985 |
| | | 223.9 | 0.498 | -0.093 | 0.0006 | 0.947 |
| | | 252.8 | 0.454 | -0.094 | 0.0002 | 0.984 |

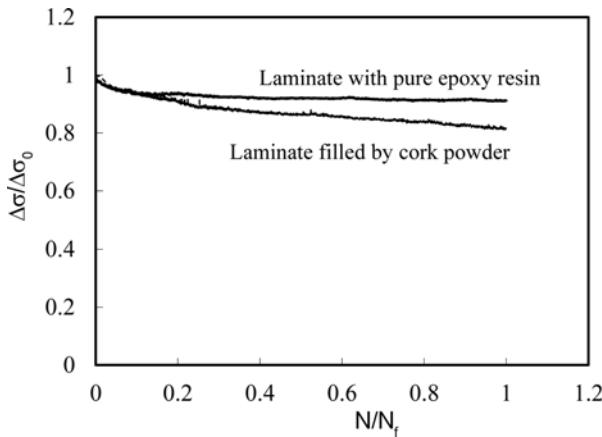


Figure 6. Comparison of the normalized stress range for initial $\sigma_{max}=135.9$ MPa.

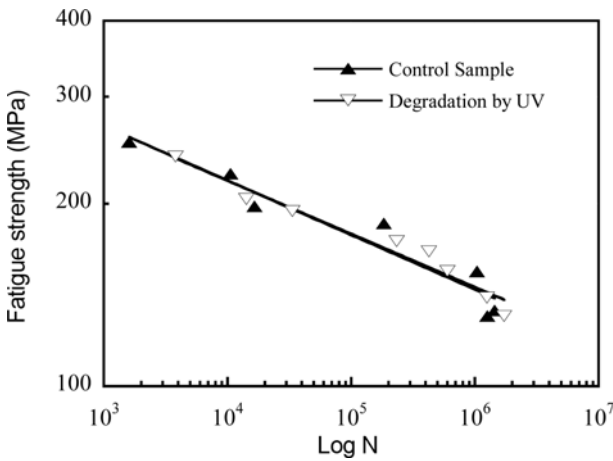


Figure 7. UV effect on fatigue strength, S-N curves.

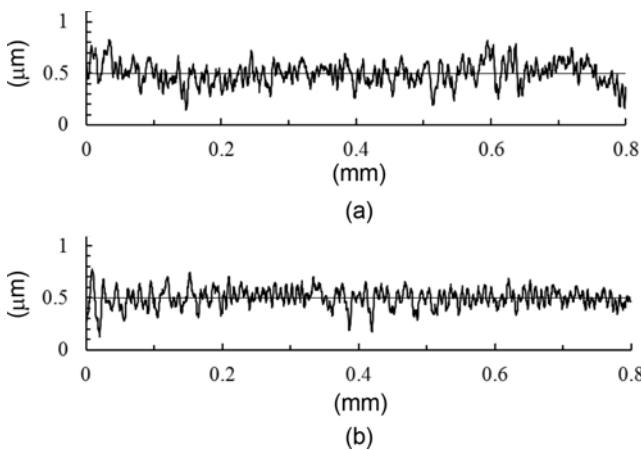


Figure 8. Typical roughness profile for: (a) control sample and (b) laminates exposed to UV radiation.

according with the static tests. This suggests that the period of exposure was relatively short to produce significant changes in terms of surface morphology (micro-cracks) and/

Table 3. Statistics of the roughness measurements

| Statistics | Control samples (μm) | After UV radiation (μm) |
|--------------------------------------|-----------------------------------|--------------------------------------|
| Arithmetic average, R_a | 0.0688 ± 0.0589 | 0.0879 ± 0.0401 |
| Root mean square, R_q | 0.0882 ± 0.0377 | 0.1111 ± 0.0101 |
| Average peak to valley height, R_z | 0.5174 ± 0.0955 | 0.5927 ± 0.0808 |
| Evaluation length, L_o | 0.8012 ± 0.001 | 0.8020 ± 0.0007 |
| Core roughness depth, R_k | 0.2158 ± 0.1155 | 0.2731 ± 0.0947 |
| Reduced peak height, R_{pk} | 0.0727 ± 0.0487 | 0.1029 ± 0.0183 |
| Reduced valley depth, R_{vk} | 0.1098 ± 0.0874 | 0.1218 ± 0.0572 |

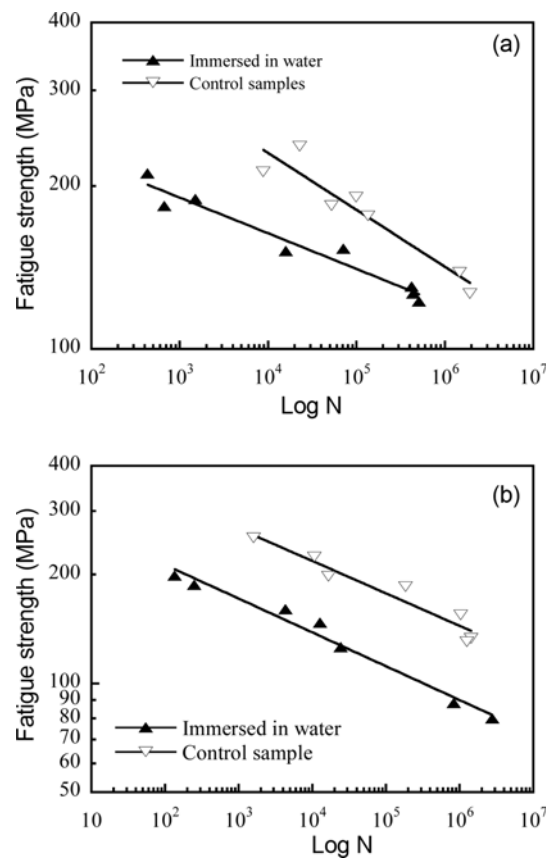


Figure 9. Water effect on fatigue strength, S-N curves: (a) pure epoxy resin and (b) epoxy resin filled by cork powder.

or chemical bonds. However, according with Woo *et al.* [42], for relatively short periods of exposure some changes in surface morphology are the main damage. In order to analyze the presence of micro-cracks, because it is well known their influence in the fatigue strength, Figure 8 shows the roughness profile for the two laminates. The roughness profiles were obtained by a Mitutoyo equipment, model SJ-500, for an evaluation length of 0.8 mm. Table 3 present the statistic of the roughness measurements, where is evident that all parameters are very close. As expected, the roughness

parameters, measured before and after UV exposition, are within the same band scatter, revealing the absence of micro-cracks promoted by the UV radiation.

Figure 9 shows the effect of the water uptake on fatigue strength. Again the criterion of failure was defined as the moment when the loss of maximum stress reaches 25 % of the initial value. For laminates with epoxy resin filled by cork powder the immersion in water, during 100 days, promotes a decrease around 36 % of the fatigue strength compared with dry samples (control samples) as shown in Figure 9(b). Similar tendency was observed in Figure 9(a)) for laminates with pure epoxy resin, however, for long cycle fatigue lives the effect of the water is less significant. As was observed in Figure 3, it is evident the hygroscopic nature of the resin and laminates. Once water infuses into matrix or interface region of composite, the water operates as plasticizer (spacing the polymer chains) promoting the deterioration of the glass transition temperature and the relieving of internal stresses. This phenomenon makes composites softer since the matrix becomes pliable due to the presence of the plasticizer, which causes significant drops of material properties. On the other hand, the degradation of the laminates includes debonding along the fiber/matrix interface, which is accelerated by the hygroscopic nature of the aramid fibers.

Conclusion

This paper has studied fatigue response effects in Kevlar/epoxy composites where cork powder was incorporated in the matrix. It was concluded that the addition of cork powder reduces the static strength. However, similar fatigue strength behaviors were found for both laminates.

The environmental conditions were studied in terms of water uptake and UV effects. The static and fatigue strength, for both laminates, is not influenced until 450 hours of UV exposure. No significant changes on roughness profiles were observed, revealing the absence of micro-cracks promoted by UV radiation. On the other hand, the water uptake promotes significant decreases of static and fatigue strengths for both laminates. However, this tendency was higher for filled resin laminates.

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