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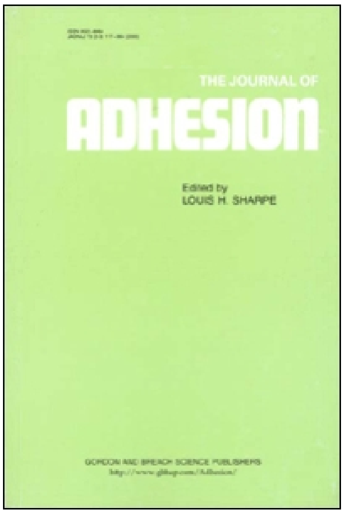

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Moisture Diffusion in Carbon/Epoxy Composite and the Effect of Cyclic Hygrothermal Fluctuations: Characterization by Dynamic Mechanical Analysis (DMA) and Interlaminar Shear Strength (ILSS)

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Moisture diffusion studies were carried out on the carbon/epoxy composite at $V_f = 0.7$ *and have been performed for three different cases* viz., *60°C/85%, relative humidity (RH), 60°C/95% RH, and 60°C/100% RH, to find out moisture absorption as a function of relative humidity. The specimens under the above conditions exhibited Fickian diffusion behavior. The effect of moisture absorption on the dynamic mechanical behavior and on the fiber/matrix interface was investigated. The humidity combined with the temperature effects may cause significant changes in the* T_g *of the matrix, and toughness affecting the laminate strength. Moisture absorption was correlated to the fracture mode of the laminate demonstrating the deleterious effect of moisture on the interface which leads to debonding between fiber and matrix. This behavior was investigated by scanning electron microscopy. The effect of cyclic thermal and humidity fluctuations is also characterized by ILSS.*

Keywords: Carbon/epoxy composite; DMA; Glass transition; ILSS

INTRODUCTION

Carbon/epoxy composites are widely accepted for use as primary aerospace components and in other structural applications due to their high performance characteristics. In service these components are generally exposed to environments usually involving temperature and humidity. Over the past few years, a great deal of experimental

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evidence has been collected to demonstrate that both physical and mechanical properties of composite materials can be strongly affected during hygrothermal ageing, affecting the composite performance. Carbon/epoxy composites do absorb moisture by a diffusion process and it is necessary to understand their behavior under varied environmental conditions, of which the hygrothermal factor is of vital importance [1].

The presence of moisture within a polymer composite can lead to significant changes in the physical and chemical characteristics of the polymeric matrix. Deformation in laminated composites can occur because of changes in temperature and the absorption of moisture. This is known as the hygrothermal effect. As polymers undergo both dimensional and property changes in a hygrothermal environment, so do composites utilizing polymers as matrices. Since fibers are fairly insensitive to environmental changes, the environmental susceptibility of composites is mainly due to the matrix. Consequently, in a quasi-isotropic composite the temperature-moisture environment has almost similar effects on the transverse, shear, and longitudinal properties [2].

Moisture absorption in the polymer matrix of a composite material can sometimes exert a greater influence on composite performance then the temperature changes. This is because polymers swell when they absorb moisture, while the reinforcement typically does not absorb moisture and, hence, does not swell. Thus, the reinforcement inhibits the swelling of the matrix, inducing internal stresses in the composite. Various polymers absorb different amounts of moisture. The amount of moisture absorbed tends to be independent of the exposure temperature, but directly proportional to the relative humidity of the environment.

Two possible mechanisms have been suggested to explain the effects of moisture on most composite systems: (a) matrix plasticization and (b) degradation of the fiber/matrix interface [2–4]. Moisture absorption by epoxy matrix composites has a plasticizer effect, as evidenced by the T_g reduction of the matrix. This effect is usually reversible when the water is removed, but exposure to moisture at elevated temperatures can produce irreversible effects, which can be attributed to the chemical degradation of the matrix and to the attack on the fiber/resin interface. This causes an increase of the internal voids of the entangling polymer chain, promoting, as a consequence, chain expansion and microcrack formation in the polymer matrix [5].

The way in which composite materials absorb moisture depends upon factors such as temperature, fiber volume fraction, reinforcement orientation, fiber nature (permeability, polarity, density, etc.), area of exposed surfaces, diffusivity, and surface protection. Moisture penetration into the composite materials governed by one major mechanism, namely, diffusion [5]. This mechanism involves the direct diffusion of water into the matrix and, to a much less extent, into the fibers. The other common mechanisms are capillarity and transport through microcracks and voids. The capillarity mechanism involves the flow of water molecules along the fiber/matrix interface, followed by diffusion from the interface into the bulk resin. Transport of moisture by microcracks and voids involves both flow and storage of water in the microcracks, and other forms of microdamage [5,6].

Long term exposures to moisture of carbon/epoxy laminates were conducted to determine diffusion coefficients and have been reported [7]. The use of accelerated moisture conditioning of graphite/epoxy composite and the comparison with regular conditioning was discussed [8–10].

The strength of the interface determines how much of the applied stress can be transferred to the load-bearing fibers. This strength is largely determined by the contact area between fibers and matrix and the level of adhesion at the contact points. However, it is not clear how the interface should be tailored to optimize composite properties [11–13].

Although many investigators have studied epoxy systems reinforced with various types of fibers and fillers [14,15], there exists the capacity for further investigations. The adhesive bonding between fiber and matrix is very important as it controls the interface profile in terms of voids and pores, which can greatly affect the absorption according to the mechanisms previously discussed. Moreover, a wide scatter of T_g values for given epoxy systems is reported in the literature [15–17]. Frequently, the variation in T_g is explained by differences associated with the material preparation. However, from experimental data in the literature, the effects of hygrothermal history (*i.e.*, exposure to moisture and temperature) on T_g are also quite significant.

This work presents measurement of diffusion parameters and the dynamic mechanical behavior of composites manufactured with prepregs of carbon fiber fabric/epoxy. Moisture absorption was correlated to the fracture mode of the laminate, demonstrating the deleterious effect of moisture on the interface. This leads to debonding between fiber and matrix. This behavior was investigated by scanning electron microscopy and dynamic mechanical analysis. Moreover, the effect of cyclic variation of temperature and humidity on interlaminar shear stress (ILSS) is also studied as most of the aerospace structures are subjected to such conditions.

EXPERIMENTAL

Materials

Prepreg was manufactured with carbon fabric style 584 (Hexcel S.A.S., Vippeuobanne, France, 8 Harness satin, 3k carbon, 370 g/m^2) and epoxy/amine resin LY564/HY2954 (Ciba-Geigy, Vautico S.A.S., Nanteore, France) LY564 is the base resin of a bisphenol A epoxy containing a reactive diluent. HY2954 is a hardener of 3,3′-dimethyl-4,4′-diaminodicyclohexyl methane. Mix ratio LY564: HY2954 (parts by weight) was 100:35.

Laminate Processing and Environmental Conditioning

The quasi-isotropic $[0°/\pm45°/90°]_s$ laminate prepared with carbon/epoxy prepreg was cured in an autoclave at 177 °C with pressure of 1 MPa by the vacuum bag method. The fiber volume fraction, V_f, of all the laminates was $70 \pm 2\%$. Test specimens were subjected to exposure at three different conditions *viz.* 60°C/85% RH, 60°C/95% RH, and 60°C/100% RH (immersion).

Moisture Absorption

The moisture absorption of carbon/epoxy laminate was measured according to ASTM D 5229/D5229M-92. All samples were cut in the dimensions of $24 \times 6 \times 2$ mm. Nine specimens were submitted to hygrothermal conditioning in a climatic chamber. Environmental chamber conditions were set as specified. The size and weight of the specimens were measured as a function of time. All specimens were dried according to ASTM C562-85 before hygrothermal conditioning. Specimens were allowed to cool down for a short period of time before weighing. The effect of removing the specimens from the chamber for a short period of time on the measurement of weight gains was seen to be negligible. The specimen weight was measured using a balance with 0.1 mg accuracy. This procedure was repeated for 9 weeks until the limit of saturation (constant weight) was reached. Some specimens were removed before saturation to yield laminates with intermediate levels of moisture absorption [18]. To ensure the removal of excessive surface (superficial) water, specimens were carefully wiped dry using clean, lint-free tissue.

The moisture gain percentage, $M_{gain}\%$, is determined from [19]:

$$M_{gain}\% = \left[\frac{(W_{wet} - W_{dry})}{(W_{dry})}\right] \times 100, \tag{1}$$

where W_{wet} is the weight of damp specimen (g) and W_{dry} is the initial weight of dry specimen (g).

Dynamic Mechanical Analysis (DMA)

Dynamic mechanical analysis (DMA) of various specimens was carried out on a DMA 2980 (TA Instruments, Newark, DE, USA). They were cut into small pieces using a diamond cutter and machined using a mechanical grinder to maintain the specified sample dimensions. The width of the samples was 12 mm and span length to thickness ratio was 10. The test was carried out according to ASTM D4065-01 [20]. The tests were run in a single cantilever beam mode with a frequency of 1 Hz and amplitude of 15 μm. The temperature was ramped from 30 to 300°C at a rate of 3°C/min. This heating rate was particularly maintained throughout the test runs so that there is a minimum temperature lag between the sample and the furnace environment.

Cyclic Humidity Variation at Constant Temperature

The specimens were initially subjected to an environment characterized by 85% RH and 60°C temperature for a period of 1 h. Then they were exposed to a second environment characterized by 40% RH and 60°C temperature for another 1 h interval. Then, 23 humidity cycles were carried out within a time period of 24 hours.

Cyclic Temperature Variation at Constant Humidity

The specimens were initially subjected to an environment characterized by 85% relative humidity (RH) and 70°C temperature for a period of 10 min. They were then exposed to a second environment characterized by 85% RH and 30°C temperature for another 10 min. interval. Finally 143 thermal cycles were carried out within a time period of 24 hours.

Inter Laminar Shear Strength (ILSS)

The interlaminar shear test (short beam shear test) was performed according to ASTM D2344/D2344M-00 [21]. Specimens were tested in order to assess the effect of environmental conditions on the ILSS. The tests were performed on an Instron model 4482 universal testing machine (flex fixture with short beam shear heads, Instron, Canton, MA, USA). The test specimens were machined from flat, finished composites. The dimensions of the sample were $24.0 \times 6.35 \times 4.10\,\mathrm{mm}$ (length × width × thickness). The thickness and width of the test specimen are measured before conditioning. The specimen is placed on a horizontal shear test fixture so that the fibers are parallel to the loading nose. The loading nose is then used to flex the specimen at a speed of 1.5 mm/min until breakage. The force is then recorded. Calculations are performed to determine shear strength by the following equation [22]:

$$ILSS = \left[\frac{0.75 \times \mathit{breaking\ load}}{\mathit{width} \times \mathit{thickness}}\right]. \tag{2}$$

Microstructural Analysis

Scanning electron microscopy (SEM) was employed to characterize the fracture surfaces of dried and damp specimens of carbon/epoxy laminates submitted to the interlaminar shear tests. The distribution, shape, and location of voids and fiber/matrix interface in the samples before and after mechanical tests were examined, using a model 982 FE-SEM (Leo Electron Microscopy Inc., Thornwood, NY, USA), with vacuum level control. No metallic coating was necessary on the samples.

RESULTS AND DISCUSSIONS

Determination of Maximum Moisture Contents, M_m, and Diffusivity Coefficient, D

Figure 1 shows plots of % moisture absorption *vs.* sq. root of time in hours for test specimens of carbon/epoxy conditioned at 60°C/85% RH, 60°C/95% RH, and 60°C/100% RH (immersion). Only those data points are shown which fit well to avoid vagueness. The nature of the curves indicate a Fickian diffusion pattern for the initial portion and thereafter remain concave to the time axis until maximum moisture content (M_m) is reached. Generally, Fickian diffusion takes place at low temperatures and for materials exposed to humid air. Deviation from Fickian behavior occurs at elevated temperature and for materials immersed in liquid. Hence, Fickian diffusion is a reasonable approximation in this case, which is evident from the figure.

Diffusivity values for all specimens were calculated, using the equation:

$$D = \pi\left[\frac{h}{4M_m}\right]^2\left[\frac{(M_2 - M_1)}{(\sqrt{t_2} - \sqrt{t_1})}\right]^2, \tag{3}$$

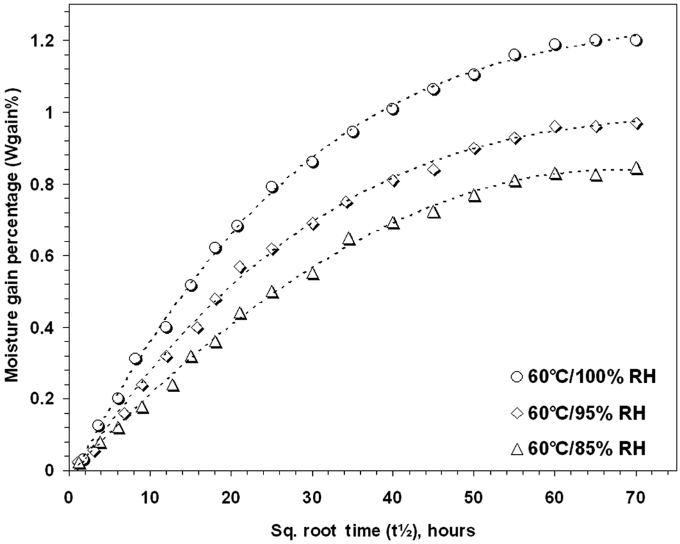

FIGURE 1 Moisture gain, % as a function of square root of time in hours.

where D is diffusivity h is the plate's thickness and M_1 and M_2 are moisture contents at times t_1 and t_2.

The average D values obtained are given in Table 1. Incomplete curing of the epoxy resin has an adverse effect for this sort of study, as secondary curing during hygrothermal exposure may render experimental observations ambiguous, promote misinterpretation of results, and may alter the value of D significantly. Moisture absorption for the cured neat resin after 10 days immersion in 23°C water was reported to be 0.23% (from Ciba-Geigy data sheet). The maximum moisture content is a strong function of the RH of the ambient. For a material immersed in liquid, M_m is a constant. For a material exposed to humid air, M_m depends on the relative humidity (ϕ), according

TABLE 1 Diffusion Parameters for Dry and Damp Specimens at Specified Conditions

Test conditions	Mm (%)	D (mm^2/sec)
60°C/85% RH	0.84	1.60×10^{-7}
60°C/95% RH	0.97	2.45×10^{-7}
60°C/100% RH (immersion)	1.2	2.87×10^{-7}

to the relationship:

$$M_m = a(\varphi)^b \tag{4}$$

$$\log M_m = \log a + b \log(\varphi). \tag{5}$$

From the data obtained at two different conditions *viz.*, 60°C/85% RH and 60°C/95% RH, "a" and "b" were evaluated for the above equation as a = 0.944 and b = 0.948.

The variation of M_m with %RH at constant temperature for this carbon/epoxy system can be estimated:

$$M_m = 0.944(\varphi)^{0.948}. \tag{6}$$

Dynamic Mechanical Analysis

From the test data, storage modulus, which gives the dynamic elastic response of the samples; loss modulus, which gives the dynamic plastic response of samples; and tan delta, which is the ratio of loss modulus/storage modulus, were determined. Figure 2 shows the curves of dynamic mechanical analysis that present similar behavior with differences in the magnitude of tan delta values that depend on the

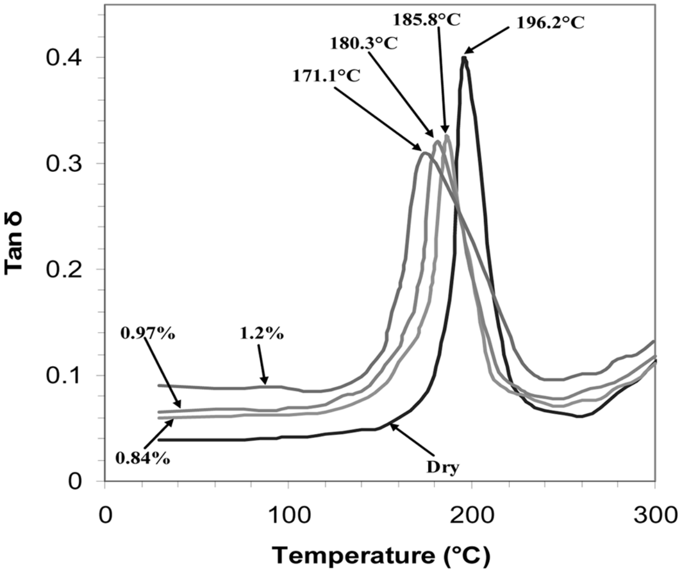

FIGURE 2 Tan δ as a function of temperature for dry and damp specimens.

polymeric matrix and the moisture absorbed during the hygrothermal conditioning.

Tg measurements can be scattered because, among other reasons, the glass transition is actually a temperature range within which long range molecular motion is activated by the temperature increase. In a dynamic mechanical experiment several characteristic temperatures in the transition region may be arbitrarily defined as T_g [23–25]. These include the onset temperature of the characteristic drop in modulus, the inflection temperature of the drop in modulus, the onset temperature of the characteristic damping peak, and the temperature of maximum damping. The problem is more complex in the case of damp polymer samples with high T_g values because the samples lose moisture to a significant extent in the glass transition region where the molecular mobility and the moisture diffusion are enhanced [26]. Thus, there is probably a gradient of moisture concentration through the samples in the T_g region and a corresponding distribution of temperature dependence of the molecular relaxation times. For this reason, in the present work, it was chosen to assign T_g to the maximum peak of tan delta for all samples.

The glass transition temperature for cured neat resin was 174°C (for the aforesaid formulation). It can be observed in Figure 2 that the samples submitted to hygrothermal conditioning showed a reduction in T_g. The curve shift increases with the increase of the moisture levels. The moisture level leads to different changes in T_g depending on the particular characteristics of the molecular structures and matrix/fiber interface interactions of the matrix system, so the behavior studied and changes in the distribution of relaxation times associated with the α-transition (T_g) could be attributed to the plasticization effect. Other possible explanations for the change in the distribution of relaxation times involves a specific evolution of the macromolecular network at the fiber/matrix interface and/or debonding during the hygrothermal conditioning. Although it is not possible to identify these processes from a chemical point of view, it is possible to conclude that these processes affect the interface sufficiently to modify the relaxation mechanisms associated with the α-transition. The movement of the polymeric chains is more difficult for higher interface adhesion. Therefore, the interface adhesion increases the T_g. Since the environmental conditioning reduces the T_g, it is expected that the interface adhesion will also be reduced. This hypothesis is corroborated by interlaminar shear strength measurements (an indirect measurement of interface adhesion) that show a decrease with moisture absorption.

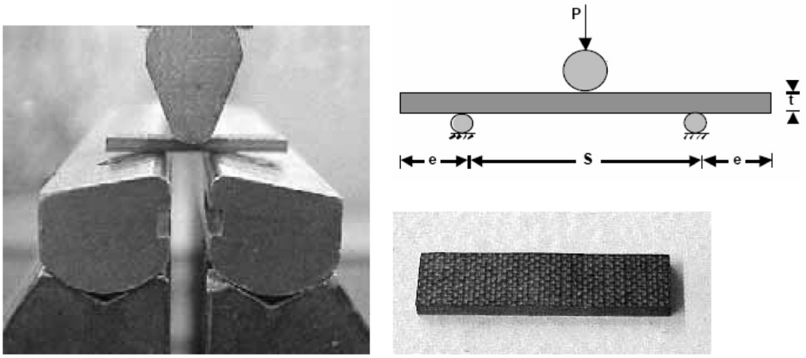

FIGURE 3 Specimen and test setup for ILSS.

Interlaminar Shear Strength (ILSS)

The effect of the moisture content on the laminate strength was assessed by interlaminar shear strength (ILSS) measurements of the dry and hygrothermally conditioned carbon/epoxy specimens. Specimens with dimensions $24 \times 6.35 \times 4.1\,\text{mm}$ were tested according to ASTM Standard D2344/D2344M-00 [21] (Figure 3).

Figure 4 presents ILSS values of specimens subjected to environmental conditions which are labelled according to the percentage of absorption water. These values reported are the average of three tests.

Figure 4 shows a decrease of 13% in the interlaminar shear strength for the laminates subjected to 60°C/100% RH (immersion). The water absorbed by the carbon/epoxy laminates, in general, causes reversible plasticization of the matrix and lowers its glass transition temperature. Combined with temperature effects, these factors cause significant changes in the matrix toughness, affecting the laminate strength. Moreover, humidity and temperature cause dimensional changes and induce stresses in the laminate that diminish the fiber matrix interface adhesion.

Microscopic Analysis

The effects of moisture on the fracture surfaces of the specimens were examined by scanning electron microscopy (SEM). Figure 5(a) depicts the fracture surface of a fractured interlaminar shear strength dry specimen of carbon/epoxy laminate. The smooth clean surface of fibers is caused by a fracture micromechanism involving an interfacial debonding in the carbon/epoxy fabric laminate. Cracks through a fiber tow, either in the warp or fill direction, can also be identified as a fracture micromechanism. The crack runs along the matrix between

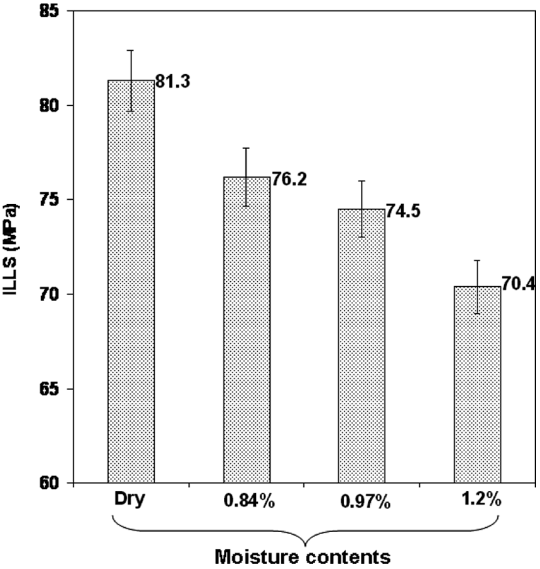

FIGURE 4 ILSS test results for dry and damp specimens.

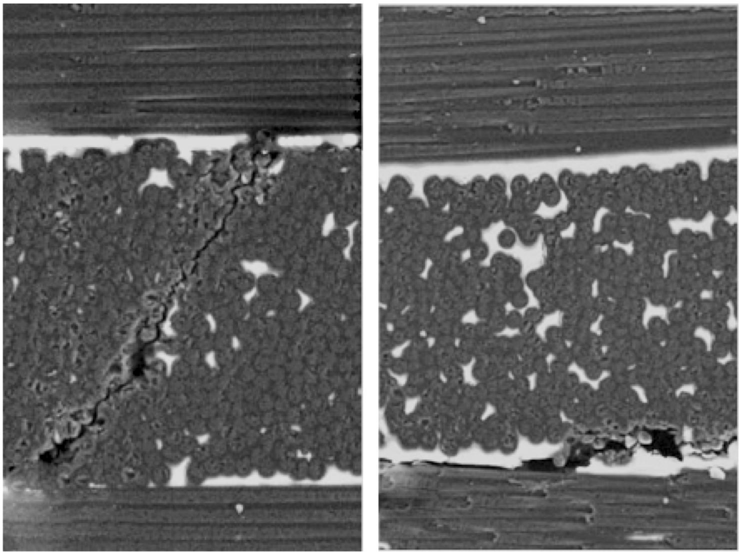
(a) (b)

FIGURE 5 SEM fractograph of carbon/epoxy laminate (a) Dry; (b) 1.2% absorbed moisture.

fibers. This feature suggests a brittle fracture micromechanism. In this material (carbon/epoxy), the microcracks initiated in the matrix at the fiber/matrix interface, growing under the action of shear loads along planes parallel to the loading direction. The growth and coalescence of these microcracks can lead to a primary crack.

Figure 5(b) shows the fracture surface of carbon/epoxy laminate conditioned at 60°C/100% RH, with 1.2% moisture content, with a fracture micromechanism to that for the dry specimen. However, the moisture absorption induced an increase of the interfacial debonding area. The effect of debonding along the warp/fill yarn interface supersedes the effect of interfacial (fiber/matrix) debonding. Large debonding along resin rich areas resulted in significant decrease of the material toughness. This could not be regarded as ply-ply failure within the laminate as the same sort of resin rich area is equally visible in figure 5(a) where no fracture is observed after similar test. So the fracture in figure 5(b) may be ascribed to moisture affected epoxy resin at the interface.

Moisture penetration through the fiber/matrix interfaces causes also interfacial debonds resulting in the rupture or degradation of the interface, exposing the carbon fibers. The absorption of the water molecules accelerated by temperature used in the hygrothermal conditioning weakens the fiber/matrix interface exposing the fibers. The sorbed moisture acts as a plasticizer and a swelling and solvent crazing agent. When the specimens are removed from the environmental chamber in the beginning of the hygrothermal conditioning process, and submitted to the ILSS test, the fibers provide some lateral restraint in the transverse direction of the polymer composite specimens, but they do not restrain the swelling of the matrix along the loading direction. This effect is reflected in very low longitudinal deformation in the polymer composite specimens. This restraining effect is necessarily offset by internal stresses within the matrix phase (mainly compression) and along the fiber/matrix interfaces (shear). The swollen matrix forms a shell around the dry matrix- and the dry matrix will be placed under a state of triaxial stresses. When the swelling stresses reach a critical value, the fracture of the matrix occurs. This mechanism can lead to the rupture of the fiber/matrix interface. Knowledge of the mechanisms that drive moisture sorption, as well as the influence of sample dimensions, temperature, and relative humidity, becomes crucial when long-term properties of the material are needed.

Cyclic Humidity Variation at Constant Temperature

Figure 6 shows the variation of ILSS as a measure of degradation due to the fluctuating environment. Each value presented is an average of

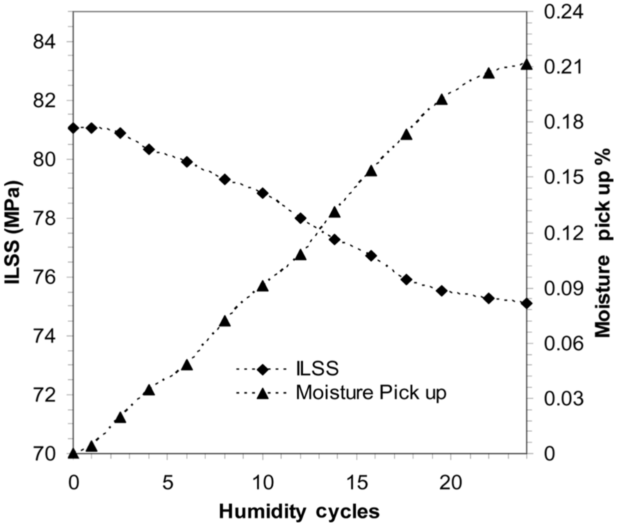

FIGURE 6 ILSS and moisture pickup as a function of number of humidity cycles.

three specimens. Experimental error is less than 3% for ILLS and up to 7% for moisture pick up.

The ILSS is seen to deteriorate over a period of time. The amount of moisture absorbed by the epoxy matrix is significantly greater than the fibers, which absorb little or no moisture. This results in significant mismatch in moisture-induced volumetric expansion between matrix and fibers, leading to the evolution of localized stress and strain fields in the composite at the interfacial region. The stress field existing in a hygrothermally conditioned composite adversely affects the interfacial bond, thereby resulting in de-adhesion. With increased moisture uptake, the nature of stresses during absorption and desorption have a more significant impact leading to a slow, but steady, fall in the ILSS values. As the moisture transport kinetics saturate, the stresses at the interface, being governed by moisture pickup, also stop fluctuating. This is reflected by the gradual leveling of the ILSS values at longer conditioning times.

Cyclic Temperature Variation at Constant Humidity

During thermal cycling, both matrix and fiber expand or contract, according to their coefficients of thermal expansion. As they were internally constrained, temperature fluctuations cause stress

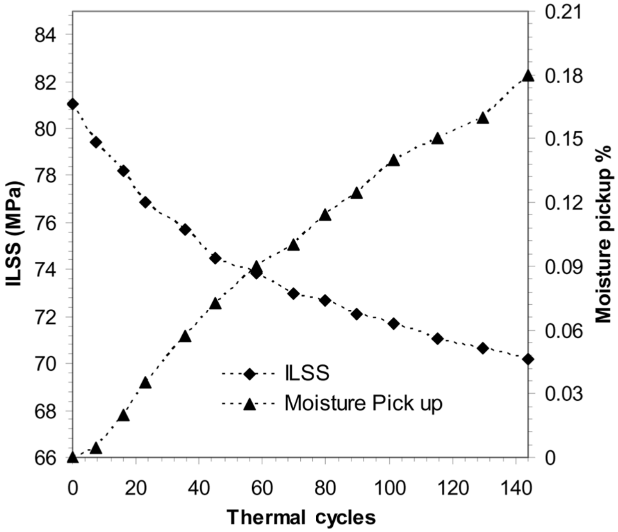

FIGURE 7 ILSS and moisture pickup as a function of number of thermal cycles.

build-up at the interface. These stresses might be relieved by one or more of the following means: (a) plastic deformation of the ductile matrix; (b) cracking or failure of the brittle fiber; (c) failure of the fiber/matrix interface. The third factor is the most contributing and characterized here by ILSS testing (Figure 7).

It is evident from the figure that ILSS values decrease rapidly and continue falling during the time frame of the experiment. During the thermal cycles the composite material was subjected to thermal stresses. Expansion and contraction caused by a temperature difference of 40°C resulted in a difference in the linear thermal expansion between the fiber and matrix. Therefore, shear stresses will occur and, as a result of these stresses, plastic deformations and micro cracks at the fibre/matrix and interlaminar interface are to be expected. Also, thermal gradients occur in the composite material during the heating and cooling phases, which result in high residual stresses in the plies. Similar to a fatigue experiment, deformations increase with the number of cycles. ILSS of the materials continued to decrease with an increasing number of thermal cycles. Comparision of Figures 6 and 7 shows that the thermal fluctuations have a more rapid effect on interlaminar adhesion than do humidity fluctuations.

CONCLUSIONS

Carbon/epoxy laminate specimens exhibited Fickian behavior at the different hydrothermal conditions studied. The maximum moisture content increased with the increase in relative humidity. The diffusivity remains practically the same for all hydrothermal conditions considered. It can be observed that the samples submitted to hygrothermal conditioning presented a reduction in T_g that increases with the increase of the moisture levels. That is, the moisture level leads to different changes in T_g that depend on the particular characteristics of molecular structures and matrix/fiber interface interactions of the system. This study demonstrated that 1.2% absorption of moisture lowered the T_g by as much as $\sim 25°C$.

It is also observed by SEM that the moisture penetration along the fiber/matrix interfaces causes interfacial debonds, leading to the rupture or degradation of the interface. The absorption of moisture accelerated by temperatures used in hygrothermal conditioning weakens the fiber/matrix interfacial adhesion, exposing the carbon fibers.

Humidity fluctuations at constant temperature affects the ILSS corresponding to moisture take up, which was fast at the start but subsequently leveled off. The enhanced moisture pickup is responsible for reducing adhesion at the fiber–matrix interface, resulting in lower ILSS values.

The residual stresses induced at the fiber/matrix and interlaminar interface as a result of thermal cycling lowered the ILSS and flexural modulus. The percentage decrease in ILSS at the end of the thermal fluctuations experiment was 3.5%. ILSS of the materials decreased with an increasing number of thermal cycles.

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