Influence of Environmental Conditioning on the Shear Behavior of Poly(phenylene sulfide)/Glass Fiber Composites

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ABSTRACT: Advanced thermoplastic composites are an alternative because of their ease of processing and storage. Poly(phenylene sulfide) (PPS) stands out among these materials because of its structural characteristics; for instance, it provides size, shape, and thermal stability, low moisture absorption, excellent chemical resistance, and good mechanical properties, including flexure, strength, and shear properties, compared to thermoset composites. Thus, the objective of this study was to evaluate the influence of environmental conditioning on the shear strength behavior of PPS/glass fiber composites. For this reason, first, some samples were treated to UV-light exposure in a chamber. Other samples were immersed in seawater and hygrothermal baths simultaneously. They were tested with the interlaminar shear strength (ILSS) and Iosipescu shear test methods. The shear values obtained for the treated samples were compared against the dry sample values. For all samples tested with the ILSS and Iosipescu methods, the results indicate that the PPS/glass fiber composites presented a decrease in shear strength after they were submitted to hygrothermal and seawater solution conditioning. The moisture absorption was not uniform throughout the material, and wet conditioning induced strong matrix plasticization, which reduced the shear strength values of the laminates. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 118: 180–187, 2010

Key words: composites; high performance polymers; mechanical properties

INTRODUCTION

In recent years, thermoplastic composite laminates have been reinforced with glass, carbon, and aramid fibers, and they have gained popularity in marine, automobile, and other engineering applications.[1–3] When compared to thermoset-fiber-reinforced composite laminates, they are easier to process, as they do not require complex chemical reactions; they can be created without a lengthy curing process; they are easily recycled; and they do not need refrigeration for storage. Furthermore, their applications are based on thermoplastic polymer engineering matrices, such as thermoplastic polyesters and polyamides, that exhibit good mechanical properties, size and shape stability, and strength.[4–7]

Furthermore, for certain applications, in addition to the high physical properties and low density provided by commonly used thermoplastic composites, some additional properties are required that cannot be met, such as polymer stability in corrosive environments and uninterrupted use at high temperatures (>150°C). For these situations, high-performance thermoplastic matrices based on a large quantity of aromatic rings in the main chain have been developed, such as certain polyurethanes, polyimide, poly(ether ether ketone), polysulfone, and poly(phenylene sulfide) (PPS).[7–11]

These and other aromatic high-performance polymers improve the mechanical properties when compared to the thermoset matrix; however, the high costs and high processing temperatures associated with them limit their applications to only those fields where prices have little importance, such as in military aircrafts and space vehicles. In particular, PPS has been considered as an alternative between the cheapest commodity and the high-performance polymers because of its price and properties.[8–11]

PPS exhibits intermediate mechanical properties combined with temperature tolerance and a resistance to chemical corrosion triggered by organic solvents, inorganic salts, and bases, which is affected by aircraft fluids.[7–10] These properties are mainly due to the atoms of sulfur being interwoven with aromatic rings synthesized from a reaction of paradichlorobenzene

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with sodium sulfide. Also, the phenol group imposes restriction to the movement of polymer chains, and its reaction produces linear highly crystallized homopolymers in the range 50–60%; this happens very rapidly at temperatures above its glass-transition temperature.[8–12]

The use of the PPS polymers in structural components demands the reinforcement of continuous fibers, such as carbon, aramid, or glass fibers, depending on the workload required. Among its applications, glass fiber is used when a light weight is needed in conjunction with good mechanical and electrical resistances, as in aircraft radomes, or resistance to galvanized or chemical corrosion and to the absorption of humidity, such as in offshore pipes.[13–15]

In comparison, glass fiber has a smaller elasticity modulus, tensile properties, and thermal expansion coefficient than carbon and aramid fibers, and it is widely used, especially because of its very attractive cost benefit and mass production throughout the world.[13–15]

In this case, glass fiber serves as a reinforcement of PPS polymers and provides high performance and low cost to thermoplastic composites and allows some application in corrosive and high-temperature environments as a good substitute to thermoset composites, such as epoxy/glass fiber composites in structural components. Because of this increased application, the transfer of properties between PPS and glass fiber is very important to structural projects. It is known that glass fiber has organic functional groups in its surface, which interact with the polymeric structure of the matrix phase; therefore, the bonding in PPS/glass fiber may be a combination of mechanical, chemical, and/or electrostatic interaction.[16,17]

One of the ways to evaluate the quality of bonding is through mechanical shear tests, such as interlaminar shear strength (ILSS), translaminar or in-plane shear strength, and transverse stretch strength tests. They must be simple enough to carry out, require small and easily made samples, and enable the measurement of very reproducible values for both the shear modulus and shear strength with a simple given procedure.[17–19]

One of the most used shear tests method is the v-notch shear test, which allows a nearly pure shear stress state at the shear plane.[20–23] This test was originally proposed by Iosipescu to determine the shear properties of isotropic materials. The test uses flat samples that are easy to fabricate and achieves a pure and uniform shear stress–strain state over the sample region tested. Many numerical and experimental investigations on the application of the v-notch shear test to different composite material systems have been carried out.[20–23]

Mechanical tests can also be used to evaluate the environmental influence in composite materials. Environmental influence is commonly considered to be responsible for failures of these materials; this results from a combination of the effects of heat, light, water, and mechanical stresses on the material.[24–27] Several studies have addressed the important effects of absorbed water and aging temperature on the physical and mechanical properties of composite materials. It has been observed that mechanisms other than simple diffusion can take place within the material above a threshold related to a given temperature and a given aging time.[28–30]

Both UV radiation and moisture have adverse effects on the mechanical properties of polymeric matrix. The polymer matrix in a fiber-reinforced composite helps to transfer applied loads to the reinforcing fibers and provide ILSS, whereas the fiber–matrix interface governs the load-transfer characteristics and damage tolerance. Thus, both components (matrix and interface) represent weak links in fiber-reinforced composites and upon degradation. They lead to reduced damage tolerance and long-term durability.[31–35]

For this reason, the focus of this article is to report the influence of hygrothermal and UV effects on the shear strength properties of PPS/glass fiber laminates. To evaluate this, samples were evaluated by Iosipescu and short-beam shear tests, followed by exposure to hygrothermal, seawater, and UV conditioning.

EXPERIMENTAL

Materials

Composite material

The PPS/glass fiber composite was supplied by Ten Cate Advanced Composite Materials (The Netherlands, Vijverdal). A laminate plate, 2.3 mm thick, was made with poly(phenylene sulfide) and a 60% volume content of glass fiber in 8HS fabric layers with an orientation of superposition of 0/90.

Sample preparation

Samples were machined with an appropriate circular saw. The dimensions were specified from ASTM D 2344 and ASTM D 5379 for use for the short-beam shear test (ILSS) and Iosipescu test, respectively.

Methodology

Evaluation of the fiber volume fraction

The composite laminate consolidation quality was evaluated by an optical microscope from Olympus BH (Tokyo, Japan) to detect the existence of manufacture-induced defects.

The fiber and matrix contents were determined with an acid digestion procedure, according to ASTM D 3171. The composite sample was weighed and then immersed in a hot sulfuric acid solution to remove the polymer matrix PPS.

Afterward, the remaining residue, containing the composite glass reinforcement, was filtered, washed, dried, cooled, and weighed. The weight percentage of the reinforcement was expressed in volume fraction, according to eq. (1):

$$\frac{m_m}{m_f} = \frac{\rho_m}{\rho_f} \times \left(\frac{1-f}{f}\right) \quad (1)$$

where m_f and m_m are weights of the glass fiber and the matrix (g), respectively; ρ_f and ρ_m are the densities of the glass fiber and the matrix (g/cm^3), respectively; and f is the fiber volume fraction (%).

Hygrothermal conditioning

Samples destined for hygrothermal conditioning were dried in a vacuum oven at 80°C for at least 24 h before testing. This procedure was done to take the weight of the material molded without the influence of atmosphere humidity. After this procedure, five samples for each shear test method were weighed and immersed in hot water with a controlled bath temperature of 80°C and maintained under these conditions for 8 weeks (Elevated Temperature Wet (ETW) conditioning). This process was based on procedure B of ASTM D 5229 M-92. The moisture level in the laminates was periodically monitored as a function of time by measurement of the mass of the traveler samples up to the moisture equilibrium state reached a steady state. Testing under room-temperature conditions was designated as dry-room-temperature testing.

Seawater conditioning

PPS/glass fiber samples were conditioned in a salt water bath (seawater). The environmental conditioning by seawater immersion was conducted according to ASTM D 1141-98. The pH of the salt solution was adjusted to 8.2, similar to the pH of seawater. Five samples for each shear test method were immersed in a recipient container containing seawater solution. In this test, the exposure time was 30 days.

UV conditioning

To reproduce damages resulting from UV radiation and water condensation, five samples for the shear test method were placed in a weathering chamber at exposure conditions of 300, 600, and 900 h. The degradation mechanisms were determined in accordance with ASTM G 154, which describes the test methodology for a Ultraviolet radiation weathering chamber (QUV/Se; Q-Panel Lab Products, Cleveland, OH).

According to this standard, damages caused by sunlight, rain, and dew were reproduced by cycles of periods of 8 h under UV-B light and 8 h under water condensation provided by the generation of vapor from a water bath.

The radiation, generated by eight fluorescent UV lamps, had a wavelength in the region 295–365 nm, which corresponded to the UV component of solar radiation. The intensity of the light emitted was constantly monitored by four Solar-Eye radiation detectors, calibrated for every 400 h of service with the objective of evaluating the quality of the lamp.

Mechanical tests

The interlaminar short-beam test method (ILSS) and the Iosipescu test method were conducted according to ASTM D 2344 and ASTM D 5379, respectively. All tests were conducted in an Instron test machine (Norwood, MA) with a test speed of 0.5 mm/min and a load cell of 500 kgf.

To obtain the shear modulus, it was necessary to assume that both stress and strain were uniformly distributed across the test area of the sample used. In this case, the apparent in-plane shear strain (γ) at the cross section along two notch tips was obtained by:

$$\gamma = \varepsilon_{+45^\circ} - \varepsilon_{-45^\circ} \quad (2)$$

where: γ = shear strain, $\varepsilon_{+45^\circ} = +45^\circ$ normal strain, $\varepsilon_{-45^\circ} = -45^\circ$ normal strain.

In this study, the shear strain was measured by the bonding strain gages at ±45 placed at the midsection between the two notch tips. Because of difficulties in fixing the strain gauges in wet specimens, it was not possible to obtain the shear modulus of the samples after they had been submitted to hygrothermal conditioning.

RESULTS AND DISCUSSION

According to the acid digestion method, it was possible to observe that the reinforcement content was 61 ± 6 vol %, which is typical for an advanced composite.

Figure 1 illustrates a typical cross section of a 0/90 glass fiber composite viewed from optical microscopy. In this case, a good infiltration of the polymer into the reinforcement was observed; this produced a homogeneous thermoplastic composite with a low porosity. In fact, the presence of pores was not clearly visible, and the manufacturing-induced

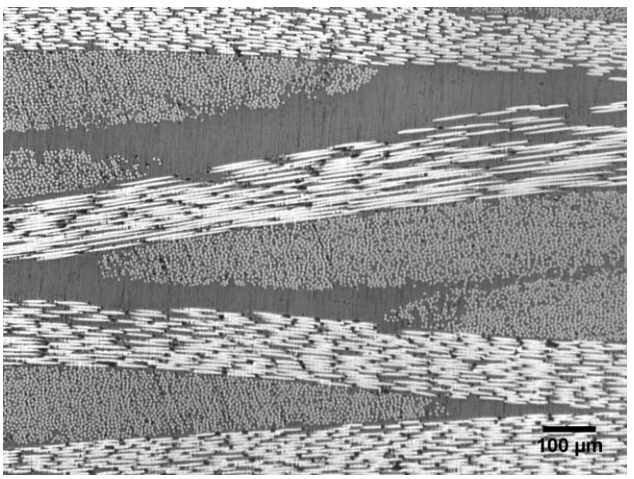

Figure 1 Optical microscopy of the fabric PPS/glass fiber laminate.

defects, such as a lack of adhesion reinforcement/matrix, excess of matrix phase, or presence of gaps or cracks, may have caused some inaccuracies in the mechanical measurements.

Figure 2 presents the average moisture absorption rate curves of the PPS/glass fiber laminates submerged in the seawater solution at room temperature and in distilled water at 80°C. The shapes of both curves show an initial linear region corresponding to a steady rate of moisture absorption up to a flat steady state of maximum moisture intake.

In both conditions, the glass-fiber-reinforced material reached this flat steady state of saturation in 30 days. During this time, the material exhibited increases in weight of approximately 0.3 and 0.7% for samples submerged in the seawater solution and hot water, respectively. Therefore, the greater absorption of humidity by the material was in hot distilled water at 80°C, and this value was very small when compared with the epoxy/glass fiber composites in the same state (ca. 2.5%[33]).

These results depended on the kinetic diffusion process of the temperature and relative moisture absorption. In other words, the absorption rate was greater when the temperature was higher than the relative moisture absorption.

At the same time, this phenomenon took place through the transportation of water molecules from areas of high concentration to areas of lower moisture concentration. For this reason, the moisture absorption coming from the atmosphere usually occurs by a diffusion process from the surface up to the core of the material.[33] On the other hand, moisture can also be transported through the material by the capillary action provided by the presence of microcavities at the fiber–matrix interfaces.

After this diffusion process, the absorption became slower because of the relaxation of polymeric chains and the filling process of voids and cavities. As a result, the absorbed moisture induced the linkage by hydrogen bonds between the polymer matrix and the water or other molecules; this, thus, changed the shear mechanical properties.

Figure 3 shows the gradual color change of the composite material surface after exposure to UV light for 300, 600, and 900 h. This change established that photooxidation resulted in the formation of chromophoric chemical groups, which absorbed the visible range of light. As shown in Figure 3, minor changes on either the surface or edge roughness caused by UV radiation were also observed by the naked eye.

Further details regarding the physical processes that caused material degradation were revealed by examination of the samples under an optical

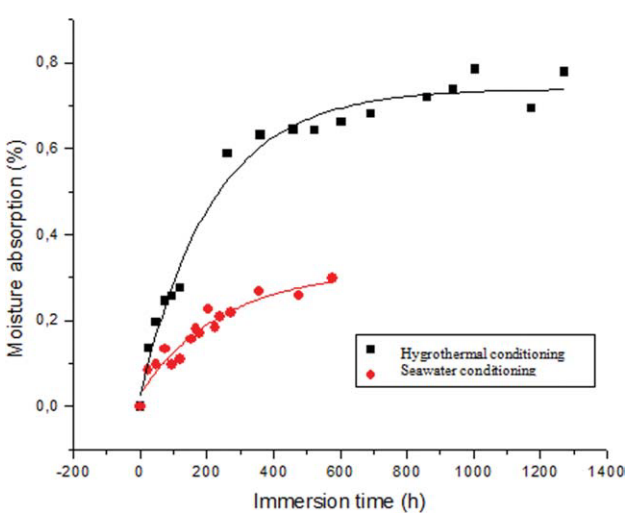

Figure 2 Hygrothermal conditioning of the PPS/glass fiber laminate. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

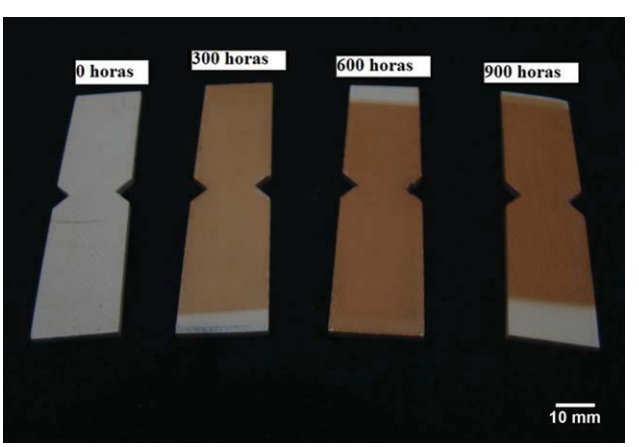

Figure 3 Change in the sample color as a function of time for UV radiation exposure. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

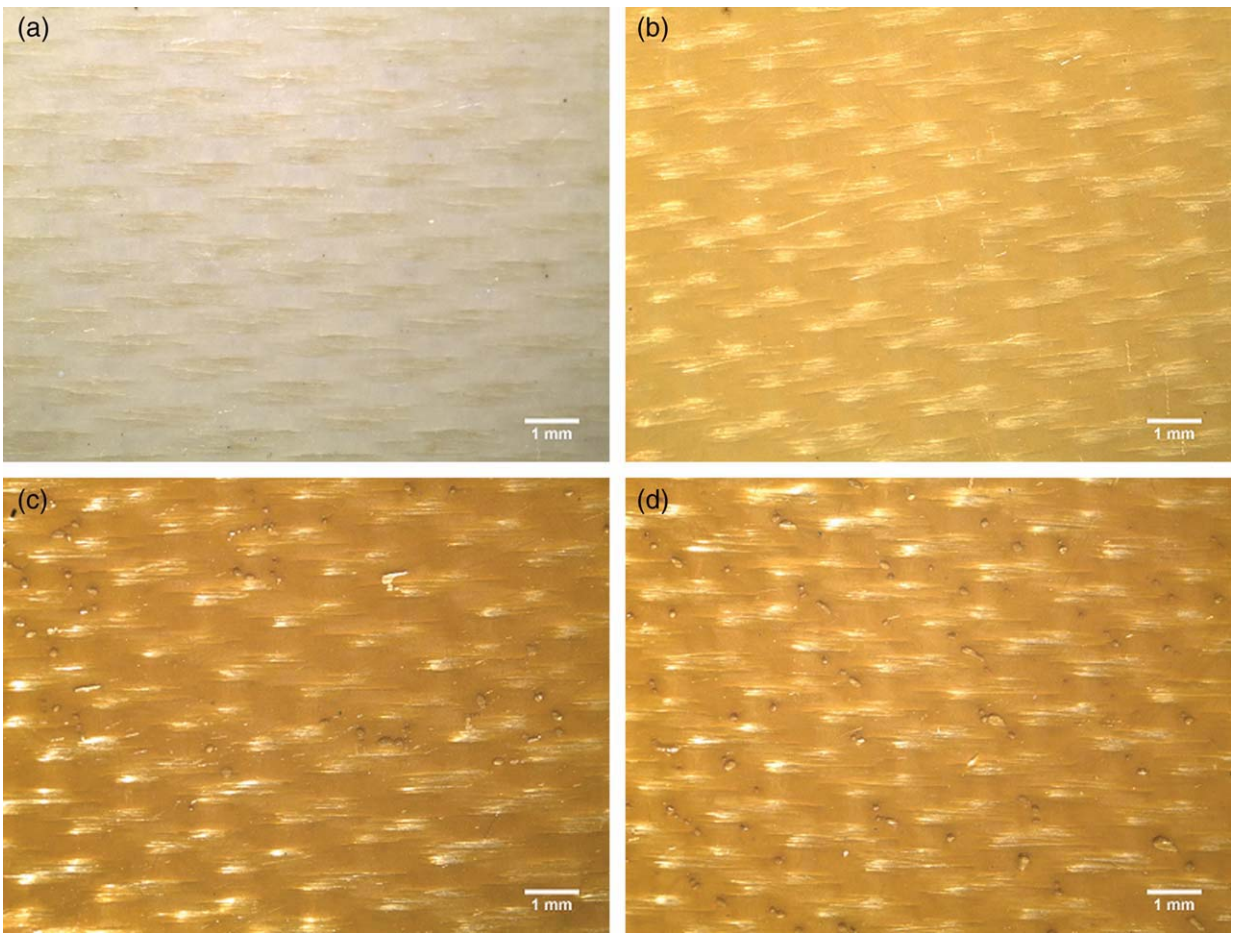

Figure 4 Morphological evaluation for the samples submitted to UV radiation exposure: (a) no exposure and (b) 300, (c) 600, and (d) 900 h of exposure. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

microscope, such as that shown in Figure 4. These higher magnified images revealed the formation of microcracks in the PPS matrix. This phenomenon was caused by the polymer matrix becoming excessively brittle because of the increased crosslinking, resulting from photooxidation reactions induced by the UV radiation. This behavior was similar to that of epoxy/glass fiber composites.[31]

The mechanical behavior of the PPS/glass fiber laminate before and after the conditioning is shown in Table I. The ultimate tension values (σ_{ult}) corresponded to the results obtained through ILSS and Iosipescu testing. As shown by the general evaluation shown in Table I, the ILSS and Iosipescu values for the no-conditioning glass fiber thermoplastic laminate decreased when it was exposed to hygrothermal and saline conditioning.

The shear strength obtained through the Iosipescu method was around 97.4 MPa for the nonconditioning laminate, and the shear modulus is not presented because the environmental conditioning made the attachment of strain gauges in wet specimens difficult to achieve. Similarly, the ILSS result was around 39.9 MPa for the ILSS test, which was close or similar to results found in the literature (30.0–70.0 MPa).[27]

Therefore, we concluded that the ILSS values showed decreases of 10% (24.0 MPa) and 37% (35.2 MPa) after the samples were submitted to seawater and hygrothermal conditioning, respectively, and the shear strength showed a similar decrease in the range of approximately 17% (73.4 MPa) to 24% (80.8 MPa) for the same conditions, as shown in Table I.

It is known that, for both saline and hygrothermal conditioning, moisture intakes always induce resin

TABLE I
Results Obtained from the ILSS and Iosipescu Tests

Conditioning	ILSS σ_{ult} (MPa)	Iosipescu σ_{ult} (MPa)
Dry	39.9 ± 0.8	97.4 ± 2.9
UV (300 h)	39.6 ± 0.4	96.4 ± 1.4
UV (600 h)	39.0 ± 0.7	94.4 ± 1.8
UV (900 h)	38.9 ± 0.8	94.3 ± 1.8
Seawater	35.2 ± 1.2	80.8 ± 2.8
Hygrothermal	24.0 ± 7.9	73.4 ± 9.6

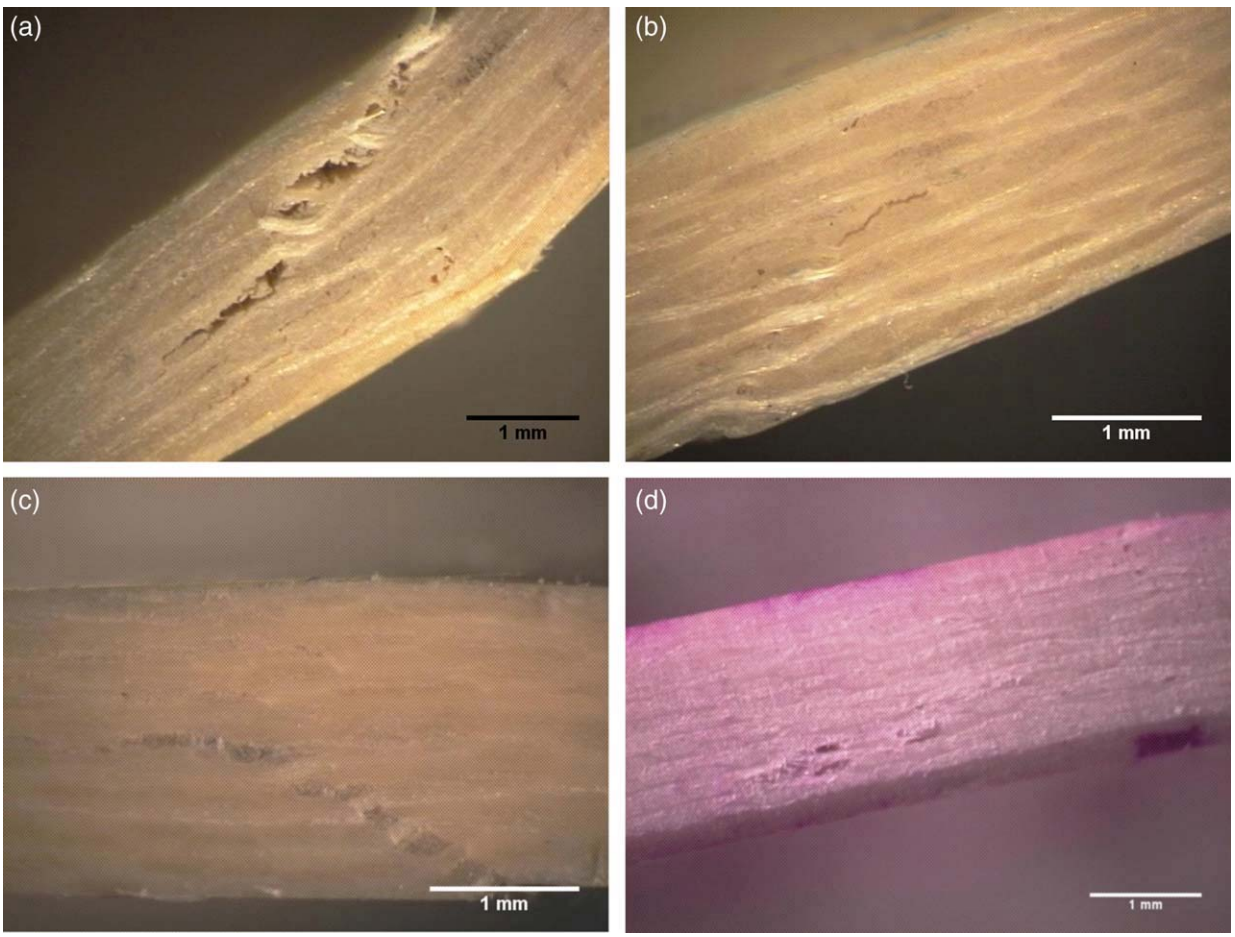

Figure 5 Representative fracture observed by the ILSS test: (a) no conditioning, (b) hygrothermal conditioning, (c) seawater conditioning, and (d) UV conditioning. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

plasticization and, consequently, reduce the shear strength values of composites. On the other hand, to evaluate the influence of both the moisture absorption and temperature effect at the same time on the mechanical properties is difficult because there is not a consensus about the magnitude of these variables.

Despite all this, evaluations of the hygrothermal effects over material are described in detail in the literature,[27] in which case, the differences between both conditions have been due to the diffusion process being accelerated with increasing temperature. Similar behavior was verified with epoxy/glass fiber composites.[31]

UV conditioning did not induce any considerable change in either shear test method under 300, 600, and 900 h of exposure, as shown in Table I. In other words, the ILSS values decreased 1% (39.6 MPa) at 300 h of exposure time, 2% (39.0 MPa) at 600 h of exposure time, and 3% (38.9 MPa) at 900 h of exposure time. We concluded that there was no significant difference between the nonconditioned material and those exposed to UV light. In fact, the same behavior was confirmed through in-plane shear strength with the Iosipescu method, with a reduction in the shear tension of only 1% for the material exposed for 300 h and 3% for those exposed for 600 and 900 h.

This small amount of actual decrease in the mechanical values was expected because neither UV radiation nor humidity condensation led to a degradation of the interface between the glass fiber and the matrix. At the same time, changes observed were due to surface phenomena; therefore, there was no molecular weight reduction of the matrix due to chain-scission reactions provoked by photooxidation from UV radiation.

Figure 5(a) presents the failure mode aspect in the PPS/glass fiber composites samples submitted to ILSS testing. This laminate exhibited multiple delaminating and interlaminar cracks at the horizontal and vertical positions. This behavior was also observed in samples after they were submitted to hygrothermal conditioning [Fig. 5(b,c)] but not to UV conditioning [Fig. 5(d)]. In this last case, the sample failure mode appeared to be compressive buckling or compressive yielding in the upper part of the beam under combined compression and shear

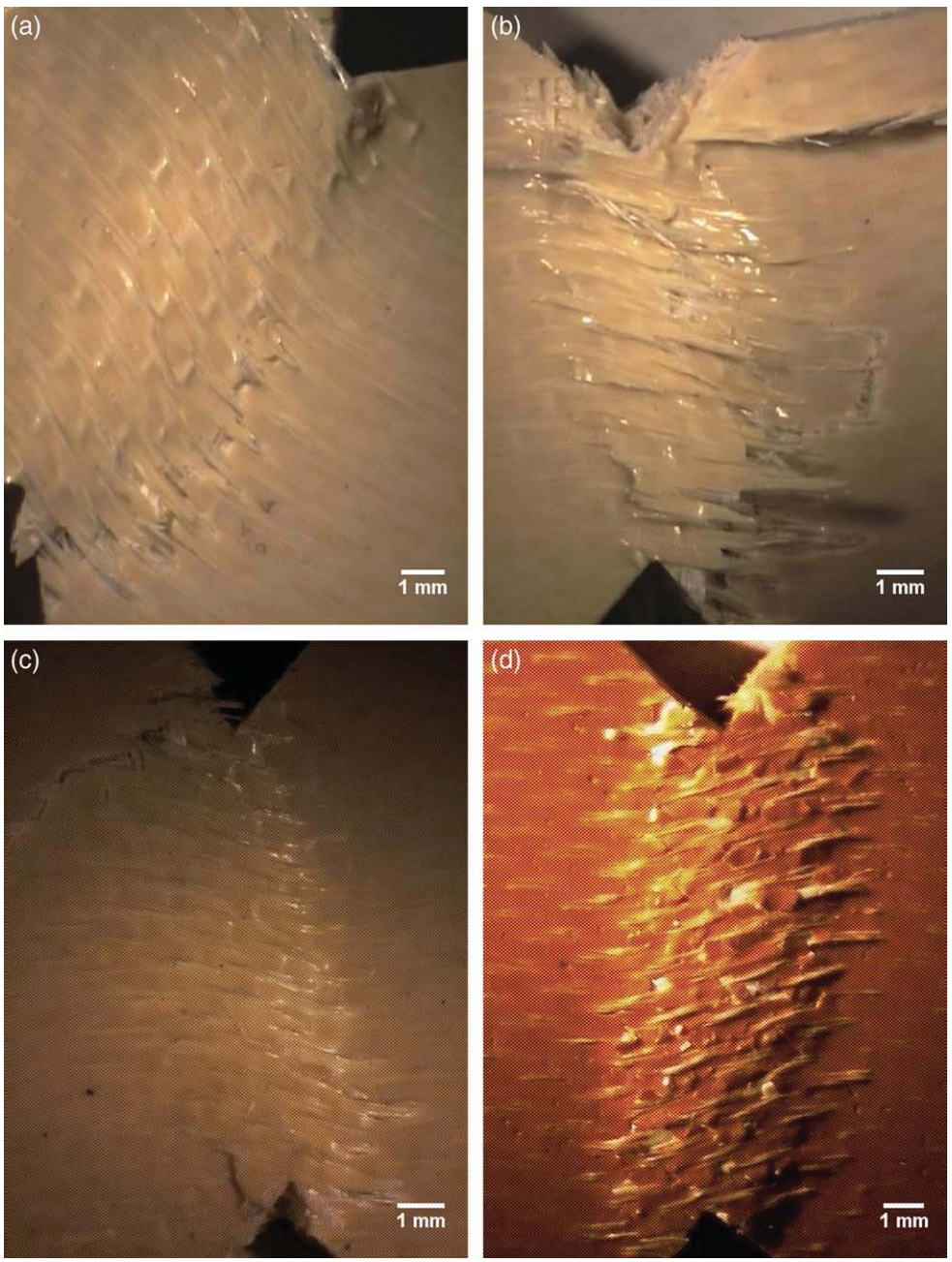

Figure 6 Representative fracture observed by the Iosipescu test: (a) no conditioning, (b) hygrothermal conditioning, (c) seawater conditioning, and (d) UV conditioning. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

efforts. Such behavior probably happened because of the increase in the ductile properties induced by UV radiation. Moreover, neither vertical nor horizontal cracks were observed.

As shown in Figure 6, the composites exhibited the same failure mode under the Iosipescu test method before and after the conditioning. In all samples, the first failure observed during loading was notch-root splitting, which started near the notch roots and propagated away from the inner loading blocks along the fibers. The splitting, in this case, was predominantly a consequence of transverse stress near the notch roots; this showed that the splits created a uniform stress distribution along the notch-root axis. Damages were also observed at the notch roots and at the inner loading blocks. The damage (crushing) caused by the loading blocks was significant at higher loads.

CONCLUSIONS

The influence of moisture on the shear properties of PPS/glass fiber composite samples was investigated.

This material absorbed approximately 0.3 and 0.7% moister in a seawater solution and hot water, respectively, after the saturation point.

For all samples tested by the ILSS and Iosipescu methods, the results indicate that the PPS/glass fiber composites presented a decrease in the shear strength after they were submitted to hygrothermal and seawater solution conditioning. The moisture absorption was not uniform throughout the material, and wet conditioning induced strong matrix plasticization, which reduced the shear strength values of the laminates.

Therefore, a small actual decrease in the mechanical value was observed from the Iosipescu and ILSS tests after the samples were exposed to UV radiation. This behavior suggests that the degradation due to UV exposure was only marginal and resulted from surface phenomena.

In this study, we also observed that the laminate submitted to the ILSS testing exhibited multiple delaminating and had interlaminar cracks at the horizontal and vertical positions before and after it was submitted to hygrothermal and seawater conditioning. However, this behavior was not observed after the exposure of the laminates to UV conditioning, probably because of the higher ductility generated by the UV radiation process. Therefore, all specimens exposed to hygrothermal conditioning, saline conditioning, and UV after going through Iosipescu testing, had similar failure modes.

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