EFFECT OF THE HYGROTHERMAL CONDITIONING ON THE MECHANICAL PROPERTIES OF CARBON FIBER/EPOXY COMPOSITES

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Abstract. *Fiber reinforced epoxy composites are used in a wide variety of applications in the aerospace field. These materials have high specific moduli, high specific strength and their properties can be tailored to application requirements. In order to screening optimum materials behavior, the effects of external environments on the mechanical properties during usage must be clearly understood. The environmental action, such as high moisture concentration, high temperatures, corrosive fluids or ultraviolet radiation (UV), can affect the performance of advanced composites during service. These factors can limit the applications of composites by deteriorating the mechanical properties over a period of time. Properties knock down is attributed to the chemical and/or physical damages caused in the polymer matrix, loss of adhesion of fiber/resin interface, and/or reduction of fiber strength and stiffness. The dynamic elastic properties are important characteristics of carbon fiber reinforced composites (CRFC). Nowadays, there are several kinds of mechanical experiments available in order to establish the mechanical properties of composite materials. The present study has been performed to explore relations between the dynamic mechanical properties (compression, tensile, shear strength and free vibration properties), damping test and the influence of high moisture concentration of carbon fiber reinforced composites (plain weave). The results show that the E', shear strength and shear modulus decrease (up to 20%) with the increased of exposed time for carbon fiber/epoxy composites specimens exposed at 80°C and 90% relative humidity (RH), during 60 days of exposure.*

Keywords: *metal/glass fiber composites; damping; behavior; elastic properties.*

1. Introduction

The high specific strength and stiffness, and good chemical resistance of carbon-fiber-reinforced polymer matrix composites make them attractive for applications in aerospace components, sporting goods, civil structures and marine vehicles (Gao and Kim, 1999). However, manufacturing defects, particularly voids, may be formed in these materials, degrading their structural performance. In polymeric composite materials, voids have been shown to reduce mechanical properties such as interlaminar shear strength, longitudinal and transverse strength, modulus, and fatigue resistance (Costa, Rezende and Almeida, 2001a, b).

Environmental effects have to be considered as the pre-impregnated can absorb moisture changing their physical and chemical properties (Thomason, 1995a, 1995b; Olivier, Cottu and Ferret, 1995). The wide range of composite materials applications results in an almost inevitable contact with liquids and vapors, either organic or aqueous, which can affect both the immediate and the long-term performance of the material. The mechanisms of water absorption, the plasticizing effect of absorbed moisture and the lowering of glass-rubber transition temperature are well known processes which have been widely studied in polymeric materials (Thomason, 1995a, 1995b; Ishai, 1975; Paplham et al, 1995; Harper, Staab and Chen, 1987; Cândido, 2001; Choi et al, 2001). Water absorption in composite materials has also been studied and it has been shown that, in general, the mechanisms of moisture penetration are much more complex than in the case of the unreinforced matrix (Thomason, 1995a, 1995b; Lee and Peppas, 1993)

Moisture absorption has been shown to lead to general reduction of the mechanical properties of composites. This has been attributed, in part, to degradation of the fiber-matrix interfacial bond (Thomason, 1995a, 1995b; Paplham et al, 1995; Lee and Peppas, 1993). It has been shown that the use of special fiber surface treatment can reduce the sensitivity of certain mechanical properties to water. Typical cases are mentioned in the literature as the manufacture of glass or carbon fiber-reinforced epoxy composites (Thomason, 1995a, 1995b; Gupta and Drzal, 1985, Delaware Composite Design Encyclopedia, 1990). Although the improvement in some properties in the wet condition is well documented, it is still not fully clear how the water and the fiber/surface treatment/matrix region interact (Madhukar, Drzal, 1991). In particular, there are questions concerning water absorption preferentially along the fiber-matrix interface (wicking effect), leading to the bonds hydrolysis between the surface treatment and the fiber, resulting in the degradation of the fiber-matrix adhesion. It is thought that the wicking mechanism is not active unless the fiber matrix adhesion is weak, possibly as a result of degradation of the interface by water. However, the exact role of the interface in the water absorption process is not fully understood and there are conflicting results in the literature (Thomason, 1995a, 1995b, a,b; Paplham et al, 1995; Harper, Staab and Chen, 1987; Cândido, 2001; Choi et al, 2001).

The way in which composite materials absorb water depends upon many factors, such as temperature, fiber volume fraction, orientation of reinforcement, fiber nature (permeable or impermeable), geometry of exposed surfaces, diffusivity and surface protection. The major mechanism of moisture penetration into composite materials is diffusion. This mechanism involves direct diffusion of water into the matrix, and, to a much lesser extent, into the fibers. The other common mechanisms are capillarity and transport by microcracks. The capillarity mechanism involves flow of water molecules along the fiber-matrix interface, followed by diffusion from the interface into the bulk resin. Transport of moisture by microcracks involves both flow and storage of water in microcracks or other forms of microdamage (Andreopoulos and Tarantili, 1998).

Aircraft composite parts are exposed to water, fuel and mechanical stresses. These combined effects may cause severe damage to composite structures. A detailed investigation of the absorption behavior of the material is necessary in order to understand the material changes induced by hygrothermal effects.

In this work, the influence of the moisture on the mechanical properties (tensile, interlaminar shear strength, compression and damping results) of carbon fiber composites were studied. Different fiber orientation in composite materials can result in the moisture penetration more or less inside of the polymeric composites. It was observed that bidirectional composites ($[0/90]_s$ and $[\pm45]_s$) leads to a lower rate of moisture absorption compared to unidirectional reinforced composites due to the edges effect.

2. Materials

Unidirectional carbon fiber/epoxy (CF/E) prepreg with F155 specification was used for composite preparation, supplied by EMBRAER (Empresa Brasileira de Aeronáutica). Three different families of laminates were produced using unidirectional carbon/epoxy prepreg tapes. The families of laminates obtained were: $[0/0]_s$, $[\pm45]_s$ and $[0/90]_s$.

In order to assess the influence of the environmental conditioning on the mechanical properties, the carbon/epoxy specimens were exposed to a combination of temperature and humidity in an environmental conditioning chamber. The condition selected to saturate the specimens before the mechanical tests were based on Procedure B of ASTM D 5229 M-92.

The tensile test was done according to ASTM D 3039-76. The tests were performed in an Instron mechanical testing machine using a test speed of 1.27 mm/min. Tensile strain was measured by bonding one strain gage type Rosette at ±45°, placed at the mid-section between the tabs (Figure 1). They were used 10 specimens for each family during this test.

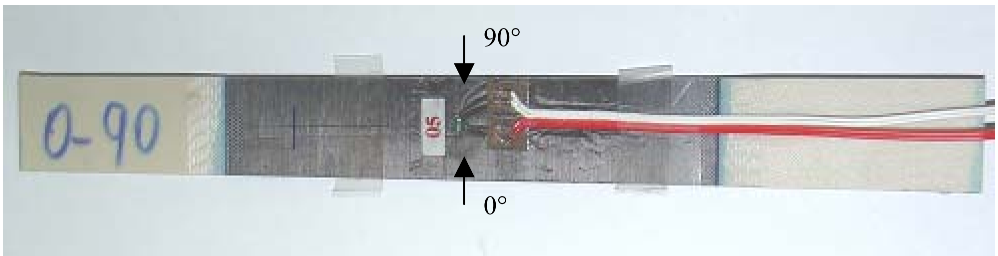

Figure 1. One strain gage type Rosette bonded at ±45°.

In order to compare the experimental results of CF-E composites in relation to theoretical values, the Fabric Geometry Model (FGM) was used program. The program allows predicting the stiffness of composite materials having spatially oriented reinforcements, from constituent material properties using composite micromechanics approach.

The compression test was done according to ASTM D 3410-87. The tests were performed in an Instron mechanical testing machine using a test speed of 1.27 mm/min. The interlaminar shear strength test (ILSS) was done according to ASTM D 2344-84. They were used 10 specimens for each family during this test.

The dynamic elastic modulus was determined by vibration damping measurements. The vibration measurements used a 0.6g accelerometer attached to the end of the rectangular beam. The vibration test gives as a result the free vibration damping decay and the frequency response function (FRF), simultaneously. They were used 3 specimens for each family during this test.

Using the vibration damping results, the storage modulus (E') was obtained according to Eq. 1.

$$E' = \frac{4\pi^2 f^2}{3I} \cdot \left[M + \frac{33}{140} m \right] \cdot L^3 \cdot \left[1 + \frac{\Delta^2}{4\pi^2} \right] \quad (1)$$

where: E'= elastic modulus; f = natural frequency (first mode of vibration); I = inertial moment; M = accelerometer weight; m = specimen weight; L = specimen lenght and Δ = logaritmic damping.

Most of the materials studied have been considerably more compliant than aluminum hence would present less problem of parasitic loss. Due to eventually problems related to parasitic damping, this work will not approach damping factor values.

3. Results

Figure 2 shows the weight increase as a function of exposed time for carbon fiber/epoxy composites specimens exposed at 80°C and 90% RH. As described in ASTM D 5229 M-92, tree specimens of each family were used in this experiment. Like any other polymers, epoxies can absorb moisture when exposed to humid environments. This takes place through of a diffusion process, in which water molecules are transported from areas with high concentration to areas with lower moisture concentration.

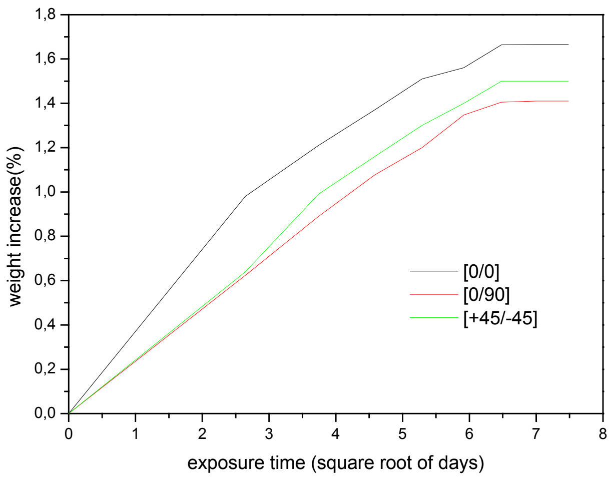

Figure 2. Weight increase of CF/E composite specimens exposed at 80°C and 90% RH**.**

Different fiber orientation in composite materials, can result in the moisture penetrate more or less inside of the polymeric composites. It was observed that bi-directional composites leads to a lower weight gain percentage (maximum of 1.4% and 1.5% in $[0/90]_s$ and $[\pm45]_s$, respectively) compared to unidirectional reinforced composites (maximum of 1.7%), due to the edges effect. Since the edge region is very narrow (of the order of 0.25 mm) the higher moisture concentration in this area should have a small effect in the total mass gain of the sample. However, the bi-directional reinforced composites presented a higher resin rich area in the edge of the composite when compared with unidirectional reinforced composite, absorbing a higher moisture quantity.

Table 1 shows the variation of the tensile strength of carbon fiber/epoxy composites. The initial cracking occurred at strains of about 1.06% and 1.74% for $[0/0]_s$ and $[0/90]_s$, in dry conditions, respectively. For these specimens, can be considered that composite components (fiber and matrix) behave elastically until the initial matrix cracking and additionally, transverse stiffness (fibers of 90° towards loading direction) contribution to axial stiffness is negligible. Therefore, the initial cracking for [±45] happened at strain of about 11%. This behavior is due to the fiber of 45° towards loading direction, allowing a bigger deformation of epoxy matrix.

According to Table 1 the tensile strength values for carbon fiber/epoxy composites, after to be submitted to hygrothermal conditioning, shows a decrease of 2.5%, 14% and 14% for $[0/0°]_s$, $[0/90°]_s$ and $[\pm45°]_s$ laminates, respectively. As can be observed, these materials show a decrease of their mechanical properties after to be submitted to hygrothermal conditioning due to the matrix plasticization and the degradation of the carbon fiber/epoxy interface properties.

Table 1. Tensile values obtained by carbon fiber reinforced epoxy resin composite.

Layup family	**σ_{ult} (MPa)**		**ε (%)**		**E (GPa)**	
	dry	**wet**	**dry**	**wet**	**dry**	**wet**
$[0/0]_s$	1965±28	1913±55	1.06±0.04	1.60±0.06	118±2	114±4
$[0/90]_s$	1160±37	999.0±39	1.74±0.06	1.51±0.05	67.2±4	64.0±3
$[\pm45]_s$	167±8	144.0±13	10.9±0.03	11.7±0.60	11.3±1	10.0±1

Both tensile strength and modulus for carbon fiber/epoxy composites depend strongly on the fiber orientation angle. With increasing fiber orientation angle, both tensile strength and modulus are reduced.

The theoretical elastic constants calculated by using composite micromechanics approach are shown in Table 2. The theoretical calculations show that the $[0/0]_s$ composite have an elastic modulus (E) higher than the other layups, as expected (134GPa). Therefore, the tensile behavior of the $[\pm45]_s$ is closer of the epoxy matrix behavior (E=2 GPa) than the $[0/0]_s$. This behavior happened due to the increase of the tensile load in $[\pm45]_s$ also creates a few intralaminar cracks parallel to the fiber directions in both –45° and + 45° plies. With increasing load, the edge delamination extends toward the center of the specimen and the matrix will have a significantly influence over the tensile behavior. This composite presented a theoretical E modulus of 14.3 GPa. The fiber and matrix mechanical properties and the fiber content used in order to calculate the engineering constants by micromechanics approach are presented in Table 3.

Table 2. Theoretical engineering constants calculated by micromechanics approach.

layup family	**E_x (GPa)**	**E_y (GPa)**	**ν_{12}**
$[0/0]_s$	134	71.3	0.26
$[0/90]_s$	71.3	71.3	0.03
$[\pm45]_s$	14.3	14.3	0.81

Table 3. Parameters used in the FGM program and the mixtures rules.

material	**Fraction content (%)**	**E_x (GPa)**	**E_y (GPa)**	**G_{12} (GPa)**	**ν_{12}**
Epoxy	43%	5.00	5.00	1.85	0.30
Carbon fiber	57%	220	20.0	15.0	0.20

A comparison of the average E moduli between the theoretical and experimental results shows little difference. It is observed that the experimental tensile modulus values exhibited a decrease of 12%, 5.8% and 21% for laminates $[0/0]_s$, $[0/90]_s$ and $[\pm45]_s$, respectively, in relation to the theoretical values. These differences were expected because the interface effect or void presence which is not considered in the theoretical calculations. The insensitivity of E_1 to the interfacial adhesion is expected, since the properties in the longitudinal direction are dominated by fibers and thus the effect of interfacial adhesion on the longitudinal elastic properties will only be marginal.

It was observed that the E modulus of the carbon fiber/epoxy composites depends on the moisture absorbed in the matrix. The tensile modulus values for carbon fiber/epoxy composites, after to be submitted to hygrothermal conditioning, shows a decrease of 3.5%, 4.5% and 11.5% for $[0/0°]_s$, $[0/90°]_s$ and $[\pm45°]_s$ laminates, respectively. This behavior can be attributed to the moisture effect of the interface between the fiber and epoxy matrix with the increase the rigidity of the composite specimen. Thus, there is an adding hygrothermal effect of the interface on tensile behavior in composites, which is more pronounced for this composite submitted in humidity atmosphere.

The influence of moisture on tensile modulus of carbon fiber/epoxy composites observed in $[0/0°]_s$, $[0/90°]_s$ and $[\pm45°]_s$ laminates are in agreement with the tensile strength results found in this work.

Table 4 presents the values of compression, ILSS and damping for the carbon fiber/epoxy composites. Due to the different orientation of reinforcement, the families present the dispersions of the data, as listed on Table 4. As can be observed, all values decrease with the increase of hygrothermal conditioning time. These values confirm the results obtained by tensile tests.

Table 4. Compression, ILSS and E' (damping) values obtained by carbon fiber reinforced epoxy resin composite.

layup family	Compression (σ_{comp} MPa)		ILSS (MPa)		E' (GPa)	
	dry	wet	dry	wet	dry	wet
$[0/0]_s$	1002±112	920±84	84.5±6.2	60.8±4.1	111±8	89±5
$[0/90]_s$	760±82	690±63	66.4±5.3	49.6±4.2	56±4	52±3
$[\pm45]_s$	810±73	780±68	98.9±8.2	83.3±7.7	13±1	12±1

4. Conclusion

In this work, the influence of the moisture in the mechanical properties (tensile, ILSS, compression and damping results) of carbon fiber composites were studied. Different fiber orientation in composite materials, can result in the more or less moisture penetration inside of the polymeric composites. It was observed that bidirectional composites ($[0/90]_s$ and $[\pm45]_s$) leads to a lower rate of moisture absorption compared to unidirectional reinforced composites due to the edges effect.

The mechanical properties values for carbon fiber/epoxy composites, after to be submitted to hygrothermal conditioning, show a decrease values with the exposure time due to the matrix plasticization and the degradation of the carbon fiber/epoxy interface properties.

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