Damping Behavior of Hygrothermally Conditioned Carbon Fiber/Epoxy Laminates

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ABSTRACT: Carbon fiber reinforced polymer composites have been used in wide variety of applications including, aerospace, marine, sporting equipment as well as in the defense sector due to their outstanding properties at low density. In many of their applications, moisture absorption takes place which may result in a reduction in mechanical properties even at lower temperature service. In this work, the viscoelastic properties, such as storage modulus (*E*') and loss modulus (*E*''), were obtained through vibration damping tests for three carbon fiber/epoxy composite families up to the saturation point (6 weeks). Three carbon

fiber/epoxy composites having $[0/0]_s$, $[0/90]_s$, and $[\pm 45]_s$ orientations were studied. During vibration tests the storage modulus (*E'*) and loss modulus (*E''*) were monitored as a function of moisture uptake, and it was observed that the natural frequencies and *E'* values decreased with the increase during hygrothermal conditioning due to the matrix plasticization. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 106: 3143–3148, 2007

Key words: carbon fiber/epoxy; damping behavior; viscoelastic properties; hygrothermal conditioning

INTRODUCTION

In recent years, fiber-reinforced composites has achieved steps of extensively use in aerospace, marine, automobile, medical, and other engineering industries. In service, temperature variations and moisture can lead to reductions in the elastic moduli and degrades the strength of the laminated material.^{1–5} Among thermoset polymers, epoxy resins are the most common matrices for high performance carbon-fiber composites because of easy processing conditions. However, they tend to absorb moisture readily, leading to a reduction in properties in a long-term service basis. Absorbed moisture in fiber/ epoxy composites are found to depend on the relative humidity of the environment only.^{6–8} In fact, hygrothermal effects on carbon fiber is negligible when compared to polymeric matrices. Therefore, water diffusion into a laminate is a matrix-dominated phenomena. As a result, the matrix glass-transition temperature may be reduced, accompanied by a reduction in stiffness and strength.⁸⁻¹⁰

Faster moisture uptake may also induce a faster material degradation. As a consequence, it is important to know how moisture absorption takes place in

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a composite. For an undamaged material, wellaccepted moisture transportation models are available. The water absorption of composite materials after a long time of use, while exposed to the environment, is considered an unavoidable phenomenon. The majority of the models developed for describing the water kinematic diffusion in the composite materials are based on Fick's law.^{11,12} One of most important parameters in Fick's law is the material diffusivity. If the material contains cracks that significantly affect the moisture uptake, then the original law of Fickian^{11,12} are no longer valid for the whole laminate, but locally they still work. Moisture absorption models for cracked materials have been presented by e.g. Weitsman¹³ and Lundgren and Gudmundson.¹⁴ In the work by Weitsman a link is made with continuum damage theories and thermodynamics, while Lundgren and Gudmundson use a micromechanical approach.

Elastic properties of composites can be determined by semi-static or dynamic mechanical tests. Static mechanical tests are destructive while the majority of dynamic mechanical test offers the advantage of being nondestructive.^{15–18} One of the most used dynamic mechanical test is the vibration damping.^{19–24} The principle of dynamic mechanical testing consists of recording the vibration decay of a material, usually in the form of rectangular plate excited by a controlled mechanism. The test identifies the materials elastic and damping properties. The frequency amplitudes are measured by accelerometers and

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Figure 1 The experimental set-up.

compared to the intrinsic frequencies calculated by using a digital method. An interactive procedure, based on the calculation of the sensitivity matrix of the resonance frequencies to variations in rigidities, makes it possible to correlate the calculated frequencies with the experimental frequencies and to obtain the actual rigidity of the plate.^{25–32} As moisture leads to reductions on mechanical properties of composite materials their significance can be monitored by dynamic mechanical tests.

In the present study we attempted to explore the dynamic mechanical properties obtained by free vibration damping test for three families of carbon fiber/epoxy composites ($[0/0]_s$, $[0/90]_s$, and $[\pm 45]_s$). The measurements were performed before and after submitting the composites to hygrothermal conditioning.

EXPERIMENTAL

Composite manufacturing

Unidirectional carbon fiber/epoxy (CF/E) prepreg tapes having F155 specification (Hexcel Co) were used for composite preparation. Three different families of composites were prepared: $[0/0]_{s}$, $[\pm 45]_{s}$, and $[0/90]_{s}$. The fiber content in each composite was ~60% (v/v). The composites were cured in autoclave, under a pressure of 0.69 MPa and vacuum of 0.083 MPa, following a heating cycle up to 180° C. After curing a 10 ply composite a 2.1-mm nominal thickness was attained.

Theoretical calculations

To compare the experimental results of carbon fiber/ epoxy composites with theoretical values were used the Fabric Geometry Model (FGM) Code.³³ The program allows the prediction of stiffness of composite materials having spatially oriented reinforcements, from constituent material properties by using composite micromechanics approach. The FGM Code allows the calculation of the elastic constants for the fiber/epoxy composites, taking into account the fiber orientation.

Environmental conditioning

All carbon fiber/epoxy composites were exposed to a combination of temperature and humidity in an environmental conditioning chamber in order to assess the influence of both factors on the mechanical properties. The condition selected to saturate the specimens before the mechanical tests were based on Procedure B of ASTM Standard D 5229 M-92 (80°C and 90% of humidity).³⁴

Measurement of viscoelastic properties

The dynamic elastic modulus was determined by free vibration damping measurements. The measurement principle consists of recording the free vibrations of a prismatic cantilever beam excited by tapping it with an appropriate hammer, as shown in Figure 1. The amplitude decay as a function of time and the vibration modes were detected by an acquisition data system from Spectral Dynamics Company and recorded using a software LMS CADA-PC. The test parameters were: analyses range of 1000 Hz; acquisition time of 200 ms; rectangular observation window and frequency resolution of 5 Hz. The amplitude decay was measured using a 0.6 g accelerometer. Beam dimensions are shown in Table I. Following the testing procedure, two types of curves were obtained: damping free vibration and frequency response function profiles.

The length, width, and thickness were measured along the beam specimens. Average values for the thickness, width and standard deviation were calculated from 10 measurements. The upper and lower

Dimensions and Weight of Specimens Used in Damping Tests					
Specimen	Length,	Width,	Thickness,	Weight	Inertia
	L (mm)	b (mm)	h (mm)	(g)	(m ⁴)
[0/0]	207	15	1.8	9.19	$\begin{array}{c} 7.29 \times 10^{-12} \\ 7.29 \times 10^{-12} \\ 8.21 \times 10^{-12} \end{array}$
[0/90]	210	15	1.8	9.06	
[±45]	210	15	1.9	9.21	

TABLE I

limits of the standard deviation of the sample dimensions were considered for the calculations.

The vibration test gives as a result the free vibration damping decay and the frequency response function (FRF), simultaneously. Considering a linear system of a single degree of freedom, the FRF response is a decomposition of the natural frequencies of a structure or specimen, which corresponds to a typical fingerprint identity of the vibration modes. The number of vibration peak frequencies (vibration modes) and the shape of the FRF response is a direct result of the rigidity of the material.

A theoretical analysis of internal damping and dynamic stiffness for aligned continuous fiber composite was developed based on micromechanics models for the complex moduli. The free vibration method results generally present a logarithmic damping (Δ) given by the eq. (1).^{29–32}

$$\Delta = \frac{1}{n} \ln \left(\frac{\delta_1}{\delta_n} \right) \tag{1}$$

where *n* is the number of peaks; δ_1 is the amplitude of the first peak and δ_n is the amplitude of the final peak analyzed.

Most of the materials studied have been considerably compliant hence would present less problem of parasitic loss.^{35–37} Because of eventually problems related to parasitic damping, this work will not approach damping factor values. In the present work only the E', E'', and tan δ values are presented.

The storage modulus (E') can be obtained according to eq. (2).^{29–32}

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

where E' = elastic modulus; f = natural frequency; I= inertial moment; M = accelerometer weight; m = specimen weight, and L = specimen length.

RESULTS AND DISCUSSION

Moisture absorption

Figure 2 shows the graph of moisture absorption for the three carbon fiber/epoxy composites: $[0/0]_{s}$, [0/ $90]_{s'}$ and $[\pm 45]_{s}$. There is a steady increase in moisture absorption for all specimens up to the saturation point (6 weeks), reaching a maximum moisture absorption around 1.4-1.7% per mass.

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.^{24–26} As a result of different fiber orientation in composite materials, moisture can follow different infiltration patterns inside the polymer matrix.^{24–26} Moisture uptake through the fiber/matrix interfaces can cause interfacial debonds leading to the rupture or degradation of the interface, and eventually exposing the carbon fibers at the edges of the composite.²⁴⁻²⁶ The absorption of the water molecules accelerated by temperature used in the hygrothermal conditioning weakens the fiber/matrix interface exposing the fibers.37

Theoretical calculations

Composite elastic constants calculations were performed by using the FGM software in order to be compared with the elastic constant changes after hygrothermal conditioning. The parameters used to calculate the elastic constants of polymer composite materials and the results for theoretical elastic constants calculated by using composite micromechanics



Figure 2 Moisture uptake of carbon fiber/epoxy composites.

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TABLE II				
Parameters	Used	in	the FGM	Program ¹⁵

			-		
	Volume	E_x	E_{y}	G ₁₂	
Specimen	content (%)	(GPa)	(GPa)	(GPa)	v_{12}
Epoxy resin	40.0	5.00	5.00	1.85	0.30
Carbon fiber	60.0	220.0	15.4	15.8	0.25

approach are shown in Tables II and III, respectively. Because of the elastic behavior of these composite materials, it can be assumed that the E' value found by vibration test is near to the E_x value found by theoretical calculations.

Theoretical calculations shows that the elastic modulus for the [0/0], [0/90], and $[\pm 45]_s$ laminates are 134 GPa, 71.3 GPa, and 14 GPa, respectively.

Influence of moisture on frequencies

Figure 3(a–c) present the resonant frequency response function results of nonconditioned specimens. For [0/0] and [0/90] families, three frequencyrelated peaks were found. For [±45] family of laminate two frequency modes were found, shifted to higher values in relation of the others families evaluated, due to a higher mass of the beam tested. From these graphs, the first mode of vibration was used in order to calculate E' and E'' moduli, by using eq. (2), due to the fact that it is the predominant wavelet level. The beam specimens have the frequency scanned up to 500 Hz. A comparison of the unidirectional, bi-directional and quasi-isotropic data for carbon fiber/epoxy laminates indicated that increasing the proportion of 0° plies in the laminates reduced damping.

Figure 4 shows the first mode frequency response function results of conditioned composite specimens. As can be observed from this Figure, the hygrothermal conditioning generates a sensitive change on the frequency values due to the matrix plasticization. This changes is enouth to produce significative changes in E' and E'' moduli.

Influence of moisture in the elastic and loss moduli (E' and E'')

Four primary mechanisms have been suggested to contribute to damping in composites: viscoelastic response of the constituents, friction, and slipping at

TABLE III Calculated Theoretical Engineering Constants

Specimen	Fiber content (%)	E _x (GPa)	E _y (GPa)	G ₁₂ (GPa)	G ₁₃ (GPa)	V ₁₂
$[0/0]_s$ $[0/90]_s$ $[\pm 45]_s$	60.0 60.0 60.0	134.0 71.3 14.0	8.14 71.3 14.0	3.86 3.86 34.6	2.92 3.39 3.39	0.26 0.03 0.81

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Figure 3 Resonant frequency results from composites laminates specimens studied: (a) $[0/0]_{s}$; (b) $[0/90]_{s}$, and (c) $[\pm 45]_{s}$.

the fiber-matrix interface, thermoelastic damping due to cyclic heat flow and damage initiation and growth. The damping ratio of a composite is dictated primarily by the viscoelastic or microplastic phenomena at the matrix interface. Excluding the contribution from any cracks and other defects, the internal damping of a composite is determined by the following variables: properties and relative proportions of the matrix and the reinforcement, dimensions of the inclusions, orientation of the reinforcement with respect to the loading axis, surface treatments of the reinforcement and void content.¹⁵

Figure 5(a-c) presents the vibration damping curves representatives of the composite laminate specimens studied in this work. As can be observed in Figure 5 the amplitude decays of [0/0] families are more pronounced due to their higher stiffness.



Figure 4 Variation of resonant frequency values with the exposure time.

As mensioned earlier, the E' for all specimens were obtained by using equation 2. These values can be found in Table IV.

The calculated elastic modulus for [0/0], [0/90], and $[\pm 45]$ composites were 17.2%, 21.5%, and 5.0% higher than the experimental result, respectively. Experimental measurements of elastic modulus of composites tend to exhibit different values from the theoretical calculations from micromechanics approach, because ideal bonding between fiber/matrix interface, perfect alignment of fibers and absence of voids and other defects are considered in the last. So, the differences between *E'* experimental and calculated *E* modulus are expected.

Table IV shows the storage modulus (E') as a function of the number of days exposed to humidity and temperature for all laminates studied in this work. As can be observed, the E' modulus values is nearly constant up to 15 days of exposure conditioning. From this period up to 60 days of hygrothermal conditioning, a decrease of 11%, 3.6%, and 7.5% of the [0/0], [0/90], and $[\pm 45]$ laminates is reached, respectively. These differences are due to differences between the moisture absorption for each specimen, i.e., in which manner the composite edges are exposed to moisture attack. After 60 days of hygrothermal conditioning, the specimens reaches moisture saturation and the E' values remain constant at 98.7, 54.0, and 12.3 GPa for [0/0], [0/90], and [±45] laminates, respectively. As mentioned before, for polymer composite materials, moisture uptake always induced resin plasticization and, consequently, reduces the E' modulus of the laminates.

The loss modulus (E'') is related to the energy dissipation mechanisms in materials and in the major of the cases their behavior is very difficult to be understood due to the complexity of combining both energy dissipation mechanisms and nonuniform interface adhesion. So, in this case, the energy dissipation due to interfacial adhesion can play the role. The values for *E*["] can be found in Table IV.

As can be observed in Table IV, the E" values for [0/0], [0/90], and $[\pm 45]$ laminates were 0.94, 2.42, and 0.20, respectively. There is no direct correlation between these values due to the complexity of the E" behavior, so in this work, it was not possible to correlate the E" behavior with the moisture absorption.



Figure 5 Damping behavior curves from composite laminate specimens studied: (a) $[0/0]_{si}$ (b) $[0/90]_{si}$ (c) $[\pm 45]_{s}$.

	1 0	
Specimens/		
conditioning (days)	<i>E</i> ′ (GPa)	E" (GPa)
$[0/0]_{s}$		
0	111	0.94
15	109	1.35
30	107	1.03
45	105	2.15
60	99	0.83
$[0/90]_{s}$		
0	56	2.42
15	56	2.43
30	55	0.47
45	54	1.23
60	54	0.67
$[\pm 45]_{s}$		
0	13.3	0.20
15	13.3	0.57
30	12.8	0.11
45	12.6	0.28
60	12.3	0.15

TABLE IV Loss Factor, Elastic, and Loss Modulus Obtained by Damping Tests

CONCLUSION

The damping behavior of carbon fiber/epoxy laminates, having configurations of $([0/0]_{s}, [0/90]_{s}, \text{ and} [\pm 45]_{s})$, was investigated. The dynamic elastic modulus (*E*') for the composites was compared with the theoretical values, and good agreement was found.

For all the composite specimens studied the moisture saturation point occurred after 6 weeks of exposure. Up to saturation point it was observed that the specimens $[0/90]_{s'}$ [±45]_s, and $[0/0]_{s'}$ presented a moisture uptake of 1.4%, 1.5%, and 1.7%, respectively.

During vibration tests, it was observed that all the natural frequencies and E' values decreased with an increase as a function of time during hygrothermal conditioning. This is due to the matrix plasticization by the moisture.

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