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Processing and hygrothermal effects on viscoelastic behavior of glass fiber/epoxy composites

E. C. BOTELHO*
Divisão de Materiais, Instituto de Aeronáutica e Espaço, CTA, São José dos Campos, São Paulo, Brasil; Fatigue and Aeronautic Material Research Group, Department of Material and Technology, UNESP, Guaratinguetá, São Paulo, Brasil
E-mail: ebotelho@directnet.com.br

M. L. COSTA, L. C. PARDINI, M. C. REZENDE
Divisão de Materiais, Instituto de Aeronáutica e Espaço, CTA, São José dos Campos, São Paulo, Brasil

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 determination 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 glass fiber reinforced composites (GRFC). They control the damping behavior of composite structures and are also an ideal tool for monitoring the development of GFRC's mechanical properties during their processing or service. One of the most used tests is the vibration damping. In this work, the measurement consisted of recording the vibration decay of a rectangular plate excited by a controlled mechanism to identify the elastic and damping properties of the material under test. The frequency amplitude were measured by accelerometers and calculated by using a digital method. The present studies have been performed to explore relations between the dynamic mechanical properties, damping test and the influence of high moisture concentration of glass fiber reinforced composites (plain weave). The results show that the E' decreased with the increase in the exposed time for glass fiber/epoxy composites specimens exposed at 80°C and 90% RH. The E' values found were: 26.7, 26.7, 25.4, 24.7 and 24.7 GPa for 0, 15, 30, 45 and 60 days of exposure, respectively.

1. Introduction

Fiber-reinforced composite materials are finding a wide range of applications in structural design, especially for lightweight structures that have stringent stiffness and strength requirements. Composite laminates have high stiffness to weight and strength to weight ratios compared to conventional materials. Hence, they are a favorable choice for many applications ranging from sports to aerospace [1–4]. With the increasing applications of there materials, more and more knowledge is needed to get a better understanding of the bonding of the materials, which can lead to different mechanical properties of the materials [5–8].

Therefore, the use of resin matrix composites in aircraft structure introduces a new range of problems. Many experiments reveal a sensitivity of resin matrix and fiber-matrix interface to both environmental degradation, with interactions between various mechanisms being possible. Assurance is therefore needed that any environmental and mechanical damage accumulated

*Author to whom all correspondence should be addressed.

during a typical aircraft life will not compromise structural integrity [9–14].

Elastic properties of composites can be determined by static or dynamic mechanical tests. Static mechanical tests are destructive while some dynamic mechanical test offers the advantage of being non-destructive. Nowadays, various experimental methods potentially applicable to determine dynamic moduli and damping of composites (free vibration, rotating-beam deflection, forced vibration response, continuous wave or pulse propagation technique) have been reviewed [15–22].

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 testing identifies the materials elastic and damping properties. The frequency amplitudes are measured by accelerometers and compared to the intrinsic frequencies calculated by using a digital method. An iterative 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 [23–27].

In the present study we attempted to explore the dynamic mechanical properties obtained by free vibration damping test of glass fiber/epoxy composites. The measurements were performed before and after submitting the composites to hygrothermal conditioning.

2. Experimental

2.1. Materials

Glass fiber/epoxy (GF/E) prepreg with F155 specification (standard epoxy resin) was used for composite preparation. It was supplied by Hexcel Composite. The fiber reinforcement was plain weave fabrics. The fiber content was 60% (v/v).

2.2. Composites processing

The glass fiber/epoxy composite was prepared by stacking alternating laminae of the prepregs. After the lay-up process, the laminate was fit inside in a vacuum bag and placed in the autoclave system. The curing cycle was done at a heating rate to 2.5°C/min up to 121°C and this final temperature was held during 1 h. The pressure and the vacuum used were 0.69 MPa and 0.083 MPa, respectively. The curing cycle was based on results obtained by differencial scanning calorimetry analyses (DSC). In this experiment, the uncured prepreg samples (F155), weighing about 5 mg, were placed in aluminum crucibles with a sealed lid and placed opposite the empty reference pan in the oven chamber. The DSC testing was performed in both dynamic and isothermal heating modes. The equipment used here was a DSC Pyris from Perkin Elmer. For dynamic testing, three linear heating rates of 2.5; 5.0 and 10.0°C/min were used over a temperature range of 30–300°C for also determining prepreg cure kinetics. Isothermal experiments were conducted at three temperatures (100, 110 and 120°C) in order to obtain both the cure rate ant the extent of cure as a function of time. The reaction was considered complete when the signal returned to the baseline. The origin $t = 0$ was obtained by rerunning the experiment on the reacted sample, under the same conditions, to obtain the true baseline.

2.3. Environmental conditioning

In order to assess the influence of the environmental conditioning on the damping results, the glass/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 Standard D 5229 M-92 [28]. The moisture level in the laminate was periodically monitored as a function of time by measuring the mass of traveler samples until the moisture equilibrium state is reached. During conditioning, the temperature was set at 80°C and the relative humidity in the chamber was set to 90%. The temperature must remain well bellow the resin glass transition temperature so as to avoid onset of irreversible damage (swelling and cracks), which permanently changes the absorption characteristics of the material.

2.4. Measurement of dynamic moduli

The dynamic elastic modulus was determined by vibration damping measurements. The measurement principle consists of recording the free vibrations (cantilever) of a rectangular plate excited by tapping the beam with an appropriate hammer as shown in Fig. 1.

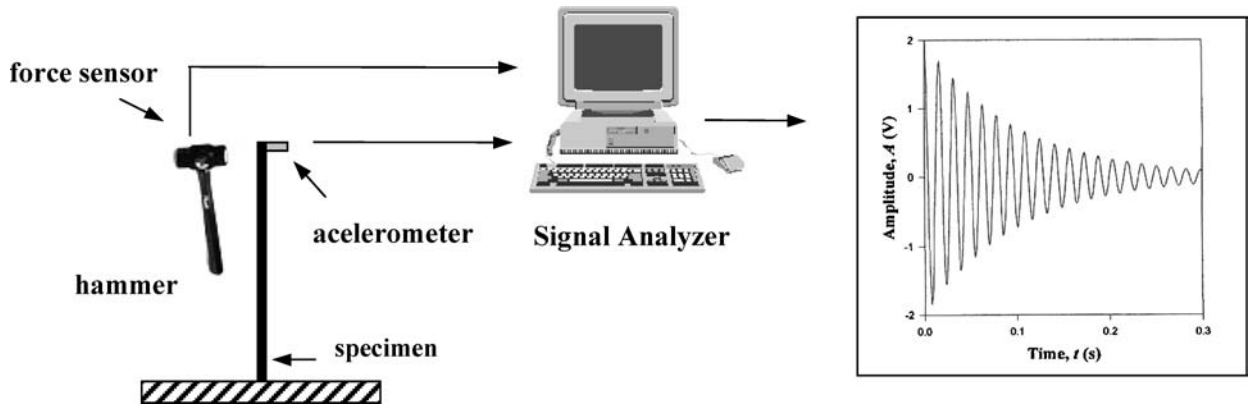

Figure 1 The experimental set-up.

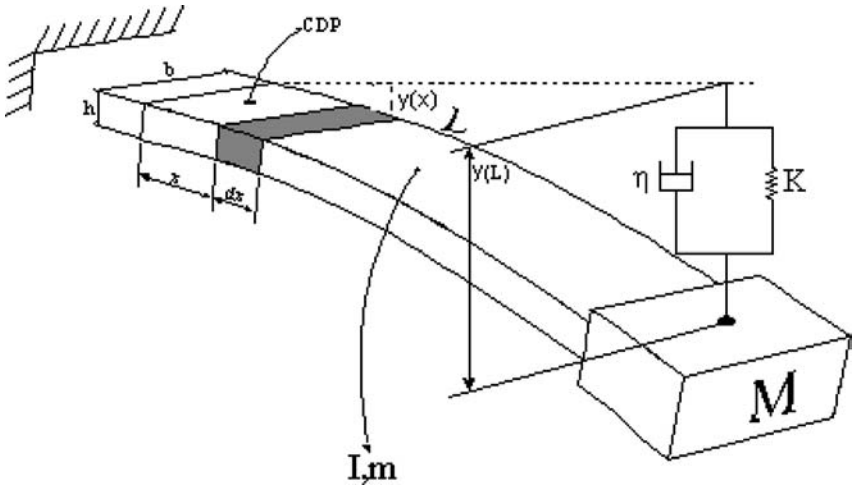

Figure 2 —Viscoelastic model for free vibration method [24–26].

The amplitude decay as function of time and the vibration modes were detected by an acquisition data system from Spectral Dynamics Company and calculated using a software LMS CADA-PC. The test parameters were: analyses range: 500 Hz; acquisition time: 200 ms; observation window: rectangular and frequency resolution: 5 Hz. The amplitude decay was measured using a 0.6 g accelerometer. Beam dimensions were: length: 0.21 m; thickness: 0.02 m and width: 0.002 m. The non-conditioned specimen weight was 19 g. Using this procedure, they were obtained two types of curves: damping free vibration and frequency curves.

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 viscoelastic response of materials under stress can be modeled by the model depicted in Fig. 2, where L is length; h is thickness, b is width, dx is deformation in x and y is the deflection of the beam [24–26].

In this work, the damping factor was obtained by:

$$\zeta = \sqrt{\frac{\Delta^2}{\Delta^2 + 4\pi^2}} \tag{1}$$

Knowing that [24–26]:

$$\omega = \sqrt{1-\zeta^2}\omega_n \tag{2}$$

and

$$2\pi f = \omega_n\sqrt{1-\zeta^2} \tag{3}$$

When δ is small, $\sqrt{1-\zeta^2} \cong 1$ and the equation becomes: $\delta \cong 2\pi\zeta$.

In this work, the storage modulus (E') was obtained according to Equation 4 [24–26].

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

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

The loss factor, tan δ, was calculated from the decaying-oscillatory damping curve as follows:

$$\tan\delta = \frac{\ln(\delta_1/\delta_n)}{n\pi} \tag{5}$$

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

The term $[\ln(\delta_1/\delta_2)]/n$, also known as the logarithmic decrement (Δ), can be obtained by fitting the experimental data.

Reorganizing Equations 2 and 5 the E'' values was calculated as:

$$2\zeta = \frac{E''}{E'}\sqrt{1-\zeta^2} \tag{6}$$

The Equation 6 allows quantifying the loss factor in composite materials through energy dissipation measurements.

The E' modulus of the composite materials was calculated using micromechanical model, in order to verify the experimental result values.

3. Results and discussion

3.1. Cure cycle

Cure cycle for composite prepregs are as a role provided by prepreg manufactures. In order to guarantee the complete cure cycle for the glass fiber/epoxy composite studied in this work, the cure kinetics was studied in order to draw an adequate cure cycle.

Cure cycle modeling involves the study of cure reaction kinetics, physical property correlation, and cure variables analysis. Several rate equations have been derived to describe the extent of reaction, or reation rate and reaction order for various thermoset systems [24–29]. In the present work, all study was based on the parameters obtained by DSC.

In order to calculate the total heat of reaction generated to reach full conversion, ΔH_0, the DSC dynamic scans at different heating rates were performed and

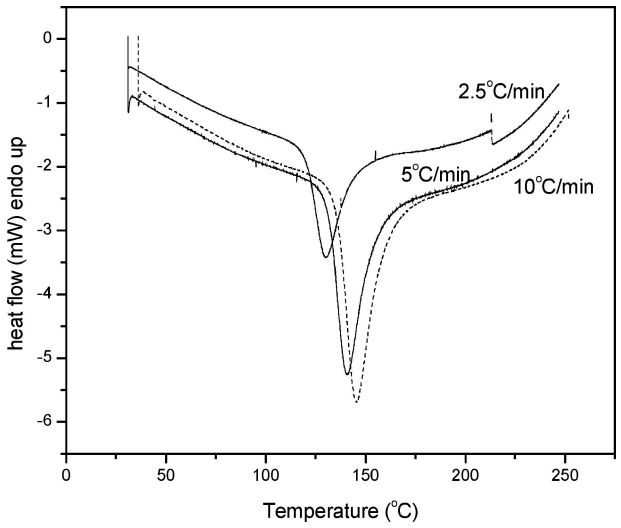

Figure 3 DSC of F155 prepreg system at different heating rates.

the total area above the thermogram was determined (Fig. 3). Replicate experiments were performed at each heating rates. The area of the peak upper the exothermic regions were used to determine the fractional conversion of the epoxy resin by assuming that the heat evolved during cure is directly related to the disappearance of epoxide groups during cure. The overall heat evolved in the reaction has been determined as the average value of reaction heats calculated in each thermogram. The corresponding value found for the F155 system was $\Delta H_0 = 125$ J/g. This value was smaller than the obtained value of the neat resin system. The glass transition, Tg, for the fully cured material was 105°C.

The value of extent of cure, α, is usually obtained by dividing heat ΔH_t evolved at time t by total heat ΔH_0 generated during the entire reaction, i.e, with shows the Equation 8

$$\alpha = \frac{\Delta \mathrm{H}_i}{\Delta \mathrm{H}_0}. \tag{8}$$

In graphical terms, ΔH_0 is the area above the dynamic DSC curve. Analytical representation of such a cure process is often studied through existing models such as *n*th order and *autocatalytic* models. In this work it was used the *n*th order model as following:

$$\frac{\mathrm{d}\alpha}{\mathrm{d}t} = k(1-\alpha)^n, \quad n\text{th order} \tag{9}$$

where: α is the degree of chemical conversion, n is the order of the reaction, k is the reaction rate and $\mathrm{d}\alpha/\mathrm{d}t$ is the derivative α with respect to time.

The temperature dependent reaction rate k is commonly described by the Arrhenius equation

$$k = A \exp\left(-\frac{E}{RT}\right), \tag{10}$$

where R is universal gas constant, T is temperature, E is activation energy and A is pre-exponential factor.

The kinetics parameters associated with the curing of carbon fabric/epoxy *F155* prepreg were shown in the Table I.

TABLE I Kinetics parameters obtained by isothermal method of carbon/epoxy resin (F155) preperg

Heating rates (°/min)	n	$-\Delta$H (J/g)	In A (s^{-1})	E (KJ/mol)	Glass transition, Tg(°C)
2.5	1.5	123.2	8.4 ± 0.1	102.0	105
5.0	1.7	124.8	8.6 ± 0.1	103.1	106
10.0	2.1	126.1	8.4 ± 0.1	102.8	107

The majority of the final properties of a composite are obtained during the cure of the matrix material of the composite and is the most important phase of the manufacturing process. Before a resin cure phase, a considerable money investment has been made into a composite component. If the part is incorrectly cured, then the outcome of the cure is irreversible and the part is usually scrapped. No amount of work before cure can ensure that an incorrectly cured part will be usable. So, it is apparent that the most critical step in obtaining good quality composites parts is the cure cycle. During the cure cycle, chemical reactions occur, which cause the matrix material (resin) to polymerize and obtain the desired properties of the material.

A cure cycle was suggested and have 4 steps. Firstly, the temperature is raised from 30 to 93°C at 2.5°C/min; holding at 93°C for 30 min (second step); following the temperature is raised until 121°C using the same heating rate (third step); holding at this temperature for at least 90 min (fourth step). Based on this information, a mathematical simulation of the degree of conversion of the *F*155−epoxy resin was carried out and represented in Fig. 4. This study was made to confirm if the steps suggested in the cure cycle were adequate to promote a good cure of laminates.

Fig. 4 presents the simulation of the prepreg system conversion degree as function of temperature for a time interval of 156.4 min; this is the time necessary to elevate the temperature from 30 to 121°C, using the heating rate of 2.5°C/min. This curve was obtained based on Equation 11, using a predetermined time and

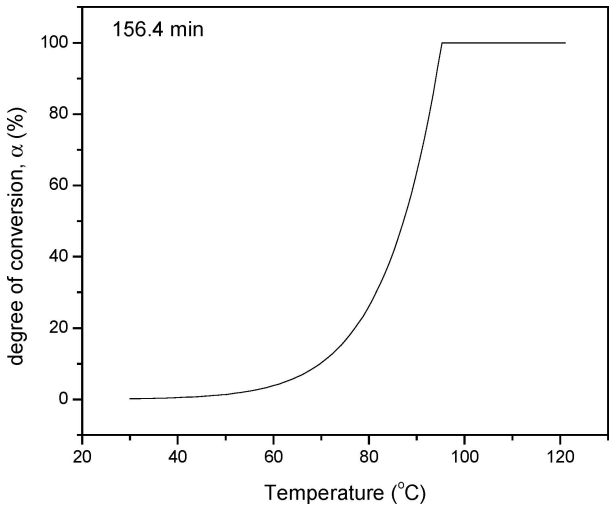

Figure 4 Total degree of conversion for F155 during the curing cycle.

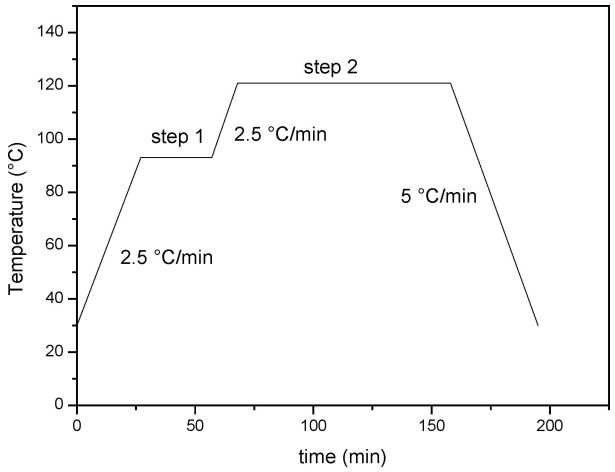

Figure 5 Cycle of curing of glass fiber/epoxy composite obtained by thermal analyses.

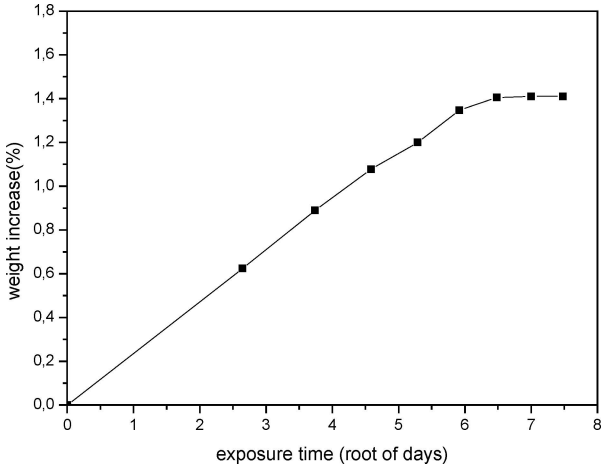

Figure 6 Moisture absorption of glass fiber/epoxy composites.

temperature range [24–29]:

$$\alpha = 1 - [1 - (1 - n).A.t.\exp \times (-E_a/RT)]^{(1/(1-n))} \quad (11)$$

Figs 4 and 5 resumes all steps described previously, showing the evolution of polimerization reaction in all cure cycle employed in the autoclave polymeric composites processing. It was observed that the steps suggested in the cure cycle were adequate to promote a good cure in the laminate, i.e, the total degree conversion was around 100%.

Considering that the autoclave processing involves several steps, such as reinforcement and resin choice, vacuum/pressure cycles, etc the system heating must consider the heating rate and the initial and final temperature of polymerization. The glass fiber/epoxy composite processing steps can be executed with safety and reliability. Using the DSC informations is it possible to establish the appropriate cure cycle of glass fiber/epoxy *F155* resin system in order to produce a composite material with quality to be used as aircraft structural part.

3.2. Moisture absorption

Fig. 6 shows a graph of the weight increase as a function of exposed time for glass fiber/epoxy composites specimens exposed at 80°C and 90% RH. 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 [10–12]. As a result of different fiber orientation in composite materials, moisture can penetrate more or less inside of the polymeric composites. It was observed that bi-directional composites leads to a lower rate of moisture absorption compared to unidirectional reinforced composites, due to the edges effect [12–14]. The bidirectional reinforced composites presented a higher resin rich area in the edge of the composite when compared with unidirectional reinforced composite, presenting a higher absorption rate [12–14].

The kinetics of the diffusion process depends on the temperature and relative moisture absorption. The higher the relative moisture absorption the greater is the absorption rate. This diffusion process can be described by Fick's law [10–12].

In was observed in Fig. 6 that the saturation for glass fiber/epoxy composites occurred at 6 weeks of exposure.

3.3. Study of natural frequencies

Fig. 7a–e and Table II present the resonant frequency results of all specimens studied in this work. As can be observed, the hygrothermal effect was not to be enough to change the frequency values of the composite studied.

The first mode of vibration was used in order to calculate the E' modulus, due to this mode is the dominant wavelet level.

It was observed in the Table II that the all the natural frequencies for the samples studied decreased with the increase of exposed time in hygrothermal conditioning. This behavior is due to the matrix plasticization by the moisture. Glass Fiber/epoxy specimens plasticized presents a lower stiffness under damping tests. The first mode value was used in the Equation 4 in order to study the E' of this composite

3.4. Influence of moisture absorption in the storage module (E')

Four primary mechanisms have been suggested to contribute to damping in composites: viscoelastic response

TABLE II Frequency values of materials exposed in hygrothermal conditions, analyzed up to 500 Hz

Exposure time (days)	First frequency (Hz)	Second frequency (Hz)	Third frequency (Hz)
0	24.0	167	475
15	24.0	168	479
30	23.4	177	492
45	23.4	179	494
60	23.2	180	497

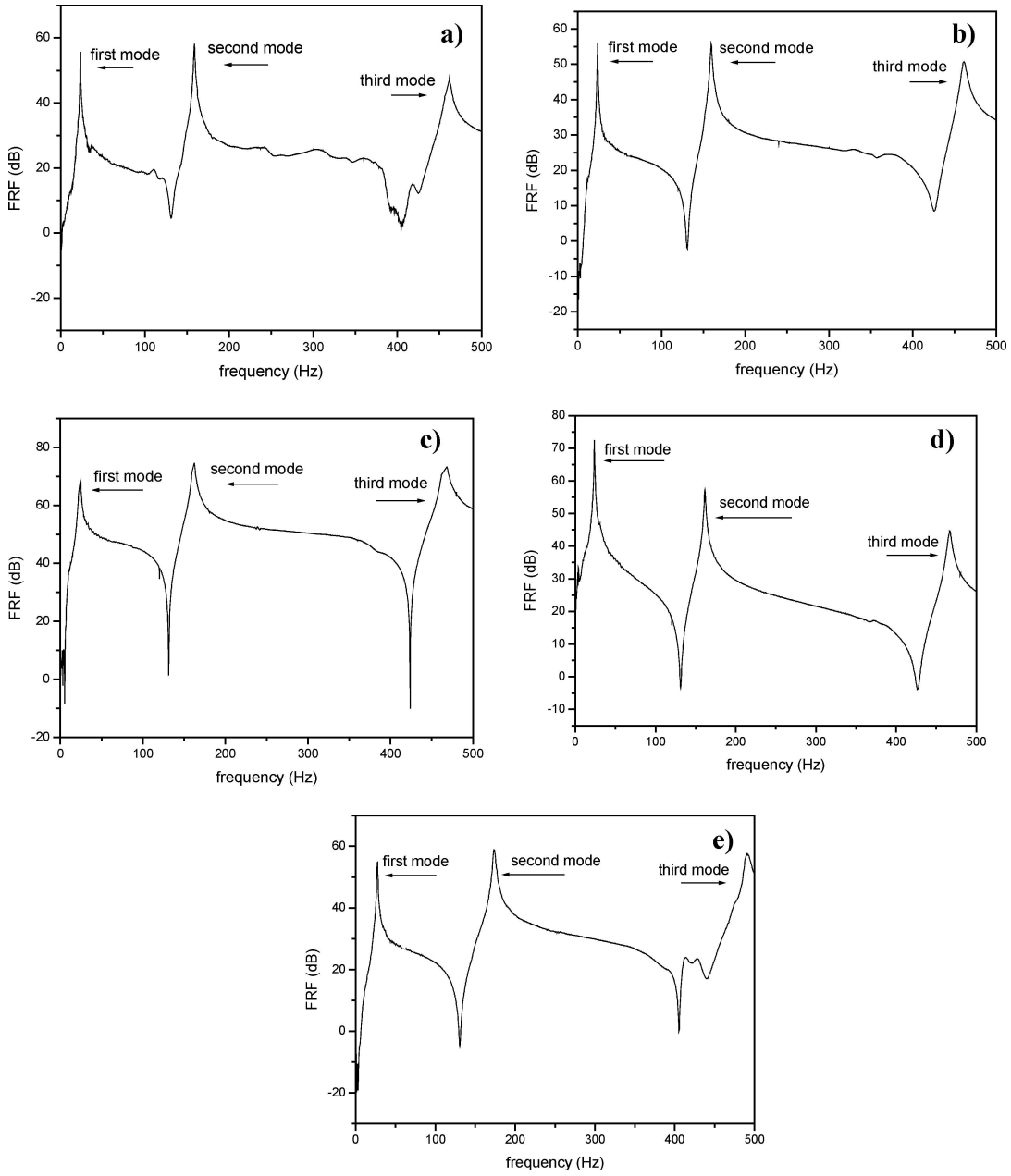

Figure 7 Resonant frequency results from glass fiber/epoxy composite laminate submitted to hygrothermal conditions: (a) 0 day; (b) 15 days; (c) 30 days; (d) 45 days; (e) 60 days.

of the constituents, friction and slipping at the fiber-matrix interface, thermoelastic damping due to cyclic heat flow and damage initiation and growth. 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 [19].

Fig. 8a–e presents the vibration damping curves representatives of the composite laminate specimens. The curves show an exponential decay of maximum peak amplitudes as a function of time. The amplitude decays of composites submitted to hygrothermal conditioning during more time are more pronounced due to the lower stiffness.

It was observed that the damping behavior of the glass fiber/epoxy composites depends on the moisture absorbed in the matrix. The damping factor of the dry glass fiber/epoxy composites ($\zeta = 1.01 \times 10^{-2}$) was very close to the values found for this composite after to be submitted during 15 days to hygrothermal conditioning. This behavior indicates that the dry specimens and the specimens of the glass fiber/epoxy conditioned during 15 days have similar stiffness and so as resilience. However, the damping factor value obtained by this specimen after to be submitted above 30 days increased with the increase of the exposure time.

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 beam specimen. Thus, there is an adding hygrothermal effect of the interface on damping behavior in composites which is more pronounced for this composite submitted in humidity atmosphere.

Results from Table III and Fig. 9 show that is also possible to model the damping factor contribution of

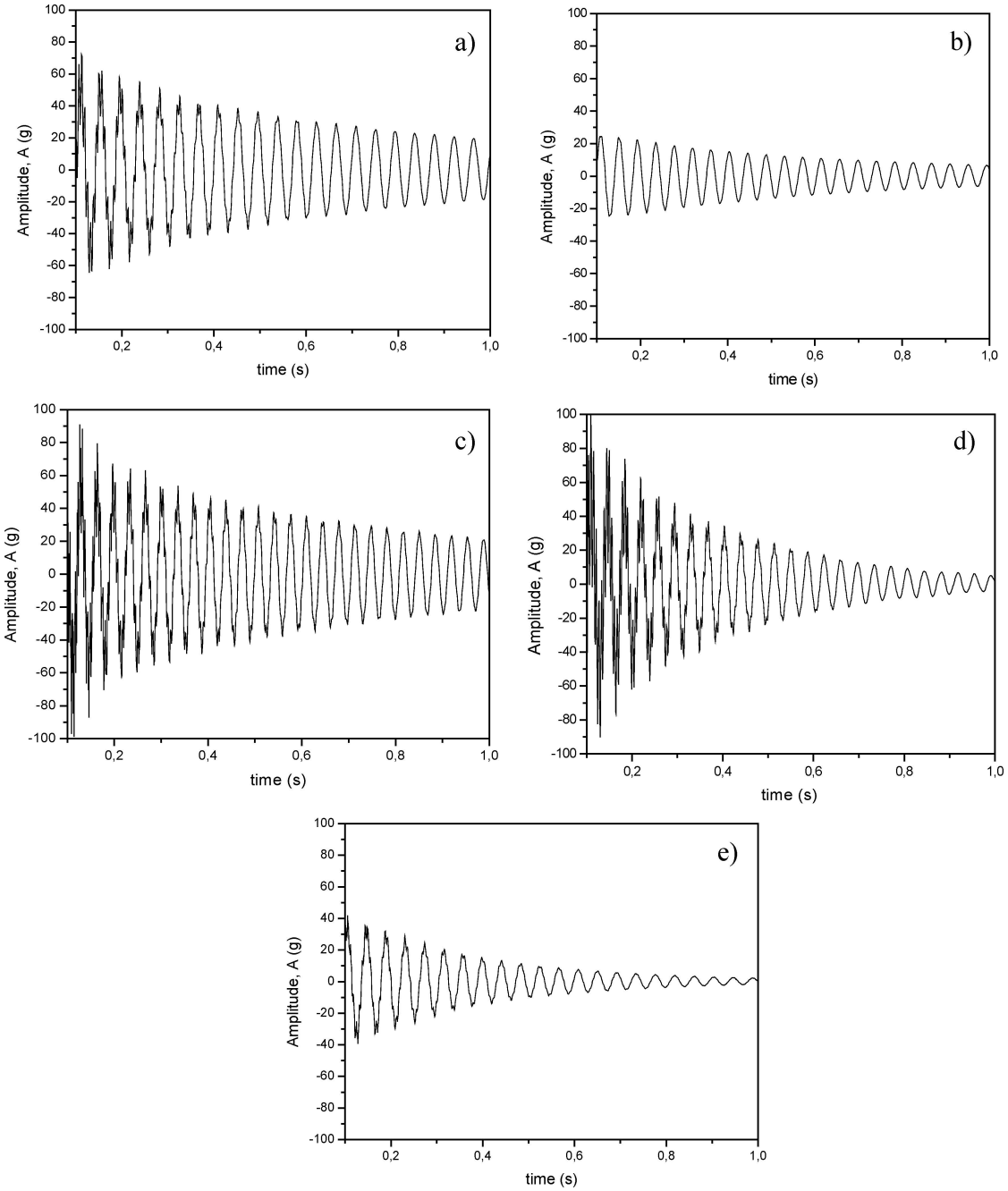

Figure 8 Damping behavior curves from composite laminate specimens studied submitted to hygrothermal conditions: (a) 0 day; (b) 15 days; (c) 30 days; (d) 45 days; (e) 60 days.

the composites by using the simple plot of moisture content versus damping factor as presented in Fig. 9. According to this Fig. 9, the damping factor increase around of 85% up to the saturation (6 weeks) and 110% up to 60 days.

TABLE III Damping results obtained for the composite materials studied

Exposure time (days)	Interia (m^4)	ζ	E' (MPa)	E (MPa)*
0	1.33×10^{-11}	$1.01 \times 10^{1-2}$	26.7 ± 2.3	30.6
15	1.33×10^{-11}	0.93×10^{-2}	26.7 ± 2.7	–
30	1.33×10^{-11}	1.13×10^{-2}	25.4 ± 2.8	–
45	1.33×10^{-11}	1.87×10^{-2}	24.7 ± 2.3	–
60	1.33×10^{-11}	2.10×10^{-2}	24.7 ± 3.2	–

*calculated by composite micromechanics approach.

The theoretical elastic constant was calculated using composite micromechanics approach. The theoretical calculations for the dry glass fiber textile/epoxy composite studied have an elastic modulus (E) of 30.6 GPa. Experimental measurements of elastic modulus of glass fiber/epoxy composite 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 (Table III) of the $\sim$13% is expected. For the composites submitted to hygrothermal conditioning there is an additional factor related to the influence of moisture, which is not considered also in the theoretical calculations. The result of the elastic modulus for the glass fiber/epoxy composite, Table III, shows a good

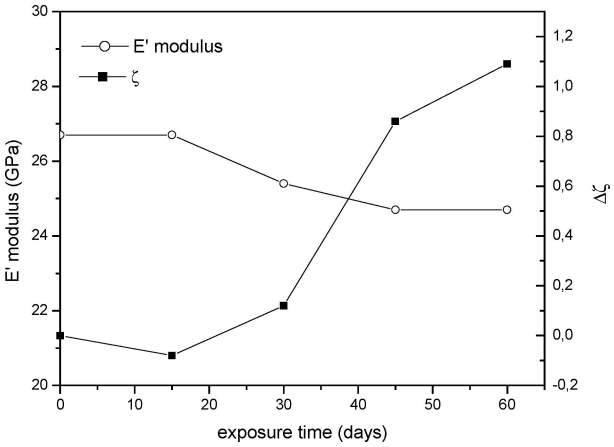

Figure 9 Elastic modulus (E') and variation of the damping loss ($\Delta\zeta$) curves from composite laminate specimens studied submitted to hygrothermal conditions.

agreement between the value found in the literature and the experimental value [27].

Table III and Fig. 9 shows the E' modulus obtained by the glass fiber/epoxy composites. In all specimens analyzed, moisture uptake always induced resin plasticization and, consequently, **decrease** the E' modulus values of the laminates. According with these results, up to the saturation, E' modulus values decreased in 19% due to the moisture effects in the interface between the reinforcement and epoxy matrix. Above the saturation, this values maintenance constant.

3.5. Influence of moisture absorption in the loss factor (tan δ) and loss modulus (E'')

The loss modulus is proportional to the E' and ζ values, and it is related to the energy dissipation mechanisms in materials. In composites, such as glass fiber/epoxy laminates, the loss modulus is also a combination of energy dissipation mechanisms from the fiber and epoxy interface between them. So, in this case the energy dissipation due to interfacial adhesion can play the role.

Table IV presents the E'' values for glass fiber/epoxy composite materials submitted during different times under hygrothermal conditioning .It can be observed in Table 4, the E'' values for dry composite and saturate composite (submitted after 60 days in hygrothermal conditioning) was 0.41 and 1.05 MPa, respectively. Thus, the wet glass fiber-composite dissipates more energy per cycle of damping than the dry glass fiber composite. The value of the dynamic modulus of the wet composite is tree times the value of the dynamic modulus of the dry composite. Moreover, the damping factor of the wet composite is twice higher than for the dry composite. Consequently, the energy dissipation in the wet composite is higher than the dry composite, as can be seen in the amplitude decay curves of Fig. 8a and e.

TABLE IV Loss factor (tan δ) and loss modulus (E'') results for the composite materials studied

Exposure time (days)	tan δ	Increase of tan δ (%)	E'' (GPa)
0	1.54×10^{-2}	reference	0.41
15	2.02×10^{-2}	31	0.54
30	1.85×10^{-2}	20	0.47
45	4.25×10^{-2}	176	1.05
60	3.77×10^{-2}	145	0.93

Table IV shows that the E'' modulus only increase significatively around of 30 days after the samples to be submitted to hygrothermal conditioning. This behavior is due to the plasticization of the sample near to saturation region (around 45 days). The behavior of E'' modulus agree with the natural frequency, E' and ζ values found in this work.

Table IV shows the values found by tan δ (loss factor) of the composites studied. The damping of composites is mainly controlled by tan δ of the interface between the reinforcement and matrix. The loss factor was calculated from the decaying-oscillatory damping curve by the Equation 5. It was observed that a higher value of tan δ reflects poor adhesion in the system. Results shown in Table IV indicate that the sample submitted to hygrothermal conditioning appear to have the weakest fiber-matrix interfacial adhesion when compared with dry specimen (strongest interfacial adhesion).

Therefore, Table IV shows that the tanδ values only increase significatively around of 45 days after the samples to be submitted to hygrothermal conditioning. This behavior is due to the plasticization of the sample near to saturation region. In the saturation region (after 45 days) the tan δ values increased of 176% when compared with dry composite. After to be submitted to hygrothermal conditioning during 60 days, the increase of tan δ was 145%.

The behavior of tan δ agree with the natural frequency, E', E'' and ζ values found in this work. It is thus even more interesting to note that the vibration damping method is more sensitive to the fiber-matrix interface, and is likely to be a more promising technique to detect the fiber-matrix interfacial adhesion.

4. Conclusion

The relations between the dynamic mechanical properties, damping test and the influence of high moisture concentration of glass fiber reinforced composites (plain weave) were studied. In was observed that the saturation for glass fiber/epoxy composites occurred at 6 weeks of exposure.

During vibration tests, it was observed that all the natural frequencies and E' values decreased and damping factor for the samples studied increased with the increase of exposed time in hygrothermal conditioning. This behavior is due to the matrix plasticization by the moisture. Glass Fiber/epoxy specimens plasticized presents a lower stiffness under damping tests.

It can be observed that the E'' values for dry composite and saturate composite (submitted after 60 days in hygrothermal conditioning) was 0.41 and 1.05 MPa, respectively. Thus, the wet glass fiber-composite dissipates more energy per cycle of damping than the dry glass fiber composite. The value of the dynamic modulus of the wet composite is tree times the value of the dynamic modulus of the dry composite. Moreover, the

damping factor of the wet composite is twice higher than for the dry composite. Consequently, the energy dissipation in the wet composite is higher than the dry composite.

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