HYGROTHERMAL EFFECTS ON VISCOELASTIC BEHAVIOR OF CARBON FIBER REINFORCED PEKK THERMOPLASTIC COMPOSITES

Rogério L. Mazur[1], Edson C. Botelho [1], Amanda M. Cunha [1], Mirabel C. Rezende [2]

[1] *Dept. de Materiais e Tecnologia, São Paulo State University-UNESP, Guaratinguetá, São Paulo, Brazil* (ebotelho@feg.unesp.br).
[2] *Div. de Materiais, Instituto de Aeronáutica e Espaço- CTA, São José dos Campos, São Paulo Brazil.*

ABSTRACT

In the last years, thermoplastic composites have received much interest for structural applications. Among the available thermoplastic composites, carbon fiber reinforced PEKK composite shows excellent balance of properties, including higher glass transition temperature, high strength and stiffness, high toughness, low moisture absorption and good environmental resistance. Thermoplastic composites can present problems due to its moisture absorption. When this occurs, the matrix suffers hydrolytic degradation due to the water attack, causing the plasticizing effect and consequently degradation of the composite mechanical properties. The aim of present work is to evaluate the influence of the hygrothermal effects on carbon fiber reinforced PEKK thermoplastic composites by dynamic mechanical thermal analysis (DMTA) and free vibration damping methods. The viscoelastic properties at room temperature, obtained by dynamic mechanical thermal analysis presented a high glass transition temperature when compared with the specimens submitted to hygrothermal conditioning. In this work it was also observed that the viscoelastic values obtained by free vibration damping were lower than the obtained by the DMTA. This behavior can be relationship with defects of specimen, such as, alignment of fibers, size, voids and other defects.

1. INTRODUCTION

Advanced composite materials have experienced a rapid development in aircraft and aerospace structures in the last years. As observed by Daniel [1], some of the reasons for this development are significant progress in materials science and technology, with high values of mechanical strength and stiffness and low density together with development of powerful and sophisticated numerical methods for structural analysis. Later, cost competitiveness with more conventional materials became equally important. For these two requirements nowadays is added the need for quality assurance, reproducibility and predictability of behavior over the lifetime of the structure [1].

Among the available advanced composites, carbon fiber/PEKK (poly (ether ketone ketone)) shows excellent balance of properties, including excellent thermal stability, low moisture absorption, and excellent flammability resistance, high toughness and tensile modulus. PEKK has a glass transition temperature, T_g of 156°C and melting temperature, T_m of 305-310°C [2-4].

In service, failures of carbon fiber-reinforced thermoplastic composites are commonly attributed to ageing of the material in its particular environment, brought about by a combination of the effects of heat, light, water and mechanical stresses on the material. Thermoplastic composites can present problems due to its moisture absorption. When this occurs, the moisture plasticizes the matrix, leading to a reduction in Tg and changes mechanical properties, such as Young's modulus. As discussed previously, the glass transition temperature, Tg, is an important material property because it defines the temperature at which material properties are drastically reduced as the matrix changes from a glass, stiff state to a pliabe one[5].

The aim of the present work is to evaluate the influence of the hygrothermal effects on carbon fiber reinforced PEKK thermoplastic composites by dynamic mechanical thermal analyses and free vibration damping methods. The measurements were performed before and after submitting the composites to hygrothermal conditioning.

2. EXPERIMENTAL

2.1 Materials

The PEKK/carbon fiber reinforced thermoplastic composite was produced at UNESP/CTA by using hot compression molding. As reinforcement were used 12 plies of carbon fiber (plain weave).

2.2 Environmental conditioning

In order to evaluate the influence of the hygrothermal effects on carbon fiber reinforced PEKK thermoplastic composite on the damping results, the specimen was exposed to a combination of temperature and humidity in an environmental conditioning chamber. The condition selected to saturate the specimen before the tests were based on Procedure B of ASTM Standard D 5229 M-92. The moisture level in the laminate was periodically monitored as a function of the 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%.

2.3 Dynamic mechanical thermal analysis

The dynamic mechanical thermal behavior of specimen was evaluated by a Thermal Analyzer TA 2980 of TA Instruments, operating in the three-point bending horizontal measuring system. The experimental conditions used were: dynamic force (0.7 Nm); oscillation displacement (10 μm); 1.0Hz frequency, heating rate (3.0 °C/min) and temperature range of 30-250°C.

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 of a prismatic cantilever beam excited by tapping it with an appropriate hammer, as shown in Figure 1.

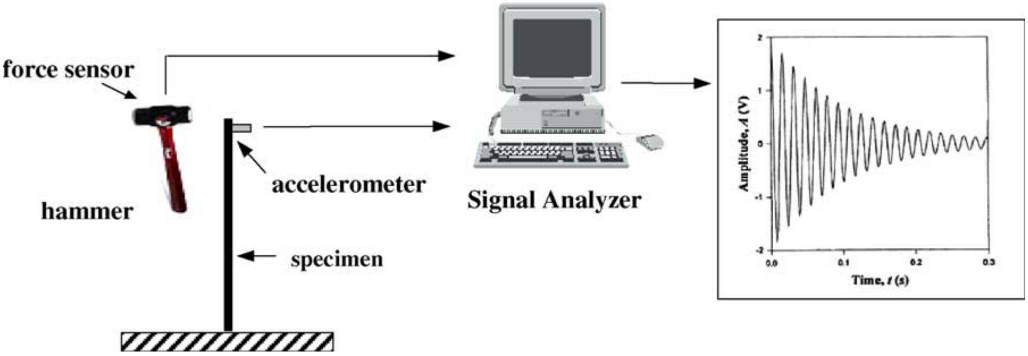


Figure 1: Experimental set-up.

The amplitude decay as a function of the time and vibration modes were detected to an acquisition data system from Spectral Dynamics Company and calculated using software LMS CADA-PC. The test parameters were: scanning analysis range up to 500 Hz; acquisition time of 200 ms; observation window, rectangular; frequency resolution, 5 Hz. The amplitude decay was measured using a 0.6 g accelerometer. Beam dimensions were: length: 0.20m; width: 0.02m and thickness: 0.0027m. The non-conditioned specimen weight was 14.54g. Using this procedure, it was obtained the free vibration damping curve.

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 equation 1.

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

where: n = number of peaks; δ_1 = amplitude of the first peak and δ_n = amplitude the final peak analyzed. In this work, the storage modulus (E') was obtained according to equation 2.

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

where: E'= elastic modulus; f = natural frequency; I = inertial moment; M = accelerometer weight; m = specimen weight and L = specimen length and Δ = damping factor. The loss factor, tan δ, was calculated from the decaying-oscillatory damping curve as follows, given by the equation 3.

$$n = \tan\delta = \frac{\ln(\delta_1/\delta_n)}{\pi} \tag{3}$$

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 analysed. Loss Modulus (E") can be calculated by using the equation 4.

$$\tan\delta = \frac{E''}{E'} \tag{4}$$

4. RESULTS AND DISCUTION

4.1 Moisture absorption

Figure 2 shows a graph of the weight increase as function of exposed time (root of hours) for carbon fiber reinforced PEKK thermoplastic composite specimen exposed at 80°C and 90% RH. It is observed in Figure 2 in the first days a linear weight increase for composite until the saturation point (50 days), reaching a moisture absorption around 0.20% mass. As observed by literature[5-7], thermoplastic resin absorb very low levels of moisture (they are generally much less polar than thermoset resins).

Moisture absorption in polymers takes place through of a diffusion process, in which water molecules are transported from areas with high concentration to areas with lower moisture concentration. The diffusion process depends on the temperature and relative moisture absorption and can be described by Fick's law. It is characterized experimentally by an uptake of moisture that reaches an asymptotic value after a period of time as showed in Figure 2.

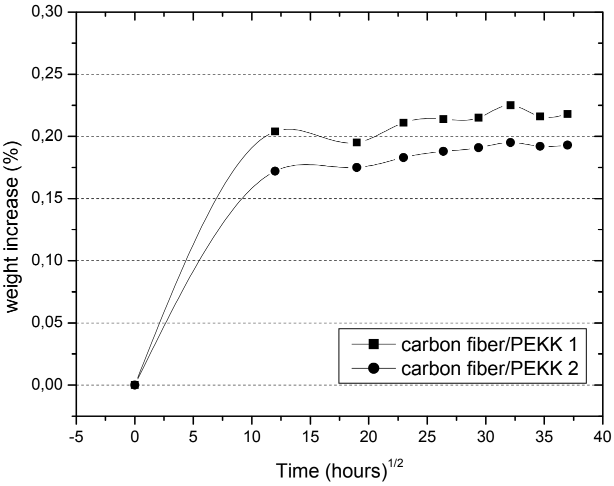
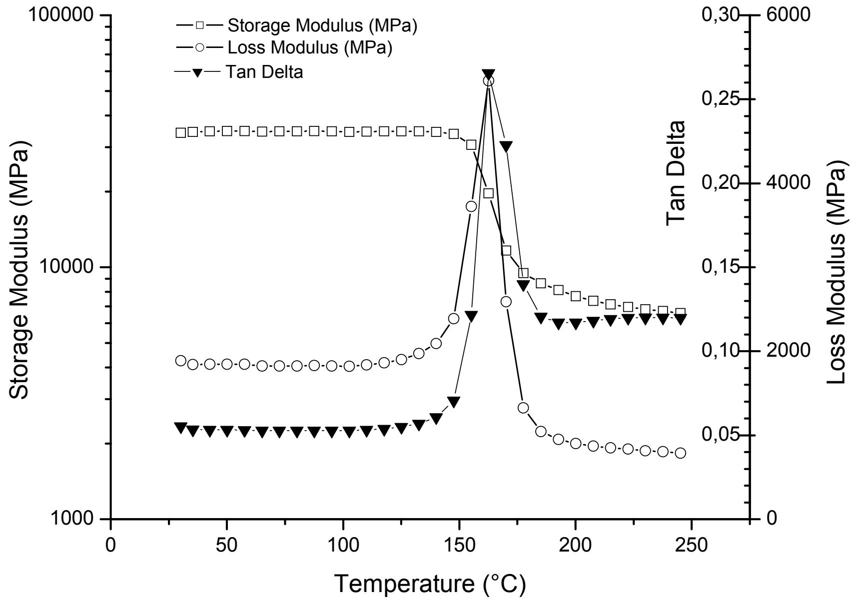


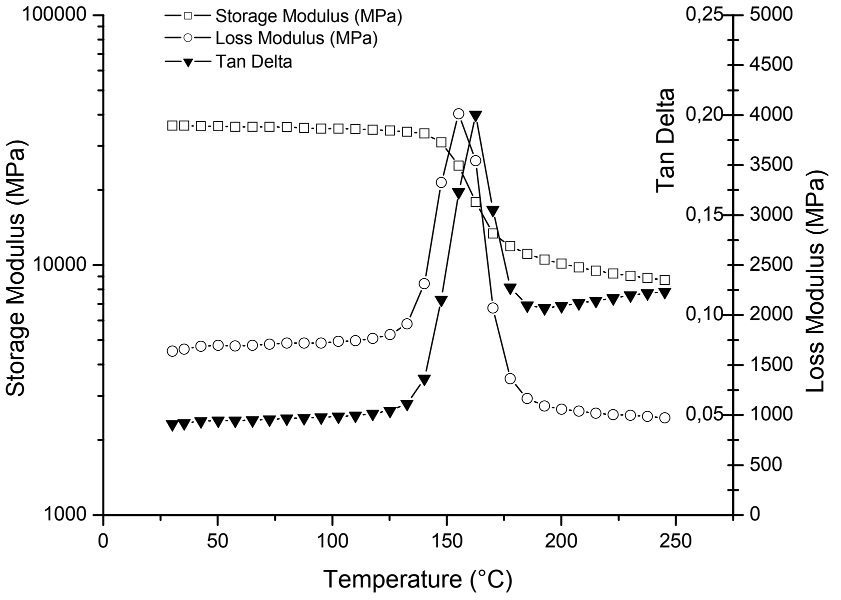
Figure 2: Moisture absorption of carbon fiber reinforced PEKK thermoplastic composite.

4.2 Dynamic mechanical thermal analysis

Figure 3a-b shows the curves of dynamic mechanical thermal analysis of dry and wet specimen of carbon fiber reinforced PEKK thermoplastic composite. As can be observed in Figure 3 the non-conditioning specimen showed a (Tg) glass transition temperature of approximately 162°C, differently from the specimen submitted to the hygrothermal conditioning (156°C). This behaviour probably can be relation with plasticization effects on polymeric matrix leading to a reduction in Tg and changes mechanical properties, such as Young's modulus.



a)



b)

Figure 3: Dynamic mechanical thermal analysis (DMTA) as function of temperature: (a) dry laminate: (b) wet laminate.

4.3 Vibrational Test

Figure 4 shows the vibration damping curves of carbon fiber reinforced PEKK thermoplastic composite. As can be observed, the curve exhibits an exponential decay of maximum peak amplitudes as a function of the time. As observed in the literature [8-9], four primary mechanisms have been suggested to contribute to damping in composites: viscoelastic response of the constituents, friction and slipping at the fiber-matrix interface, thermoelastic damping due to cycle heat flow and damage initiation and grow.

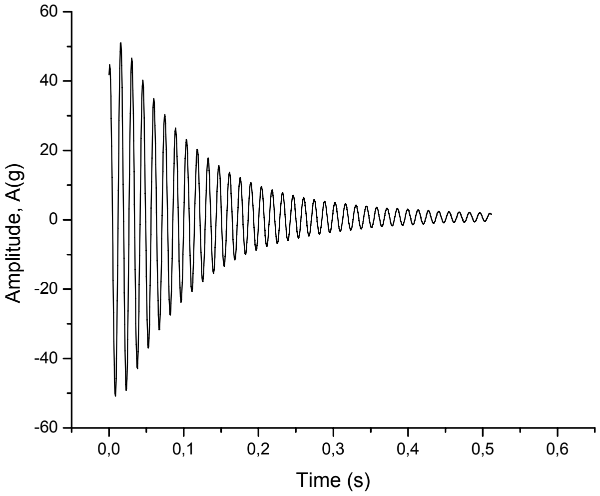


Figure 4: Vibration damping curves from composites laminates specimen studied.

As mentioned earlier, the storage modulus (E') for specimen was obtained by using Eq. 2 and the parameters such as: damping factor (Δ) and frequency (*f*) were obtained from Figure 4. Table 1 shows the frequency (Hz), damping factor (Δ), storage modulus (E'), loss modulus (E") and loss factor (tan δ) values for the tested specimen.

Table 1: Elastic properties of the laminate studied of carbon fiber reinforced PEKK thermoplastic composite.

Properties	**Vibration test of carbon fiber/PEKK**
Frequency (Hz)	66.66
Damping factor (Δ)	0.1137
Storage Modulus E' (GPa)	29.38
Loss Modulus E" (GPa)	1.07
Tan δ	0.0364

In this work it was observed that the storage modulus for carbon fiber reinforced PEKK thermoplastic composite for non-conditioned specimens, obtained by free vibration damping test was ~16% lower when compared with elastic modulus (35 GPa) obtained by DMTA. The same behavior was observed about the loss modulus (32% lower) and tan δ (27% lower) values. This behavior can be relationship with defects of specimen, such as, alignment of fibers, size, voids and other defects. So, the differences between elastic modulus (E') obtained by vibration test and dynamic mechanical thermal analysis analysis are expected.

6. CONCLUSIONS

In this work, it was observed that for all the composite specimens studied the moisture saturation point occurred after 50 days of exposure. During DMTA tests, it was observed that the E' and glass transition temperature values decreased with an increase as a function of time during hygrothermal conditioning. This behavior happened due probably to the matrix plasticization by the moisture.

When compared E' values obtained by free vibration damping and DMTA tests, it was observed a diffence of aroud 16%. This behavior happened probably due to the diference of the specimen sizes and, consequently, due to a higher presence of voids, and alignment of fibers into the laminates used as specimens for free vibration damping tests.

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REFERENCES

1. Daniel I.M.; Ishai O.; "*Engineering Mechanics of Composites Materials",* New York, Oxford University Press,1994, 1-395.
2. Marchand V., Authesserre J., Pouyet J., Bacon C.;"Determination of the elastic constants of materials, in the form of plates, by a free vibration method", *Journal of Sound and Vibration*, 1996:194(4):497-512.
3. Zhang Z, Hartwig G.;"Low-temperature viscoelastic behavior of unidirectional carbon composites", *Cryogenics*: 1998:38:401-405.
4. Shokrieh M.M., Najafi A.;"Experimental Evaluation of Dynamic Behavior of Metallic Plates Reinforced by Polymer Matrix Composites", *Composites Structures*, 2006:75:472-478.
5. Scida D., Aboura Z., Benzeggagh M.L.;"The effect of Ageing on the Damage Events in Woven-Fibre Composite Materials under Different Loading Conditions", *Composites Science and Technology*, 2002:62:551-557.
6. Botelho E.C., Rezende M.C, Pardini L.C.;"Hygrothermal Effects on Damping Behavior of Metal/Glass Fiber/Epoxy Hybrid Composites", *Materials Science and Engineering A*, 2006:399:190-198.
7. Jang. B.Z., "Advanced Polymer Composites: Principles and Applications". First edition, ASM International, OH, USA, 1994.
8. Botelho E.C., Costa M.L., Pardini L C., Rezende M.C.; "Processing and hygrothermal effects on viscoelastic behavior of glass fiber/epoxy composites", *Journal of Materials Science,* 2005:40:23615-3623.
9. Botelho E.C., Campos A.N., Barros E., Pardini L.C., Rezende M.C.; "Damping Behavior of Continuous Fiber/Metal Composite Materials by the Vibration method", *Composites: Part B*, 2006:37:255-263.