VISCOELASTIC PROPERTIES OF COMPOSITE LAMINATED BEAMS BY DYNAMIC MECHANICAL ANALYSIS

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Abstract. *Viscoelastic material characterization has become a topic of great interest for engineers working with design and analysis of composite material structures. Recently, an approach leading to the complete 3-D viscoelastic characterization of transversely isotropic laminae, using a reduced number of measured parameters, was developed. The approach allows viscoelastic properties to be evaluated from experimental data collected from bend-beam oscillatory tests. In this investigation, viscoelastic properties of cross-ply laminated beams are measured using Dynamic Mechanical Analysis (DMA) equipment. The DMA measured properties of laminates are compared with predicted properties, computed according to an existing mechanics model. The results indicate good agreement between measured and predicted laminate properties. Thus, this study demonstrates the capability of this DMA-based approach to measure viscoelastic properties of laminates, which ultimately can be used in the design and analysis of composite structures.*
Keywords*. Damping; Composite Laminates; Dynamic Mechanical Analysis.*

1. Introduction

The design of modern structures often requires material capabilities, which are difficult to obtain with traditional materials. Such design requirements may include combinations of low-density, high-strength, high-stiffness, high damping, chemical resistance, thermal-shock resistance and many others. Unlike most traditional materials, properties of composite materials can be tailored to meet the design criteria and therefore offer a unique design solution. Due to these unique characteristics, composite materials have achieved wide use in aerospace structures, automotive parts, marine structures, and sporting goods such as tennis rackets, golf clubs, skis and snowboards. Moreover, in many structural applications where an improvement in specific stiffness and in damping characteristics can be beneficial, advanced composite materials are an appealing option as a substitute for conventional materials. This is the case, for example, in high-speed machinery, where dynamic stability and positioning accuracy are important design requirements. Further, it may be that tailorable damping becomes an enabling technology in the advancement of important consumer applications such as faster, higher storage density computer hard drives.

Damping is an essential material property for the design of many advanced structures especially when vibration and noise control are critical. In fact, some structures require specified levels of damping as a major design criterion. Variations in damping can also be potentially used as a way to understand processing related problems. Therefore, unlike the design of traditional structures, in the case of tailored damping, information about the elastic properties is very important, but not sufficient. Determining accurate viscoelastic properties of the material becomes fundamental.

Dynamic mechanical analysis (DMA) is a widely accepted research technique in the polymer industry, measuring modulus and damping over a wide range of frequency and temperature, and providing important information about the cure of thermoset resins and the aging of thermoplastics (Menard, 1999). This technique offers great potential for investigating damping properties of composite materials (Gibson, 2000; Sichina, 1987; Gill, 1983; Reed, 1980 and Melo, 2002). The method provides fast and reliable results using a very small amount of material, which can, in many cases, be taken directly from the part. In addition, DMA test equipment allows precise temperature and atmosphere control.

In most viscoelastic models of fiber-reinforced laminae, the number of independent loss factors to be determined requires experimental measurements that are not very practical (Saravanos and Pereira, 1995 and Wei et. al., 2001). Consequently, micromechanics methodologies are often used for the prediction of damping properties of composites. However, these methodologies are based on the properties of the fiber, matrix, and fiber volume fraction and, therefore, the accuracy depends upon the proper measurement of the constituent material properties, which is difficult to accomplish. Furthermore, in micromechanics based models, it is difficult to incorporate effects such as temperature and manufacture in the viscoelastic properties. This is due to the fact that these changes will not only affect the constituent material properties but also the fiber/matrix interface, which plays an important role in the mechanical behavior of the material (Brantseva et. al., 1999; Jeng and Chen, 1999 and Gaertner et al., 1999).

A DMA-based approach was recently developed to investigate viscoelastic properties of fiber-reinforced composites (Melo and Radford, 2003). A mechanics model was also developed, which describes the 3-D viscoelastic properties of a transversely isotropic material, based on five independent storage moduli and three damping loss factors (tan δ), which reduces the number of experimental procedures necessary. Based on this mechanics model, it has been determined, that, the computation of all 3-D viscoelastic properties can be accomplished from three DMA bending tests, providing that two Poisson's ratios, ν_{12} and ν_{23}, are known from elastic measurements. This approach has been successfully applied in the viscoelastic characterization of a carbon fiber reinforced unidirectional tape (Melo and Radford, 2003).

Following this previous work, the present investigation evaluates the feasibility of the experimental approach developed, using Dynamic Mechanical Analysis, to measure viscoelastic properties of laminates. Viscoelastic properties of cross-ply laminated beams are measured and the measured properties are compared with predicted laminate properties, based on measured laminae properties, and computed according to existing mechanics models.

2. Experimental procedure

2.1. Material and Test Specimens

The material tested in this investigation was APC-2/IM7, unidirectional prepreg tape, manufactured by Cytec Engineered Materials Inc., with a resin content of 32% and a fiber areal weight of 145g/m^2. Two 150.0 x 200.0 mm laminates, were produced in a hot-press: a 8 ply unidirectional $[(0)_8]_T$ and a cross-ply $[(0/90)_2]_s$. The laminates were processed at a temperature of 395°C for 45 minutes, under an applied pressure of 690 kPa (100 psi).

Beam specimens were cut from the processed laminates using a diamond abrasive saw. Unidirectional laminated beams were cut from the $[(0)_8]_T$ plate with fibers oriented at 0°, 30°, and 90°, with respect to the beam length. In this research, the properties of these unidirectional specimens are assumed to be a true representation of the properties of one unidirectional layer. Therefore, the measured properties of the unidirectional specimens are referred to as lamina properties. From the $[(0/90)_2]$s cross-ply laminate, laminated beams were also cut in three different angles, 0°, 45°, and 90°, resulting in specimens with stacking sequences of $[(0/90)_2]_s$, $[(+45/-45)_2]_s$, and $[(90/0)_2]_s$, respectively.

After the cutting procedure, the specimen edges were sanded flat for better dimensional precision. Finally, the specimens were conditioned to reduce effects of residual stresses and moisture introduced during specimen preparation. The conditioning procedure consisted of heating the specimen in an oven from room temperature to 70°C, holding for 2 hours, then heating from 70°C to 90°C followed by a hold for 2 hours, and finally, heating from 90°C to 110°C with a final hold for 2 hours. Upon cool-down, accurate dimensions of each specimen were measured using a hand-held micrometer. The specimen dimensions were nominally 50.0 x 4.0 x 1.0 mm (LxWxH), as shown in Fig. (1).

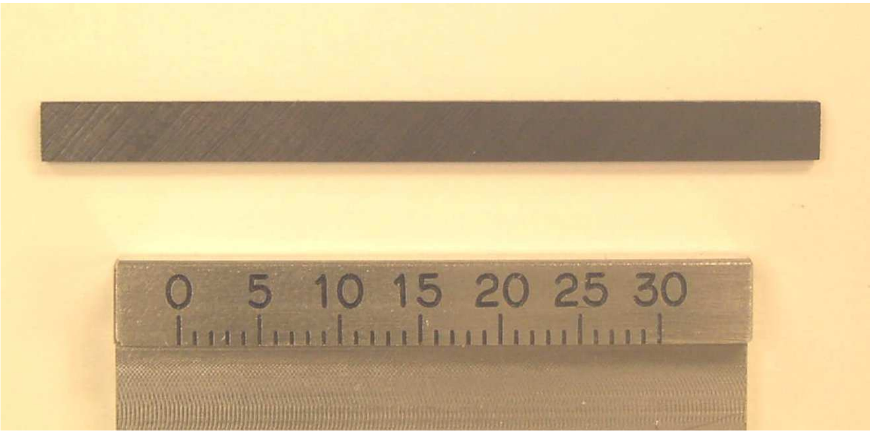

Figure 1 - Bend-beam specimens for viscoelastic characterization.

2.2. Laminae viscoelastic characterization

The viscoelastic properties of PEEK/IM7 unidirectional prepreg were determined according to a previously developed experimental approach, which is based on a mechanics model using a reduced number of measured parameters (Melo and Radford, 2003). According to this reduced model, the complex moduli of transversely isotropic materials, can be written in matrix form as,

$$\left[C^*\right] = \left[C'\right] + i\left[C'\right]\left[\eta\right] \quad (1)$$

Where: C^* are the complex moduli, C' are the storage moduli and η are the loss factors (tan δ).

Then, if symmetry conditions of transversely isotropic materials are considered, only five storage modulus parameters and three damping coefficients, are independent. Thus, the two matrices can be expressed in contracted form as (Melo and Radford, 2003).

$$\begin{bmatrix} C'_{11} & C'_{12} & C'_{12} & 0 & 0 & 0 \\ C'_{12} & C'_{22} & C'_{23} & 0 & 0 & 0 \\ C'_{12} & C'_{23} & C'_{22} & 0 & 0 & 0 \\ 0 & 0 & 0 & C'_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C'_{66} & 0 \\ 0 & 0 & 0 & 0 & 0 & C'_{66} \end{bmatrix} \quad (2)$$

and

$$\begin{bmatrix} \eta_1 & 0 & 0 & 0 & 0 & 0 \\ 0 & \eta_2 & 0 & 0 & 0 & 0 \\ 0 & 0 & \eta_2 & 0 & 0 & 0 \\ 0 & 0 & 0 & \eta_2 & 0 & 0 \\ 0 & 0 & 0 & 0 & \eta_6 & 0 \\ 0 & 0 & 0 & 0 & 0 & \eta_6 \end{bmatrix} \quad (3)$$

Therefore, according to this mathematical model, the viscoelastic constitutive relationships of transversely isotropic materials can be described based on eight parameters: five independent dynamic stiffness parameters, E'_1, E'_2, G'_{12}, $\nu'_{12} = \nu_{12}$, and $\nu'_{23} = \nu_{23}$, plus three independent damping loss factors (tan δ), η_1, η_2, and η_6.

An experimental investigation was conducted to determine the independent viscoelastic parameters. First, the Poisson's ratios, ν_{12} and ν_{23}, for the prepreg material were determined according to an experimental technique, which uses coefficient of thermal expansion measurements in laminates (Melo and Radford, 2002). Then, sub-scale flexure tests in unidirectional beams with fibers oriented at 0°, 30°, and 90°, with respect to the beam length, were conducted. According to the procedure previously developed (Melo and Radford, 2003), the storage moduli, E'_1 and E'_2, and the damping loss factors, η_1 and η_2, can be measured directly from the oscillatory bend-beam tests of unidirectional laminae with fibers oriented at 0° and at 90°, with respect to the beam length. Then, the storage shear modulus, G'_{12}, and the damping constant, η_6, are computed based on the measured storage moduli, E'_1, E'_2, and E'^b_{xx}, damping loss factors, η_1, η_2, and η_x, and a measured value of the Poisson's ratio, ν_{12}, where E'^b_{xx} and η_x are measured from a bending test performed in an off-axis unidirectional beam specimen, with a predetermined intermediate angle θ, where $0° < \theta < 90°$. In this study, the included angle of the off-axis beam specimen was 30°. The equations used for the computation of the shear properties are (Melo and Radford, 2003):

$$\eta_x = \frac{\Delta W}{2\pi W} = \frac{1}{\bar{S}'_{11}} \left\{ \frac{\eta_1}{E'_1} \cos^4(\theta) + \frac{\eta_2}{E'_2} \sin^4(\theta) + \left[\frac{\eta_6}{G'_{12}} - (\eta_1 + \eta_2) \frac{\nu_{12}}{E'_1} \right] \sin^2(\theta) \cos^2(\theta) \right\} \quad (4)$$

$$\bar{S}'_{11} = S'_{11} \cos^4(\theta) + S'_{22} \sin^4(\theta) + (2S'_{12} + S'_{66}) \cos^2(\theta) \sin^2(\theta) \quad (5)$$

The dynamic mechanical tests were performed in a Rheometric Scientific DMTA-V, in a 3-point bending mode, with a 40.0mm span between the supports. All tests were carried out under sinusoidal strain-control. A very small strain amplitude (0.01%) was used throughout the measurements since the model applied, for viscoelastic material characterization, assumes that the specific damping capacity is independent of the amplitude of strain, which is a valid assumption only for very small strains.

Thus, using the measured viscoelastic properties for the beam specimens with fiber orientations of 0°, 30°, and 90°, and the Poisson's ratios, ν_{12} and ν_{23}, the viscoelastic characterization of PEEK/IM7 is complete, according to the previously developed experimental approach.

2.3. Laminate properties

After the determination of the lamina viscoelastic properties, these properties are used with a previously published mechanics model to predict viscoelastic properties of laminates. The predicted laminate properties are then compared with DMA measured properties for verification.

The laminate model considered uses the relationship between strain energy and dissipated energy for the determination of the loss factors of the laminated beams (Adams, and Maheri, 1994). Thus, considering a thin laminated beam composed of transversely isotropic layers, the damping factor is given by

$$\eta^b = \frac{\Delta W}{2\pi W} = \frac{\sum_{k=1}^{n} f^{(k)} g^{(k)} \left(z_k^3 - z_{k-1}^3 \right)}{3 d'_{11}} \quad (6)$$

where $f^{(k)} = \overline{Q}'^{(k)}_{11} d'_{11} + \overline{Q}'^{(k)}_{12} d'_{12} + \overline{Q}'^{(k)}_{16} d'_{16}$, $g^{(k)} = \overline{\eta}_{11}^{(k)} d'_{11} + \overline{\eta}_{12}^{(k)} d'_{12} + \overline{\eta}_{16}^{(k)} d'_{16}$, k is the layer number, z is the distance from the mid-plane of the beam, $\overline{Q}'^{(k)}_{ij}$ are the components of the reduced stiffness matrix in laminate coordinates, and d'_{ij} are the components of $[d'] = [D']^{-1}$, where the bending stiffnesses, D'_{ij}, are primed to indicate that they are based on storage moduli.

For the determination of the beam's storage modulus, the beam equation is written as

$$\kappa_x = \frac{M}{E'^b_{xx} I} = d'_{11} M_x \tag{7}$$

Thus,

$$E'^b_{xx} = \frac{12}{h^3 d'_{11}} \tag{8}$$

where $M = M_x b$ is the applied moment, and b is the beam width.

The model presented in Eqs. (6)-(8), for the computation of the loss factor and storage modulus of a laminated beam, has been presented in previous research (Adams, and Maheri, 1994). This model is consistent with the Eqs. (4) and (5) used, in this work, for unidirectional beams. Therefore, for a beam where all layers have the same included angle θ, Eqs. (6)-(8) produce the same properties as the model used for unidirectional beams given in Eqs. (4) and (5).

The application of the measured viscoelastic properties of the prepreg laminae, in the laminate model presented, Eqs. (6)-(8), allows the determination of the viscoelastic properties, storage modulus and loss factor, of the laminated beams. These calculated properties are then compared with experimentally measured data, for verification.

3. Results and discussion

The measured dynamic properties of the three unidirectional PEEK/IM7 beams are presented in Tab. (1). The table shows the storage moduli and the loss factors (tan δ) for unidirectional beams with fibers oriented at 0°, 30°, and 90°, with respect to the beam length.

Table 1. Measured Properties for PEEK/IM7 unidirectional beams.

E'_0 (GPa)	154.9
E'_{90} (GPa)	10.0
E'_{30} (GPa)	28.5
η_0 (10^{-3})	5.1
η_{90} (10^{-3})	7.2
η_{30} (10^{-3})	9.5

Using the DMA-measured properties shown in Tab. (1) and the two Poisson's ratios, ν_{12} and ν_{23}, previously determined (Melo and Radford, 2002), the viscoelastic parameters that characterize the unidirectional lamina were determined. The 3-D viscoelastic properties of transversely isotropic PEEK/IM7 are presented in Tab. (2).

Table 2. 3-D Viscoelastic Properties of PEEK/IM7 unidirectional lamina.

E'_1 (GPa)	154.9
E'_2 (GPa)	10.0
G'_{12} (GPa)	7.2
ν_{12}	0.34
ν_{23}	0.50
η_1 (10^{-3})	5.1
η_2 (10^{-3})	7.2
η_6 (10^{-3})	10.6

After the lamina viscoelastic characterization was complete, the viscoelastic properties, storage modulus and loss factor, of the three cross-ply laminated beams were experimentally measured. Also, the application of the measured lamina viscoelastic properties shown in Tab. (2) in the laminate model produced the predicted laminate viscoelastic properties. The property predictions for the laminates were based on laminae properties measured by the same equipment and according to the same procedures as the measured laminate properties. The measured viscoelastic properties for the three laminates are compared with the calculated values, using measured laminae properties, for verification. The results are presented in Tab. (3).

Table 3 – Measured and calculated viscoelastic properties of PEEK/IM7 laminated beams.

	$[(0/90)_2]_s$		$[(+45/-45)_2]_s$		$[(90/0)_2]_s$	
	E' (GPa)	η (10^{-3})	E' (GPa)	η (10^{-3})	E' (GPa)	η (10^{-3})
Measured	97.3	5.0	21.9	8.6	51.6	5.0
Predicted	110.2	5.2	24.3	9.9	55.6	5.4
Difference	11.7%	3.8%	9.9%	13.1%	7.2%	7.4%

The data presented in Tab. 3 show that the measured laminate storage modulus were, in all cases, about 10% smaller than the properties predicted by the laminate model. Further, for all three stacking sequences studied, the predicted loss factor, for each of the laminates, shows good agreement with the measurements.

A previous investigation with composite laminae did not indicate smaller modulus measurements by the DMA bend-beam tests as a trend (Melo and Radford, 2003). However, the laminate model used in this study to predict the properties of the laminated beams, assumes that all layers are perfectly bonded together. Thus, the lower measured values, compared to the predictions, may be an indication of debonding, or at least not perfect bonding, between layers. Further, the spacing between the fibers at the ply interface of a cross-ply specimen is expected to be greater compared to the distance at the ply boundary in unidirectional specimens. This resin rich region, not predicted by the laminate model, would also contribute for a lower measured modulus. This hypothesis would explain why the lower modulus was only observed in cross-ply laminates and not in the unidirectional specimens.

Table (3) also shows the difference between measured and predicted loss factors, which was more significant for the $[(+45/-45)_2]_s$ beam. This difference was also observed in a previous research (Adams and Maheri, 1994). This previous investigation, which used beams of various widths, has indicated that a better correlation between measured and calculated properties can be obtained from narrower beam specimens.

In general, the data presented in this study show good agreement between the predicted and measured viscoelastic properties for all three laminates studied. This agreement indicates that the DMA-based approach used in this investigation, for laminae and laminate viscoelastic characterization, is able to produce valid properties. Therefore, the technique using Dynamic Mechanical Analysis offers a great potential for studying viscoelastic behavior of laminae and laminates, producing critical information, which ultimately can be used to design and analysis of composite structures.

4. Conclusions

This study evaluated the feasibility of using Dynamic Mechanical Analysis to measure viscoelastic properties of laminates. Viscoelastic characterization of unidirectional fiber-reinforced laminae and also measurements in three different cross-ply laminates, were conducted. The laminate measurements were compared with predicted properties, using a published model from the literature, based on measured laminae properties. The results show good agreement between measured and predicted loss factors for the laminates. The measured storage modulus was, for all cross-ply laminated beams tested, about 10% smaller than the predicted values, suggesting a presence of a resin rich region in addition to possible delaminations or imperfect bonding between adjoining layers. In general, the results of laminate testing indicated that the DMA-based approach used can produce valid lamina and laminate properties, since laminate predicted properties, that were based on laminae measured properties, agreed properly with the measured laminate values.

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