Characterization of the dynamic behaviour of flax fibre reinforced composites using vibration measurements

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Abstract. Experimental and numerical methods to identify the linear viscoelastic properties of flax fibre reinforced epoxy (FFRE) composite are presented in this study. The method relies on the evolution of storage modulus and loss factor as observed through the frequency response. Free-free symmetrically guided beams were excited on the dynamic range of 10 Hz to 4 kHz with a swept sine excitation focused around their first modes. A fractional derivative Zener model has been identified to predict the complex moduli. A modified ply constitutive law has been then implemented in a classical laminates theory calculation (CLT) routine.

1. Introduction

Structural parts of vehicles are submitted to dynamic loading. Excitations coming from the powertrain, road surfaces and aerodynamic flows cause mechanical vibrations. In order to improve the acoustical and vibrational comfort, natural fibre reinforced polymer composites, exhibiting interesting dissipative properties, can be used. Indeed, compared to conventional composite materials, the damping performance of flax fibre reinforced polymer (FFRP) can be respectively twice or three times higher than that of glass or carbon fibre reinforced polymer composites [1,2].

Damping in undamaged composite materials is induced by several microscopic level mechanisms, such as viscoelastic elongation of the matrix and/or fibres, friction between both components at their interfaces. Moreover, in the particular case of natural fibre reinforced polymer composites, the friction between fibres inside bundles can also increase this phenomenon.

In order to assess the damping within materials, vibration techniques have the advantage of rapidly exploring a wide range of frequencies (10 Hz to 1 kHz). Thus, many authors have studied the frequency dependence of composite materials [3,4,5,6].

The frequency dependence is a typical feature of viscoelastic materials. This dependence induces variations of the complex moduli when the frequency of excitation changes. In order to assess these variations, it is necessary to understand the material constitutive equations that relate the stresses to the strains with respect to time or frequency [7], with the help of the linear viscoelasticity theory. These relations are expressed by linear differential equations or convolution integrals. They have the main benefit of being expressed both in frequency and time domains.

The present study aims at presenting a method dedicated to the identification of the evolution of complex moduli of FFRP laminates on a large frequency band with the help of a fractional derivative Zener model. This identification has been done thanks to experimental tests on a specific device, between 10 Hz and 4 kHz.
2. Linear viscoelastic model

Materials submitted to mechanical loading store energy by elastic deformation. For purely elastic materials, this energy is totally and immediately returned during the unloading phase. However, in the case of viscoelastic materials, the energy stored is completely returned but with some delay due to inner rearrangements. For such materials the stress depends on the strain history [7]. Several models describing the behaviour of a viscoelastic material in the structural dynamic domain are available in the literature. Among them, the fractional derivative Zener model (Fig. 1) has been used by many authors to describe the viscoelastic behaviour. This model is mathematically described in Eq. 1, where \(\sigma(t), \varepsilon(t), \tau, \alpha\) are respectively the stress and strain as a function of time, the relaxation time and the \(\alpha\)-order fractional derivative coefficient \((0 < \alpha < 1)\). \(D^\alpha[\bullet]\) is an \(\alpha\)-order fractional derivative operator. The mechanical stiffness \(E_0\) and \(E_\infty\) are respectively the dynamic modulus at very low frequency and at high frequency. The fractional derivative model is described in Fig. 1.

\[
\sigma(t) + \tau^\alpha D^\alpha[\sigma(t)] = E_0\varepsilon(t) + E_\infty \tau^\alpha D^\alpha[\varepsilon(t)]
\]  

(1)

In the case of a steady state harmonic excitation, the complex modulus \((E^*)\) is derived from Eq. 1, where the fractional derivative operator \(D^\alpha[\bullet]\) is replaced by a multiplication by \((j\omega)^\alpha\). \(j\) is the imaginary unit and \(\omega_n = \omega \tau\).

\[
E^*(\omega) = \frac{\sigma^*(\omega)}{\varepsilon^*(\omega)} = \frac{E_0 + E_\infty (j\omega_n)^\alpha}{1 + (j\omega_n)^\alpha}
\]  

(2)

Thus, it is possible to write Eq. 2 in the form of Eq. 3, in which \(\eta(\omega) = E''(\omega)/E'(\omega)\) represents the material’s loss factor. The real part \(E'(\omega)\) of \((E^*)\) represents the elastic behaviour of the tested material. It is the storage modulus while the imaginary part \(E''(\omega)\) or loss modulus represents the viscous or damping behavior of the material.

\[
E'(\omega) = E'(\omega) + jE''(\omega) = E'(\omega)[1 + j\eta(\omega)]
\]  

(3)

Using the constitutive equation (Eq. 1), \(E'(\omega)\) and \(\eta(\omega)\) can be expressed by Eqs. 4 and 5, where \(c = E_\infty/E_0\).

\[
E'(\omega) = E_0 \frac{1 + (c+1)\cos(\alpha\pi/2)\omega_n^\alpha + c\omega_n^2\alpha}{1 + 2\cos(\alpha\pi/2)\omega_n^\alpha + \omega_n^{2\alpha}}
\]  

(4)

\[
\eta(\omega) = \frac{(c-1)\sin(\alpha\pi/2)\omega_n^\alpha + \omega_n^{2\alpha}}{1 + (c+1)\cos(\alpha\pi/2)\omega_n^\alpha + c\omega_n^2\alpha}
\]  

(5)

3. Classical laminate theory applied to viscoelasticity

With the help of the individual properties of each ply, the CLT allows one to compute the elastic properties of a stack of unidirectional (UD) composite plies (i.e. longitudinal and transverse storage moduli, Poisson’s ratios and shear moduli). In order to establish a viscoelastic version of CLT, the so-called correspondence principle has been used. Initially introduced on homogeneous materials, this principle has been extended to heterogeneous and composite materials by Hashin [9]. In the case of a sinusoidal steady-state excitation, it is possible to measure and replace the elastic properties by the appropriate complex ones, i.e. the conversion of the elastic solutions to viscoelastic ones. Thus, the laminate’s relations derived from the CLT can be used to predict the overall viscoelastic moduli of the
laminate from the elementary ply properties. According to the elastic CLT method, the complex constitutive relation linking forces (\(N^*\)) and bending moments (\(M^*\)) to strains (\(\varepsilon^*\)) and membrane curvatures (\(\kappa^*\)) can be expressed by Eq. 6.

\[
\begin{bmatrix}
N^*(\omega) \\
M^*(\omega)
\end{bmatrix} = 
\begin{bmatrix}
A^*(\omega) & B^*(\omega) \\
B^*(\omega) & D^*(\omega)
\end{bmatrix}
\begin{bmatrix}
\varepsilon^*(\omega) \\
\kappa^*(\omega)
\end{bmatrix}
\]

(6)

The complex laminate stiffness matrices \([ABD]^*\) are given by Eqs. 7 to 9, based on the complex reduced stiffness matrix \([\bar{Q}^*_{ij}(\omega)]\), where \(h_n\) represents the position of the \(n^{th}\) ply in the thickness of the laminate and \(N\) is the total number of plies (Fig. 2).

\[
A_{ij}^* = A_{ij}' + jA_{ij}'' = \sum_{n=1}^{N}[\bar{Q}^*_{ij}]_n (h_n - h_{n-1})
\]

(7)

\[
B_{ij}^* = B_{ij}' + jB_{ij}'' = \frac{1}{2} \sum_{n=1}^{N}[\bar{Q}^*_{ij}]_n (h_n^2 - h_{n-1}^2)
\]

(8)

\[
D_{ij}^* = D_{ij}' + jD_{ij}'' = \frac{1}{3} \sum_{n=1}^{N}[\bar{Q}^*_{ij}]_n (h_n^3 - h_{n-1}^3)
\]

(9)

The \(n^{th}\) layer in which plies are oriented with an angle \(\alpha_n\) with respect to the loading direction, the coefficients \([\bar{Q}^*_{ij}]_n\) are obtained from \([Q^*_{11}(\omega)]_n\) using Eqs. 10 to 15, where \(p_n = \cos \alpha_n\) and \(q_n = \sin \alpha_n\). The \([Q^*_{11}]_n\) are the complex reduced stiffness.

\[
[\bar{Q}^*_{11}]_n = [Q^*_{11}]_n p_n^4 + [Q^*_{22}]_n q_n^4 + 2([Q^*_{12}]_n + 2[Q^*_{66}]_n)p_n^2q_n^2
\]

(10)

\[
[\bar{Q}^*_{12}]_n = ([Q^*_{11}]_n + [Q^*_{22}]_n - 4[Q^*_{66}]_n)p_n^2q_n^2 + [Q^*_{12}]_n(p_n^4 + q_n^4)
\]

(11)

\[
[\bar{Q}^*_{16}]_n = ([Q^*_{11}]_n - [Q^*_{12}]_n - 2[Q^*_{66}]_n)p_n^2q_n^2 + ([Q^*_{12}]_n - [Q^*_{22}]_n - 2[Q^*_{66}]_n)p_nq_n^3
\]

(12)

\[
[\bar{Q}^*_{22}]_n = [Q^*_{11}]_n p_n^4 + [Q^*_{22}]_n p_n^4 + 2([Q^*_{12}]_n + 2[Q^*_{66}]_n)p_n^2q_n^2
\]

(13)

\[
[\bar{Q}^*_{26}]_n = ([Q^*_{11}]_n - [Q^*_{12}]_n - 2[Q^*_{66}]_n)p_nq_n^3 + ([Q^*_{12}]_n - [Q^*_{22}]_n - 2[Q^*_{66}]_n)p_nq_n^3
\]

(14)

\[
[\bar{Q}^*_{66}]_n = ([Q^*_{11}]_n + [Q^*_{22}]_n - 2([Q^*_{12}]_n + [Q^*_{66}]_n))p_n^2q_n^2 + [Q^*_{66}]_n q_n^4 + q_n^4
\]

(15)

For each of the \(n\) layers, the \(Q^*_{ij}\) are determined in the principal directions of the UD layer based on the engineer’s moduli (Eqs. 16 to 19). \(E'_{L}\) and \(E'_{T}\) represent, respectively, the storage moduli in the fibres and the transverse directions of the elementary ply. \(\eta_L\) and \(\eta_T\) are longitudinal and transverse loss factors. \(G'_{LT}\) is the ply shear modulus. \(v'_{LT}\) and \(v'_{TL}\) are its Poisson’s ratios.

\[
Q^*_{11}(\omega) = \frac{E^*_L(\omega)}{1 - v_TL^2v_{LT}} = \frac{E^*_L(\omega)[1 + j\eta_L(\omega)]}{1 - v_TL^2v_{LT}}
\]

(16)

\[
Q^*_{22}(\omega) = \frac{E^*_T(\omega)}{1 - v_TL^2v_{LT}} = \frac{E^*_T(\omega)[1 + j\eta_T(\omega)]}{1 - v_TL^2v_{LT}}
\]

(17)
Thus, the effective bending stiffness and the loss factor of a composite cantilever beam are given by relations 20 and 21.

\[
E^*(\omega) = \frac{12}{h^3D_{11}^{-1}(\omega)} = E' + jE''
\]

\[
\eta(\omega) = \frac{E''(\omega)}{E'(\omega)}
\]

4. Experimental method

Materials and specimens

The FFRE specimens used were made of the Hermès variety of flax cultivated in northern France. A commercial dry roll of unidirectional (UD) flax fabric was supplied by BioRenforts, a local textile company. The fabric consisted of aligned flax UD fibres stitched by a cotton thread, without any treatment. The matrix was an epoxy system based on the resin SR 8200 with the SR 8205 hardener provided by SICOMIN. Fibre layers were first cut from the roll and manually impregnated with the liquid matrix. The plies were hand-laid, before being stacked under a pressure of 7 bars in a hydraulic press equipped with heating plates.

Slender composite specimens made of four layers, i.e. [0]4, [90]4, [±45]s and [±60]s with a thickness (t) of 1 mm, a width (b) of 10 mm and a free length (L) of 170 mm, were cut out from laminated plates with a high speed rotating grinding disk.
Figure 3. Schematic diagram of the specimen and test set-up.

The test apparatus is shown on Fig. 3. A PCB® accelerometer was fixed to the end of the shaft of the shaker, at point $M_1$, in order to measure the acceleration ($a(M_1)$) imposed to the sample. Then, the vibration velocity ($v(M_2)$) of the free end of the beam, at point $M_2$, was measured by a vibrometre sensor head Polytec-OFV-503 coupled to a controller unit Polytec-OFV-500, via a mirror inclined at 45° above $M_2$ allowing the laser signal to enter in the chamber through a small transparent window. A real-time signal analyser Pulse LabShop-B&K® gave the frequency response function ($H(\omega)$) between $v(M_2)$ and $a(M_1)$ (Eq. 22).

The storage modulus ($E'(\omega_i)$) is computed from the beam theory (Eq. 23). The loss factor ($\eta(\omega_i)$) has been calculated by the “-3 dB bandwidth” method, in the vicinity of $\omega_i^2$.

\[
\omega = \frac{v(M_2)}{a(M_1)}
\]

\[
E' = \frac{12 \rho L^4}{X_h^2 h^2} \omega_i^2
\]

5. Results and discussion

5.1. Material properties identification

The linear viscoelastic Zener’s parameters ($\tau$, $\alpha$, $E_0$ and $E_\infty$) have been identified for $[0]_4$ and $[90]_4$ using Eqs. 4 and 5 in an iterative procedure minimizing the error between the experimental data and the theoretical values. The results of the identification are given in Table 1.

| Laminate | $E_0$ (GPa) | $E_\infty$ (GPa) | $\tau$ (s) | $\alpha$ |
|----------|-------------|-----------------|-------------|---------|
| [0]_4    | 19.2        | 20.7            | $1.59 \times 10^{-5}$ | 0.422   |
| [90]_4   | 5.03        | 5.63            | $1.59 \times 10^{-5}$ | 0.422   |

Experimental data with standard deviations and Zener model curves for $[0]_4$ and $[90]_4$ laminates are plotted on Figure 4. The analytical calculation of the moduli and loss factors show good agreement with the test measurements, despite the inherent scattering of the properties of fibres. The measured moduli increased with the frequency of 10 and 17% on average, respectively for $[0]_4$ and $[90]_4$ laminates in the bandwidth (Figure 4a). The loss factors exhibited important increase with a non-linear trend of 22 and 20% on average, respectively for $[0]_4$ and $[90]_4$ samples (Figure 4b). Conversely, $\eta$ was dependent on
frequency. Despite the inherent scattering of plant fibres, the analytical predictions show good agreement with the test measurements. Thus, the Zener model was assumed to be relevant.

5.2. Validation method

After the identification of the parameters of the Zener model \((E_L, E_T, \eta_L, \eta_T)\) on \([0]_4\) and \([90]_4\) specimens presented above, the identification of the storage modulus and loss factor have been extended to additional \([\pm 60]_1\), \([\pm 45]_1\), laminates. The coefficients \([\hat{Q}_{06}]\) were computed by the CLT, and used to determine the \([ABD]^*\) matrices. Inversion of these matrices gave the desired complex modulus. \(v_{LT}\) was supposed to be frequency-independent and was measured at 0.43 by quasi-static tensile testing. \(v_{TL}\) was determined using the conventional relationship \(v_{TL} = v_{LT} \times E_T / E_L\). The frequency dependence of the shear modulus \(G_{LT}\) was similar to that of the complex modules insofar as it uses the fractional derivative Zener model. The model coefficients that minimize the difference between the theoretical values and the experimental data were estimated at \(G_{LT,0} = 0.15\) GPa and \(G_{LT,\infty} = 1.5\) GPa.

The comparison between the theoretical values and the experimental data for \([\pm 60]_1\), and \([\pm 45]_1\), laminates appears on Fig. 4.

As for \([0]_4\), and \([90]_4\), laminates, storage modulus for \([\pm 45]_1\), and \([\pm 60]_1\), laminates seems to depend slightly on the frequency (a). The difference between theoretical values and experimental data is relatively small for the \([\pm 60]_1\), laminate, but it is around 12% for \([\pm 45]_1\), at kHz. When the frequency increases, this difference tends to reduce to less than the experimental standard deviation.

Loss factor curves follow a non-linear law, with a stabilization beyond 1 kHz (b). Once again, the difference between the theoretical curves and experimental values is lower for \([\pm 60]_1\), but with increasing frequency, it remains within the standard deviation for \([\pm 45]_1\), laminate.

![Figure 4](image)

Figure 4. Experimental and analytic frequency dependence of \(E_L\) (a) and \(\eta\) (b) for \([0]_4\) (---: model, •: experimental data), \([90]_4\) (---: model, *: experimental data), \([\pm 45]_1\) (----:Model, ♦: experimental data) and \([\pm 60]_1\) (---: model, ▼: experimental data)
6. Results and discussion

An experimental vibration technique for fast identification of the dissipative properties of flax fibres reinforced epoxy composites has been presented. The characterization method allowed the measurement of Young’s modulus and loss factor of beams in a large frequency band (10 Hz to 4 kHz). Parameters of the fractional derivative Zener models identified in fibre and in-plane transverse directions of UD composites have been used to predict the frequency evolution of both moduli and loss factors. Based on the elastic-viscoelastic correspondence principle, CLT has been successfully used to predict the linear viscoelastic behaviour of different composite laminates. This prediction method was validated by comparison with the experimental results obtained for [0], [±45], [±60], and [90]_4 laminates.

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