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# Measurements and predictions of the viscoelastic properties of a composite lamina and their sensitivity to temperature and frequency

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## ABSTRACT

We perform finite element analysis of the mechanical response of random RVEs representing the microstructure of a unidirectional (UD) fibre composite, predicting its anisotropic stiffness and damping properties and their sensitivity to temperature and frequency, using as inputs only the measured response of the constituents. The simulations are validated by DMTA measurements on a UD composite; then, the numerical predictions are compared to those of previously published theoretical models. New equations are proposed to predict the viscoelastic constants, providing better accuracy than existing models. The accuracy of these new equations is tested, over wide ranges of fibre volume fractions and stiffness ratios of the constituents, against the numerical predictions.

Keywords: Microstructure, Vibrations, Viscoelastic, Composite, Finite Elements.

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## 1. INTRODUCTION

Fibre composites are being adopted in automotive and aerospace constructions due to high specific stiffness and strength, superior to that of metallic alloys. Composites with a viscoelastic polymer matrix also exhibit relatively high damping, compared to metals of similar stiffness, which makes them suitable for all components subject to vibrations. There has been growing research to measure and predict such damping properties, to allow the design of optimised components. Damping of laminates can be increased by incorporation of viscoelastic damping layers in the laminate [1–7], however such layers reduce the stiffness, thereby offsetting weight saving. Composite laminae with higher intrinsic damping are therefore needed and it is therefore essential to be able to predict the viscoelastic response of a UD composite over a range of operating conditions, including different temperature and pressure. Predictions should be performed using as inputs only the measured properties of the constituent materials, to allow an assessment of the composite's response prior to manufacturing.

Damping properties of fibre composites can be tailored by controlling different parameters at both microscopic level (constituent materials, fibre volume fraction, fibre aspect ratio) and at laminate level (stacking sequence); we refer the reader to recently published reviews on such aspects of the design [8,9]. The majority of the research to date has focused on laminate level analyses [10] and specifically on the optimisation of composite layups to maximize damping and stiffness, using as an input the properties of individual UD plies and making use of the elastic-viscoelastic correspondence principle [11,12]; Adams and co-workers [13,14] also carried out extensive measurements on the damping response of UD fibre composites and laminates.

Several studies have analysed the effect of the properties of the constituent materials and of fibre volume fraction upon the stiffness and damping properties of a single UD ply, taking analytical [15,16], numerical [17–20] or experimental [1,2] approaches to the problem; this is also the purpose of the present study. Willway and White [21] and Akay [22] found that existing theoretical models under-predict the measured flexural damping and this was attributed to a relatively low length to thickness ratio, inducing shear deformation in the specimen, as well as to frictional losses at the fibre-matrix interface. Bogren et al. [23] found that theoretical models predict elastic properties with a margin of error of order 10% but underpredict the damping properties by more than 60%; again this was attributed to frictional losses due to poor adhesion between fibres and matrix. Similar observations were made by Shokrieh et al. [24] for the case of glass-epoxy composites. Chandra et al. [25] performed a comparative study between FEM and existing micromechanical models to assess their effectiveness, however these results were not validated with experiments. Tsai and Chi [26] and Pathan

et al [27] assessed the effect of different periodic arrangement of fibres on damping and found the loss factors to be strongly dependent on the spatial distribution of the fibres.

The main drawback of existing research is in the fact that the mechanical properties of the constituent materials used in predictions are taken from the work of others or from the manufacturer's datasheets. In addition, only a few existing studies are measuring or predicting the full transversely isotropic viscoelastic tensor, but most are limited to selected loading cases. Much of the published research investigated either elastic properties or damping properties; comparison of predictions to experiments is very often conducted on the response of laminates rather than of single plies, making validation of the predictions difficult. Existing experimental studies come to contrasting conclusions and measurements of damping of similar materials may differ by up to an order of magnitude. The study on the dependence of damping on temperature and frequency has been limited[28], despite knowledge of this dependence is needed by design engineers, who also lack simple equations to calculate accurately the viscoelastic ply properties.

This study addresses all the limitations outlined above. We manufacture, in a controlled laboratory environment, thin UD composite plates made from carbon fibres impregnated with an epoxy resin. We measured the viscoelastic response of the constituents and that of the UD plates along different directions and at different temperatures and frequencies. We then construct random RVEs of the ply microstructure employing a previously developed algorithm [27,29,30] which guarantees effectively random arrangements of fibres and perform Monte Carlo analyses in the commercial FE solver ABAQUS/standard. The numerical predictions are validated by the measurements and are compared to a number of existing theoretical models, in order to rank their effectiveness. Finally, we propose new equations to make accurate predictions of the viscoelastic properties of UD composites.

The outline of the paper is as follows: in Section 2 we discuss the manufacturing of the composite and the testing methods. Section 3 presents the details of the numerical models, while results are discussed in Section 4.

## **2. EXPERIMENTAL METHODS**

### **2.1 Material manufacturing**

To manufacture a neat resin panel with minimal air bubbles we used the vertical sandwich panel method as described by Figliolini [31]. A mould cavity was created using  $250 \times 250 \times 10$  mm heat treated glass plates, which were sealed on three sides using a rubber-o ring and toolmakers clamps. The inner surfaces of the glass plates were sprayed with multiple coats of a release agent (FreeKote 700NC) to allow for easy demoulding. The resin used was Prime20LV with the slow hardener, due to

its low viscosity and longer setting time, to allow manufacturing of void-free thin UD composite plates. The resin/hardener was mixed in the suggested ratio and degassed for 15 min at 30 °C in a vacuum oven. The mixture was then poured in the mould cavity and was allowed to stand for 10 min to allow most air bubbles to travel to the top of the plate. The entire assembly was then transferred to the oven to cure at 50 °C for 16 hr, according to the manufacturer's instructions. Beams of size  $50 \times 6 \times 2$  mm were manufactured using a water jet cutter and conventional milling; the specimens were stored in a plastic bag with a desiccant to avoid moisture absorption. The glass transition temperature (corresponding to a peak of loss factor) was found to be 81 °C via a DMTA temperature sweep test (soak time of 5 mins, temperature ramp rate of 1 °C/min); this value is in good agreement with the manufacturer's value of 82.6 °C.

Elastic and damping properties of the T700 carbon fibres employed in this study were measured in single fibre tests, following ASTM C1557-14 [32]. Mounting tabs were made from a 200gsm paper sheet via automatic cutting. Single fibres were randomly pulled from a bundle using rubber-tipped tweezers; they were aligned and mounted on the tabs using a thin layer of a two-part epoxy adhesive. The adhesive was allowed to cure for three days, following which the specimens were analysed under an optical microscope to measure the diameter. The specimens were tested using a flat-faced tensile grips in a DMTA; the gauge length was limited to 30mm due to the limitations of the apparatus. We note that the carbon fibres were coated with an epoxy sizing to improve handling characteristics of the fibres, however the amount of sizing was  $< 1\%$  in weight and hence its effect on the overall composite property is expected to be negligible.

A UD fibre composite plate measuring  $300 \times 300$  mm was manufactured using three layers of T700 fibre fabric (Sigmatex UK, 150 gsm, 12K) and Prime20LV/Slow using the resin infusion under flexible tooling method. The panel was cured at atmospheric pressure for 16 hrs at 50 °C, i.e. in identical conditions as for the neat resin. In order to determine the fibre volume fraction, void fraction and nominal thickness, the cured plate was analysed in an optical microscope; the specimens were ground and polished to a surface roughness of  $3\mu\text{m}$  and mounted on a Zeiss optical microscope system. High resolution images of regions of size approximately  $550 \times 450 \mu\text{m}$  were taken, as in Fig. 1a. These were then analysed using ImageJ. The measured fibre volume fraction was 63%, the average fibre diameter was  $6.77 \mu\text{m}$ , with a standard deviation of  $0.335 \mu\text{m}$ . Beams of size  $50 \times 6$  mm were extracted from the plate such that the longitudinal axis of the beam formed angles of  $0^\circ$ ,  $90^\circ$  and  $45^\circ$  with the carbon fibre direction.

The micrograph in Fig. 1a shows a resin region at the top of the composite plate; this represents a section of the resin-rich pattern left on the plate by the resin infusion mat used in the manufacturing.

This influenced the thickness of the specimen, which was irregular and varied periodically in the plane of the plate, measured at an average of 0.32 mm using several micrographs. The resin rich layer on average consisted of around 8% of the total specimen thickness.

## 2.2 Mechanical testing

Measurements of the stiffness and damping properties of the constituents and the UD composite was performed by the forced vibration (non-resonant) technique, making use of a DMTA analyser with load capacity of 35 N (RSA-G2 by TA instruments). Isothermal frequency sweeps in the range 0.1-10 Hz were conducted at three different temperatures (25, 40 and 50°C). The maximum temperature was chosen to ensure a linear viscoelastic response of the matrix material, in line with the constitutive modelling strategy adopted in this paper; as the glass transition temperature for the resin used was of 81°C, the test temperature was therefore limited to 50°C. In these tests, after applying a small pre-load to the sample, a sinusoidal displacement history of constant amplitude and varying frequency is imposed to the specimens, and the amplitude and phase of the resulting sinusoidal force are measured. Such measurements allow calculation of the storage and loss moduli of the material, which quantify stiffness and viscoelastic dissipation, respectively. The low capacity of the DMTA results in relatively small specimen size and, in turn, in the need for very precise manufacturing and positioning of the specimens.

Tests on the single carbon fibres were conducted in the axial mode, subjecting the fibre to an axial tensile force; measurements on the neat resin and on the composite beams were performed in three-point bending mode, due to the limited capacity of the DMTA. Preliminary experiments showed that clamped bending fixtures and simple support by knives provided similar measurements of elastic moduli, however clamped bending fixtures provided more repeatable damping measurements, especially for the case of the thin composite laminae tested. For simply supported composite laminae, any small non-planarity of the specimen or edge defects, as well the coupling between in-plane normal and shear strains (for off-axis composite specimens), resulted in rubbing of the specimen against the supporting rollers and in a higher and less repeatable measured damping. For this reason clamped bending fixtures were preferred in this study. The clamped boundary condition induces stretching along the axis of the beam which scales, in first approximation, as  $2(\delta / L)^2$ , where  $\delta, L$  are the deflection and length of the specimen; calculating the bending strain field via beam theory, the ratio of the strain energies associated to axial stretching and beam bending is  $(\delta / H)^2 / 4$ , where H is the beam thickness. For the small peak deflections in our tests, this ratio is smaller than 0.1 for the thin composite beams (and negligible for the thicker neat resin beams); for this reason axial strains are

neglected in the analysis. A correction for the compliance of all loading fixtures was automatically performed by the DMTA, making use of factory calibration curves.

Tests were performed using the available fixture of maximum span (32mm). This gave a length-to-thickness aspect ratio for the neat resin specimen of  $l/t= 16$ , in accordance with the ASTM D5418 [33] testing standard for homogenous materials. For the case of fibre composite specimens, the corresponding aspect ratio was 50, which is in line with the recommendations of ASTM D790 [34] for flexural testing of fibre composites.

### 2.2.1 Response of the neat resin

Epoxy beams were first subject to a strain amplitude sweep, to identify the limiting strain amplitude to give inelastic deformation. This was done by imposing sinusoidal deflection histories of increasing amplitudes on the beams at 1 Hz frequency and until loss of the linear correlation between modulus and strain. Subsequently, frequency sweeps were conducted in the range 0.1-10 Hz, with a pre-load of 1 N and a strain amplitude of 0.04%, chosen to give a maximum deflection well below the linearity limit.

Five samples were tested at 3 selected temperatures and the response was repeatable within a 10% margin; Fig. 2 shows averages of the storage modulus and the loss factor of the matrix measured in the DMTA experiments. Note that the loss factor is defined as the ratio between the imaginary and real parts of the complex modulus. The storage modulus and loss factor are sensitive to frequency and temperature; the modulus increases monotonically with the applied frequency (i.e., strain rate); the loss factor decreased with frequency, as expected for viscoelastic materials in glassy region. An increase in temperature results, as expected, in a decrease of storage modulus and an increase in the loss factor in a non-linear manner. Experimental results of the elastic storage modulus and loss factor for neat resin are given in Table 1 for 1 Hz loading frequency at different temperatures.

*Table 1: Stiffness and damping properties of neat resin obtained from DMTA testing at different temperatures at 1Hz loading frequency*

	25°C	40°C	50°C
E (GPa)	2.70	2.42	2.22
$\eta_E$ %	1.55	1.64	2.18

### 2.2.2 Response of the single carbon fibres

Both the axial modulus and the axial damping of the T700 carbon fibres were measured, to inform numerical models. The longitudinal damping of UD fibre composites is substantially influenced by the damping of the fibres, which is often neglected in the literature on the ground that loss factors for fibres are typically one order of magnitude lower than those of the matrix. On the other hand the fibres occupy the majority of the composite volume and store a very large fraction of the total strain energy in the composite. The test method used is unable to measure the radial properties of the fibres; in the numerical simulations presented below we shall assume that fibre damping is isotropic, while the radial modulus is taken as  $\approx 10\%$  of the measured axial modulus, following [35–38].

Frequency sweeps were performed in the DMTA in axial mode and at different temperatures, following a preliminary strain amplitude sweep test. The imposed pre-load was 0.08 N, while the imposed strain amplitude was 0.1%. Tests were repeated 15 times on different specimens; averages of the measurements at 25°C are shown in Fig. 3, where the error bars indicate the measured ranges. The mean measured storage modulus of 225 GPa is in good agreement with the value reported by the manufacturer (230 GPa), as well as with measurements from other authors [39]; the scatter in modulus was of order 7%. The loss factor of the fibres had mean 0.0033 but showed a greater variation around this value, of order 100% as indicated by the error bars in Fig. 3b.

It is clear from Fig. 3(a) that the modulus of carbon fibre is scarcely sensitive to the imposed frequency in the range investigated. In view of the large scatter observed in the measured loss factor of carbon fibres in Fig. 3(b), no definitive trends could be established and hence it was assumed that the damping of carbon fibre was insensitive to frequency. Preliminary experiments conducted at 50°C showed no difference with measurements at 25°C; this is expected for this material, and in line with results in the literature: Saunder et al. [40] found a decrease of less than 5% in modulus at 1000°C respect to room temperature, for various types of carbon fibres; Feih and Mouritz [39] found similar results up to 500°C for the T700 fibre considered in this study. Given the narrow temperature range investigated here, the viscoelastic properties of the carbon fibres will be considered insensitive to temperature in the numerical simulations.

### 2.2.3 Anisotropic response of the UD composite

The UD composite was tested along three different directions, such that fibres were oriented longitudinally, transversely or at 45° with respect to the axis of the beam. Following a strain amplitude sweep, frequency sweeps were conducted in the range 0.1-10 Hz, with a strain amplitude of 0.02%. Again, testing was performed at temperatures of 25, 40 and 50°C.

Experiments were repeated 5 times and beam theory was used to extract the axial storage modulus and loss factor of the material at each orientation. The scatter in these measurements was less than 15% for the stiffness and less than 50% for the damping properties. Averages of the measurements are presented in Figs. 4-9; Figures 4-6 compare the measurements to existing analytical models and numerical predictions described below; Figures 7-9 show measurements and numerical predictions of the response at different temperatures. As expected it was found that, for all orientations, storage moduli increase and loss factors decrease with increasing frequency, as it is visible in Figs. 4-6. Conversely, an increase in the test temperature resulted in a decrease of storage moduli and an increase in loss factors (see Figs. 7-9).

### 2.2.5 Data reduction for axial shear modulus

The in-plane complex shear modulus  $G_{12}^*$  is calculated from the measured complex bending stiffness  $E_{xx}^*$  of the off-axis ( $45^\circ$ ) specimen, using the rule of material property transformation [41]

$$\frac{1}{G_{12}^*} = \frac{\left[ \frac{1}{E_{xx}^*} - \frac{\cos^4 \theta}{E_{11}^*} - \frac{\sin^4 \theta}{E_{22}^*} \right]}{\sin^2 \theta \cos^2 \theta} + \frac{2\nu_{12}}{E_{11}^*} \quad (1)$$

where,

$\nu_{12}$  is the composite Poisson's ratio (calculated using rule of mixtures)

$E_{11}^*$  Axial complex Young's modulus

$E_{22}^*$  Transverse complex Young's modulus.

## 3. NUMERICAL ANALYSIS

### 3.1 Generation of the random RVEs

Virtual microstructures of UD composited were generated using an algorithm based on optimization techniques and previously proposed by the authors [29]. In brief, this algorithm allows randomly placing a number of fibres, of arbitrary shape, in a stochastic volume element (SVE) of square cross-section in the isotropic plane. Geometric analysis of the SVEs revealed that the microstructures generated are effectively random when their size  $L$  is greater than 8 times the fibre radius  $R$ , i.e.  $\delta = L / R \geq 8$ . Mechanical analysis [27] revealed that in order to obtain predictions of the viscoelastic properties insensitive to SVE size, and associated to an intrinsic of scatter less than 5%, it is necessary

to enforce  $\delta = L / R \geq 24$ . In other words, the minimum size of an RVE [19] is equal to 24 times the fibre radius for the case of circular fibres. For this reason in this study we analyse random RVEs of size  $\delta = 24$ . These RVEs have thickness  $t = 4R$  along the fibre direction, as it was previously shown in [42] that predictions are insensitive to  $t$  for  $t > 4R$ . The radius of the carbon fibres was taken to as  $6.77 \mu\text{m}$ , as suggested by image analysis of optical micrographs. Example of an analysed RVE of  $\phi_f = 0.63$  and  $\delta = 24$  is as given in Fig. 1(b). A total of 10 repeated simulations were performed, in each case, on different realizations of the microstructure.

### 3.2 Details of the FE simulations

Analyses of the response in the frequency domain were conducted with the commercial FE software Abaqus/standard [43], performing periodic steady-state analysis (steady-state direct in the jargon of Abaqus). The RVEs were subject to three different harmonic loading cases, i.e. uniaxial stress along the fibres and in the transverse direction, as well as axial shear. The macroscopic strains imposed on the RVEs were pure sine waves of amplitude arbitrarily set to 0.01 and varying frequencies; the analysis allowed calculation of the corresponding macroscopic stress histories; such histories were interpreted as phasors and split into two components, in-phase and out-of-phase with respect to the imposed strain. The ratio of the in-phase stress amplitude to the corresponding strain amplitude provided the values of the storage moduli; similarly, the ratio of the out-of-phase stress amplitude to the strain amplitude gave the imaginary (or loss) modulus.

The microstructures were meshed using a combination of hexahedral and tetrahedral finite elements with linear shape functions (C3D8 and C3D6). A mesh sensitivity study was performed to determine the optimal element size in each loading case, to guarantee mesh-insensitive predictions. It is widely accepted that periodic boundary conditions (PBC) are the most appropriate boundary conditions to analyse a geometrically periodic RVEs [44,45] and PBCs are imposed on the domains analysed here, following, e.g., [46] or [42]. Loading was applied by imposing nodal displacements on appropriate dummy nodes via the method of macroscopic degrees of freedom, as introduced by Michel et al. [47] and used by Tucker and Liang [48]. A steady-state dynamic analysis in the frequency domain was performed and allowed calculation of the corresponding stress histories, from which real and imaginary components of the stiffness matrix were extracted from resultant forces at the dummy nodes.

For a linear viscoelastic solid, the shear and volumetric response are independent and characterized by the complex shear and bulk modulus,  $G^*$  and  $K^*$ , or equivalently by the shear storage modulus

$G'$ , the corresponding shear loss factor  $\eta_G$ , the bulk storage modulus  $K'$  and the volumetric loss factor  $n_K$ , where the following identities hold

$$G^* = G' (1 + i\eta_G); \quad K^* = K' (1 + i\eta_K). \quad (2)$$

Due to lack of availability of independent characterization of matrix in shear mode of deformation, the matrix was modelled as isotropic viscoelastic solid and the damping response was assumed to be same in both dilation and shear i.e. ( $\eta_K^m = \eta_G^m$ ). However, the true viscoelastic behaviour of the resin is expected to lie somewhere in between the two extreme assumptions of (i) same damping in dilation and shear and (ii) viscoelastic only in shear and elastic in dilation ( $\eta_G^m \gg \eta_K^m$ ). In order to study the effect of this assumption on the homogenized macroscopic properties, we performed two additional simulations on same microstructure, each modelled with the above mentioned assumption. We find that the homogenized elastic properties as well as the axial loss factor ( $\eta_{11}$ ) are insensitive to this assumption. However, we obtain on average 20% higher prediction of the axial shear loss factor ( $\eta_{12}$ ) and 8% lower prediction of the transverse loss factor ( $\eta_{22}$ ) of the composite for the assumption of ( $\eta_K^m = \eta_G^m$ ) as compared to ( $\eta_G^m \gg \eta_K^m$ ). Hence, it is expected that compared to the true material property of resin, current modelling technique will lead to higher (lower) predictions for  $\eta_{12}$  ( $\eta_{22}$ ).

The frequency and temperature dependence of the matrix was explicitly modelled by providing frequency- and temperature-dependent modulus and loss factors, according to the measurements in Fig. 2. Three sets of simulations were performed imposing a uniform temperature field of 25, 45 and 50 °C, as in the experiments. The Poisson's ratio of the polymeric matrix was taken as  $\nu_m = 0.38$  based on literature [49,50] and was assumed to be independent of temperature and frequency for the given testing range. To determine the sensitivity of the numerical macroscopic predictions on the assumption of constant matrix Poisson's ratio, we performed two simulations on the same microstructure considering two 'extreme' values of matrix Poisson's ratio of 0.32 and 0.45, at 25 °C. We found that the predictions of homogenized elastic moduli (loss factors) were higher (lower) for higher matrix Poisson's ratio, with 18% and 7.8% higher predictions for the case of transverse Young's moduli and axial shear moduli, respectively and 10.7% and 1% lower predictions of the transverse and in-plane shear loss factors, respectively. However, in the current study we only examine narrow ranges of temperature and frequency, in which the matrix Poisson's ratio is only expected to only increase by 5% in the frequency range of 0.1-10 Hz [51,52]. With regards to temperature dependence, appreciable

variations in the matrix Poisson's ratio are only seen in the vicinity of glass transition temperature [53], which in current study is 82 C, while maximum testing temperature is 50 C. Hence, it is concluded that the assumption of constant matrix Poisson's ratio scarcely affects numerical predictions in this study.

In this study we take into account the damping of fibres by modelling them as transverse isotropic elastic solid with isotropic damping (i.e.  $\eta_{11} = \eta_{22} = \eta_{12} = \eta_{23}$  in the usual notation). The measured axial fibre modulus is used to calibrate the constitutive model; due to difficulties in measuring the transverse Young's modulus of carbon fibre,  $E'_{22f}$  was taken as 25 GPa i.e. ( $\approx 10\% E'_{11f}$ ) with  $G'_{23f} = 9.62$  GPa and  $\nu_{12f} = 0.2$ . In absence of consensus on the axial shear modulus of carbon fibre in literature, we used a value of  $G'_{12f} = 40$  GPa based on inverse modelling to fit elastic predictions of the numerical model to the experiments. The above mentioned fibre elastic properties are assumed to be independent of temperature and frequency. As there is no support for anisotropic viscoelastic constitutive response in Abaqus, we developed a UMAT (user material interface) to implement linear viscoelasticity in the frequency domain. The UMAT was validated by performing uniaxial stress tests (along different directions) on a single C3D8 finite element. We omit here the details of the implementation of this subroutine for the sake of brevity.

#### 4. RESULTS AND DISCUSSION

In this section we compare the DMTA measurements to the numerical predictions and to a number of established theoretical models. Several theoretical approaches have been proposed to date to predict the viscoelastic properties of fibre composites. Here we choose to assess the following models: (i) a direct rule of mixture (ROM), or Voigt bound [54], based on the assumption of equal strains in the fibres and in the matrix; (ii) an inverse rule of mixtures (IROM), or Reuss bound [55], derived upon assuming equal stress in fibres and matrix; (iii) upper and lower bounds developed by Hashin [56] and Hill[57] (denoted as (Hashin+, Hashin- in the following, respectively); (iv) a model developed by Mori and Tanaka[58] (denoted as Mori-Tanaka) based on the Eshelby's result for dilute strain-concentration tensor [59] and the assumption that the far-field strain equals the average strain in the matrix, and (v) a model developed by Lielens (Lielens, [60]).

We begin by considering measurements and predictions of the response to frequency sweeps at a temperature of 25°C; then, we examine separately the effects of an increasing temperature on the material response. We stress here that in the following we provide ensemble averages of the FE predictions; the scatter in these predictions did not exceed 5%, as discussed in Section 3.1.

#### 4.1 Anisotropic viscoelastic response of UD composite ply at 25°C

Measurements and numerical predictions of the material response at 25°C and frequency in the range 0.1-10 Hz are presented in Figs. 4-6. The figures include theoretical predictions from the models detailed in Section 4. Figure 4 refers to loading in the fibre direction (or direction 1); the axial storage modulus (fig. 4a) is scarcely dependent upon frequency and temperature in the ranges explored, as expected, and in good agreement with the FE simulations and all theoretical predictions shown in the figure. The corresponding measured axial loss factor (Fig. 4b) shows significant scatter and appears scarcely sensitive to frequency; the simulations agree with all the theoretical models shown, but appear to underestimate the loss factor with an error of around 15-20%.

We anticipate that for all load cases the measured loss factors of the UD composite will be slightly higher than the corresponding numerical predictions. We recall that, in the real tests, other dissipative mechanisms exist, in addition to the internal viscosity of the material, and these cause an increase of the apparent measured material damping; such mechanisms are the interaction of the specimen with the surrounding air [61] and the loading fixtures. The order of magnitude of the energy dissipated by aerodynamic forces can be calculated by simple considerations and it can be shown to be negligible, compared to the material dissipation. On the other hand friction between the loading fixture of the DMTA and the material specimens can be considerable, in consideration of the very small volume of the composite specimens, but is difficult to quantify. The discrepancy of 15-20% between predicted and measured damping is small compared to previously published studies. We conclude that our simulations predict correctly the axial loss factor.

The results for transverse loading are presented in Fig. 5. The measured storage modulus in direction 2 is in excellent agreement with the numerical predictions (Fig. 5a). In contrast, none of the theoretical models provides effective predictions of the transverse modulus; experimental results lie roughly mid-way between the predictions of the Lielens and Mori-Tanaka models. The measurements of transverse loss factor show significant scatter and are scarcely sensitive to the imposed frequency. The corresponding numerical predictions are in good agreement with the measurements (again, the FE simulations slightly underestimate the measurements) while again the theoretical models fail to predict accurately this material property; the Mori-Tanaka model provides the most accurate predictions.

For the case of the axial shear modulus, shown in Fig. 6a, we find that the experiments are again in broad agreement with the FE predictions, while the theoretical models fail to estimate the transverse storage modulus; both measurements and FE predictions show clear frequency dependency, with the modulus increasing with frequency. The measured sensitivity to frequency is higher than that predicted by the FE simulations; this may be a consequence of the fact that epoxy exhibits higher strain rate sensitivity in shear as compared to tension [62], while the input data used to calibrate the constitutive

model for the matrix was obtained from bending tests. Figure 6b presents predictions and measurements of the axial shear loss factor; the experiments are in good agreement with the FE predictions. The Reuss bound, the Hashin's lower bound and the Mori-Tanaka model provide reasonably good results, while Lielens model underestimates the loss factors. The measurements and the FE predictions show a decreasing shear loss factor with increasing frequency.

#### **4.2 Effect of temperature on the anisotropic viscoelastic response.**

We proceed to examine the sensitivity of the measurements and FE predictions to temperature, by comparing the average measurements to the ensemble averages of the FE predictions in Figs. 7-9; the predictions of theoretical models are not included in the figures for the sake of clarity.

For the case of axial loading we find that FE predictions and measurements of the axial storage modulus (Fig. 7a) are in broad agreement; however the FE predictions are insensitive to temperature and frequency, while the measurements show a small sensitivity to temperature (with the storage modulus decreasing with increasing temperature). Similar results are observed for the corresponding loss factor, shown in Fig. 7b.

The sensitivity of axial viscoelastic properties to temperature is unexpected, as the axial response is dominated by fibre properties, which are known to be insensitive to temperature in the range explored. The measurements shown in Fig. 7a were conducted by clamping the specimens when both specimens and fixtures were at the correct test temperature; this practice reduced the sensitivity of the measured modulus to the test temperature. The measured sensitivity to test temperature was due to the presence of a resin-rich, irregular layer on the surface, as shown in in Fig. 1a. Additional simulations were performed to investigate this further, details of which are given in Appendix A; in brief, the presence of the layer resulted in a lower measured stiffness and higher measured damping.

For the case of transverse loading (Fig. 8), the FE simulations effectively predict the observed dependence of the response on temperature and frequency. The experiments show a sensitivity to temperature slightly higher than that predicted by the FE simulations, however measurements and predictions are in good agreement.

In Fig. 9 we report measurements and predictions of the shear modulus and corresponding loss factor at different temperature and frequencies. Again the FE simulations capture correctly the effects of frequency and temperature observed in the experiments. A comparison of the numerical and experimental results is provided in Table 2 for the case of 1 Hz loading frequency and different temperatures.

Table 2: Comparison of experimental results and numerical predictions of the anisotropic viscoelastic response of UD CFRP at 1 Hz frequency at 25, 40 and 50 °C.

	25°C		40°C		50°C	
	Numerical	Experimental	Numerical	Experimental	Numerical	Experimental
$E_{11}$ (GPa)	140.75	138.64	141.21	136.64	140.84	133.00
$E_{22}$ (GPa)	9.24	9.22	9.13	8.99	8.87	8.68
$G_{12}$ (GPa)	4.74	5.17	4.53	4.72	4.37	4.63
$\eta_{11}$ %	0.338	0.357	0.339	0.298	0.342	0.423
$\eta_{22}$ %	1.05	1.18	1.15	1.45	1.47	1.71
$\eta_{12}$ %	1.51	1.72	1.59	2.08	1.92	2.24

### 4.3. New equations to predict the viscoelastic properties of a composite lamina

The FE predictions conducted in this study are in broad agreement with the measurements on a composite of fibre volume fraction 0.63. We have also shown, in Section 4.1 and Figs. 4-6, that none of the theoretical models examined provides accurate predictions for all viscoelastic properties. In this section we propose a new model to allow calculations of the viscoelastic properties with improved accuracy, using as input the properties of the constituent materials.

The existing theoretical models which better captured the measured response were Mori-Tanaka and Lielens. We recall that while Mori-Tanaka is physically-based, the approach of Lielens [60] is to conduct a simple weighted averaging of the results of the Mori-Tanaka model [58] and of the Inverse Mori-Tanaka Tanaka model [27]. If the complex stiffness tensors predicted by the Mori-Tanaka (MT) and Inverse Mori-Tanaka models are denoted by  $C_{(MT)}^*$  and  $C_{(IMT)}^*$ , respectively, Lielens proposed a complex stiffness tensor calculated as

$$C_{LIELENS}^* = [(1 - f(\phi_f))C_{(MT)}^{*-1} + f(\phi_f)C_{(IMT)}^{*-1}]^{-1} \quad (3)$$

where  $f(\phi_f)$  is a non-dimensional weighting function of the fibre volume fraction,  $\phi_f$ . Based on the observation that experimental results tend to be closer to Mori-Tanaka model for low fibre volume fractions, while approach the Inverse Mori-Tanaka model at higher volume fractions, Lielens assumed

$f(\phi_f) = (\phi_f + \phi_f^2) / 2$ ; this choice however is not effective for the material investigated here. We therefore propose to use a different weighting function of general form

$$f(\phi_f) = \frac{1}{n} \sum_{i=1}^n \phi_f^i \quad (4)$$

We note that for  $n = 2$  this equation coincides with that proposed by Lielens (eq. (4)).

We proceed to find the optimal value of  $n$  which yields predictions in line with the validated FE calculations presented here, over a wide range of volume fractions. To do this we perform additional simulations on RVEs of different volume fractions, in the range 0.2-0.7. For simplicity we consider a single temperature and frequency, arbitrarily chosen as 25 °C and 1 Hz, respectively. Ten repeated FE simulations are conducted and ensemble averages of all predicted viscoelastic properties are shown as a function of the volume fraction in Figs. 10-12, for the 3 loading cases investigated. The figures include our measurements, for reference, and the theoretical predictions of the Lielens and Mori-Tanaka models. After a curve-fitting exercise on the data of Figs. 10-12, we find that the choice  $n = 5$  provides a better fit through the results of FE predictions, for all loading cases. This corresponds to a complex stiffness tensor calculated as

$$C_{TP}^* = \left[ \left( 1 - \frac{1}{5} \sum_{i=1}^5 \phi_f^i \right) C_{(MT)}^{*-1} + \left( \frac{1}{5} \sum_{i=1}^5 \phi_f^i \right) C_{(IMT)}^{*-1} \right]^{-1} \quad (5)$$

The proposed  $C_{TP}^*$  tensor accurately predicts all viscoelastic lamina properties for the UD composite examined in this study. We proceed to investigate if this accuracy is maintained as the material properties of the constituents change; in particular we explore the effect of the contrast in stiffness of the constituents,  $E_f' / E_m'$ , as this is known to lead to increased RVE size [63] and to decreased accuracy of certain theoretical models [64].

We perform additional simulation exploring the range  $E_f' / E_m' = 1 - 1000$ . We consider  $\phi_f = 0.6$  and cylindrical isotropic elastic fibres of properties  $E_f = 1$  GPa and  $\phi_f = 0.2$ ; the matrix is modelled as an isotropic viscoelastic solid with storage modulus  $E_m'$  of 1, 0.1, 0.01, 0.001 GPa; the loss factor of the matrix  $\eta_m$  is given corresponding values of 0.001, 0.01, 0.1, 1, respectively. Note that the mesh sensitivity studies were repeated for this set of simulations, due to the different contrast in material properties. The FE predictions are summarised in Fig. 13 for the case of transverse and shear loading; the case of axial loading is omitted because it is trivial (in the sense that all theoretical predictions coincide with the FE predictions, as in Figs. 4 and 10). It is clear that over the 3 orders of magnitude

investigated for the stiffness contrast  $E_f' / E_m'$ , the proposed model (eq.(5)) effectively predicts the results of FE simulations, independently of the choice of viscoelastic properties of the constituent material. The experimental validation of the proposed analytical model over a given range of fibre volume fraction is left for a future study.

## 5. CONCLUSIONS

Measurements and FE predictions of the anisotropic viscoelastic response of a UD carbon composite were presented and compared in this study; predictions are found to be in broad agreement with the experiments. The predictions inspired the construction of new simple equations to provide accurate values of the full set of viscoelastic properties of the UD composite. The concluding points of this study are follows:

- In all load cases, the macroscopic material stiffness increases with increasing imposed frequency and decreases with increasing temperature. Conversely, material damping decreases with increasing frequency and increases with increasing temperature. The axial modulus and corresponding loss factor are scarcely sensitive to frequency and temperature for the carbon fibre composite considered here.
- Conducting Monte Carlo analysis of repeated FE simulations of the response of a random RVE was shown to be an effective method to predict the viscoelastic lamina properties at different frequency and temperature. Predictions of elastic properties are more accurate than those of loss factors, which are lower than measurements. The model was calibrated using exclusively the measured responses of the constituent materials and did not include any dissipation at the fibre-matrix interface, suggesting that this dissipation is not an important contributor to the loss factors of this CFRP lamina, in the range of frequency and temperatures investigated.
- Several established theoretical models were tested against the experiments and the numerical predictions conducted in this study, and their accuracy was ranked. New equations were formulated to conduct accurate predictions of all the viscoelastic lamina properties of UD composites. These are expected to aid prototyping, design and optimisation of components made from fibre composites.

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## APPENDIX A.

### Effect of a resin-rich layer on the specimens' surface

An uneven resin-rich region was observed on one side of all composite specimens, as shown in Fig. 1; the mean thickness of this layer was 8% of the total thickness. This was due to the presence of resin infusion mat and peel-ply during composite manufacturing. Here we quantify the effect of presence of such layer by explicitly modelling the DMTA experiments conducted via FE simulations.

We simulate steady state vibrations of fully clamped beams, for a single frequency of 1 Hz and with displacement amplitude of 0.2 mm, as in the experiments. Two types of specimens are considered in the FE simulations: (a) a homogeneous CFRP beam with a fibre volume fraction of 0.63 and (b) a CFRP beam of fibre volume fraction 0.68 adhered to a resin layer on the top surface, of 8% of the total thickness, as in the micrographs of Fig. 1.

The anisotropic viscoelastic constitutive response of the composite is modelled via a UMAT subroutine, similar to the UMAT used to model anisotropic viscoelastic response of single CF fibres considered in this study; the resin-rich layer is modelled by the same technique used in our Monte Carlo simulations of the response of RVEs. The viscoelastic properties of the uniform composite beam ( $v_f = 0.63$ ) are taken from the Monte Carlo simulations of RVEs of the same volume fraction; to obtain the properties of the composite layer with volume fraction 0.68 we use the analytical model proposed in this paper (this is to avoid repeating our computationally expensive Monte Carlo simulations). Two orientations of the composite layers were considered, with fibres oriented longitudinally or transversely to the beam span.

Two different types of boundary conditions were considered: in a first set of simulations only the free length of the beams was modelled, with the clamped conditions imposed by restraining the displacements of the beam's ends. In a second set, the clamps, of width 6 mm, were explicitly modelled as pairs of rigid plates making contact with the top and bottom surface of the beam; such contacts were modelled as tie constraints.

For the 'smeared' composite beams of volume fraction 0.63, the predictions of the Monte Carlo simulations were recovered. The simulations on the inhomogeneous beams provided different values of elastic moduli and loss factors; the percent differences between such simulations and the Monte Carlo analyses are given in Table A1.

TABLE A1: Summary of FE clamped beam bending analysis of homogenous CFRP beams and CFRP beams with resin rich layer on top, for different fibre orientations, temperatures and modelling techniques.

	0° CFRP Orientation				90° CFRP Orientation			
T	$\frac{RF_{CFRP+Resin} - RF_{CFRP}}{RF_{CFRP}}$		$\frac{\eta_{CFRP+Resin} - \eta_{CFRP}}{\eta_{CFRP}}$		$\frac{RF_{CFRP+Resin} - RF_{CFRP}}{RF_{CFRP}}$		$\frac{\eta_{CFRP+Resin} - \eta_{CFRP}}{\eta_{CFRP}}$	
	Without clamps	With Clamps	Without Clamps	With Clamps	Without Clamps	With Clamps	Without Clamps	With Clamps
25 °C	-12.21%	-10.88%	0.58%	163.09%	-4.19%	2.26%	-1.28%	22%
40 °C	-14.83%	-14.92%	0.93%	189.14%	-9.87%	-9.45%	-1.97%	24%
50 °C	<b>-17.35%</b>	-14.91%	1.57%	<b>256.73%</b>	-8.33%	<b>-10.72%</b>	-0.42%	<b>26%</b>

\*RF = Reaction Force, subscripts CFRP+Resin denote beams with CFRP and resin-rich layer on top.

For the case of elastic properties (represented by RF in Table 1a) both simulations (with and without rigid clamps modelled) give e similar results, with a maximum discrepancy in modulus of -17%. However for the case of corresponding loss factor, we found that simulations considering the clamps predicted a much larger discrepancy, due to the high additional dissipation, in shear, of the complaint resin-rich region sandwiched between the rigid clamps and the stiff composite beam. The maximum differences between the two simulation approaches are found at high temperature and in the longitudinal directions. FE simulations predict higher loss factors in presence of the resin-rich layer. These trends are in line with the observations in Figs. 4-9 and indicate a broad agreement between DMTA measurements and FE predictions.

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