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# ACOUSTICS 2012

## Evaluation of transverse elastic properties of fibers used in composite materials by laser resonant ultrasound spectroscopy

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We apply the laser resonant ultrasound spectroscopy (RUS) technique to evaluate the mechanical properties of carbon fibers that are used in reinforced polymers. The mechanical vibrations of a cross section are excited by the beam of a pulsed subnanosecond laser focused and shaped onto the fiber in order to form a line parallel to the fiber. The vibrations are detected optically by the probe beam of an interferometer focused at the zone of excitation. Eigenfrequencies are measured in the 100–500 MHz range for glass and carbon fibers. Using finite element modeling, the eigenmodes of a fiber can be identified, and by solving an inverse problem, the mechanical properties of the fiber can be evaluated. The transverse Young modulus of the tested carbon fibers differs significantly from the longitudinal Young modulus. In contrast, glass fibers exhibit isotropic mechanical properties.

## 1 Introduction

The mechanical behavior of fiber-reinforced polymers depends strongly upon the mechanical properties of elementary fibers, as the longitudinal Young modulus of the fiber. The longitudinal Young modulus of fibers is generally well known for the fibers used in reinforced polymers. But the transverse Young modulus of micrometric fibers cannot be determined by tensile test. However, it is important to determine both the longitudinal and the transverse elastic properties of fibers to predict accurately the behavior of a fiber-reinforced polymer. Using the laser picosecond ultrasonics technique, the transverse Young modulus of carbon fibers was determined and the mechanical anisotropy of carbon fibers was demonstrated [1]. In the paper, we propose a method based on laser Resonant Ultrasound Spectroscopy (RUS) [2] to determine the transverse elastic properties of fibers. This method uses a pulsed laser to excite the vibration eigenmodes of a single fiber. The generated vibrations are measured using the probe beam of an interferometer. We applied the laser RUS technique to determine the transverse Young modulus of carbon fibers. In order to validate the experimental procedure, we applied the laser RUS technique to a E-glass fiber, which is expected to have isotropic mechanical properties.

## 2 Laser Resonant Ultrasound Spectroscopy

### 2.1 Laser ultrasonic setup

The vibrations of a fiber are excited using a Q-switched Nd:YAG microchip laser which delivers optical pulses with a repetition rate of 7 kHz at the wavelength 1064 nm. The pulse width and energy are 0.5 ns and 10  $\mu$ J, respectively. The pump laser beam is focused on the fiber surface using a cylinder lens to form an elliptical spot, with dimensions 6  $\mu$ m x 100  $\mu$ m, aligned along the fiber axis. A semi transparent gold film, deposited on the fiber, plays the role of an ultrasonic transducer. The pump pulse heats the gold film and then excites ultrasonic waves in the fiber. This transducer gold film is particularly important for glass fiber, which cannot absorb the pump light. For carbon fibers, the gold film is mainly used to enhance the reflectivity of the fiber and improve the signal to noise ratio of the measurement. The gold film thickness, about 10-20 nm, is small compared to the fiber diameter, so that it induces no significant changes in the mechanical behavior of the fiber. With a 0.5 ns pump pulse, it is possible to excite vibration eigenmodes up to 1-2 GHz, which is sufficient for fibers with diameters in the 5-20  $\mu$ m range. In order to excite acoustic waves that propagate in the transverse direction,

the length of the pump spot must be much greater than the fiber diameter. The probing of fiber vibrations is achieved optically with an interferometer. The beam of a continuous wave (CW) probe laser at 532 nm is focused to form a spot of about 2  $\mu$ m in diameter centered with the pump line. The probe beam of the interferometer can only measure the radial displacements of the surface of the tested fiber. The vibrations are recorded by a digital oscilloscope during about 1  $\mu$ s and the vibration spectrum is obtained by calculating the Fast Fourier Transform (FFT), with a spectrum resolution of 1 MHz (Figure 2). By measuring the line width of eigenfrequencies, the damping of eigenmodes can be evaluated.

### 2.2 Finite element modeling of the eigenmodes of a fiber

In order to evaluate the elastic properties of the fiber, we solve an inverse problem by fitting modeled eigenfrequencies with the experimental eigenfrequencies. For this purpose, we carried out a 2D-model of a fiber and we calculated the vibration eigenmodes with the software "COMSOL multiphysics" (Figure 1).

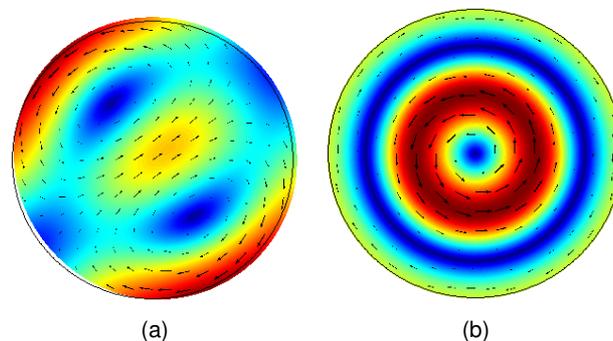


Figure 1: Display of the displacement field for two particular modes of a circular fiber. (a) The first dipolar mode:  $(m, n) = (1, 1)$  and (b) The first torsional mode:  $(m, n) = (0, 1)$ . The arrows show both the direction and magnitude of the displacement vectors. The color scale represents the magnitude of the displacement, the red color corresponds to the maximum of the displacement field and the deep blue color corresponds to nodal points.

We consider that the vibrations are homogeneously distributed along the fiber and that the displacement vectors lie in the plane of the fiber cross-section. Such modes can be named according to the number  $m$  of plane of symmetry going through the fiber axis. In addition, a second number  $n$ , orders the eigenmodes of a given type  $m$  according to their increasing eigenfrequencies. Eigenmodes with  $m \neq 0$  exhibit two-fold modal degeneracy, which differ only by a rotation of  $\pi/(2m)$  radian of the displacement fields. For a mode with  $m = 0$ , the displacement field has no plane of

symmetry; this case corresponds to torsional modes (Figure 1(b)). Modes having only radial displacements have an infinite number of reflection planes, so they are named  $(\infty, n)$  modes. The fundamental breathing mode is the  $(\infty, 1)$  mode.

By fitting the modeled eigenfrequencies with the experimental eigenfrequencies some mechanical parameters of the fiber (Young modulus, Poisson ratio, density, fiber diameter ) are determined.

### 3 Experimental results

#### 3.1 Glass fiber

The laser RUS method has been applied to a E-glass fiber. The fiber diameter  $d$ , evaluated by optical microscopy, ranges between  $21 \mu\text{m}$  and  $28 \mu\text{m}$ . Figure 2 represents the experimental spectrum of a fiber.

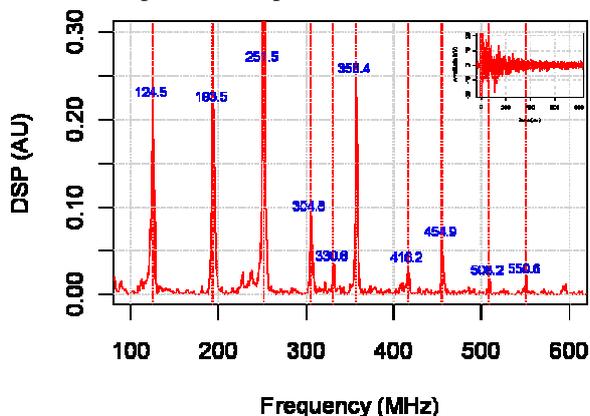


Figure 2: The vibration spectrum of a E-glass fiber. The vertical scale is the power density of the displacement signal shown in the inset.

The eigenfrequencies are indicated for the lines of strongest amplitudes. The amplitude of the lines may vary from one measurement to another but the positions of the eigenfrequencies are reproducible within 1 MHz.

The fiber diameter was adjusted in the numerical model to fit the experimental frequencies. Figure 3 shows the result of the fit. A diameter of  $20.5 \mu\text{m}$  with an uncertainty of  $0.4 \mu\text{m}$  was obtained with a residual standard deviation of about 1 MHz. The diameter uncertainty is calculated from the following mechanical parameters and uncertainties of the E-glass material: Young modulus:  $E = (75 \pm 5) \text{ GPa}$ , Poisson ratio:  $\nu = 0.25 \pm 0.05$ , and density:  $\rho = (2540 \pm 50) \text{ kg/m}^3$ .

The value of the diameter is consistent with the expected value and the fact that the actual diameter may vary slightly along the fiber. The experimental results validate the laser excitation of vibration eigenmodes of a fiber cross-section. Nevertheless, the 2D modeling is actually an approximate approach, which does not take into account of the finite length of the pump line and the propagation of guided waves along the fiber. The vibration eigenmodes of a cross-section depend on the transverse mechanical properties. Thus, it is possible to determine the transverse Young modulus of a fiber if the diameter is known. In the case of glass fibers, there is no evident difference between the longitudinal and the transverse

Young modulii, so that the glass fiber material is mechanically isotropic.

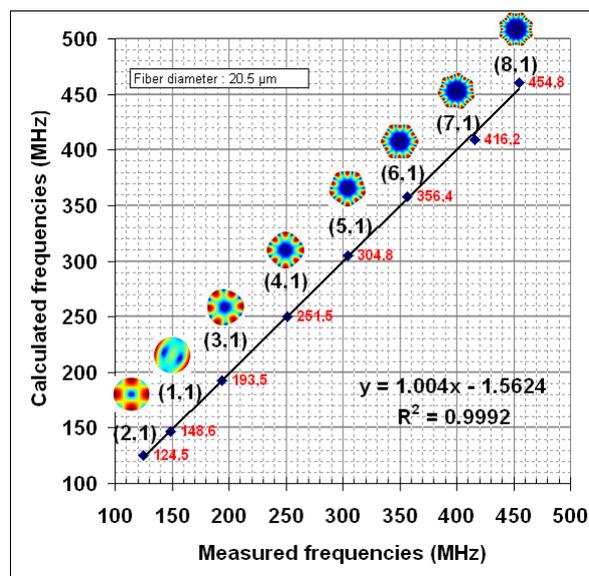


Figure 3: The best fit of the calculated frequencies with the measured frequencies (red values) giving a fiber diameter  $(20.5 \pm 0.4) \mu\text{m}$ . The modal shape of eigenmodes  $(m, n)$  are represented.

Moreover, from symmetry considerations an elliptical cross-section of the fiber would induce a lifting of the modal degeneracy. That is not observed, which confirms the quite circular shape of the glass fiber.

#### 3.2 Carbon fiber

The Resonant Ultrasound Spectroscopy technique was applied to determine the transverse Young modulus of a T700 carbon fiber. It well known that the longitudinal Young modulus of a carbon fiber depends strongly on the crystalline texture of the fiber [3]. It is also expected from the crystalline texture that the elastic properties of a carbon fiber may be anisotropic. Indeed, this property was already demonstrated on carbon fibers using laser picosecond ultrasonics [1].

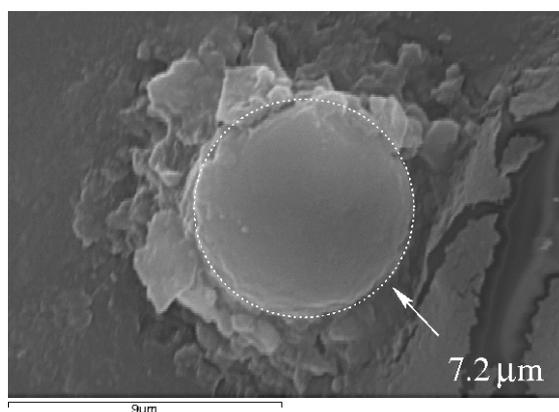


Figure 4: SEM image *post mortem* of the fiber cross-section close to the point of laser ultrasonic measurements. The cross-section is nearly circular with a diameter of  $7.2 \mu\text{m}$ .

We used alternatively the laser ultrasonic configuration that was previously described for the glass fiber. The

Young modulus of the fiber was adjusted to get the best fit of the calculated eigenfrequencies with the measured frequencies. The cross-section of the carbon fiber was determined accurately using a scanning electron microscope, SEM (Fig. 4). The fiber was embedded in acrylic resin in order to get a polished cross-section of the fiber close to the point of ultrasonic measurements. From Figure 4, we obtain a diameter  $(7.2 \pm 0.2) \mu\text{m}$  for the carbon fiber.

Figure 5 shows the vibration spectrum of the carbon fiber. The amplitude of the vibration lines may vary with the focusing of both the pump and the probe laser beams and on the fiber surface. The line at 133 MHz, due to spurious electric oscillations, cannot be attributed to any eigenmode. In reality, this line is observable when the pump beam is suppressed. The other lines in grayed boxes correspond to vibration eigenfrequencies, though they have very weak amplitudes. The line widths are smaller for the carbon fiber than for the glass fiber. It seems that the damping is weaker in the case of carbon fibers.

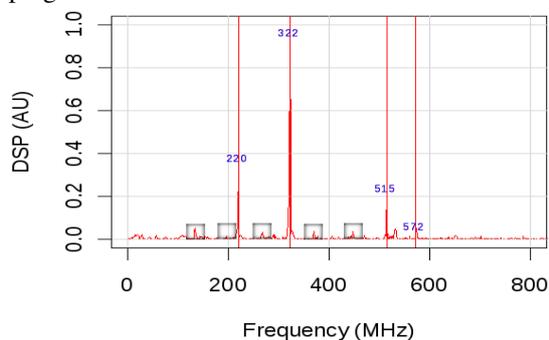


Figure 5: Vibration spectrum of a T700 carbon fiber. The vertical axis represents the power density.

The best fit was obtained for a transverse Young modulus  $E^t = (16 \pm 1.3) \text{ GPa}$ . The Poisson ratio  $\nu^t$  and the density  $\rho$  and were set respectively to  $\nu^t = 0.31$  and  $\rho = 1800 \text{ kg/m}^3$ . These two last parameters could not be determined accurately from the fit. In fact, these parameters have too weak effects on most eigenfrequencies of Figure 5. Moreover, the effects of the mechanical parameters differ with eigenmodes. For example, the Poisson ratio has an important effect on the torsional mode (Figure 1b). Except for torsional modes, the transverse Young modulus has the most important effect on eigenfrequencies.

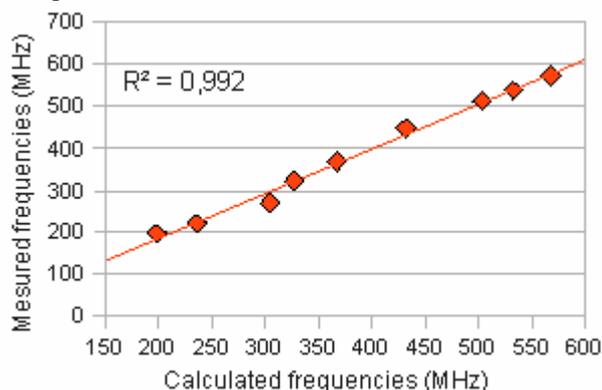


Figure 6: Best fit of the calculated frequencies with the measured frequencies.

The Young modulus was evaluated with the same method on another T700 carbon fiber, giving  $E^t = (17 \pm 1.3) \text{ GPa}$ . The transverse Young modulus of a T700 carbon fiber is significantly different from the longitudinal Young modulus  $E^L \approx 230 \text{ GPa}$ . Thus, T700 fibers exhibit a strong mechanical anisotropy with a ratio  $E^L / E^t \approx 15$ .

## 5 Conclusion

The laser Resonant Ultrasound Spectroscopy was applied to determine the mechanical properties of fibers used in reinforced polymers. To solve the inverse problem, we used a 2D finite element modeling to calculate the vibration eigenfrequencies of a fiber. The technique was validated on an E-glass fiber. Then the method was applied to a carbon fiber and the transverse Young modulus was evaluated. The tested carbon fibers display a strong mechanical anisotropy, whereas the E-glass fibers exhibit isotropic properties.

The laser RUS method provides the opportunity to determine the mechanical damping of the fiber material and thus the complex Young modulus of the material. Nevertheless, it would be necessary to carry out a 3D modeling in order to take into account the part of the attenuation due to the propagation of the ultrasonic waves along the fiber axis, which would certainly contribute to increase the apparent damping of vibration modes.

Preliminary results were obtained with flax fibers using the laser RUS method. Flax fibers have diameters in the 10–30  $\mu\text{m}$  range and they can be used to fabricate reinforced polymers, which have interesting damping properties in some applications. Eigenfrequencies of flax fibers were found in the 5–20 MHz range and eigenmodes show a particular strong damping, greater than the damping of the measured damping of glass and carbon fibers.

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