Tensile behaviour of natural fibres. Study of the stiffness increase phenomenon under cyclic loading

Vincent Placet Department of Applied Mechanics- FEMTO-ST Institute – UMR CNRS 6174 University of Franche-Comté - 24 Chemin de l'Epitaphe. F-25000 Besançon.

1. Introduction

With growing ecological concern and exigencies of legislative authorities related to the material recycling, cellulosic fibres coming from annual plants such as hemp, flax, coir, sisal, and abaca represent an interesting alternative to glass fibres in composite applications. The use of these biological resources in high performance composite requires specifically an accurate understanding of the mechanical behaviour of the fibres themselves.

This paper is focused on the understanding of a particular mechanical behaviour of natural fibres: the fibre stiffering under cyclic loading. When submitted to periodic tensile loading, the rigidity of the natural fibres increases until a constant value is reached, after a certain number of cycles. This was evidenced in literature for at least 3 fibre species: flax [1], sisal [2] and hemp [3], and could positively impact the fatigue or long-term behaviour of composites reinforced with natural fibres.

Since several years, in our team, we have developed sophisticated experimental setup and numerical tools to investigate and highlight this intricate tensile behaviour of natural fibres. The 2nd ICNP is an excellent opportunity to present the current research and the major findings collected on hemp fibres on this subject.

2. Experimental investigation of the stiffering phenomenon

2.1. Plant materials and specimen preparation

Hemp fibres (*Cannabis sativa L.*) were procured from LCDA Company in France. Elementary hemp fibres are isolated by hand from the initial bundles. According to the ASTM standard D3379 and also to simplify handling of the fibres, thin paper with glue is used as mounting tabs.

2.2. Tensile set-up for elementary fibres

A commercial Dynamic Mechanical Analyser (DMA Bose Electroforce 3230) was implemented with a thermal chamber and a humidity generator to control the environment conditions around the samples. This specific experimental set-up is able to perform quasi-static, harmonic and fatigue tests on elementary or bundle of fibres in environmental-controlled conditions. The typical operating ranges are: 0.01 to 200 Hz for the loading frequency, -70°C to 300°C for the temperature and 2% to 97% with a maximum temperature of 75°C for the relative humidity. The high resolution of the magnetic actuator and of the sensors (1 mN and 0.1 μ m) allows an accurate determination of the mechanical properties of elementary fibres.



Natural fibre in clamps

Figure 1: Tensile set-up for the quasi-static and dynamic characterization of elementary fibres

The typical clamping length is about 10 mm. Results of harmonic tests are presented as storage modulus (E'), loss modulus (E'') and loss factor (tan δ = E''/E') and are plotted according to time, temperature, RH or loading frequency with regular interval.

3. Modelling

A theoretical tool was derived to link the microstructure and the mechanical properties of the constitutive components of the cell wall to the apparent mechanical properties of the elementary fibres. This model, developed with F. Trivaudey is detailed in a specific paper [5]. It is based on the laminate theory applied to thin-walled cylindrical composite specimen and uses the continuum mechanics formulation. The elementary fibre is considered as a laminate made of a stacking of five perfectly cylindrical layers. Each layer is modelled as long fibres reinforced composite material. Microfibrills whose behaviour is supposed to be transverse isotropic, reinforce a matrix modelled as a mixture of isotropic lignin and transverse isotropic hemicellulose.

4. Results and discussion

Figure 2 depicts the characteristic behavior of hemp fibres submitted to tensile fatigue test. The rigidity of the fibre increases until a constant value is reached. After 12.5h of cycling, it is 1.45 times greater than its initial value. This result suggests an "accommodation phenomenon".



Figure 2 : Longitudinal elastic properties of a hemp fibre vs. time during fatigue test: evidence of an "accomodation phenomenon" (*Ambient relative humidity, temperature:* 25°C, loading frequency: 1 Hz, stress amplitude: 10% of the ultimate stress)

The stiffering phenomenon was showed to be time-dependent, partially reversible and also to be highly sensitive to the temperature as soon as to the moisture content [3, 4]. We particularly demonstrated that the fibre stiffness can be raised of an unexpected and remarkable value of about 2.5 times its initial value when the fibre is submitted to particular environmental conditions during cyclic loading [6]. A significant average longitudinal elongation (2.25 %) is also observed in combination to the fibre stiffering. The absorption and desorption of moisture involve in addition a substantial rotation of the fibre about its axis. These experimental results seem to be consistent with a hypothesis of re-orientation of the cellulose microfibrils during tensile tests.

The numerical results (Fig. 3) corroborate the potential role of the angle of cellulose microfibrils (MFA) in the fibre stiffering. A complete re-orientation of the cellulose microfibrils from 11° to 0° engenders a stiffering of the fibre modulus of about 2.3 times, from 33 GPa to 76 GPa.



Figure 3: Evolution of the fibre Young's modulus according to the MFA angle in the S2 layer.

5. Conclusions

Natural fibres depict a complex micro-mechanical behaviour. The stiffness of the fibre can be increased of an unexpected and remarkable value of about 2.5 times its initial value when the fibre is submitted to cyclic loading. Numerical data, collected using a comprehensive composite model, seem to corroborate the potential role of the microfibril angle in the fibre stiffering phenomenon. Recently, wide angle X-ray diffraction (WAXS) technique was used to determine the crystalline structure of cellulose and try to evaluate the MFA [7]. The first results are encouraging and seem to confirm the involvement of microfibrils rearrangements in the cell wall during cyclic loading, as observed on coir fibres by Martinschitz *et al.* [8].

References

- 1. C. Baley. Analysis of the flax fibres tensile behaviour and analysis of the tensile stiffness increase. Compos: Part A **33**, 939-948 (2002)
- F.A. Silva, N. Chawla, R.D. Toledo Filho. An experimental investigation of the fatigue behaviour of sisal fibers. Mat Sci Eng A 516:90-95 (2009)
- 3. V. Placet. Characterization of the thermo-mechanical behaviour of hemp fibres intended for the manufacturing of high performance composites. Compos: Part A **40**, 1111-1118 (2009)
- V. Placet. Tensile behaviour of natural fibres. Effect of loading rate, temperature and humidity on the "accommodation phenomena". 14th International Conference on Experimental Mechanics 4-9 July 2010, Poitiers France.
- 5. V. Placet, F. Trivaudey. Tensile properties of natural fibres. Part I: Influence of the lumen size and fibre diameter on the elastic properties. Submitted to J Mater Sci
- 6. V. Placet, F. Trivaudey. Tensile properties of natural fibres. Part II: Investigation of the fibre stiffering phenomenon under tensile cyclic loading. Submitted to J Mater Sci
- 7. V. Placet, A. Bouali, P. Perré. The possible role of microfibril angle of Hemp fibre during fatigue tests and its determination using Wide-Angle X-raydiffraction. Submitted to Mat & Tech.
- K.J. Matinschitz, P. Boesecke, Č.J. Garvey, W. Gindl, J .Keckes. Changes in microfibril angle in cyclically deformed dry coir fibers studied by in-situ synchrotron X-ray diffraction. J Mater Sci 43:350-356 (2008)