Composites: Part A 51 (2013) 62–70

Contents lists available at SciVerse ScienceDirect

Composites: Part A

journal homepage: www.elsevier.com/locate/compositesa

# Effect of flax fibres individualisation on tensile failure of flax/epoxy unidirectional composite

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ARTICLE INFO

*Article history:*
Received 22 November 2012
Received in revised form 12 February 2013
Accepted 27 March 2013
Available online 11 April 2013

*Keywords:*
A: Natural fibre composites
B: Strength
B: Microstructure
C: Micro-mechanics

ABSTRACT

The study of plant fibres composites is a widespread research topic; nevertheless, the reinforcement mechanism understanding of these materials must be still improved. The paper presents a study of the effect of the mechanical properties, the dispersion and the fibre/matrix interface property of elementary fibres on the tensile properties of unidirectional composites. Our work shows that the mechanical performances of unidirectional composites could be linked to those of the elementary fibres as well as to the composites microstructure. Flax fibres individualisation, linked to the homogeneity of the microstructure, is highly dependent on the fibre extraction process. The importance of the composites homogeneity has been confirmed by the Rosen model, which could be used thanks to interfacial shear strength measurements.

## 1. Introduction

Using vegetal fibres such as flax fibres in composite materials as reinforcement makes it possible to obtain interesting mechanical properties [1], while reaching high environmental performance [2]. Flax fibres are present in the outer periphery of flax stems in the form of unitary fibres ($5\ \mu m < \text{diameter} < 40\ \mu m$) assembled into bundles of 10–40 single fibres [3]. Each elementary fibre has a complex multi-scale microstructure. The key parameters of this structure are the microfibrillar angle, the organisation of amorphous and crystalline phases, the size of the lumen [4], the biochemical composition and interactions between cellulose fibrils, hemicelluloses and pectins matrix [5]. These parameters are governed by the genetics and the growing conditions of the plant [6], which explain the wide range of mechanical properties that flax fibres have, one of which being their tensile strength [7].

The Weibull cumulative distribution function can be used to describe the tensile failure behaviour of brittle materials. It is based on the weakest link theory and on the assumption that only one kind of flaw leads to failure [8]. The Weibull distribution is given by:

$$P(\sigma) = 1 - \exp\left[\frac{-L}{L_0}\left(\frac{\sigma}{\sigma_0}\right)^m\right] \quad (1)$$

where $L_0$ is the gauge length reference, $P$ is the cumulative probability of fibre failure under $\sigma$ stress at $L$ gauge length. Parameters $m$ and $\sigma_0$ are respectively the Weibull modulus and the Weibull scale parameters. The Weibull modulus is a statistical value without any physical meaning. However this value could be linked to the flaws size distribution [9,10]. When the Weibull modulus is high the tensile failure is near the theoretical strength [10], which means small and homogeneously distributed flaws. This model has been successfully applied to synthetic fibres [8,11] and flax fibres by means of a two-parameter Weibull law [12].

In a composite material, the interface has a major role to play in the composite behaviour. The fibre/matrix adherence can be directly done through micromechanical tests such as fibre fragmentation tests [13], pull out tests [14] or microbond tests [15]. Then, taking into account the shear stress uniformity, it is possible to determine an apparent shear strength $\tau_{app}$, which is characteristic of the fibre/matrix bond. Thanks to high calculation hypothesis, this allows for more qualitative analysing [16].

To reach high mechanical performances, composites have to be reinforced with highly aligned fibres, and the fibres need to have a homogeneous distribution into the matrix. Using flax fibres as reinforcement in a composite highlights a mixing of elementary fibres and bundles into the composite. The proportioning of the mix changes according to the quality of the extraction process (retting,

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http://dx.doi.org/10.1016/j.compositesa.2013.03.018

hackling) [17]. Bundles contain single fibres linked together by a middle lamella which has been characterised and modelled by Charlet and Béakou [18]. The presence of this lamella induces a modification of the damage mechanism [19]. Bundles have lower mechanical properties than single fibres [20]. Rask et al. [21] studied the damages processes of PP/flax UD by using X-ray diffraction; they concluded that well separated fibres are recommended for composite reinforcements. Andersons and Joffe [22] enounced similar conclusions by demonstrating that a probabilistic model, assuming perfect separation and regular spacing of fibres, yields an upper limit of strength for UD flax composites.

Among the analytical models used to estimate the strength of unidirectional composites, these proposed by Rosen [23] is fundamental. This model takes into account the statistical behaviour of fibre tensile failure to describe the damage accumulation of tensile strength. Rosen [23] supposes a homogeneous repartition of the load on the fibres. Unbroken fibres carry the load initially carried by the breaking fibres, without any load concentration effect. Rosen's model was found to overestimate composite failure strengths [24] but predict a satisfactory upper bound of the tensile failure in unidirectional composites [25].

The aim of this paper is to present a multi-scale analysis of the tensile failure in a unidirectional composite reinforced with flax fibres. Tensile tests on single fibres from three different varieties of flax were conducted in order to characterise the mechanical behaviour of the fibre, and to determine the influence of variety on fibre properties. These fibres have been treated through different extraction process this lead to several individualised fibre content which have been characterised thanks to image analysis. To complete the microscale analysis, microbond tests were carried out to determine the interfacial shear strength between the fibre and the epoxy matrix. Unidirectional composites reinforced with the three different fibre varieties were tested to study their tensile behaviour. The experimental data were compared with those used by Rosen for its respective analytical model which includes the microbond test results and the statistical analysis of single fibre failure.

## 2. Materials and method

### 2.1. Material

Three different varieties of flax fibres cultivated in France were used in this study (Table 1). Two of them are currently cultivated in France (Marylin and Andrea) and the third one (Hermes) is a well-known flax variety, whose mechanical or biochemical properties have been measured previously by our research group [3,26]. These varieties have been chosen for their differences and dispersion of their mechanical properties induced by various growth conditions (hail for Andrea) or years (2003 for Hermes and 2009 for Marylin and Andrea). They were dew retted on the field before being scutched and hackled. E-glass fibre extracts from industrial roving will be the reference of the study. The fibres were textilo-plastic sized.

An epoxy resin (Axson, Epolam 2020) was used as matrix. It was mixed with its amine hardener at 100:34 ratios. After hardening for 24 h at 25 °C, all samples were post-cured following supplier recommendation (3 h – 40 °C; 2 h – 60 °C; 2 h – 80 °C; 5 h – 100 °C).

**Table 1**
Characteristics of flax and glass.

| Material | Variety | Year | Hackling | Weather information |
|---|---|---|---|---|
| Flax | Hermes | 2003 | Yes | – |
| Flax | Andrea | 2009 | No | Hail |
| Flax | Marilyn | 2009 | No | – |
| Glass | E | – | – | – |

### 2.2. Preparation of the unidirectional composite

Fibres locked of a length of 10 cm were impregnated with epoxy. They were then put into a 100 mm long aluminium mould with a $6 \times 2\ \text{mm}^2$ section opened on each side. Fig. 1 shows a sketch of the system.

The small cross section of the mould induces the unidirectional resin flow during the compression and preferential orientation of the fibres in the composite. The control of fibre content is described below. Fibreglass tabs (±45°) were bonded at the end of the tensile sample with an Araldite adhesive to reduce the risk of breakage in the jaws during the tensile test.

### 2.3. Density and fibre content measurements

The measuring of the fibre content was carried out by density measurement, with a balance Mettler Toledo. Samples are weighed both in air and in pure ethanol. The density calculation is given by Eq. (2). Then, the fibre volume fraction ($V_f$) and the density are linked together through a simple mixture law, such as the following:

$$\rho_{\text{composite}} = \frac{M_{\text{air}}}{M_{\text{air}} - M_{\text{ethanol}}}(\rho_{\text{air}} - \rho_{\text{ethanol}}) + \rho_{\text{ethanol}} \quad (2)$$

where $\rho_{\text{composite}}$, $M_{\text{air}}$, $M_{\text{ethanol}}$, $\rho_{\text{ethanol}}$ and $\rho_{\text{air}}$ are respectively the density of the composite, the sample mass in the air, the sample mass in ethanol, and the density of ethanol and air at experiment temperature.

### 2.4. Tensile test on single fibres

Tensile tests on elementary fibres were carried out at a controlled temperature (23 °C) and relative humidity (48%) to measure longitudinal mechanical properties (Young's modulus, ultimate strength and failure strain) of elementary flax fibres were determined. The length of the elementary fibres being in average in the range of 20–30 mm, they were stuck on a paper frame to have a gauge length of 10 mm. The fibre was clamped on a universal MTS type tensile testing machine equipped with a 2 N capacity load cell, and loaded at a constant crosshead speed of 1 mm/min up to rupture. The determination of the mechanical properties was made in accordance with the NFT 25-704 standard which takes into account the compliance of the loading frame. For each variety of fibre, at least 90 fibres were tested. Before the tensile test, the diameter of every fibre was measured from six points taken along the fibre with an optical microscope. A statistical analysis based on the Weibull model was performed on the tensile strength results.

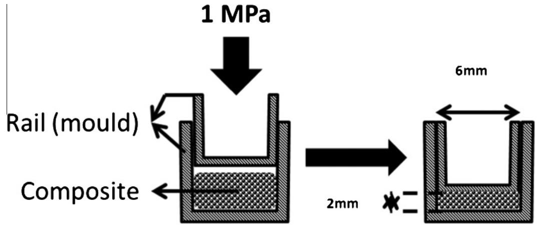

**Fig. 1.** Moulding the unidirectional composite.