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Tension–tension fatigue behaviour of woven hemp fibre reinforced epoxy composite: A multi-instrumented damage analysis

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ABSTRACT

The purpose of this work is to characterise the tensile–tensile fatigue behaviour of a woven hemp fibre reinforced epoxy composite, adding up analysis of fatigue damage mechanisms by combining different techniques: optical microscopic and X-ray micro-tomography observations, temperature field measurement by infrared camera, and acoustic emission monitoring (AE). Two different stacking sequences: [0°/90°] and [±45°] are compared. A power law based model is used to fit *S–N* curves of experimental results. $[\pm 45°]_7$ layups show better fatigue strength than $[0°/90°]_7$ ones, in relative terms. This is explained by the difference of their damage behaviour, in concordance with the local shear stresses developing in $[\pm 45°]_7$ laminates. Moreover, high resolution micro-tomography pictures allow one to clearly visualise the yarn/matrix interface damage in these materials. The obtained results give a complete description of fatigue damage mechanisms, and a damage scenario during fatigue tests is proposed for these eco-composite materials.

1. Introduction

In recent years, there is a growing scientific and economic interest on plant fibres because of their eco-friendly production and disposal [1,2], accompanied by interesting mechanical properties [3]. Plant stem fibres (hemp, flax and jute) and lamina fibres (sisal) have been presented as possible substitute candidates of traditional fibres as plastic reinforcements for semi-structural applications, specially for E-glass fibres composites [4]. In this context, it is necessary to completely characterise the mechanical properties of plant fibre composites (PFC) but there is still few data on atypical structural loading for this material (off-axis, multi-axial, high-strain rate, creep and fatigue) [5,6].

Focusing on the fatigue behaviour, there are several studies on traditional fibre composites [7–18]. Already in the 80s one can find studies on the fatigue damage development of composites materials [16,19], where critical data in the fatigue behaviour of these materials are pointed out as: progressive and dispersed damage, distinct damage modes and the dominance of fibre damage modes on the final failure. Later, papers are focused on producing experimental data [8,9,17,18] and/or on modelling fatigue lifetime [8,9,11,13,15]. On the other hand, data on plant fibre composites (PFC) are more recent. Gassan [20] did a complete initial characterisation, analysing the influence of several parameters on the fatigue behaviour, such as: type of fibre (UD-flax/epoxy versus UD-jute/epoxy), textile architecture (UD-jute/epoxy versus woven-jute/epoxy), fibre–matrix adhesion (treated and non-treated jute as reinforcement of epoxy, polyester and polypropylene resins) and fibre volume fraction. Later, Yuanjian and Isaac [21] studied the impact and tension–tension fatigue behaviour of a mat-hemp/polyester composite and compared it with a ±45° glass fibre cloth/polyester. Towo and Ansell [22,23] studied the behaviour of treated UD-sisal/polyester and treated UD-sisal/epoxy composites in tension–tension and tension–compression fatigue tests. Shahzad [24,25] contributed characterizing tension–tension fatigue behaviour of hybrid mat-hemp-E-glass/polyester and mat-hemp/polyester composites. More recently, Liang et al. [26] studied tension–tension fatigue behaviour of a UD-flax/epoxy composite and compared the results with those of a similar UD-glass/epoxy composite. And finally, Shah et al. [5] performed also an extensive analysis of fatigue behaviour of polyester PFC, presenting the influence of: fibre volume fraction (for UD-jute/polyester); textile architecture of flax/polyester composite, comparing UD (0°), UD (90°) and stitched biaxial (±45°) fabric; fatigue stress ratio on UD-hemp/polyester composite ($R = 0.1, 0.3, 0.5, -1, 2.5$).

The present work aims to characterise the tensile–tensile fatigue behaviour of a woven fabric hemp/epoxy composite, which had its static tensile behaviour previously characterised by Bonnafous [27,28]. This study also adds up an extensive analysis of fatigue damage mechanisms by combining different techniques: mechanical parameters evolution, acoustic emission monitoring, X-ray mi-

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cro-tomography observation and temperature field measurement by infrared camera. This analysis was done for two different stacking sequences ([0°/90°] and [±45°]), because they present different local stress states.

2. Materials and methods

2.1. Tested materials

The studied composite material is made of 7 plies of a plain woven hemp fabric impregnated with epoxy resin. The hemp fabric is non-treated, has a fabric weight of 267 ± 1 g/m^2, has three hemp yarns for each warp and weft thread, has a thread count of 2362 × 1575 (warp and weft per metre) and it was produced by *Lin et ĹAutre*, France. The yarns are composed of hemp fibres which have an average diameter of 13 ± 5 μm [27]. Those yarns are produced with twist level of 324 tpm (yarn surface twist angle of 11°) and a linear density of 83*tex*. Besides of the irregular cross-section, the hemp yarns have an apparent diameter of 300 ± 60 μm based on Weibull distribution [27]. The epoxy resin is an EPOLAM 2020 from *Axson Technologies* with density of 1.10 g/cm^3, for cured resin (according to the manufacturer datasheet) [27]. The composite plates were manufactured at the enterprise *Valagro*, France, by a Resin Transfer Moulding process (RTM), with resin injection (at pressure of 7 bar) in a rigid aluminium mould tool with an initial vacuum of 30 mbar (absolute). The hemp fabric is pre-dried at 40 °C for 24 h. The plates were cured by the following cycle: 24 h at ambient temperature, 3 h at 40 °C, 2 h at 60 °C, 2 h at 80 °C and 4 h at 100 °C. The resulting composite plates are characterised by a density of around 1.24 ± 0.01 g/cm^3[27], a fibre volume fraction of 36 ± 2% and a maximum void content of around 4%. These two last parameters were measured from X-ray micro-computed tomography observation. Two different composite plates were fabricated: one, referred as $[0°/90°]_7$, has the warp direction of each ply oriented at 0° from the tensile axis (*x* axis). The other one, referred as $[\pm45°]_7$, has the warp direction of each ply oriented alternately at +45° and −45° from the tensile axis. From these plates, rectangular specimens were cut (without any lubrication) with the dimensions of 150 mm × 20 mm × 3 mm. The jaws of test machine take 30 mm of each extremity. The specimens edges were polished to remove mechanical damage caused by the cutting and 80 grit sand papers were used in the jaws to improve clamping. No failure was observed in the tab area.

2.2. Experimental procedure

In order to define the stress levels for fatigue testing, static ultimate tensile strengths (UTS) had to be determined. Static tensile tests were performed by using an INSTRON 4505 electromechanical machine with a cross-head speed of 0.5 mm/min, a 100 kN cell force and strain was measured by a 10 mm extensometer. Three specimens of each layup were tested. The fatigue tests were performed with an INSTRON 8501 servo-hydraulic machine. Constant amplitude loads were applied in a sinusoidal waveform at the frequency of 1 Hz (which avoids self-generated heating in the specimen) under load control, in tensile loading exclusively, and with a fatigue stress ratio (R), i.e. ratio between minimum (σ_{min}) and maximum (σ_{max}) stresses, of 0.01. This value of fatigue stress ratio was chosen to approach the "zero load", enabling a correct measurement of the minimum strain value at each cycle. It indicates the permanent strain, which is a damage indicator [29]. Maximum and minimum cycle strain values were calculated as the machine cross-head displacements divided by specimen gauge length. The accuracy of this calculation was not an issue as it was used to analyse relative strain evolution only qualitatively and a comparison with extensometer measurement showed equivalent evolution of cycle strain. Fatigue tests were stopped at specimen failure or when reached 10^6 cycles. At least 9 specimens of each layup were tested. In order to do a detailed analysis of the damage process, the mechanical tests were multi-instrumented. *In situ* measurements were carried out by using: acoustic emission (AE) monitoring, infrared camera and high resolution observations of specimen surface. After fatigue tests, some specimens were also observed with X-ray micro-computed tomography (micro-CT).

The acoustic emission monitoring was performed by using the AE system from *Physical Acoustics SA*. The sensors are of type Micro80, with ceramic face and diameter of 10 mm. Two sensors were placed at the gauge extremities, with a distance of 80 mm between their centres. The measured amplitude values of AE events were corrected by the corresponding attenuation curve, as proposed by Mechraoui et al. [30]. Acquisition parameters and acoustic events definition were taken from the work of Bonnafous et al. [27,31] on the same material.

In this previous work, experimental tests were performed on single hemp yarn, neat epoxy resin and composite specimens. Then, statistical analysis of AE amplitude signals were realised and correlated with microscopic observations. Results have enabled to identify three types of damage in this composite material and it has been shown that AE amplitude was a discriminating signature. Based on normal distribution of AE amplitudes, three amplitude ranges with high probability to be related to each damage mode have been proposed: 35–53 dB for epoxy resin cracks, 58–63 dB for interface damage mechanisms, and 66–100 dB for hemp fibre damage. AE signals with amplitude in the intervals between 53–58 dB and 63–66 dB have high uncertainty to be classified in a single damage mode, so were not considered.

Specimen surface was observed during tests with a Nikon D3X digital single-lens reflex camera (DSLR), with a 35.9 mm × 24 mm CMOS sensor and a resolution of 5.95 μm/pixel. Observations of specimen surface were also performed after tests with an optical digital microscope from *Leica Microsystems*, with a 6× objective and a resolution of 1.36 μm/pixel. The X-ray micro-computed tomography (micro-CT) observations were made at the *Jean Lamour Institute* in Nancy, France. A 20 mm × 20 mm × 3 mm volume near the failure zone was observed with a resolution of 14.8 μm/pixel, for several specimens. In addition, micro-CT observations of a 10 mm × 2.5 mm × 3 mm volume zone of a damaged specimen with a resolution of 0.7 μm/pixel were performed at the *European Synchrotron Radiation Facility* (*ESRF*), in Grenoble, France. An infrared camera from *Cedip Infrared Systems* with a detector resolution of 90 μm/pixel and a sensitivity of 0.1 °C was used to measure the temperature evolution at the surface of some specimens during the fatigue tests.

2.3. Data analysis

In order to analyse fatigue *S*–*N* curve results for composite materials, power law based equation regressions have been widely used [5,12,14,15,17,18]. In this work, an empirical model developed by D'Amore et al. [11], also based on power law equation but which considers in addition the influence of stress ratio, was used to fit fatigue *S*–*N* curve results. This model was initially developed and verified for random glass–fibre-reinforced plastics (RGFRP) with thermosetting resin. Later, Caprino and Giorleo [9] checked the applicability of the model to a glass fabric/epoxy composite. These verifications were made with four-point bending fatigue tests and for different stress ratios. The model is based on the following assumptions:

- The mechanical degradation of the material in fatigue is in accordance with a power law.