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Influence of hygrothermal ageing on the damage mechanisms of flax-fibre reinforced epoxy composite

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ABSTRACT

This paper describes the influence of hygrothermal ageing on mechanical properties and damage behaviour of quasi-unidirectional flax-fibre reinforced epoxy (FFRE) composite. The evolution of water absorption for FFRE composite appears to be Fickian and the kinetics parameters are influenced by the temperature variation. Young's modulus and tensile strength are clearly affected by the hygrothermal ageing because a significant reduction in Young's modulus is shown while tensile strength decreases much less for water-saturated composites. The decrease of both properties could be explained by a reorientation of flax microfibrils and the plasticiser effect of water on the matrix, which are both stimulated by moisture absorption. Acoustic emissions analysis combined with scanning electron microscopy enabled investigating the effects of hygrothermal ageing on the process of degradation of flax-fibre composite.

1. Introduction

Today, the needs for more environmentally-friendly products in the industry are in strong growth resulting from ecological concern, environmental attentiveness and new rules and regulations. The development of vegetal fibre reinforced polymer presents numerous advantages because of their biodegradability and their low density. Besides, vegetal fibres, used as reinforcement of composite materials, constitute an interesting alternative to glass fibres because of their easy-processing and their competitiveness in both mechanical properties and cost. Vegetal fibres, which originate from renewable resources, nowadays form a new class of materials having the potential to be a substitute for mineral and organic fibres in several structural applications.

However, because of the hydrophilic nature of the fibre, the use of vegetal fibre reinforced composites requires to know how they behave in a wet environment. Research investigations showed that the exposure of vegetal fibre composites in a wet environment leads to a decrease of the mechanical properties when water spreads in the material. This loss of properties due to water absorption was noticed on various vegetal fibre composites, as composites with fibres of sisal [1], bamboo [2], jute [3], hemp [4] or flax [5–11]. The knowledge of environmental ageing is essential for the development of vegetal fibres reinforced composites. Dhakal et al. [4] showed, in the case of hemp fibre reinforced polyester composites, that the moisture uptake increased with the fibre volume fraction and induced a significant degradation of the mechanical properties of the composites at an elevated temperature. Alix et al. [5] studied the hydric interface in unsaturated polyester matrix reinforced with treated flax-fibres. The use of treatments on the flax-fibres improved their mechanical properties and enhanced the water resistance and the permeametric properties of composite films. Le Duigou et al. [6] studied the influence of seawater ageing on the properties of bio-composites. They showed that the weakening of the flax-fibre/PLLA matrix interface is one of the main factors of damage mechanism induced by water uptake. The efficiency of the interface is critical both for short term properties and long term durability. The strength of the interfacial bond depends on many parameters, such as the chemistry of the matrix and the roughness of the fibre. These parameters can be modified by different treatments [12,13]. The interfacial bonds between vegetal fibres and relatively hydrophobic polymer matrices could be weakened with high water uptake. This weakened interface causes the reduction of the mechanical properties of the composites. In this regard, the water resistance of the natural-fibre composites could be improved by modifying the surface of the fibres and by using coupling or compatibilizing agents. The use of upgraded Duralin flax-fibre in natural-fibre-mat-reinforced polypropylene thermoplastics allowed the reduction of the moisture absorption by about 30% compared to that of untreated Green flax composites, after immersion in water for about 60 days [14]. The treatment of flax-fibres improved the mechanical properties of composites and

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these properties showed that the tensile strength of composites was not as much affected by the water uptake as the stiffness. The modification of the flax-fibre surface and also of polypropylene matrix led to the improvement of the mechanical properties of composites and to a significant decrease in the water uptake [7].

To notice the damage development in composite materials, many different methods have been used for many years, as the technique of Acoustic Emission (AE) combined with Scanning Electron Microscopy (SEM), which have been used in this work for flax-fibre reinforced epoxy (FFRE) composite. The possibility of determining the failure modes of composites reinforced with natural fibres using AE technique does not need to be demonstrated any more [15]. From static tensile tests, Romhány et al. [16] distinguished three ranges of acoustic events in biodegradable composites (containing thermoplastic starch and flax-fibre in various contents). Fibre–matrix debonding and splitting of the elementary flax-fibres occur at low loading. When increasing loading, fibre pull-out becomes significant while the breakage of fibre bundles represents the final failure. Czigány [17] has carried out a multi-parameter AE study during various types of loading to characterize the evolution of damage over different composites, such as matrix deformation at the beginning (21–35 dB), then delamination (35–45 dB) and fibre pull-out (45–60 dB), and ultimately fibre breakage and the failure of the composites (over 60 dB).

This paper deals with the effect of hygrothermal ageing of epoxy composite materials reinforced with quasi-unidirectional flax-fibres. From experimental procedures, the characteristics of the water absorption and the effect of the water absorption on the tensile properties of the composites have been studied. AE has been used to examine how hygrothermal ageing affects the different mechanisms of damage of FFRE composite. Finally, electron microscopy was used to observe the various failure modes after ageing.

2. Experimental procedure

2.1. Materials

The composite materials were manufactured from several layers of quasi-unidirectional flax/epoxy prepregs. The polymer matrix used for the prepregs was an epoxy system consisting of an Araldite LY 5150 resin and an Aradur 5021/XB3471 hardener, at a ratio of 100:16 parts by weight, both produced by the Huntsman company. The reinforcement is made of twisted flax-fibre yarn coming from wet-spinning (24 Nm, 471 revolutions per meter). The weight of unidirectional fabrics was 180 g m^{-2}. The weft and warp ratio was 1/19; the yarn numbering in the warp direction was 41.6 g km^{-1} while, in the weft direction, it was 21.7 g km^{-1}. The tested material, which is 11 ply unidirectional laminate, was prepared by hot platen press process with prepregs from epoxy resin and flax-fibre, as described in [18]. A plate of 480 mm by 480 mm was manufactured with a plate thickness of 2.5 mm and with a fibre content of 44% by volume fraction and a porosity content of approximately 6% by volume fraction. These values were measured, by weighing the plates, by taking into account the density of fibres, resin, and fabrics (1540 kg m^{-3}, 1200 kg m^{-3} and 180 g m^{-2} respectively), and by using the following equation:

$$\upsilon_{\mathrm{p}} = 1 - \frac{m}{l^p \cdot w^p \cdot h^p \cdot \rho_m} + \frac{n^p \cdot \rho_f^s}{h^p} \cdot \left(\frac{1}{\rho_m} - \frac{1}{\rho_f}\right) \tag{1}$$

where m is the mass of the plate; l^p, w^p and h^p are the dimensions of the plate; ρ_m and ρ_f are the mass density of the matrix and the dry fibres respectively; ρ_f^s is the surfacique density of the dry fabric; and n^p is the number of layers.

2.2. Hygrothermal ageing of FFRE composite

Specimens from each batch of tensile samples were subjected to hygrothermal ageing. After being dried during 24 h, they were put into an environmental test chamber with a relative humidity (RH) of 90% at different temperatures (20 C and 40 °C). The samples were not protected on the edge in order to accelerate the process of diffusion. During the ageing experiment and at certain periods of time, specimens were periodically taken out of the chamber. To assess the weight change, they were wiped dry with tissue paper and weighed using an analytical balance having a precision of ±1 mg. The weighing of the specimens was stopped when weight reached saturation point, that is to say when the weight gain was nearly constant.

After various periods of time, the water absorption characteristics of the composites were evaluated by the relative uptake of weight defined by M_t according to:

$$M_t = \frac{W_t - W_0}{W_0} \times 100(\%) \tag{2}$$

where W_0 is the weight of dry specimen and W_t is the weight of wet specimen at time t.

The degradation behaviour of vegetal fibre composites exposed to environmental conditions such as humidity is characterized, in most cases, by a phenomenon of water absorption. The way in which the material absorbs water depends on many parameters, such as hygrometric rate, temperature, fibre fraction, fibre nature, porosity fraction, reinforcement geometry, matrix type. Furthermore, water could penetrate inside the reinforcement itself. It is particularly possible for composite reinforced with twisted fibre yarn. The transport of water can be facilitated by its diffusion inside the resin, by the presence of defects within the matrix, formed during the compounding process (micro-space, pores or cracks) or by capillarity along interfaces which present some defects between fibres and resin. Even if these mechanisms are jointly active, taking into account the diffusion mechanism only can be enough to model the global behaviour. The different categories of diffusion behaviour (as Fickian or Non-Fickian) can be distinguished theoretically by the shape of the sorption curve represented by:

$$\frac{M_t}{M_m} = k \cdot t^n \tag{3}$$

where M_t is the moisture uptake at time t, M_m is its maximum moisture uptake, at equilibrium state, and k and n are the diffusion kinetic parameters. The diffusion exponent n indicates the mode of diffusion. When n is equal to 0.5, diffusion obeys Fick's law. The mechanism is non-Fickian when $n = 1$ (or $n > 1$) while the diffusion is anomalous when n shows an intermediate value, between 0.5 and 1. Moisture uptake in vegetal fibre reinforced plastics usually follows a Fickian behaviour. Fick's laws, in the case of one-dimensional approach, shows that the water uptake increases linearly with the square root of time, and then gradually slows until an equilibrium plateau is reached.

For values M_t/M_m lower than 0.6, the initial part of the curve can be correlated by:

$$\frac{M_t}{M_m} = \frac{4}{h}\sqrt{\frac{D \cdot t}{\pi}} \tag{4}$$

where D is the diffusion coefficient and h is the thickness of the specimen.

For the second half-sorption, for M_t/M_m upper than 0.6, Shen Springer [19] proposed the following approximation:

$$\frac{M_t}{M_m} = 1 - \exp\left[-7{,}3\left(\frac{D \cdot t}{h^2}\right)^{0{,}75}\right] \tag{5}$$

The diffusion coefficient D, which is an important parameter in Fick's law, is determined from Eq. (4), in the case where values M_t are less than 60% of the equilibrium value M_m:

$$D = \frac{\pi}{(4M_m)^2}\left(\frac{M_t h}{\sqrt{t}}\right)^2 = \pi\left(\frac{k}{4M_m}\right)^2 \qquad (6)$$

where h is the specimen thickness and k is the slope of the linear part of the curve $M_t = f(\sqrt{t}/h)$.

2.3. Tensile tests

Tensile tests were carried out using an universal mechanical testing machine Instron, model 3382. The tensile tests were conducted at room temperature, with a cross-head speed of 2 mm min^{-1}. A clip-on extensometer with 50 mm gauge length was used to measure the longitudinal strain. According to ASTM standard D3039, the tensile test specimens are in rectangular form (20 mm × 200 mm) and aluminium tabs were bonded with a suitable adhesive. For each set of samples, all the results were taken as the average value and the standard deviation of five samples.

2.4. Acoustic emission

With the aim of analysing specific damage phenomena occurring in composites, the data acquisition system made by Mistras Group and equipped with two channels, a sampling rate of 5 MHz and a 40 dB pre-amplification was used to record AE data. AE measurements were achieved by using two piezoelectric sensors with a frequency range 100 kHz–1 MHz, placed on one of the sides of the specimens with silicon grease, as described in [10].

3. Results and discussion

3.1. Water absorption

Fig. 1 shows the percentage of water absorbed for the samples after 90% RH ageing at 20 °C and 40 °C, as a function of square root of ageing time divided by thickness. The solid curves show the theoretical water uptake behaviour predicted from Fick's second law of diffusion. The curves were calculated by using Eq. (4) for the linear part and by using Eq. (5) for the second part. Saturation weight gains and diffusion coefficients are shown in Table 1. Both curves in Fig. 1 are found to follow Fickian type behaviour. It can be seen that temperature has an influence on moisture uptake of FFRE composite. Results shown in Fig. 1 reveal that the increase of the temperature decreases the time necessary to reach the equilibrium plateau, which is characterised by an increase of the slope of the curve. When the ageing temperature is increased from 20 °C to 40 °C, the diffusion coefficient, D, calculated by Eq. (6), increases by 5.55 to 10.97 × 10^{-7} mm^2 s^{-1}. The same trend is observed for saturation level of water uptake. The value stabilizes around 4.10% at 20 °C after 62 days of ageing and around 6.94% at 40 °C after 38 days of ageing. This difference in water uptake at 40 °C compared to 20 °C may indicate that the composite will be more degraded, which should have an impact on the mechanical properties and on the damage mechanisms.

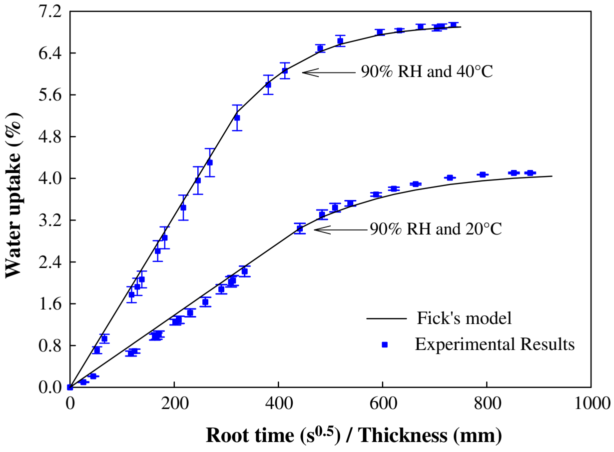

Fig. 1. Evolution of water uptake of FFRE composite after 90% RH ageing at 20 °C and 40 °C.

Table 1
Values of equilibrium water uptake M_m and diffusion coefficient D.

Ageing conditions	M_m (%)	D*10^{-7} (mm^2/s)
90% RH/20 °C	4.10	5.55
90% RH/40 °C	6.94	10.97

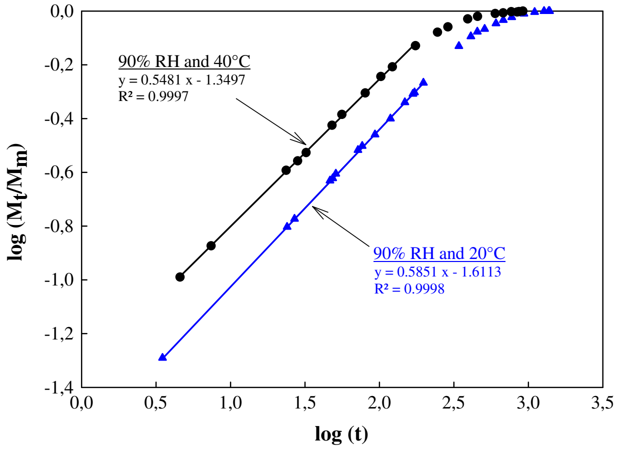

Fig. 2. Diffusion case fitting plots for FFRE composite at 20 °C and 40 °C.

Table 2
Diffusion kinetic parameters.

Ageing conditions	n	k
90% RH/20 °C	0.585	0.024
90% RH/40 °C	0.548	0.045

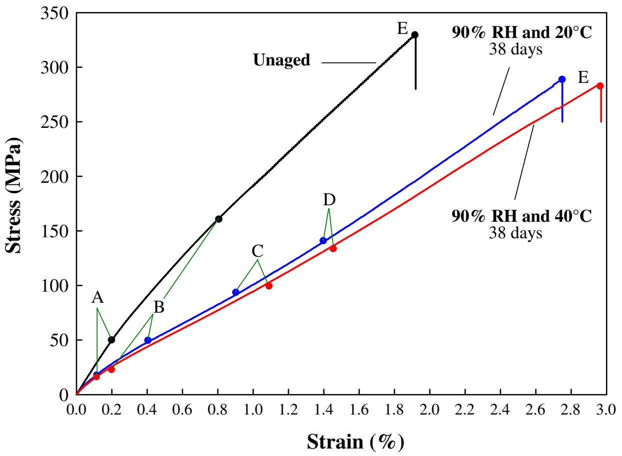

Fig. 3. Stress–strain response under tension loading for FFRE composite.

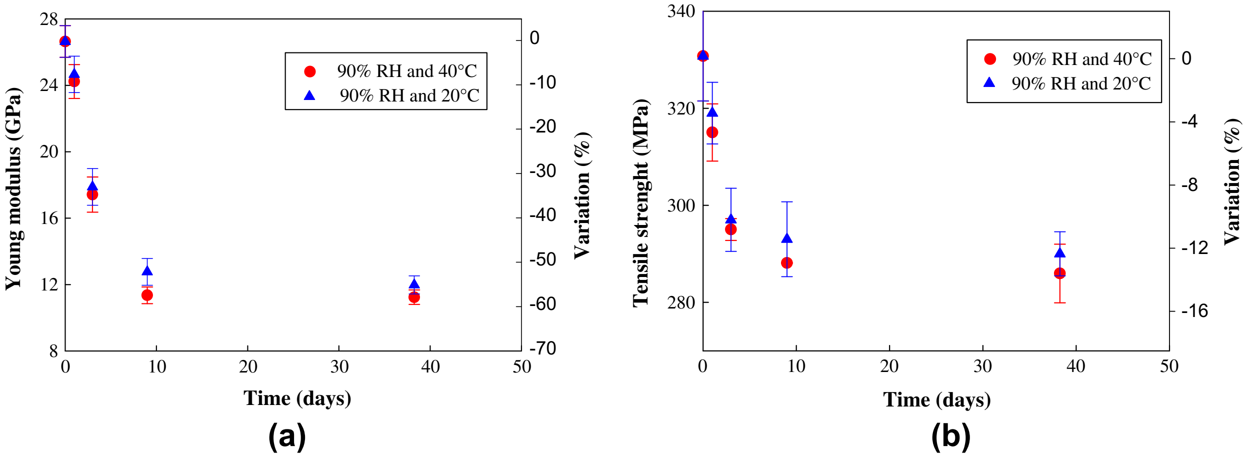

Fig. 4. Evolution of mechanical properties according to ageing time: Young's modulus (a), and tensile strength (b).

Table 3
Maximal strain according to the time of ageing.

Ageing tests	90% RH/20 °C	90% RH/40 °C
Unaged	1.56 ± 0.13	1.56 ± 0.13
1 day	1.60 ± 0.10	1.69 ± 0.10
3 days	1.71 ± 0.11	2.02 ± 0.08
9 days	2.33 ± 0.13	2.79 ± 0.21
38 days	2.72 ± 0.05	2.95 ± 0.15

Fig. 2 reports the fitting in the logarithmic form of experimental data to Eq. (3) for FFRE composite. The values of the kinetic parameter n and k resulting from the fitting are summarized in Table 2. The values of n, close to 0.5, show that the process of absorption is close to the Fickian behaviour in the case of FFRE composite. Experimental results also indicate that the diffusion parameter k depends on the temperature, because k increases when the temperature increases. The increase in the value k also indicates an increase of the interaction between the moisture and the composite material. Similar observations were reported by other researchers for other natural fibre-reinforced composites [20,21].

3.2. Mechanical properties

In order to study the influence of temperature and humidity on mechanical behaviour, we must look at Fig. 3 which shows typical stress–strain curves under tension loading for FFRE composites, unaged and aged at 90% RH during 38 days at 20 °C and at 40 °C. Note that 38 days of ageing are equivalent to 100% of M_m at 20 °C and to 97.5% of M_m at 40 °C. As it may be observed, the shape of the stress–strain curves allows identifying distinct portions, some of which are linear. The initial portion of the curves (O–A) which is linear – up to a strain of 0.2% for the unaged composite and of 0.10% for the aged composites – is associated to the elastic domain. The difference between the unaged and aged composites in the modulus calculated in this linear portion is significant (unaged: 26.6 GPa vs. aged: 12.0 GPa at 20 °C and 11.2 GPa at 40 °C). After the first portion, a knee in the curves corresponding to a change in the slope is observed (A–B). For the unaged composite, the stress–strain curve becomes non-linear from 0.2% strain (A) with a moderate decrease of slope, up to a strain of 0.8% (B). From 0.8% strain, the curve becomes linear again until failure (E). These two different portions have been previously described on several flax/epoxy prepreg composites and were attributed [18,22] to the reorientation of the microfibrils which was described for individual flax-fibre during tensile test in [23,24]. For aged FFRE composites, the non-linear portion (A–B), illustrated by a knee in the curve, becomes shorter than that of the unaged composite. In the following regions, the curves are not any more described by one linear portion but by two linear portions, B–C and D–E. There is a drop in stiffness (up to 0.9% strain at 20 °C and up to 1.1% strain at 40 °C) but then, we notice a moderate increase of the curve slope (at a strain of 1.4%), until failure (E).

This behaviour, with three linear phases, was described for plant fibre [23] as well as for plant tissue [25], which are considered as composite material i.e. an assembly of matrix and reinforcement. In the case of FFRE composite, the description of the three linear phases was previously observed only for unidirectional FFRE composite [18,22]. The same observation for aged quasi-unidirectional FFRE composites is understandable if we admit that the collective reorientation of flax microfibrils is stimulated by the moisture absorption.

Fig. 4 illustrates the evolution of the mechanical properties of FFRE composite according to the duration of hygrothermal ageing. Results of Fig. 4 and Table 3 illustrate that Young's modulus, the tensile strength and the maximal strain are affected by hygrothermal ageing. For composite aged at 90% RH / 20 °C, Young's modulus decreases by 33% during the first 3 days and by 55% after 38 days of ageing. The evolution is different for the tensile strength because the ageing leads to a decrease of this property by 10% and by 12%, respectively at 3 and 38 days. The maximal strain increases furthermore by about 10% and by 74%, respectively. These three trends are slightly amplified with the ageing at 40 °C. The mechanical results indicate that the hygrothermal ageing modifies the mechanical behaviour of FFRE composite. This change induces a great variation of Young's modulus and maximal strain, whereas it induces a smaller drop of tensile strength.

3.3. Acoustic emission

In order to obtain information about damage mechanisms of FFRE composite, the acoustic signals recorded during tensile tests were analysed according to amplitude and duration signal. Fig. 5 shows a study of the percentage of the number of hits versus amplitude (Fig. 5a) and versus signal duration (Fig. 5b), for samples that are unaged and aged during 38 days. Focusing on amplitude frequencies (Fig. 5a), all specimens have a maximum percentage of the number of hits centred around 40–45 dB. These amplitude ranges of acoustic events are considered to be characteristic of damage matrix, such as deformation, tearing and cracking of the matrix [15,17]. Note that AE amplitudes higher than 55 dB are

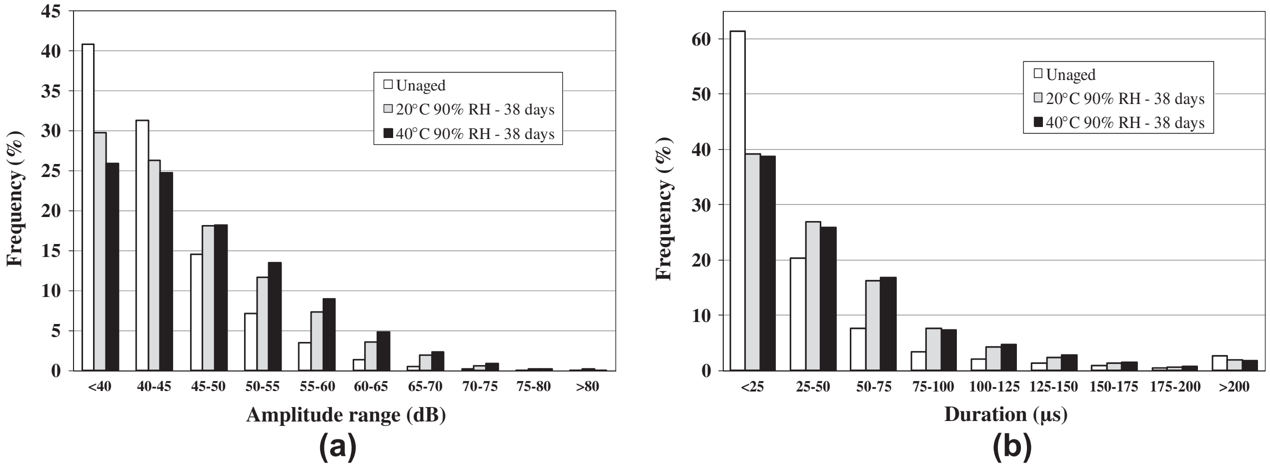

Fig. 5. Comparison of distribution of AE signals during tensile tests on FFRE composite (unaged and after 38 days of ageing) according to: amplitudes (a) and duration signal (b).

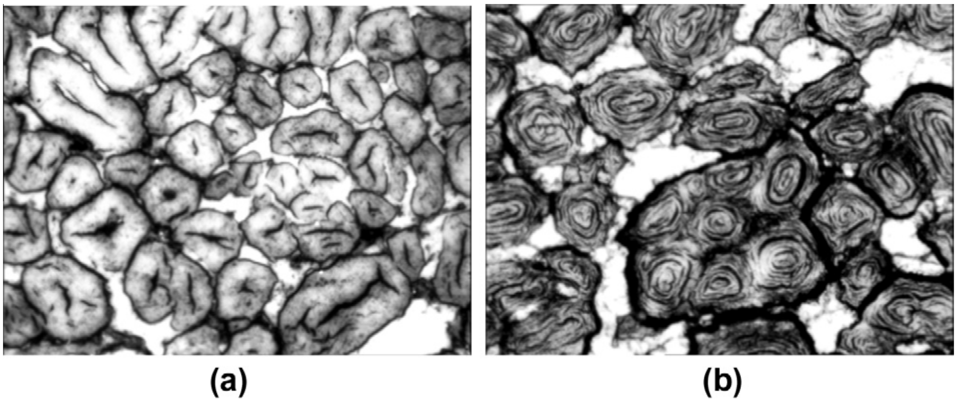

Fig. 6. Pictures of FFRE composite polished with specific lubricant (a) and with water (b), perpendicularly to the fibre axis.

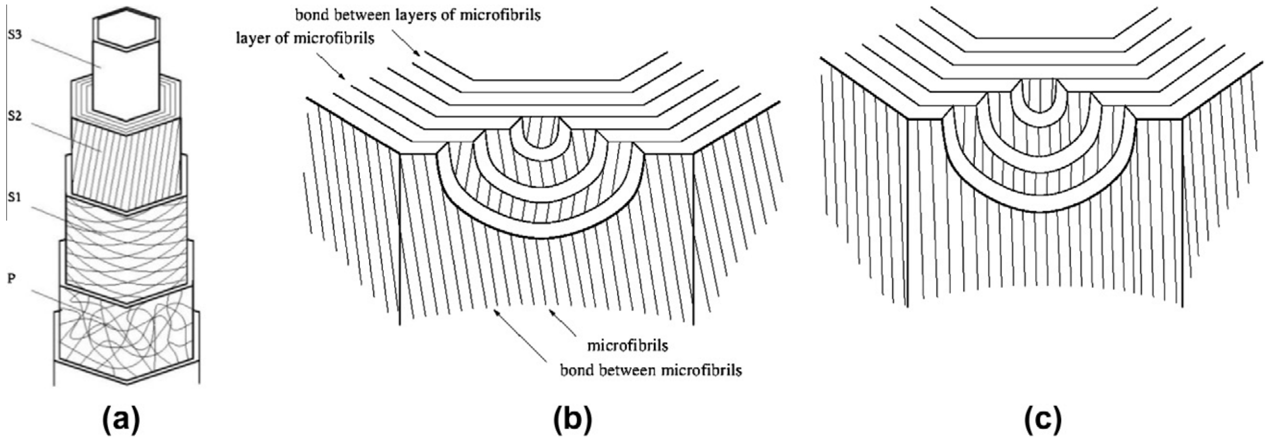

Fig. 7. Schematic representation of individual flax-fibre (a) main walls (b) initial orientation of microfibrils inside the S2 wall (c) orientation of microfibrils inside the S2 wall (with hygrothermal ageing) under tensile loading.

more significant with ageing: the proportion of these hits is equal to 19% for the 40 °C specimens, to 15% for the 20 °C specimens and to 7% for the unaged specimens. The effect of the ageing due to relative humidity is characterized by a shift of histograms toward higher amplitudes. The temperature, from 20 °C to 40 °C, makes this shift even more important. Focusing on duration frequencies (Fig. 5b), the maximal number of events occurs at low duration (lower than 25 μs), with a frequency around 60% for the unaged composites and around 40% for the aged ones. The signal duration increases with the ageing due to relative humidity only, while the temperature does not modify it. The signal duration is higher than 50 μs for 32% of proportion of hits for the aged specimens, against

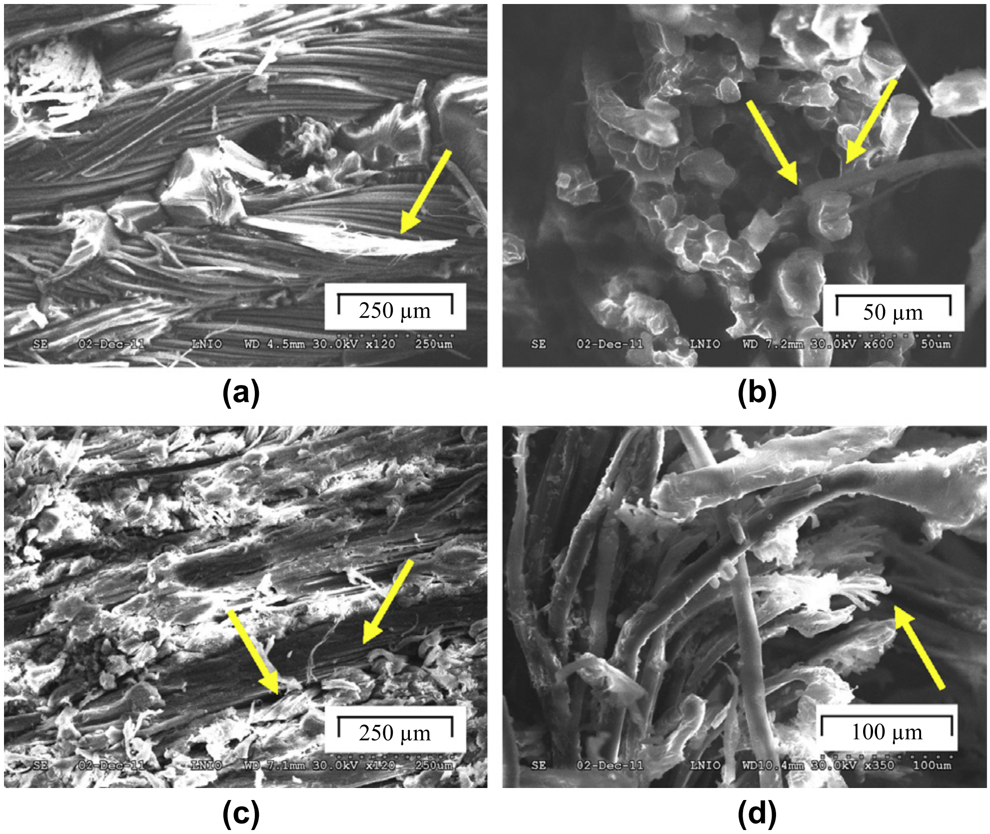

Fig. 8. SEM pictures taken from the fracture surfaces of FFRE composite: unaged (a–b), after 38 days of ageing at 20 °C and 90% RH (c) and after 38 days of ageing at 40 °C and 90% RH (d).

16% for the unaged specimens. As observed during the analysis in amplitude, ageing induces the increase of the proportion of hits toward higher durations.

3.4. Synthesis

In order to analyse the influence of the hygrothermal ageing, damage mechanisms have been evaluated at various stages of damage. The stress–strain curve was divided into the following three ranges on the basis of the maximum stress value: (I) 0–$0.8\sigma_{max}$, (II) $0.8\sigma_{max}$–$0.9\sigma_{max}$ and (III) $0.9\sigma_{max}$–σ_{max}. The choice of these three ranges is explained by AE activity and by the shape of the curves of the cumulative events as already mentioned in other research articles [16]. About 70% of the number of AE events has been detected during the second and third ranges (i.e. in the final linear phase of the stress–strain curves). It could be noted that very few AE events have been detected below the yield stress (A) whatever the ageing. The low percentage of events in the first range (where stress–strain curves show distinct portions some of which are linear) is explained by the reorientation of flax microfibrils rather than by damage.

In case of living plant cells, water generates the mobility of microfibrils [26]. To illustrate our interpretation for dead cells, Fig. 6 shows the same sample of FFRE composite polished by specific lubricant in (6a) and by water in (6b). The comparison of these pictures shows that some (essentially pectin) bonds between individual fibres, i.e. inside bundles, and some (essentially hemicellulose) bonds between constitutive layers of individual fibre, i.e. inside the S2 wall (Fig. 7a), are not only softened but are broken by the action of water. The same observation occurs for the bonds between microfibrils, which are the reinforcement of constitutive layers of individual fibres [26]. Thus, the mobility of microfibril layers and microfibrils themselves is privileged with the presence of water, due to a plasticising effect, as previously mentioned in previous articles [8]. Fig. 7b and c (illustrated from [27]) shows the mechanism of reorientation. Due to the fact that the water acts as a plasticiser, microfibrils can move more freely because the internal bonds are softened (or broken in some cases). With an application of tensile loading, the microfibrilar angle (between the microfibrils and the fibre axis) changes in such a way that the microfibrils align themselves in the direction of the fibre. Consequently, this phenomenon has an effect on FFRE composite, even if the reinforcement is made of twisted yarns and the epoxy matrix does not let the yarns come loose. In fact, the bonds between fibres and epoxy are considered stronger than the natural bonds inside bundles and inside individual fibres [28]. These natural bonds are themselves approximately two orders of magnitude weaker than microfibrils [26]. The microfibrils reorientation is not clearly distinguished from AE signals but clearly explains the global tensile behaviour of FFRE composite.

In order to distinguish the different damage mechanisms due to the ageing, that occurred on the microstructure of composite, the specimens' surfaces were examined using a Jeol SEM. Small sections of the FFRE composite from the selected failed specimens unaged and after ageing at 20 °C and 40 °C were cut transversely to the beam axes 5 mm away from the failed centre region. Fig. 8 shows representative SEM images taken at suitable magnification for each failed specimen. The analysis shows that the failure of the tested composite materials (unaged, 90% RH/20 °C, 90% RH/40 °C) is essentially induced by the following damage mechanisms: micro cracking of the matrix (Fig. 8c), fibre pull-out and fibre–matrix debonding (Fig. 8b), flax-fibre fracture (Fig. 8a). The flax-fibre

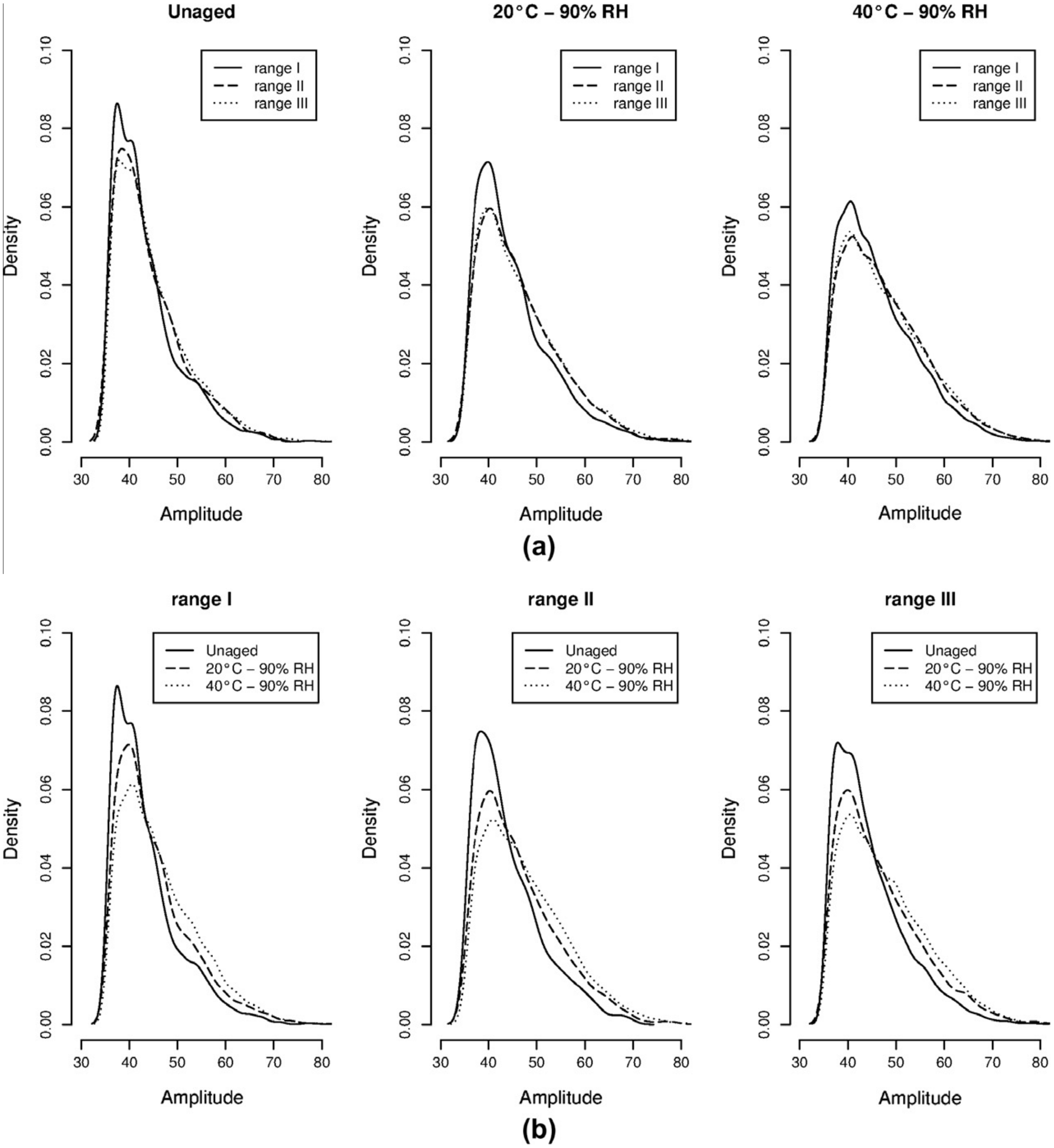

Fig. 9. Density of the AE events versus amplitude in the stress ranges (I, II and III) for FFRE composite (unaged and after 38 days ageing at 20 °C and 40 °C): (a) synthesis according to the ageing and (b) synthesis according to the stress ranges (I, II and III).

failure occurs according to several processes: axial splitting of the pectin boundary layer, pull-out of microfibrils, explosion of microfibrils (Fig. 8a and d). These observations also indicate that hygrothermal ageing does not induce other mechanisms of damage but only accelerates the process of degradation, particularly concerning the flax-fibres, helped by the plasticisation of bonds and the reorientation of the microfibrils.

The density of the AE events versus amplitude in the stress ranges (I, II and III) for the unaged and aged composites are depicted in Fig. 9. For the unaged material, Fig. 9a indicates that around 80% of the recorded signals show amplitudes lower than 50 dB whatever the stress ranges and that the maximal density appears in range I for amplitudes centred at 38 dB. With the ageing, this peak of density curve decreases and shifts towards higher amplitudes (range I in Fig. 9b). These results show that, by the epoxy plasticization effect, the hygrothermal ageing induces less matrix damage. In ranges II and III, the amplitude curves are similar whatever the ageing (Fig. 9a).

In fact, results of this study show that only the moisture absorption affects negatively the elastic properties, whereas both temperatures of ageing are far fewer penalising. Indeed, increasing the temperature of ageing accelerates the kinetic of water diffusion probably by a combination of matrix dilatation and reinforcement drying. The evolution of mechanical properties with ageing cannot only be explained by interface weakness and by yarns untwisting because the decrease of ultimate stress is very moderate with the ageing. The reorientation of microfibrils, which comes from individual fibres plasticisation, seem to be a major damage of FFRE composite due to hygrothermal ageing.

4. Conclusion

This study aimed at investigating the influence of hygrothermal ageing on the damage mechanisms of quasi-unidirectional flax-fibre reinforced epoxy composite. This study was undertaken from the water absorption results, the static tensile mechanical characteristics, the AE analysis and the SEM observations. From this work, the following conclusions can be drawn:

- Water absorption on FFRE composite was proven to follow a Fickian diffusion mode, where the kinetics parameters, such as M_m, D and k, are influenced by the temperature of ageing.
- The tensile mechanical behaviour is clearly affected by the hygrothermal ageing, both on the shape of the stress–strain curves and on the properties' values. A significant reduction in stiffness is shown while strength decreases much less for the water-saturated composites. The modification of the shape is explained by the reorientation of the microfibrils. The decrease in stiffness and strength are explained by a plasticiser effect of water on the matrix.
- The analysis of the signals of AE enabled, on the one hand, to identify the chronology of the various damage mechanisms in FFRE composite and, on the other hand, to characterize the evolution of this chronology according to the hygrothermal ageing. SEM observations allowed a better understanding of this evolution, revealed by the AE signals detected during the different stress ranges. These observations proved that several modes occur during the flax-fibre failure: axial splitting of the pectin boundary layer, pulling-out of microfibrils, explosion of microfibrils.

The results analysis shows that the hygrothermal ageing damages mainly the matrix, which induces the presence of the other damage mechanisms (fibre/matrix debonding and fibre breakage) at lower loading than without the ageing process. Extensions of this work would be, on the one hand, to study other matrix less sensitive to water absorption, and on the other hand, to use multi-variable analyses to distinguish, more in details, the AE signals from different damage mechanisms.

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