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CURE AND DAMAGE MONITORING OF FLAX FIBER-REINFORCED EPOXY COMPOSITE REPAIRS FOR CIVIL ENGINEERING STRUCTURES USING EMBEDDED PIEZO MICRO-PATCHES

Mingfa Zhang, Olivier Bareille, Michelle Salvia

mingfa.zhang@ec-lyon.fr, olivier.bareille@ec-lyon.fr, michelle.salvia@ec-lyon.fr

Ecole Centrale de Lyon, LTDS CNRS UMR 5513, Université de Lyon, France;

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ABSTRACT:

In recent years, high interests of repair of civil engineering structures were concentrated on the fibre-reinforced thermosets as a structural component for repairing use. The properties of the resulting mechanical composites are directly in relation to the reinforcement type but also to viscoelastic matrix properties. This study, proposes to in-situ follow-up the cure mechanism of a flax reinforced bio-epoxy-amine resin as repairing patches using piezoelectric elements embedded in the composite structure. After curing, the sensors remain in the part and was used as a damage detector for structural health monitoring (SHM). [Curing results compared to DMA analysis show that embedded piezoelectric cells are well suited to in-situ monitor the reaction progress. Moreover, this technique was found to be reliable for detecting preliminary signal of critical state for the composite by comparison with Acoustic Emission and Digital Image Correlation \(DIC\).](#)

1. Introduction

The built heritage in France is wide but often aged. Many civil engineering structures have defects that need to be repaired in a more or less short-term period to prevent the rapid development of structural disorders that may lead to tragedies such as Morandi Bridge (Italy) collapse.

In recent years, increasing interests of maintenance and repair of civil structures have concentrated on the fibre reinforced thermosets as structural components for repairing applications. They are expected as a repair and/or reinforcement method for structural rehabilitation for their high specific stiffness, specific strength and chemical resistance compared to traditional materials such as steel and concrete. Those composite patches are usually fabricated by hand lay-up manufacturing process and then bonded on the cracked structures to prevent the growth of the failures. Among the thermosets, room temperature curing epoxies get the most attention due to their merits for fabrication and easy to control. The properties of composites are directly in relation to reinforcement type, stacking sequence but also to viscoelastic matrix properties and reinforcement/matrix interactions. The most important factor, which controls the properties of the matrix and of the reinforcement-matrix

interface, is in particular the cross-linking density. The cross-linking density is influenced by manufacturing processes, and it is linked to the degree of cure. However, the cure of thermosets is a complex process to form a three-dimensional (3D) macromolecular network, and the final morphology of this particular 3D network determines the properties and applications of the final material.

That is, how to monitor and control the curing process of thermosets to the desired level is an essential issue. As a result, there is a growing interest in utilizing the sensors which can be embedded within a structural material and provide real-time, performance feedback. In previous, several systems with a sensing function, which could be embedded within the composite material to analyse, were used to evaluate the different phenomena during on-line polymerization of thermoset materials, as for instance dielectric sensor, optical fibres, or piezoelectric sensors. These sensors can be used to monitor the health state of composites, from their curing process to the end of their life cycle.

Dielectrics sensors consist of interdigitated electrodes deposited on an insulating substrate. They are used to measure the sample complex permittivity ($\epsilon^* = \epsilon' + j \epsilon''$) and conductivity (σ) changes during curing. From these measurements, it is possible to follow the progress of chemical reactions and to obtain information on relevant phenomena in term of different level of polymerization [1-2]. Implantation of optical fibres in composite structure during cure, helps to follow the progress of chemical reaction in term of degree of conversion using for example the variation of its density by refractive index measurements (intensity-based sensors) [3] or by the use of Bragg gratings sensors (wavelength based sensors) that measure the local strain changes associated to thermoset shrinkage [4]. The embedding of small piezoelectric inserts through the material to be monitored overcomes coupling reproducibility with conventional ultrasonic non-destructive testing (NDT) technique. These inserts can be used to have in-situ information about material state from the matrix liquid state to the end of their life cycle [2-5].

This study, in the frame of a collaborative research (MICRO ANR project), proposes to use flax reinforced bio-epoxy-amine resin as repairing patches. Up to now continuous carbon fiber reinforced epoxy composites (CFRP) are mainly used especially due to their lightness and durability to repair existing infrastructures and to increase their life-span. Indeed, the density of carbon fibers (1.77 g/cm^3 for the high strength carbon fibre which is the mostly used) is significantly lower than that of E-glass fibres (GF) (2.6 g/cm^3), and alkaline environment of concrete can cause degradation to the latter.

Natural fibre reinforcements especially flax fibres, show interesting characteristics for civil engineering applications for which the reduction of carbon emissions is becoming one of the main challenges [6]. They are environmentally friendly, relatively cheap to produce and renewable. The stiffness (E) and strength (σ_{ult}) along the axis of the fibre are in the range (45–55 GPa) and (800–1000 MPa) respectively, and due to their lightness (density = $1.4\text{--}1.5 \text{ g/cm}^3$), their specific properties are comparable to those of the E-glass fibre (E= 73 GPa ; σ_{ult} = 2000 MPa) [7].

Small PZT piezoelectric discs fully embedded in the composite were chosen as sensors. These sensors have high sensitivity and are well suited for monitoring composite repairs. They are manufactured directly on site. They are indeed more easily embeddable than optical sensors for which precautions must be taken: (i) to prevent excessive bending during handling,

(ii) to embed them parallel to the reinforcing fibres to avoid the occurrence of large resin pockets. These kind of flaws can have a detrimental effect on the durability of the composite repair.

The change in the impedance spectrum, which is linked to the changes of matrix viscoelastic properties as cure progressed, was used to analyse the different steps of the epoxy cure and their consequences on the mechanical properties of the material. Moreover, after curing, this sensor could be used as structural health monitoring (SHM) sensor for the composite material. Indeed, during their use, the composite patches could be subjected to different damages, such as aging processes or debonding from the concrete structure. The SHM capability of such a system is based on the existence, during the damaging processes of acoustic emission, which can be registered by piezoelectric sensors in a passive or active way.

2. Materials

The thermoset resin system is designed for ambient and low temperature cure. The DGEBA epoxy prepolymer is a low molecular weight “green” epoxy resin made from bio-based epichlorohydrine (EnviPOXY®530 from SPOLCHEMIE) (28% bio-based carbon). The bio-hardener is a solvent-free, low viscosity phenalkamine curing agent made through the Mannich reaction of cardanol from cashew nuts, formaldehyde, and amines (Cardolite NX5619) (100 % bio-based carbon). Resin and hardener were mixed in stoichiometric ratio. The glass transition temperature (T_g) of the reactive mixture, T_{g0} was -39°C determined from DSC heat flow curves (midpoint), recorded at a heating rate of 10 K/min (DSC1 device from METTLER-TOLEDO). In this work, the curing was carried out at room conditions ($T = 23^\circ\text{C}$; HR = 40%). The reinforcement is a quasi-unidirectional flax fabric made of untwisted rovings (LINCORE® FF FLAX FABRIC) supplied by DEPESTELE Group. The weft and warp ratio is 9/91 and the areal density is 200 g/m^2 . The relative moisture content of the fabric is about 5-6 % under ambient-condition storage and it was used as received, without any preliminary treatment. In this work, the curing was carried out at room conditions ($T = 23^\circ\text{C}$; HR = 40%). The PZT discs sensors are 0.2 mm thick disk with a diameter of 7 mm from STEMINC® working in radial vibration mode (resonant frequency is about 300 kHz). The Curie temperature of the ceramics is around 250°C . Ag electrodes are deposited on each side of the ceramic and the electrical wires for connection were soldered on one side.

3. Experimental

3.1. In situ curing monitoring method

The technique used in this work was based on the measurement of the electrical impedance of a piezoelectric PZT ceramic disc embedded in the composite materials as its impedance is linked to the characteristics (stiffness, damping) of the surrounding medium, which vary as cure progresses. Indeed, the curing process is the transition that thermosettings change from viscous liquid state to infusible cross-linked solid (3D-network) [8-9]. This transformation may involve two main transitions: gelation when the viscous liquid prepolymer is transformed in an infusible, insoluble gel or rubber-like material and vitrification, which corresponds to rubber-glass transition. At vitrification, the rate of the cure reaction may be reduced due to molecular mobility. This phenomenon occurs only if the reactive mixture is cured at a temperature lower

than the infinite T_g of completely cross-linked system. This takes place when the T_g equals the cure temperature.

The composites were fabricated using wet hand lay-up to be representative of materials used in the rehabilitation of civil engineering structures. A first UD fabric layer was arranged on a wooden mold coated with release agent. The resin was then applied by using a squeegee and a paint roller to ensure enough infiltration of the reinforcement with the resin and removing excess resin and entrapped air. Once the first layer was fully impregnated, the second reinforcement ply in which two piezoelectric discs were placed is applied and impregnated. Figure 1 shows the plate after manufacturing. To ensure the wetting of the outer ply and to minimize air pocket rate, careful roll-out was done again. Indeed, poor wet-out and large amount of porosities can induce a structural weakness of the composite repairs and affect their long-time behavior [10]. In civil engineering rehabilitation field, skilled labor is required, but due to difficult workability environment conditions, composites may exhibit some defects originating from manufacturing processes. This justifies the need for in-situ control in this procedure.

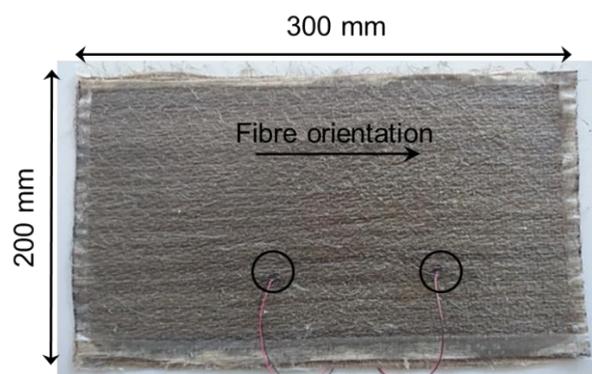


Figure 1: Flax reinforced epoxy composite material with embedded PZT discs; the position of the sensors is given by the black circles

The purpose of embedding two piezo-cells between the two layers of the flax fabric was to ensure a way to perform an analysis of the composite material integrity, by signal transmission through the plate. However, it was here used to confirm the recorded signal shape by comparison with one another. The PZT sensors were connected to an impedance analyser and the impedance was monitored as a function of time at room temperature (RT) at 4 kHz. The principle of the measurement was to record the voltage at the poles of the electrical resistor which is mounted in series with the piezo-disc. The ratio of this voltage to the voltage supplied to the two elements (piezo + resistor) gives the value of the piezo's electrical impedance. Due to the electromechanical properties of the piezo-component, this electrical impedance is linked to the mechanical ones and therefore to the level of curing for the matrix. The choice of the signal frequency is led by the signal-to-noise ratio of the mounted electromechanical system. The choice of the resistor's value is made in order to have the same order of magnitude as the expected piezo impedance. The recorded signal is the time-dependent electrical impedance of the piezo-disc. They confirmed the efficiency and the reliability of the method when compared to conventional methods as shown further in the paper in section 4.2.

3.2. Dynamical mechanical analysis

Dynamical mechanical analysis (DMA) was performed for a better understanding on piezoelectric results. This technique provides convenient and sensitive determination of thermo-mechanical properties of polymers and reinforced polymers at solid or liquid state as a function of frequency and temperature [11]. The dynamic mechanical characteristics (complex modulus, E^* and loss factor, $\tan \delta$) were determined using a DMA50 of 01dB METRAVIB in tension-compression mode on rectangular specimens ($30 \times 10 \times 2 \text{ mm}^3$) with fibres aligned perpendicular to the load axis. Tests were carried out at controlled dynamic displacement ($\pm 5 \text{ }\mu\text{m}$) in the linear viscoelasticity domain within a temperature range of -50 at 150°C (heating rate = 1 K/min) at a constant frequency of 1 Hz .

3.3. Tensile test and damage monitoring

Experimental investigations on SHM capability of piezoelectric sensor were performed using tensile test. In this case, the sensor's time DC voltage was directly measured. The purpose of integrating two sensors was initially to address the capability of analysing the composite material's level of wear by signal transmission from one piezo to the other: one piezo would have been used as an emitter and the other as a receiver. This strategy was harder to implement than expected since the choice of the frequency range can change significantly the vibration's interaction with the singularities and the breaks. The idea has not been abandoned, just postponed at this stage. However, integrating a couple of sensors was here used to confirm the recorded signal shape by comparison with one-another.

After curing and aging at room conditions during 12 months, samples for tensile tests with the fibers oriented at 0°C to the main specimen axis were cut at LMC2 laboratory in the area where the sensors were embedded. Specimen dimensions were 200 mm long, 25 mm wide and 2 mm thick. Composite end-tabs (50mm-long) were bonded on the ends of the tested specimens in order to minimize the stress concentration due to the tightening of grips. The gage length is 140 mm . The volume fraction of flax fibres for the composite samples is about 0.20 (Image analysis using Image J).

Monotonic tensile tests (Figure 2) were performed using an INSTRON tester equipped with a load cell of 30 kN at a cross-head speed of 2 mm/min and digital image stereo correlation (DISC or 3D-DIC) was used to determine in and out of displacements and strain maps on deformed surfaces [12]. For this purpose, the samples were painted with aerosol white and black spray paint on one face to create a speckle pattern with adequate contrast. A region of interest (ROI) was defined on the specimen surface. The correlation algorithm compared gray level of each pixel in subset defined in the region of interest (ROI). Once the matching was done, the displacement components of the center of the reference and deformed subset can be determined. In this study, strain computation was carried out by Vic3D[®] software developed by Correlated Solutions[®].

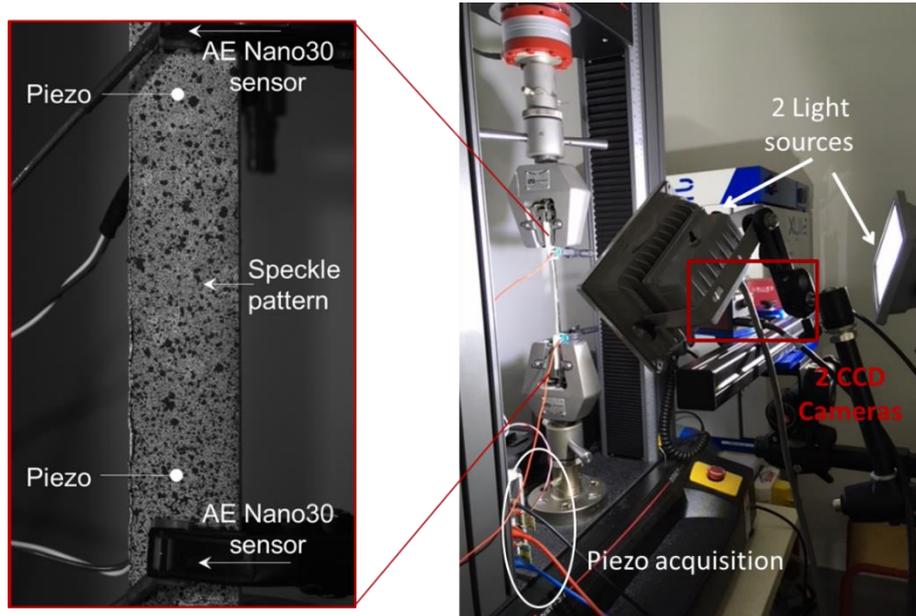


Figure 2: (a) Position of the PZT piezoelectric and AE sensors and the speckle pattern; (b) Tensile test Configuration

In order to better analyse piezoelectric insert response, conventional acoustic emission (AE) signals were monitored during tests using Mistras 2001 system from Physical Acoustics Corporation (PAC). The AE sensor (Nano30) were attached to the sample by silicon grease and a mechanical specific device. The detection threshold of AE was fixed at 35dB.

4 RESULTS AND DISCUSSION

4.1. *In situ* curing monitoring

Once the impregnation process was completed, the impedance was monitored at 4 kHz every 10 min at room temperature (23°C). A comparison of normalized change in impedance vs. time obtained on two different plates manufactured two weeks apart, shows the high reproducibility that was achieved (Figure 3).

The variation of impedance versus time had a sigmoidal shape. At the very early beginning, a slight increase in impedance appeared. Beyond this lag-time, the impedance increased sharply, then levelled off until it reached a quasi-asymptotic value. At the very beginning, the thermoset mixture was composed of low-molecular pre-polymers. As the reaction proceeded, chain extension occurred to produce relatively high molecular weight reaction products resulting in an increase in impedance. Gelation which corresponded to the first appearance of an infinite cross-linked network may be associated to the time at which the first significant increase in stiffness. As mentioned previously, vitrification was associated with the transition from a rubbery modulus to a glassy modulus. Above vitrification, the mobility of the reacting groups was restricted with the reduction of free volume. Thereby, this phenomenon led to an extremely slow reaction, as the reaction became diffusion-controlled. Therefore, vitrification was associated to the instant at which the impedance reached an almost constant value. Beyond, the impedance continued to increase slowly long after the vitrification: the reaction dramatically slowed, since the cure mechanism was controlled by the molecular diffusivity, but did not come to end.

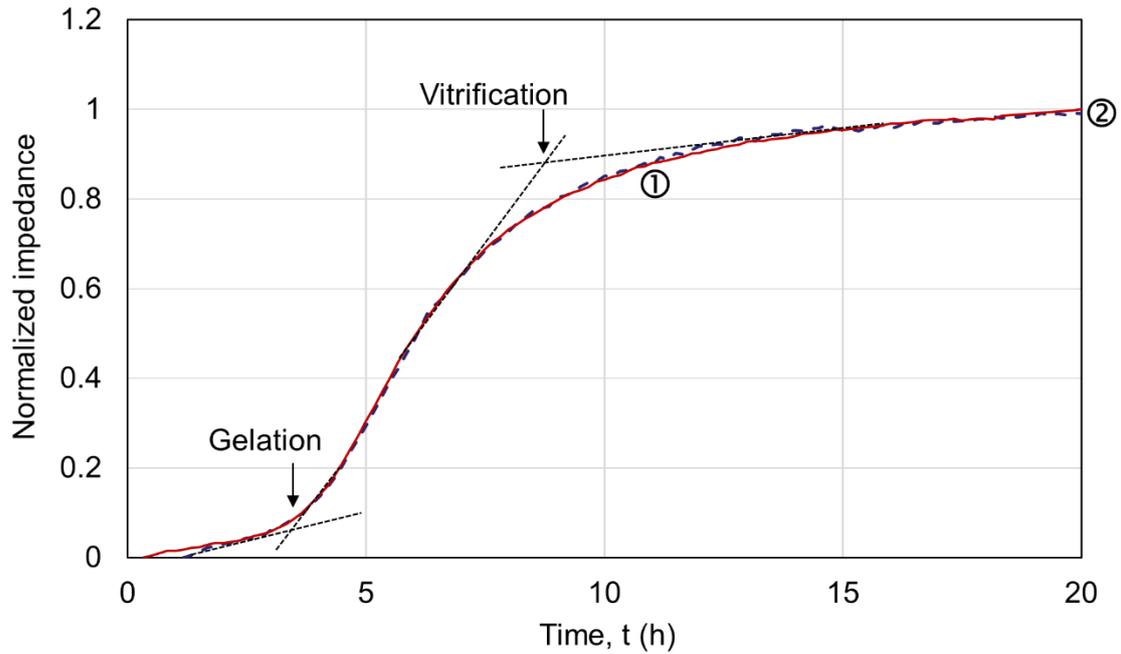


Figure 3: Normalized impedance vs time during cure at RT of two different plates (arrows indicate gelation and vitrification)

The influence of the curing time on the progress of polymerization at ambient conditions (23°C, 50% HR) was also investigated by DMA analysis, on samples cut in transverse directions with respect to the fibre direction in a plate after curing at various cure times (12 hours (sample 1), 2 (sample 2), 8 (sample 3) days).

Typical plots of the loss factor $\tan \delta$ and the storage modulus E' versus temperature were given in Figure 4a and 4b respectively. In the range of temperature studied, the $\tan \delta$ spectra showed two peaks whatever the sample. The highest temperature peak is concurrent in a large drop in the storage modulus (near 2 decades); this relaxation, named α -relaxation is associated with the glass transition and corresponded to the coordinated motion of relatively long chain segments by debonding of low energy bonds (hydrogen bonds). It is well known that the measurement of α -relaxation temperature which is associated with T_g can be used as an accurate indication of cross-link density of a thermosetting. The small process occurring just below the α -relaxation, so called ω -relaxation was attributed to different mechanisms: the presence of water in the material, physical aging [13], and to unreacted chain segments in the network [14].

As shown in Figure 4, the onset of the storage modulus drops and the maximum of $\tan \delta$ associated with α -relaxation were shifted towards high temperatures and the area under the curve of loss factor was reduced when the curing time increases from 12h (sample 1) to 24h (sample 2), and the magnitude of the ω -relaxation decreased. These results were consistent with a progressive decrease in chain motion due to a degree of crosslink increase during curing. Conversely, the α -relaxation temperature is similar for the samples cured during 2 (sample 2) or 8 (sample 3) days. But, it is interesting to note that from sample 2 to 3, the height of the $\tan \delta$ peak associated with the glass transition decreased slightly, and the intensity of

the ω -relaxation dropped sharply. These observations show that the crosslinking density can continue to increase slightly long after the vitrification.

The joint analysis of the impedance (Figure 3) and DMA (Figure 4) results shows that the in-situ measurement of impedance of PZT insert embedded in a composite provides a powerful tool for monitoring the changes in physical properties of thermoset during cure.

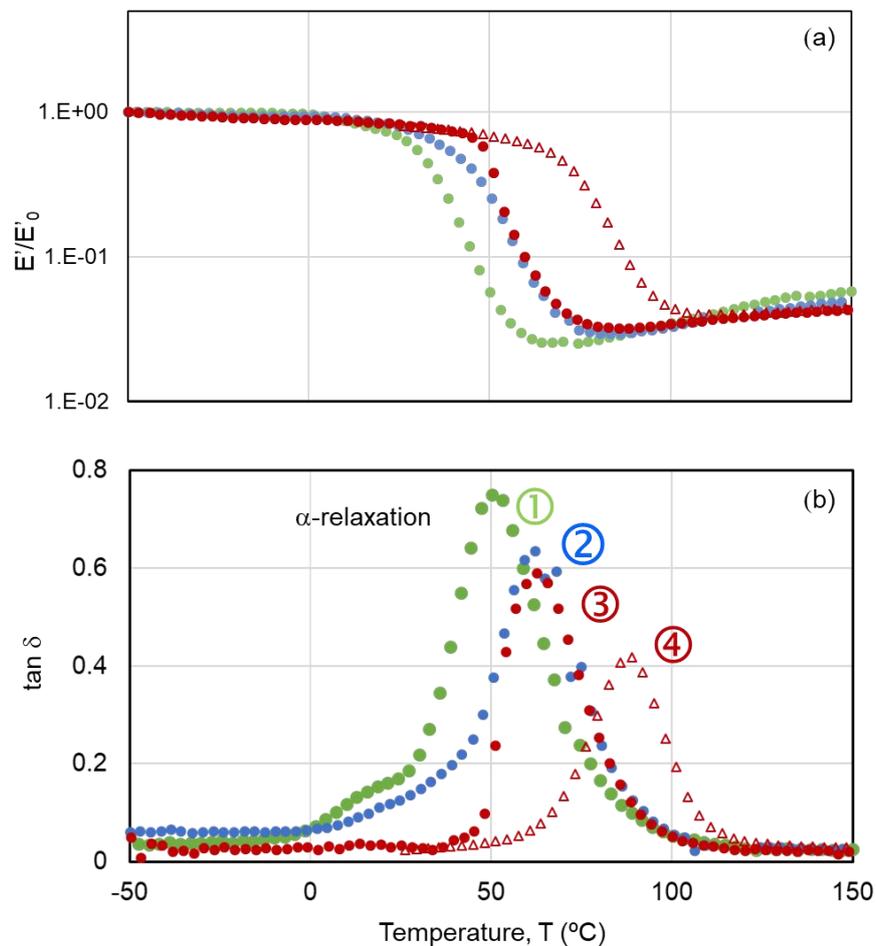


Figure 4: Temperature dependence of (a) $\tan \delta$ and (b) E'/E_0' response at $F=1$ Hz for flax reinforced composites at various curing states: (●) 12 hours; (●) 2 days; (●) 8 days; (△) 8 days + sweep until 150°C

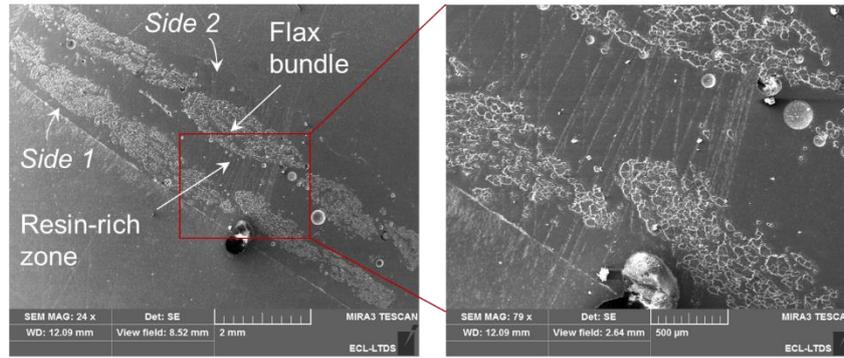
4.2. Damage monitoring

At the end of the curing process, the sensors remain in the part and could be used as damage or aging sensors during all the life of the composite patches. The monitoring of the structural integrity of the repairing patches is of particular interest. Indeed, due to their manufacturing process (in situ hand lay-up, cold-curing resin), [as previously mentioned](#), it is difficult to obtain very high quality composites:

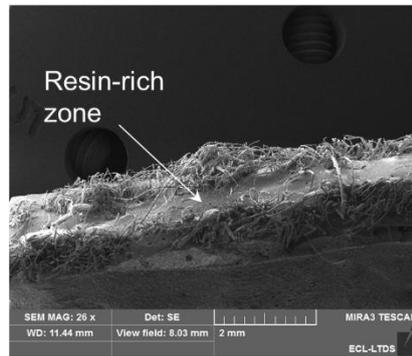
- The curing conditions lead to a slight incomplete matrix cure. Indeed, as can be observed from Figure 4, the α -relaxation of the epoxy composite, after 8 days at room conditions, was about (61°C), and increases by about 25°C after a second DMA scan

(sample 4). Moreover, a slight and continuous increase in the rubbery modulus is observed above the α -relaxation temperature for samples cured at RT (samples 1 to 3). This also confirms that the curing at RT led to a partially cured network, and underwent a post-curing during DMA test. Indeed, it is well known that the rubbery modulus of networked structures is inversely proportional to the average mass of the segments between crosslinking nodes (concept of rubber elasticity [15]) which decreases with the degree of cure, up to the completion of curing. When exposed to a temperature higher than T_g , residual reactions occurred as the mobility of the residual reactive epoxy and amine groups is increased.

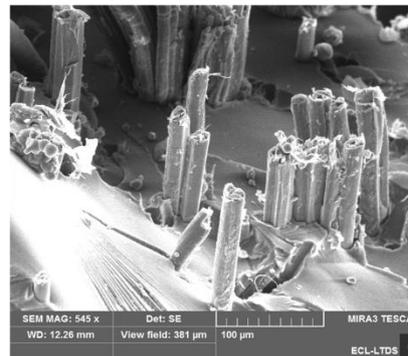
- SEM micrographs of a polished cross-section observed perpendicular to the axis of fibres (Figure 5a) and of a post-mortem failure feature after tensile test (Figure 5b) of the flax reinforced bio-epoxy composite, show different defects: voids due to air trapped, large resin-rich zone between the two reinforcement layers and around the bundles. Figure 5c shows a magnified view of the fracture feature. In this figure, it can be observed, even if the fibres were not treated, that bonding between flax fibers and matrix was strong enough. Indeed, the process of failure mainly involved cohesive phenomena with fracture of the surrounding matrix shown by the presence of matrix debris on the fibres. Probably, the adhesion is associated with two main processes: (i) fibre surface roughness resulting in mechanical anchoring, (ii) hydrogen bondings between hydroxyl (OH) groups in the backbone chain of the epoxy resin and the flax fibres, which also contain OH groups (cellulose) [16].



(a)



(b)



(c)

Figure 5: (a) Polished cross-section of flax reinforced epoxy composite; (b) and (c) Post-mortem scanning electron micrograph after tensile test up to failure

These defects may be responsible for different damages and aging processes which could affect the behavior of the final patches during service. In the aim to evaluate the capability of the embedded sensors for detecting and assessing damage in flax reinforced epoxy composite under load, tensile tests were performed. The results from the PZT sensors were compared to conventional acoustic emission (AE), force sensor (unfiltered) results and digital image correlation analysis (VIC3D).

Figures 6a to 6c show the results obtained on one specimen. Unfiltered Force, sensor voltage, and cumulative AE hits vs. time (or displacement) are given in Figure 6a. Figure 6b gives the amplitude of the AE signals according to time. The strain distribution observed in the region of interest (ROI) for different times t_1 , t_2 and t_3 is shown in Figure 6c.

The most relevant contribution of the piezo-sensor was at the early stage of the damage evolution. Indeed, as visible in Figure 6a, the voltage exhibits a sharper and stronger evolution than the acoustic counting. At a low level of stress, it is more sensitive than acoustic sensors bonded on the surface of the sample. A correlation between sensor voltage measurement and the other results can be exhibited in a specific phase of the damage evolution (framed area in Figures 6a and 6b). On both the force and the cumulative acoustic counting records, there is a change in the intensity of the damage processes. This change is perceived by a change on the slope of the cumulative acoustic counting and the occurrence of disturbances on the uniform increase of the force. The measurement of the force is unfiltered on purpose.

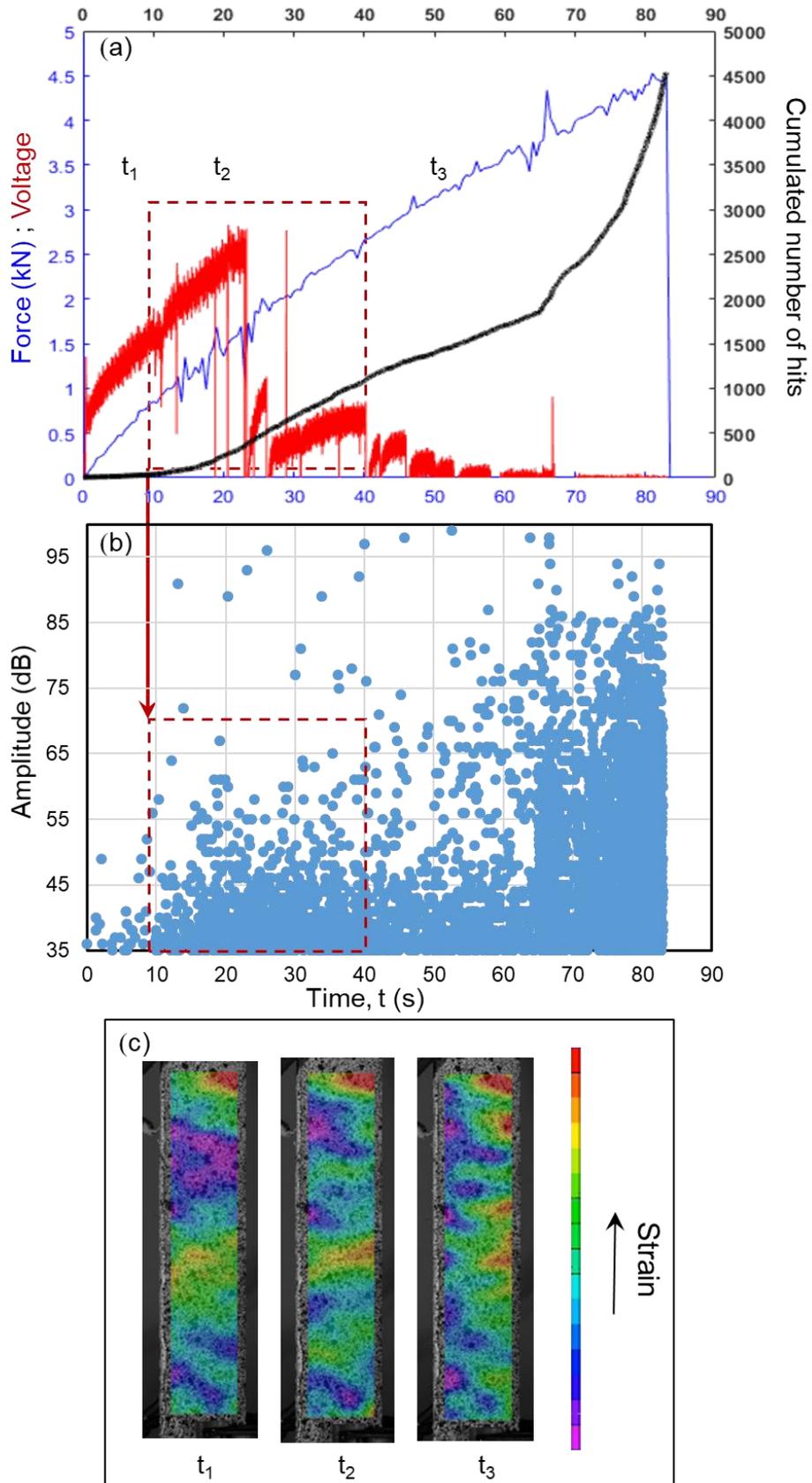


Figure 6: (a) Unfiltered Force, sensor voltage, and cumulative AE hits vs. time; (b) Amplitude of the AE signals vs. time; (c) Strain distribution at different times

However, it seems that the piezo-sensor is inoperative during the last phase of the testing. Yet, the setting of the sensor, inside the composite material, is the key element to explain such behavior. Indeed, the piezo sensor is placed inside the matrix-rich zone between the two fibre layers (Figure 5). Hence, it is only sensitive to events occurring in this zone. Whether the matrix is broken or is debonding from the plies, a vibrational signal is recorded. However, once the separation between the ply and the matrix is locally initiated, the sensor is less loaded. This loss of efficiency of the sensor is a sharp and preliminary signal that the material is entering a critical state, not yet breaking, but prone to do so in the short term.

Still, it is coherent with the acoustic emission and DIC results which show damages associated with matrix breakages and delamination in the same time-range:

- AE signals are detected very early and until the specimen fails (Figure 6b). In the first phase, up to 16% of the maximum force (Figure 6a), events of small amplitude (between 35–50 dB) occur. These events are often related to matrix cracking [17]. In the second phase, events of low to average amplitude are measured. These aspects combined could indicate, in addition of resin failures, friction due to matrix–matrix debonding (delamination), microcracking coalescence (50 dB to 60dB) and matrix/fiber debonding (60 dB-70 dB). The third phase is when a failure occurs: multiple high amplitude events indicate fiber/matrix friction associated with pull-out (70 dB-85 dB) and multiple fibre breakages (>85 dB).
- As shown Figure 6c, at the very beginning (t1), the axial strain distribution is layer distributed in the height of the specimen, associated with local morphological differences (fibre distribution, matrix layer thickness, thickness) along the specimen axis due to the manual manufacturing method. This distribution tends to change in the second phase (t2), and the strain distribution is no longer axisymmetrical in the last phase (t3) which may be related to a large extent of the damage especially on one side of the specimen.

The fact that acoustic emission is able to give information up the failure of the sample is related to the fact that its sensors are directly in contact with the fabric layer, on the surface of the sample. This ensures a permanent transmission of the breaks vibration signal, throughout the different components of the layer.

4 CONCLUSION

A novel PZT ceramic/flax-reinforced epoxy resin composite was fabricated by a hand lay-up manufacturing method in the purpose to be used as smart repairing patches for civil engineering structures.

The curing behavior of this system was investigated by means piezoelectric impedance. The variation tendency in the impedance spectrum is in accordance with the transitions of matrix

viscoelastic properties as cure progressed. The results showed that the piezoelectric transducers are well suited to in-situ monitor the reaction progress during isothermal curing of flax reinforced epoxy materials. Tests on moisture effect on natural fibre reinforced epoxy are in progress.

Besides, after curing, the sensor may be further applied as structural health monitoring (SHM) sensor for the composite material. In order to assess the efficiency of such a system for health monitoring, tensile tests were performed with conventional AE and optical deformation system associated with scanning electron microscope analysis. The results show that sensor is a sharp and preliminary signal that the material is entering a critical state, not yet breaking, but prone to do so in the short term.

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REFERENCES

- [1] A. A. Skordos, P. I. Karkanas, I. K. Partridge, A dielectric sensor for measuring flow in resin transfer moulding, *Meas. Sci. Technol.*, 11 (2000), 25-31. <http://dx.doi.org/10.1088/0957-0233/11/1/304>.
- [2] E. Chailleux, M. Salvia, N. Jaffrezic-Renault, Y. Jayet, A Mazzouz, G. Seytre, In situ multidetection cure monitoring of an epoxy-amine system, *J. Adv. Sci.*, 12 (2000), 291-297. <http://doi.org/10.2978/jsas.12.291>.
- [3] E. Chailleux, M. Salvia, N. Jaffrezic-Renault, V. Matejec, I. Kasik, In situ study of the epoxy cure process using a fiber optic sensor, *Smart Mater Struct.*, 10 (2001), 1-9. <https://doi.org/10.1088/0964-1726/10/2/304>.
- [4] U. Sampath, H. Kim, D. G. Kim, Y. C. Kim, M. Song, In-Situ Cure Monitoring of Wind Turbine Blades by Using Fiber Bragg Grating Sensors and Fresnel Reflection Measurement, *Sensors*, 15 (2015), 18229-18238. <https://doi.org/10.3390/s150818229>.
- [5] M. Scheerer, Z. Simon, M. Marischler, B. Rittenschober, A Multifunctional Piezo and Temperature Sensor for Process and Structural Health Monitoring of CFRP Structures Made by Resin Transfer Molding, In: *International Workshop on Structural Health Monitoring Volume 1*, Stanford, 2017. doi: 10.12783/shm2017/14155.
- [6] K. T. Lau, P. Y. Hung, M. H. Zhu, D. Hui, Properties of natural fibre composites for structural engineering applications, *Compos. Part B-Eng.*, 136 (2018), 222-233. <https://doi.org/10.1016/j.compositesb.2017.10.038>.
- [7] A. Lefeuvre, A. Bourmaud, C. Morvan, C. Baley, Elementary flax fibre tensile properties: Correlation between stress–strain behaviour and fibre composition, *Ind. Crops Prod.* 52 (2014) 762– 769. <https://doi.org/10.1016/j.indcrop.2013.11.043>.
- [8] C. Billotte, F. M. Bernard, E. Ruiz, Chemical shrinkage and thermomechanical characterization of an epoxy resin during cure by a novel in situ measurement method, *Eur. Polym. J.*, 49 (2013), 3548-3560. <https://doi.org/10.1016/j.eurpolymj.2013.07.013>.

- [9] J. Enns, J. Gillham, Time-temperature-transformation (TTT) cure diagram: modeling the cure behaviour of thermoset, *J. Appl. Polym. Sci.*, 28 (1983), 2567-2591 <https://doi.org/10.1002/app.1983.070280810>.
- [10] M. Mehdikhani, L. Gorbatikh, I. Verpoest, S. V Lomov, Voids in fiber-reinforced polymer composites: A review on their formation, characteristics, and effects on mechanical performance, *J. Compos. Mater.* 53 (2019), 1579–1669. <https://doi.org/10.1177/0021998318772152>.
- [11] J. D. Ferry, *Viscoelastic Properties of Polymers*, 3rd Ed, Wiley, New York, 1980.
- [12] Camille Flament, Michelle Salvia, Bruno Berthel, Gérard Crosland, Local strain and damage measurements on a composite with digital image correlation and acoustic emission, *J. Compos. Mater.* 50 (2016), 1989-1996. <https://doi.org/10.1177/0021998315597993>.
- [13] D. Colombini, J.J. Martinez-Vega, G. Merle, Dynamic mechanical investigations of the effects of water sorption and physical ageing on an epoxy resin system, *Polymer* 43 (2002) 4479-4485, [https://doi.org/10.1016/S0032-3861\(02\)00272-0](https://doi.org/10.1016/S0032-3861(02)00272-0).
- [14] J. P. Foreman, D. Porter, S. Behzadi, K.P. Travis. F. R. Jones, Thermodynamic and mechanical properties of amine-cured epoxy resins using group interaction modelling, *J Mater Sci.* 41 (2006), 6631-6638. <https://doi.org/10.1007/s10853-006-0202-9>.
- [15] J.P. Pascault, H. Sautereau, J. Verdu, R.J.J. Williams, *Thermosetting Polymers*, 1st edition, Marcel Dekker, New York, 2002.
- [16] [16] A. K. Mohanty, M. Misra, L. T. Drzal, *Natural Fibers, Biopolymers, and Biocomposites*, Boca Raton, FL: Taylor & Francis/CRC Press, 2005.
- [17] S. Barre, M. L. Benzeggagh, On the use of acoustic emission to investigate damage mechanisms in glass fibre-reinforced polypropylene, *Compos Sci Technol*, 52 (1994), 369-376. [https://doi.org/10.1016/0266-3538\(94\)90171-6](https://doi.org/10.1016/0266-3538(94)90171-6).