

# EMBEDDED SENSING MICRO-COMPONENTS FOR FIBRE REINFORCED COMPOSITE MATERIAL SYNTHESIS AND MONITORING

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## 1. Introduction

The failure of materials involved in large infrastructures, such as bridges or roads, may cause a huge loss of life, economy or a loss of services. An attractive solution to upgrade existing infrastructures and increase their life-span is to bond fibre reinforced thermosetting composite patches to strengthen the damaged structure and to prevent failure growth. And in doing so, understanding the properties as well as failure mechanism of the bonded thermosetting composites is essential. Initiations and growths of defects occur during almost all life of the composite up to final failure which occurs by coalescence of previous damages (cumulative damage). Hence, how to detect the latent cracks in materials and how to fix the damaged materials with more cost-effectively ways are research emphasizes.

As a non-destructive inspection method, in-situ Structural Health Monitoring (SHM) implies the use of sensors, such as piezoelectric ceramics (PZT), which are embedded within a structural material and provide real time performance feedback based on the measurement, such as electrical impedance. These sensors can be used to monitor the health state of thermosetting composites, from their curing process to the propagation of microcracks then to the end of their life cycle, which is useful for analyzing the fabrication or failure procedure of materials. By monitoring the change in the impedance spectrum which is linked to the changes of matrix viscoelastic properties as curing progressed, the different steps of the epoxy curing regarding molecular motion, viscosity, crosslinking density and their consequences on the mechanical properties of the material can be understood.

## 2. Experiment and method

### 2.1. Monitoring method with PZT

The monitoring circuit is shown as figure.1, an oscilloscope is applied as a voltage signal source. In

output option, the parameters are set as followingg: sinusoidal, frequency 4kHz (the resonance frequency of PZT), 2.5V offset. An electric resistance with fixed value (6.8M $\Omega$  or other values in the order of mega ohms, which is in similar to the order of magnitude of impedance of PZT, in order to minimize the measuring error) and a PZT ceramic disk are connected in series with the source. A data acquisition system (DAQ) is connected to the circuit, detecting the electric tension of the source and the electric resistance mainly by a differential operational amplifier, and then the data is sent to a laptop. According to the tension relation in the series circuit, the following equation can be given:

$$\frac{U_0 - U_R}{U_R} = \frac{Z_P}{R}$$

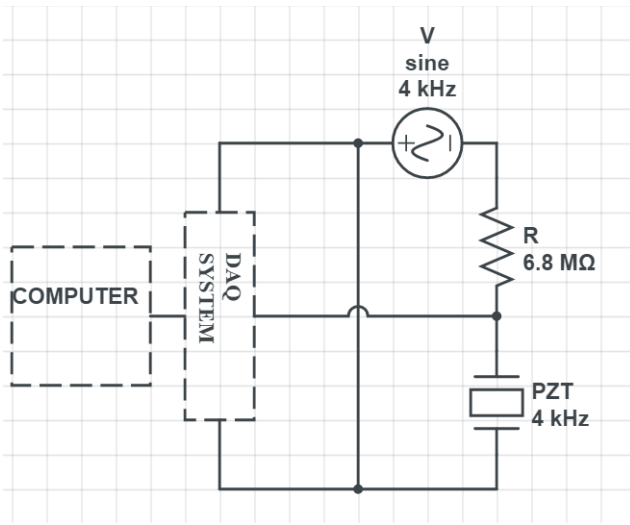
where  $U_0$  represents the total electric tension applied to the circuit,  $U_R$  represents the electric tension applied on the piezoelectric ceramic,  $R$  is the electric resistance value,  $Z_P$  is the impedance of PZT concerned in this monitoring. The expression of impedance  $Z_P$  can be written as following:

$$Z_P = A \cdot R$$

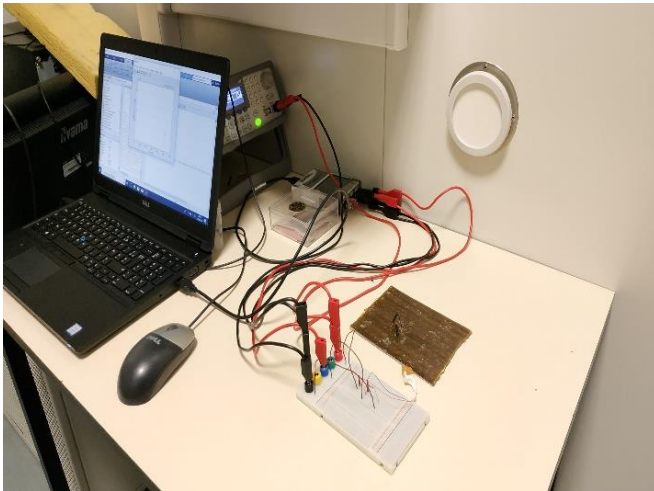
where

$$A = \frac{U_0 - U_R}{U_R}$$

A series of programs based on MATLAB are running with the monitoring process. Variables such as total monitoring time and sampling frequency should be set before experiments, and should be adapted to monitoring requirement. For long-time monitoring such as epoxy resin curing monitoring or epoxy/flax composite monitoring, the system calculates and records the impedance every minute. But if this system is applied for monitoring the behaviour of material during tensile test, there would be greater sampling frequency.



**Fig. 1.** Circuit diagram of monitoring system



**Fig. 2.** Monitoring electric circuit and equipment

## 2.2. Material preparation

In this work, the resin for fabricating the patch composite is designed for ambient temperature curing. Its prepolymer is a low molecular weight “green” epoxy resin that made from bio-based epichlorohydrin. The hardener used is Cardolite NX5619, a solvent-free, low viscosity phenalkamine curing agent made through the Mannich reaction of cardanol from cashewnuts, formaldehyde, and amines. The natural fiber used is flax, and a quasi-unidirectional fabric made of untwisted rovings was used. The weft and warp ratio is 9/91 and the areal density is 200 g/m<sup>2</sup>. The manufacturing of the natural fibers reinforced epoxy composite was accomplished by the use of wet hand lay-up process (2 plies) at room temperature.

## 2.3. Epoxy resin curing monitoring

Epoxy and its hardener are fully mixed in room temperature with mass ratio of 1.8:1, and stored in a cylindrical container of 50ml, which is show as figure.3. A PZT sensor (0.2 mm thick disk with a diameter of 7 mm) is placed inside the resin right after the mixture. Then the epoxy resin and PZT are transferred to the inside of a constant temperature chamber which is preheated to 40°C. The monitoring is carried on within this temperature during 16 hours. For the epoxy resin curing monitoring, a fixed value electric resistance of 9.68MΩ is used.



**Fig. 3.** Specimen of epoxy resin curing monitoring

## 2.4. Epoxy/flax composite curing monitoring

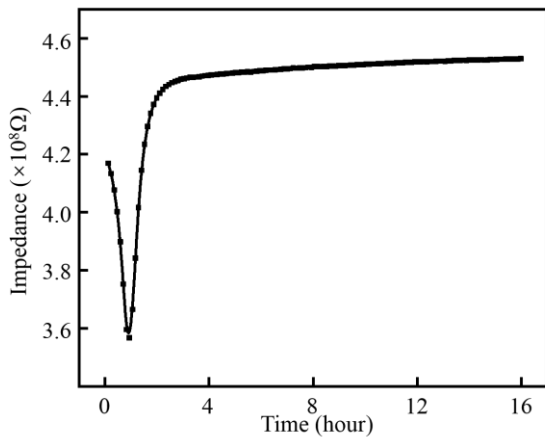
As a consequence of hand lay-up molding process, the proportion of natural fibers in the final composite can not be precisely controlled. In this experiment, composites containing about 14% wt flax fiber are used. A PZT sensor is embedded between the two plies of flax fabric during the manufacturing process at room temperature. Before the experiment, the composite plate has cured for 4 days under ambient temperature, and preheated 1 hour inside the constant temperature chamber. Different from the epoxy resin curing monitoring experiment, a fixed value electric resistance of 1.0MΩ is used, which indicates more intense electric current in the monitoring circuit. The composite plate with embedded sensor is similarly keep inside of the constant temperature chamber under 40°C



**Fig. 4.** Specimen of epoxy/flax composite monitoring

### 2.5. Composite monitoring in water environment

In order to measure the influence of water environment to the composite, another composite plate (manufactured with the same process) with PZT embedded is dipped in deionized water during a week. The change of impedance is recorded by the monitoring system. This experiment is implemented under ambient temperature, which is variable in a day.



**Fig. 5.** Impedance of PZT sensor embedded in the specimen of epoxy resin

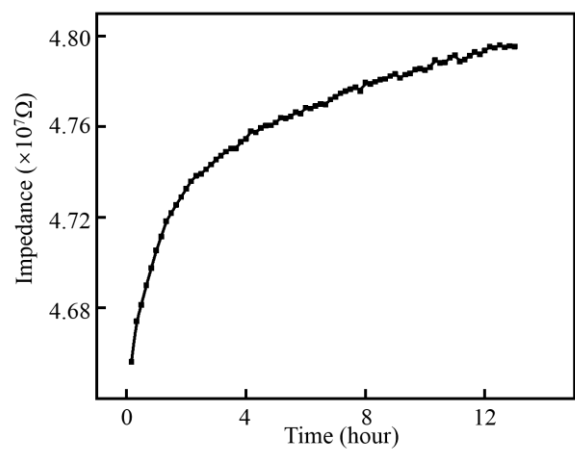
## 3. Results

### 3.1. Epoxy resin curing monitoring

The results of epoxy resin curing are shown in Fig. 5. According to the figure, the impedance of PZT has a rapid decrease in the first hour from the beginning of experiment. Then the impedance increases similarly fast as the decrease period. After about 2 hours, the impedance arrives at a platform,

where only slight but continuous augmentations can be observed.

The decreasing section is considered caused by the change of temperature, which indicates that, comparing to room temperature, the augmentation of temperature surely reduce the viscosity of epoxy resin. The fast increase section indicates an abrupt change of viscosity of epoxy resin, which matches the gelation of polymer. The gel point of epoxy resin should be found before the lower pole of impedance curve, synthesizing the heat-related decrease.



**Fig. 6.** Impedance of PZT sensor embedded in the specimen of epoxy/flax composite

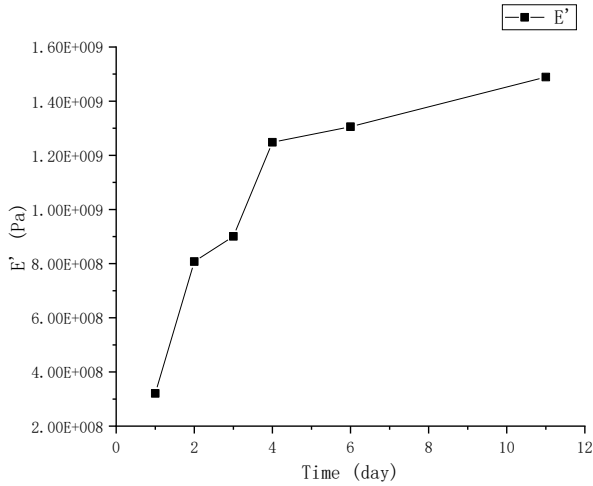
### 3.2. Epoxy/flax composite curing monitoring

The behavior of pre-cured epoxy/flax composite is principally different from that of pure epoxy resin. After the four-day-long curing, the epoxy resin has already passed its gel point for rather long time, the cross-linking reaction has well developed. The epoxy resin has mainly lost its fluidity. The characteristic of composite should be described as viscoelasticity, and storage modulus is used as a reference of curing process.

The increasing rate of impedance during the whole composite curing monitoring is about 0.2% per hour, which is very close to the rate found in epoxy resin monitoring, at the platform section. Although, a relatively faster augmentation is observed at the first hours of monitoring, as a result of higher temperature in monitoring experiment than curing temperature. The curing process is accelerated by heat, as shown in Fig. 6.

Aiming at qualitative analysis of the relation between storage modulus and curing of epoxy, a

series of DMA (Dynamic mechanical analysis) tests were carried on. The composite was tested after 1, 2, 3, 4, 6 and 12 days of curing. The storage modulus under 40°C are shown in Fig. 7. An evident increasing tendency of storage modulus is found, which is associated with polymerization of epoxy.

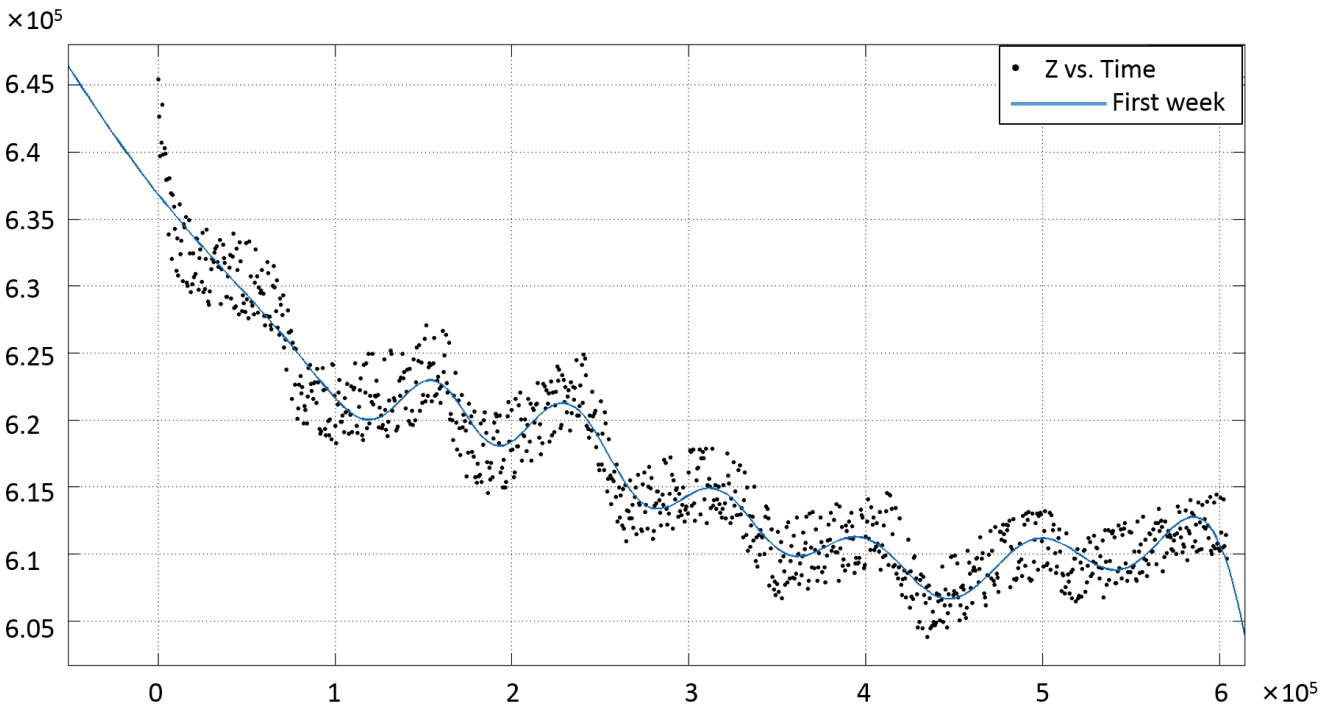


**Fig. 7.** Storage modulus of epoxy/flax composite measured by DMA tests in a series of days

### 3.3. Composite monitoring in water environment

After a week's monitoring, the impedance variation is shown as Fig. 8. A curve of tendency (blue line in Fig. 8) is added by Gaussian fitting which is only utilized to give a clearer view.

According to the figure, the curve is a kind of combination of two parts. One is the decreasing tendency which is driven by water absorption of composite. The hydrophilic groups in cellulose or other molecules allow flax fiber to have relatively high water absorption rate, comparing to synthetic fibers, which causes degradation of mechanical properties of composite. The epoxy resin was still curing, thus a balance appears with saturation of water absorption, and then the curing is more dominant, which explains the final slight augmentation of impedance. The other part is a sinusoidal within an envelope. This part is caused by the variation of room temperature during a day, which can be proved by its period about 8000 seconds



**Fig. 8** Impedance of PZT embedded in epoxy/flax composite steeped in water

#### 4. Conclusion

The change in the impedance spectrum which is linked to the changes of matrix viscoelastic properties as curing progressed was used to follow the different steps of the epoxy matrix curing.

The results showed that the piezoelectric transducers are well suited to in-situ monitor the reaction progress during isothermal curing of a flax reinforced epoxy materials. Other factors being able to influence the mechanical properties of composite can be correlated with the change of impedance and be monitored by PZT sensor. After curing, the sensor was used as damage detector. In order to assess the efficiency of such a system for health monitoring, tests will be performed based on tensile measurements using digital image correlation (DIC), classical acoustic emission and scanning electron microscope (SEM).

#### References

- [1] F. Regina, R. M. William, B. Derrick, A. Purvis, A. Tiwari, and G. D. M. Serugendo "Self-healing and self-repairing technologies". *The International Journal of Advanced Manufacturing Technology*, 69(5-8),1033-1061, 2013.
- [2] G. Park, H. Sohn, C. R. Farrar and D. J. Inman "Overview of piezoelectric impedance-based health monitoring and path forward". *Shock and Vibration Digest*, 35(6), 451-463, 2006.
- [3] D. G. Bekas, K. Tsirka, D. Baltzis and A.S. Paipetis "Self-healing materials: a review of advances in materials, evaluation, characterization and monitoring techniques". *Composites Part B: Engineering*, 87, 92-119, 2016.
- [4] Dae-Hyun Han, Lae-Hyong Kang "Piezoelectric characteristics of PNN-PZT/Epoxy paint sensor according to the poling conditions". *Sensors and Actuators*, A269 (2018), 419-426.
- [5] P. Negi, T. Chakraborty, Naveet Kaur, Suresh Bhalla "Investigations on effectiveness of embedded PZT patches at varying orientations for monitoring concrete hydration using EMI technique". *Construction and Building Materials*, 169 (2018), 489-498.
- [6] Hidemi Mutsuda, Yoshikazu Tanaka, Rupesh Patel, Yasuaki Doi, Yasuo Moriyama, Yuji Uminod "A painting type of flexible piezoelectric device for ocean energy harvesting". *Applied Ocean Research*, 68 (2017), 182-193.
- [7] T. Visalakshi, S. Bhalla, A. Gupta "Monitoring early hydration of reinforced concrete structures using structural parameters identified by piezo sensors via electromechanical impedance technique". *Mechanical Systems and Signal Processing*, 99 (2018), 129-141.
- [8] Gyuhae Park, Hoon Sohn, Charles R. Farrar and Daniel J. Inman "Overview of Piezoelectric Impedance-Based Health Monitoring and Path Forward" *The Shock and Vibration Digest November 2003*, 451-463.
- [9] Ismael Payoa, J.M. Hale "Sensitivity analysis of piezoelectric paint sensors made up of PZT ceramic powder and water-based acrylic polymer" *Sensors and Actuators A*, 168 (2011), 77-89.