

Monitoring in situ in real time of resin infusion for thermoset composite structures

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Abstract. The presented work investigates changes in electrical resistance of embedded sensory yarns as a method to monitor the resin flow front position and curing degree of resin during manufacturing of composite structures by vacuum infusion technology. The sensor concept is based on Piezo-resistive sensors integrated to the flax fabric, having almost identical propriety and dimensions as the flax threads used for the production of reinforcements. In the first time sensors have been characterized and first measures of the resin infusion have been realized in order to demonstrate the feasibility of the proposed approach. Then, the measures in real time were realized with fibrous sensors added to the flax fabric (green composites) to monitor the flow front of resin. A large amount of data recorded, filtered, examined, analysed and processed in order to understand and to optimize the infusion and polymerization process.

1. Introduction

The composites are more and more used in various fields such as aerospace, aeronautics, defense, sport and biomechanics. They are used due to their remarkable mechanical properties such as: density, rigidity mechanical resistances, lightweight, but also lifetime duration, impenetrability, electrical insulation, resistance against shocks [1] etc. A robust and reliable infusion process is vital for the quality of composite parts. Therefore, monitoring of resin flow and cure development in liquid composite molding processes was the subject of extensive researches in the past. A variety of sensor concepts based on thermocouples [2], pressure transducers [3], optical fibers [4-5], and electric resistance sensors [6-7] was investigated. In situ monitoring in real time of the resin diffusion inside the composite is the main objective of this article. The understanding of resin behavior during the infusion process of composite materials in press molding is a major concern, enabling their optimal mechanical properties.

A disadvantage of methods using vision systems or camera is the impossibility to see inside composites materials and the difficulty of adapting this process to industrial production. The use of thermocouples or optical fibres within composites could also damage their structures.

2. Measuring system

In situ monitoring of the resin infusion has been realized using piezo-resistive sensors embedded to composite reinforcement. A protocol has been set up in order to follow the resin movement supposed to modify sensors electrical resistance. To achieve this objective, conductive multi filament yarns (polyamide silver coated) were sewn onto flax fabric used as composite reinforcement, with electrical resistance ranging from 30 to 100 Ω /m. The flax fabric used as reinforcement is twill 2/2 with the reference WD55018113, 13X13 105 DTEX (305 g/m²). The stitching of sensory yarns has been realized on the X axis (same direction as the resin infusion), with a distance of 5 cm between two consecutive sensors. The flax fabric with high damping factor [8] is used in regards the specification of “green composite” with the specific rigidity superior compared to those of glass fibres (Figure 1).



For the electrical resistance measurements, Keithley 3706A was used, able of over 14 000 measurements per second, storing data to the memory with optional high performance resolution. Camera has been used to follow from outside the resin advance.

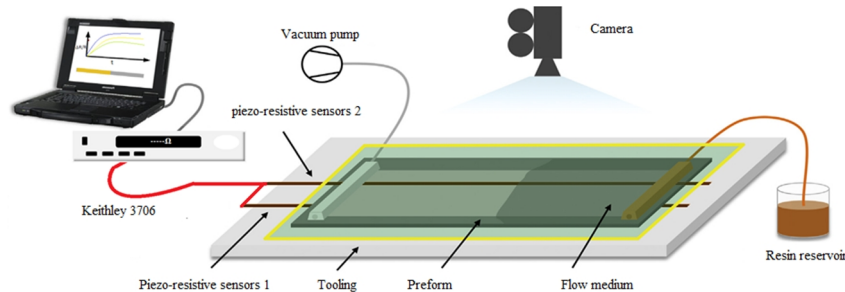


Figure 1. The structure of multilayer flax fabric reinforced composite with embedded sensors in order to measure the resin movement during the infusion phase

Measurements in real time were realized by sensory yarns [9] embedded to the flax based reinforcements of the composite. Methods for production, placement, and calibration of the sensors adapted to our measurement protocol have been developed and data filtering algorithms set up. Moreover, experiments and manipulations have been established in order to characterize and verify the sensitivity, the rapidity, the response time and repeatability of measures of our sensors.

2.1. Result and Discussion

The computation of the relative resistance variations is realized using the following expression:

$$R_{rel} = \frac{\Delta R}{R} \quad (1)$$

First experiment of resin flow monitoring has been conducted in our lab in order to verify the feasibility of the concept and to test fibrous sensors embedded to the flax fabric in the direction of the resin advance. Figure 2 shows the outputs of 5 embedded sensors. It could be considered that the feasibility has been proven and that the selected polyamide silver coated yarns composed of a number of multifilaments may be used as sensors for detection of resin flow. As the resin used in infusion process is non-conductive, it reduces the conductive surface of the yarn (multifilaments) and implies an increase of its electrical resistance. It is possible to observe in Fig. 2 the increase of the sensory yarns resistances (5 sensors) and to conclude consequently, that the resin has progressively covered all the sensory yarns. The infusion process has been conducted over 10 minutes approximately and during this time the electrical resistance of sensors increased. It may also be observed that the increase in the electrical resistance is faster in the beginning of the infusion process and that it slowed down after approximately 5 minutes. It means that the resin front flow was faster in the beginning of the infusion that has also been observed visually during the experiment and that is quite normal for this kind of processes. It could also be noticed that the fifth sensor (violet colour) generated lower output than other sensors because it has been located close to the border of the flax fabric and the resin flow was delayed at that position comparing to other sensory yarns. A decrease of electrical resistance in the beginning of the infusion process for this sensor may be explained by the change in the pressure of plastic sheets because the resin has arrived close to this sensor, but it has not yet been “coated” by the non-conductive resin.

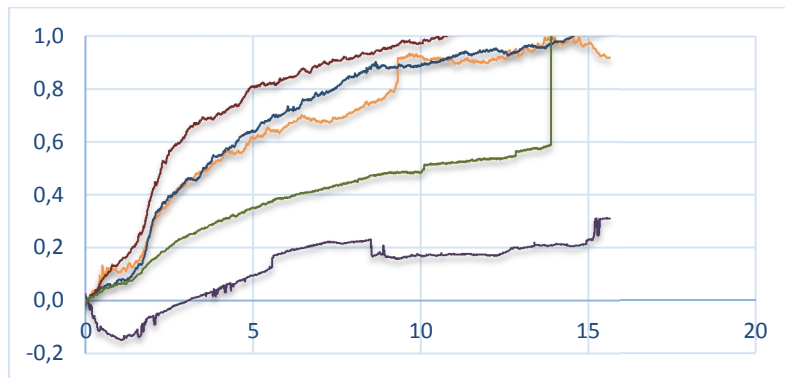


Figure 2. The variation of electrical resistance as function of time

3. Sensors' Characteristics

In this chapter, static and dynamic characteristics of sensory yarns will be presented. Their importance will be highlighted and their influence on the operation of sensing systems will be described.

3.1. Measurement Range

The maximum and minimum values of the measurand that can be measured with a sensing system are called the measurement range, also defined as the dynamic range or span. This range results in a meaningful and accurate output for the sensing system.

All sensing systems are designed to perform over a specified range. Signals outside of this range may be unintelligible, causing unacceptably large inaccuracies, and may even result in irreversible damage to the sensor [10].

3.1.1. Experiment

A protocol has been set up in order to check the measurement range of sensors. The conductive multi filament yarns (polyamide silver coated) were sewn onto flax fabric used as composite reinforcement, with electrical resistance ranging from 30 to 100 Ω /m. The sensor has been located in the middle of the reinforcement. The resin used, is Epoxy SR 8200 mixed with the epoxy hardener SD7201 (37 g of hardener for 100 g of resin). The density $\rho = 1.2$ g/cm³ and the volume part $V_{Resin} = 0.6 V_{Tot}$.

A multimeter was used to measure the electrical resistance before the beginning of infusion process, then the pump was switched on and the infusion started. The process of liquid resin infusion lasted 2 minutes and 23 seconds. The dimensions of the sample were: 20×17cm², and the sensory yarn length: 17 cm. Measures of electrical resistance were realized with the same multimeter at different time intervals. The following table summarizes the evolution of electrical resistance as a function of time.

Table 1: The change of electrical resistance of sensor as a function of time

Time (min)	Electrical resistance R(Ω)	(R _t - R ₀)/R ₀
0	R ₀ = 15.21	0
5	16.71	0.0986
60	17.64	0.1598
1440	17.63	0.1571
2880	18.22	0.1966
4320	18.51	0.2163

3.1.2. Results

According to the results obtained in our tests of infusion phase, it may be deduced that the range of sensor varies between 15.21 Ω (0 mm of resin) and 18.51 Ω (150 mm of polymerized resin).

3.2. Sensitivity

The sensitivity of the sensor is defined as the minimum input of physical parameter that will create a detectable output change. In some sensors, the sensitivity is defined as the input parameter change required producing a standardized output change. In other terms, it is defined as an output change for a given change in input parameter [11].

3.2.1. Experiment

In order to check the sensitivity of our sensors, the following manipulation has been set up:

On a reinforcement made of one-layer flax fabric with dimensions (20×20 cm²), using the sewing machine, four polyamide silver coated conductor yarns have been integrated, with electrical resistance ranging from 30 to 100 Ω/m. A distance of 5 cm between the yarns has been left. The resin used in this process was Epoxy SR 8200 mixed with the epoxy hardener SD7201. For the electrical resistance measurement, ohmmeter was used. 1 ml of resin on each sensor was dropped using a pipette, and the dose was increased progressively (1 ml by 1 ml) to reach the value of 10 ml. A time interval of 90 seconds has been left between the drops, 1 ml each.

Table 2: The evolution of electrical resistance as a function of dose used and covered distance

Dose of resin (ml)	Covered distance of sensors (cm)	Sensory yarn1 R ₀₁ = 13.34	Sensory yarn2 R ₀₂ = 8.51	Sensory yarn 3 R ₀₃ = 14.462	Sensory yarn4 R ₀₄ = 13.32	(R _i -R ₀)/R ₀
		(R _{i1} -R ₀₁)/R ₀₁	(R _{i2} -R ₀₂)/R ₀₂	(R _{i3} -R ₀₃)/R ₀₃	(R _{i4} -R ₀₄)/R ₀₄	
0	0	0	0	0	0	0
1	1.8	0.0022	0.0012	0.0028	0.0015	0.0019
2	3.1	0.0029	0.0047	0.0077	0.0053	0.0052
3	4.6	0.0052	0.0094	0.0055	0.0091	0.0073
4	6.4	0.0090	0.0129	0.0137	0.0143	0.0125
5	8.8	0.0142	0.0247	0.0158	0.0158	0.0176
6	9.7	0.0157	0.0329	0.0184	0.0189	0.0214
7	11.3	0.0187	0.0364	0.0322	0.0211	0.0271
8	12.4	0.0202	0.0529	0.0343	0.0255	0.0332
9	13.5	0.0232	0.0611	0.0389	0.0308	0.0385
10	15.3	0.0262	0.0659	0.0388	0.0421	0.0432

3.2.2. Results

In the previous section, it is mentioned that the sensitivity is defined as the ratio between the output signal and measured property. The following graph represents the change of electrical resistance as a function of time, the dose used and the covered distance by resin on the flax one layer reinforcement.

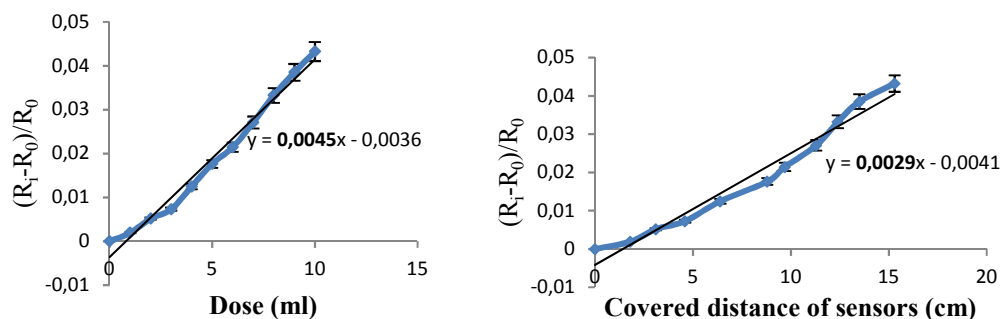


Figure 3: The evolution of electrical resistance as a function of dose used and covered distance Two different sensitivities S_{dose} and S_{covered} could be computed. The values obtained from the plot are: $S_{\text{dose}} = 0.0045 \text{ } \Omega/\text{ml}$, $S_{\text{covered}} = 0.0029 \text{ } \Omega/\text{cm}$, and it may be noticed that they are rather linear as expected.

3.3. Repeatability

This is the ability of a sensor to repeat a measurement when put back in the same environment. It is often directly related to accuracy, but a sensor can be inaccurate, yet be repeatable in making observations [12].

3.3.1. Experiment

Flax fabric twill 2/2 was applied with dimensions of (20×17) cm². Two sensors were integrated to the flax fabric with a distance of 7 cm between the yarns. The objective of this trial is to check that the two sensors give the same responses with regard to the repeatability.

For this experiment two ohmmeters have been used, the process of liquid resin infusion lasted 2 minutes and 30 seconds. The electrical resistance is measured for 30 minutes.

The following table summarizes the evolution of electrical resistance for the first and the second sensory wire as a function of time.

Table 3: The evolution of electrical resistance as a function of time of 1st and 2nd yarn

Time (min)	Sensory yarn 1. $R_{01} = 15.00 \Omega$	$(R_{i1}-R_{01})/R_{01}$	Sensory yarn 2. $R_{02} = 16.02 \Omega$	$(R_{i2}-R_{02})/R_{02}$
0	15	0	16.02	0
5	16	0.0667	16.928	0.0567
10	16.3	0.0867	17.256	0.0771
15	16.3	0.0867	17.33	0.0818
20	16.7	0.1133	17.42	0.0874
25	16.6	0.1067	17.384	0.0851
30	16.6	0.1068	17.358	0.0835

3.3.2. Results

As stated before, repeatability is defined as the range of actual positions the system takes while being repeatedly commanded to the same location under identical conditions (The same dimension and weight of flax fabric, the same the sensory yarn length, the same pumping speed, the same resin and the same temperature of the test room). Even if the LRI (Liquid Resin Infusion) process is not uniform at sensory yarns 1 and 2 location, it is considered that it has been identical. From the data obtained from the first and the second sensors, it may be deduced that the output of two sensors is almost the same.

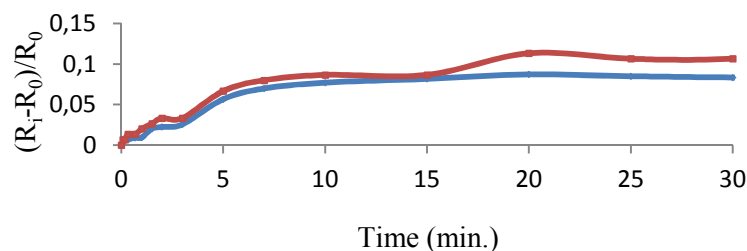


Figure 4: The evolution of electrical resistance as a function of time of 1st and 2nd yarn.

3.4. Response and Recovery Time

Generally, Recovery Time is defined as the time for a sensor to return to baseline value after the step removal of the measured variable. Usually specified as time to fall to 10% of final value after step removal of measured variable. In real-time systems the response time of a task or thread is defined as the time elapsed between the dispatch (time when task is ready to execute) to the time when it finishes its job (one dispatch).

3.4.1. Experiment

As it was impossible to measure the recovery time of our sensors, because it is impossible to remove the resin from composite once it has been infused and polymerized, only a response time has been evaluated. To do that following trial was conducted:

For this manipulation the same flax fabric and sensory yarns has been used with dimensions of $5 \times 17 \text{ cm}^2$. The length of our flax fabric was reduced to 5 cm to ensure that the resin arrives and coats the sensor all over 5 cm in the same time. It means that the step input (Heaviside function) has been applied to three sensors at different times, scheduled by the velocity of the resin flow. In this way three different step responses have been obtained from the infusion process applied on three sensors for statistics purposes.

The stitching has been realized on the X axis (parallel to the direction of the resin infusion in order to generate a step response), and a distance of 1 cm has been set up among the sensory yarns. To observe the change of the electrical resistance three ohmmeters were used.

3.4.2. Results

The time constant relative to the response time if the system could be modelled as a first order, or a second order well damped system, in response to a step input as it is a case for our lateral infusion process.

First order system transfer function was used to model the sensory yarn (Equation 2), the input is defined as $x(t)$, where x is the front resin position and $\frac{\Delta R}{R}(s)$ is the sensory yarn output. If the input is equal to the step input (Heaviside function), the output is presented in Figure 5.

The transfer function identification gives the static gain $K = 0.025$ and the time constant $\tau = 5$ second (simulated step response is given in Fig. 6).

$$\frac{\Delta R}{R}(s) = \frac{K}{1+\tau s} = \frac{0.0}{1+5s} \quad (1^{\text{st}} \text{ order system}) \quad \text{and} \quad \frac{\Delta R}{R}(s) = \frac{K}{\frac{s^2}{\omega_n^2} + \frac{2}{\omega_n} s + 1} = \frac{0.0}{6.2 \frac{s^2}{2+3.5s} + 1} \quad (2^{\text{nd}} \text{ order system})$$

Where $K = 0.025$, proper frequency $\omega_n = 0.4$ and the damping factor $\xi = 0.7$ (see simulated step responses in Fig 6).

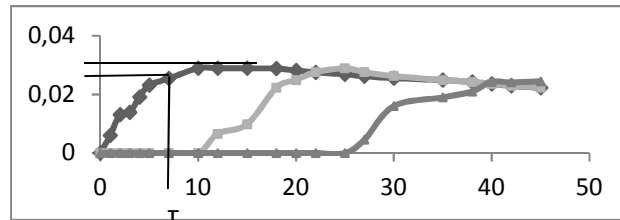


Figure 5. The step response of 3 sensory yarns perpendicular to resin flow advancement

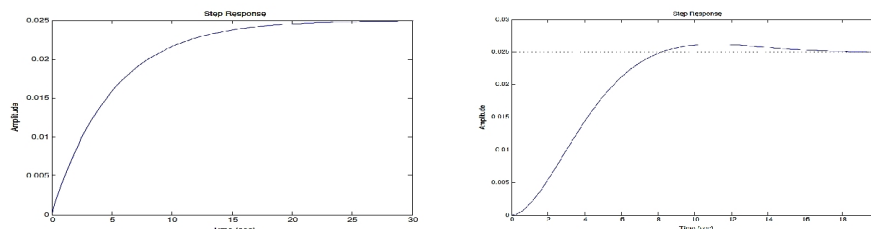


Figure 6. The step responses of the system defined by the equation 2 and 3

References

- [1] Antonucci V, Giordano M, Nicolais L, Calabro A, Cusano A and Cutolo A. 2003 Resin flow monitoring in resin film infusion process. *J Mater Process Tech.* **143**: 687e92
- [2] Di Fratta C, Klunker F and Ermanni P. 2013 A Methodology for flow-front estimation in LCM processes based on pressure sensors. *Compos Part A Appl Sci*; **47**: 1e11
- [3] Danisman M, Tuncol G, Kaynar A and Sozer EM. 2007 Monitoring of resin flow in the resin transfer molding (RTM) process using point-voltage sensors. *Compos Sci Technol*; **67**(3):367e79
- [4] Gupta N and Sundaram R. 2009 Fiber optic sensors for monitoring flow in vacuum enhanced resin infusion technology (VERITy) process. *Compos Part A Appl Sci*; **40**(8):1065e70
- [5] Lekakou C, Cook S, Deng Y, Ang TW and Reed GT. 2006 Optical fibre sensor for monitoring flow and resin curing in composites manufacturing. *Compos Part A Appl Sci*; **37**(6):934e8
- [6] Luthy T and Ermanni P. 2002 Linear direct current sensing system for flow monitoring in liquid composite moulding. *Compos Part A Appl Sci*; **33**(3): 385e97
- [7] Danisman M, Tuncol G, Kaynar A and Sozer EM 2007. Monitoring of resin flow in the resin transfer molding (RTM) process using point-voltage sensors. *Compos Sci Technol*, **67**(3):367e79
- [8] Lutpil L, Bensadoun F and Pariset J. 2015 Why are designer fascinated by flax and hemp fiber composites? *Ignas VERPOEST*
- [9] Trifigny N, Kelly F.M, Cochrane C, Boussu F, Koncar V and Soulat D. 2013 Pedot:PSS-based piezo-resistive sensors applied to reinforcement glass fibres for in-situ measurement during the composite material weaving process, *Sensors* n°8, pp.10749-10764, 203
- [10] Chu-pak L. 2013 The Range of sensors and Algorithms used in rate adaptive cardiac pacing. sensors, algorithms, rate adaptive pacing, *sensor combination*
- [11] Zachary J, Davis D and Anja B. 2005 Aluminum nanocantilevers for high sensitivity mass sensors. *Applied physics letters* 87
- [12] Brett L, Bousek J, Castello P, Salyk O, Harskamp F and Aldea L. 2009 Reliability of commercially available hydrogen sensors for detection of hydrogen at critical concentrations. *part I – testing facility and methodologies*