IN SITU AND QUANTITATIVE CONTROL OF RESIN TRANSFER MOULDING (RTM) USING FIBRES OPTIC SENSORS

X.A. Aduriz[1,2], C. Lupi[1], N. Boyard[2], J.L. Bailleul[2], D. Leduc[1], D. Delaunay[2] and C. Boisrobert[1]

[1] IREENA, Faculté des Sciences et Techniques, 2 rue de la Houssinière F- 44322 Nantes
[2] LTN–UMR 6607 CNRS / Université de Nantes, rue Christian Pauc, BP50609, F-44306 Nantes cedex 3.

ABSTRACT

In this paper, we present an application of fibres optic sensors to the studies of the Resin Transfer Molding process. We present in a first time our experiments devices. Then, two fibres optics sensors are described and their associated exploitation means too. Finally few experimental results are presented and described. So we show that it is possible to measure temperature, cure degree and material change with a simple sensor. With a more complicated one we can also measure curing reaction stress.

1. INTRODUCTION

The RTM process is mostly used in aeronautics and space industries. In this process the reinforcement is placed inside a mould and a low viscosity resin is injected and heated until it polymerizes. The impregnation of the reinforcement and the polymerization step are two key-elements to reach good piece quality. The knowledge of the front position of the thermosetting resin, polymerization degree in the piece and associated stresses are then of crucial interest in the context of the optimization process to avoid dry areas and to limit stresses. The filling step can be monitored by thermocouples or more recently with dielectric sensors or fibre Bragg sensors. Real time measurements of dielectric permittivity or heat flux on industrial parts are already performed to estimate polymerization degree. Finally, thermal and chemical strains are generally estimated using strain sensors. The use of fibre Bragg grating is now in constant development.
To overcome several problems such as sensor intrusion, averaged measurements, destructive experiments or necessity of specific sensor for each monitoring parameter, we report on the control of the RTM processing of composite materials by using fibre optics sensors, simply based on the Fresnel reflection [1]. We apply this optical technology to the monitoring of the injection and polymerization of the resin in an industrial mould. Finally, we characterize local strain distribution using an optical fibre Bragg grating embedded in samples molded in industrial conditions.

2. EXPERIMENTS

In our experiment, we used an experimental mould developed in the laboratory (figure 1) with which we can measure simultaneously 4 thermodynamics variables: pressure (P), volume (V), temperature (T) and cure degree (α), as a function of time.

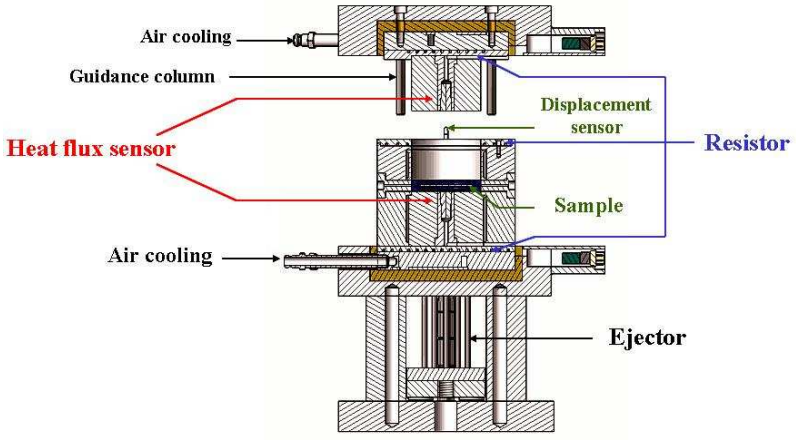

Figure 1: Scheme of the experimental mould.

The sample is introduced in a press which imposes the pressure (P) from 5 bars up to 100 bars. In the same time, we measure the variation of our sample thickness by a displacement sensor, in order to get the variation of the volume. The temperature is increased slowly during our experiments (3°C/min), so that the thermal gradient inside the sample can be negligible. Two non-intrusive heat flux sensors, equipped with 3 micro thermocouples of 25µm diameter, are located on both side of the mould cavity. We use an inverse sequential method [2] to calculate the mould side temperature and the heat flux density from which we derive the cure degree (α) [3].

We used this original mould to test and validate two different fibre-optics sensors introduced in the mould cavity: Fresnel sensor and fibre Bragg grating. The sensor is made of a single mode fibre (standard telecom G 652) and delivers a signal due to the Fresnel reflection at the end of the fibre. We consider a dioptre surface common boundary between two media with different refractive indices n_1 and n_2 (Figure 2). The relations between the angles of the incident, refracted and reflected beams are given by the Descartes-Snell and Fresnel laws [2].

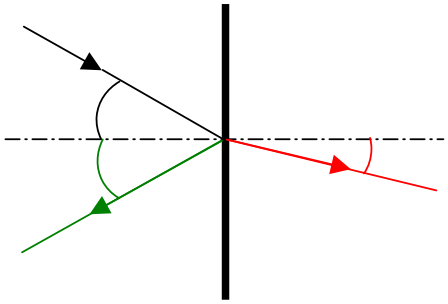

Figure 2 : Fresnel reflection

P_i is the incident radiant power. When the incident beam is normal to the dioptric surface the reflected radiant power is given by:

$$P_r = \left| \frac{n_1 - n_2}{n_1 + n_2} \right| . P_i \qquad (1)$$

The reflected radiant power depends on the indices n_1 and n_2 of the two media. We now consider an optical fibre (figure 3) whose core refractive index is n_{fibre}. The fibre is dipped into a medium whose refractive index is n_{ext}. The radiant power reflected at the fibre end face is then given by:

$$P_r = \left| \frac{n_{fiber} - n_{ext}}{n_{fiber} + n_{ext}} \right| . P_i \qquad (2)$$

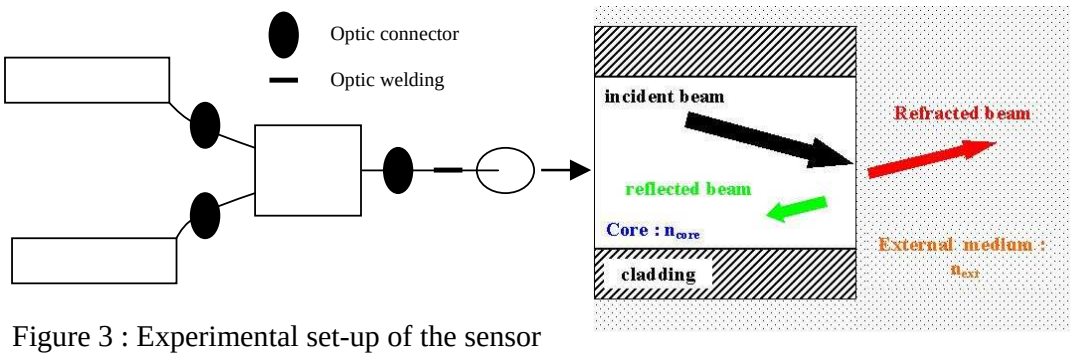

Figure 3 : Experimental set-up of the sensor

The experimental sensor set-up is displayed in figure 2. A 1550 nm laser source is connected to the fibre through a "2 to 1" fibre coupler. 50% of the laser output radiant power propagates along the fibre. The fibre end face is cleaved normal to its axis and reflects part of this incident radiant according to Fresnel law. 50% of this reflected part propagates along the back to the coupler and can then be detected through the coupler on a photo-detector connected to the second input fibre pigtail of the coupler. Previous article [3, 4] demonstrated that the reflected radiant power can be used to obtain the resin refractive index variations with temperature or the polymerisation degree. The resin refractive index can be calculated from the relations:

$$\eta = \frac{n_{fiber} - n_{air}}{n_{fiber} + n_{air}} . \sqrt{\frac{P_{resin}}{P_{air}}}$$

$$n_{resin} = n_{fiber} . \frac{1+\eta}{1-\eta} \qquad (3)$$

where n_{resin}, n_{fibre} and n_{air} are the refractive indices of the resin, the fibre and the air respectively. P_{resin} and P_{air} are respectively the radiant powers (at the photo-detector level) reflected by the resin or the air (external medium).

Equations 2 and 3 show that if the incident radiant power at the fibre end facet and n_{fibre} remain constant in time, it is possible to follow the evolution of the external medium refractive index. Different physical phenomena may cause changes of this refractive index during the RTM process:

1. the resin front edge passes the fibre end face,
2. temperature variations of the resin (leading to refractive index variations),
3. changes of the resin structure (polymerisation).

The fibre we use (standard G 652) has a 8 µm core diameter and an external silica optical cladding diameter of 125 µm. The refractive index is 1.457 with a thermo-

optic coefficient of $dn/dT = 9.2\ 10^{-6}\ °C^{-1}$ [2], which will be taken into account for the fibre refractive index thermal evolution.

In a second time, in order to determine the stress inside the resin, we use a fibre Bragg grading sensor. A fibre Bragg grating is made of a periodic longitudinal modulation of refractive index of the optical fibre (figure 4). It is characterized by 3 functions:

1. the amplitude of modulation $\Delta n_{ac}(Z)$,
2. the mean effective index $\Delta n_{dc}(Z)$,
3. the variation of the period of the modulation $\Lambda(Z)$.

In practice, the influence of the grating is evaluated through its complex coupling coefficient $\Omega(Z)$, which amplitude is proportional to $\Delta n_{ac}(Z)$ and which phase depends on both $\Delta n_{dc}(Z)$ and $\Lambda(Z)$.

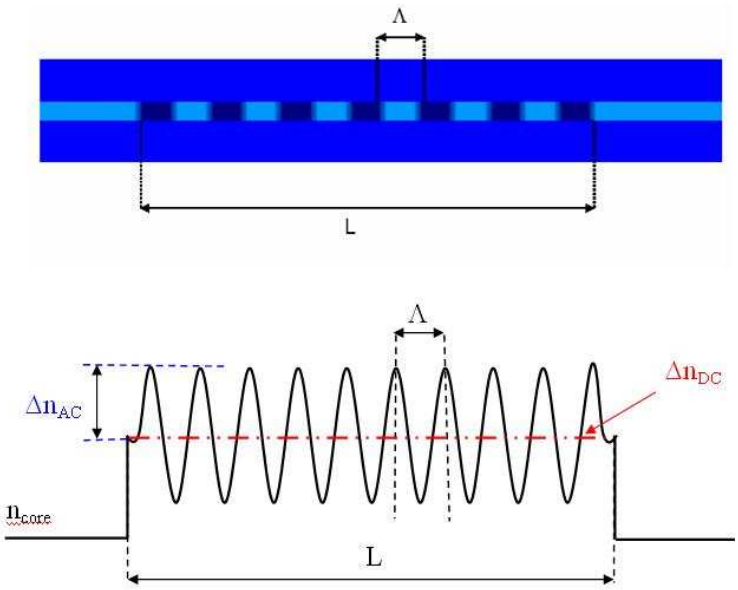

Figure 4: Bragg network representation.

When an external strain is applied to the grating, its length and its refractive index change. Hence, the local strain is encoded in the phase of the complex coupling coefficient of the grating. In order to get local information on the grating sensor, it is necessary to measure the complex reflection coefficient of the grating and then use as Optical Low Coherence Reflectometer to measure the complex reflection coefficient. The set-up used is presented on figure 5.

Our reflectometer is the association of two Michelson-interferometers.

The first one is almost entirely constituted of single mode fibres and uses a broadband source emitting in the wavelength range 1525 – 1575 nm. The incident light is split by a 2 by 2 fibre coupler. One of its outputs is connected to the Bragg sensor and the other is used as a reference arm where the propagating wave is bent on a sliding corner cube reflector and oriented toward a fixed plane mirror. Interferences of the waves propagating along the different arms of the fibre coupler are then obtained.

The second “free space” Michelson interferometer is used as a fringe counter to sample the infrared interferogramme every 80 nm. The complex reflection coefficient of the Bragg grating is computed from Fourier Transform of the interferogramme that it

produced. It is then used as input data of a layer peeling algorithm in order to characterize the amplitude of modulation and the phase of the grating [6].

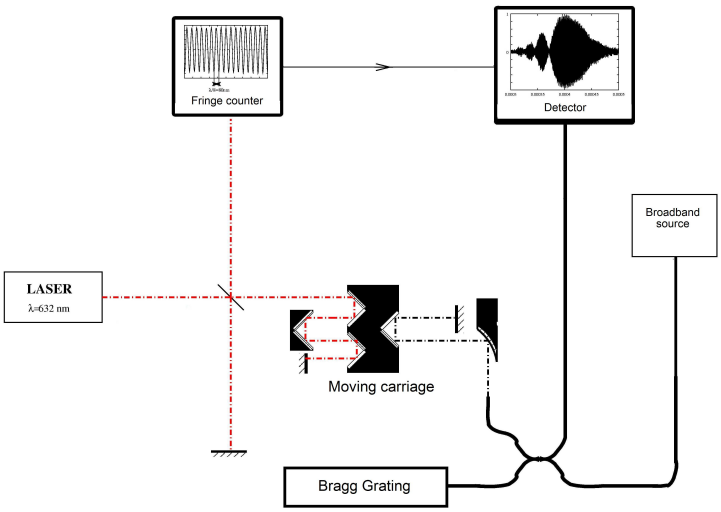

Figure 5: Reflectometer.

3. EXPERIMENTAL RESULTS

A Fresnel sensor was inserted in a thermo set resin sample (RTM6 from EXCEL ©), which was placed in the PVT-α mould. Figure 6 displays the response of the optic fibre according to a given thermal cycle.

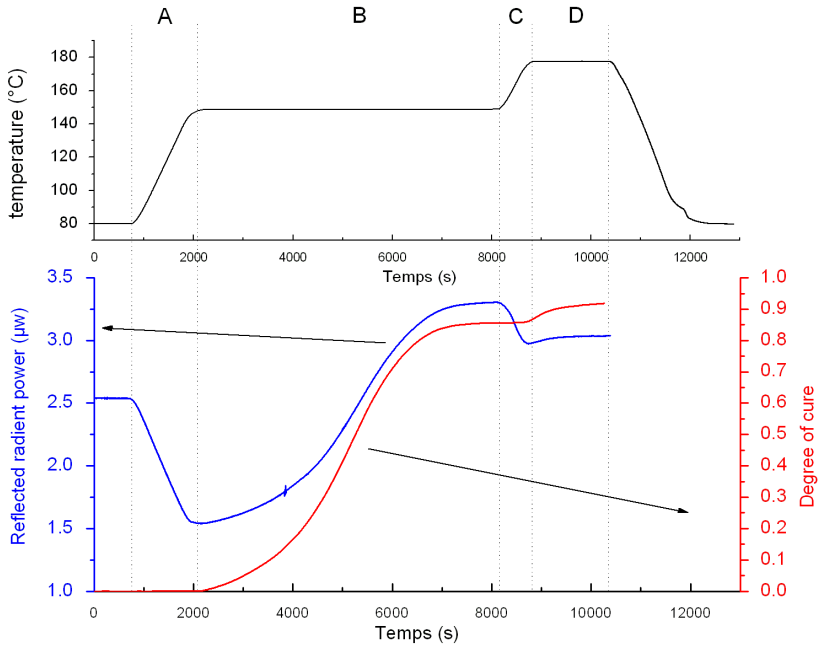

Figure 6: Reflected power measure with a Fresnel sensor.

On this figure, we also presented the cure degree evolution versus time according to the given thermal cycle. So, we can divide the time in five different zones A-B-C-D.

After an isothermal plateau at 80°C, the temperature increases up to 150°C. During this first sequence A, we observe that the cure degree is constant and equal to zero and the reflected radiant power decreases. This power decrease is due to thermal dependence of our resin optical properties. The temperature dependence of the reflected power is close to linear as shown on figure 7. From the relation (1) and measurements, we define the optical index evolution versus temperature. Then the thermo-optical coefficient of the RTM6 resin equal to $-3.58\ 10^{-4}\ °C^{-1}$, for a 1550 nm wavelength and with a relative standard deviation of 9%. The relative standard deviation is obtained for ten different sensors measurements.

During the sequence B, we consider that the temperature is constant and uniform inside our sample because of the small sample thickness. So, the reflected radiant power evolution was only due to the cure degree evolution. The cure degree dependence of the reflected power is also close to linear as shown on figure 8. We define the optical index evolution versus cure degree. An optical cure degree coefficient is define as $dn/d\alpha$ and is equal to $4.53\ 10^{-2}$ (standard deviation = 8%).

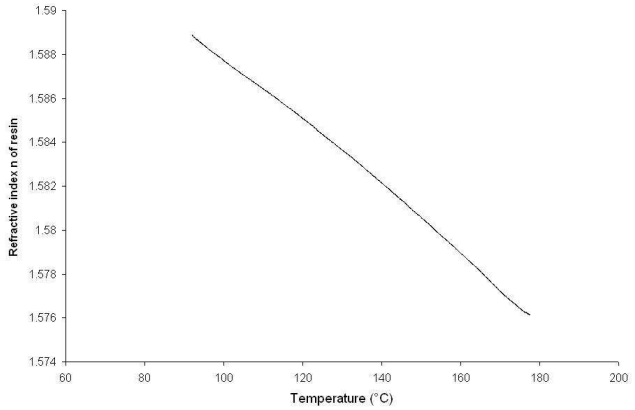

Figure 7: Evolution of the cured resin optic index as a function of temperature

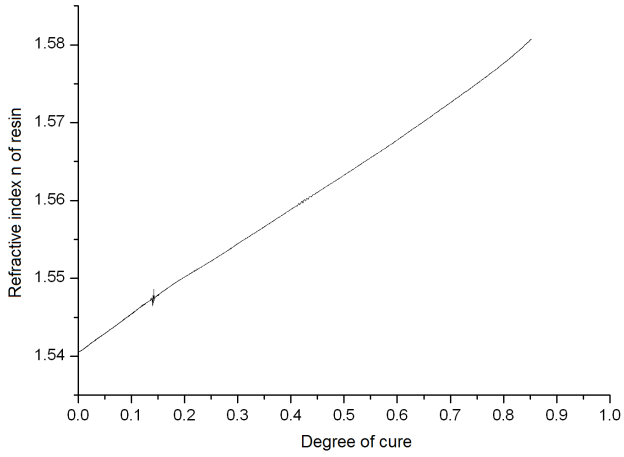

Figure 8: Evolution of the resin optical index versus cure degree.

With these defined optical coefficients, we developed a law to describe the refractive index evolution during curing cycle and we compared measurements and calculations (figure 9).

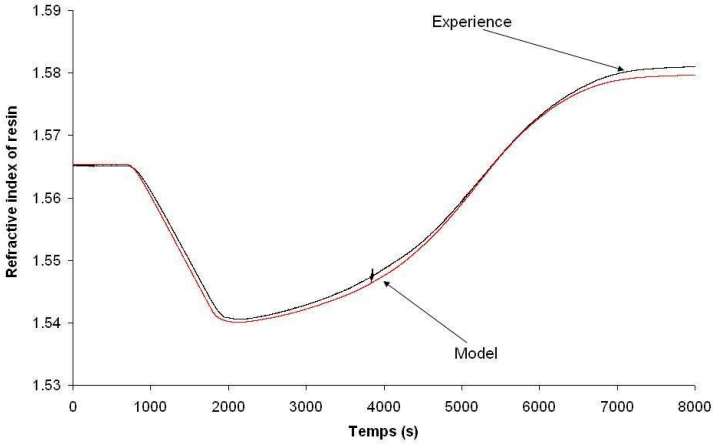

Figure 9: Comparison between experimental and measured resin optic index.

$$n(\alpha, T) = \alpha.\left(1.594 - 3.696.10^{-4}.T\right) + (1-\alpha)\left(1.604 - 1.289.10^{-4}.T\right) \quad (2)$$

After the thermal plateau at 150°C, the temperature increases up to 180°C. During the first time of the sequence C, the measured temperature increases but the cure degree is still constant because of the glass state of our resin. With the optic sensor, we defined the glass transition temperature at 159°C. This result was confirmed with DSC experiment for the same conditions. During the last sequence D, the cure degree increase up to the glass transition at 180°C.

To conclude this part, we showed that an optic Fresnel sensor can be used to measure temperature and cure degree of a resin sample. Moreover, they can be used to detect resin during its injection because of the great difference of optic index between vacuum and resin material. We monitored successively the injection and the polymerization of the resin in industrial conditions. As the response of the sensor exhibited an abrupt variation when its surrounding medium changed, this sensor was also able to detect the resin front inside a mould. Twelve fibre optic sensors thus equipped a steel mould designed for making a rectangular 350 mm × 350 mm × 6 mm piece. The results clearly exhibited a preferential channel along the edge of the mould. In a second time, we recorded the reflected optical power on several sensors to monitor the polymerization of the resin. Using the calibrations results we estimated the final conversion degree of the reaction (T_{cure}=150 °C) to be close to 0.85 (standard deviation = 8 %).

Fibre Bragg sensor have also been used during reticulation process. In the following figures, we present 3 measures at the same temperature (20°C) because of the measurement deviation due to the press. The first one is before curing with raw resin. The second one is after a curing cycle at 150°C, and the last one after a post-curing at 180 °C. On the figure 10 and 11, the mean effective index $\Delta n_{dc}(Z)$ and the optical index modulation amplitude processed from OLCR measurements are presented respectively.

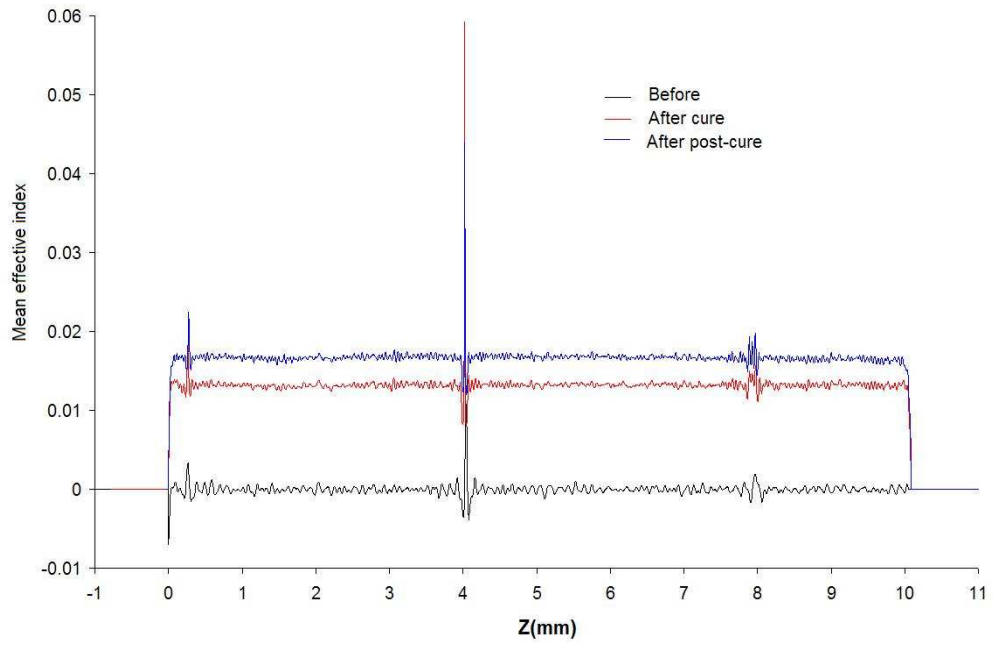

Figure 10: Mean effective index evolutions

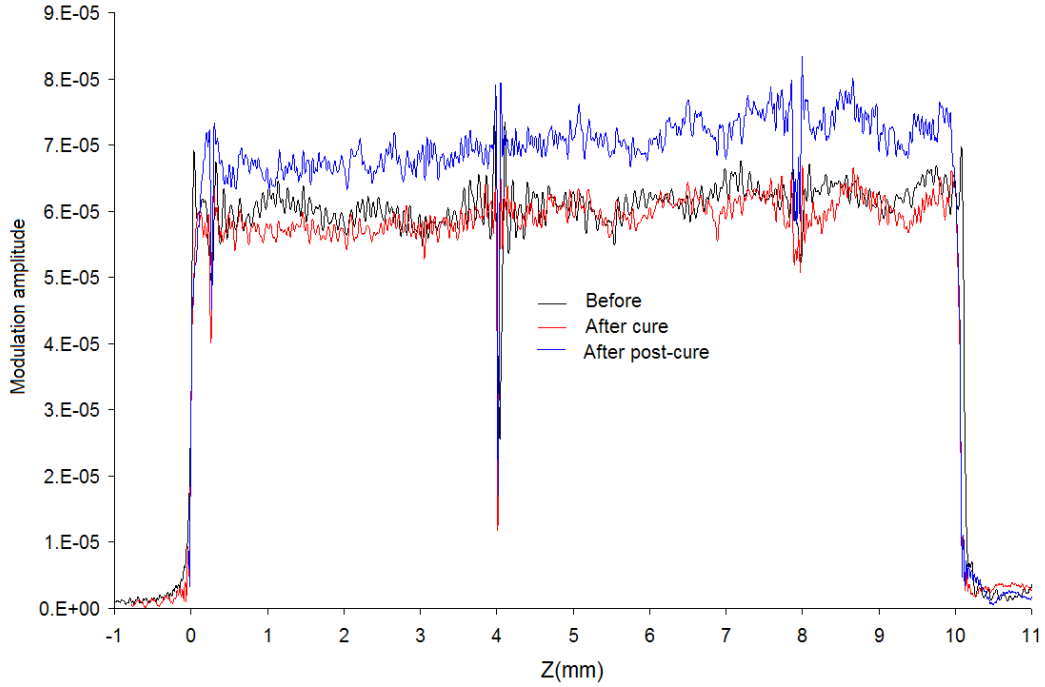

Figure 11: Bragg sensor optical index modulation amplitude after curing and post-curing treatment.

After the first curing, we observe an increase of the mean effective index due to the curing reaction stress. The post curing increase a little more the measurement due to the cure degree evolution between a curing at 150°C and a post curing at 180°C. A homogeneous increase of the amplitude modulation is observed along the sensor after resin post-cure, no significant effect is observed after resin cure on the magnitude of

modulation amplitude. But a decreased of the length of the sensor is determined after the resin cure and did not change after the post curing (figure 12).

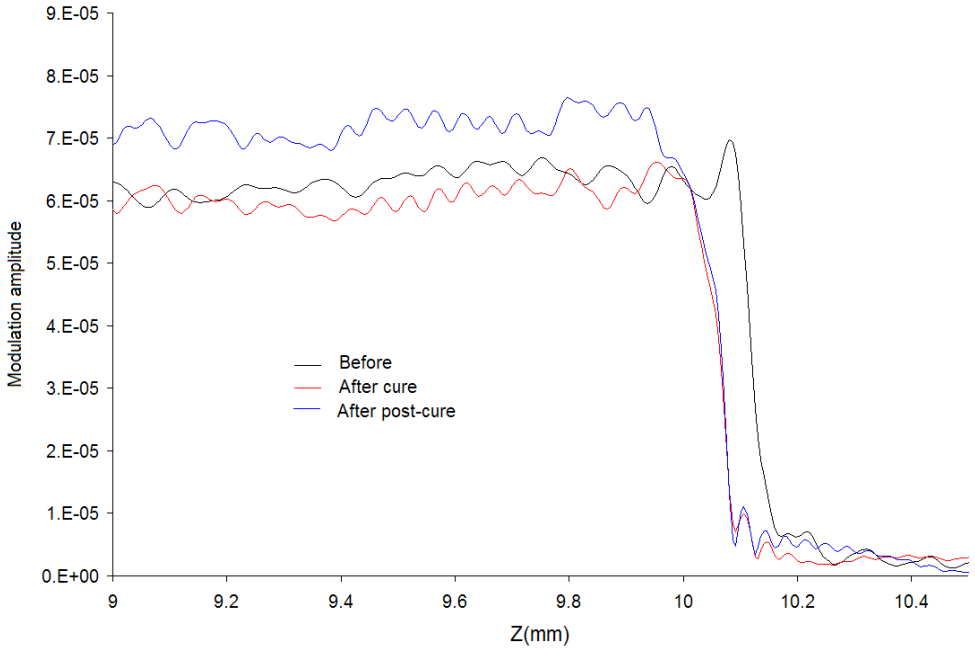

Figure 12: Sensor length modification

4. CONCLUSIONS

In this work, we study a fibre optic sensor to monitor the fabrication of a composite material piece from RTM process. As the response of the sensor exhibits an abrupt variation when its surrounding medium changes, it is also able to detect the resin front inside a mould. Its characterization clearly shows that the response of sensor is linear with T and α. This implies that we can also use this sensor to measure easily the degree of polymerization of the resin inside the mould during curing. We demonstrated that a single and simple optical sensor could bring important information on composite materials processing. Moreover, the probe of Bragg grating sensor with OLCR technique is of great interest to determine strains distribution.

REFERENCES

1. Born M. and Wolf E. *Principles of Optics*. 7th Edition. Cambridge University Press, 1999.
2. Beck, J.V., Blackwell B. and St. Clair Jr. "*Inverse heat conduction : Ill posed problems*.", New-York, John Wileys and Sons, 1985.
3. Pu S., Chen X., Chen Y., Liao W., Chen L., Xia L, « *Measurement of the refractive index of a magnetic fluid by the retroreflection on the fibre-optic end face.* » Applied Physics Letters 86, 171904 (2005).
4. Chang S., Hsu C.-C., Huang T.-H., Chuang W.-C., Tsai Y.-S., Shieeh J.-Y. and Leung C.-Y., "*Heterodyne interferometric measurement of the thermo-optic coefficient of single mode fibre.*", Chinese Journal of Physics, Vol. 38, n° 3-1, pp 437 – 442 (2000).
5. Sobotka Vincent, PhD Thesis, "*Optimisation thermique du procédé resin transfer molding.*", University of Nantes, 2004.

6. Chapeleau X., Casari P., Leduc D., Scudeller Y., Lupi C., Le Ny R., Boisrobert C., "*Determination of strain distribution and temperature gradient profiles from phase measurements of embedded fibre Bragg gratings*". Journal of Optics A: Pure and Applied Optics. Vol.8, No 9, pp 775-781, 2006.
7. Aduriz X.-A., Lupi C, Boyard N., Bailleul J.-L., Leduc D., Sobotka V., Lefevre N., Chapeleau X., Boisrobert C., Delaunay D., "RTM6 epoxy resin optical index determination and real time curing monitoring for a quantitative control of the industrial process.", Composites Sciences and Technology, 67, 2007, pp 3196 – 3201.
8. Botsis J., Humbert L., Colpo F. and Giaccari P. "*Embedded fiber Bragg grating sensor for internal strain measurements in polymeric materials.*", Optics and Lasers Engineering, 43, pp 491-510, 2005.