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FIBRE OPTIC SENSORS APPLIED TO RESIN TRANSFER MOLDING: A POWERFUL WAY FOR IN-SITU QUANTITATIVE CONTROL AND OPTIMIZATION

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SUMMARY: RTM6 epoxy resin curing is usually characterized by the polymerisation degree. We report in this paper on a refractive index measurement technique applied on an experimental mould to control, quantitatively and *in-situ*, the industrial RTM process. The calibration of the optic fibre sensor using a specific mould enables a quantification of the RTM6 epoxy resin polymerisation process. It is also very useful to follow the filling of a mould. The recorded data, coupled to PAM-RTM simulations, can be then used to estimate the permeability along one direction. Finally, it is important to underline that the optoelectronic system is connected to a data processing unit and is easy to use in an industrial environment.

KEYWORDS: fibre optics, sensors, Resin Transfer Molding (RTM), thermosetting resin

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 polymerises. The impregnation of the reinforcement and the polymerisation step are two key-elements to reach good piece quality. The knowledge of the front position of the thermosetting resin and polymerisation degree in the piece and associated stresses are then of crucial interest in the context of the process optimisation 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 grating. Real time measurements of dielectric permittivity or heat flux on industrial parts are already performed to estimate polymerisation degree. Finally, thermal and chemical strains are generally estimated thanks to strain sensors but 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 propose here the use of fibre optics sensors, simply based on the Fresnel's reflection. We report on the control of the processing of composite materials by RTM. We study and describe the use of a fibre optic sensor. It provides important information linked to the process such as the polymerisation degree of the resin. We also apply this technique to the monitoring of the injection and polymerisation of the resin in an industrial mould.

FIBRE OPTIC SENSOR

The sensor is made of a single mode fibre (standard telecom G652) and delivers the Fresnel reflection signal from the cleaved end faced of the fibre. We consider a dioptre surface common boundary between two media with different refractive indices n_{core} and n_{ext} (Fig. 1). The relations between the incident, refracted and reflected beams are given by the Descartes-Snell and Fresnel's law [1].

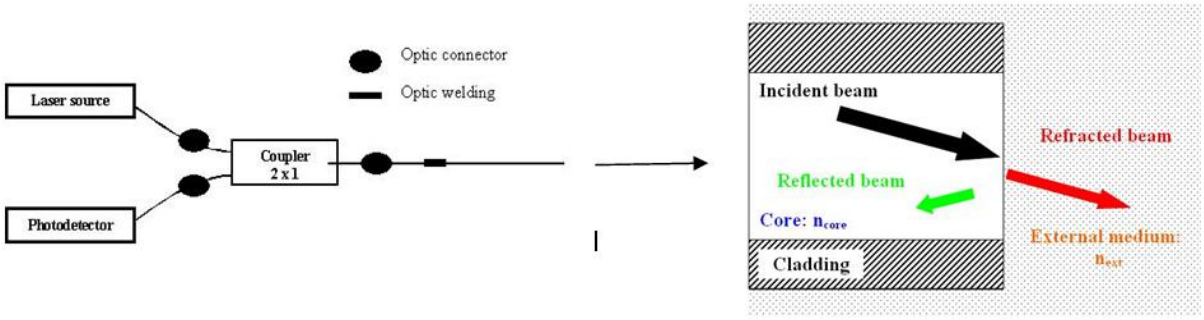

Fig. 1 Principle of the fibre optic sensor.

The principle of the sensor is displayed in Fig. 1. 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 power according to Fresnel law. This reflected part propagates along the fibre back to the coupler and can be detected through the coupler on a photo-detector connected to the second input fibre pigtail of the coupler. Previous articles [2, 3] demonstrate that the reflected radiant power can be used to obtain the resin refractive index variations with temperature or the polymerization degree. The resin refractive index can be then calculated from the relations

$$n_{resin} = n_{fibre}\frac{(1-\eta)}{(1+\eta)}$$
$$\text{with} \quad \eta = \frac{n_{fibre}-n_{air}}{n_{fibre}+n_{air}} \times \sqrt{\frac{P_{resin}}{P_{air}}} \qquad (1)$$

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 the radiant powers (at the photo-detector level) reflected by the resin or the air (external medium), respectively.

The fibre we use has a 8 µm core diameter and an external silica optical cladding diameter of 125 µm. The refractive index is 1.457 and the thermo-optic coefficient $dn/dT = 9.2\ 10^{-6}\ °C^{-1}$ [5] will be taken into account for the fibre refractive index thermal evolution.

EXPERIMENTAL SET UP

We use an experimental mould named "PvT-α" to characterize and calibrate our Fresnel reflection sensor. This mould is equipped with different sensors and is a replica of an RTM process one. It allows us to control and/or record simultaneously the applied pressure (P), the specific volume (v), the temperature (T) and the polymerisation degree (α) during all the experiment. It is represented with the added optical devices in Fig. 2.

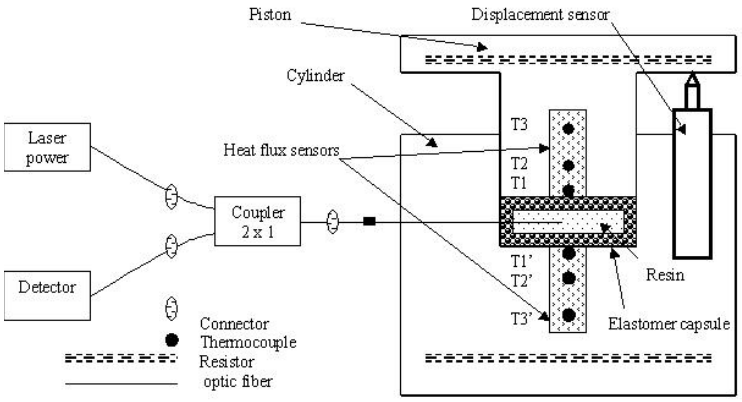

Fig. 2 Experimental set-up (optic fibre sensor and PvT-α mold).

Our sample is placed inside a 50 mm diameter weatherproof elastomer capsule, which is itself placed inside the cylindrical cavity of the mould. A piston applies a pressure on the resin sample. Furthermore, we can apply temperature cycles using resistor heaters, which are installed in the piston and the bottom of the mould cavity. The mould is also instrumented with two heat flux sensors located face to face in the piston and the bottom of the mould. These sensors are made of three 25 µm diameter thermocouples located approximately at 0.4, 3.5 and 6.5 mm from the surface of our sensor. The recorded data are processed using the Beck sequential inverse method to compute the heat flux density $\Phi(t)$ exchanged between the sample and the mould [4]. This method is based on the solving of the Fourier equation in one dimension. We get by this way the evolution of the thermal flux caused by the polymerization and it is then easy to determine the polymerization degree defined by the equation below:

$$\alpha = \frac{\Delta H_t}{\Delta H_{total}} = \frac{\int_0^t \phi(t)dt}{\int_0^\infty \phi(t)dt} \qquad (2)$$

where ΔH_t is the reaction enthalpy measured at time t and ΔH_{total} is the total enthalpy due to the polymerization reaction. This special experimental mould "PvT-α" has already been described and presented in several publications. It has been used to characterize the kinetics and the polymerization shrinkage of several thermosetting resins [5-7].

Calibration of the Optical Fibre Sensor

The studied thermosetting polymer is the RTM6 resin provided by Hexcel®. We proceed to the Fresnel's reflection sensor calibration following the same temperature cycles as those used in industry composed of two isothermal steps at T = 150 °C and 180°C (see Fig. 3). The first one is reached with a heating rate of 3.6 °C/min. The temperature is then kept constant during 10 min. The polymerization (crosslinking) of the resin occurs during this step. This auto-catalytic reaction is characterized by the formation of a 3D network of amorphous resin: the thermosetting resin start is initially in a liquid visco-elastic state and becomes a visco-elastic solid at the end of the reaction. Thus an increase of the polymerization degree corresponds physically to a densification of the resin network, which induces a decrease of the mobility of the macromolecular chains and the reactive species, leading to a gradual decrease of the reaction rate. Furthermore, when the glass temperature (T_g) of the resin tends to the imposed temperature (i.e., T = 150 °C), the resin passes to a glassy state. As a consequence, the reaction rate is dramatically reduced and then stops quickly. This phenomenon implies that the reaction is not complete at the end of the first isothermal step. In order to restart the polymerization, the resin has to pass to the rubbery state (when $T > T_g$) since in this state, the specific volume and the mobility of the macromolecular chains are greatly improved. A second temperature plateau at 180 °C is then applied during 5 min to achieve the polymerization (post-curing).

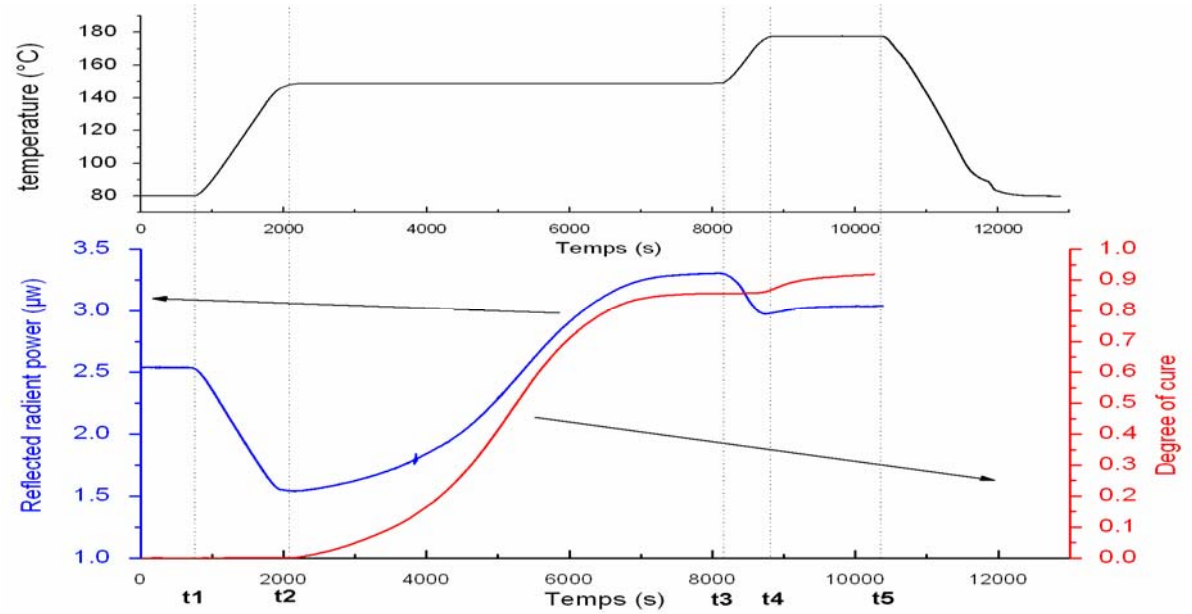

Fig. 3 Polymerisation degree, reflected optical power and temperatures applied to the resin plotted versus time.

Fig. 3 shows a temperature cycle applied to a sample, the polymerisation degree computed from the heat flux and the response of the optical fibre sensor. During the first step (from t_1 to t_2), the sensor response versus temperature gave us a linear resin refractive index (derivate from Eqn. 2) versus the temperature. These results led us to determine the thermo-optical coefficient equal to -3.696 10^{-4} °C^{-1} for a 1550 nm wavelength. This value is the average of ten measurements with a relative standard deviation of 4 %. From t_2 to t_3, the evolution of the refractive index depends only on the resin polymerization since we operate at a constant temperature (150°C) and constant pressure. The refractive index of the resin is also a linear function of the conversion degree with a slope of $dn_{resin}/d\alpha$ = 4.63 10^{-2} for a 1550 nm wavelength. Several measurements have been realized, the reproducibility has been confirmed and the standard deviation is 4 %. During heating up to the second temperature plateau (from t_3 to t_4), the refractive index varied due to two coupled phenomena: polymerization restarting and the increase of temperature. During the last step (from t_4 to t_5), we observe an increase of the reflected light, which corresponds to the residual polymerisation.

Application to an Industrial Mold

In a second stage, we investigate the feasibility to monitor successively the injection and the polymerisation of the resin in industrial conditions. When the resin is injected in the mould, the refractive index of the external medium measured by the fibre end faced swings from n_{air} = 1.000 to n_{resin} = 1.565. This change causes an abrupt drop of the reflected radiant power, as shown in Fig. 4. This is then possible to detect and to follow the pass of the resin front during the filling of the mold.

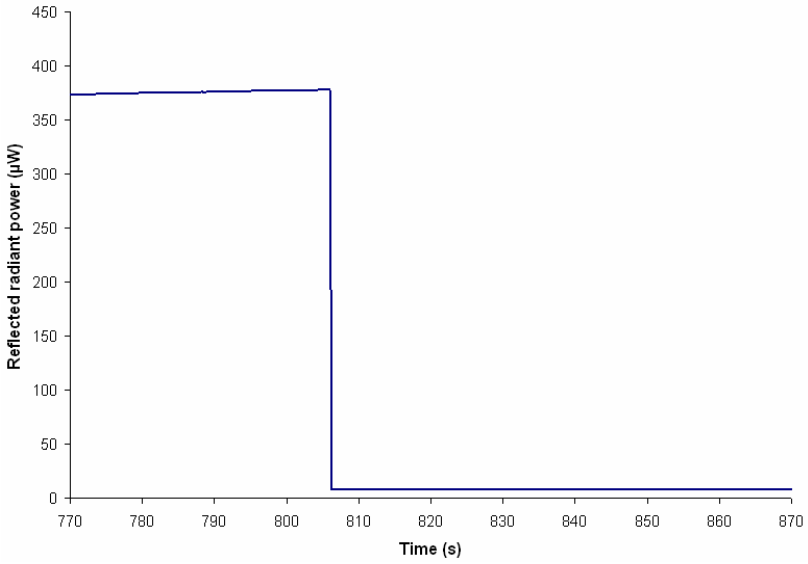

Fig. 4 Passage of the resin front.

Twelve fibres optic sensors equip a steel mould designed for making a rectangular 350 mm x 350 mm x 6 mm piece. The reinforcement of the piece is made of 8 plies carbon fibre fabric. These sensors detect the resin front during the injection as well as the conversion degree of polymerisation at different locations inside the mould. For symmetry reasons, the sensors have

been carefully located in the middle of the plies and half of the surface as displayed in Fig. 5. To detect the resin front correctly, the end face of fibres are placed in front of the resin flow.

The mold is pre-heated in an oven at T = 150°C during 12 hours before the beginning of the experiment. The temperature of the mould has been kept constant during the experiment and the resin has been heated at T = 80°C before its injection. Each fibre optic sensor has detected the arrival of the resin. The lines in Fig. 6 represent the resin front for different times during the filling step. In the beginning of the injection, this front is not plain. A preferential channel obviously appeared along the edge of the mould. During the injection step, the curvature front is attenuated. The injection time is 337 s.

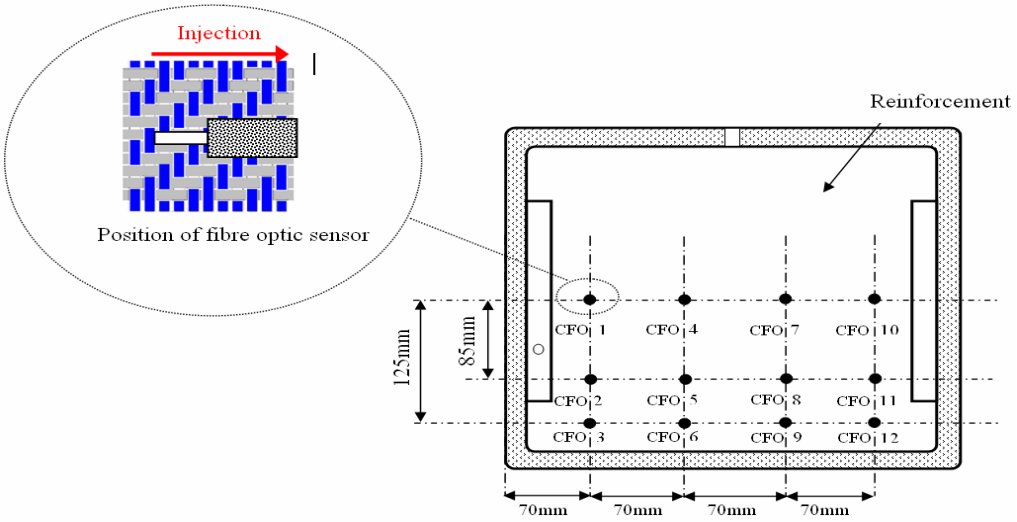

Fig. 5 Positions of the sensors.

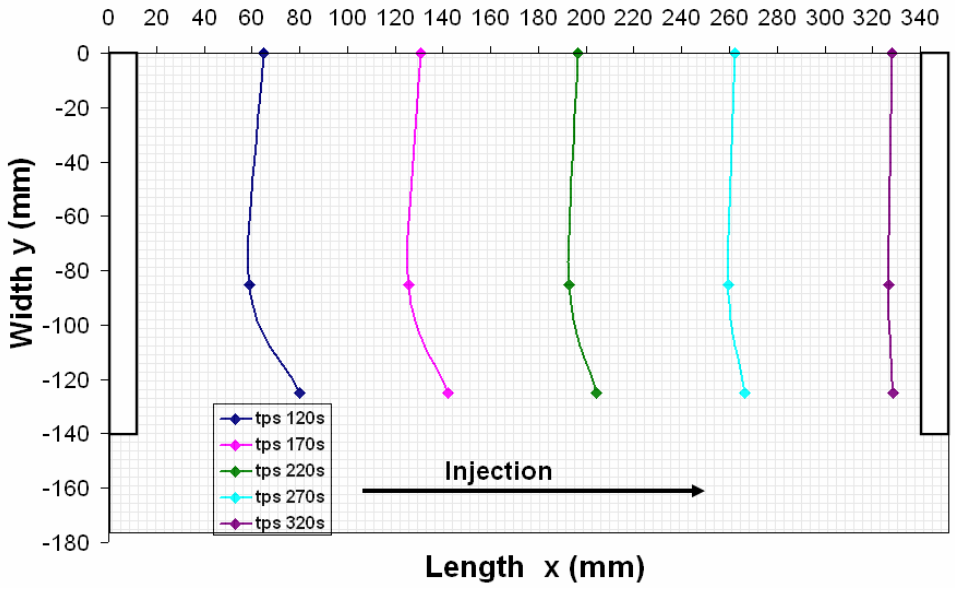

Fig. 6 Evolution of the resin front edge during the filling stage.

Once the filling stage of the RTM process has been completed, the reflected radiant power is recorded at several sensors. Fig. 7 depicts an example of the data recorded by the CFO 7 sensor (see Fig. 5). We observe an increase of the reflected radiant power, which is characteristic of resin polymerization. Using the calibration results presented in the previous sub-section, we can therefore give an estimation of the final conversion degree of the reaction of 0.85 (after curing at 150°C) with an error of 4%.

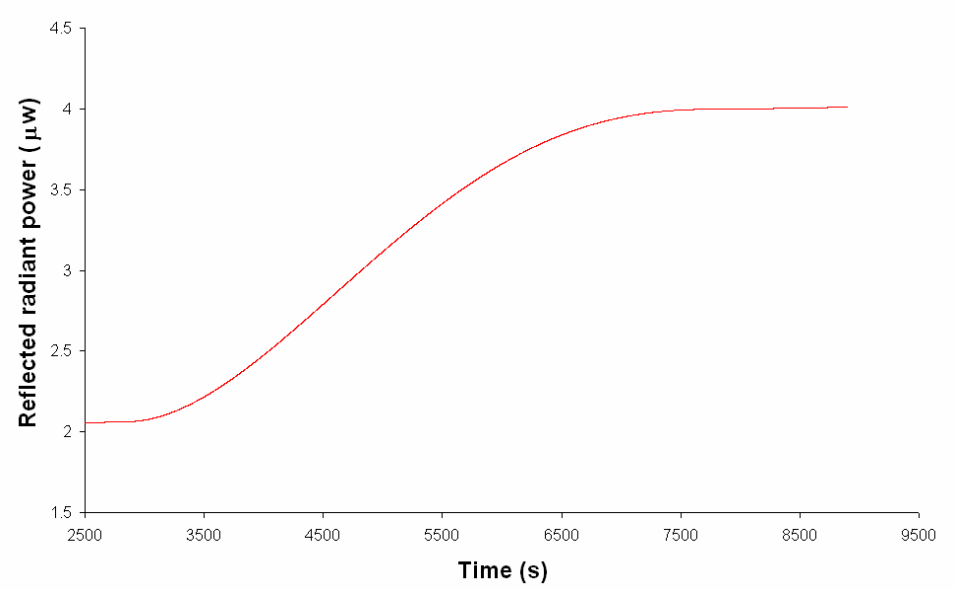

Fig. 7 Response of Fresnel reflection sensor during resin cross-linking.

These experimental data have been processed in order to determine the permeability of the reinforcement along the *x*-direction. The permeability can be obtained by an iterative algorithm detailed in Fig. 8. Simulation and experimental results are compared to finally determine the permeability.

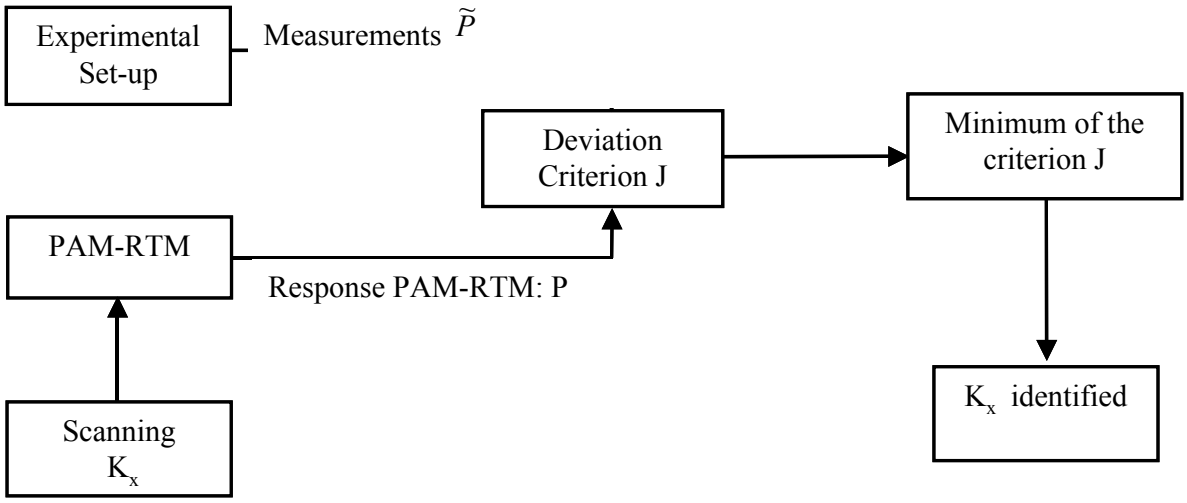

Fig. 8 General procedure of permeability estimation.

The deviation criterion between experimental measurements and results of PAM-RTM software simulations is defined by

$$J(K_x) = \sum_t (\tilde{P}_t - P_t)^2 \qquad (3)$$

where P_t and $\tilde{P}_t$ are the calculated and experimental pressures for a time step "t", respectively. Value of deviation criterion depends on the unknown K_x parameter. The criterion for the estimation versus the permeability is represented in Fig. 9. The minimum of the criterion corresponds to the wanted permeability and is equal to 4.8 10^{-10} m² (the fibre volume fraction is 39 % and the thickness of the part is 7.88 mm).

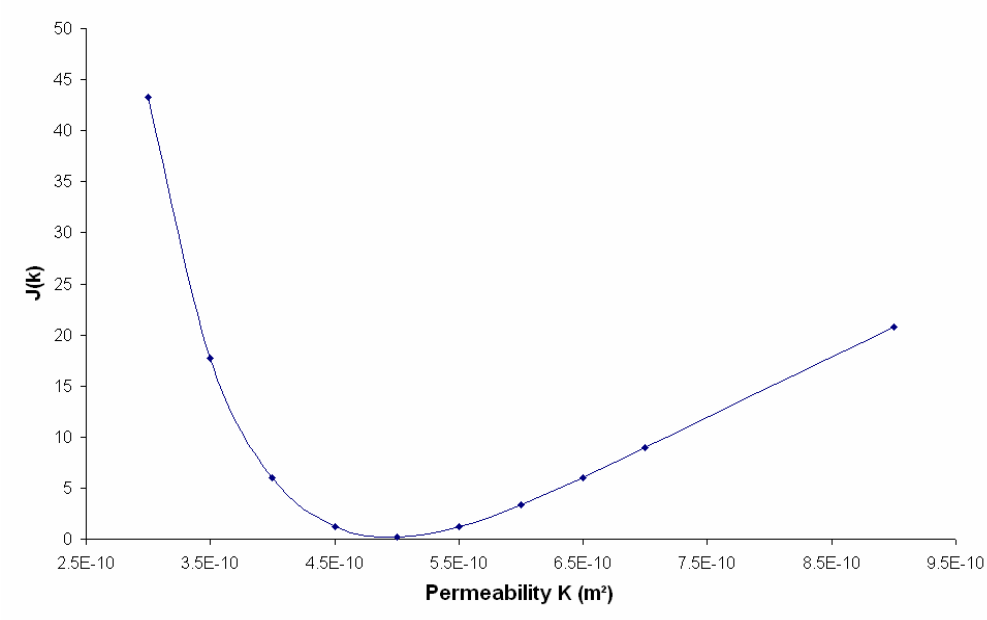

Fig. 9 Deviation criterion as a function of permeability K_x.

CONCLUSIONS

In this work, a fibre optic sensor was studied to monitor the fabrication of a composite material piece using the RTM process. As the response of the sensor exhibits an abrupt variation when its surrounding medium changes, this sensor is able to detect the resin front inside a mold. Moreover, the response of sensor is linear with the temperature with a rate of -3.696 10^{-4} °C^{-1} and linear with the degree of polymerization with a rate of 4.63 10^{-2}. This implies that we can also use this sensor to measure the degree of polymerization of the resin inside the mould during the heat treatment. We demonstrated that a single and simple optical sensor can bring important information in composite materials processing. From an academic point of view, the propagation rate of the resin is an essential parameter to estimate the permeability of the material in order to simulate the filling step. From an industrial point of view, it is necessary to measure the degree of polymerization in order to determine when the process can be stopped.

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