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A dielectric sensor for measuring flow in resin transfer moulding

Alexandros A Skordos, Panagiotis I Karkanas and Ivana K Partridge
Advanced Materials Department, School of Industrial and Manufacturing Science
Cranfield University, Beds, MK43 OAL,UK

Abstract: The development, analysis and experimental validation of a novel flow and cure sensor for use in the resin transfer moulding of composites are presented. A linear relationship is established between the flow front position in the mould and electrical admittance measurements gathered using the sensor setup, allowing accurate flow front location. The sensor performance as an indicator of flow front position is evaluated using visual verification. Its efficiency for monitoring of the curing stage is assessed by comparison of the measurements with data obtained from more conventional microdielectrometry. Experimental results demonstrate that the sensor can locate the flow front accurately. The measurement output is in the form of a complex number; this offer a potential qualitative self-assessment method. The monitoring of the cure process using the new sensor shows performance similar to that of the established microdielectrometric techniques.

Keywords: flow monitoring, cure monitoring, RTM, dielectric sensors, impedance

1.Introduction

Resin transfer moulding (RTM) is a relatively recently developed production method for the manufacture of continuous fibre reinforced thermoset composites. The processing is performed in three stages. Initially a dry fibrous reinforcement (pre-form) is placed in a rigid mould cavity. Then, forced impregnation by a liquid resin occurs (filling stage). Finally the mould is heated up, cross-linking of the thermosetting matrix material takes place and the composite cures. Although many problems of the conventional laying-up/autoclaving process connected to prepreg preparation and green composite handling are eliminated, other important matters arise in the RTM process. The impregnation stage becomes and the necessity to decouple the infiltration from the curing stage leads to the need for specialized resins and adapted thermal cycles.

In recent years the filling stage of RTM has been studied extensively. Flow models based on Darcy's law have been developed [1-3] and the mechanisms of resin impregnation have been analyzed [4,5] in order to predict filling behaviour. These analyses have provided an ability to estimate filling time, evaluate the risk of dry spot formation and examine the effects of various process parameters on them. Additionally, resin cure kinetics [6] and heat transfer phenomena [7,8] have been investigated and modelled, aiming to predict the component behaviour during the cure. Modelling has been utilized combined either with inversion techniques as a design optimization tool [9] or with measurements as a process control tool [10].

The difficulty in predefining some of the material properties involved in the process does not allow the implementation of a complete simulation as production practice at present. Thus, the need to use direct monitoring has arisen. Cure monitoring techniques based on dielectrics and infrared spectroscopy developed in previous composites manufacturing studies have been adapted for monitoring the resin cure in RTM processing [11,12]. The monitoring of the filling stage has so far focused on visual measurement of the flow front position used in permeability estimations [13-15]. Experimental configurations used in these studies involve partially transparent tooling. Therefore they cannot be extended to production or pilot scale moulding. Some studies have been carried out on the implementation of automatic non-visual flow monitoring. Dielectric or electric multiple sensing, typically comprising a grid of sensors, has been used to identify the time of wetting at a specific location [16-18]. Mathur et al [19] have investigated the application of a flow sensor based on evanescent wave fluorescence, capable of locating the flow front.

The present paper describes the development and application of a novel dielectric sensor with a dual monitoring role. During the filling it follows continuously the flow front position and during the cure it monitors globally the progress of the reaction.

2.Sensor Setup

2.1 Description and principle of operation

The geometry of the sensor is given in Figure 1. It comprises two parallel flat copper electrodes embedded between two layers of a polymeric film. A very thin layer of adhesive exists between the electrodes and the polymeric film and between the two layers of the polymeric film. Some air is trapped in the area between the two electrodes. One side of the sensor is covered by metal foil. Consequently, when voltage is applied between the two electrodes, electric field is formed only on one side of the sensor. The semicylindrical field penetrates a portion of the space where the filling of the fabric by liquid resin takes place.

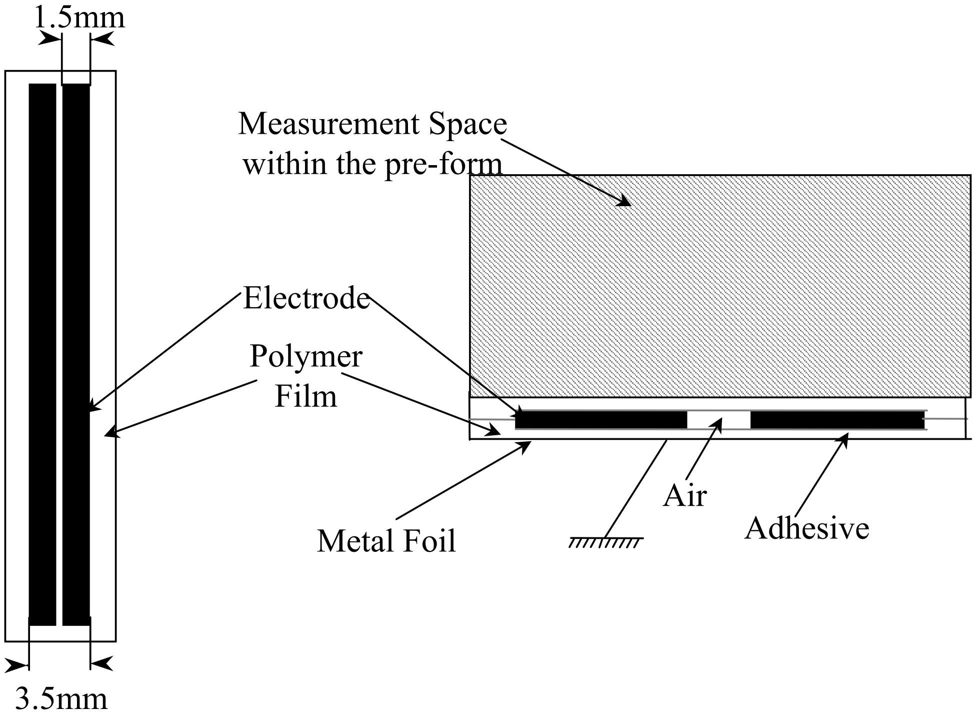

Figure 1. Geometry of the sensor setup, showing the sensor and its placement with respect to the pre-form.

During operation the sensor is aligned parallel to the expected direction of the resin flow front as illustrated in Figure 2. The sensing area is divided into two parts, the impregnated by resin 'wetted' area and the 'dry' part. As the impregnation progresses the 'wetted' area percentage increases and the 'dry' area percentage decreases. The process is illustrated by the photographs shown in Fig. 3. In Fig. 3a the sensor, placed on the bottom of the mould, is completely hidden by the dry glass fabric. As resin impregnation progresses (Fig. 3b) it becomes possible to see the sensor through the wetted part of the fabric. Fig. 3c shows a completely impregnated pre-form, with the sensor visible across the entire length of the mould. Since the electrical properties of the liquid resin differ significantly from those of the air, monitoring the evolution of the sensor dielectric response can provide quantitative information about the mould filling process.

Furthermore when the filling is completed the sensor can perform macroscopic dielectric cure monitoring.

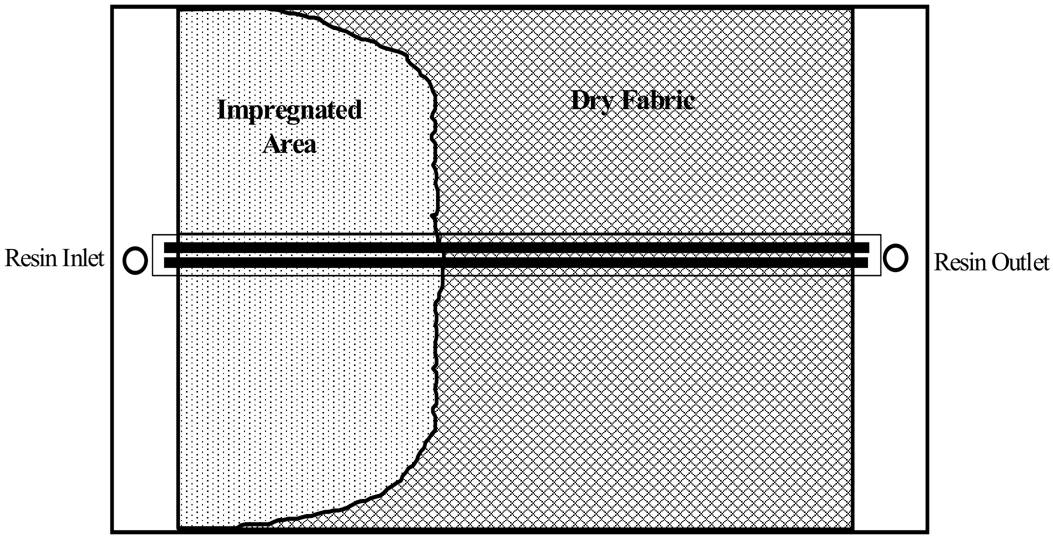

Figure 2. Schematic representation of the operation principle of the sensor.

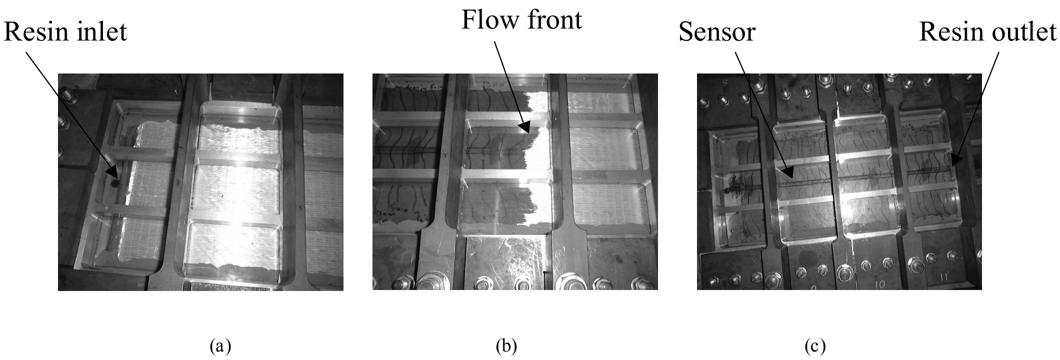

Figure 3. Filling of the RTM mould containing dry glass fabric with RTM6 resin (a) dry glass fabric (b) partially wetted (c) fully wetted. The photographs are taken from the top, through the toughened glass top of the mould.

2.3 Analysis of the sensor response

Analysis of the electric response can be performed using an electric circuit equivalent to the sensor. Each of the previously described components of the sensor contributes an impedance element to the equivalent circuit. In order to establish the component connections, the respective potential and current conditions that are imposed on them are considered. Thus, components under the same potential are considered as connected in parallel and components carrying the same current are considered as connected in series.

The substrate of the sensor has three components: the polymeric film, the adhesive and the entrapped air. The film is connected in series to the adhesive and the resulting element is connected in parallel to the air entrapped between the electrodes, as shown in Figure 4(a). Consequently, the total impedance of the substrate per unit length is given by the relation:

$$z_s = \frac{1}{\dfrac{1}{z_f + z_g} + \dfrac{1}{z_a}} \qquad (1)$$

where z_f, z_g and z_a represent the impedance per unit length of the film, the adhesive and the entrapped air respectively.

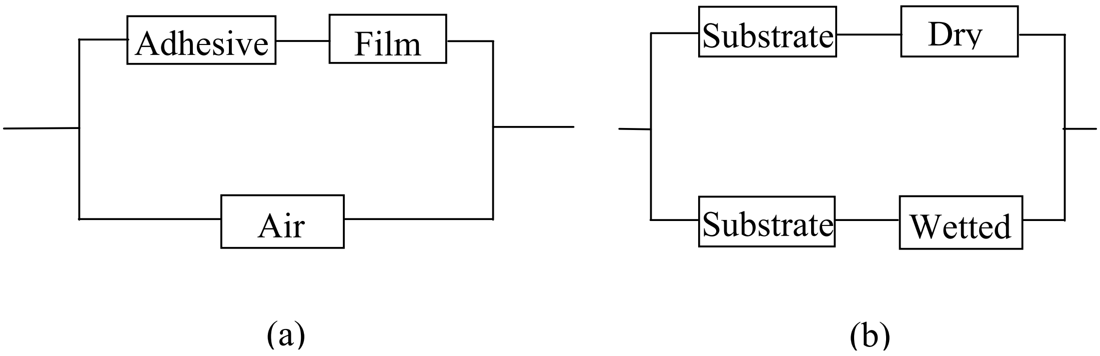

Figure 4. Circuits representing the electrical response of the sensor (a) substrate circuit (b) general circuit.

The sensor can be considered as a parallel circuit of the wetted and the dry regions. Each of these two regions can be analysed further into a serial circuit of the substrate and the corresponding measurement area. According to these the circuit representing the electrical behaviour is the one shown in Figure 4(b).

In order to express the total impedance explicitly, it is required to find an expression of the dry and the wetted areas impedances as functions of the impedance per unit length and of the length of each of them. For this reason we consider a uniform part of the sensor with impedance per unit length z and length l. If the sensor is conceptually divided into l parts each of them will be connected in parallel to the rest. Therefore the total impedance will be:

$$Z = \frac{z}{l} \qquad (2)$$

Application of that expression to the circuit of Figure 4(b) results in the relation:

$$Z_{sensor} = \frac{1}{\dfrac{l_w}{z_s + z_w} + \dfrac{l - l_w}{z_s + z_d}} \tag{3}$$

where l denotes the total length of the sensor, l_w the length of the covered by resin (i.e. wetted) part, z_w the impedance per unit length of the wetted part and z_d the impedance per unit length of the dry part.

Algebraic manipulation of equation (3) leads to the expression:

$$Z_{sensor} = \frac{z_s^2 + z_s z_w + z_s z_d + z_d z_w}{l z_s + l_w z_d + l z_w - l_w z_w} \tag{4}$$

Consequently the admittance of the sensor is:

$$Y_{sensor} = l_w \left(\frac{z_d - z_w}{z_s^2 + z_s z_d + z_s z_w + z_w z_d} \right) + \left(\frac{l z_s + l z_w}{z_s^2 + z_s z_d + z_s z_w + z_w z_d} \right) \tag{5}$$

It can be observed that according to equation (5), the admittance is a linear function of the wetted sensor length.

The two terms in brackets of the linear equation (5) are constant, if the impedances of the materials involved do not change during the filling. The intercept corresponds to the admittance of the dry sensor Y_{dry}. In order to calculate the slope, a measurement of the admittance at some point is required. Moreover, it is observed that the slope is not dependent on the total length of the sensor, so its value is constant for sensors of different lengths in the same molding. Thus equation (5) becomes:

$$l_w = \frac{Y_{sensor} - Y_{dry}}{Y_{cov} - Y_{dry}} l_t \tag{6}$$

where Y_{cov} is the measured admittance when length l_t of the sensor is covered by the resin at time t of the filling stage.

3.Experimental details

Individual sensors were fabricated by embedding two flat copper wires between two layers of a polymeric film. A single electrode width was 1.5 mm and the interelectrode distance was 0.5 mm. The sensor performance was validated during the filling of a glass cavity with silicone oil

(Dow Corning 2000) and during the filling and curing of an RTM6/glass (non crimped fabric) reinforced composite in a partially transparent RTM tool.

In the first case the total sensor length was 27.8 cm and the cavity thickness was 8 mm. The filling was performed at ambient temperature. In the second case the sensor length was 73 cm and the mould thickness 3.3 mm. The filling temperature was 100 ^oC. Admittance data were gathered over a range of frequencies using a Solartron 1260 impedance analyser. Visual determination of the flow front position was recorded against time in both cases. After completion of the filling step the mould temperature was increased to 160 ^oC to cure the resin.

In order to validate the cure monitoring performance of the sensor, the curing of unreinforced RTM6 resin, subjected to an equivalent thermal programme, was monitored using commercial GIA microelectrodes [20].

4. Results and discussion

4.1. Flow monitoring

Equation (6) has been used to calculate the flow front position from the admittance data. The admittance measured when the sensor is fully covered by resin has been used as Y_{cov}. That means that knowledge of the final measurement was necessary in order to perform the covered length calculation. In a real application where on-line estimation of the flow front position is required another characteristic length should be used. One possibility is a measurement from an additional short length sensor, which would be fully covered by resin before the wetting of the actual sensor commences. Alternatively measurement from the actual sensor can be used, with the field interrupted by electrode screening over a short length at a specific point.

It can be observed from equation (6) that the calculated length is a ratio of two complex numbers. In the ideal case the nominator and denominator of the ratio would be in phase and consequently, the resulting length would be a real number. However, measurement inaccuracies and occurrence of phenomena which alter the impedance of the materials involved (e.g. local temperature variations), can cause some phase difference resulting in the existence of an imaginary component of the calculated length.

The real and the imaginary parts of the wetted length, combined with the visual flow front position measurements, for the filling of the cavity with silicone oil and the filling of the RTM mold with epoxy resin are illustrated in Figures 5 and 6 respectively. The corresponding measurement statistics are given in Figures 7 and 8. It can be observed that in both cases the calculated flow front location closely follows the visual observation. The statistical analysis shows that there is a correlation between the measured imaginary length and the error in the flow front location. Thus, high values of the imaginary length imply a significant error in the flow front location. In all measurements the imaginary part never exceeded 0.6 cm which is low in comparison with the sensor length; therefore the final accuracy can be considered to be satisfactory.

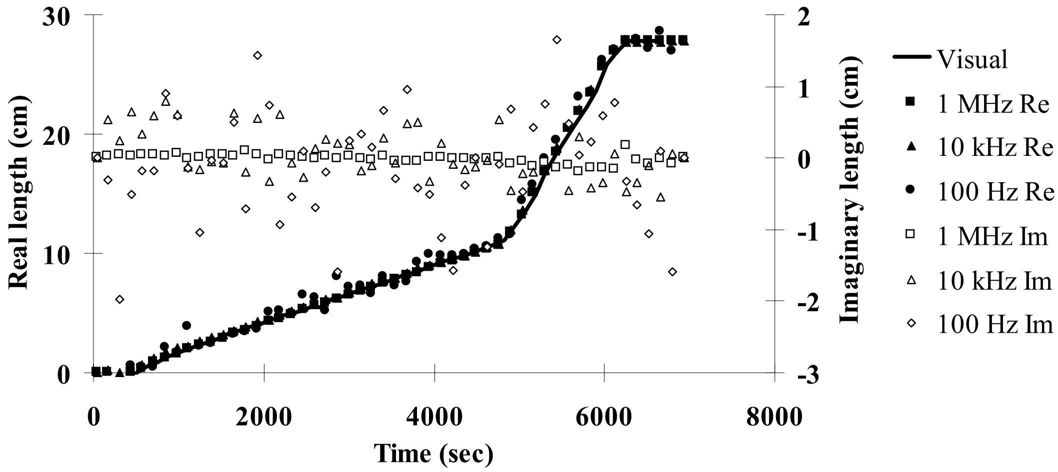

Figure 5. Comparison of visual measurement with dielectric flow measurement for silicone oil filling.

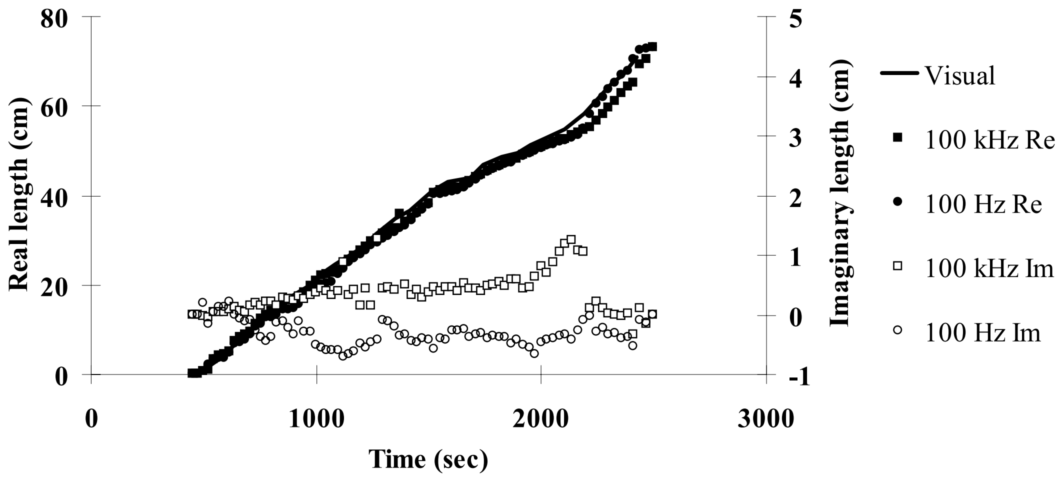

Figure 6. Comparison of visual measurement with dielectric flow measurement for RTM6 resin filling.

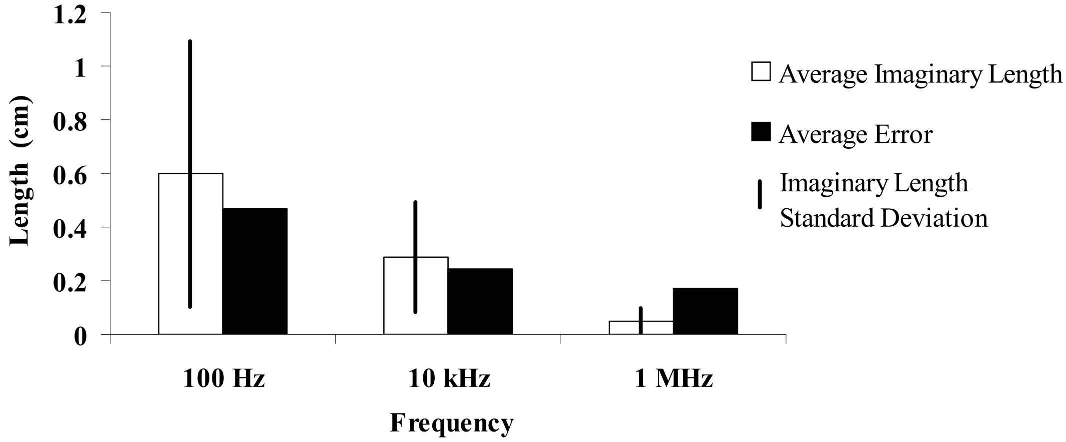

Figure 7. Correlation between the imaginary part of the measured length and the error of the measurement for silicone oil filling.

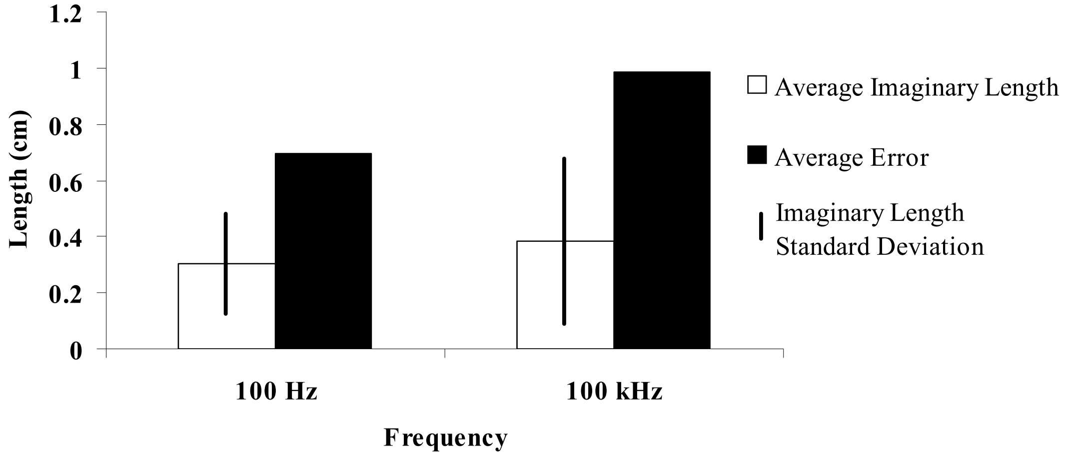

Figure 8. Correlation between the imaginary part of the measured length and the error of the measurement for RTM6 resin filling.

4.2. Cure Monitoring

The data of the imaginary impedance gathered by the flow sensor are given in Figure 9. Results from a microdielectrometry experiment using a simulation of the thermal profile followed in the RTM mould are shown in Figure 10.

The logarithm of impedance values have been normalized using the following expression:

$$Normalized\ lg\ Z'' = \frac{log_{10} Z'' - log_{10} Z''_{min}}{log_{10} Z''_{max} - log_{10} Z''_{min}} \qquad (7)$$

where Z''_{min} Z''_{max} are the minimum and maximum values of the imaginary impedance during the cure.

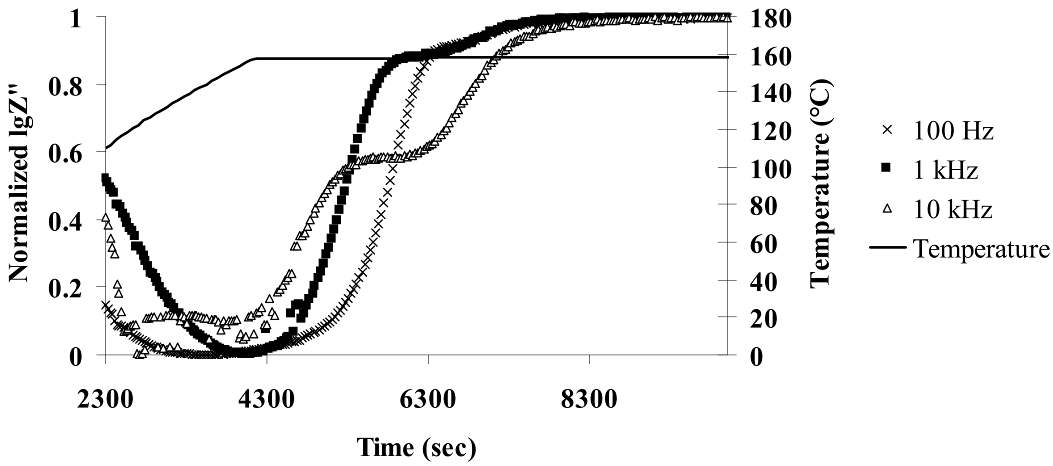

Figure 9. Results of cure monitoring obtained with the new sensor. Logarithm of imaginary impedance is normalized against its minimum and maximum. during the cure.

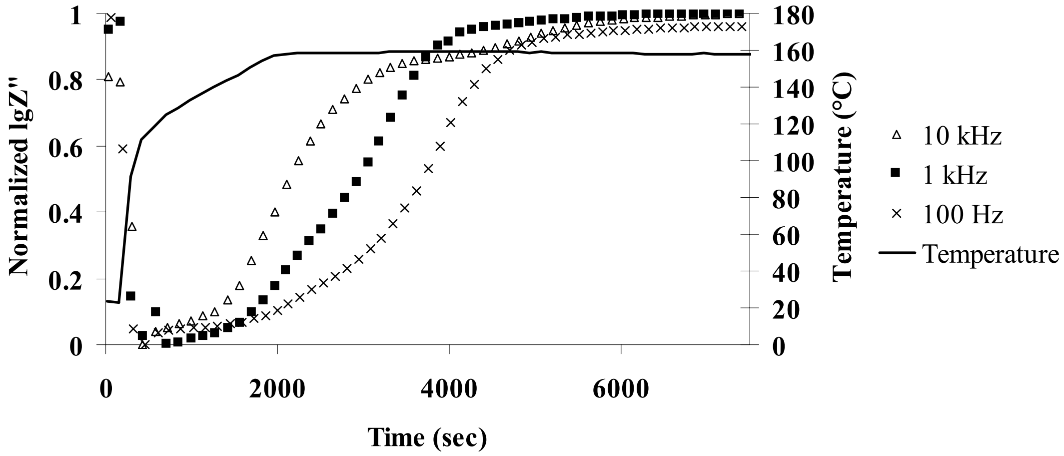

Figure 10. Results of cure monitoring using microdielectrometry. Logarithm of imaginary impedance is normalized against its minimum and maximum during the cure.

The information derived from the two different experiments is similar. During the cure of such a system the imaginary impedance signal presents a two step pattern. The first step corresponds to the progressing reaction but it is frequency dependent and consequently cannot be identified as a characteristic point of the cure. The second slight step or 'knee' corresponds to the vitrification of the resin [21]. The time ranges where the first step appears at different frequencies are within 300 seconds of each other. The vitrification is indicated in both cases approximately 4000 seconds after the beginning of the thermal program. In the microdielectrometry experiment the vitrification occurs slightly earlier, therefore the knee is obscured by the reaction step at low frequencies. During the cure of the component, significant temperature gradients can be expected to exist within the sensing region of the global sensor. The microdielectrometry experiment was performed at the temperature representing the bottom layer of the curing component, which is higher than the average temperature in the RTM mould. Therefore vitrification in the microdielectrometry experiment occurs earlier than in the RTM experiment.

5. Conclusions

A dielectric sensor for measuring resin flow in resin transfer moulding has been developed and tested. Equivalent circuit analysis demonstrated the ability for the filling stage of the RTM process to be monitored through the existence of a linear dependence of the admittance signal, measured by the presented sensor setup, upon the flow front position. Experiments confirmed that the flow front position can be located continuously with a satisfactory accuracy. In addition, the existence of an imaginary component of the flow front position can be utilized as self-assessment tool of the measurement performance. The sensor setup also presented the ability to monitor the cure of the resin after the end of the filling stage. Results from this macro-dielectric cure monitoring performed by the sensor were in fair agreement with results from conventional microdielectrometry experiments.

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Skordos, Alexandros A.

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