Applied process monitoring and control for CFRP production

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ABSTRACT

An electrical resistance and temperature monitoring system focused on the in-situ process monitoring of composite manufacturing processes is presented and applied in high-end composites applications. Durable non-intrusive sensors have been used for the sensing of resin arrival, viscosity changes as well as curing. Simultaneous measurements of temperature and electrical resistance of the matrix provide in-situ real-time indication of the resin viscosity during processing but also to identify and handle issues such as the aging of premixed resins or prepregs. Additionally, the correlation of the measurements with kinetic and viscosity models resins prove that the proposed system is able to monitor the process from the minimum viscosity level up to the end-of-cure. Also, comparison to dielectric systems proves the present system's superiority for this type of applications. Furthermore, the feedback of the monitoring system has been employed successfully for the real-time control of the composites processing to ensure product quality and cycle time reduction. The monitoring and control systems did prove their advantages in monitoring and control of an RTM epoxy CFRP production achieving curing cycle acceleration by 36% and maximum cycle temperature reduction by 5%.

INTRODUCTION

In composite materials production there is a real need for durable non-intrusive sensing system that would provide complete information of what is happening in a mould or die for real-time process control and quality control purposes. As the installation and operation of such systems is not easy in real production environment, it is very important that these systems are robust and durable. Previous works have shown that the processing of thermoset (ref. 1, 2) and thermoplastic (ref. 3) matrices in composite materials production can be monitored in real-time and *in situ* using the dielectric monitoring technology. The concept of this approach is based on the assumption that the electrical properties of a polymer matrix can provide the state of the polymer i.e. viscosity, gel point, vitrification point and end-of-cure. The ultimate target is to link these electrical properties with quality and performance criteria so the electrical and temperature measurements should be combined with material models in order to convert them to resin viscosity η and degree-of-cure or glass transition temperature T_g. These models can be used

offline to provide the optimal process cycle (ref. 4) but also online to provide real-time optimal process control in composite materials manufacturing (ref. 5).

In the same context similar accuracy can be achieved by measuring directly the electrical resistance of a resin using a steady-state electrical excitation instead of a range of sinusoidal excitations. In the literature several sensors and DC amplifiers have been proposed but the first commercial system has been recently available and tested in lab and industrial scale applications (ref. 6). At the present paper new results from the use of this new process monitoring system are presented together with results from the correlation between the viscosity and the conductivity. Furthermore, the possible deviations during composite production are speculated and possible control techniques are analyzed.
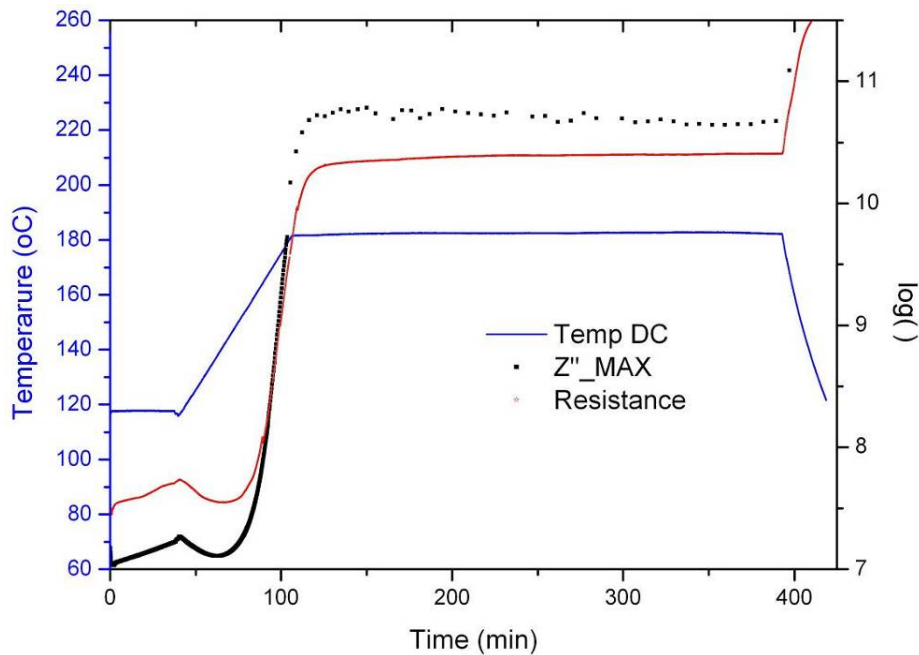

Fig.1. Simultaneous dielectric (max imaginary impedance- Z''max) and DC (resistance) and temperature measurements during the cure cycle of a high-temp epoxy system.

PROCESS MONITORING USING DC SENSING

In dielectric cure monitoring, a frequency range of sinusoidal voltage or current excitations are applied to a pair of electrodes which are in contact with the material under investigation so that the post-processed feedback provides information about the material state. Although significant effort has been devoted in this technology for more than 30 years only laboratory and limited industrial scale applications exist. On the other hand, the use of DC conductivity for process monitoring purposes is even older but no significant progress was presented in extending this idea to monitor the complete composites processing until recently. Recently a new DC-based process monitoring system (ref. 7) has been presented. With a clear focus on composite materials' industrial applications the new system measures the materials' resistivity and temperature using durable sensors and suitable electronic systems capable of the in-situ monitoring of the full transformation of a thermoset resin i.e. from very low viscosities at high temperatures to fully cured resins at room temperature where the measured resistance varies from

10^6 Ohms up to 10^{14} Ohms. Comparisons between the DC sensing and commercial dielectric systems which measure the impedance of a resin showed that the superiority of the DC sensing is observed particularly after gelation where conductivity is measured in a more reliable manner and reduced sampling time. As can be seen in fig.1 in the curing of a monocomponent resin at high temperature both monitoring systems advance almost in parallel but after gelation the measurement sampling time increases significantly due to the need of very low frequency scanning (well below 1 Hz) so the measuring quality deteriorates. Additionally the continuation of the DC monitoring during the cooling stage highlights the great potential of the DC cure monitoring system to measure even at very high resistance levels a feature which is very important at low temperature curing. Similar conclusions can be drawn from the results of the cure monitoring of another epoxy system cured at lower temperature which are depicted in fig. 2. Obviously, there is a deficiency of the dielectric system to measure higher than 0.80 degree of cure. The increase of the size of the dielectric sensor that was used could improve its performance but other problems would have occurred such as greater sensitivity to carbon fibres. On the contrary, the measurement quality of the DC monitoring system is very stable even at the end-of-cure region and at temperatures as low as 20°C. Furthermore, the DC sensing equipment is more affordable than dielectrics and corresponding sensors can be more flexible and robust and can be installed in various locations in the mould, the die, the feeding or the evacuation lines so global process monitoring is possible.

Last but not least, in contrast to the through-thickness measuring capability of the dielectric systems, DC sensing is less vulnerable to the existence of carbon fibres in the cavity due to its inherited "surface" measuring nature so it may be used in industrial production of carbon fibre parts even without protection. The durable DC sensors used in this study had 16 mm diameter and were flush mounted in areas that can touch the resin during its transformation. The sensor had an integrated temperature sensor providing the temperature close to the polymer matrix which is absolutely necessary for the calculation of the material's state in conjunction with its conductivity.

IN-MOLD VISCOSITY MONITORING

Besides the end-of-cure area, the electrical measuring system can be used for monitoring the viscosity of the resin in the cavity in order to verify the onset of viscosity rise, the quality of the resin matrix or other process deviations. The viscosity of a polymer is closely related to its electrical resistance as it has been experimentally shown (ref. 7) for a non-reactive polymer:

$$\sigma(T) \cdot [\eta(T)]^k = Const \qquad [1]$$

where σ is the electrical conductivity of the polymer and η is its viscosity. To calculate the viscosity from the in-mold measuring resistance eq.(1) can be modified as:

$$\eta(T) = C \cdot [R(T)]^K \qquad [2]$$

Where R is the measured resistance and K and C are constants which should be calculated from a series of combined viscosity and resistivity measuring trials.

To verify this in practice four samples of a neat epoxy resin were step-wise heated from room temperature to 90°C. During heating, viscosity, temperature and resistance were recorded simultaneously using a Brookfield viscometer and an integrated Synthesites process monitoring system. The results from the interpolation of viscosity and resistance following eq.[2] can be seen in fig. 2 which provides a fairly accurate prediction of the resin's viscosity from the real-time measured resistance as can be seen in fig.3.

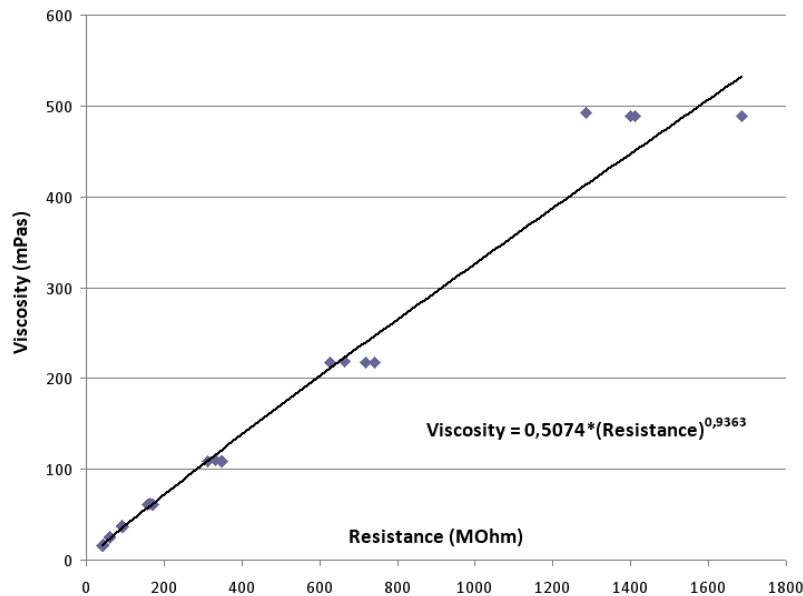Viscosity = 0,5074*(Resistance)^{0,9363}

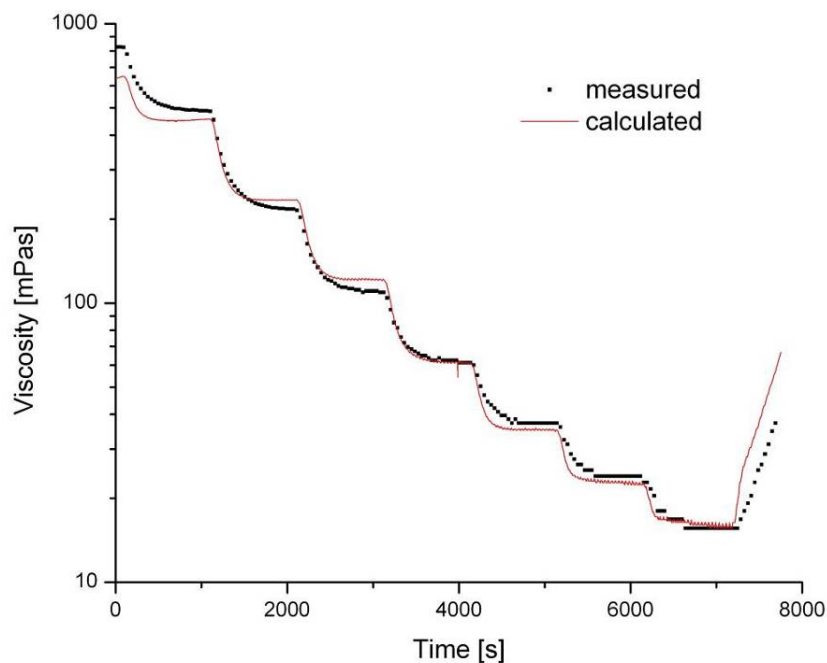Fig.2. Interpolation of viscosity with respect to electrical resistance.

Fig.3. Comparison of measured and calculated viscosities from the measured electrical resistance based on eq.[2] for a neat epoxy resin (no hardener).

IN-MOLD CURE MONITORING

For monocomponent resins which are used in advanced composites, thermal history of a batch when kept at 80°C for several hours as well as when it is mixed with fresh resin may lead to process deviations. Furthermore, the discard of aged or expired resins besides their economic loss cause large environmental burden. The use of the DC monitoring system can reveal the exact state of a resin and can give the right feedback to a control system to maintain product quality. As can be seen in fig.4 an epoxy system (case e) exposed at 80°C for 16 hrs more than the maximum recommended time (case a) showed in a simulated production cycle (80°C-120°C-180°C) a considerable deterioration of its minimum viscosity value and duration which was fully correlated to its measured electrical resistance.
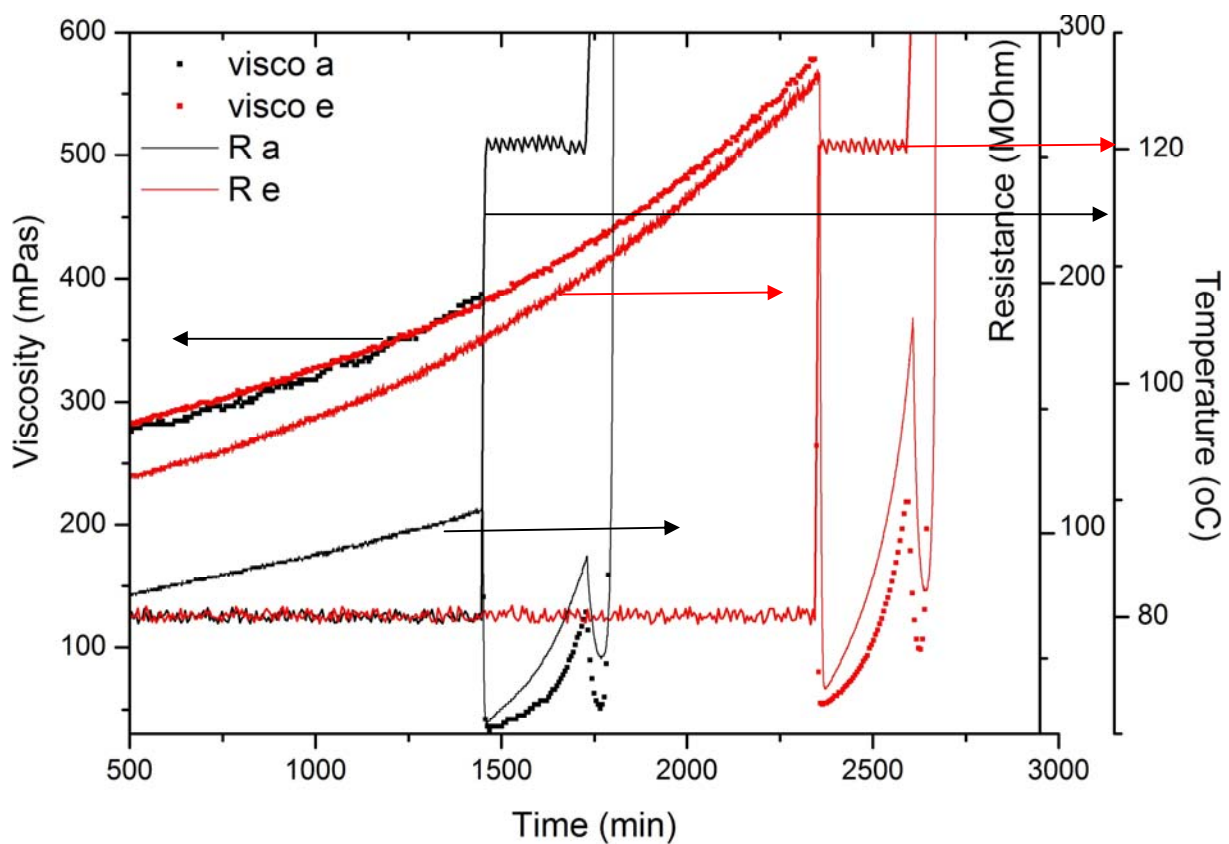

Fig.4. Resistance (solid lines), viscosity (symbols) and temperature (dashed lines) of two samples of a monocomponent epoxy system with different extended preheating duration @ 80oC. Longer preheating by 16 hrs (case e) caused an increase of the lowest viscosity point during injection @120oC and shorter injection time allowance which was picked-up by the measured resistance.

End-of-cure can be also identified when monitoring the electrical resistance of the resin as depicted in fig. 5. The slight time shift between the monitored resistance and the theoretical prediction of the degree of cure was due to the exotherm of the 10 mm thick resin sample.
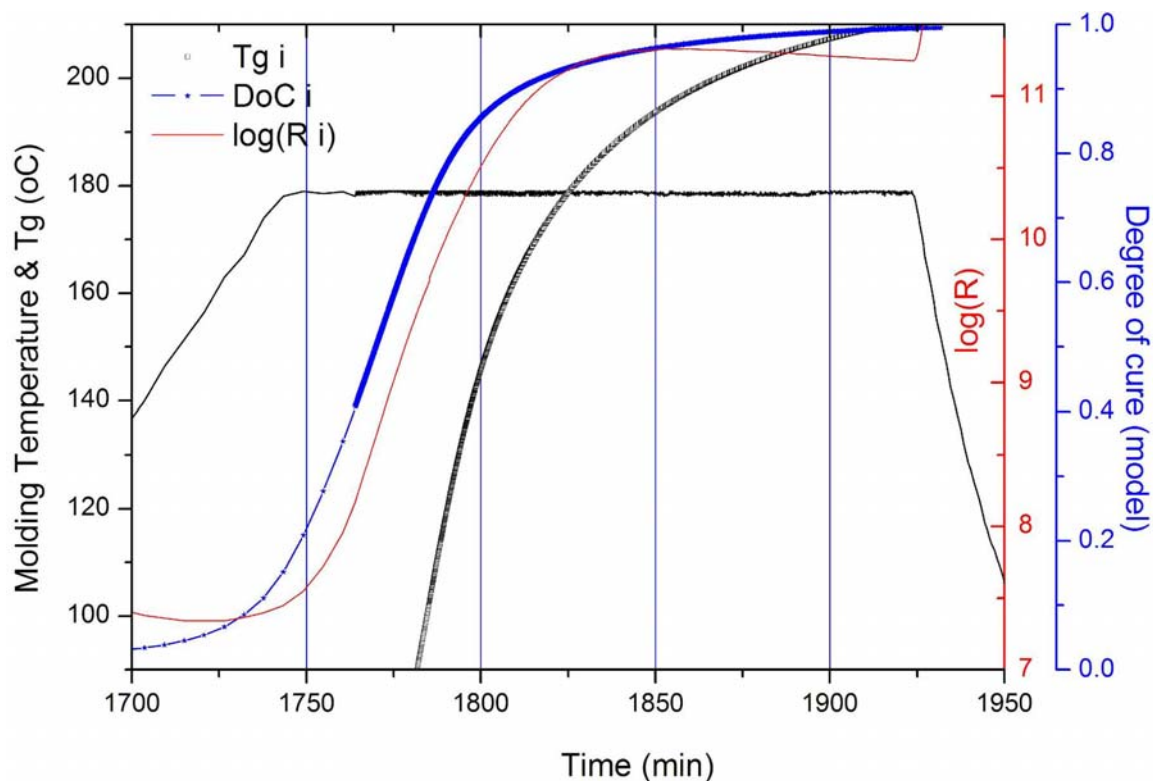
Fig.5. Electrical resistance, molding temperature as well as theoretical Tg and degree of cure during the recommended curing cycle of a monocomponent epoxy system.

PROCESS DEVIATIONS AND CONTROL

One of the major disadvantages of composites over homogeneous materials e.g. metals is their complex and delicate manufacturing which is not forgiving at all. In principle the concept in advanced composites manufacturing is to eliminate process deviations by using fully controllable materials and conditions such as prepregs and autoclaves so their manufacturing costs were very high. Nowadays, the wide spreading of composites and the need of cost reduction have pushed materials and craftwork to their limits so the need for automation is prerequisite to increase productivity and minimize scrap and waste. Especially the latter is very important as materials and recycling are very expensive especially in carbon fibre parts. In order to reduce costs in moulding industries, production has moved to heated moulds, dry fabrics and liquid resins. These changes introduced several deviations in the manufacturing process which should be kept under control for optimum output. For example the in-situ mixing of two or three different components of a resin system just before injection may result in variations in the mixing quality or mixing ratio in some instances. In a fixed cure cycle these variations even if they are small might cause some quality problems. It has been shown (ref. 7) using the DC-based process monitoring system we can clearly distinguish when the matrix is above or below the required mixing ratio even for deviations as low as 2.2% so corrective actions can be taken to maintain quality and speed. Other deviations that can be picked up by the process monitoring system can be the quantity of other additives in the resin such as thermoplastics or the nano-size additives.

In order to take full advantage of the process monitoring capabilities process control tools and platform are under development in the EC supported research project "iREMO". In this project simplified control methods based in a combination of events and rules (rule-based control (ref. 3)) will be combined with very complex methods using real-time process models (model-based control (ref. 5)) in conjunction with self-learning modeling and control tools for the optima and direct control of the manufacturing process.

INDUSTRIAL APPLICATION

To verify the performance of the process monitoring and the rule-based control tools in industrial applications, the systems were installed in a fast RTM production scale for manufacturing of epoxy/carbon-fibre sandwich beams that has been already operating for more than 2 years at Technika Plastika's production site in Greece. In the basic set-up the resin was injected at one end of the cavity and exited the cavity on the other end of the beam under constant flow so the pressure was reaching 15 bars. In order to speed-up curing, the thermal cycle was a constant heating after the end-of-injection at maximum rate up to 100°C, dwell at this temperature for 3 minutes before cooling according to the resin's specification sheet.

Fig.6. Flush mounted sensor at the cavity's margin near the inlet (left) and outlet (right) gates.

For the process monitoring, two durable sensors were installed near the injection and venting gates of the cavity (fig.6). Both sensors were measuring resin's electrical resistance and temperature and their feedback was recorded and used for quality and process control purposes. During the production the sensors did not receive any special treatment from the operators and did not present any problem due to the existence of carbon fibres in the mould cavity. Based on the feedback from the sensors two rules were introduced to optimise the process: firstly, to increase mould injection temperature by 5°C based on the resin state at the outlet gate sensor and secondly to conclude curing as soon as the resistance curve levels off. As these rules were confirmed by the quality control tests they were used consecutively to control the process during normal production. During three days where we were observing the system the mean curing time

was reduced from a mean cycle duration of 1980 s for the production without control to 1267s achieving a speed-up of 36%.

CONCLUSIONS

An innovative process monitoring system has been used for the real-time monitoring of cure and viscosity in the lab but also in real industrial environment. The system's performance has been verified at the lab-scale for identifying various issues useful in production and used to model viscosity changes of a resin. Furthermore, installed in a real manufacturing line, the system demonstrated exceptionally stable measurements and robustness without requiring any special treatment from the staff. The development of a rule-based control strategy taking advantage of the feedback from the process monitoring system led to 36% decrease of processing time allowing for an overall increase in the production rate from 4-5 parts per shift to 8 parts per shift.

Next step which is under development in the iREMO project by a European Consortium is an intelligent control system with an innovative hybrid control based on neural networks and optimised model based control as it will enhance the performance of the control, increasing also its flexibility and robustness.

ACKNOWLEDGEMENTS

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