

PROCESS MONITORING AND CURE CONTROL APPLIED IN RTM PRODUCTION OF EPOXY/CARBON FIBRE PARTS

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SUMMARY

An innovative process monitoring system of Synthesites (Athens, Greece) was employed for the real-time sensing of composite manufacturing. The system uses durable non-intrusive electrical and temperature integrated sensors and can monitor the complete process cycle i.e. resin arrival detection, viscosity changes up to the end-of-cure. Based on the monitoring feedback a control algorithm decides when to terminate the curing cycle of each part leading to shorter and more reliable production. The monitoring system was installed at a manufacturing plant and successfully used in the production of epoxy/ carbon fibre parts using RTM. Sensors were installed near the injection and venting gates and real-time information of the resin state was used for the quality and process control of the production. Using the system a 30% reduction of the curing time was attained while the monitoring system helped in improved fine tuning of the production.

Keywords: polymer composites, cure monitoring, process control

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1. 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 [1, 2] and thermoplastic [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 [4] but also online to provide real-time optimal process control in composite materials manufacturing [5].

In the same context to the dielectric process monitoring similar performance 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 [6, 7, 8] but the first commercial system has been recently available and tested in lab and industrial scale applications. At the present paper new results from the use of this new process monitoring system are presented together with initial 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. Finally, an industrial application in liquid composite moulding is presented where a significant acceleration of the cure cycle was achieved.

2. 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, e.g. [9] 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 [10] 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.

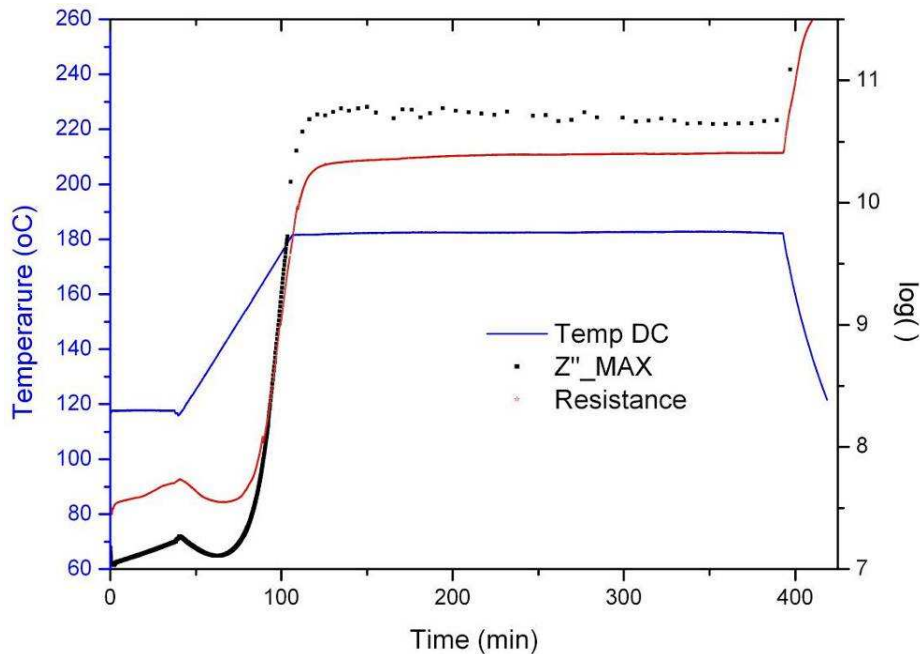


Fig.1. Simultaneous dielectric (max imaginary impedance- Z''_{max}) and DC (resistance) and temperature measurements during the cure cycle of a high-temp epoxy system.

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 is relatively cheaper than dielectric monitoring and requires simpler sensors which can be more flexible and robust and can be installed in several locations in the mould, in the die, in the feeding or in the evacuation lines so a global process monitoring is possible.

Last but not least, in contrast to the through-thickness measuring nature of the dielectric systems, the 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 an outer diameter of 16 mm and were flash 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.

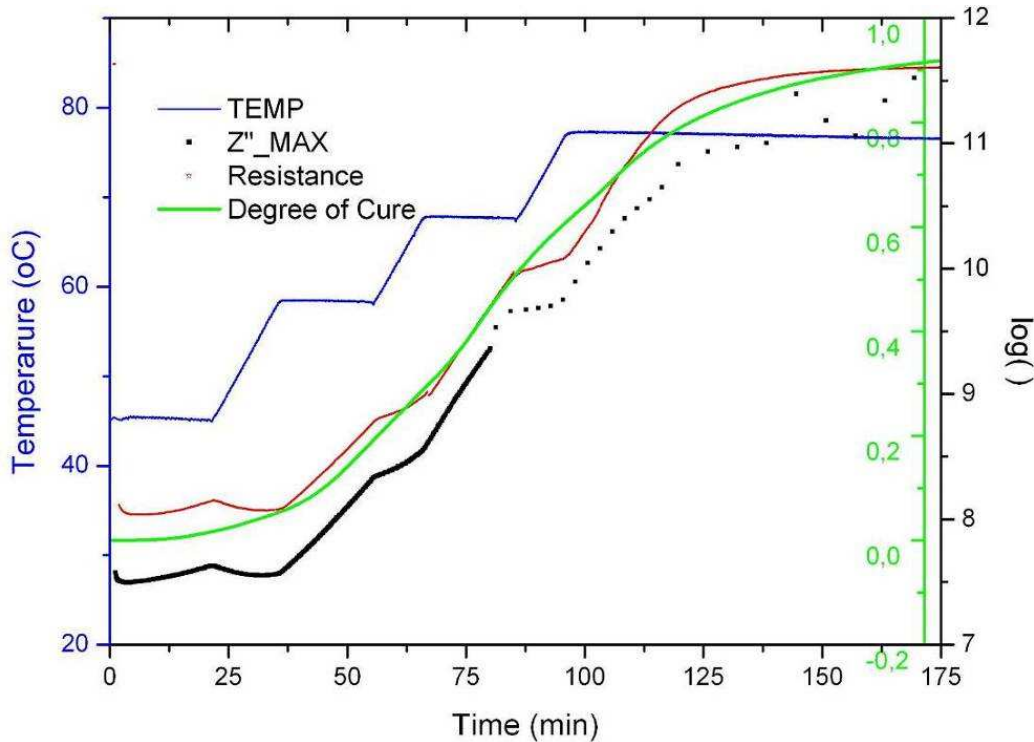


Fig.2. Simultaneous dielectric (max imaginary impedance- Z''_{max}) and DC (resistance) measurements and comparison with the theoretical prediction (degree-of-cure), during the mid-temp curing of an epoxy system.

3. PROCESS DEVIATIONS

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 the initial steps of composites manufacturing the concept was to eliminate process deviations by using fully controllable materials and facilities 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 craftsmanship to their limits so the need for automation is evident to increase productivity and minimize rejection ratio. Especially the latter is very important as materials and recycling are very expensive especially in carbon fibre parts. For example in order to reduce costs in moulding industries, production has moved from autoclaves and prepregs 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. As can be seen in fig.3 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.

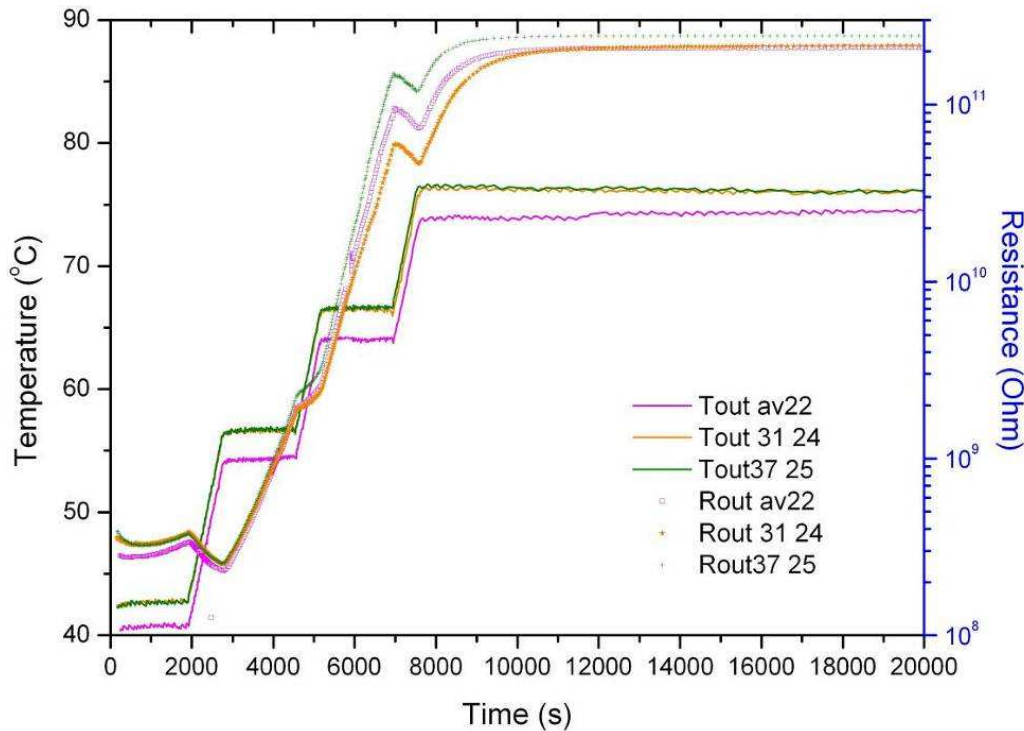


Fig.3. Electrical resistance and temperature during the curing of an epoxy system with nominal mixing ratio (av22), 2.2% more hardener (Rout37 25) and 2.2% less hardener (Rout31 24).

In the case of monocomponent resins which are used to avoid such mixing problems, the thermal history of a batch when defrosted and reheated several times as well as when it is mixed with fresh resin batch may cause other process deviations. Furthermore, the discard of expired resins besides their economic loss can cause large environmental burden so the use of these resins would save money and reduce pollution. 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 a fresh and a thermally-aged uncured monocomponent epoxy system has been tested in a simulated injection cycle, measuring simultaneously viscosity and resistance. As depicted in fig.4 the aged resin's viscosity and resistance presented higher values than their corresponding values of the fresh resin. Furthermore, during the injection steps at 120°C the viscosity of the aged resin increases faster than the fresh resin and a similar behaviour is recorded for the corresponding resistances.

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.

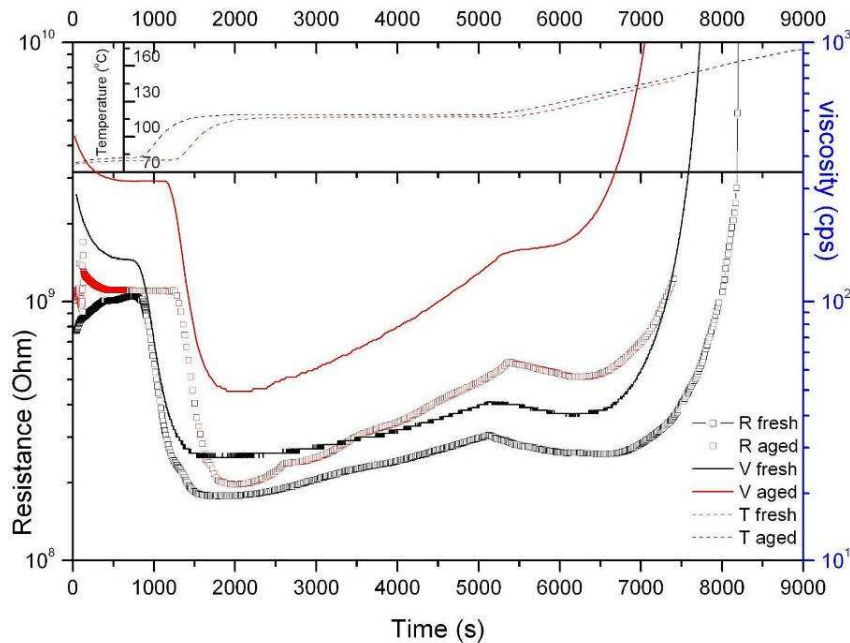


Fig.4. Electrical resistance (symbols), viscosity (solid lines) and temperature (dashed lines) during the injection of a fresh and an expired monocomponent epoxy system.

4. CURE CONTROL

In order to take full advantage of the process monitoring capabilities a process control algorithm should be developed. This algorithm can be from a rather simplified one based in a combination of events and rules (rule-based control [11]) to a very complex one using real-time process models (model-based control [5]). The former can be applied in a straightforward manner using basic knowledge of the process and existing experience but it cannot be optimal, the rules need to be updated for each process and the specific rules need testing and verification. On the other hand, model-based control requires detailed information and process models as well as quantified targets and process constraints but it can be optimal and can be used in more complicated processes such as curing of thick components with significant exotherms. In the present study, the rule-based control has been developed as it is more general and can be applied directly. One of the main variables that can be used for control actuation during the process is the thermal cycle i.e. the temperature profile which can be extended, shortened or increase maximum temperatures. In order to enforce smooth control actions the rules' handling can be combined with fuzzy logic.

5. INDUSTRIAL APPLICATION

To verify the performance of the process monitoring and control systems 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 Plastica'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 until 100°C and remained there for 3 minutes before cooling according to the resin's specification sheet.



Fig.5. Flash 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.5). Both sensors were measuring resin's electrical resistance and temperature and their feedback was recorded (fig.6) and used for quality and process control purposes.

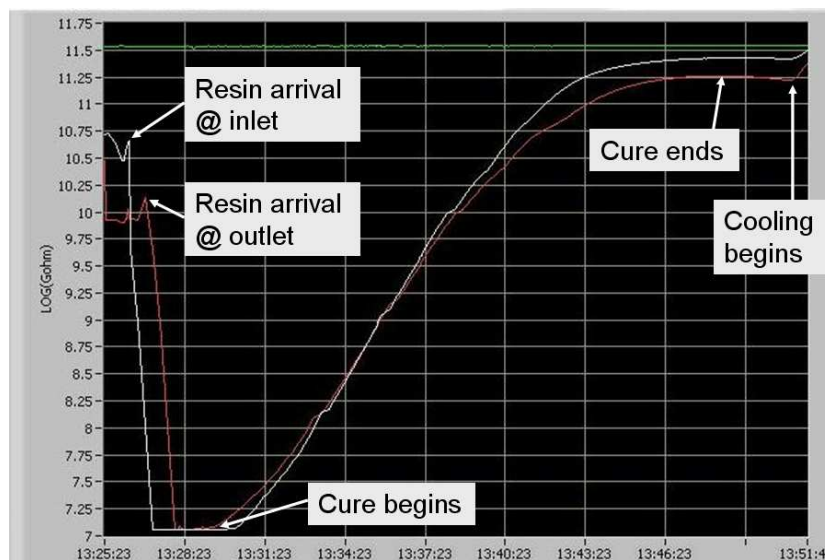


Fig.6. Screenshot of the real-time monitoring of the process milestones.

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 (fig.7) achieving a speed-up of 36%.

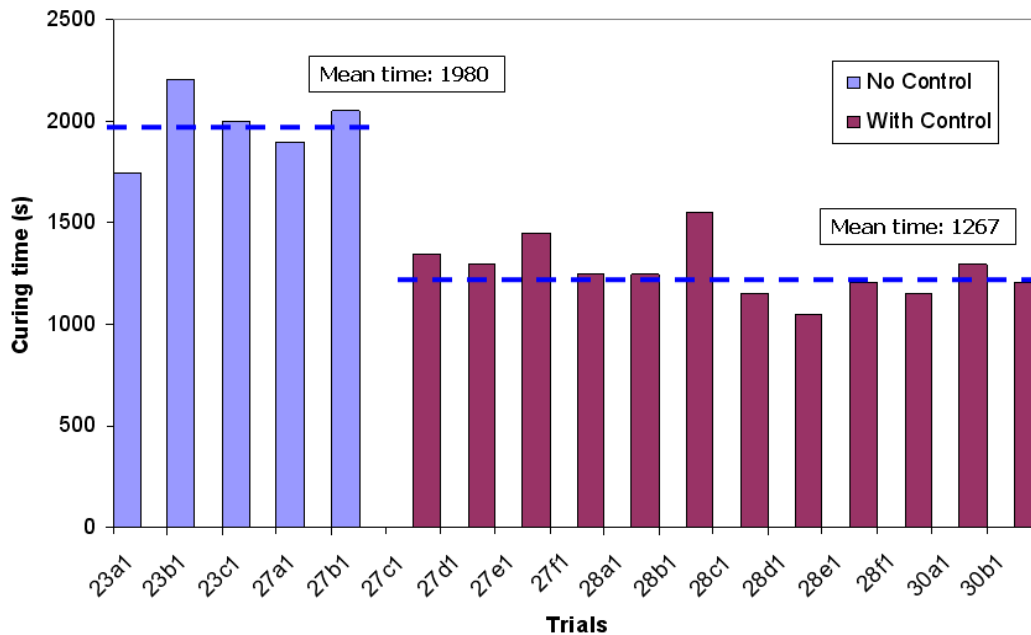


Fig.7. Curing times for an RTM production line before and after cure monitoring and control shows mean cure cycle time acceleration of 36%.

6. 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 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.

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