In-situ process monitoring and quality control for advanced liquid composite moulding

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INTRODUCTION

An electrical monitoring system has been used for the in-situ process monitoring of composite manufacturing in aerospace. The correlation of the electrical resistance with the viscosity and the degree of cure evolution for aerospace resins proves its accuracy and repeatability for the cure prediction up to the end-of-cure while the comparison to other dielectric systems in this application area shows its superiority with respect to performance, applicability, versatility and cost. Two types of durable non-intrusive sensors have been benchmarked for the sensing of resin ageing, resin arrival, viscosity evolution as well as curing. The sensor's feedback is recorded and processed by the system providing real-time material state information (viscosity, degree of cure etc.). Simultaneous monitoring of resin viscosity and electrical resistance of monocomponent resins has demonstrated the potential to provide in-situ real-time information of the resin viscosity during processing but also to identify and control issues such as resin or prepreg ageing.

PROCESS MONITORING USING DC SENSING

In dielectric cure monitoring, a range of sinusoidal electrical excitations are applied to the electrodes of a sensor 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 [1]. On the other hand, the DC conductivity for process monitoring has been studied [2, 3, 4] with no significant applications in industrial monitoring of the complete composites processing.

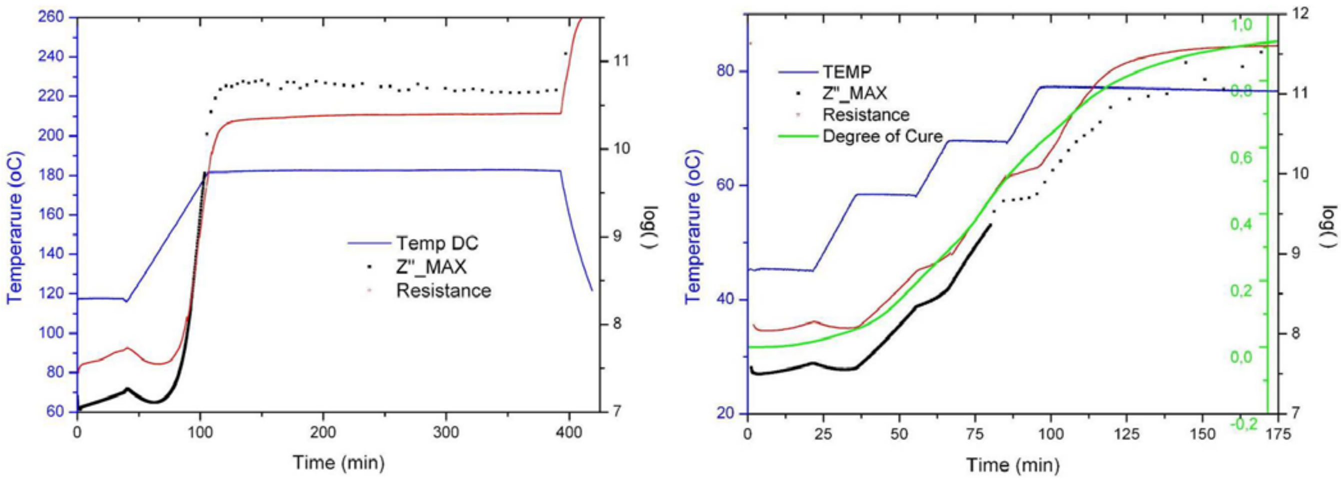

Fig.1. Simultaneous dielectric max imaginary impedance (Z''max), DC resistance, temperature and theoretical prediction of degree-of-cure of curing of a high-temp (left) and mid-temp (right) epoxy systems.

Recently, a new DC-based process monitoring system was presented [5] with a clear focus on composites manufacturing industrial applications. The new system measures the materials' resistivity and temperature using durable non-intrusive 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 bothe using durable sensors showed the superiority of the DC sensing which is observed particularly after gelation where conductivity is measured by the DC-system in a more reliable manner with reduced sampling time. As can be seen in Fig.1 (left) in the curing of a monocomponent resin at high temperature both monitoring systems advance almost in parallel but after gelation the sampling time of the dielectric system increases significantly due to the scanning of very low frequencies (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 temperatures as depicted in Fig. 1 (right). Obviously, there is a difficulty on the dielectric system to measure over than 0.80 degree of cure. On the contrary, the measurement quality of the DC monitoring system is very stable even at the end-of-cure region and at curing temperatures as low as 20^{o}C. Furthermore, the DC sensing is relatively cheaper and requires simpler sensors which can be more flexible in geometry 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 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 sensors used in this study had an outer diameter of 16 mm and were flash mounted into the tool. The sensor had an integrated temperature sensor providing the temperature close to the polymer which is absolutely necessary for the calculation of the material's state in conjunction with its conductivity.

QUALITY CONTROL

One of the major disadvantages of composites over homogeneous materials e.g. metals is their complex, delicate and non-forgiving manufacturing process. If the concept of eliminating the process deviations is adopted by using fully controllable materials and equipments such as prepregs and autoclaves the quality is ensured but the manufacturing cost is high. However, the need of lowering the cost and increasing the complexity have driven to more advanced manufacturing techniques such as RTM and vacuum infusion. Nowadays, the wide spreading of composites and the need of cost reduction have pushed materials and knowledge to their limits so the need for automation is necessary to increase productivity and minimise scrap. Especially the latter is very important as recycling and component costs are very high especially for carbon fibre parts. Furthermore, in order to reduce manufacturing costs, production technology has moved from autoclaves and prepregs to heated moulds, dry fabrics and liquid resins. These changes introduced several deviations in the manufacturing process which must be kept under control for optimal performance. 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 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 may cause process deviations. Furthermore, the discard of expired resins besides their economic impact can have large environmental burden so the use of such resins would save money and reduce environmental

pollution. The monitoring of the resin's electrical resistance with a non-intrusive sensor in the resin pot can reveal the exact state of the resin and can provide the right feedback to a control system in order to maintain product quality. As can be seen in Fig.2 and Fig. 3, fresh and thermally-aged monocomponent epoxy resins have been mixed in various mixing ratios and tested in a simulated resin pot application and in a representative injection cycle measuring simultaneously viscosity and resistance, respectively. In Fig.2 the measured resistivity at 80^{o}C is shown against a variety of mixing ratios between fresh and very aged resin batches in repetitive and random trials. According to these trials the resin quality can be safely concluded by measuring the resistivity of the resin.

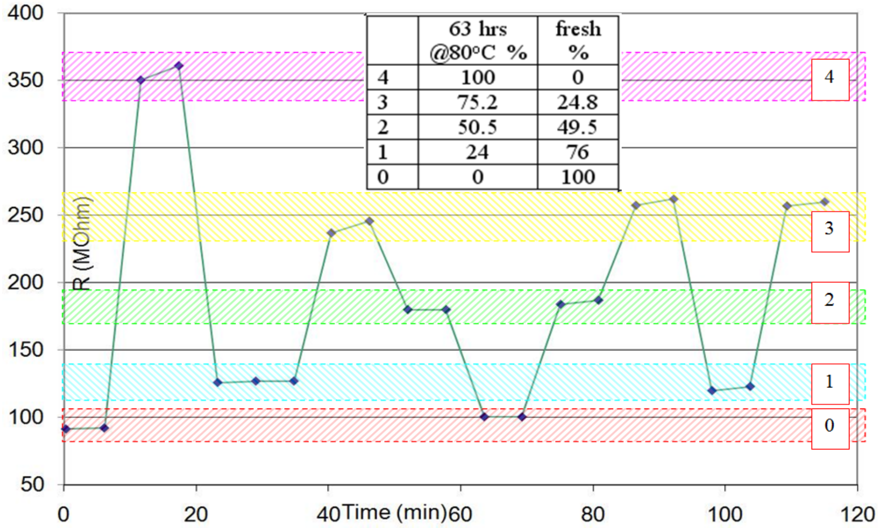

Fig.2. Fresh and thermally-aged monocomponent epoxy system in various mixing ratios in a simulated resin pot produced distinctive and reproducible measurements of their corresponding resistivity.

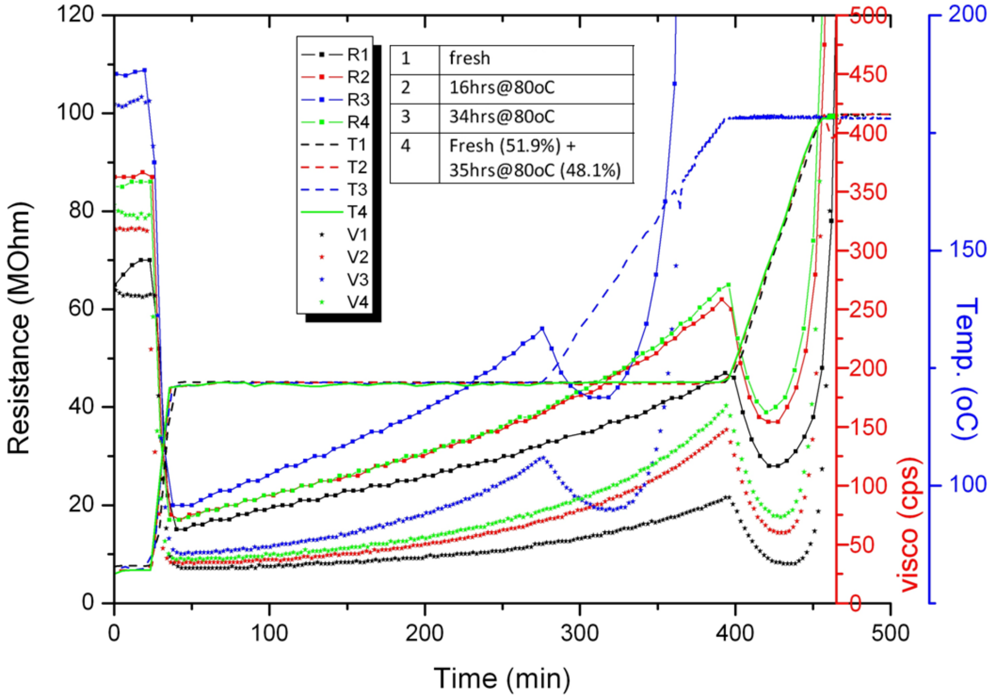

Fig.3. Resistance (R1, R2, R3, R4), temperature (T1, T2, T3, T4) and viscosity (V1, V2, V3, V4) simultaneously measured in simulated production cycles of fresh (case 1) and thermally aged (cases 2 and 3) monocomponent epoxy resin batches and their mixture (case 4).

Simulated production cycles (80°C-120°C-180°C) were executed with fresh and aged resins in a Brookfield viscometer equipped with a durable resistance sensor at the bottom of the cavity. The resin’s specifications allow for maintaining the resin at 80°C for 24 hours. In these trials a fresh resin batch (case 1 in black colour), an ‘in-specs’ aged resin (case 2 in red colour), an ‘out-of-specs’ aged resin (case 3, blue colour) were tested and an equal mixture of case 1 and case 3 resins.
As can be seen in Fig.3 during the initial stage at 80°C the resins’ viscosity is directly related to the measured resistance and are in accordance to their ‘degree of ageing’. As expected the ‘degree of ageing’ affects directly the viscosity of the resins at 120°C (injection phase) as well as the time where resins’ viscosity is below a threshold. Furthermore, the direct correlation of the viscosity and the resistance of all the resin batches should be highlighted allowing for the secure prediction of the resin viscosity by measuring the resistivity and the temperature of the resin. Finally the behaviour of the mixture (case 4) coincides well with the equally aged resin batch of case 2 both in viscosity and resistance. Preliminary trials at industrial level showed good performance of the durable sensors.

CONCLUSIONS

An innovative process monitoring system has been used successfully 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.
Next step will be the installation of the pot sensors and systems at industrial production for real-time monitoring of resins’ quality. The use of the monitoring system in autoclave environment for real-time monitoring of resin arrival and cure is also under investigation.
It must be highlighted that in other industrial scale applications such as automotive a significant acceleration of the production cycle was made possible. Other process deviations that can be picked up by process monitoring can be the quantity of other additives in the resin such as thermoplastics or the nano-size additives.

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