ONLINE INTELLIGENT CURE MONITORING FOR AEROSPACE APPLICATIONS

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ABSTRACT

The constant growth rates in the field of fiber composite materials show a potential that has been rising for years. Fiber composite materials are contributing to this growth. Above all, autoclave and RTM processes for the production of large components and large series components for the aerospace industry have become essential in the area of fiber composite construction. Therefore high quality and safety requirements apply to the products produced, as well as a continuous need for economical efficiency improvement for components and processes. The detection of these components and process relevant information is one of the key points for improving quality and efficiency. An intelligent process monitoring system has been employed for the in-situ process monitoring of the manufacturing of aerospace composite structures. The simultaneous measurement of the resin's temperature and electrical resistance allow for the real-time prediction of the viscosity, the degree of cure and the T_G as well as deviations in mixing ratio. The correlation of the electrical resistance with the viscosity and the degree of cure evolution of widely-used aerospace resins proves the accuracy and the repeatability for the cure prediction up to the end-of-cure. Due to the different application areas a common ultrasonic system was used. It will discuss how the measurement data of both methods can be correlated and supported.

1. INTRODUCTION

Due to their special properties, fibre composites are an established material in industry. The high stability and tensile strength at low weight are of great importance, especially in medicine, sports, automotive industry and aerospace. Because of their advantage properties, fiber composites components are of increasing interest, as well as of increasing production numbers. They are used in structural components in many areas of aviation. Therefore the production of components in space travel is subjected to high safety requirements. It is of great importance to obtain precise knowledge about the condition of the component during the process. This applies to flaws, but above of all to the degree of cure and the mixing ratio of the fiber matrix. To make an accurate statement about the condition of the component, the *German Aerospace Center* in Stade investigates different sensor technologies. In cooperation with *Synhesites Innovative Technologies L.T.D.*, the *DLR* is using the electric resistance to get information about the component during the process. The focus is on sensors from *Synthesites*, which are based on electrical resistance. In addition to this the ultrasonic sensors of *Grasse Zur Composite Testing* are used. Due to the fact that the two technologies work with different mechanisms of action, their fields of application differ. Concerning to this, sensors from *Synthesites* are working reliably, but requires a direct contact to the resin. On the other hand, ultrasonic sensors are non-contact sensors, but cannot be used in open-mould components, due to the requirement of two sensors. This gap is attempted to close. [1, 2]

2. CURE MONITORING SYSTEM

2.1 Process Monitoring using DC-based Sensing Tools

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. On the other hand, the DC-measured conductivity was studied [3, 4] but only in 2009 a new DC-based process monitoring system, the Optimold system from *Synthesites* [5], was presented with a clear focus on industrial manufacturing. The Optimold system measures the resin's resistivity and temperature using specialised 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 with the measured resistance ranging from 10^5 Ohm up to 10^{14} Ohm. Comparison between the DC cure monitoring and commercial dielectric systems demonstrated the superiority of the DC sensing particularly with carbon fibres and after gelation where conductivity measured by the DC-based system is more reliable and robust. Furthermore, the DC-based monitoring is cheaper and requires simpler sensors which can be more flexible in geometry and robust. Last but not least, in contrast to the through-thickness measuring nature of the dielectric systems, the DC sensing is significantly 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 has an outer diameter of 16 mm and were flush mounted into the tool. The sensor has an integrated temperature sensor measuring the temperature close to the resin which is absolutely necessary in conjunction with its conductivity for the calculation of the resin state.

2.2 Correlation of Electrical Resistance with Processing parameters

In liquid composite moulding viscosity is the most critical property of the resin at the injection where viscosity should be maintained below a certain level to ensure good product quality. Using the DC-based monitoring system it is possible to monitor this viscosity in real-time and in the mould in order to check whether fibre impregnation is progressing as planned. Furthermore, the direct correlation of the viscosity and the resistance of all the resin batches should be highlighted allowing for the secure estimation of the resin viscosity by measuring the resistivity and the temperature of the resin in the mould [6]. As can be seen in fig.1 (left) in a combined measurement of electrical resistance, viscosity and temperature of a neat resin, viscosity and resistivity are dropping simultaneously when temperature is rising while this behaviour was confirmed in four consecutive repetitions using the different resin batches. Using such sensor the increase of resin's viscosity can be observed in real-time and possible control actions can be decided accordingly to speed-up the injection.

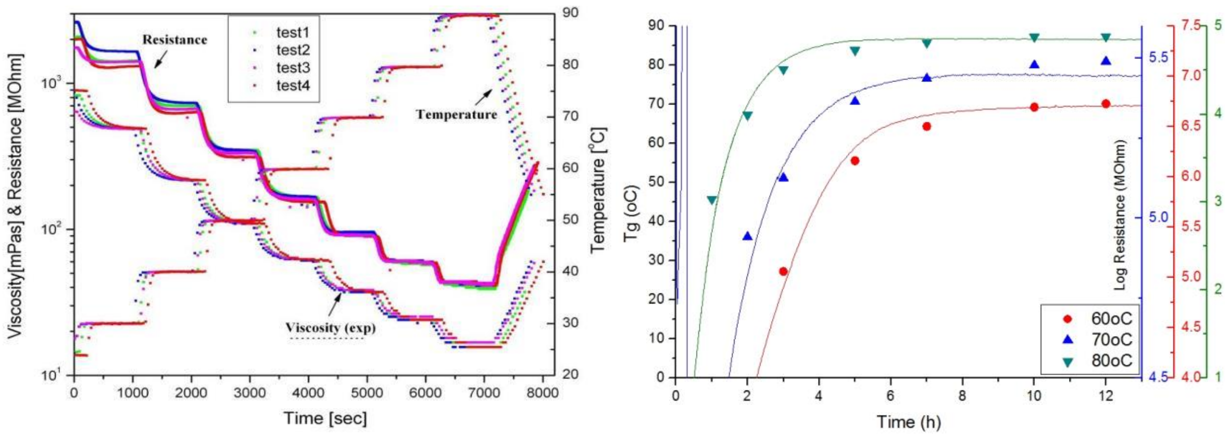

Figure 1 - Left: Correlation of viscosity and resistivity with changing temperature for a neat epoxy resin. Right: Electrical resistance of an epoxy system at three different isothermal cure cycles and the corresponding Tg evolution as provided by the resin supplier

After the end of the injection and the beginning of the heating ramp it is important to identify the gelation point and the development of the Glass Transition temperature. The good correlation between the electrical resistance and the Tg build-up in the curing of thermoset resins has been extensively studied (see for example [7]) and proved for a range of resins such as the typical epoxy system the curing of which is shown in fig. 1 (right) for three different temperatures.

In the case of monocomponent resins or prepregs which are used mainly in aerospace, the thermal aging of a resin batch when defrosted and/or mixed with fresh resin may cause filling problems. 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 to ensure product quality.

Furthermore in the case of two (or more) component resin system it is possible to reveal mixing ratio deviations using the Optimold system either based on the different electrical properties of each component or measuring directly the speed of reaction. In the first option by measuring the final resistivity of the mixed resin it is possible to identify which of the components is missing or deviates significantly from the expected resistivity of the mixture [8].

3. EXPERIMENTATION

To study the cure monitoring performance of the DC-based dielectric and the ultrasonic systems several trials have been performed with two popular aerospace resins: the RTM6-2 low viscosity two-component resin and the M21 prepreg system both from *Hexcel Composites*. The test setup depends on the state of the semi-finished product to be monitored. As well in dielectric cure monitoring as in ultrasonic cure monitoring the usage of neat resin is being preferred if available. For the DEA testing the setup of a simple open mold with a durable cure sensor in an oven has been used. Hereby the main problem is a reproducible application of temperature and sample geometry. As prepregs are frequently used in aircraft application it is also a main goal to monitor such materials with the existing monitoring systems. Unfortunately, such materials as the ones used in this paper are rarely available as separate components so the test setup bears additional requirements regarding the treatment of the semi-finished product.

3.1 RTM 6

The RTM6 monocomponent resin system from Hexcel has been already extensively investigated in its use in aircraft manufacturing reaching Tg higher than 200°C. However, its two component counterpart, RTM6-2, has not been studied as it is considered an identical resin to RTM6. In the current study both the curing behavior as well as the mixing deviations of RTM6-2 are being studied. In Figure 2 a standard cure cycle has been monitored using Optimold cure monitoring system where the evolution of Tg has been also shown.

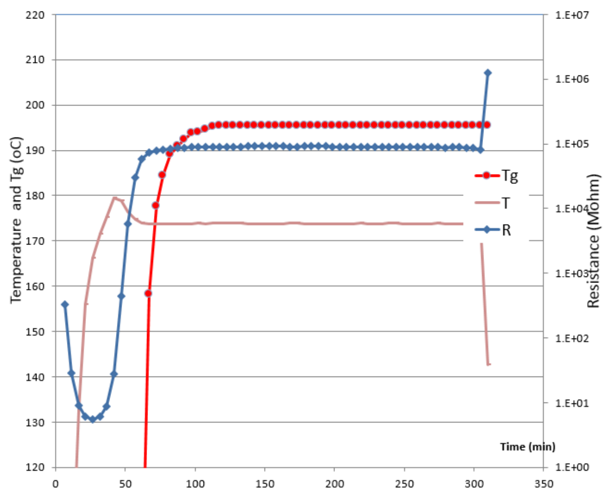

Figure 2 - Measured resistance of an almost isothermal 175°C curing of neat RTM6-2 resin.

To ensure the reproducibility of the results a test setup for the open mold application was designed with a defined diameter. The ground plate includes a hole where the durable sensor is applied with direct contact to the mold. The tool and the resin are preheated at 80°C in a conventional furnace. As soon as the tool temperature reaches 80°C the measurement is started. Right after a defined resin volume is applied to the tool to ensure that the thickness of all resulting disc-shaped parts is identical. The furnace is then immediately heated to the determined isothermal temperature until vitrification and beyond. The samples resistance and temperature are tracked with the Optimold software and correlated afterwards with a kinetic model of RTM6-2.

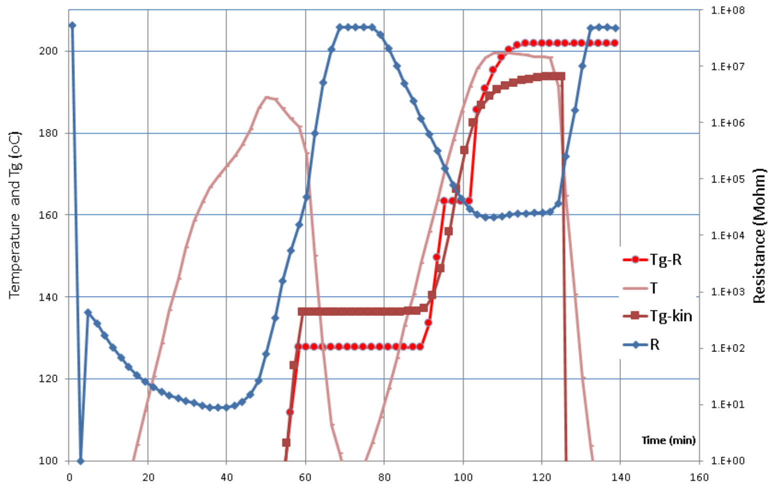

Figure 3 - Monitoring of a peculiar non-isothermal cure cycle of neat RTM6-2 resin: The estimated Tg is correlating well to the Tg predicted by a kinetic model.

In Figure 3 a more peculiar cure cycle has been tested in order to check the robustness of the cure monitoring method to estimate online the Tg evolution where the good correlation of the Tg estimation with the prediction with a kinetic model can be seen.

3.2 M21 prepreg

Using the Optimold cure monitoring system combined with a durable cure sensor, the M21 prepreg from *Hexcel Composites* has been studied. Two fresh prepreg samples and one aged sample were cured at 180°C. A comparison of the three samples shown in Figure 4 indicates that the fresh samples reach a much lower resistivity (as opposed to viscosity) with respect to the aged sample. Furthermore, the two fresh prepreg samples have produced quite repeatable resistivity curves which are very well correlated to the evolution of the Glass Transition temperature (Tg) as measured using a modulated DSC as can be seen in Figure 5.

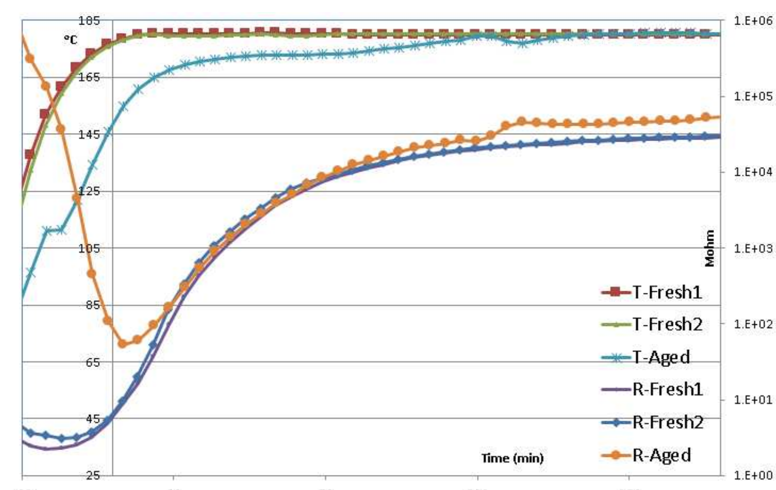

Figure 4 - Comparison of one aged and two fresh M21 samples cured at180°C. The lowest resistivity of the aged sample was kept at quite high levels with respect to the fresh samples corresponding to the higher minimum viscosity of the aged prepreg.

For the measurement a small prepreg layup with a defined number of plies is applied to the ground plate. Above the prepreg layers a layer of peel ply is placed and connected to a breather next to the prepreg layup where vacuum is applied. The whole layer structure is covered and sealed with a vacuum foil. The furnace is preheated to a temperature of 80°C and the measurement is started, when the tool is placed inside. In most cases the sensor is already in contact with the resin of the prepreg material once the measurement starts. Doing this the resins viscosity decreases due to the temperature increase and wets the sensor shortly after the tool is placed inside the furnace. Following the process is immediately heated again to the determined isothermal temperature until vitrification and beyond. The samples resistance and temperature are tracked with the Optimold software and correlated afterwards with Tg-measurements of M21 carried out in a DSC-device.

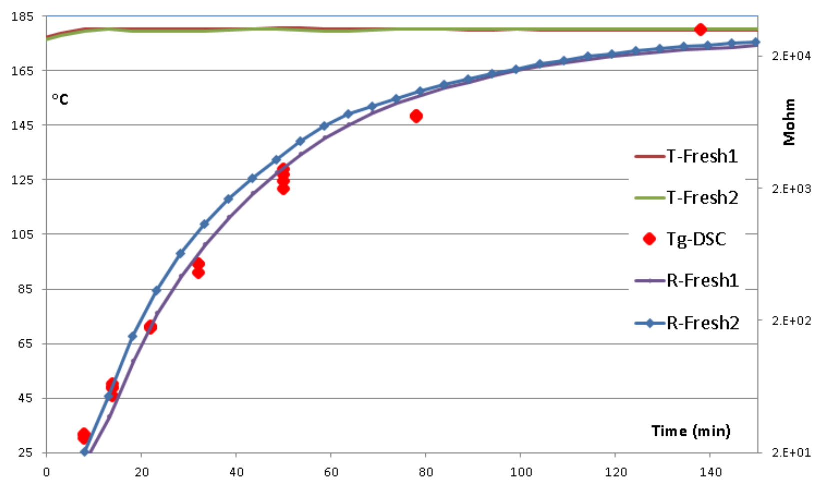

Figure 5 - Correlation of the measured resistance of the isothermal 180oC curing of two M21 samples and the measured Tg with respect to curing time.

According to these results it is expected that the real-time age estimation of the prepreg is possible as well as the online accurate estimation of the Tg and the degree of cure during curing. In this way it will be possible to terminate curing at a specific Tg for process acceleration or further processing.

3.3 Comparison US DEA RTM 6

The following trial compares the ultrasonic sound speed to the electrical resistivity during the non-isothermal curing of RTM 6. The data were measured by the Grasse Zur US Plus system in combination with Krautkreamer K4V1 sensors and the Synthesites Optimold system. Both data were measured simultaneously in a laboratory scale sensor tool, tempered in a furnace up to 180°C. Therefore, no defined heating rate can be mentioned. However, the aim was to identify the characteristic cure points and the relationship between both technologies, as shown in Figure 6.

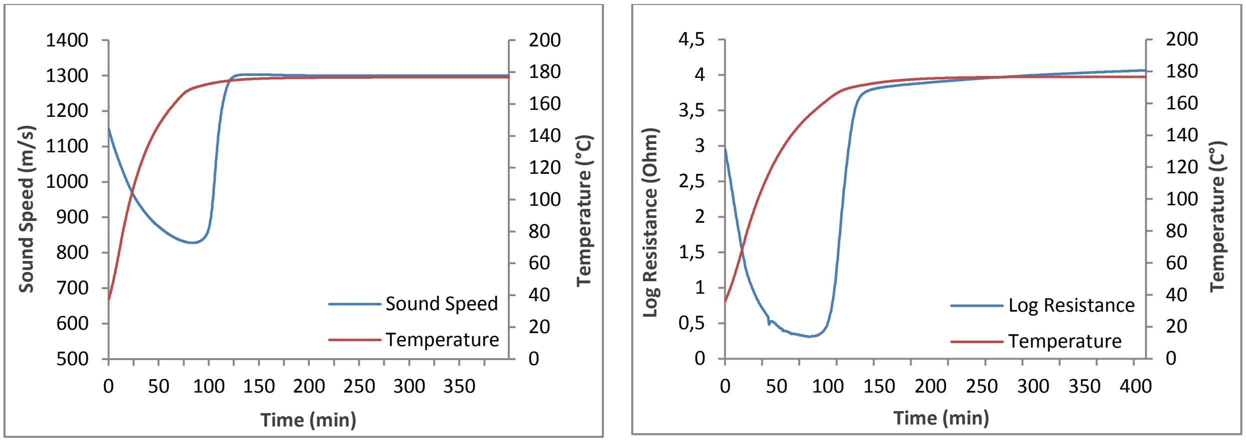

Figure 6 - Comparison of cure Monitoring of RTM 6 by means of ultrasonic and dielectric sensors

During the entire process and especially the heating ramp, there is an excellent correlation recognizable. However, during the isothermal part, the sound speed reaches a plateau and doesn`t increase further. On the other hand, the dielectric curve is further increasing during the isothermal part. That means, this part of curing can`t detect by the ultrasonic system. The dielectric sensors are very sensitive and because of this able to measure the ion mobility, which is still changing in this area. The ultrasonic sensors measure the sound speed and the elasticity, which stagnate in this part of the curing process.

4. CONCLUSIONS

Concerning to the comparison trial between the electrical resistivity and the ultrasonic sound speed a good correlation is observed. This has already been confirmed by Shepard et al. [9]. For the special application in an autoclave, both systems have advantages and weaknesses. The ultrasonic technology can measure over the entire thickness and can be used to draw conclusions on the modulus of elasticity. On the other hand, the dielectric sensors can only measure on the surface of the part via durable sensors or through-thickness through disposable ones and provides information about the degree of cure. The electrical resistance-based curing system from *Synthesites* is an already reliable operating system in the area of the neat resins and prepreg materials but requires direct contact with the matrix of the component. Hence, if contact with the component is not allowed several options exist to allow the cfrp manufacturer to use them in an optimal way. On the other hand, ultrasonic analysis is a non-contact system, but due to numerous physical conditions, it is a very complex system and requires an individual adaptation to the particular application. This adaptation relates to the method used and the sensors used in the context.

5. OUTLOOK

With regard to conventional aerospace components, especially to open mold components and autoclave processes, the outlook for this paper is to show what research and developments are to be expected. The detection of the degree of cure can, as already described, be investigated by means of ultrasonic and dielectric sensors. The two sensor systems have both advantages and disadvantages, as already described by Sheppard et al [9]. This can be used according to the current state of research and technology, to use both systems in a correlative manner. In order to be able to use the ultrasonic sensor system in this process, it has to be further developed. In collaboration with *Grasse Zur Composite Testing*, the ultrasonic-based pulse-echo process is currently under development. By using only one sensor there are a much longer signal delay and, based on the impedances, a lot of reflections. The tool-mounted puls-echo sensor acts as a transmitter and a receiver and enables the operation without influencing the surface. This system will be particularly important for open mold components in an autoclave process. Component-relevant data can thus be obtained online during an autoclave process. The challenge is the development of suitable sensors, because the sound transmission and evaluation is of fundamental importance. A sensor consists of a piezo ceramic, a damping mass and a sensor housing [10]. These components must be adapted for the particular application and can not be used in a standardized manner. Furthermore, in the field of dielectric sensor technology further TG modules are tested for different resin systems and the detection of the mixing ratio has to be developed. Due to the advantages and disadvantages and the resulting application areas, it can be helpful to use both sensor technologies in a correlative manner and to use these for a simulation if necessary. In this, the information of the respective sensors would converge.

6. REFERENCES

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