Industrial cure monitoring and control of the RTM production of a CFRP automotive component

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Abstract

A self-learning process monitoring and automatic control system has been developed to provide optimal and straightforward solutions for a wide range of resins and applications without particular investment and knowledge from the manufacturers' side. Three different liquid moulding applications were used as the testing bed for the technologies covering a wide range of processing conditions (polyesters and epoxies, fast and slow cure cycles, carbon and glass fibres) with products from automotive to composite bridges. At the first application in high-speed RTM, the new system did prove its advantages in monitoring and control an RTM epoxy CFRP production achieving acceleration of more than 10% in existing production and potentially up to 40% through an additional optimisation step.

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 [1, 2] but only recently a new DC-based process monitoring system was presented with a clear focus on industrial manufacturing. The system developed by Synthesites [3] measures the materials' 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 varying from 10^5 Ohms up to 10^{14} Ohms. Comparisons between the DC sensing and commercial dielectric systems both using durable sensors showed the superiority of the DC sensing particularly after gelation where conductivity is measured by the DC-based system in a more reliable and faster. 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 flush mounted into the tool. The sensor had an integrated temperature sensor providing the temperature close to the polymer which is absolutely necessary in conjunction with its conductivity for the calculation of the material's state.

2. Correlation of Electrical Resistance and Process Variables

Viscosity is the most important property for the first step of liquid composite moulding which is the filling of the mould cavity. During this step it is important to maintain the viscosity below a certain limit in order to ensure good product quality. Using this DC-based monitoring system it is possible to monitor this viscosity in real-time and in the mould in order to check that the fibre impregnation is progressing as planned. After that it is important to identify the gelation and the end of cure.

Figure 1 shows the simultaneous comparison between the measured viscosity, the degree of cure and Tg evolutions versus the measured electrical resistance for low-temp epoxy resin. Tests were performed at lab scale conditions show that the evolution of viscosity at the early stages of the process can be directly correlated with resistance. Then, there is a point where a sharp increase of viscosity occurs due to curing and the two curves start to diverge. This divergence becomes more obvious at the calculated gelification point at α=0.69 where the resistance curve starts to follow the calculated degree of cure and Tg. If we compare the 3 models with the signal, in this particular case, it can be concluded that from α=0 to α=0.3 the DC monitoring system provides information about the viscosity evolution and from α=0.3 to the end of the reaction the signal can be correlated with the evolution of the degree of cure or even better, with Tg. When vitrification occurs and reaction stops ($d\alpha/dt$=0), resistance curve flattens and depends only on temperature.

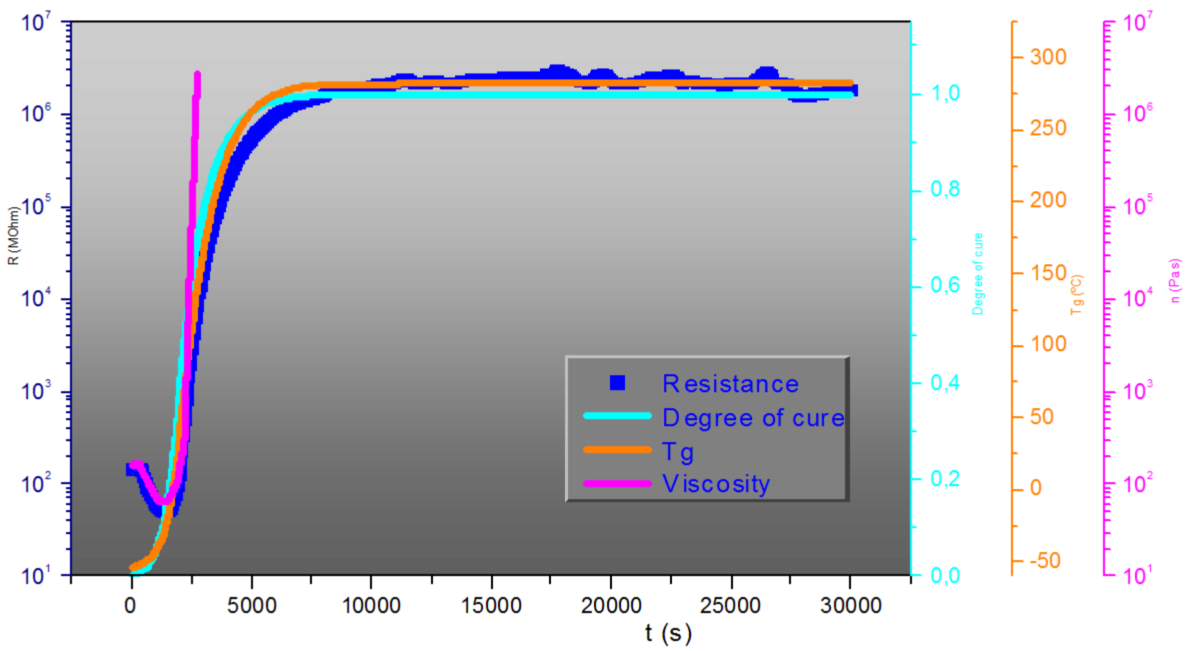

Figure 1. Electrical resistance, degree of cure, Tg and viscosity correlation at an isothermal curing of an epoxy resin.

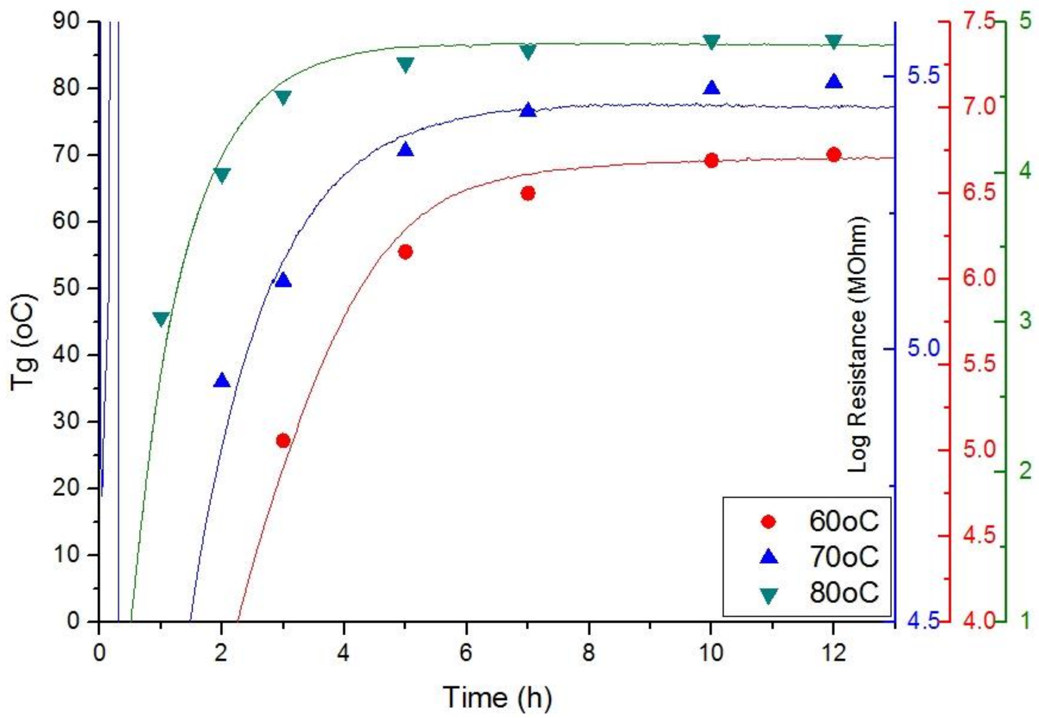

Figure 2. Electrical resistance of an epoxy system at three different isothermal cure cycles and the corresponding Tg evolution as provided by the resin supplier datasheet.

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 [4]) and is clearly demonstrated in fig. 2 for a typical epoxy system in isothermal cures at different temperatures. In the case of monocomponent resins 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. As can be seen in fig. 3, fresh and thermally-aged monocomponent epoxy resins were mixed in various mixing ratios and tested in a simulated resin pot and a representative injection cycle (80^{o}C-120^{o}C-180^{o}C) measuring simultaneously the resin's viscosity and resistance. The resin supplier specifications allow the resin at 80^{o}C for 24 hours. In these trials a fresh resin batch (case 1 in black), an 'in-specs' aged resin (case 2 in red), an 'out-of-specs' aged resin (case 3 in blue) and a 50-50 mixture of case 1 and case 3 resins (case 4 in green) were tested. As can be seen in the same figure, during the first stage at 80^{o}C the resins' viscosity is directly related to the measured resistance and are in accordance to their 'degree of aging'. As expected the 'degree of aging' affects directly the viscosity of the resins at 120^{o}C (injection phase) as well as the time where resins' viscosity is below a threshold. The behaviour of the mixture (case 4) coincides well both in viscosity and resistance with the equally aged resin batch of case 2. 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 [5].
In the case of a two-component epoxy system, before starting the injection the mixing ratio can be checked in-situ using the same DC-sensing system in order to avoid injecting resin without or with low hardener content. As can be seen in fig.4 there is significant difference between the resistance measured without any hardener (70 MOhm) and resistance measured for the recommended mixing ratio (100:34) (34 MOhm) so the real-time verification of the mixing ratio is possible for immediate actions.

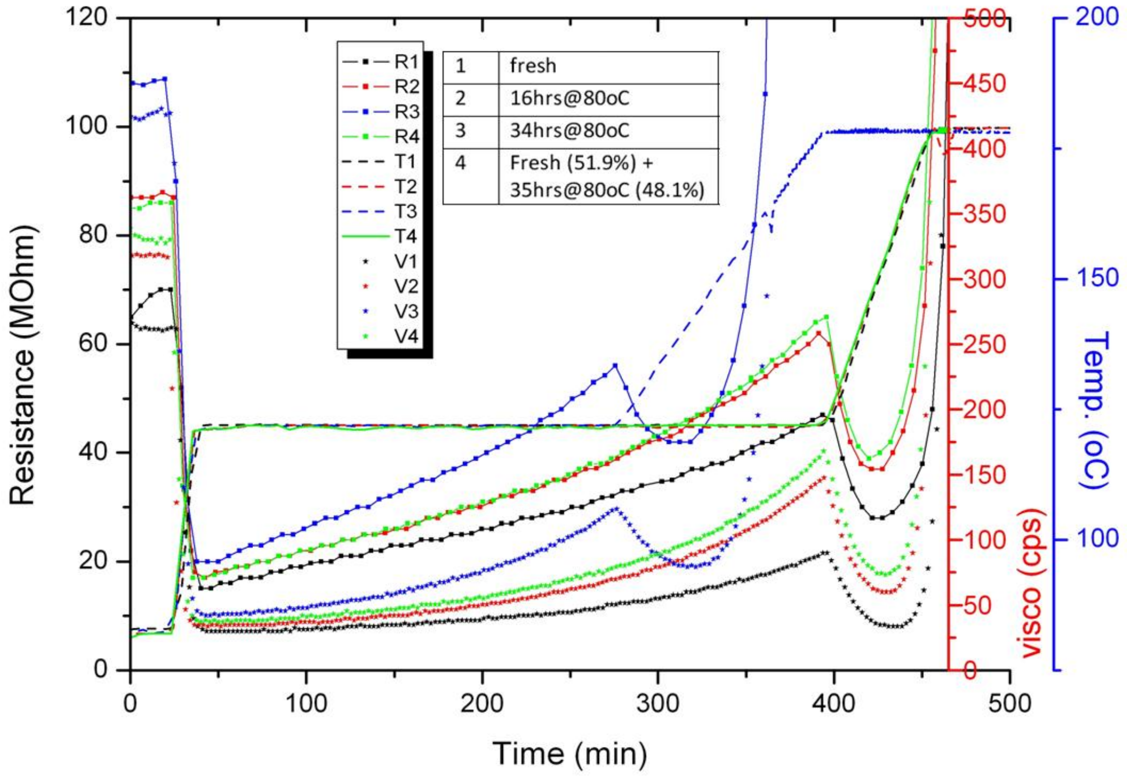

Figure 3. Resistance (R1, R2, R3, R4), temperature (T1, T2, T3, T4) and viscosity (V1, V2, V3, V4) measured in simulated production cycles of fresh (case 1) and thermally aged (cases 2 and 3) monocomponent epoxy resin batches and their mixture (case 4).

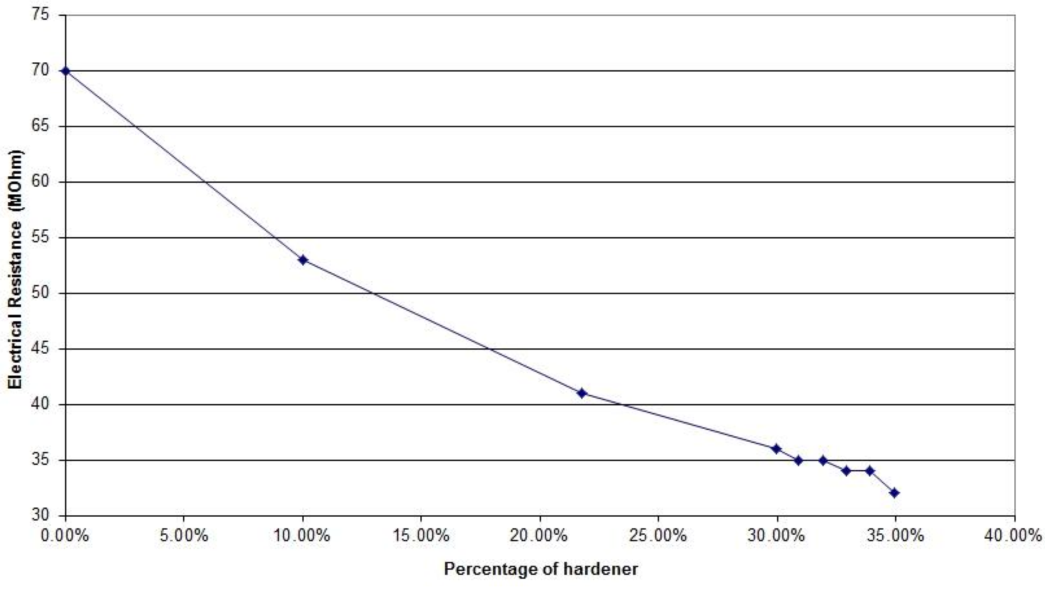

Figure 4. Electrical resistance of an epoxy/ hardener system at various mixing ratios.

3. Self-learning and Prediction with Neural Networks

In the bootstrap aggregated neural networks, several neural network models are developed to model the same relationship. Instead of selecting a “best” single neural network model, these individual neural networks are combined together to improve model accuracy and robustness. The overall output of the aggregated neural network is a weighted combination of the individual neural network outputs. This can be represented by the following equation:

$$f(X) = \sum_{i=1}^{n} w_i f_i(X) \tag{1}$$

where $f(X)$ is the aggregated neural network predictor, $f_i(X)$ is the ith neural network, w_i is the aggregating weight for combining the ith neural network, n is the number of neural networks, and X is a vector of neural network inputs. The aggregating weights can be obtained using a number of ways, such as simple averaging, i.e. the stacked neural network output is an average of the individual network outputs, or using principal component regression (PCR). Instead of using constant stacking weights, the stacking weights can also dynamically change with the model inputs. Another advantage of bootstrap aggregated neural network is that model prediction confidence bounds can be calculated from individual network predictions. Neural network models were developed using industrial data as provided by the industrial partners of the iREMO project. Based on data from three days of process operation were used to build and validate the neural network models. The data set contains 94 runs where constant curing temperature policy was applied. Variations in mould temperature exist due to exothermal effect. Data from 15 runs were selected as the model building data, which were randomly split into training set (50%) and testing set (50%). The final developed model was tested on all other runs. The developed neural network based dynamic model is of the following form:

$$logR(t)=f(logR(t\text{-}1),\ logR(t\text{-}2),T) \tag{2}$$

where R is the resistance, T is the average temperature during the first 4 minutes, t is discrete time, $f()$ is a nonlinear function represented by the neural network.

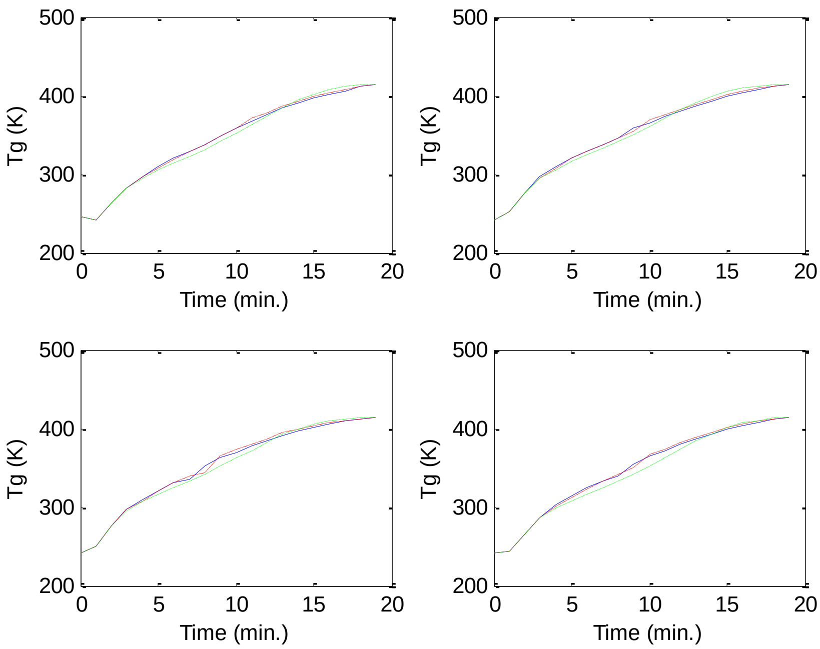

Figure 5. Dynamic neural network model predicted T_g on 4 unseen runs

A bootstrap aggregated neural network containing 30 single hidden layer neural networks was developed. The number of hidden neurons in each network was determined through cross validation. The networks were trained with Levenberg-Marquardt algorithm with regularization and early stopping. In figure 5 the actual predicted Tg values are shown as the solid lines, one-step-ahead predictions are shown as dash-dotted lines, and multi-step-ahead predictions are shown as the dashed lines. It can be seen that the neural network one-step-ahead predictions are very accurate. The multi-step-ahead predictions are also very accurate, though not as accurate as the one-step-ahead predictions.

4. Process Optimisation

One of the strongest advantage of using the system is the optimization of the moulding process as can be seen in the Sotira case. The first set of trials was performed at the real moulding set-up and there were focused on the optimisation of the process. Based on the numerical process optimisation results it was shown that the moulding temperature should be increased to 135^{o}C in order to reduce processing time significantly. Apart from verifying this performance the only question was to check whether the proposed set-up cause dimensional or surface quality issues. To check this it was decided to dedicate a whole production day in consecutively increasing temperature during the day to check the validity of the optimal recipe. In table 1 the temperature increase during the day measured by the Optimold monitoring system is shown with respect to the moulding cycle number. As expected the overall measured resin resistance decreases with temperature although the resistance rate increases which can be clearly seen in the dR curves also increases with temperature showing that the reaction rate has been also increased. Especially when focusing at the end of the cycles the dR (figure 6) have been already decreased considerably after 13 minutes for the highest temperature cycles whereas for the low temperature cycles this limit is reached after 20 minutes.

Case	20_1	20_2	20_3	20_4	20_5	20_6	20_7	20_8	20_9	20_10
Mould Tempe-rature (^{o}C)	112	116	119	119	119	126	129	128	131	132

Table 1. Moulding temperature during the corresponding curing cycle.

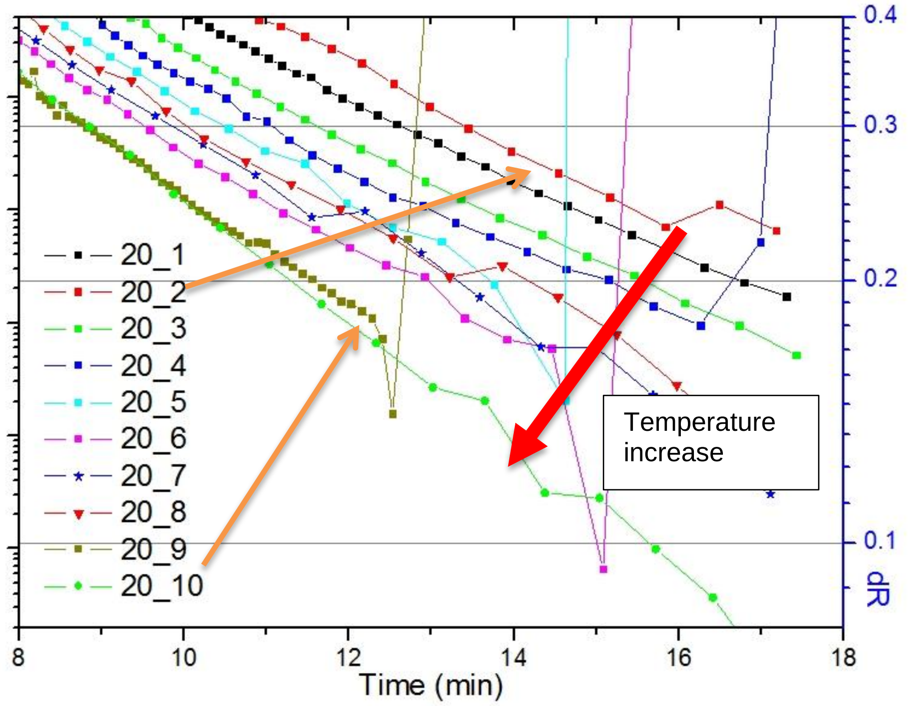

Figure 6. Focus on resistance derivative at the end-of-cure

5. Real-time Quality and Process Control

Cure control is necessary not only for the shortening of the cure cycle but also for the real-time quality monitoring and/or for unexpected events. The key issue in this target is the real-time measurement of the real state of the resin that cures in the mould. It has been shown that using the DC-based cure monitoring system from Synthesites there is not only a qualitative correlation between resistivity and degree of cure or glass transition temperature (Tg) but, more important, a quantitative one. As discussed before for a typical epoxy system at various isothermal cycles there is a certain correlation between resistivity and Tg evolution. This correlation has been materialized in the iREMO project to calculate in real-time and in-situ the Tg of the matrix. For example it has been shown [6] that a small variation of mixing ratio around the optimal one affects also the curing speed. So, as can be seen in fig. 7, by varying the mixing ratio of the resin around the optimal one (100:17) the Tg evolution can be calculated in real-time so that the opening of the press can be decided accordingly i.e. to extend the cure cycle if the mixing ratio is below the nominal one (100:15 and 100:14) or to shorten the cure cycle in the opposite case (100:20).
The control program and a user-friendly interface have been developed in Labview (fig. 6) providing a powerful and easy-to-use environment for process control. The use of the control system can provide significant speed-up of the curing especially if combined with exotherm simulations and cure cycle real-time optimisation [7].

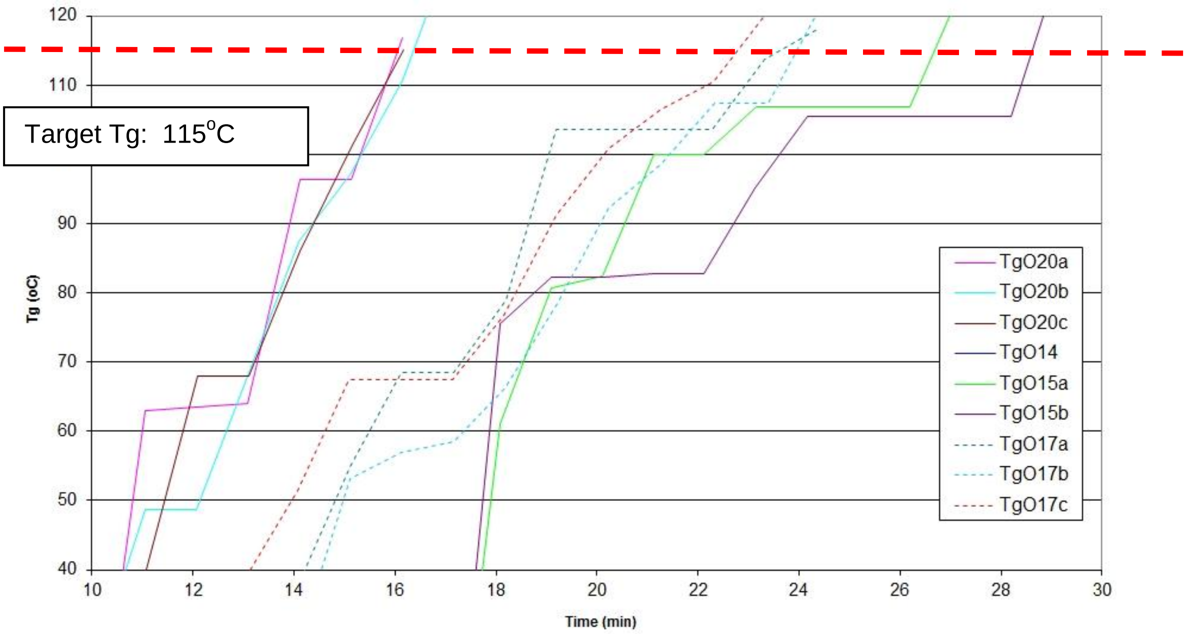

Figure 7. Real-time Tg evolution as calculated by the control algorithm developed in the iREMO project for various mixing ratios (100:14, 100:15 and 100:20) around the nominal one (100:17).

6. Conclusions

An innovative process monitoring system has been used for the real-time control of filling and curing in the lab but also in real industrial conditions. 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 exceptional stability and robustness without requiring any special treatment from the staff. The development of a control system can provide real-time information about the Tg evolution and the degree of cure with good accuracy and repeatability.

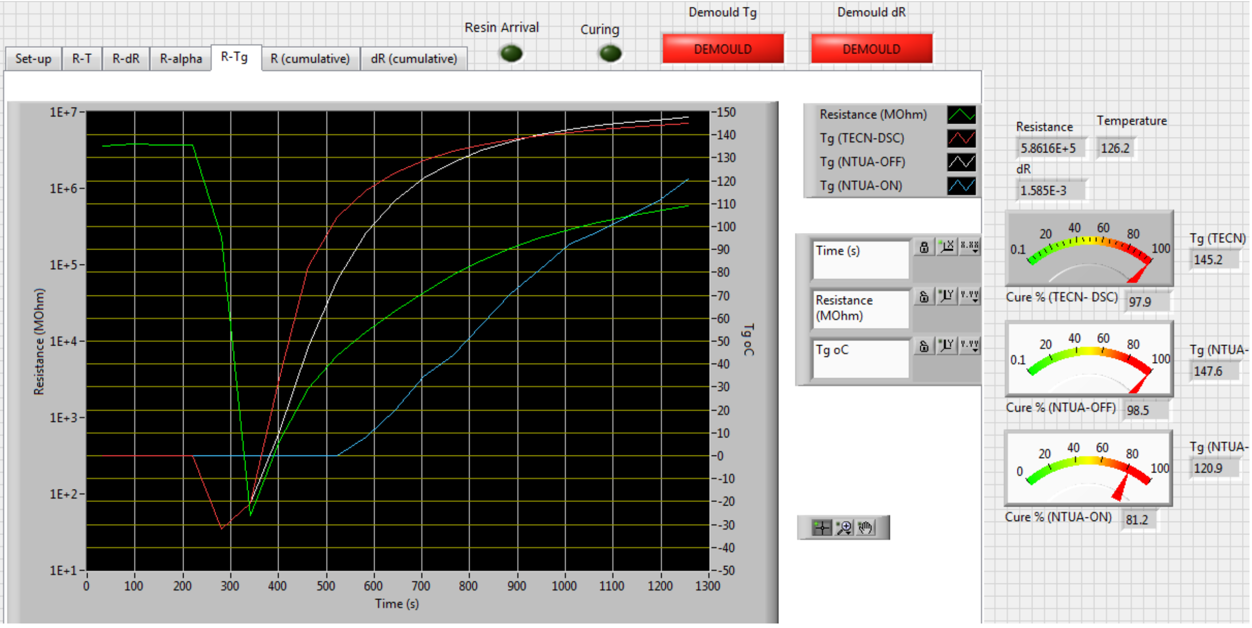

Figure 8. Screenshot from user-interface of the control showing the two demoulding decisions on the upper right corner as an indication for the operator to open the press.

7. Acknowledgements

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