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In-Line Monitoring of a Curing Process With a Multisensor Concept

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Abstract—In the production of electrical isolators based on thermosetting polymers, material is cured in a mold under temperature and pressure. As these products undergo a highly exothermic curing reaction during production, an appropriate production control which considers the effects of the curing reaction is crucial to ensure highest product quality. The objective of determining the physical parameters such as temperature and pressure in the workpiece during production was achieved by including sensors in a mold cavity. The manufacturing process from the inflowing material to the hardened and finished workpiece was recorded with data loggers. Three different sensors successfully determined the temperatures and pressure as follows: Pt1000, NTC10k, and a digital combi sensor. The determined sensor values will serve as input parameters for the creation of a simulation model, which is to represent reality based on the measured data.

Index Terms—Sensor systems, curing, electrical isolator, in-line monitoring, keyhole technology, multisensing.

I. INTRODUCTION

An isolator is an electrical component with very high resistance and high dielectric strength. Due to this property, isolators are used, for example, in transformers, high-voltage power lines, trams, or trains. With the isolator attached as an intermediate piece, high-voltage lines can be laid without current flowing between the attached line and the Earth. Environmental influences, such as rain, dirt, or snow can form an electrically conductive film on the isolator [1], [2], [3]. Therefore, most isolators have a ribbed structure to minimize leakage currents by increasing the path through the ribs, which reduces the likelihood of short-circuits. A disadvantage of, e.g., ceramic isolators is their brittleness, which is why polymers can be used as an alternative. The good thermal, chemical, and mechanical properties of highly reactive resins, such as thermosetting materials, are well suited for the production of isolators. Electrical isolators based on thermosetting polymers are subject to a highly exothermic curing reaction during production. The aim of the research is to use sensors only during process development in order to find a suitable production process and to know the effects of the curing reaction at different points in the cavity of the mold [4], [5], [6], [7]. The parameters temperature and pressure, which are measured directly in the cavity filled with material, play a key role for the curing process. Given the limited space and the need to seal the mold during production to avoid leakage, when filling the mold with the liquid resin, the measurement tasks under these harsh conditions are challenging. In this letter, a multisensor concept is presented which, in addition to the standard temperature measurement, specifically shows the use of a small pressure sensor which, enclosed in material, represents the entire process up to the cured product and provides further information. Since temperature measurement is also very important, three different temperature sensors, Pt1000, NTC10k, and an I2C digital sensor, have been tested to suitability and are compared under the same measuring conditions. Both, in-line pressure and temperature information, provide improved monitoring of the curing process [8], [9], [10], thus serving as a basis for creating a digital twin using finite element (FE) simulation.

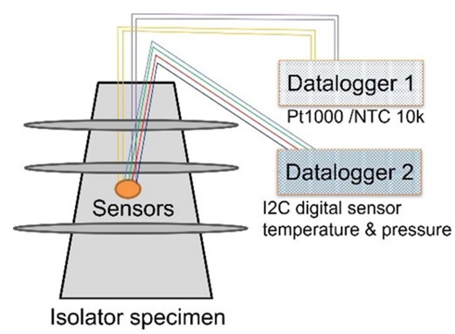

Fig. 1. Schematics of the measurement setup.

II. METHODS

A. Measurement Setup and Sensor Concept

The concept for connecting the sensors to a data logger is presented in Fig. 1. A mold cavity is equipped with sensors that lead to the respective data logger via thin wire lines. The resistance values of the temperature sensors are recorded at data logger 1, the values of the digital sensor for temperature and pressure via I2C protocol at data logger 2, both with a recording interval of 2 s.

B. Sensors for the Setup

First, a negative temperature coefficient (NTC) resistor was selected as a suitable sensor for the measurement task in the mold. The specs of the used sensor are: Tru Components; 10-kΩ thermistor; type: DHT0B103F3553SY; range: −40 to +200 °C; B value: 3550 K; dimensions: Ø2 × 4 mm; Tolerance: ±2 °C; and price per piece ∼1 €.

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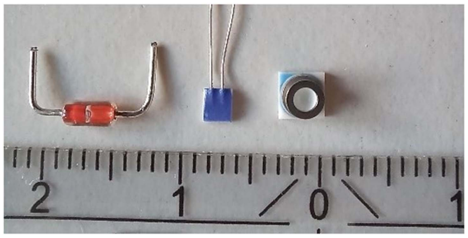

Fig. 2. Different sensors in use for temperature, NTC10k (orange) and Pt1000 (blue), as well as temperature and pressure with a digital I2C combi sensor.

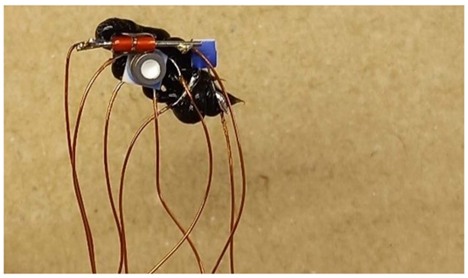

Fig. 3. Multiple sensor unit glued with temperature stable silicone.

Second, a standard platinum sensor Pt1000 was selected, which can be used at temperatures up to 500 °C. The specs of the used sensor are: Heraeus; 1000-Ω thermistor; type: M222; range: −70 to +500 °C; temperature coefficient 3850 ppm/K; dimensions: 2.3 × 2.1 × 0.9 mm; Tolerance: ±1.3 °C; and price per piece ∼ 1.50 €

As the third choice, a digital pressure–temperature combination sensor with I2C bus was used, which is only specified up to 85 °C, but in the experiment, it was able to withstand temperatures up to 200 °C. The specs of the used sensor are TE connectivity; type:MS5837-30BA; dimensions: 3.3 × 3.3 × 2.75 mm; range: 0 to 30 bar; −20 to +85 °C; Tolerances: Temp: ±0.6 °C; Pres: ± 30 mbar; and price per piece ∼7 €.

In Fig. 2, the used sensors are shown in a direct comparison and on a centimetre scale. The NTC10k and Pt1000 thermistors are easy to connect, since each sensor exhibits only two wires, whereas the digital sensor needs four wire connections.

In order to fix the sensors in close proximity and in exact position with respect to the mold, as well as to create strain relief for the connecting cables, a temperature-resistant silicone adhesive was used (UHU - high temperature silicone, resistant up to 180 °C). The respective "multiple sensor unit" is shown in Fig. 3 and covers an area of approximately 1 cm^2. The insulated wires, connecting the sensors to the data loggers, are made of enameled copper wire, each with an outer diameter of 0.224 mm and a temperature resistance up to 230 °C.

The multiple sensor units were positioned precisely at the desired measuring points with the aid of a support structure consisting of carbon fiber tubes. The wires of the sensors are led outside the mold using a cable guide, which in turn was sealed with adhesive to prevent the leakage of liquid resin during the production process. The structure was additionally attached to a metal bolt, which was also cast in place (Fig. 4). This metal bolt is used as standard in the workpiece in order to be able to fasten the isolator with a screw in the respective application.

After installing the sensors and closing the mold, the connecting leads of the Pt1000 and the NTC10k were each connected to a data logger 1 (Thermo Fischer, Data Taker DT85) according to the two-wire measuring principle. The wires of the temperature–pressure combination sensors were connected to an Arduino Due, here data logger 2 (Fig. 5).

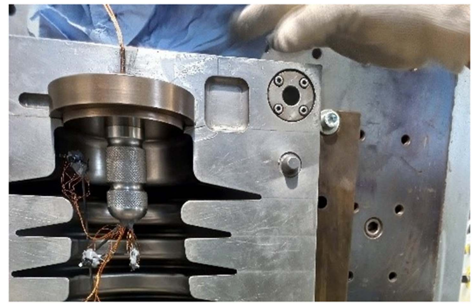

Fig. 4. Multiple sensor unit in fix position in mold and outgoing cables.

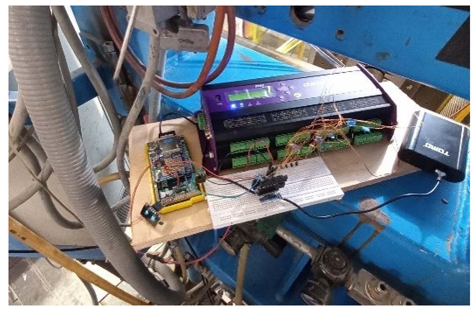

Fig. 5. Setup of data loggers next to the mold.

Fig. 6. Cured electrical isolator with integrated sensors.

Fig. 6 shows the completed casting process of the isolator with the sensor connection leads shortly before the isolator is demolded. The material is a two-component mix (silica-filled epoxy), which reacts exothermically when heated, going from liquid to gel-like to solid. The process duration is about 20 min.

III. RESULTS

Fig. 7 shows a direct comparison of the measurement results for the temperatures and pressures of the different sensors. In the graph, three temperature curves and one pressure curve are plotted against time. The pressure is plotted on the right and second *y*-axis, while the temperature is on the left *y*-axis.

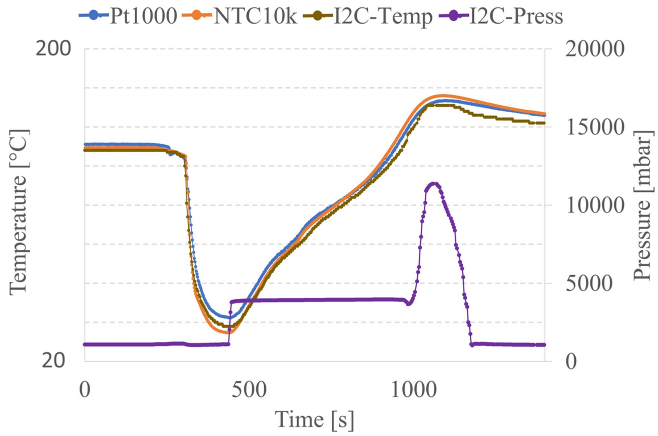

Fig. 7. Comparison of temperature curves and pressure curve over time for one position for all three sensors in the multiple sensor unit area.

At the beginning, the target temperature is constant until cold material is filled into the mold and a temperature drop occurs. In the period from 500 to 1000 s, the temperature in the cold material rises again, on one hand, due to the heating step of the preheated mold, and on the other hand, due to the additional heat from the chemical reaction. Due to the exothermic behavior of the epoxy material, there is an overshoot compared to the initial temperature set by the preheated mold. After the temperature reaches its maximum at 1100 s, it starts to decrease again. The atmospheric pressure at the beginning in the still empty mold is measured in the time range from 0–500 s. When the material is in the mold, an additional pressure is applied to press the material into the mold which is seen in the time range from 500–1000 s. In the time range from 1000 to 1200 s, a rising and falling pressure back to the initial state can be observed.

IV. DISCUSSION & CONCLUSION

The measurement in the mold cavity directly in the material was solved with several sensors. Four sensor values were transmitted (3× temperature, 1× pressure). Due to the small design of the sensors and the use of keyhole technology, the measuring task could be solved in the smallest of spaces. By using the smallest sensors, and thus, the minimum heat sink, the production process can be recorded with little influence and interference in the system. Particularly noteworthy is the successful pressure measurement at temperatures of almost 200 °C, which would have been limited to 85 °C due to the specifications and data sheet information for this sensor type. However, previous measurements in the climate chamber have shown that the combined pressure and temperature sensor can also withstand higher temperatures and still be able to send data, but it was noticed that the tolerance specifications for temperature and pressure could no longer be met. Instead of the value of 180 °C measured by a calibrated climate chamber, the sensor displayed 179 °C. This would be a deviation of 1 °C. At 25 °C, the atmospheric pressure was still displayed as 1.0 bar, while at 200 °C it was already 1.1 bar. That would be a deviation in displayed pressure of 10% only due to a temperature difference.

When comparing the three temperature sensor values, it is noticeable that a deviation of the temperature value was observed with a maximum temperature difference of 8 °C in the time range of 400 s. There can be three reasons for this: 1) The millimeter distance between the sensors influences the results because the temperature in the reactive material varies despite the small distance between the sensors; 2) the sensors themselves have different response times; 3) the tolerances of the sensors cumulatively cause this deviation. The specified temperature tolerances are cumulatively ±4.3 °C. The pressure sensor's tolerance of 1 °C at high temperatures has been taken into account. This means that a measured deviation of the temperature sensors from each other of 8 °C is still within this cumulative tolerance. This is acceptable because the target deviation of ±5 °C, specified by the manufacturer, was not reached and the measuring system is therefore suitable. Nevertheless, the small distance in the position of the sensors to each other could also have an influence on the deviation, even if this distance corresponds to about 0.5 cm, as shown in Fig. 3. The difference in thermal mass could also play a role, as the deviations from each other increase in the range of a temperature change, while the readings are closer together when the temperature change is small. From considerations, such as thermal mass, small distance to each other as well as tolerances, it can be concluded that temperature comparisons with different sensor types can produce larger deviations than with static processes, especially with dynamic processes. Pressure measurements at a point in the cavity of a mold can give an indication of when something is happening in the mold. On one hand, pressing pressures can be checked, but changes in the state of the material can also be tracked. In this case, the fact that the pressure deviates more from its real value due to the operation of the sensor outside of its specifications is of secondary interest. Of particular interest is the pressure change, which in Fig. 7 begins at time 1000 s, indicating the start of a change in the material, and continues until time 1200 s, which in turn indicates the end of a change. One explanation for a change in pressure measured in a two-component thermoset material would be during a transition from liquid to gel to solid as the material expands or contracts.

Both events, the exothermic temperature and pressure rise, correlate very well with each other indicating that a transformation of the material takes place at this point. Since this work focused on the measurement technique behind it, a qualitative statement about the process progress as well as the solidification of the material via the parameters temperature and pressure is preferred instead of tracking the curing degree. The chemical degree of cure of a progressing curing reaction might correlate with the two parameters, but nevertheless a "postcuring" might take place afterward, and a degree of cure can only be accurately determined by other analytical methods.

In summary, using pressure sensors in combination with temperature sensors in such an application is a smart way to extract status data for process control and monitoring. Depending on the application and mold, a process engineer can decide at which points such sensors should be used, since this sensor concept can be expanded.

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REFERENCES

[1] A. Al-Gheilani, W. Rowe, Y. Li, and K. L. Wong, "Stress control methods on a high voltage isolator: A review," *Energy Procedia*, vol. 110, pp. 95–100, 2017, doi: 10.1016/j.egypro.2017.03.112.

[2] A. A. Salem et al., "Pollution flashover under different contamination profiles on high voltage isolator: Numerical and experiment investigation," *IEEE Access*, vol. 9, pp. 37800–37812, 2021, doi: 10.1109/ACCESS.2021.3063201.

[3] E. S. M. El-Refaie, M. K. Abd Elrahman, and M. K. Mohamed, "Electric field distribution of optimized composite isolator profiles under different pollution conditions," *Ain Shams Eng. J.*, vol. 9, pp. 1349–1356, 2016, doi: 10.1016/j.asej.2016.08.012.

[4] C. Pasco and K. Kendall, "Characterisation of the thermoset prepreg compression moulding process," in *Proc. Soc. Petroleum Engineers Automot. Composites Divisions-16th Annu. Automot. Composites Conf. Exhib.*, 2016, pp. 1–18.

[5] M. Hall, X. Zeng, T. Shelley, and P. Schubel, "In situ thermoset cure sensing: A review of correlation methods," *Polymers*, vol. 14, 2022, Art. no. 2978, doi: 10.3390/polym14152978.

[6] M. Etchells, N. Pantelelis, and C. Lira, "Cure monitoring of highly reactive resin during high-pressure compression resin transfer moulding," in *Proc. 18th Eur. Conf. Composite Materials*, 2018, pp. 25–28.

[7] M. Kahali Moghaddam, M. Hübner, M. Koerdt, C. Brauner, and W. Lang, "Sensors on a plasticized thermoset substrate for cure monitoring of CFRP production," *Sensors Actuators A: Phys.*, vol. 267, pp. 560–566, 2017, doi: 10.1016/j.sna.2017.10.007.

[8] A. Fabregat-Sanjuan, X. Fernández-Francos, and F. Ferrando-Piera, "Controlled composite processing based on off-stoichiometric thiol-epoxy dual-curing systems with sequential heat release (SHR)," *J. Appl. Polym. Sci.*, vol. 139, no. 17, 2022, Art. no. e52009, doi: 10.1002/app.52009.

[9] H. Ming-Shyan, C. Jian-Yu, and X. Yu-Qi, "Quality monitoring of micro shrinkage defects in thick-walled injection molded components," *Measurement*, vol. 201, 2022, Art. no. 111733, doi: 10.1016/j.measurement.2022.111733.

[10] S. Krizsma and A. Suplicz, "Comprehensive in-mould state monitoring of Material Jetting additively manufactured and machined aluminium injection moulds," *J. Manuf. Processes*, vol. 84, pp. 1298–1309, 2022, doi: 10.1016/j.jmapro.2022.10.070.