Deutscher Luft- und Raumfahrtkongress 2012
DocumentID: 281405

EFFECTIVE AND FLEXIBLE ULTRASOUND SENSORS FOR CURE MONITORING FOR INDUSTRIAL COMPOSITE PRODUCTION

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Abstract

Fiber reinforced plastics production is growing at high rates in aeronautic and automotive as well as wind energy industry. On the other hand the production of these composite materials is complex – therefore difficult to automate – and cost-intensive due to the high amount of necessary manual work. The curing of the thermoset polymer matrix is one of the key steps in the process chain, where process-integrated cure monitoring sensors could play an important role for quality assurance, process optimization and automation. While ultrasound sensors are well suited for cure monitoring, in practice – especially in regard of industrial production conditions – the reliability is not sufficient for automated processes. Instead of the commonly used ultrasound transducers a method is presented where tool-mounted piezoceramics are used instead. These sensors are tested for their capability and efficiency as cure monitoring sensors and furthermore integration techniques and application examples are presented.

1. INTRODUCTION

With their excellent mechanical properties and low density carbon fiber reinforced plastics (CFRP) are gaining interest as structural components for effective weight reduction. Especially in the background of energy reduction for ground and air transportation, but also in other fields like the blades of wind turbines, the production volume of composite parts is growing. On the other hand the production of composite parts is very complex and today requires still a high amount of manual work as the automation of process steps is very challenging. Today the high demands of commercially competitive composite production concerning productivity, quality and cost effectiveness cannot be met by processes largely depending on manual work.

In order to contribute to overcome these technical challenges for process automation the Centre for Lightweight-Production-Technology in Stade of the DLR (German Aerospace Center) composite production processes are developed at an industrial scale filling the gap between research and industrial application.

One of the most important steps in composite production is the curing of the polymer matrix, where the material's final properties are broadly set. In today's cure processes the temperature profiles are based on the resin's data sheet or experience and furthermore adapted and optimized by extensive trials. Nevertheless the process steps contain high safety margins to ensure a sufficient degree of cure and to compensate process deviations like resin age, process temperature tolerances and laminate thickness. In state of the art for process monitoring temperature sensors such as thermocouples are applied. However by temperature measurements only indirect information about the current part quality can be derived. Through the temperature the cure rate of the resin is controlled while small temperature variations can cause considerable cure rate deviations or insufficient degree of cure. Regarding this, sensors which can obtain direct information about the resin's cure state are of high interest. These so called cure monitoring sensors can be a powerful tool in composite production. Besides quality assurance and documentation they offer as well a high potential for effective process control and optimization, trouble shooting and short run up time for new processes. [01, 02, 03]

2. CURE MONITORING SENSORS

Cure monitoring sensors are amongst others dielectric, resistive and ultrasonic sensors [04, 05]. The sensors are most commonly installed directly into the mould and are able to sustain the harsh environmental conditions of temperatures over 200 °C and pressure over 20 bars. As the principle of the dielectric and resistive sensors is based on measurements of the electric properties of the resin which undergo a change during cure they require direct contact to the resin. Thus they affect the part surface, the mould tightness and furthermore the process reliability. Hence the dielectric and resistive sensors are difficult to integrate into the mould and the sensor locations are limited, especially for complex moulds. Also their information content is limited as they obtain only the cure state on the surface. Due to the resin's low thermal conductivity the heat transfer into the inner laminate is delayed and therefore the cure reaction as well. But as heat is released during cure which cannot be transferred to the surface the cure often is strongly accelerated inside thick laminates once the cure reaction is started. Therefore it is not always sufficient to monitor the cure on the surface.

Ultrasonic sensors on the other hand do not need direct contact to the resin and provide the degree of cure averaged over the whole specimen thickness. The ultrasound waves are sent from an emitter through the mould, pass through the composite part and then are registered by a sensor located on the opposite. With rising degree of cure as the elasticity increases sound waves are propagated faster by the resin which allows a sensitive production-integrated cure monitoring. Also morphologic changes, i.e. gelation and vitrification, can be detected by ultrasound.

3. ULTRASOUND CURE MONITORING IN PRACTICE

In practice on the other hand trials with ultrasound transducers showed unstable measurements where often no signal could be received during long periods of the production process. The problem occurred often in heating or cooling phases and at high temperatures. The source of these measurement failures has been located in the unreliable acoustic coupling between the transducers and the mould. When the transducers are not properly coupled the sound waves cannot propagate into the mould and the part in production, thus the monitoring fails.
The transducers are commonly integrated by adapters into the mould – or in case of open-mould-processes into the vacuum bagging (Fig. 1). The adapters have to hold the transducers with some pressure in an upright position against the coupling surface. To apply also enough pressure at high temperatures naturally a simple screw fixation is not sufficient and thus has to be extended by a spring.

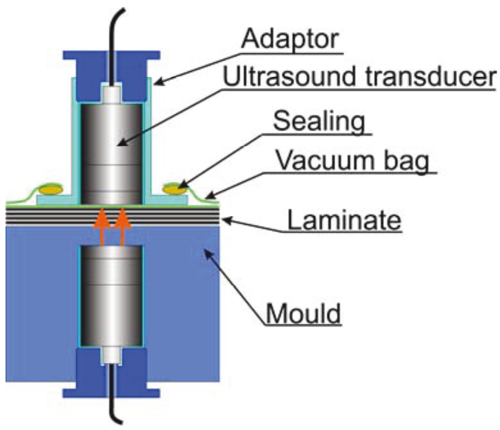

Fig. 1: Integration scheme of ultrasound transducers

Fig. 2: Integration of ultrasound transducer in vacuum bagging

The coupling between the transducer and mould is commonly ensured by a thin layer of soft metals (such as copper or lead), rubber or coupling paste. In [06] several coupling materials were investigated, where lead was to be found to be the most effective and ageing resistant coupling material. Nonetheless the coupling layer is a critical point when high temperature differences occur and the transducer and mould are not perpendicular. All efforts optimize the coupling layer and adapters only resulted in insignificant improvements of the reliability.
Instead of further optimizing the coupling layer and adapter the problem was resolved by a surprisingly simple solution. Instead of using a whole transducer – working with a piezoceramic actuator – the piezoceramic itself has been applied to the mould by an adhesive layer (Fig. 3) [07]. By using this method remarkable signal amplitudes and high measurement reliability has been achieved.

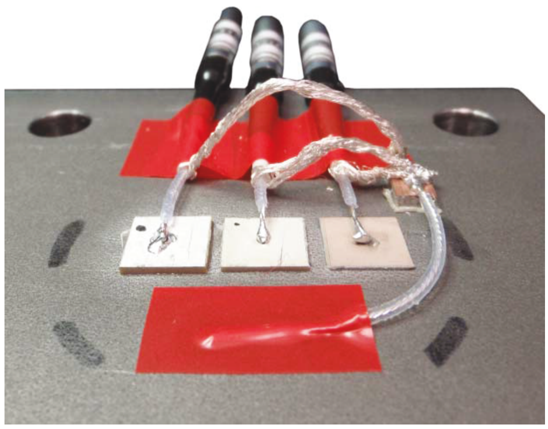

Fig. 3: Piezoceramics with different thicknesses applied to mould

4. PIEZOCERAMICS AS CURE-MONITORING SENSORS

The conservative ultrasound transducers were compared to monolithic piezoceramics of different thicknesses and materials. As the sensors are required to sustain temperatures of more than 200 °C in RTM and autoclave processes piezoceramics with high curie temperatures ($T_C \geq 300$ °C) were chosen to prevent depolarization [08]. As experimental set-up served a small tool of two steel plates of 15 and 5 mm thickness in which epoxy resin of 18 mm thickness was cured. The transmitters were placed on the 15 mm plate and the receivers on the 5 mm plate. As resin Araldite LY564 with hardener Aradur 22962 (*Huntsman International LLC*) was used mixed with a mass ratio of 100 : 25 which was cured at room temperature.
As result the piezoceramic sensors reached signal amplitudes of more than 50 times the amplitudes of the transducers like showed in Fig. 4 for the 1.0 mm piezoceramic of the material “255” who was found to be the most effective transmitter and sensor. The signal delay in the transducer’s diagram is due to the additional distance the sound waves have to pass inside the transducer’s housing. As the transducers include a damping material their impulse is briefer and clearer while the impulse of the piezoceramics decays more slowly. The clear impulse is preferable for signal analysis, but on the other hand the strong signal of the piezoceramics contain a very low noise level. Thus even during gelation, where the sound waves are strongly

attenuated, it was still possible to receive a clear signal transmitted through the thick epoxy specimen, while the transducer's signal was highly disturbed (Fig. 5).

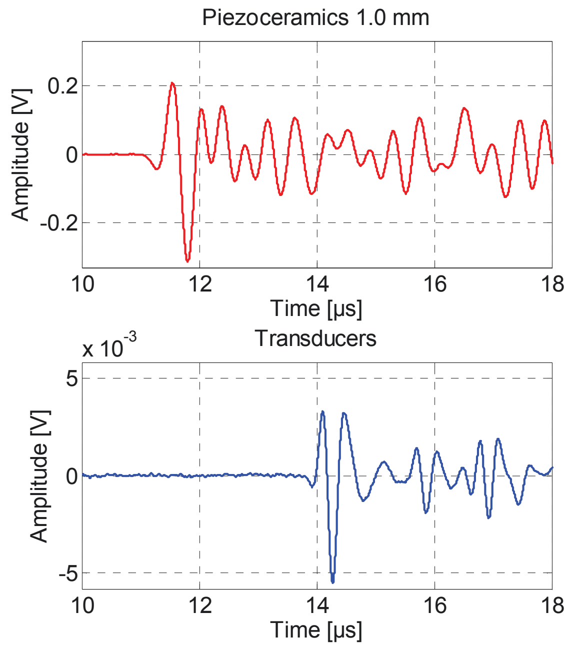

Fig. 4: Signal of piezoceramics and transducers of through-transmitted epoxy resin at the beginning of cure

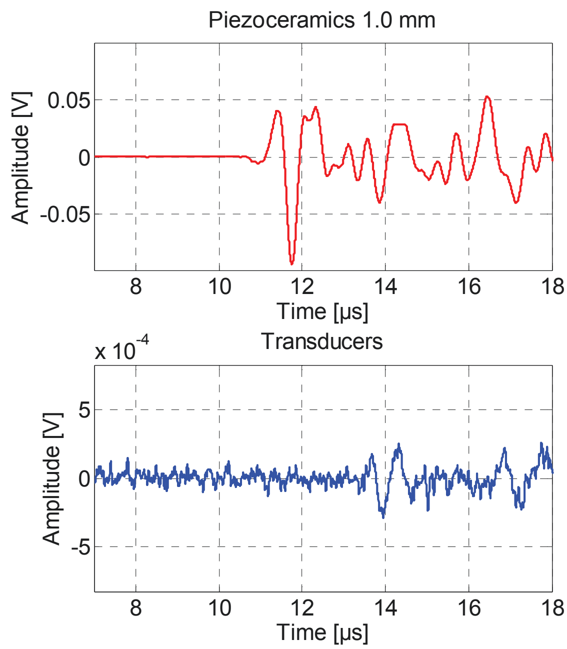

Fig. 5: Signal of piezoceramics and transducers of through-transmitted epoxy resin strongly attenuated during gelation

The significant high amplitudes cannot be explained only by the slightly lower impulse frequency by the piezoceramic actuators of about 3.1 MHz against the 3.5 MHz impulse of the transducer, which is assumed to have a relatively minor influence. The essential reason for the signal enhancement is the reduction of the numbers of interfaces the sound waves have to pass (Fig. 6). At every interface a fraction of sound is reflected due to impedance discontinuity. The reflection factor R can be calculated from the acoustic impedances Z_1 and Z_2 of the two interfacing materials (eq. 1), where the acoustic impedance Z is the product of density ρ and sound velocity c (eq. 2) [09].

(1.) $$R = \frac{Z_2 - Z_1}{Z_2 + Z_1}$$

(2.) $$Z = \rho c$$

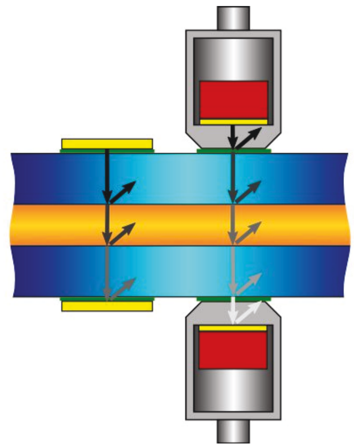

Fig. 6: Interfaces of piezoceramics (left) and ultrasound transducers (right) as cure monitoring sensors

With their high efficiency as actuators and sensors the piezoceramics allow more precise measurements and monitoring the cure in thick laminates. The exact limitations of laminate thickness has not yet been investigated, former trials using the transducers showed that a laminate thickness of about 4 mm outline already the limitations at high temperatures. Although it has to be stated, that the trials took place under the harsh conditions of an autoclave resin infusion process. Because of the high signal amplitudes achieved with the piezoceramics it is expected to increase the thickness limitations considerably.

Besides their high efficiency piezoceramic cure monitor sensors they have the advantage of being very compact. Therefore they are easier to integrate into moulding tools as well as they are considerably less expensive than ultrasonic transducers. Altogether they offer a broader field of application.

5. SENSOR INTEGRATION

For fixing the sensors to the mould adhesives have been found to be the most practical way, alternatively the sensors can be fixed by a vacuum membrane or a mechanical fixation like clamping or screwing. Several adhesives have been tested for signal amplitude and temperature influence. Three of the evaluated adhesives are compared in Fig. 7. The diagram shows the normalized measured signal amplitude of piezoceramics applied to an aluminum block as function of temperature during cooling from high temperature.

The first adhesive "Hysol 9492" (*Henkel*) is curable at room temperature and therefore has a relatively low glass transition temperature resulting in a quick amplitude drop under the influence of heat. In contrast to the two others the Hysol 9492 curve displays the measurement during heating as it was thermally damaged during the test. Nonetheless it is a well-suited adhesive for low temperature applications with low preparation time. "RTM6" (*Hexcel*) is a low viscosity injection resin for aeronautical applications with a high glass transition temperature of up to 220 °C, but has a minimum required cure temperature of 160 °C. It is a one-component epoxy needed to be stored at -18 °C. With increasing temperature the signal drops approximately linear to 70 % at 180 °C. The fabric-reinforced FM300 adhesive film (*cytec*) has to be cured at 180 °C developing high temperature resistance and moreover the signal strength stays almost constant during temperature change. As their disadvantage it has to be noted that due to the reinforcement the grounding contact of the sensors cannot simply be connected to the mould. The unreinforced adhesives form very thin layers thus the sensors are in electrical contact with the mould.

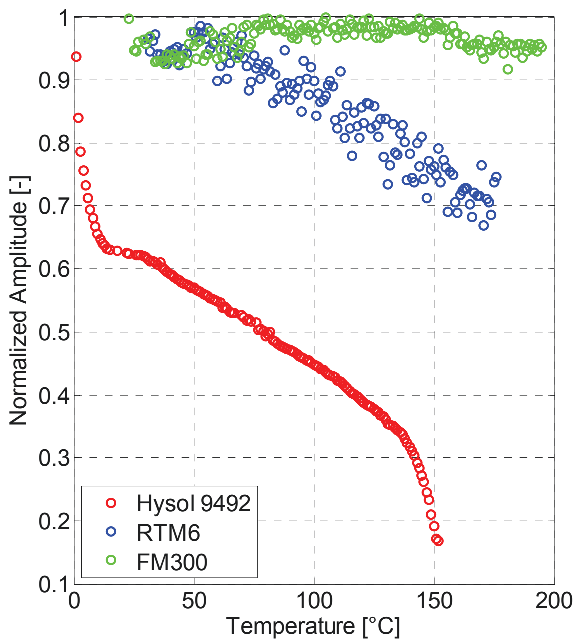

Fig. 7: Temperature influence of used adhesive on signal amplitude

For optimal measurement conditions the sensors have to be placed coaxial and in such manner that the sound waves are transmitted perpendicular to the part surface. Also the part surface should be flat to transmit a maximum sound energy through the part to the sensor, although when measuring at curved surfaces the effect of focusing can be used when the transmitter is placed on the convex side.

The sensor integration can be realized by simple construction measures like milled pockets and grooves for cable placement (see example in Fig. 8). The sound attenuation in metal is negligible compared to the one in the composite (approximately 0.1 % [05, 09]), so that it is not required to drill close to the surface for metal moulds. Also the near-field of the sensors have to be considered, which can be calculated from eq. 3., where D is the sensor diameter and λ the wavelength. The wavelength can be calculated from eq. 4. For example when using a 10 mm sensor with a frequency of 3.1 MHz mounted to a steel mould the near-field-length l_0 is approximately 12.9 mm. Only after the near-field-length the generated sound waves form a uniform wave front. The thickness of the mould where the sensors are placed should be minimum this value. If necessary the near-field-length can be reduced by smaller sensors or lower impulse frequencies.

(3.) $$l_0 = \frac{D^2 - \lambda^2}{4\lambda}$$ [09]

(4.) $$\lambda = \frac{c}{f}$$ [09]

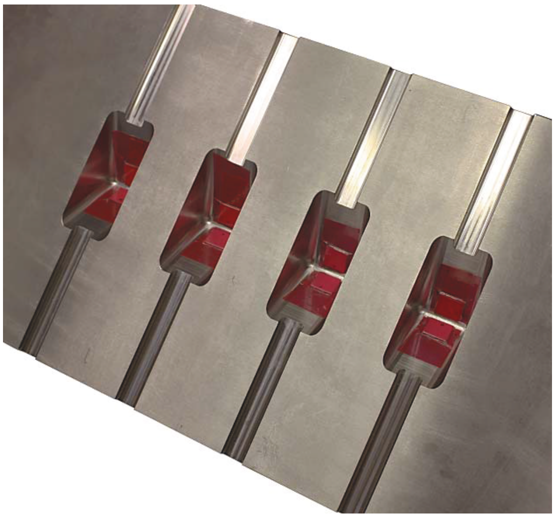

Fig. 8: Sensors mounted on tool (8 pcs.)

In open-mould processes the sensors are placed on or underneath the vacuum bagging (Fig. 9). In this case as well the piezoceramics are easier and less invasive to integrate. The transducers have to be placed in an adapter and sealed into the vacuum bag which is a high preparation effort and leakage risk. The piezoceramics can be placed inside the vacuum bag, for example between peel ply and breather as disposable sensors or on the vacuum bag fixed by another bag or adhesive layer. The vacuum bag does not have to be cut or draped while the sensor cables can be sealed on the side by sealing tape.

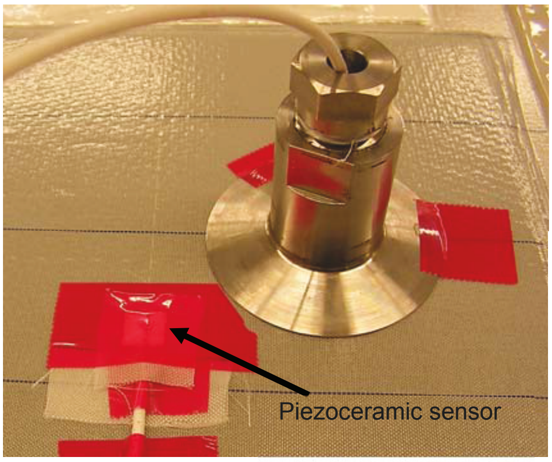

Fig. 9: Sensor integration of piezoceramics and transducers into vacuum bagging

All in all the presented sensors provide much simpler, less cost consuming and process invasive integration methods. On the other hand the high preparation time and effort for placing the sensors into the vacuum bagging in open-mould-processes is not always acceptable, even though the preparation time is suspected to be minor compared to the gain in process time using cure monitoring based process control. In consequence a method has been developed, where the sensors only have to be mounted on the tool side. As these sensors can stay installed permanently they only need to be connected before starting the process. This method is described in the following chapter.

6. REFLECTION METHOD FOR SINGLE SIDE ACCESS

Cure monitoring based on ultrasound by through-transmission has the disadvantage of requiring access from both sides of the composite part. For ultrasound monitoring where only the part is only accessible from one side the sender and receiver have to be placed on the same side. One possible way is the pulse-echo method where the sound waves are sent through the mould and composite part, then are reflected back, for example on the opposite mould or vacuum bag. The reflections can be detected by the same sensor sending the sound waves or a separate one. Like described in 5. the sound attenuation in composite material is considerably greater than in metal so that using the pulse-echo method the sound which is reflected repeatedly inside the mould overlay the sound waves who passed the composite part. Therefore the resin's sound velocity or attenuation cannot be derived by this method.
Inspired by [10] an alternative method was developed based on the fact, that the amplitude of the reflected sound is a function of the acoustic impedances of the two interfacing materials (eq. 1). As the impedance is the product of sound velocity and density (eq. 2), the sound velocity of the resin can be calculated from the reflection signal amplitude changing during cure. Of course it has to be noted, that the derived degree of cure describes only the material state at the surface of the composite part and not inside like by through-transmission. Therefore the method is preferable for thin laminates where the degree of cure is more homogeneous. This method provides still the advantage of not requiring direct contact to the resin and at the same time almost no preparation time is needed. The measurement of the reflection amplitude can be realized by applying the sensors in milled pockets in a steep angle to the part surface (Fig. 10). The reflection test trials correlate well with the simultaneous performed sound velocity measurements by through transmission (Fig. 11).
The drop in amplitude during cure is strongly depending of the tool material which defines the cure monitoring sensitivity. For example with a steel mould the acoustic impedance difference with resin is relatively high and therefore the reflection factor R changes hardly during cure. For aluminum and especially composite moulds the impedance difference is lower thus the method significantly more sensitive.

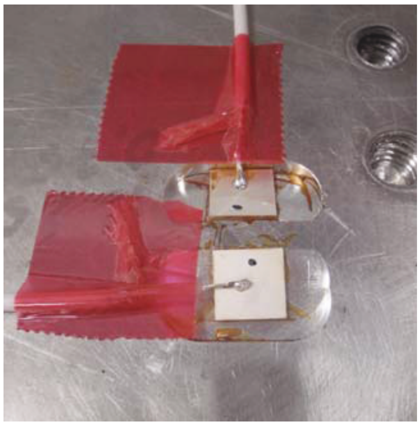

Fig. 10: Tool-mounted piezoceramics for reflection method

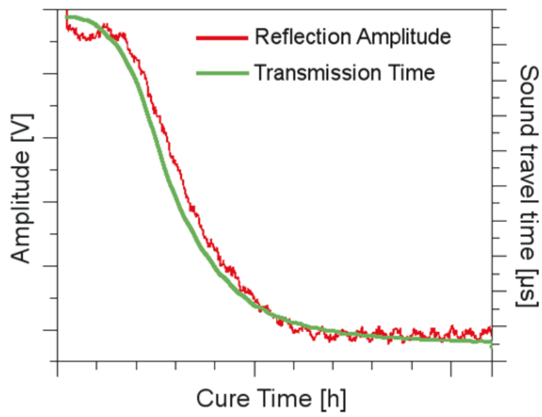

Fig. 11: Correlation between classic through-transmission and reflection method during resin cure

7. NOVEL CORRELATION TECHNIQUE FOR ULTRASOUND CURE MONITORING

To give an example for the broad application range owing their numerous advantages, their small size even allow integrating the sensors into a rheometer [11] (Fig. 12). The rheometer is a precise instrument to obtain the viscosity and the points where the gelation and vitrification occur during cure in function of the temperature, which are important data for composite processing. With this technique the rheological properties of a resin while curing can be measured such as elastic and loss modulus and acoustic parameters i.e. sound velocity and attenuation simultaneously. In difference to separate experiments this permits very exact correlation between the established laboratory method for cure characterization and ultrasound measurement, while in separate experiments the resin cure deviates due to inevitable temperature differences. The valuable results permit better understanding or "calibration" of the ultrasonic cure monitoring method. In the next step extensive measurement series will be performed with the goal of an empiric model for calculating the degree of cure and morphologic state of the resin in function of the acoustic parameters and the resin's cure. The gained knowledge and material models permit more precise online cure monitoring and thus more effective process and quality control.

Fig. 12: Piezoceramis integrated into rheometer

8. CONCLUSIONS

Even though cure monitoring by ultrasound is a powerful tool for process and quality control, the commonly used ultrasound transducers lack in measurement reliability under industrial production conditions. The crucial point is the unstable coupling between the transducer. The reliability could be realized by replacing commonly used transducers with piezoceramic plates fixed to the mould by an adhesive layer. Different piezoceramics were tested as cure monitoring sensors and compared to the transducers. The novel sensors reached significantly higher amplitudes and stability, as by this method the number of interfaces where the sound waves are reflected is reduced. With their small size the piezoceramics are easy to integrate while they are less expensive than transducers. Furthermore a method for single sided access to the mould – for example for open-mould processes – as well as a new calibration method for ultrasound cure monitoring were presented giving further application examples for the piezoceramic sensors.

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