ULTRASONIC INJECTION AND CURE PROCESS MONITORING: ENHANCING EFFICIENCY AND QUALITY ASSURANCE

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1 ABSTRACT

Ultrasound-based process monitoring offers a notable advantage: transducers operate without direct contact with the part, preserving the integrity of part surfaces and mold vacuum tightness. In-house developed, small and low-cost transducers facilitate seamless integration in a high number – if necessary. Advanced signal analysis allows not only the detection of flow front arrival time but also its velocity. Previously, cure monitoring required through-transmission with a transducer on each side of the part to measure sound velocity for deriving cure degree. Recent findings indicate the possibility of cure monitoring using pulse-echo configuration, simplifying transducer integration not only for open-mold applications and allows cure monitoring in parts with sandwich reinforcement. In addition to pressure waves, lamb waves expand process monitoring capabilities and offer another alternative to the through-transmission method. The sensor system accelerates process development and design, enhances quality assurance, allows process automation and reduces non-destructive testing (NDT) effort.

2 INTRODUCTION

In-process monitoring using sensor systems during the manufacture of thermoset composite structures can accelerate process development, enable new manufacturing methods or help to assure quality in series production. For instance, in closed-mold Liquid Composite Molding processes (LCM) like RTM (Resin Transfer Molding) the flow front evolution can only be observed by sensors to reconstruct where flow fronts join and might lead to dry spots. Without this capability the development of new LCM processes can require many trials in order to design a robust process.

Temperature sensors are often incorporated into molds for process monitoring, typically using thermocouples. The temperature measurements can be used as flow front detectors, when the resin and mold temperatures differ, and hence a temperature change upon flow front arrival can be detected. The method requires sensor integration close to the inner mold surface and works best with high temperature differences and less conductive mold materials [1]. The temperature measurement can also be used as boundary condition input for cure modeling in parallel to the running process [2]. This method does not account for the cure progression before and during injection and might be inaccurate due to measurement uncertainties. Since the reaction rate is derived from the measured temperature and then used to calculate the degree of cure by numerical integration, the measurement error has a significant influence on the degree of cure. Pressure transducers can be used to detect the flow front and furthermore to derive the pressure gradient from different sensor position allowing flow front position estimation [3].

A widely used measurement principle for flow front and cure monitoring are dielectric sensors. Electrodes are used to create an electric field on the composite surface and changes in electrical properties such as phase angle or the electrical resistance can be used to derive flow front arrival and degree of cure. [4]–[6] A disadvantage of these sensors is that they require direct contact with the composite part, making them more difficult to integrate and leaves marks on the part surface. In addition, the degree of cure is only measured on the part surface.

Ultrasonic sensors on the other hand can be mounted on the outer mold surface, as the sound waves are sent through the mold wall towards the cavity and composite part. Either classical ultrasonic transducers are used or as developed by the DLR bare piezoelectric ceramic disks can be mounted on the mold to transmit and receive sound signals [7],[8] as shown in Figure 1. They are not only easier and more flexible to integrate but also significantly more cost effective. In addition, this method achieves higher signal quality as less interfaces need to be overcome.

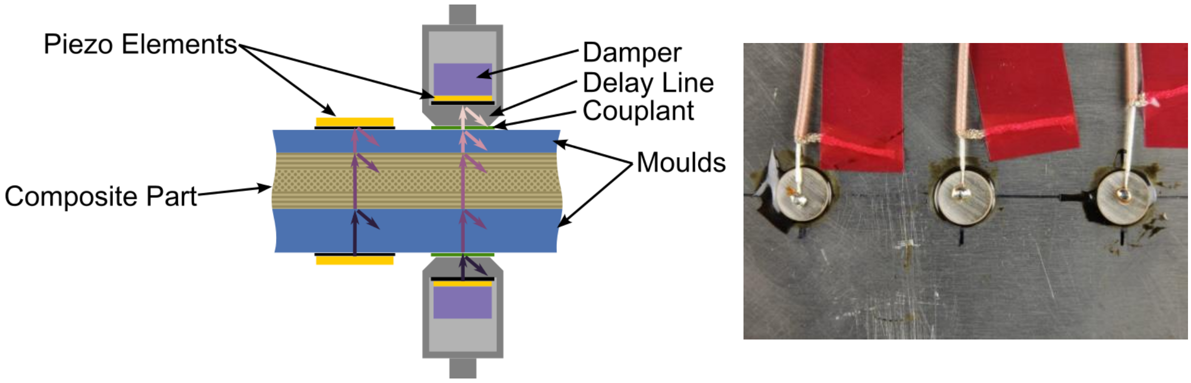

Figure 1: Comparison of tool-mounted bare piezoelectric ceramic disks and classical transducers for ultrasonic process monitoring

Cure monitoring is most commonly performed using through-transmission method, where the sound transmitter and receiver are located on opposite sides of the composite part as shown in Figure 1. The degree of cure can be derived from the sound velocity measurement as it is strongly related to the mechanical properties which in turn depend on the degree of cure.

The flow front can also be determined from the amplitude of the through-transmission signal or alternatively from the amplitude of the pulse-echo signal reflected from the interface of the mold and cavity (Figure 2). Prior to front arrival the sound signal is totally reflected and upon flow front arrival the reflection amplitude drops. The pulse-echo method indicates wetting of the mold surface and the through-transmission method the impregnation over the whole thickness between the transducers. By combining the pulse-echo signals from both sides and the through-transmission, the shape of the flow front can be derived, e.g. wedge or U-shaped. The duration of the reflection amplitude during flow front passage is used to calculate th flow front velocity. [7],[9],[10]

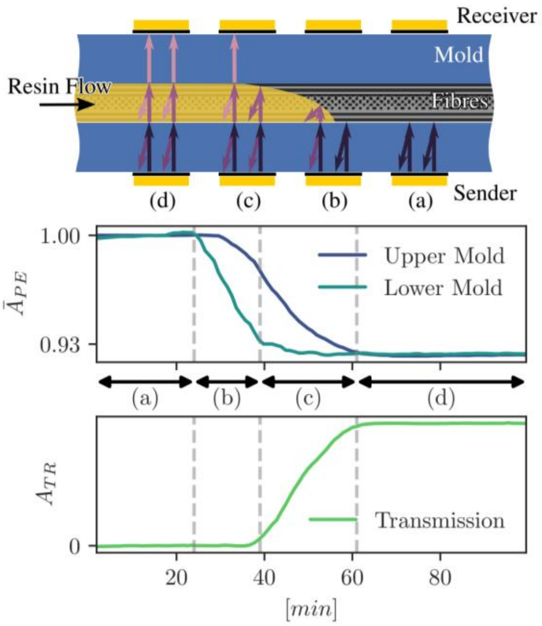

Figure 2:Ultrasonic flow front detection using pulse-echo and through-transmission methods

The previously described ultrasonic method applies pressure waves, also other modes of sound waves have been used to monitor impregnation or cure. The application of lamb waves are well known for structural health monitoring (SHM), also of composite structures. Some authors have used piezoelectric actuators mounted directly on top of the composite [11]–[14], while Hudson et al [15] used a thin Aluminum caul plate and Mehrabi et al [16] mounted the actuators on a thick Aluminum mold (as shown in Figure 3). The integration into or respectively on top of the laminate is often not desirable but in the case for later use as SHM and the cure is limited to only higher degrees of cure [11],[14]. When the mold is used as a waveguide the cure can be monitored over a larger range [15]. Flow front can be monitored with both methods, enabling the flow front position estimation on the sound propagation path [17],[18]. For industrial and series production the tool side sensor integration is desirable.

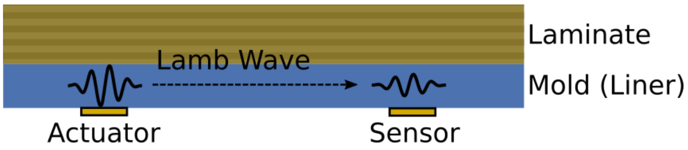

Figure 3: Lamb wave based flow front and cure monitoring using tool mounted piezoelectric actuators and sensors

3 EXPERIMENTATION

To evaluate the feasibility of sound (pressure and lamb wave) based process monitoring on various mold materials a series of experiments were conducted. For each experiment a different mold material or thickness was used. Three different polymer mold materials and one steel mold as a reference were used. For each polymer material the mold thickness was varied as well as listed in Table 1.

Table 1: Mold materials and thicknesses used for sound-based process monitoring evaluation

Experiment Nr.	1	2	3	4	5	6	7	8	9
Material	Sika Lab 975 New			PET		PA6			Steel
Thickness [mm]	5	10	30	2	5	1	2	5	5

One scope of this study is to examine the functional capability of the sensors on these mold materials and another scope is to assess the polymer materials as an impedance matched material to the composite part to enable pulse-echo based cure monitoring over the whole range from uncured to fully cured resin. For cure monitoring using the pulse-echo method the reflection factor at the interface between the mold and composite part must be low. In metallic molds, especially steel molds, approximately 92 % of the sound signal is reflected back to the transducer. Only a small ratio of the signal propagates into the composite part, where it is attenuated while traveling through the part thickness twice. So, a very small signal carrying the information of time of flight through the composite thickness arrives back at the transducer, while echoes still are reflected back and forth inside the mold as outlined in Figure 4. These echoes superimpose the small looked-for signal. Hence, a low reflection factor is required in order to allow pulse-echo based cure monitoring. Polymer materials are expected to have similar impedances, but may inhabit high acoustic damping. As acoustic properties are often not available or only broad value ranges are indicated, they have to be obtained experimentally.

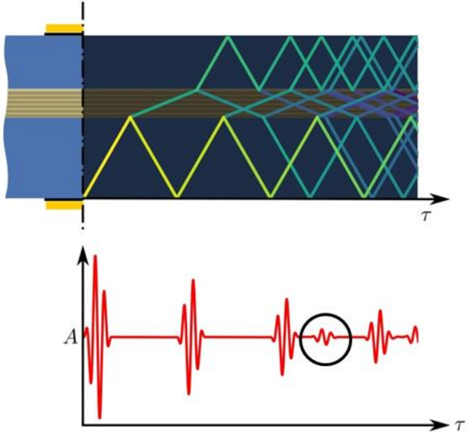

Figure 4: Signal composition by echoes inside an RTM mold calculated by ray tracing (the circle marks the part of the signal carrying information about the time of flight through the composite part thickness required for cure monitoring)

For each mold material and thickness a flat plate with the dimensions of 500 x 500 mm^2 was used. The piezoelectric transducers were mounted at the positions shown on the left hand side in Figure 5 using an instant adhesive with a copper mesh between the transducers and the mold for electrical connection. At positions 9 and 10 transducers were also mounted on the vacuum bag side between first and second vacuum film (positions 11 and 12) for using the transmission method as a reference.

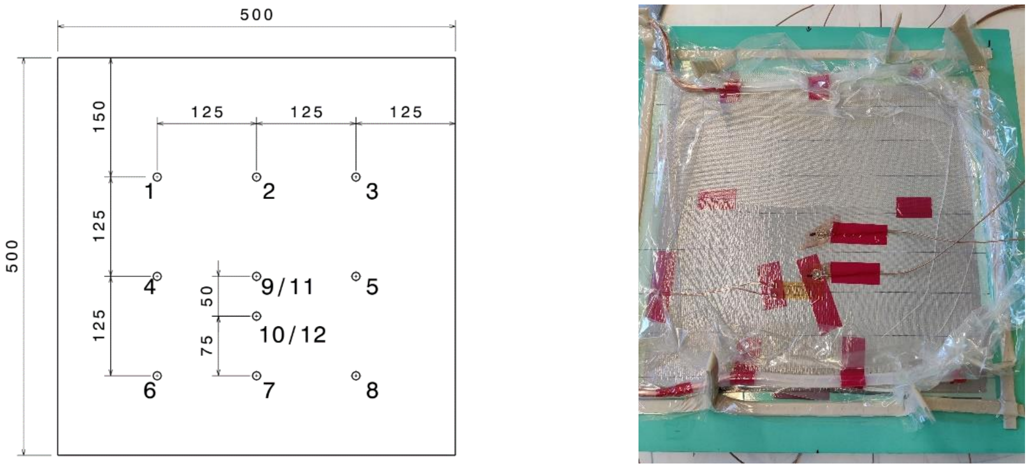

Figure 5: Transducer positions for each test mold plate (left) and photo of test laminate inside vacuum bagging with integrated piezoelectric transducers on top (right)

For signal generation and acquisition a pulser-receiver *US-Wave* of *lecoeur electronique [19]* and a self-house developed multiplexer and measurement software was employed. The pulser has implemented an arbitrary waveform generator. As excitation signal a sine wave with a Hanning window was used. For impulse-echo and through-transmission a center frequency of 2 MHz and a signal length of two periods was chosen. For the generation of Lamb waves the center frequencies 50, 70, 80, 100, 120 and 150 kHz and a signal length of five periods were used. The pulse-echo method was employed at all positions, the transmission method between positions 9 → 11 and 10 → 12 and the lamb waves on the paths 10 → 9, 10 → 7, 7 → 9 and 12 → 11 (see Table 2). The pulse-echo and through-transmission signals were acquired at a sampling rate of 125 MHz and the Lamb wave signals at 10 MHz All signals had a length of 5000 samples.

Table 2: Summary of measurement configurations

	Excitation Signal (Hanning windowed sine wave)		**Sound Path**
	Center Frequency [kHz]	**Periods**	**Sensor positions Transmitter → Receiver**
Pulse-Echo	2000	2	1-12 → 1-12 (transmitter = receiver)
Through-Transmission	2000	2	9 → 11, 10 → 12
Lamb Wave	50, 70, 80, 100, 120 and 150	5	10 → 9, 10 → 7, 7 → 9 (tool side), 12 → 11 (vacuum side)

For the infusion trials on each mold plate a dry fiber layup of four layers of carbon fiber fabric (HexForce G0926) of the size of 350 x 350 mm^2 were built up with the orientation $[0,90]_s$ with a peel ply top layer. As resin system the resin *SR1710* and hardener *SD8822* of *Sicomin* was chosen. The recommended cure consists of 24 hours cure at ambient temperature and then 16 hours at 60°C. The measurements were only performed during the first cure step at ambient temperature. For infusion a line injection and a flow aid spanning over about a third of the layup was used (from top to bottom in Figure 5).

4 RESULTS

4.1 Through-Transmission Method

At first the results of the through-transmission measurements as a well-known method for cure and flow front monitoring are presented. In Figure 6 an example is shown using four diagrams.

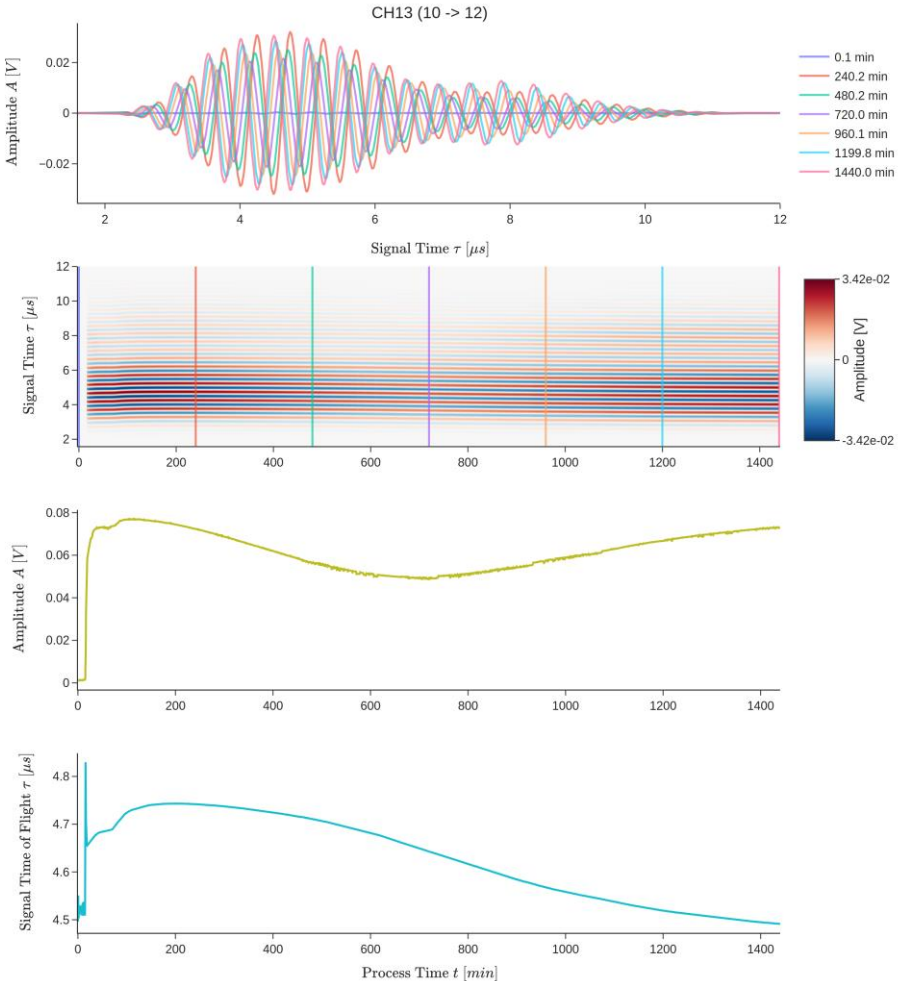

Figure 6: Representative example (PA6 2 mm) of measurement results using through-transmission method during infusion and cure (from top to bottom: signals at different times, pseudo-color representation of signal evolution over measurement time, signal amplitude and signal time of flight)

The two top graphs show the signal evolution over the measurement time of 25 hours in different visualizations. The other two graphs are the calculated signal amplitude and time of flight (TOF) from transmitter to receiver. At the beginning of the measurement the signal amplitude raises promptly after flow front arrival at the sensor position. The TOF seems to rise and drop at that moment, which is due to the calculation method and the noise registered before flow front arrival. After that the TOF increases with the laminate thickness during ongoing infusion. At about 180 minutes the TOF starts to decrease caused by curing and rising mechanical properties. At the end of the measurement duration the TOF continued to decrease as the cure progress was not finished.

4.2 Pulse-Echo Method

As mentioned before, the flow front arrival and velocity can be derived from the amplitude evolution of the pulse-echo signal reflected at the interface between mold and cavity. In Figure 7 an example of the same experiment as above is given. The flow front arrival can be seen very clearly by a sharp amplitude drop of more than 50 %. As expected, the reflection factors of PA6 can be assumed to be close to the factor of the impregnated laminate. In the appendix the amplitude plots of the pulse-echo signals on different mold materials and thicknesses are shown for comparison. Using a steel mold, the amplitude drop is less pronounced compared to those of the examined polymer molds. In the comparison it can be seen that also the mold thickness has an influence on the amplitude drop. On thin molds the TOF through the mold thickness is shorter than the sound signal length leading to signal overlaps. As each manually mounted and soldered piezoelectric transducers inhabits different characteristics, the signal shape and hence overlaps lead to diverging amplitude evolutions during flow front detection (Figure 11, Figure 12, Figure 13, Figure 14).

After flow front arrival in the pseudo-color representation of the signal evolution in Figure 7 a significant change in the signal characteristics and a similar TOF change due to laminate thickness growth is visible similar to the through-transmission results. Due to the low reflection factor it seems that a sound signal carrying the TOF information through the laminate thickness is received. The TOF plots of this pulse-echo measurement is similar to the TOF of the through-transmission, but due to the propagation twice through the thickness the value change about double as high. The significant TOF drop between 800 and 1000 minutes is assumed to be a calculation error due to complex echo signal overlaps. It has to be mentioned that most pulse-echo measurements on the polymer molds show less clear signals returning from the opposite laminate side. Anyways the results show clearly the feasibility of one- and tool-sided cure monitoring using TOF measurements. Further investigation and development should focus on more reproducible sensor integration, sensor dampening for shorter sound signals, optimal mold material and thickness and analysis of the pulse-echo signals.

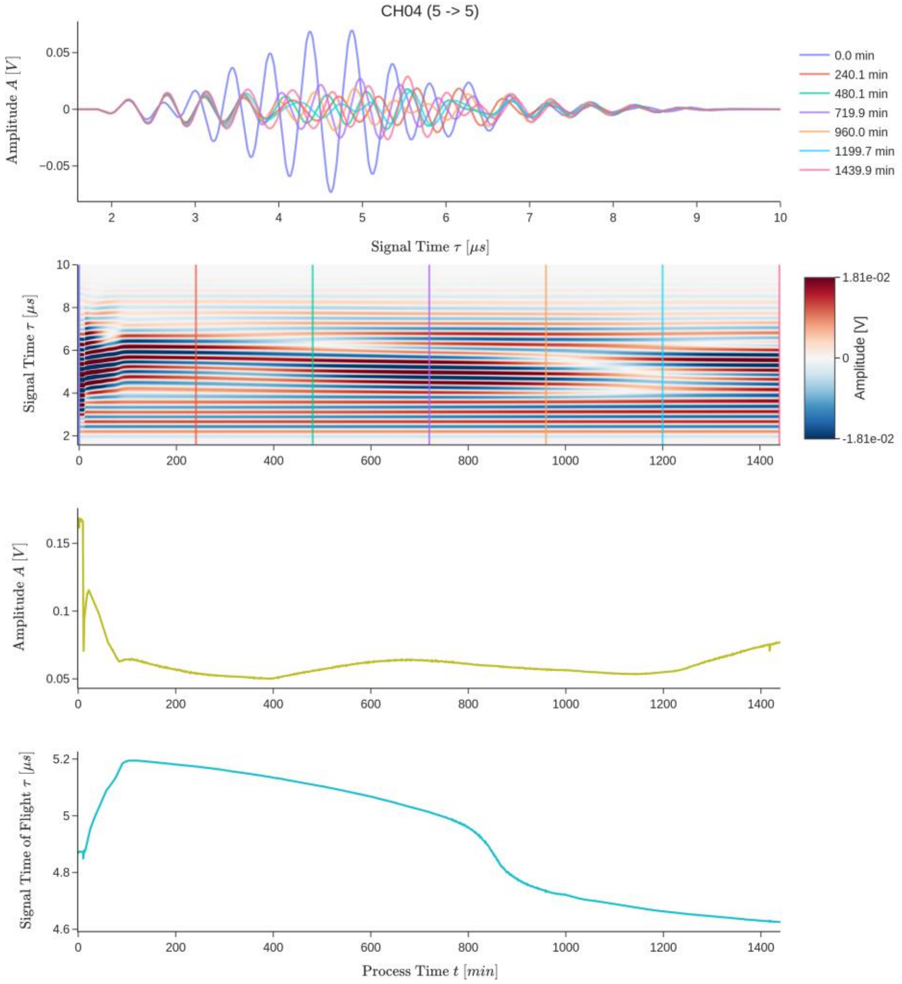

Figure 7: Representative example (PA6 2 mm) of measurement results using pulse-echo method during infusion and cure (the scale of the pseudo-color signal graph has been adapted to make smaller signals clearer)

4.3 Lamb Wave Method

In Figure 8 the results of the Lamb wave method during the infusion process with sender and receiver mounted on the tool side are shown. The method was found to work best with thin mold plates, where lamb waves propagate better and are easier to generate. For comparison the flow front measurements by pulse-echo at the two sensor positions is given in Figure 9. The flow front were detected after about 6 and 15 minutes, which corresponds well with the time range where an amplitude drop and phase increase of the lamb waves can be seen. Both outputs correlate with the flow front position on the sound path between sender and receiver and hence can be used as a more precise flow front detection method compared to the point wise detection using pulse-echo or through transmission methods.

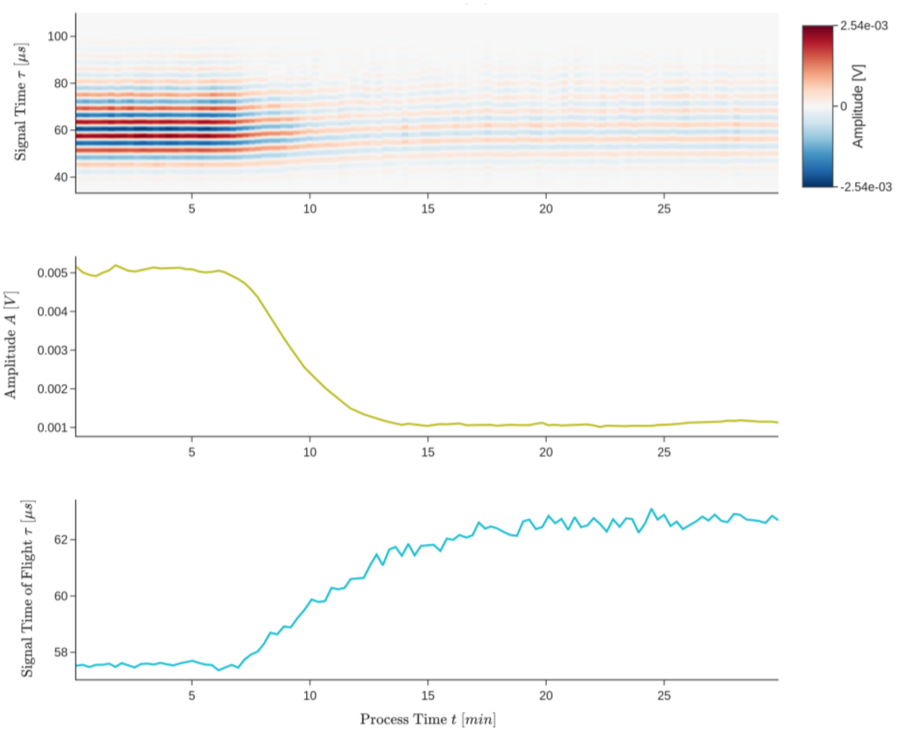

Figure 8: Representative example (PA6 1mm, 150kHz, 10 → 9) of measurement results using tool-side Lamb wave method during infusion

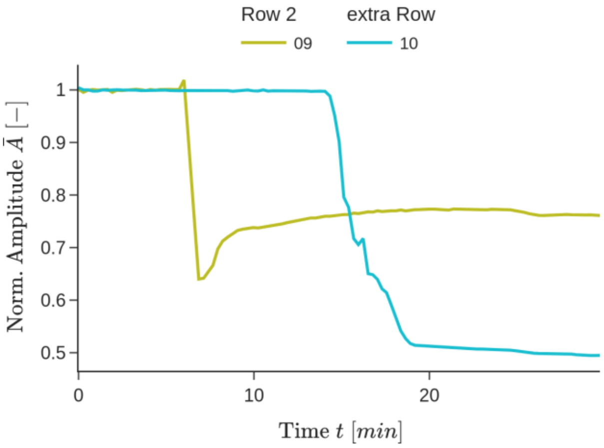

Figure 9: Results of Pulse-echo method for flow front detection (PA6 1mm) for comparison with lamb wave method

The degree of cure on the other hand could not be monitored over the whole measurement time using the tool-side Lamb wave method. Due to the amplitude drop during impregnation caused by leaking of the lamb waves into the laminate the received signals are weak and inadequate for analysis. As it can be seen in Figure 10 the amplitude starts to rise only after about 900 minutes at a higher degree of cure as stated in all literature references (see introduction).

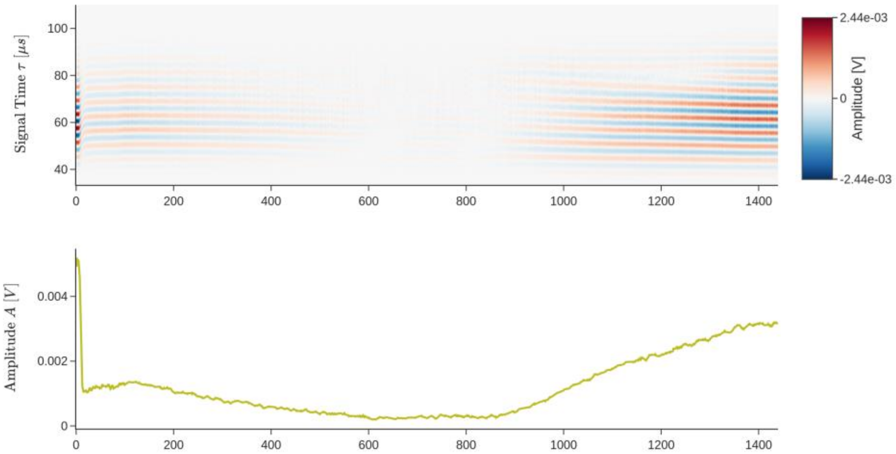

Figure 10: Representative example (PA6 1mm, 150kHz, 10 → 9) of measurement results using tool-side Lamb wave method during cure

5 CONCLUSIONS

Different measurement principles for monitoring the infusion and cure processes of thermoset liquid composite molding processes were introduced and compared. The sound-based methods were put into focus for their advantage not requiring direct contact to the composite part. To show their potential a series of experiments with different mold materials and thicknesses was carried out. The scope was on polymer mold materials and a steel mold was used as reference. Different sound-based methods were used: the pressure wave-based pulse-echo and through-transmission methods and the Lamb wave method.

The through-transmission method served as a well-known reference and could be used successfully for flow front and cure monitoring on all mold materials and thicknesses. The pulse-echo method is established for flow front monitoring, but could be used also for cure monitoring based on time of flight measurements on the polymer tools. This is due to the similar acoustic impedance compared to the wet end also fully cured laminate and hence the low reflection factor at the interface between mold and composite part. Furthermore, it has been shown that the Lamb wave-based method using tool mounted transducers can be used for flow front monitoring of the flow front position on the sound path between transmitter and receiver. The cure can only be monitored at high degrees if cure. The Lamb wave method is limited to thin molds.

6 REFERENCES

[1] G. Tuncol, M. Danisman, A. Kaynar, E.M. Sozer, Composites Part A: Applied Science and Manufacturing, *Constraints on monitoring resin flow in the resin transfer molding (RTM) process by using thermocouple sensors* 38 (2007) 1363–1386.

[2] K.-T. Hsiao, R. Little, O. Restrepo, B. Minaie, Composites Part A: Applied Science and Manufacturing, *A study of direct cure kinetics characterization during liquid composite molding* 37 (2006) 925–933.

[3] C. Di Fratta, F. Klunker, P. Ermanni, Composites Part A: Applied Science and Manufacturing, *A methodology for flow-front estimation in LCM processes based on pressure sensors* 47 (2013) 1–11.

[4] A. McIlhagger, D. Brown, B. Hill, Composites Part A: Applied Science and Manufacturing, *The development of a dielectric system for the on-line cure monitoring of the resin transfer moulding process* 31 (2000) 1373–1381.

[5] M. Kazilas, Cranfield University, *Acquisition and Interpretation of Dielectric Data for Thermoset Cure Monitoring*, Cranfield University, 2003.

[6] N. Pantelelis, E. Bistekos, *Process monitoring and control for the production of CFRP components,* SAMPE, Seattle, USA, 2010, accessed 5 March 2014.

[7] N. Liebers, D. Bertling, *Reducing NDT Effort by Coupled Monitoring and Simulation of Liquid Composite Molding Processes,* 11th International Symposium on NDT in Aerospace, Paris, France, 2019, https://www.ndt.net/search/docs.php3?id=25037.

[8] N. Liebers, *Ultraschallsensorgeführte Infusions- und Aushärteprozesse für Faserverbundkunststoffe*. Dissertation, Braunschweig, 2018, https://elib.dlr.de/121155/.

[9] N. Liebers, *Verfahren und Vorrichtung zur Herstellung eines Faserverbundbauteils*, 2016, https://worldwide.espacenet.com/patent/search/family/059285085/publication/EP3266597B1?q=pn%3DEP3266597B1.

[10] N. Liebers, *Verfahren und Vorrichtung zur Herstellung eines Faserverbundbauteils*, 2016, https://worldwide.espacenet.com/patent/search?q=pn%3DDE102016112263B4.

[11] J.S. Chilles, A.F. Koutsomitopoulou, A.J. Croxford, I.P. Bond, Composites Science and Technology, *Monitoring cure and detecting damage in composites with inductively coupled embedded sensors* 134 (2016) 81–88.

[12] S. Pavlopoulou, C. Soutis, W.J. Staszewski, Plastics, Rubber and Composites, *Cure monitoring through time–frequency analysis of guided ultrasonic waves* 41 (2012) 180–186.

[13] K. Mizukami, T. Ikeda, K. Ogi, Ultrasonics, *Measurement of velocity and attenuation of ultrasonic guided wave for real-time estimation of cure-dependent anisotropic viscoelastic properties of carbon fiber-reinforced plastics* 99 (2019) 105952.

[14] C.-A. Holst, V. Lohweg, K. Rockemann, A. Steinmetz. “Lamb wave-based Cure Monitoring of Carbon Fibre Reinforced Polymers for On-site Aircraft Repairs,” in: Innovation to shape the future: 5th International Forum on Research and Technologies for Society and Industry Centro Didattiko Morgagni, Firenze, Italy, September 9-12, 2019 2019 forum proceedings, IEEE, Piscataway, NJ, 2019, pp. 384–388.

[15] T.B. Hudson, F.-G. Yuan, Journal of Nondestructive Evaluation, Diagnostics and Prognostics of Engineering Systems, *Automated In-Process Cure Monitoring of Composite Laminates Using a Guided Wave-Based System With High-Temperature Piezoelectric Transducers* 1 (2018).

[16] M. Mehrabi, M. Soorgee. “The Use of Ultrasonic Guided Waves in Cure Monitoring of Adhesives,” in: 2019.

[17] X. Cui, Y. Yu, Q. Liu, X. Liu, X. Qing, Smart Mater. Struct., *Full-field monitoring of the resin flow front and dry spot with noninvasive and embedded piezoelectric sensor networks* 32 (2023) 85021.

[18] X. Liu, Y. Li, J. Zhu, Y. Wang, X. Qing, Polym. Compos., *Monitoring of resin flow front and degree of cure in vacuum-assisted resin infusion process using multifunctional piezoelectric sensor network* (2020).

[19] J.M. Lecoeur, *US-Wave*, Chuelle, France, 2020, https://lecoeur-electronique.net/us-wave.html, accessed 13 May 2024.

7 APPENDIX

7.1 Pulse-Echo Flow front Monitoring Comparison

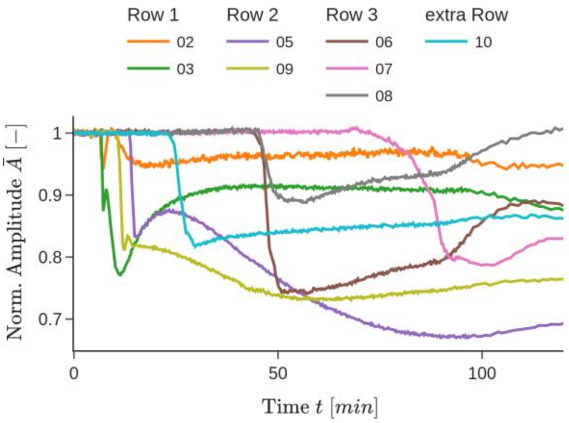

Figure 11: Steel 5 mm

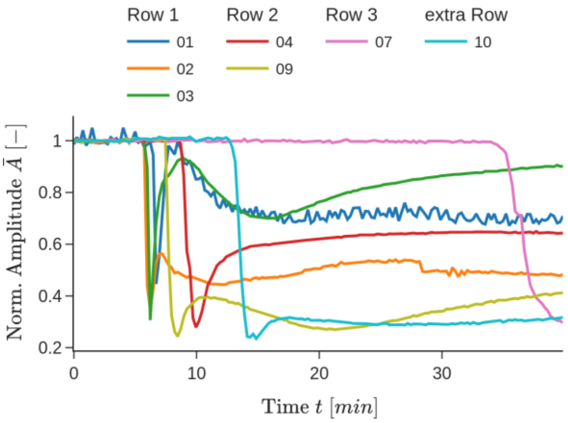

Figure 12: Sika 5 mm

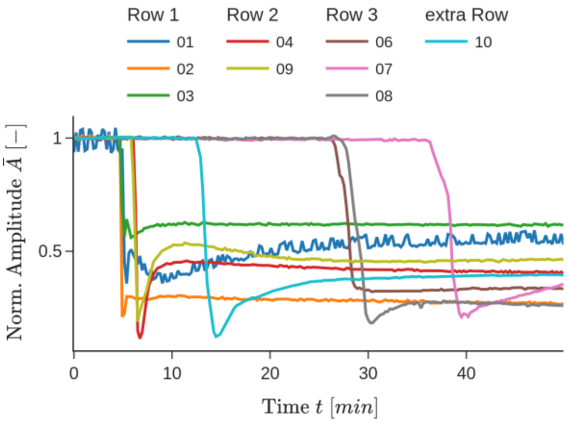

Figure 13: Sika 10 mm

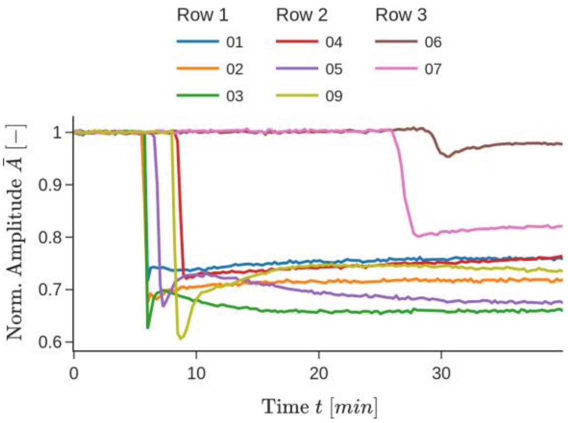

Figure 14: Sika 30 mm