Development of a Fully Automated Guided Wave System for In-process Cure Monitoring of CFRP Composite Laminates

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A guided wave-based in-process cure monitoring technique for carbon fiber reinforced polymer (CFRP) composites was investigated at NASA Langley Research Center. A key cure transition point (vitrification) was identified and the degree of cure was monitored using metrics such as amplitude and time of arrival (TOA) of guided waves. Using an automated system preliminarily developed in this work, high-temperature piezoelectric transducers were utilized to interrogate a twenty-four ply unidirectional composite panel fabricated from Hexcel® IM7/8552 prepreg during cure. It was shown that the amplitude of the guided wave increased sharply around vitrification and the TOA curve possessed an inverse relationship with degree of cure. The work is a first step in demonstrating the feasibility of transitioning the technique to perform in-process cure monitoring in an autoclave, defect detection during cure, and ultimately a closed-loop process control to maximize composite part quality and consistency.

1. Introduction

In the polymer composites industry, cure cycles are typically developed from a "trial and error" or a more effective "processing science" approach to reduce the final porosity level in the composite laminate [1]. Porosity [2] is defined as "large number of microvoids … which collectively may reduce the mechanical properties of the components to an unacceptable degree," but "each of which is too small to be of structural significance or to be detected individually by a realistic inspection technique." Typically, porosity occurs because of entrapped air, moisture, or volatile products during the curing cycle [2]. Although this work does not address the issue of porosity

directly, an automated guided wave-based system is preliminarily being developed to perform cure monitoring with the future goal of defect detection during cure.

In previous work with thermoset resins, ultrasonic velocity has been used to infer the degree of cure because of its association with the modulus of the resin [3-5]. Bulk wave ultrasound, in pulse-echo mode, can monitor the completion of resin cure based on when the time delay plateaus (i.e., the ultrasonic velocity stops increasing) in graphite/epoxy composites [6] and epoxy matrices [7]. Non-contact, air-coupled transducers can be used for ultrasonic velocity measurement in resins when line of sight is available during cure [8]. Using piezoceramic actuators and sensors mounted on the mold can alleviate the unreliable coupling between an ultrasound transducer and the mold which can occur, especially during phases of heating and cooling [9]. Other ultrasonic phenomena have also been used for monitoring degree of cure including attenuation (i.e., amplitude of signal) [2,5,6,10,11], instantaneous phase, and the "mean value of each frequency curve weighted by the maximum corresponding spectral amplitude" [12].

This work examined the amplitude and time of arrival (TOA) of guided waves in a carbon fiber reinforced polymer (CFRP) panel during cure in real-time. The significance of this approach was that unlike conventional ultrasound that provides information about the quality of the part directly underneath or near the ultrasonic transducer in a point-by-point (discrete) inspection, the guided wave approach interrogated a continuous wave path through the thickness of the panel along the line from actuator to sensor. Using a guided wave approach in CFRP laminates, it has been recently demonstrated that the group velocity of guided waves propagating in-plane, normal to the carbon fiber, increased as final degree of cure of the composite increased and the expected porosity level decreased [13]. However, this work was performed offline, after cure, on three separate composite panels. If key cure parameters can be estimated during cure, the process parameters can be dynamically tuned based on the measurements. This would prevent the operator from following a non-optimized fixed cure cycle.

2. Materials and Methods

A twenty-four ply panel was laid up by hand using IM7/8552, 35%RC, 190 gsm unidirectional prepreg (Hexcel® Corporation, Salt Lake City, UT). The panel was 203 mm × 152 mm × 4.65 mm (nominal) and the layup was $[0_{24}]$. The panel was cured in an oven (Figure 1) following the cure cycle recommended by Hexcel® (Ramp to 107°C at 2.8°C/min, hold one hour, ramp to 177°C at 2.8°C/min, hold two hours, cool down). The panel was interrogated during the entire cure period through guided waves. A guided wave was excited into the plate using a five-cycle Hanning windowed sinusoidal toneburst signal emitted from a waveform generator (Agilent Technologies: 81150A) to an amplifier (Krohn-Hite Corporation: Model 7602M) to a piezoelectric transducer (Physical Acoustics Corporation: Nano-30 (⌀7.9 mm, height 7.1 mm)) that was rated for use up to 177°C. The amplifier magnified the input signal to a peak-to-peak voltage of approximately 120 V. The plate response, normal to the fiber direction, was recorded by identical piezoelectric transducers in a pitch-catch configuration on an oscilloscope (Agilent Technologies: MSO9064A)

(Figure 1). Note that normal to fiber direction refers to the in-plane direction that is ninety degrees to the fiber and not through-the-thickness of the plate.

Release film was placed on the top and bottom of the panel. The sensors were bonded to a thin (0.1 mm) sheet of steel ("caul" plate) that was placed on top of the part which prevented the sensors from being pressed into the part during cure while still allowing the guided wave in the part to be measured in the composite. Rubber was placed around the sensors to help even out the pressure. A breather cloth and vacuum bag covered the part and full vacuum was applied using a vacuum pump (Figure 1). An oven was used to cure the panel as a part of the building-block approach to developing the guided wave system for use in an autoclave where the recommended 690 kPa pressure can be used. By following this approach, the port in the back of the oven could be utilized for the ingress/egress of all cables during development of the system prior to making modifications to the autoclave onsite. It should be noted that the goal of the current work was not to produce a high quality composite panel but rather to develop and test whether the system can excite and record guided waves during cure at elevated temperatures and whether meaningful information could be derived from the results.
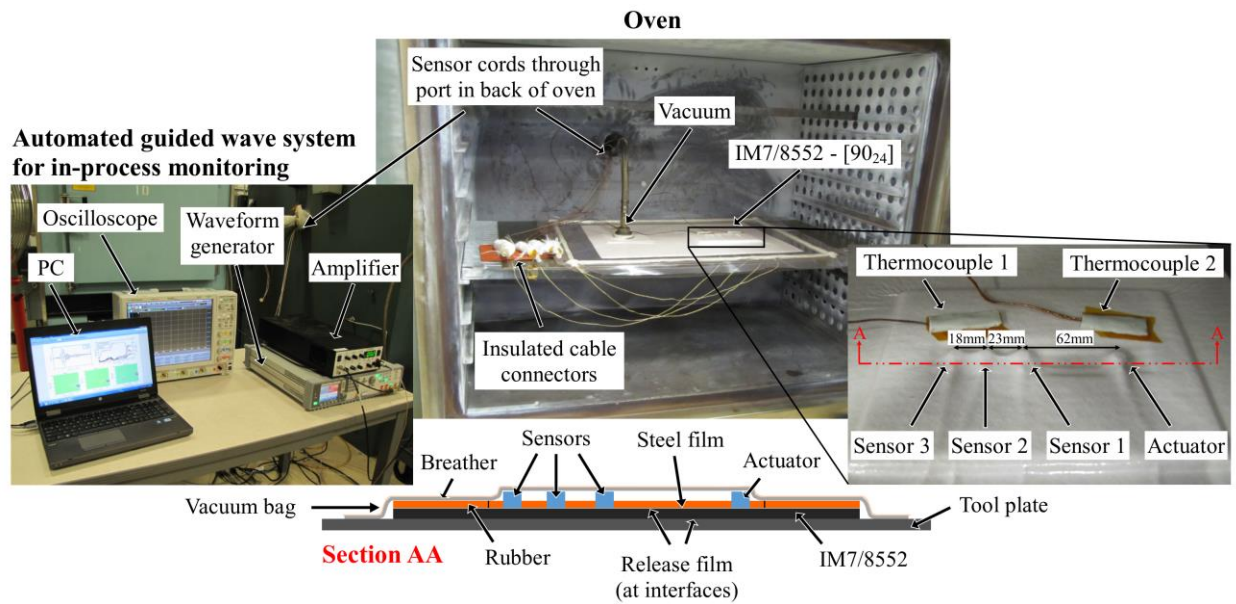

Figure 1. Automated guided wave system for in-process cure monitoring.

The automation code for the sensing system was written in MatLab® and utilized the Instrument Control Toolbox to control both the waveform generator and the oscilloscope. The general procedure of the algorithm is outlined in Figure 2. The center frequency of the five-cycle, Hanning windowed sinusoidal toneburst signal was set on the waveform generator. During each iteration, the center frequency was cycled through 105 kHz, 115 kHz, 125 kHz, and 135 kHz. The range of voltages to be measured by the oscilloscope was set based on the peak voltage recorded at that center frequency on the previous iteration. Dynamically scaling the range on the oscilloscope based on the previous iteration ensured that the range was minimized to increase signal to noise

ratio (SNR) while also keeping it sufficiently large enough to prevent the recorded voltage from being cut off. After the equipment was set, sixteen measurements were averaged on the oscilloscope and transferred to the computer. These data were processed through a bandpass filter and analyzed in real-time in MatLab®. The instantaneous recorded waveforms, the full time history waveforms, and key metrics such as peak voltage were all displayed on-screen during cure. This process was iterated throughout the cure of the part.

Figure 2. Automated algorithm for data collection and analysis of guided waves for in-process cure monitoring.

3. Results

The plate response at Sensor 2 (Figure 1) for five-cycle Hanning window toneburst actuation with center frequency 125 kHz at oven time around 285 minutes in the entire cure period is shown in Figure 3. Two wave packets were observed and analyzed separately. Figure 4 is the time-frequency analysis of this waveform. The frequency content of the recorded waveform corresponds to the center frequency of actuation (125 kHz) as would be expected (Figure 4).
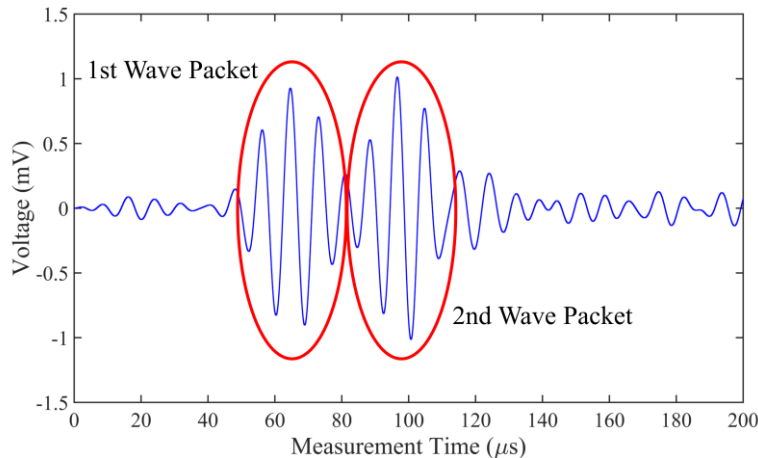
Figure 3. Plate response at Sensor 2 for five-cycle Hanning window toneburst actuation with center frequency 125 kHz at oven time around 285 minutes.
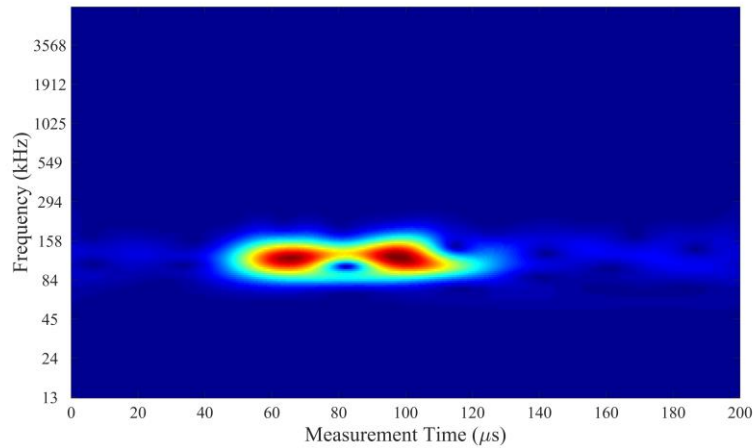
Figure 4. Time-frequency analysis of wave response at Sensor 2 for 125 kHz actuation at oven time around 285 minutes in Figure 3.

The full time history of the plate response at Sensor 2 excited by a five-cycle Hanning window toneburst actuation with center frequency 125 kHz is shown as a three-dimensional surface and contour plot in Figure 5. The amplitude of the recorded waveform sharply increased from around oven time equal to 185 minutes to 200 minutes.
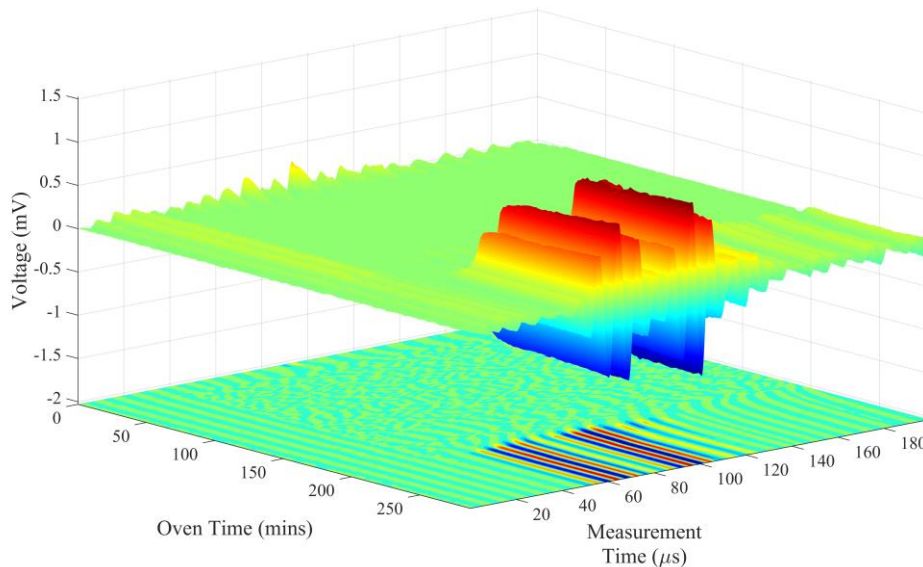

Figure 5. 3-D surface and contour plot of wave response for 125 kHz actuation at Sensor 2 throughout the cure.

In addition to the experimental study, a one-dimensional simulation of the cure response (Figure 6) was performed using RAVEN® composite process simulation software. The recorded air temperature of the oven and the average temperature of the two part thermocouples were utilized as inputs to the model. The two thermocouples were taped to the outside of the vacuum bag using sealant tape (Figure 1) and modeled as the temperature of the vacuum bag. Heat transfer coefficients were applied at the boundaries of the model which included each material layer listed

in the materials and methods section. The part temperature, degree of cure, and resin viscosity were direct outputs of the simulation. The glass transition temperature (T_g) was calculated using the DeBenedetto equation

$$T_g = T_{g_0} + \frac{\lambda \sigma}{1 - (1 - \lambda)\sigma}\left(T_{g_\infty} - T_{g_0}\right) \tag{1}$$

where σ is the degree of cure and $\lambda = 0.78$, $T_{g_0} = -7°C$, and $T_{g_\infty} = 250°C$ are model parameters for Hexcel® 8552 resin [14].

The transformation of the resin from the liquid to rubbery state is typically referred to as gelation. The subsequent transformation from the rubbery state to the glass state is defined as vitrification [15]. Vitrification occurs around the point at which the temperature of the composite resin becomes less than the glass transition or vitrification temperature [16] (Figure 6).
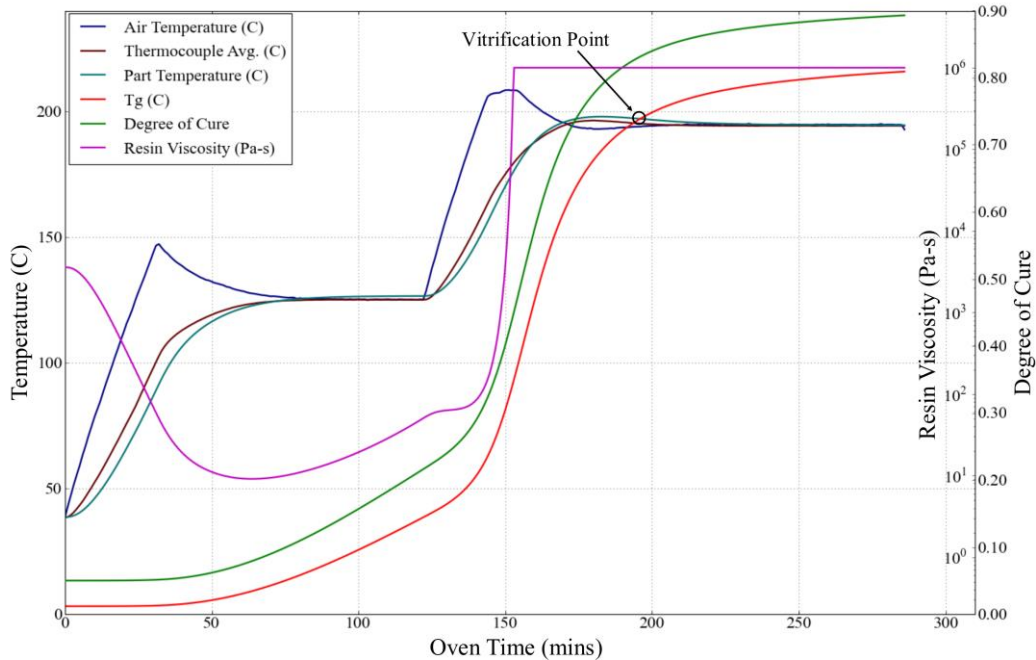

Figure 6. Cure response (part temperature, Tg, degree of cure, resin viscosity, and vitrification point) as predicted by RAVEN® composite process simulation software.

Figure 7 and Figure 8 indicate the average part thermocouple temperature as well as the peak voltages and time of arrivals (TOA) of the guided waves from the four excitation frequencies (105 kHz, 115 kHz, 125 kHz, and 135 kHz) at Sensor 2 of the 1st and 2nd wave packet, respectively. The sharp increase in peak voltage beginning at around oven (cure cycle) time equal to 185 minutes mentioned earlier was also observed at each of the four actuation frequencies for both wave packets. A sharp increase in the peak voltage curve ceases at around oven time equal to 200 minutes (vitrification point). From the first point at which the TOA can be accurately determined (oven time around 185 minutes) to an oven time of 195 minutes, the TOA of each wave packet for

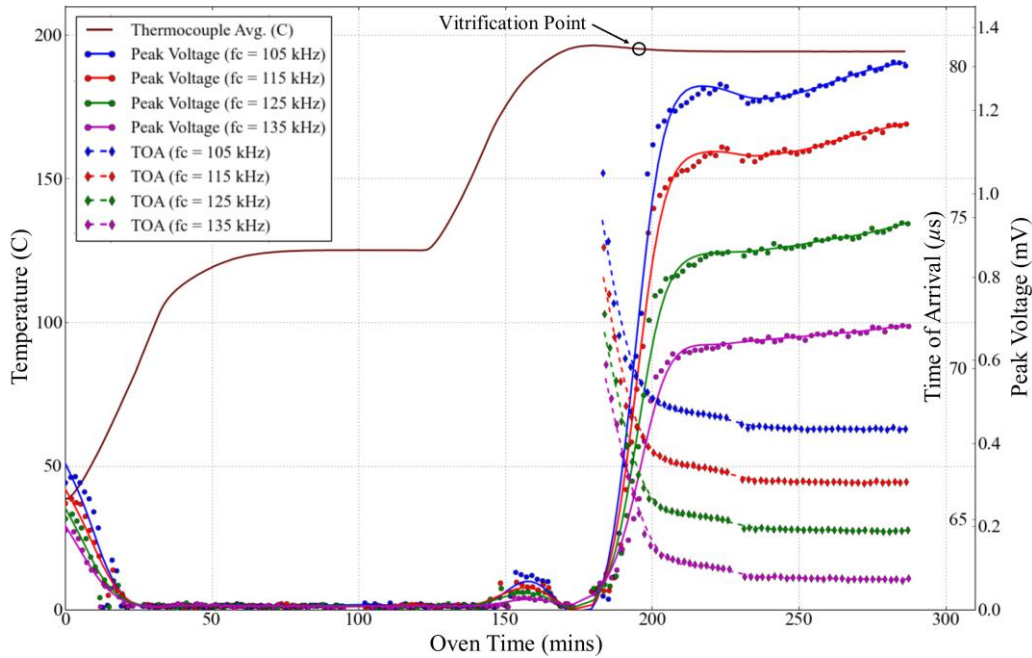each frequency drastically decreased. After which, the TOA of each wave packet decreased at a slower rate.

Figure 7. Peak voltages and time of arrivals (TOA) of the guided waves from four excitation frequencies at Sensor 2 of the 1st wave packet.
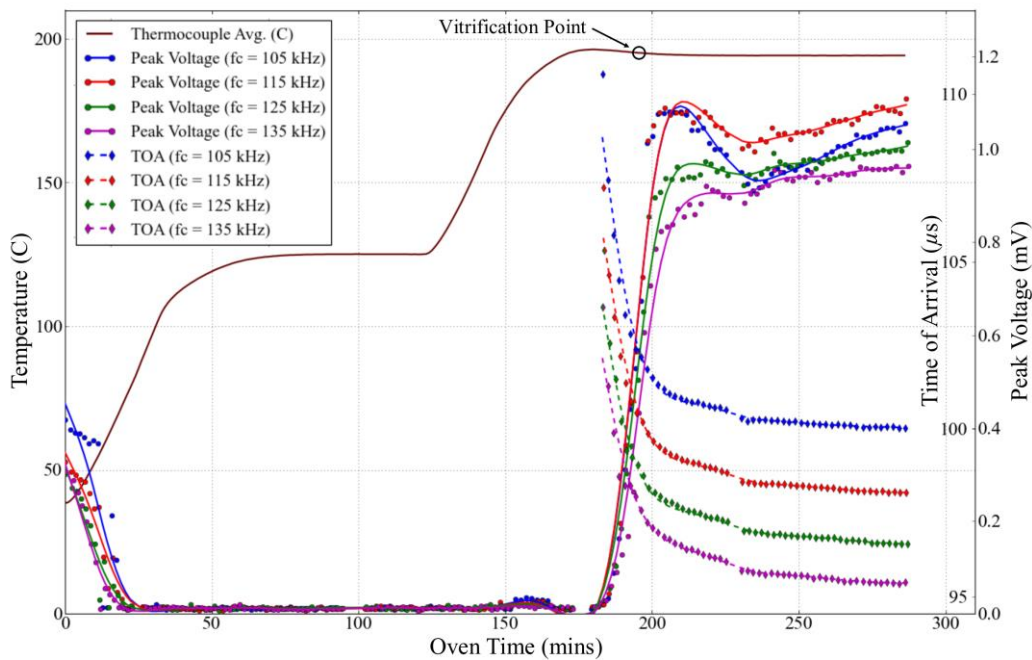
Figure 8. Peak voltages and time of arrivals (TOA) of the guided waves from four excitation frequencies at Sensor 2 of the 2nd wave packet.

4. Discussion

Several key cure transition points can be identified from these results. First, a sharp change of slope of the peak voltage occurred near the vitrification point which was predicted by RAVEN® (around oven time equal to 200 minutes). The time of vitrification was also consistent with previously published works on composites fabricated with Hexcel® 8552 resin [17-19]. Second, as the degree of cure simulated by RAVEN® increased, the TOA of the guided waves decreased (i.e., degree of cure was inversely proportional to TOA). In addition, the TOA curve flipped vertically resembled the shape of the degree of cure curve. From the curing time ranging from 180 to 195 minutes, both the TOA and degree of cure curves exhibited a sharp slope. The magnitude of both slopes decreased in a transition range from around 195 minutes to 225 minutes. This was followed by only a small decrease in TOA and increase in degree of cure until cool down began at 287 minutes. Although a quantitative equation for degree of cure based solely on TOA was not presented in this work, the TOA curve could be used as a qualitative measure to predict the degree of cure of the composite. Each of these trends were consistent over the frequency range investigated. Using guided waves throughout vitrification pushes the technology forward as guided waves are typically applied to solid state structures. Possible future research includes investigating a wider frequency range and developing dispersion curves for the composite laminate during cure by understanding the TOA frequency dependence.

The peak voltage prior to gelation (occurs before vitrification) was masked by the noise level of the oscilloscope and thus was not useable to make predictions about the state of the composite in the early stages of cure. However, preliminary results from additional experiments indicate that when the plate response is measured along the fiber direction, the peak voltage is high enough to record measurements throughout the entire cure cycle including when the resin in the prepreg is in the liquid, rubbery, glassy, and solid state. Before comparing the signals collected at multiple stages of the cure cycle, temperature compensation would be required. This was not necessary for this work because all of the measurements analyzed were taken during the second isothermal hold. In addition to measuring the guided waves in a direction where a fiber bed is present, preamplifiers could also be incorporated into the system to increase the SNR to a point where measurements are possible throughout the entire cure cycle.

Future work also includes investigating group velocity and attenuation. By using the other sensors from this experiment, the group velocity can be calculated as the distance between the two sensors divided by time of flight (TOF). It is expected that group velocity of the guided waves could also be used as a metric to track degree of cure since it is inversely proportional to TOA and thus should be directly proportional to degree of cure. Also by using multiple sensors, attenuation could be investigated by the change in peak voltage between sensors. More sensors will be needed, however, to provide an appropriate fit to the attenuation rate.

As mentioned previously, the goal of this work was not to produce a high quality composite panel but rather to develop and test the system and quality of the signals recorded during cure at elevated

temperatures. Additional modifications will be required to ensure the panels fabricated have no sign of mark-off in the regions where the sensors were placed above the part on the caul.

In summary, a preliminary automated cure monitoring system employing high-temperature piezoelectric transducers was developed to interrogate a twenty-four ply unidirectional composite panel fabricated from Hexcel® IM7/8552 prepreg during cure. The system and process initially developed in this work has the potential to be used in the future to dynamically control the cure cycle in a closed-loop process to maximize composite part quality and consistency. This is possible because the acquisition of the guided waves and the analysis of peak voltage and TOA metrics are done almost simultaneously in real-time during cure.

5. References

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