

Correlation Between Electromechanical Impedance and Thermal Analysis for Adhesive Cure Time Evaluation in SHM GW Applications

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Piezoelectric ultrasonic sensors used in Structural Health Monitoring (SHM) systems are usually attached to components by using adhesives with intrinsic properties that end up affecting their frequency-response. In this work, the electromechanical impedance of sensors attached to an aluminum sheet with three different epoxy resins was monitored during curing. Impedance change indexes (CIs) were calculated and compared with cure degree estimations from Differential Scanning Calorimetry (DSC). Results show that resonance frequencies were displaced by up to 30 kHz, and amplitudes changed considerably during the curing process of the selected resins. Results indicate that certain CIs show a high positive correlation value ($R=0.96$) with DSC data, and could therefore be used in practical applications to evaluate the curing status of the adhesive in SHM applications.

Keywords: *Adhesive, electromechanical impedance, change index.*

1. Introduction

Structural Health Monitoring (SHM) in the aerospace, nuclear and civil sectors can provide operators with information that can help to assess safety of processes and to schedule maintenance procedures. Ultrasonic Guided Waves (USGW) have successfully been used to monitor structures and to detect cracks in rails and airplane fuselage or corrosion in pipelines^{1,2}, presence of water on the surface of an aluminum plate³ and sensor/material interface degradation^{4,5}. A typical monitoring program includes the measurement of USGW signals of the pristine condition of a structure and a comparison with later measurements. A change in the signals indicates the appearance of an anomaly, and environmental effects can be partially suppressed through signal processing methods⁶⁻⁸. However, changes in the sensor itself are highly detrimental to the operation of such systems since they can mask indications caused by defects, especially those with slow growth rates.

Two-part epoxies are commonly used to attach piezoelectric elements to the structure to be monitored. These adhesives have a good adherence to a wide range of materials provided the interface is in appropriate conditions and the resin was properly selected. Manufacturers usually inform the curing times of these adhesives under controlled conditions, but in real field-conditions these times may vary. The frequency-response of the transducer is highly influenced by the thickness, uniformity and mechanical properties of the resin layer, and the inspection team must somehow decide how long it takes for the system to stabilize in order to collect

signals from the pristine condition and start the monitoring process. If monitoring starts too early, changes in the Young's Modulus of the resin (caused by incomplete curing) will increase the background noise and are likely to generate false calls (the system signals the presence of a defect when there are no defects in the component). On the other hand, if monitoring starts too late, potential events can be missed.

Curing of the resin can be affected by external factors that can alter the structure temperature such as direct incident sunlight, contact with fluids (in the case of pipelines), season of the year and regional atmospheric conditions (wind and rain). Curing at different temperatures also affects resin properties such as Young's modulus (E), yield strength and glass transition temperature (T_g), as shown by Carbas et al.⁹ (in general, for Araldite AV 138M, higher cure temperatures yielded higher values of T_g and E). Additionally, water absorption¹⁰ can alter polymers through plasticization and UV radiation¹¹ can cause degradation processes, so the ideal solution would be a method that can measure transducer stability integrated into the same equipment being used to carry out the SHM.

The degree of resin cure can be measured by methods found in laboratories such as Differential Scanning Calorimetry (DSC) but the equipment is usually a stand-alone machine that is not easily taken to the field, and alternative more portable methods are required for cure evaluation of a transducer in SHM applications in the field. A study carried out by Lindrose¹², for example, showed that ultrasound bulk wave velocity changes as the resin is cured, and Lim et al.¹³ evaluated the curing of Sikadur 330 epoxy by monitoring the displacement of the

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resonance peaks in electromechanical admittance readings of a sensor completely embedded in the resin.

Electromechanical impedance is a technique that can use piezoelectric sensors to monitor the integrity of components. The transducer is attached to a surface to be inspected and is excited by an alternating electrical pulse, causing it to change dimensions and mechanically load the structure. Readings of the resulting voltage and current can be modeled as an electric circuit^{5,14}, and the response of the sensor can be measured as a function of frequency. Wilcox et al.¹⁵ showed that electromechanical response is also a factor of density, stiffness and $\tan \delta$ of the medium being modeled, therefore impedance is affected by the local mechanical properties of the sensor, structure and interface. Studies such as in Giurgiutiu and Kropas-Hughes¹⁶ showed that the technique can detect the presence of cracks in aluminum samples with attached piezoelectric sensors by evaluating the response and searching for shifts in the existing peaks, the appearance of new peaks and peak splits. Tseng and Naidu¹⁷ monitored strips of aluminum and noticed that the damage index calculated based on impedance measurements could be related to holes drilled in the sample. Impedance readings taken by Qing et al.¹⁸ showed that peak position and amplitude are affected by the thickness of the resin used to couple piezoelectric elements to an aluminum plate. These results suggest that impedance can be used to evaluate the degree of cure of a piezoelectric transducer attached to a metallic sample, and could be a valuable tool for nondestructive inspection of components and structures.

The purpose of this work is to show how electromechanical impedance measurements taken with a custom build (US/impedance) portable system can be used to measure peak changes caused by the hardening of the resin, and thus to evaluate the stabilization of the mechanical behavior of three different epoxies. An impedance change index (CI) is calculated and compared with cure degree data obtained from the DSC technique for different adhesive cure times. Once this procedure is validated, it can be used to give better estimates of the time required for the system to settle in non-ordinary conditions, such as transducers attached to a pipe carrying hot fluids or sensors attached to an airplane wing that is exposed to the sun.

2. Materials and Methods

2.1. Adhesives tested

The three resins used in this work are two-part epoxies that were mixed according to the manufacturer’s instructions

and cured at room temperature. The following descriptions and additional information presented in Table 1 were obtained from the resin datasheets. Araldite professional (Ar. Prof.)¹⁹ is an adhesive mostly used in house repairs for joining non-structural components such as low pressure water pipes, wood, concrete and tiles. It has an initial hardening stage in 90 min and fully cures after 24 h. Fusor380²⁰ is an adhesive mostly used for joining fiberglass reinforced plastics, sheet molded compounds and metals. It can be handled 2 h after curing at room temperature. Araldite AV138²¹ is an adhesive used for joining plastics and metals exposed to high temperatures or aggressive environments.

2.2. DSC analysis

A small volume (approximately 5 mg) of each of the three combinations of resin and hardener was mixed and put inside a crucible for DSC analysis. The equipment used was a DSC Q20 equipment from TA Instruments, with a heating rate of 10 °C/min and N₂ flux of 50 ml/min. Measurements were taken after the resin had cured for the following periods: 0, 2, 4, 6, 8, 24 and 72 h. The first run was a heating cycle, where the enthalpy (heat released) was automatically calculated by the software (TA Instrumental Analysis) by integration of the area under the peak (peak start and end positions were hand-picked by the operator). This was followed by a cooling cycle and a second heating cycle, which should show that the peak has disappeared and that all functional groups of the epoxy have reacted. The degree of cure was obtained by comparing the enthalpy of the current measurement and the reference (measurement at 0 h).

The main objective of this analysis was to provide data concerning the percentage of cure achieved for each resin after the chosen periods. A correlation between these results and those obtained from impedance measurements is presented in Section 3.

2.3. Impedance monitoring

To simulate a monitoring case in an airplane fuselage, lead zirconate titanate (PZT) piezoelectric discs (PZT5A, diameter 20 mm, thickness 1 mm) were attached to an aluminum sheet (thickness 2 mm) through the use of the resins described in Section 2.1. The aluminum sheet was cleaned with 70% alcohol and a mask was used to control the thickness of the adhesives for the three sensors (one sensor for each resin) in the first part of the study. Copper contacts (thickness 0.2 mm) were placed between the PZT and the aluminum sheet to provide better electrical contact and also to allow a better control of the adhesive

Table 1. Characteristics of the epoxies used in the study.

Company	Epoxy		
	Huntsman	Lord	Tekbond
Resin (part A)	Araldite AV138-2BR	Fusor 380 NS	Ar. Prof. (A)
Hardener (Part B)	HV 998	383 NS	Ar. Prof. (B)
T _g (°C)	110*	90**	63*
Cure conditions	24 h at 23 °C	24 h at T>18 °C	15 h at 20 °C
Mix ratio by weight	100 resin 40 hardener	100 resin 95 hardener	80 resin 100 hardener

* ASTM D-4065. ** ASTM D882-63A.

thickness, even though this resulted in the sensor not being omnidirectional. A 3D printed plastic spacer was placed on top of the sensor to protect the upper contacts and a steel block weighting 110 g was used to mechanically load the sensor against the sample. A previous test, carried out to check if it was possible to make the electrical connections directly through the resin layer without the use of the copper contacts, resulted in signals that were unreliable and noisy, being affected by small displacements of the cable (coaxial RG174) that connected the sensor to the impedance analyzer. Therefore, the configuration with the copper contacts was implemented (Figure 1).

The aluminum sheet was placed inside a furnace to allow curing of the resin at approximately 25 °C. The furnace also acted as a Faraday cage and the measurements were less noisy than those carried out in the open (sheet placed on top of a desk in the lab). Coaxial cables were used to connect the sensors to a custom-made ultrasonic equipment capable of conducting Lamb wave excitation and electromechanical impedance measurements by switching electrical pathways inside the hardware. Calibration of the custom equipment was carried out by comparing results with a Keysight E4990A impedance analyzer and showed good agreement. Impedance measurements were taken every 5 minutes after the specimen was ready to cure for a period of 72 h and the following parameters were used:

- excitation was carried out sequentially with three Ormsby waveforms operating at the 2–319 kHz, 316–633 kHz and 630–947 kHz ranges, with a voltage of 300 Vpp.
- data were acquired with an acquisition board with a sampling rate of 2 MHz.

In the second and third parts of the study, three new PZTs (one for each resin) were attached to the same aluminum sheet and the impedance test was repeated to increase the confidence of the test results and provide repeatability.

The frequency range for impedance monitoring was chosen based on works conducted on similar samples or

which had similar objectives. In Lim et al.¹³, PZT patches were embedded in samples made of Sikadur 330 resin and monitoring was performed in the 5–900 kHz range (peaks were found at 150 kHz, 230 kHz, 450 kHz and 750 kHz). In Giurgiutiu and Kropas-Hughes¹⁶ three frequency ranges (10–40 kHz; 10–150 kHz; 300–450 kHz) were used to detect the presence of cracks in an aluminum sample. Tseng and Naidu¹⁷ detected the presence of holes drilled in aluminum strips by monitoring the peaks in the 100–150 kHz and 400–450 kHz ranges. Qing et al.¹⁸ studied how the thickness of the resin affects the peak position and amplitude in the 50–600 kHz range for PZTs attached to aluminum.

2.4. Change index

The Change Indexes (CI) used in this work are based on the Damage Indexes presented in Lizé²². Each CI is a measure of the distance between the current signal and a reference signal and belongs to one of three classes: index based on the energy, phase or amplitude of the signal. The reference signal adopted was the last signal collected, corresponding to a moment when the resin was considered to be fully cured, and so the CI initially had a higher value and converged to zero as time passed. The first collected signal was not chosen as the reference because the mounting of each PZT in the aluminum sheet took at least 2 min (the last sensor was ready when the first was already curing for 4 min), and thus the use of the last measurement as a reference represented a more uniform condition for all cases.

In total, 26 estimators were calculated based on the impedance data obtained from a window of a specific frequency range (Table 2). For the sake of brevity, only the equation for the DSEN (Diff shared energy norm) is presented below, Eq. (1), since this specific CI showed the best results. The equations for the other change indexes can be found in Lizé²² (chapter 4).

$$DSEN(x_1, x_2) = \frac{E_s \left(\frac{x_1}{\max(x_1)} - \frac{x_2}{\max(x_2)} \right)}{2 \left(E_s \left(\frac{x_1}{\max(x_1)} \right) + E_s \left(\frac{x_2}{\max(x_2)} \right) \right)} \quad (1)$$

where $E_s(x)$ is the energy (RMS) of the signal; x_1 is a vector of amplitudes corresponding to the reference signal and x_2 is a vector of amplitudes corresponding to the reference signal.

3. Results and Discussion

3.1. DSC thermal analysis

Figure 2 shows examples of curing curves for all resins at a period of 0 h (Figure 2a) and for Ar. Prof. at three different

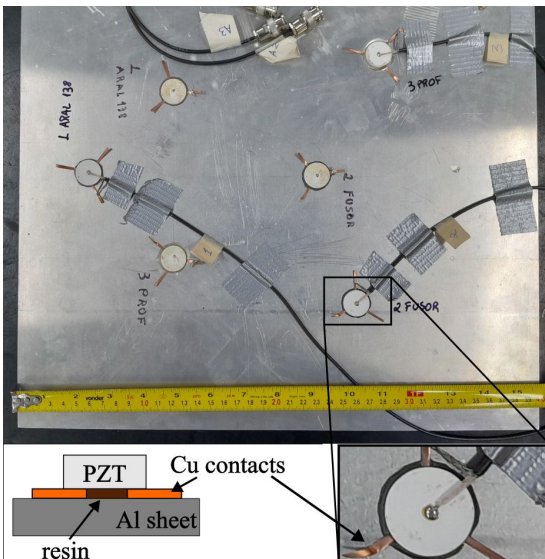


Figure 1. Mounting of the PZT sensors on the sheet with the use of Cu contacts and resin.

Table 2. Ranges of frequency chosen for the CI estimations.

Window	Frequency range (kHz)	
1	70 – 220	1st resonance
2	220 – 320	2nd resonance
3	70 – 550	All four resonances

cure periods (Figure 2b). In all cases, the second heating curve has no distinguishable peaks, showing that all species reacted in the first cycle and there were no leftover reactions. Also, as expected, there were fewer unreacted groups as the resin cured (from 0 to 4 and 72 h), and therefore the size of the peak decreased accordingly (Figure 2b). However, for 72 h, Ar. Prof. exhibited an anomalous behavior with energy absorption, which was also seen for AV138, but not for Fusor 380 (which showed the expected behavior for the reticulation of a thermosetting material).

The enthalpies calculated for the whole set of resins and cure times are presented in Figure 3 and in Table 3 (which also contains the estimated cure percentage). The AV 138 and Fusor 380 resins released smaller quantities of heat during cure and also cured faster (approximately 8 h is the time needed for them to converge to cure degrees >90%). After 72 h the resins should be fully cured, and the small deviations from zero in the enthalpy for some resins might be related to experimental imprecision and are not relevant.

The differences observed in the behaviors of the epoxies are likely to have been caused by the different formulations of the resins, which contain large percentages of unknown

compounds as listed in the datasheets provided by the suppliers. Ar. Prof. cures with diamine/triethylenetetramine (part B) and part A contains bisphenol A with two molecular weights, which might explain the apparent presence of two stages of curing: the first one occurring during the first 6-8 h and the second one occurring up to 24 h for a full cure, as seen in Figure 3. It also released heat levels almost two times higher than the other two adhesives, with a peak position generally occurring at higher temperatures (108 °C, Figure 2a) than AV138 and Fusor 380 (peaks at 86 °C and 87 °C respectively). AV138 is cured with a mixture (part B) composed primarily of fatty acids with a smaller quantity of amines and bisphenol A, while part A contains some compounds with lower concentrations and two major components: bisphenol A with epichlorohydrin and barium sulphate (both at 30-50%). This last component is considered to be an inert addition and should not participate in the cure reaction, but it interferes in the absorption/dissipation of heat when the adhesive is heated for long periods of time. Fusor is the resin with the least amount of available information regarding composition, being composed of a proprietary epoxy resin (55-60%)

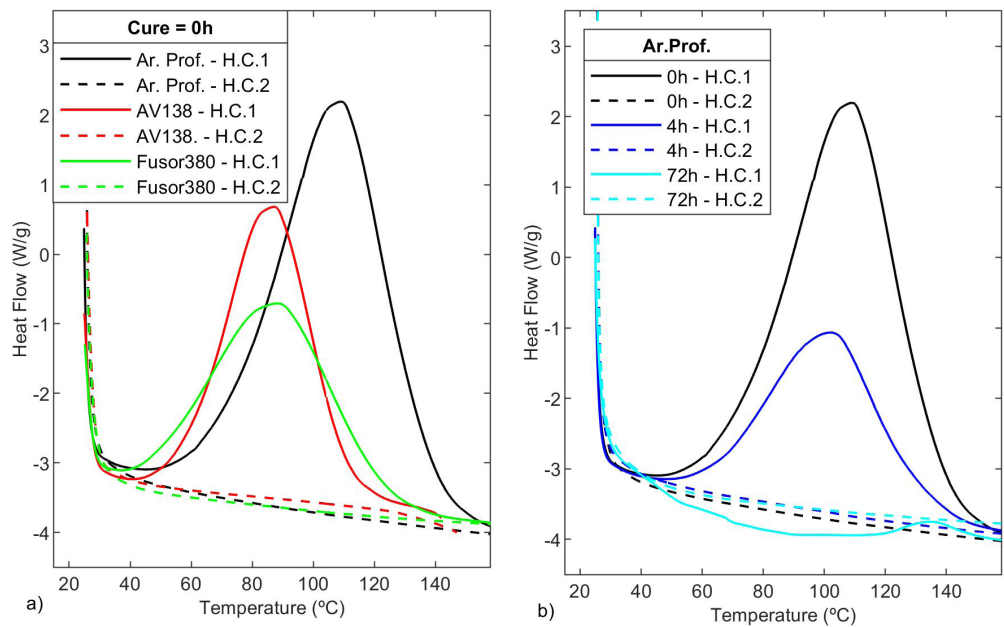


Figure 2. DSC thermograms for (a) all resins studied for a cure of 0h; (b) Araldite professional for multiple cure periods; H.C.1 and H.C.2 stand for heating cycle 1 and 2 respectively.

Table 3. Enthalpies and cure degree as a function of time passed after mixture.

Cure time (h)	Cure ΔH (J/g)			Cure (%)		
	Fusor 380	AV 138	Ar. Prof.	Fusor 380	AV 138	Ar. Prof.
0	105.4	139.8	289.3	-	-	-
2	62.46	54.35	260.8	40.5	61.1	9.9
4	31.73	28.68	127.6	69.9	79.5	55.9
6	19.24	14.18	98.31	81.7	89.9	66.0
8	8.673	12.0	87.06	91.8	99.9	69.9
24	2.722	13.77	6.345	98.4	91.4	97.8
72	0.276	5.232	4.207	99.7	96.3	98.5

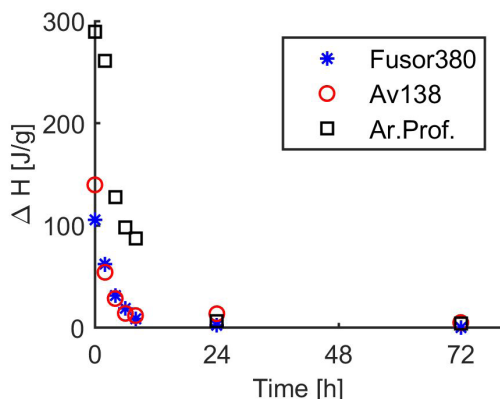


Figure 3. Enthalpy versus time plotted for the 3 resins tested.

that could be assumed to be diglycidyl ether bisphenol A (DGEBA). Fusor 380 cures with a mixture of a polyamide resin and amine compounds (also proprietary information), with P-chlorophenol and carbon black. Both AV138 and Fusor 380 are described as thixotropic pastes and have viscosities significantly higher than Ar. Prof., which is probably caused by the addition of the inorganic compounds (barium sulphate and carbon black) previously mentioned. Although this higher viscosity makes the resin more difficult to be spread evenly in the PZT sensor, it has a positive aspect: the sensors were less likely to be displaced after being attached to a sample. This displacement can degrade coupling and might be caused by tension in the wiring connecting the PZT to the equipment, or by gravity if the sensor is mounted on a vertical wall.

3.2. Impedance monitoring

The curves obtained during the monitoring of test 1, test 2 and test 3 exhibited very similar behaviors, with some minor differences at higher frequencies, which were associated with the contact placements. For brevity and since all graphs showed similar behaviors, only the results of round 3 are presented in Figure 4 for frequencies up to 530 kHz, and show a displacement of the four first resonances to higher frequencies as time passed. The peaks at higher frequencies (>530 kHz) also moved to the right and showed a relatively constant amplitude, being of little interest for this application.

For all resins in Figure 4 the first peak of resonance (approx. 120 kHz) displayed a considerable monotonic decrease in amplitude. The amplitude of the second peak generally increased for AV138 (Fig. 4a, b) and Fusor 380 (Fig. 4c, d), and oscillated up/down for Ar. Prof. (Fig. 4e, f). Both first and second resonance peaks showed a frequency displacement that is easily measurable and are likely to be good indicators of the cure process (frequency changed from 120 to 150 kHz for peak 1; from 260 to 290 kHz for peak 2). For all resins, the third peak was not visible in the early stages of curing and slowly started to appear as time passed. In all cases (Fig. 4a – Fig. 4e), the fourth peak showed the largest displacement in frequency content but also showed a parabolic behavior in amplitude (noticeable in Fig. 4b, d, f) with the lowest values in Fig. 4e very close to the baseline, which hindered monitoring of the exact peak position. A displacement of the

peaks to higher frequencies was also observed by Lim et al.⁹, although in that work the peaks initially (first 6 h of curing) moved to lower frequencies and only later moved to higher frequencies (the rate decreased as time passed and apparently stabilized after 14 days). Despite the fact that the resins used by Lim et al.⁹ were different from those analyzed in this work, and that the authors monitored PZTs completely embedded in a full epoxy sample, the resonance peaks were shown to increase nearly 30 kHz (from 132-138 kHz to 157-172 kHz), which agrees with the results shown in Figure 4. A parabolic behavior of the impedance peaks was also reported by Lizé²², although in that case the resin was already cured and the author was studying the effect of temperature on the frequencies of resonance. Since the resins have different chemical compositions and inorganic additions, it is expected that they will show the parabolic amplitude behavior for different resonance peaks.

During the monitoring of the second test, some data were lost due to a power outage which occurred approximately at 8.5 h and turned off the system for a few hours. Although it was a nuisance, this power outage did not have much impact in the experiment since the correlations with the DSC were taken at 8 h and 24 h, and the impedance data for these periods were available.

3.3. Change indexes and correlation between impedance and cure

The CI calculations for the first, second and third tests are shown in Figure 5a, 5b and 5c respectively. In general, the Fusor 380 resin exhibited a behavior similar to AV138, with the CI decreasing quickly (reaching convergence at approximately 7 h), although AV138 presented a larger standard deviation between 4 and 9.5 h (Figure 5a), mostly caused by data scattering in test 3. The reason for this is unclear; it might be the result of the aging of the reagents (part A and B of the resin), electrical contact placement between the PZT and the aluminum plate or a non-centered positioning of the steel block on top of the PZT. The CI for the Ar. Prof. resin decreased a bit slower than the others and shows an inflection at 4 h, eventually converging to values near zero at a later time (12 h approximately), which agrees with the results obtained from the DSC. Since the electrical properties of the cured resins are not likely to have changed significantly, the resulting changes in CI reflect a change in mechanical behavior of the system, which is inferred to be related to changes in the mechanical properties and cure of the resins.

Figure 6 shows the results of the DSEN for window 1 (70-230 kHz) versus the DSC data, where the perfect Pearson correlation ($R=1.00$) would be represented by a straight line 45° from the origin. Although the AV138 and the Ar. Prof. showed larger standard deviations for CIs smaller than 0.4, all resins showed a positive behavior and a good correlation between Normalized CI and Normalized Enthalpy. This relatively similar trend between resins is an encouraging result and shows the potential of the technique, although more tests with different resins should be performed in the future to confirm these results. For each frequency window, the average of all the nine R values (3 resins x 3 rounds) was calculated and resulted in correlations of 0.95, 0.79 and 0.96, for windows 1, 2 and 3, respectively. For this CI, window

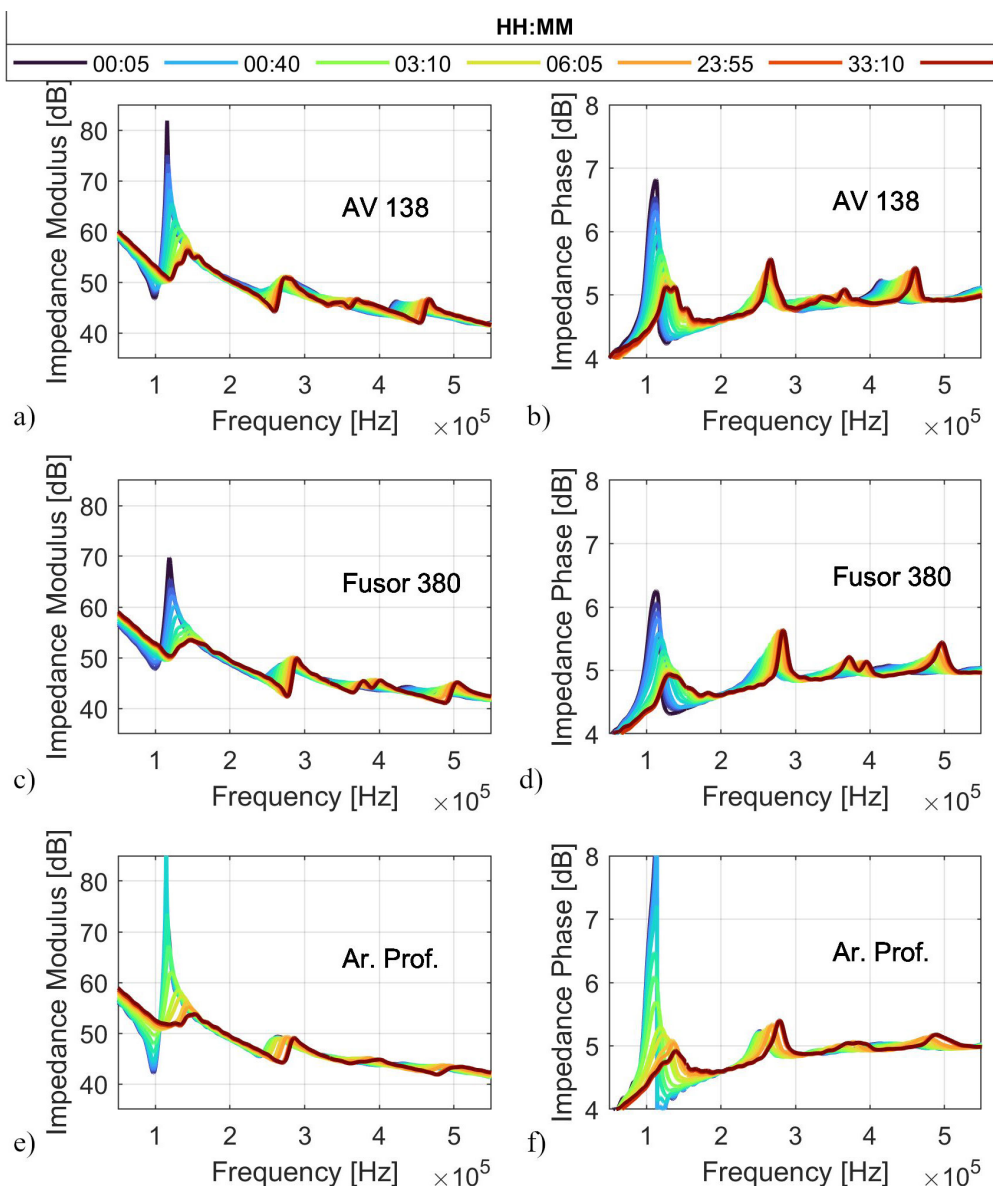


Figure 4. Electromechanical impedance of the resins in test 3; (a) magnitude and (b) phase for AV138; (c) magnitude and (d) phase for Fusor 380; (e) magnitude and (f) phase for Ar. Prof.

2 apparently did not provide useful information, since it showed the lowest R value. It should be noticed that the frequency range in window 3 encompassed both window 1 and 2, and the correlation found for window 3 was only marginally superior to the one observed in window 1.

Analysis of the other CIs revealed some indexes with non-linear positive behaviors, which were associated with lower values of R, as shown in Figure 7. Also, in several cases, the Ar. Prof. resin stood apart from the others and this caused a worse result since the mean of the three curves was used to calculate the final group correlation. The data measured for the Ar. Prof. suggests a second curing stage, and since there is limited DSC data between 6 and 24h (Figure 3) this could cause a worse result for the correlation.

A summary of the correlation for all CIs for the three frequency ranges is shown in Figure 8, where the CIs are

grouped by class (phase, energy, or amplitude). Some Energy CIs yielded a high negative correlation, and could potentially be used as an alternative method for evaluating resin cure, but for ease of use, a positive linear behavior is preferable. For the CIs that presented a positive non-linear behavior, a similar argument could be used, but a non-linear behavior represents a resolution problem since some regions are better represented than others. The energy group of CIs showed higher correlations in general (higher means, but also higher spread), followed by the amplitude CIs and the phase CIs in the last place. This encourages the choice of DSEN as a proper CI, since the other CIs that are based on energy, although calculated differently, also provided good results, and thus the good performance of the DSEN is unlikely to be due to chance. Concerning the analysis of the preferred frequency ranges, all windows showed a similar

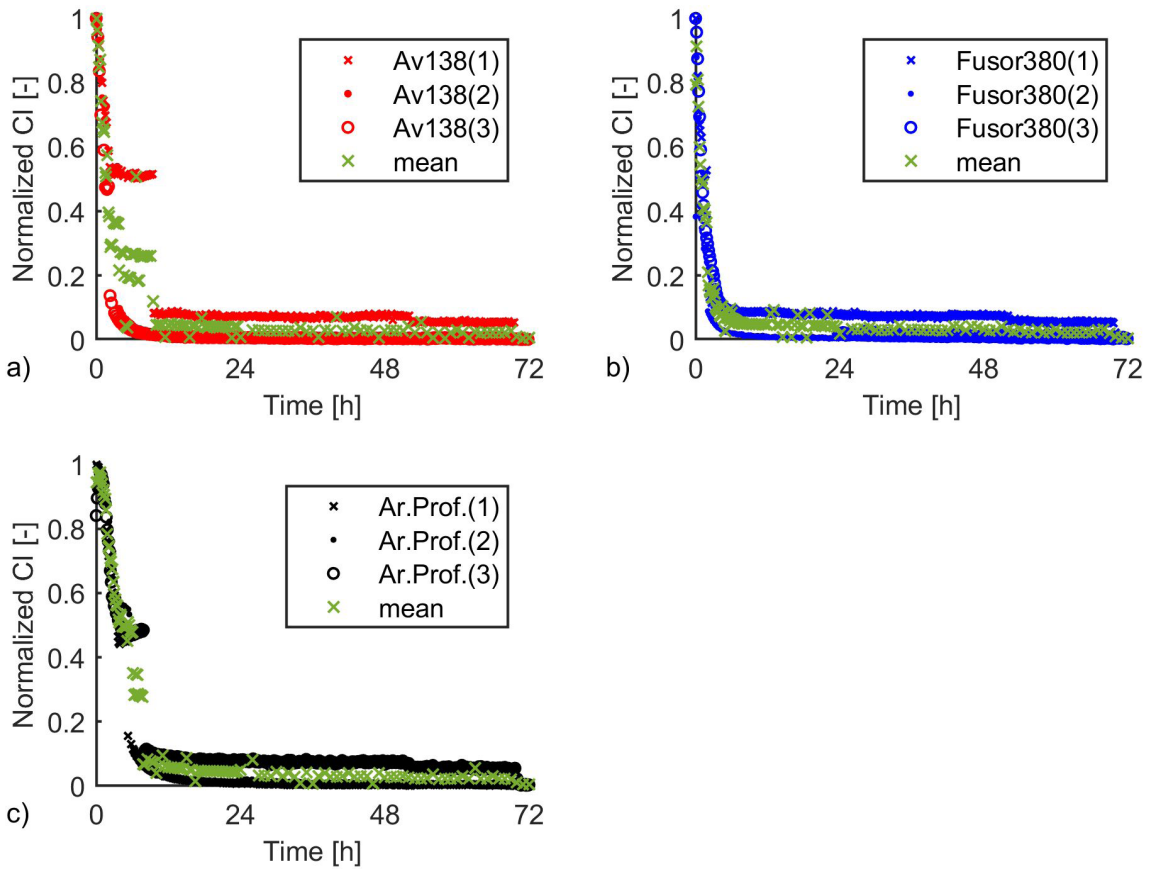


Figure 5. DSEN (data for tests 1, 2, 3 and mean values) in the 70 - 220 kHz range vs curing time for Av138 (a), Fusor 380 (b) and Ar. prof. (c).

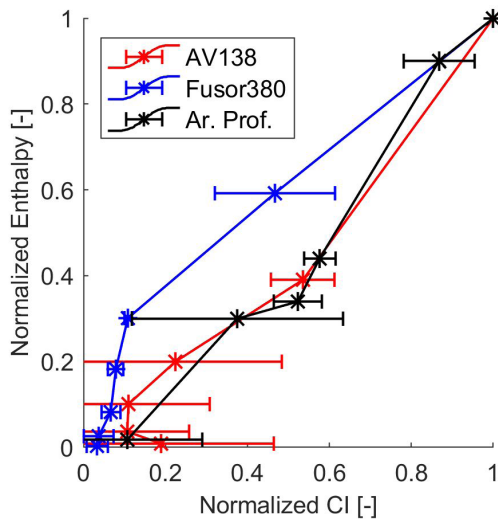


Figure 6. Mean values (stars) and standard deviation (bars) for the Normalized Enthalpy versus Normalized CI (DSEN) (calculated from window 1) for each resin.

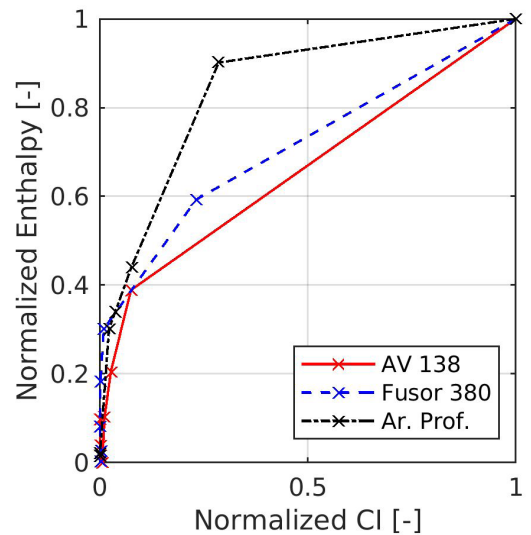


Figure 7. Normalized Enthalpy versus Normalized CI "SignalDiffCoef" (calculated from window 1).

pattern. The median for the energy group in window 1 was 0.85, which is equal to or higher than in the others (0.78 for window 2 and 0.85 for window 3), so the frequency range of 70-220 kHz (window 1) is the one recommended to be used.

A qualitative analysis was also performed, where the CIs were ranked from 1-3 according to the agreement (distance) between each curve and a hypothetical 45° straight line from the origin. After ranking, the mean for each CI

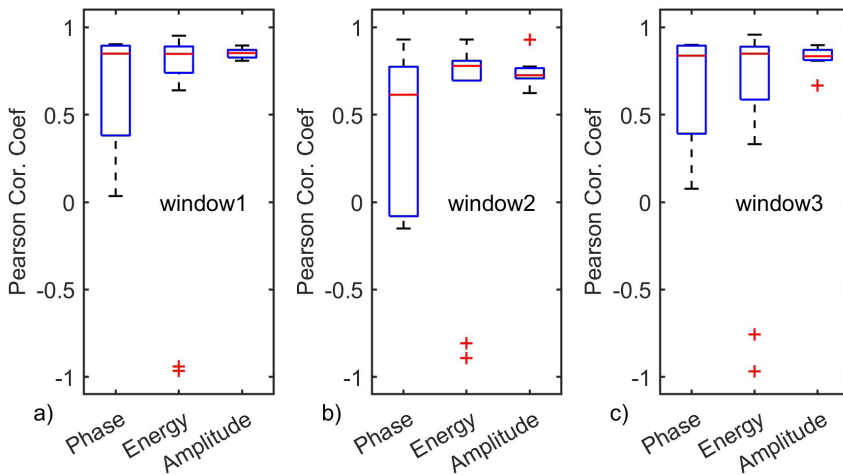


Figure 8. Box-plot representation of Pearson correlation coefficients for window 1(a), window 2(b) and window 3 (c).

in each window was calculated to provide a group value. This evaluation resulted in the choice of the Energy class of CIs for the experiments in both test rounds, with window 2 (for round 1) and window 1 (for round 2 and 3) being selected as best. Despite being subjective to the operator, these results agreed with those obtained from the Pearson Correlation calculations and reinforce the suggestion of using the DSEN and window 1 as a method for evaluating resin curing. The DSEN belongs to the CI class (energy) with the best group results in general for all windows, and window 1 showed good results over the whole range of parameters studied. It should be highlighted, though, that in some specific cases a high correlation was obtained for a specific CI and window combination, but when either the window or CI was held constant and the other condition was changed, the correlations dropped significantly. These cases were considered as outliers, or cases with minor practical significance, and were disregarded for future use.

4. Conclusion

The data obtained in these experiments showed it was possible to monitor the change in the response of the resins to an electromechanical excitation as time passed, by inspecting the resonance peaks with the electromechanical impedance technique. Significant changes in amplitude of the first and second resonances were observed as well as a 30 kHz peak displacement. Change indexes were calculated and the best results were obtained for the DSEN and a frequency range of 70-220 kHz. These changes were correlated to results obtained from DSC experiments and it is reasonable to infer that curing of the resin caused the observed impedance changes due to changes in mechanical properties. Therefore, the technique could be used as a means to signal that the resin has fully cured and the transducers are ready to acquire data for the pristine condition of the structure being monitored. Specific procedures should be chosen by each inspection team, but could follow a rule such as the system is considered to be ready to start monitoring when the change index has converged to a point where additional readings only change the DSEN value by 0.2%.

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