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Huikangyue Bao, Philippe Marguerès, Philippe Olivier. OUT-OF-AUTOCLAVE CURE MONITOR-ING OF CFRP LAMINATES USING AN INNOVATIVE AND LOW-COST ELECTRICAL SYS-TEM. 21st European Conference on Composite Materials, Jul 2024, Nantes (France), France. pp.599-606, 10.60691/yj56-np80. hal-04638875

HAL Id: hal-04638875 https://hal.science/hal-04638875

Submitted on 8 Jul 2024

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OUT-OF-AUTOCLAVE CURE MONITORING OF CFRP LAMINATES USING AN INNOVATIVE AND LOW-COST ELECTRICAL SYSTEM.

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Keywords: CFRP, Cure monitoring, Electrical Impedancemetry, material functionalization, smart material

Abstract

This work introduces an innovative real-time cure monitoring method for Carbon Fibre Reinforced Polymer (CFRP) manufacturing, using Electrical Impedancemetry (EI) and self-sensing material. The method monitors changes in the electrical complex impedance, which can be related to the degree of cure of the thermoset matrix. The study was initially conducted using a commercial single-channel impedance analyzer, HIOKI, which had limitations in terms of measurement channels and data acquisition rate. To overcome these limitations, a new EI measurement bench was developed. This multi-channel bench, based on the Digilent PmodIA module and an Analog-Front-End (AFE), costs only 15% of the HIOKI analyzer and has a data acquisition rate 48 times higher. It maintains an equivalent impedance measurement range and allows for the monitoring of the degree of cure and oven temperature, as well as the detection of potential cure cycle issues. The method was validated on different CFRPs, showing a significant difference in cure behavior between unreinforced resins and composites. The EI results were in good agreement with those obtained by rheometer and differential scanning calorimetry. This approach will contribute to the Structures Health Monitoring (SHM) of CFRP and the functionalization of carbon/epoxy composites and will be used for refining electrical-mechanical modelling during curing and mechanical loading.

1. Introduction

In the context of carbon fibre reinforced polymer (CFRP) manufacturing process, conventional online cure monitoring techniques are usually based on expensive measurement systems using dedicated sensors. As a solution, a low-cost real-time cure monitoring method, Electrical Impedancemetry (EI), is proposed. Unlike Dielectric Analysis (DEA), which is commonly used for Glass Fiber Reinforced Polymers (GFRP) or unreinforced matrix, EI can be used for CFRP, which has an anisotropic electrical response due to the presence and orientation of carbon fibres. The approach uses the material itself as a sensor, correlating changes in rheological properties with the electrical impedance of CFRP, enabling the identification of different physicochemical transition phases of polymerization. The cure behaviour of CFRP is represented by the evolution of the modulus and argument over time, and their first and second derivatives, allowing for the monitoring of four phase transitions during curing: liquefaction, gelation, vitrification, and end of cure. In a recent previous work [1] we studied phases transitions in CFRP during their curing by EI showing that changes in the electrical complex impedance Z can easily be related to those of conventional parameters such as the thermoset matrix degree of cure α . This work was carried out using a commercial single-channel impedance analyser: HIOKI. In addition to its high acquisition cost, this device presented certain limitations in terms of measurement channels (only one) and data acquisition rate (60s for a frequency sweep).

These facts led us to develop a new EI measurement bench for monitoring the impedance changes in CFRP part for aeronautical applications (airframe structures). The innovative new multi-channel (8) bench we designed and manufactured is based on the Digilent PmodIA module (never used previously for this purpose) and an Analog-Front-End (AFE) developed for EI measurements. It costs only 15% of the HIOKI analyser and has a data acquisition rate 48 times higher (1,23 s versus 60 s). All this while maintaining an equivalent impedance measurement range: $100m\Omega$ to $1M\Omega$. This approach not only

makes it possible to monitor the degree of cure α (DOC) by the EI and the oven temperature, but also to detect potential cure cycle issues. The sweep frequency response analysis of the material (using impedance modulus |Z| and argument θ and their time derivatives during curing), enables the identification of the most critical points of polymerization: Liquefaction (PL), Gelation (PG), Vitrification (PV) and Finish of reaction (PF). The paper is organized into sections discussing preliminary works, samples and experimental setup, correlation between cure monitoring results of DSC and EI, and a summary of main results and future perspectives.

2. Thermal analysis of thermoset

This work aims to correlate the curing process of Carbon Fibre Reinforced Polymer (CFRP) with its electrical behaviour. It identifies critical rheological points such as Liquefaction (PL), Gelation (PG), Vitrification (PV), and Finish of reaction (PF). The changes in electrical impedance (Z) are studied as a function of the degree of cure (α), which is determined by thermal analysis and cure kinetics. This correlation can contribute to future multi-physics modelling of CFRP. The Electrical Impedancemetry (EI) method used for cure monitoring offers several advantages over conventional techniques, making it more applicable in CFRP manufacturing. Thermal analysis techniques are used to understand the properties of polymers, their thermal stability, and decomposition behavior. These techniques, including Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA), Dynamic Mechanical Analysis (DMA), and Thermal Mechanical Analysis (TMA), help predict performance and optimize the cure cycle. Our methodology involves comparing physicochemical results to Electrical Impedancemetry (EI) results. DSC is used to determine the glass-transition temperature of the polymer and its cure kinetic parameters. The degree of cure (α) determined by DSC is crucial for linking the electrical behavior with the physicochemical transitions of the thermoset matrix.

During a Differential Scanning Calorimetry (DSC) scan, a sample is cured at a constant heat rate, isothermal temperature, or using a specific cure cycle. The heat flow is measured by DSC. The enthalpy H of the reaction is calculated as the integral of heat flux over time to determine the progress of the sample's polymerization. The degree of cure, $\alpha(t)$, is given by the ratio of H(t) to HT, where HT is the total enthalpy for the complete polymerization of the thermoset resin. The cure kinetics of the polymeric matrix allows for the determination of different critical points of polymerization by analyzing changes in α during a cure cycle. The DSC results can differ between unreinforced resin and carbon/epoxy prepreg due to the presence of carbon fibers, which have higher thermal conductivity than the polymeric resin, thus modifying the material's coefficient of thermal conductivity.

The rheological critical points, denoted by PL, PG, PV, and PF, of the unreinforced resin epoxy M21 were determined through DSC and parallel-plate rheometer experiments. As described in Figure 1, these points correspond to various stages of the curing process, including:

- PL can be found with the minimum points of viscosity $\alpha(t)$ and tan δ , the onset point of $\alpha(t)$ and the first maximal value $\alpha''(t)$.
- PG can be found with the major inflection point after PL of the viscosity and degree of cure (between 0.4 and 0.6 [6-11]), $\tan \delta = 1$ and the first minimal value of $\alpha''(t)$.
- PF can be found with the final maximum points of viscosity and degree of cure $\alpha(t)$, a constant value of the conservative shear modulus G, loss shear modulus G" and tan δ .
- PV can be found with the transition between the end of gelation and PF, a local maximum of G" and tan δ after the end of gelation, and $\alpha(t)$ tending between 0.9 and 0.95 [6, 11].

Figure 2 gives the obtained points for an unreinforced M21 epoxy resin. Two different time scales are used to indicate when these points occur: one is the chronological cure time, and the other uses the onset of isothermal dwell as the origin of times. For instance, PL happens 67 minutes after the start of the cure cycle or -6 minutes before the onset of the curing dwell at 180°C. The polymerization is not complete due to the cure cycle, resulting in α at PV being around 0.86 and at PF around 0.95.

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Figure 1. Identification of PL, PG, PV, and PF using $\alpha(t)$ for a complete polymerization (α reaches 1)



Figure 2. Identification of PL, PG, PV, and PF using α for an unreinforced M21 epoxy resin during cure-cycle.

3. Cure monitoring using Electrical Impedancemetry

Various methods have been developed since the 1970s to monitor changes in composite materials during curing and mechanical loading. These include Bragg gratings, ultrasonic or infrared sensors, eddy current control, and piezoelectric sensors. Dielectric Analysis (DEA) is particularly valued as it monitors real-time cure of polymers and is based on the same principles as Electrical Impedance Analysis (EI). Changes in the complex dielectric permittivity of the polymer during curing correlate with the physicochemical transition of the polymer [11, 12]. Dielectric sensors, crucial for Dielectric Analysis (DEA), can be disrupted by the electric field of carbon fibers, limiting DEA's application to insulating materials like unreinforced polymers or Glass Fiber Reinforced Polymers (GFRP). To address this, a new EI method has been developed, using Carbon Fiber Reinforced Polymers (CFRP) themselves as sensors. This method analyzes the complex electrical impedance, which contains resistive information about the carbon fibers and capacitive information about the resin.

CFRP structures exhibit anisotropic properties, including electrical conductivity. Carbon fibers show pure resistivity longitudinally, while transverse conduction depends on inter-fiber contacts and fiber volume fraction, modeled as a network of resistors and capacitors. This has led to a homogeneous conduction model of the CFRP structure, composed of parallel RC circuits. The relationship between the function of impedance \bar{Z} and the equivalent RC circuit of the model is illustrated in the following equations. ω (rad/s) is the signal pulse with $\omega = 2\pi f$ (Hz); R is the resistance (Ω) and C is the capacitance (F) of the material; j is an imaginary number such that j²=-1.

$$\bar{Z} = \frac{R}{1 + j\omega RC}$$
(1)

$$|Z| = \frac{R}{\sqrt{1 + \omega^2 R^2 C^2}}$$
(2)

$$\theta = -\arctan(\omega RC)$$
 (3)

In preliminary research, CFRP laminates (made from T700/M21 carbon/epoxy prepreg) were monitored in real-time during their polymerization in an oven or autoclave. This was done using a bench that included the Hioki IM3570 impedance analyzer. The samples were continuously excited by a sinusoidal sweep signal, and polymerization behaviors were observed using time functions such as |Z|=f(t) and $\theta=f(t)$. Several stacking sequences were studied, including unidirectional samples ($[0^{\circ}_8]$, $[0^{\circ}_{16}]$, and $[0^{\circ}_{32}]$) and quasi-isotropic samples ($[0^{\circ}+45^{\circ}/-45^{\circ}/0^{\circ}]_s$, $[0^{\circ}+45^{\circ}/-45^{\circ}/0^{\circ}]_{2s}$, and $[0^{\circ}+45^{\circ}/-45^{\circ}/0^{\circ}]_{4s}$). The global tendencies of electrical responses of the quasi-isotropic (QI) and unidirectional (UD) samples were found to be similar. For QI samples, the electrical conductivity was enhanced compared to unidirectional samples, possibly due to a better percolation network. Two types of electrodes were compared: a copper tape and a copper mesh. From a microstructural perspective, no defects were found in the area of the copper mesh after manufacturing of samples. However, plain copper tapes hindered resin flows during curing and generated local porosities. Mechanical tests showed that the copper mesh electrodes had no harmful effect, unlike the ones using plain copper tape. This led to the choice of only using electrodes made of copper mesh.

The samples studied here are CFRP laminates made from NC66/NA1808 prepregs and unreinforced NA1808 matrix samples, to highlight differences in behavior and the effect of carbon fibers.

4. Experimental setup and materials

DSC and EI experiments were conducted on NA1808 resin and NC66/NA1808 prepreg samples using a homemade bench, an impedance analyzer (Hioki IM3570), a thermocouple data logger (PicoLog TC-08), a curing oven, and a DSC device (Q50-TA instrument). The samples were oven-cured under the same conditions, and the results were compared to validate the consistency of the curing process. Samples are 10 x 10 cm² square unidirectional plates: 2 mm-thick $[0^{\circ}_{8}]$ and 4 mm-thick $[0^{\circ}_{16}]$ laminates. DSC data were used to build cure kinetics based on Kamal-Sourour modelling [7].

Thin, flexible copper fabric electrodes are embedded in CFRP laminates in various configurations to ensure current flow in different directions. In the longitudinal and transverse configurations, two electrodes are inserted between the two highest plies of the laminate. In the diagonal-through-the-thickness-longitudinal and diagonal-through-the-thickness-transverse configurations, the first electrode is inserted between the two highest plies, and the second is inserted between the two lowest plies.

Former results have demonstrated the equivalence between diagonal thickness and through-thethickness direction EI measurements and the non-intrusiveness effect of copper fabric electrodes. After curing, the electrodes remain in the CFRP laminates for further structural health monitoring.

Unreinforced matrix samples are made by placing NA1808 on a silicone rubber frame. The frames are sealed with an insulating adhesive with a higher dielectric constant than the resin to prevent short-circuiting during high-frequency electrical excitation. Two identical electrodes are placed on the top and bottom surfaces of the resin sample.

4. Measurement bench

A new homemade bench has been developed, featuring eight channels for Electro-Impedance (EI) measurements and two channels for temperature measurements. EI measurements are conducted with eight PmodIA impedance analyzers and a multiplexer (MUX). The samples are excited by a sinusoidal sweep voltage, and the instantaneous response in |Z| and θ is calculated and converted to a digital signal.

A Data Acquisition Board (DAQ) is used for transmission from the bench to the PC. The samples, located in the oven, are connected to the bench using HiFi wires, which have a resistance lower than 0.01Ω and an inductance lower than 1×10^{-5} H. The connection between the HiFi wires and the copper electrodes is made by soldering. Open-circuit and short-circuit experiments were performed to measure the parasitic elements and to demonstrate that the wired system does not have a thermal response. The short-circuit impedance of the system is around 4.6 Ω , and impedances below this value will be distorted.

The accuracy of the new bench was evaluated by measurements on T700/M21 cured samples. The Hioki IM3570 was used in continuous acquisition mode for comparison purposes during curing. The proposed scanning frequency range is 5 kHz to 50 kHz with a 5 kHz step, including the optimal frequency F=10 kHz for the T700/M21. The data acquisition time in an EI channel is approximately 1.23 seconds, which guarantees the accuracy of the first- and second-time derivatives of |Z| and θ . A PicoLog module was used to measure temperature during curing via up to 8 thermocouples. The thermocouples, linked to the bench and to the PicoLog module, are placed next to the laminates under the vacuum bagging film. To ensure longitudinal EI measurements, a specific Analog Front End (AFE) has been developed to enable four-wire measurements with PmodIA. The results obtained with and without the AFE will be discussed in the next section.

4. Results

Transverse through the thickness EI measurements on NC66/NA1808 $[0^{\circ}_{8}]$ samples were performed during curing with a sweep frequency from 5 kHz to 50 kHz with a peak-to-peak voltage (Vpp = 0.84 V). As shown in Figure 3, the |Z| curves overlap, while the θ curves remain generally parallel, tending towards 0° at the end of curing. The samples are so almost purely resistive in the thickness direction because of a high fiber content (65%). The results obtained on the T700/M21 samples (fiber content equal to 51%), used in previous works, were more frequency-dependent.

To validate the measurements, two identical cure cycles were also measured using a Hioki impedance analyzer, with similar results trends for all $[0^{\circ}_{8}]$ samples (Figure 4 left). The trends given by the EI measurements and cure critical point detection for NC66/1808NA using the new bench are identical to those obtained for T700/M21 samples using the previous bench, confirming the effectiveness of the new low-cost bench (Figure 4 right). Based on changes in |Z| and θ during curing (Figure 5), PL and PG are identified by a local minimum and maximum of |Z| respectively. PV and PF are identified using θ measurements and the first and second-time derivatives of |Z|. After PG, the |Z| curve can be divided into three stages: dynamic, damping, and stationary.



Figure 3. Transverse through the thickness EI measurements on a NC66/NA1808 [0°_{8}] sample: frequency response in θ (left) and |Z| (right).



Figure 4. |Z| and θ measurements at 10 kHz for several curing (noted C_i) on several samples (noted E_i): (left) NC66/NA1808 [0°₈] samples using the new homemade bench and Hioki impedance analyzer, (right) T700/M21 [0°₈] samples using Hioki impedance analyzer.



Figure 5. PL, PG, PV and PF identified on NC66/NA1808 $[0^{\circ}_{8}]$ samples using: (left) |Z| and θ and (right) first and second derivatives of |Z|.

Measurements on the longitudinal direction on NC66/NA1808 $[0^{\circ}_{8}]$ samples using PmodIA and AFE showed that while PmodIA results were distorted by system parasitic impedance, AFE could measure small impedances with higher sensitivity. Despite incorrect |Z| values from PmodIA, overall behavior matched AFE, allowing identification of critical points. Thus, monitoring the cure cycle relies on changes and variation in |Z|, not its value. All critical points can be detected using |Z| and |Z|' as for measurements in transverse through the thickness direction.

Unreinforced resin samples, unlike CFRP laminates, show isotropic behavior when excited by a sweep frequency voltage. Their response in |Z| and θ is frequency dependent. At the end of the liquefaction phase, the resin reaches the minimum viscosity and the maximum ionic conductivity. This correlates with the main peak that corresponds to the minimum of |Z| and the maximum of θ . The resin exhibits a high capacity before and after PL due to the predominance of dipolar rotation over ionic conduction. After PL, the θ curve can be divided into two stages: a dynamic part and a more global stationary part. The transition point between these two parts, PG, can be identified using the asymptotic method. In the more stationary part, two slight transitions can be detected using |Z|' and θ' , and are associated with PV and PF. These points can be detected on all samples and at all frequencies.

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Figure 6. PL, PG, PV and PF identified on NA1808 unreinforced matrix.

Table 1 presents the results derived from both EI and DSC. The EI results are an average of several samples across different curing cycles. The EI method was successful in identifying all critical points, and these findings generally align well with the DSC results. The observed discrepancies in the results, especially for PL and PG, could be attributed to the presence of carbon fibers and the mass effect. The thermal inertia's impact during the curing process can also contribute to these differences.

uniennoiced matrix samples.				
Points	Matrix EI (min)	Matrix DSC (min)	Laminate EI (min)	Laminate DSC (min)
PL	-1.28±0.04 s	-0.63±0.04	-0.25±0.06 s	-0.70 ± 0.11
PG	6.00±0.6 s	6.06 ± 0.06	6.91±0.05 s	7.16±0.33
PV	36.10±0.33 s	35.66±0.20	36.88±0.63 s	35.55±0.72
PF	82.50±0.46 s	81.03±.0.27	76.13±0.71 s	78.00±0.23

 Table 1. Comparison of PL, PG, PV and PF obtained by EI and DSC for laminates and NA1808 unreinforced matrix samples.

Two tests for detecting defects during the curing process were performed. The first test examines the impact of a lack of vacuum on the curing process. A CFRP $[0^{\circ}_{8}]$ sample was placed outside the vacuum bag and its behavior was compared to a similar sample inside the vacuum bag. The lack of vacuum significantly altered the cure behavior, particularly before the vitrification phase, and resulted in a final |Z| value almost twice the normal value. The second test involved interrupting the cure cycle for 4 minutes, 14 minutes before the isotherm dwell arrival time. This was done on two different stacking sequences: $[0^{\circ}_{8}]$ and $[0^{\circ}_{16}]$ samples. The interruption caused a time delay for PL and PG but did not alter the overall behavior of the |Z| curve. In conclusion, both the lack of vacuum and the cure interruption can be detected by the proposed EI approach. The lack of vacuum results in significant changes in the cure behavior, while the cure interruption simply results in a time delay.

3. Conclusions

This study presents a new approach for cure monitoring of Carbon Fiber Reinforced Polymer (CFRP) using electrical impedancemetry (EI). This method uses a low-cost homemade bench with PmodIA analyzers. The study used different samples of NC66/NA1808 prepreg and NA1808 unreinforced resin to identify four critical points in the resin polymerization process: the liquefaction point (PL), the gelation point (PG), the vitrification point (PV), and the end of reaction (PF). The results showed good agreement in defining these points using the EI methodology, despite some discrepancies between the samples. The approach also demonstrated the ability to detect faults during curing, such as a lack of vacuum and a break in the cure cycle. The presence of carbon fibres results in a significative difference

between the unreinforced resins and the composites in their cure behaviour. Compared to the results of a preliminary study, a similar behaviour is obtained on different CFRPs (commercial references Hexcel composites T700/M21 and CTMI NC66/1808NA laminates) using two different benches, highlighting the value of the proposed approach. The EI results are in good agreement with α obtained by rheometer and differential scanning calorimetry (DSC). The Electrical Impedance (EI) method applied to cure monitoring has several advantages: the CFRP material is used as its own sensor, the method does not induce significant changes in the overall manufacturing process, and the copper mesh electrode remaining in the CFRP structure can be used for Structural Health Monitoring (SHM) or for active Non-Destructive Evaluation (NDE), such as InfraRed thermography, or de-icing in the aeronautics field (wing leading edges). The total cost of the measurement system is only 15% compared with the previous bench using Hioki impedance analyzer.

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