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CONTACTLESS DIELECTRIC PROCESS MONITORING (CDPM) OF POLYMER COMPOSITES MANUFACTURING

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Abstract: *A feasibility study has been performed systematically to develop a real-time contactless measurement of the dielectric properties (DP) of composite surface by means of a high-frequency, free-space vector network analyser (VNA) associated with one steel conical horn antenna (mono- static method). Examinations of the dielectric spectra of thermoset epoxy systems over a frequency range of 1kHz to 10GHz have shown* α *and* γ *relaxations across a range of cure temperatures [1]. The* γ *relaxation occurring at relatively high frequencies is related to the motion of the molecules while the* α *relaxation is attributable to epoxide groups in the reacting systems . A thermoset epoxy resin was selected for this feasibility study to assess its relaxation characteristics (sensitive to the degree of cure) in real-time using a remote VNA. The assessments were carried out at 60 °C and 80 °C curing temperatures.*

Keywords: polymer composites ; real-time cure monitoring; dielectric properties ; vector network analyser (VNA).

1. Introduction

Carbon fibre-reinforced polymer (CFRP) composites are widely used for high performance, light weight structures in various industrial sectors and applications due to their superior mechanical properties (e.g. high stiffness and low weight), thermal and chemical resistance and low shrinkage properties [2]. Therefore, different manufacturing techniques for CFRP composites have been developed. However, Conventional CFRP composites manufacturing techniques (autoclaved or out-of-autoclave) can be expensive at large scales, and are time consuming (mainly due to allow thermal energy conducting through the polymer medium and the composite), and are associated with effects from uncertainties associated with the manufacturing process parameters control (e.g. temperature, pressure and resin flow) which may have a significant effect on the quality of the composite structure and thus the structure's mechanical performance.

In recent years various real-time manufacturing process monitoring technologies (e.g. embedded dielectric sensors, pressure sensors, fibre optic sensors etc.) have been developed to track the critical process parameter variations during the composite manufacturing, and to associate reliable closed-loop feedback systems with the manufacturing. However, all the techniques mentioned above are point contact type and suffer from several limitations such as possible interference with the in-process composite, not fully scalable to relatively large scale, non-flat, curved structures, and may require severe considerations in terms of practical implementations (e.g. increasing number of sensors for large scales hence, increasing the overall cost and associated electronics) [3]. Such challenges must be overcome for real-time measurement of a composite process before it can be applicable to large structures and/or for mass manufacturing.

The present study focuses on the development of a real-time contactless dielectric process (cure) monitoring (CDPM) technique for polymer composite manufacturing. The main objective of the present study is real-time measurement of the dielectric properties (DP) of composite surface, remotely, by means of a high-frequency, free-space vector network analyser (VNA) associated with two steel conical horn antenna.

Thermal imaging of the composite surface, which produces a real-time heat pattern (HP) is the sole existing measurement relying upon remote sensing with no interference with the composite structure. Developing a HP of the surface is necessary for a closed-loop process, however, a temperature increase at a specific point occurring for a relatively short period of time may not mean a high degree of cure at that location, and indeed may result from a contribution from uncertainties about the material such as an area enriched by carbon fibres. On the other hand, non-uniform heating (and cure) can be caused by the combination of material uncertainties (e.g. fibre volume fraction). Hence, process control relying solely on the HP measurement over the composite surface may not be reliable. Moreover, it has been shown that there is a strong correlation between dielectric data and the cure state (e.g. degree of cure) at a specific location in the material, as it interprets the polymerisation state in real-time [3]. Therefore, using dielectric sensing technique to identify the dielectric pattern (DP) across the surface of the composite is advantageous as it correlates with the material's characteristics under cure.

Since the change in the dielectric properties of composites during the curing is mainly contributed by the matrix material, current work focuses on the study of a pure epoxy resin, as a first step to fibre reinforced composite. Therefore, a thermoset epoxy resin was selected for this feasibility study to assess its relaxation characteristics (sensitive to the degree of cure) in real-time using a remote VNA. The assessments were carried out at 60 °C and 80 °C temperatures.

2. Experimental Methods

2.1 Materials and Manufacturing

A low viscosity thermoset epoxy resin, 'Araldite LY 5052' (Huntsman, UK), was used. The epoxy was crosslinked using 'Aradur HY 5052' (Huntsman, UK), a mixture of polyamines curing agent. The epoxy and the hardener were liquid at room temperature. Araldite LY 5052 and the hardener Aradur HY 5052 were mixed using a mixed ratio of 100:38 by weight. The epoxy-hardener system was cured at two different temperatures (60 °C and 80 °C) using a hot plate 'Stuart SD500' from Cole-Palmer, UK (Figure 1). Firstly, the hot plate was cleaned using acetone and a layer of vacuum bagging film of 50 µm thickness, 'VB200' (supplied by Easycomposites, UK), was attached to the hot plate, using flash tape, 'Flash/release tape' (supplied by Easycomposites, UK), to protect the surface of the hotplate. An 8 mm thick sealed wall was built around the edge of the hotplate using layers of 'Tacky tape' ('Vacuum Bagging Gum Sealant Tape' supplied by Easycomposites, UK) in order to make a mould for curing the epoxy system, as seen in Figure 1.

Figure 1. Hot-plate mould for curing epoxy-hardener system

The dielectric properties of approximately 718 ml (6 mm thickness layer) of epoxy-hardener system was monitored during the curing cycle using a (VNA) equipped with a steel conical antenna. The temperature variation of the epoxy during the curing cycle was observed using a FLIR T420 thermal imaging camera at every 5 min. The Schematic of the experimental set-up is illustrated in Figure 2.

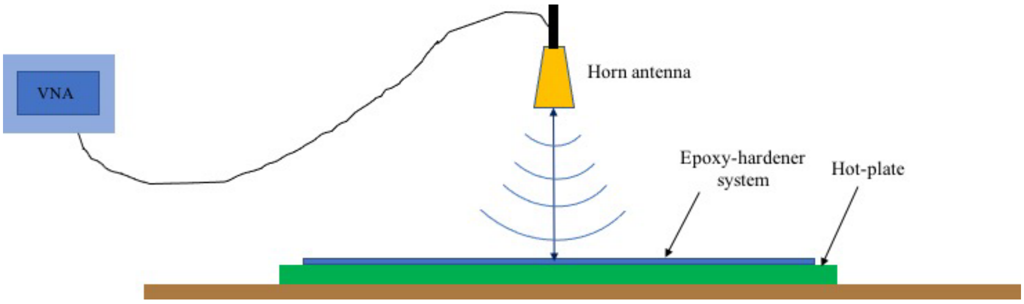

Figure 2. Schematic of the experimental set-up equipped with remote dielectric measurement

The equipment used for the dielectric measurement are VNA 37937c, Anritsu; phase stable cables, K-connector; GPIB-to-USB cable, National Instruments; Horn Antennae, Narda 640, Standard Gain; coaxial cables, SMA-to-SMA; photographic tripod; Laser rangefinder; Digital inclinometer, DXL360, 2-axis; Plexiglas flat plate of 30cm × 30cm × 6mm; Four (4) RF foam absorbers and tyrofoam block of 30cm × 30cm× 15cm .The experimental setup is show in Figure 3. Four RAM panels were positioned around the hot plate where the resin was later poured for curing. Above the hot plate, the horn antenna, mounted on a metal base and fixed onto the tripod, which regulated in pitch and roll. The hot plate was aligned with the antenna through the use of a 2-axis digital inclinometer (pitch and roll) and a laser rangefinder for the antenna to point towards the centre of the hot plate (see Figure 4). The width and height of the horn lips were 8 cm, thus producing, at 8.2 GHz (f_{min}, λ_{max}), a maximum beam width of 26.18 degree The far field at the aforementioned frequency was 0.35 m and at this downrange the beam footprint was 0.16m in both directions (azimuth and elevation). The Plexiglass block was placed at 1.176895 m downrange (1.176895, 1.188895) m, thickness 6 mm, while the resin was placed at (1.18007, 1.18607) m, thickness 6 mm. At these distances, the footprint of the beam was no greater than 54.3 cm × 54.3 cm. The size of the usable surface of the hot plate was 50 cm × 30 cm. Of this surface, an area of 46 cm × 26 cm was utilised to cure the resin. A 2 cm margin in width and length was reserved for the restraining wall (see Figure 5) as mentioned above.

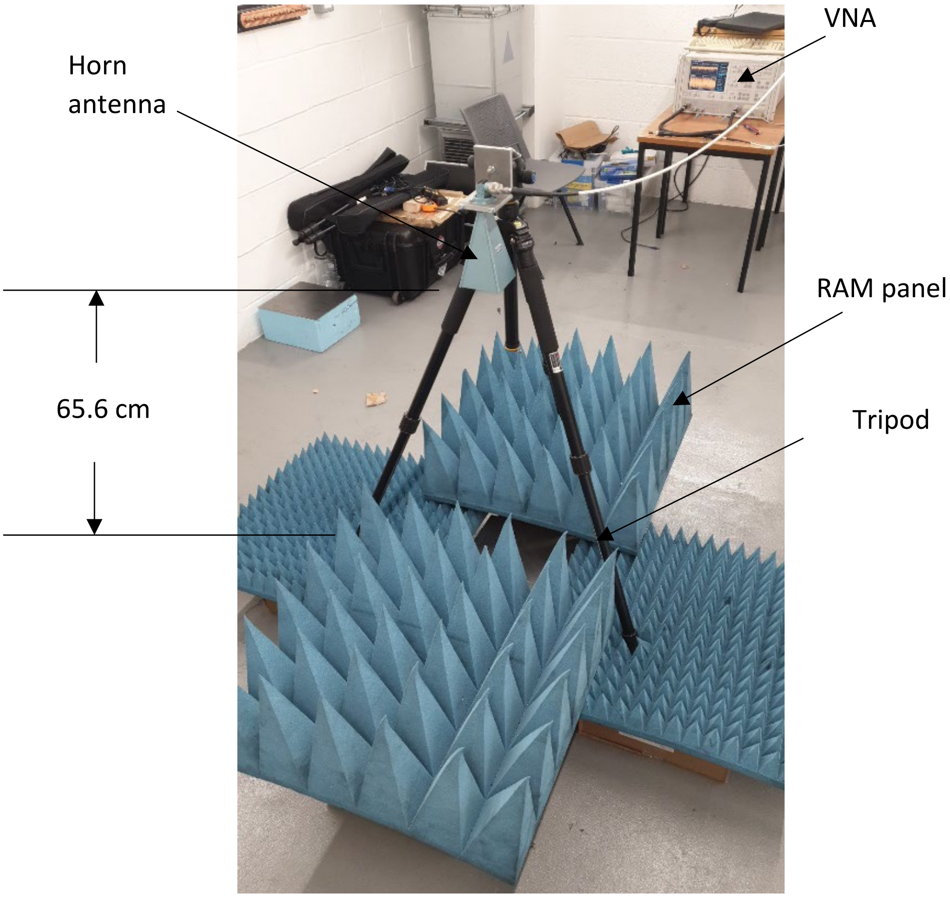

Figure 3. Dielectric Measurement set-up

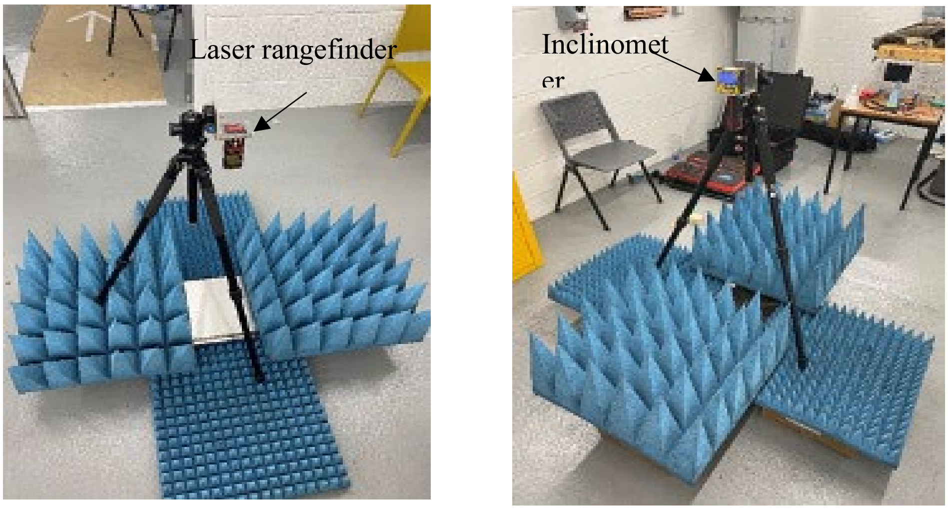

Figure 4. Hot-plate and antenna mount alignment

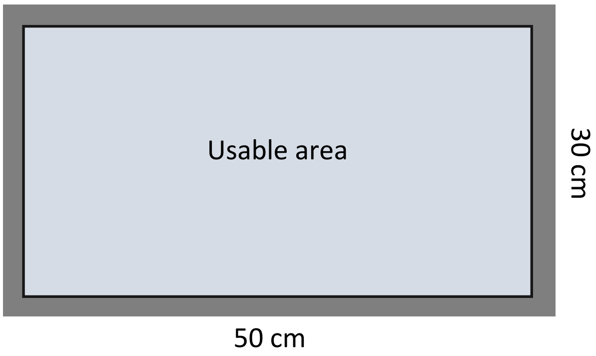

Figure 5. Experimentation area for resin curing and dielectric measurement

2.2 Data Acquisition and Processing

Two measurements were made; one on a Plexiglas 50 cm ×30 cm × 6 mm plate which was mounted on a 50 cm × 30 cm × 30 cm styrofoam block on top of the hot plate, and a second measurement of the resin over two hours in five minute intervals. Two separate backgrounds were captured for coherent background subtraction. The S_{11} channel of the VNA was captured using the GBIP cable and a laptop. The captured data were magnitude (dB) and phase (deg) and enabled the recreation of the complex signal.

3. Results

3.1 Curing of Epoxy Resin

The surface temperature of the epoxy during the curing process was observed using a thermal imaging camera. Figure 6 shows the temperature variation of the resin during the epoxy curing at 60 °C. The plot clearly shows that the temperature of the resin overshoot the mould temperature (60 °C) confirming the cure progression of the resin as the cure reaction of epoxy is exothermic.

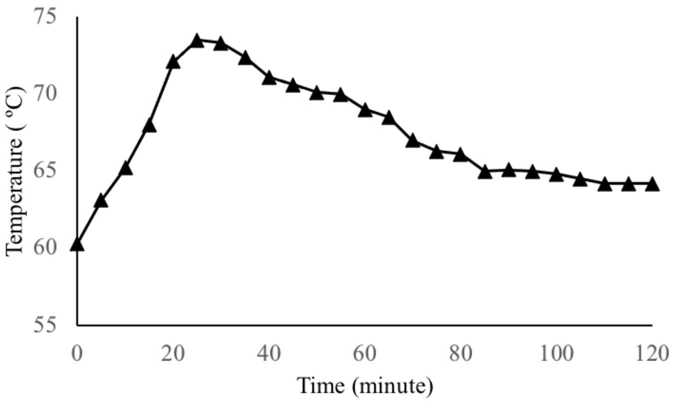

Figure 6. Temperature variation during cure of the Araldite LY 5052/Aradur HY 5052 epoxy resin

The decrease in temperature at 35 min indicates the slowdown of the exothermic reaction. The foam RAM panels used in the experiment were not heat resistant, and therefore a slight burning and melting of one of the panels were observed at the end of the epoxy curing cycle. The burnt portion of the foam absorber stuck to the tacky tap boundary on the hot plate. The epoxy was set to cure at 80 °C, however within 7 min the epoxy temperature increased dramatically to 150 °C due to exothermic reaction and smoke was also detected. Therefore, further heating of the epoxy was stopped to avoid the hazardous situation. Extreme caution must be taken as such extreme state as in the previous case.

3.2 Post Processing of Dielectric Data

A measurement was performed to check that the plexiglass background was free of unintentional interference. The signal in the frequency domain should be Gaussian white noise. In particular, when subtracting two background signal captures, only the hot plate surface and the minimum signal duration should be visible, and one or two multipath features of the room. It was found that the presence of the floor, the signal duration, the frequency dependence of clutter and not perfect subtraction cause phase linearity from 8.2 GHz to approximately 9 GHz. This was corrected by range gating.

The epoxy resin was cured for 1 hour at 60 °C and the S_{11} VNA channel was captured every 5 minutes (Figure 7, Figure 8 and Figure 9). The permittivity was calculated for every time slot.

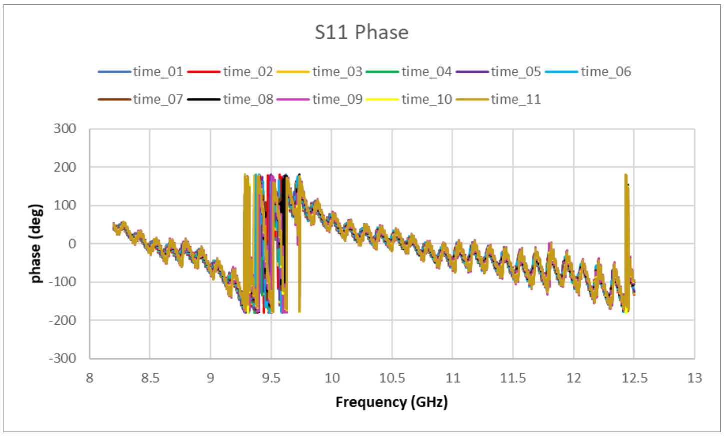

Figure 7. Resin S11 phase (background subtracted and range gated)

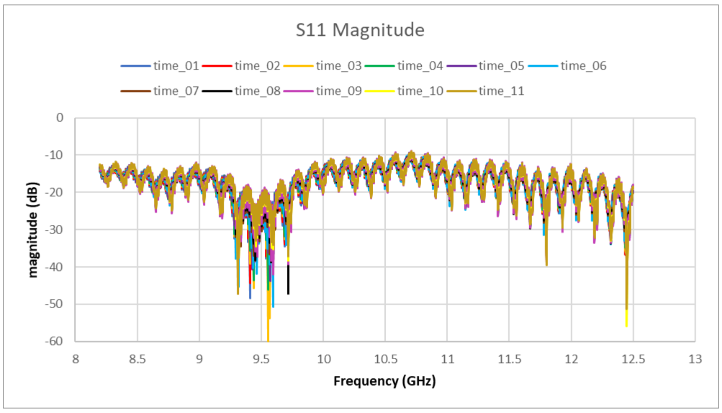

Figure 7. Resin S11 magnitude (background subtracted and range gated)

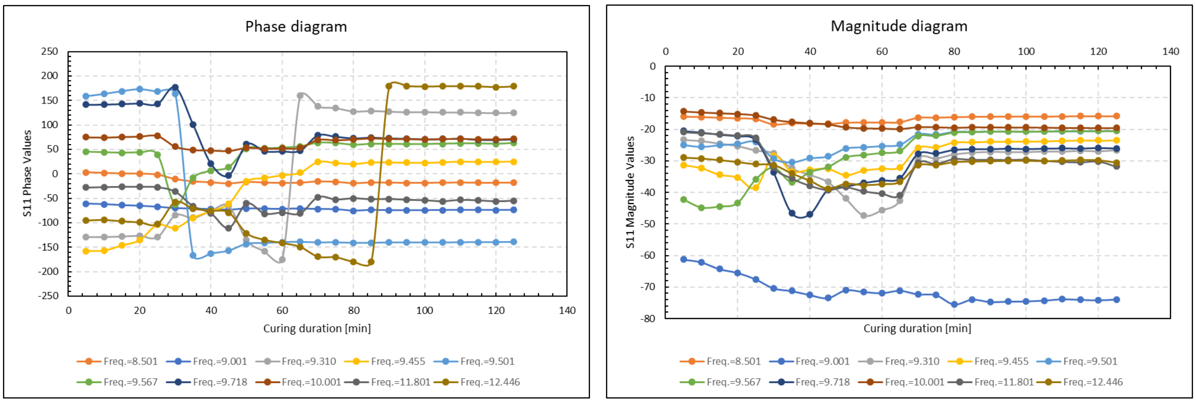

Figure 9. Resin S11 phase and magnitude vs curing time (background subtracted and range gated)

Three main regimes in the evolution of S11 phase data with time are identified (Figure 9). Regime 1: constant phase with increasing time for all frequencies for time < 25 min ; regime 2: varied phase with increasing time for all frequency for 25 min <time < 70 min; regime 3: constant phase with increasing time for all freq. for time > 70min at frequency 8.5 zero phase occurs with varied (increasing) frequency, swapping signs in the non-zero phase data, all phase values drop after time = 25 min at all frequencies except for frequency = 9.310 and 9.455. Frequency 9.310 exhibits an abrupt increase in the phase values between 25 min and 45 min, and an abrupt decrease between 45 min and 60 min. Frequency 9.455 exhibits a gradually increasing trend with time, indicates an abrupt change (drop) at time 25min (starting of regime 2), continue to rise within regime 2, and change to constant trend at time 70 min. Frequency 12.446 constant regime 3 starts later than the others' regime 3 point, post 85 min. All frequencies exhibit a short term sudden increase at the end of regime 2 at 65min, before reaching regime 3's constant values.

Three main regimes in the evolution of S11 magnitude data with time are identified (Figure 9). Regime 1: constant magnitude with increasing time for all freq. for time <25 min; regime 2: varied magnitude with increasing time for all freq. for 25 min < time < 70 min; regime 3: constant magnitude with increasing time for all frequencies for time > 70 min; all S11 magnitude data are negative across all curing time; with varied (increasing) frequency, fluctuations (rise and drop) in the magnitude data, across all times; magnitude values drop after time = 25 min at all frequencies except for frequency 9.455 and 9.567; frequency 9.455 exhibits an abrupt increase in the magnitude values between 25min and 30min, and a slight decrease between 30 min and 60 min. Frequency 9.567 exhibits a gradually increasing trend with time, indicates an abrupt change (drop) at time 30 min (approximately at the starting of regime 2), continue to rise within regime 2, and change to constant trend at time 70 min. All frequencies exhibit a short term sudden increase at the end of regime 2 at 65 min, before reaching regime 3's constant values. Frequency 9.001 is an anomaly in the magnitude trend with no indicative representation of regimes 1 and 2, however clearly shows a constant trend within regime 3 which consistent with the data from the other frequencies.

4. Conclusions

The focus of this feasibility study was to develop a real-time contactless measurement of the dielectric properties (DP) of composite surface by means of a high-frequency, free-space vector network analyser (VNA) associated with one steel conical horn antenna (monostatic method), therefore making it portable and scalable with no structural interference with the composite. A thermoset epoxy resin (Aradur HY 5052/Araldite LY 5052) was selected for this feasibility study to assess its relaxation characteristics (sensitive to the degree of cure) in real-time using a remote VNA. The assessments were carried out at 60 °C and 80 °C curing temperatures.

The methods used in the feasibility study proved to be reliable to measure the variation of electrical permittivity of thin layer of epoxy or composites over a wide bandwidth. However, further research should be performed to understand the interference of the hot plate surface with the material under test.

The feasibility study has been performed systematically to develop a real-time contactless measurement of the dielectric properties (DP) of composite surface by means of a high-frequency, free-space vector network analyser (VNA) associated with one steel conical horn antenna (mono- static method). However, through the course of this study some potential areas of further research have emerged and are addressed.

Acknowledgements

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