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Sensors on a plasticized thermoset substrate for cure monitoring of CFRP production

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ABSTRACT

Embedding sensors for in-situ monitoring of composite manufacturing guarantees high quality and cost-optimized products. For minimally invasive sensor integration, the ideal sensors should be made of identical material of the final host product. Frequently, thermoset resins are used for high-performance fibre reinforced composite in aerospace and automotive industries. A thin layer of cured thermoset resin is rigid and brittle and it cannot be reshaped. Thus, it cannot afford to be the substrate of a sensor if flexibility is required. By adding a plasticizer to the resin, a flexible substrate is produced. The substrate is tested to produce a sensor by using a hollow mask to screen-print interdigital structures. This sensor is used to measure the curing of resin during production of Carbon Fibre Reinforced Polymers (CFRP), known to be electrically conductive. Mechanical tests show a better adhesion of the developed substrate to the host structure compared with commonly used polyimide.

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1. Introduction

Since a few decades, using Carbon Fibre-Reinforced Polymer (CFRP) became more interesting in a variety of applications like automotive and aerospace. CFRP shows better mechanical and chemical properties than metals like steel or aluminum. One of the common problems in the production of CFRP is the under- or over-curing of the resin. The resin is a polymeric substance, which fills the empty volume between the fibres; bonding the fibres into a composite. The cured resin itself has a low tensile strength. When a structure made of composite undergoes a load, the resin transfers the load to the high tensile fibres. If this resin is cured improperly, the expected crosslinking does not exist in the matrix and ultimately the plies will be delaminated under operating load. Thus, it is necessary to monitor the curing of the resin during manufacturing. There are different methods for in-situ cure monitoring of the resin. The most common methods are ultrasonic transducers [1,2], optical fibre interferometers [3,4], and planar interdigital structures [5–7].

Mismatches in the coefficient of thermal expansion between the sensor and host structure can induce stress. Moreover, a sensor to

be embedded in CFRP must be up to some extent flexible to sustain the manufacturing process. In order to eliminate these two problems, the sensor ideally has to be made of either the resin or the fibres and must be flexible enough. For the first time a flexible substrate is developed of RTM6, a high-performance resin for aerospace application [8]. An interdigital structure is screen-printed on this thin and flexible substrate. This work shows how such a sensor is used for in-situ cure monitoring of pure RTM6 and CFRP during the manufacturing.

2. Measurement background

2.1. Differential scanning calorimetry (DSC)

One of the most important and common methods for thermal analysis and characterization of polymers is Differential Scanning Calorimetry (DSC). DSC is in accordance with ISO 11357 and it is based on the fact that any changes of energy during thermal treatment of liquid or solid material can be studied. DSC measures the heat flow of the sample pan with the material under test and compares it to the reference pan, which is usually empty. The material under test is tested isothermally or is heated up at a controlled rate from room temperature or below to a temperature above glass transition temperature (T_g). The DSC can be run also by cooling the

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material under test from high temperature (above T_G) to the room temperature or even below.

Using DSC technique, different information about polymeric resins can be obtained such as melting point, T_G and exothermic energy of cure. The most relevant aspects regarding polymers that can be studied by DSC are:

- Determination of thermo-physical properties of polymeric resins such as specific heat transfer during thermal treatment,
- Characterization of polymeric resins, such as identifying the cross-linking temperature, cross-linking enthalpies and glass transition temperature T_G ,
- Finding decomposition temperature.

In this paper, modulated temperature DSC (MTDSC) in accordance with ASTM E2602 – 09 (Reapproved 2015) “Standard Test Methods for the Assignment of the Glass Transition Temperature by Modulated Temperature Differential Scanning Calorimetry” was used to measure the T_G of the developed flexible RTM6 substrate and RTM6 samples isothermal cured at 160 °C for 8 h.

2.2. Dielectric analysis (DEA)

Another important method to characterize the polymeric resins is Dielectric Analysis (DEA). A fringing field interdigital structure has the same operating principle as many conventional cylindrical dielectric cells and parallel-plate sensors. In all these designs, the electric field penetrates the material under test. The capacitance and conductance between two conductors depend on the dielectric properties of the material under test and the electrode's geometry [5]. A sinusoidal voltage called excitation voltage is applied to the IDS contact pads. The output current and voltage are monitored in multiple frequencies. These output current and voltage are related to the resin's dielectric properties (dielectric constant and loss factor). The changes of dielectric properties reveal the point of minimum viscosity, gelation point and vitrification of the resin [10].

The material under test is polarized due to the oscillating voltage applied, which causes changes in the amplitude of the response current. The amplitude of the applied voltage is compared to the response current as well as the phase shift. From the changes of amplitude and phase shift, the conductance and capacitance of the material can be studied as a function of time, temperature, and frequency. These parameters are used to determine the dielectric properties. There are ions and dipoles exciting in the resin and causing the dielectric behavior of that. The ions can move and dipoles can be aligned in the electric field when the resin is liquid. Once the resin is cured, it is solidified and no more ion migration or dipole alignment occurs.

The complex impedance can be used to monitor the curing of thermoset resins or adhesives as explained by Mijovic et al. in [9]. Considering the equivalent circuit of an interdigital structure [10] given in Fig. 1, the measured impedance from the sensors contact pads is expressed by Eq. (1)

$$\frac{1}{Z} = G + i\omega C \quad (1)$$

where Z is the complex impedance, G is conductance, i is the square root of -1 ($i = \sqrt{-1}$), ω is the angular frequency and C is capacitance. $V=0$ shows the equipotential surface between each two adjacent electrodes. $R_{I,MUT}$ stands for resistance of material under test between two interior electrodes while $R_{E,MUT}$ refers to the exterior electrode. $C_{I,MUT}$ refers to capacitance of material under test measured by interior electrodes, while index E refers to exterior electrode. Similarly, $R_{I,SUB}$ and $C_{I,SUB}$ are resistance and capacitance of substrate between interior electrodes, while index E refers to

these values for exterior electrodes. Further mathematical interpretation of these terms is given in [10].

The dielectric permittivity of the material under test is given by Eq. (2)

$$\varepsilon = \frac{Im\left(\frac{1}{Z}\right)}{\omega K} \quad (2)$$

where ε is the dielectric permittivity of the material under test, ω is the angular frequency and K is the constant value coming from the sensor geometry and also called cell constant. Generally, by monitoring the changes of ε , the alignments of the dipoles in the resin can be determined.

The ion conductivity σ is mainly correlated to the charge migration and is given by Eq. (3)

$$\sigma = \frac{Re\left(\frac{1}{Z}\right)}{K} \quad (3)$$

where σ the conductivity is proportional to the real part of the impedance and reciprocal value of K , cell constant. The dielectric properties are calculated from the loss-factor. The measure of absorbed and lost energy for moving ions depends on ion mobility, which is known as ionic conductivity [11,12].

Knowing the dielectric loss-factor value, the curing degree can be calculated by Eq. (4) as shown in [11]

$$\alpha(t) = \frac{\log(\varepsilon''_0) - \log(\varepsilon''_t)}{\log(\varepsilon''_0) - \log(\varepsilon''_\infty)} \quad (4)$$

Where $\alpha(t)$ is the curing degree at time t , ε''_∞ is dielectric loss-factor at infinity, ε''_t is dielectric loss-factor at time t and ε''_0 is dielectric loss-factor at the point of minimum viscosity. The dielectric loss factor is a measure of energy loss in a dielectric material and it is complex relative permittivity of dielectric material under test. In our case, the dielectric material is the thermoset resin, which is used in production of CFRPs to keep fibers in place and protect them in different thermal, chemical and mechanical circumstances. In existence of an alternative electric field, there is dipole alignment and ion migration in resin. These both cause loss of energy in the material that can be interpreted by measuring loss factor. Now, going back to Eq. (4), we have to derive one parameter which breaks down the complex function. Following Han [12] we use the impedance Z for that purpose. The curing degree of the resin is given in Eq. (5)

$$\alpha(t) = \frac{\log(Z_t) - \log(Z_0)}{\log(Z_\infty) - \log(Z_0)} \quad (5)$$

Where Z_0 is the impedance modulus at the point of minimum viscosity, Z_t is the impedance modulus at time t and Z_∞ is the impedance modulus at infinity.

3. Experimental

3.1. Plasticized RTM6 with dibutyl sebacate

RTM6 is a thermoset resin with an irreversible chemical curing reaction. The resin forms a three-dimensional rigid polymeric cross-linked network, which cannot be reshaped once the resin cured. A thin layer of cured RTM6 is brittle, therefore it cannot be handled nor can the interdigital structure be fabricated on it. By adding a plasticizer, a thin and flexible substrate of RTM6 can be produced.

Dibutyl Sebacate from TH. Geyer is a colourless and odourless liquid at room temperature, which is used as a plasticizer in cosmetics and pharmaceutical products. To find the proper mixing ratio of the resin and plasticizer a try-and-error procedure is performed. Table 1 gives some results of this procedure.

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