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Design, fabrication and embedding of microscale interdigital sensors for real-time cure monitoring during composite manufacturing

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ABSTRACT

In this study, microscale interdigital capacitive sensors are designed, fabricated and embedded in glass fiber composite for real-time cure monitoring of resin. The microscale interdigital capacitive sensor offers great advantages due to its miniaturized size, high flexibility and high temperature stability. Most importantly, after integration, due to the small footprint and only 5 μm thickness, the embedded sensor minimally downgrades the composite mechanical properties. Apart from all these merits, the substrate foil is perforated to provide an opportunity for the resin to go through the substrate and bridge it. The sensor consists of two interdigitated arrays. There are two different sensor designs, which offer 900 and 450 electrodes in arrays. The electrodes are made out of tantalum and are fully insulated with about 50 nm tantalum oxide. Therefore, it is possible to embed the sensor even in conductive fibers, e.g. carbon, and to use the sensor out of the Cleanroom without getting contaminated with conductive particles to shortcut the arrays. Polyimide is chosen for the substrate of the sensor. This specific polymer has excellent flexibility and its geometry stays unchanged even at high temperature. The Dielectric Analysis (DEA) measurement proves the effective and real-time tracking of resin polymerization during laminate production.

1. Introduction

In the past few decades, the use of Fiber Reinforced Polymer (FRP) has been increased in demanding industries like aerospace, aircraft, civil engineering, automotive and wind energy. This is due to excellent stiffness and strength of composite and their light weight compared to metals. The quality of the final product is directly influenced by the manufacturing process which is based in some cases on an **infusion process** (filling of dry textiles with resin) and **curing of the resin** during manufacturing. Mainly, the quality of a thick and complex shape composite is affected by the existence of dry spots, porosity, voids and micro- voids as well as under-curing of the resin. By monitoring the manufacturing process, the cost- effectiveness and optimum properties will be achieved.

To ensure complete infusion process which leads to the fully impregnation of the fibers in a preform different methods can be used; **Non-embedded** sensing uses cameras (for transparent mold) [1] or ultrasound reflection [2]. **Embedded** sensing comprises pressure sensors [3], optical fiber Refractometers [4], thermocouples [5] or an optical fiber spectrometer [6]. Tracking the flow of the resin and the impregnation of the fibers is necessary to achieve a uniform product without dry or non-saturated parts.

After fully impregnation of the fibers, it is crucial to monitor the complete curing of the resin to guarantee a high quality product, which can afford the expected mechanical properties. To measure the degree of resin cure various techniques could be utilized such as: **Electromagnetic properties** (Dielectric Analysis (DEA)) [7], Direct Current (DC) analysis [8], Electrical time domain Reflectometry [9]), **Mechanical properties** (Optical fiber interferometers [10], Ultrasonic transducers [11]), **Optical properties** (Optical fiber Refractometers [4], Spectrometers [6]). Among these three approaches for cure monitoring, DEA shows the biggest potential in online cure-monitoring. This is while in ultrasonic transducers and optical fiber methods have some disadvantages. The former uses a sensor and a source for detecting the state of cure in a manufacturing mold; there is a big influence of the fiber volume content of the ultra-sound signal. The latter is very expen-

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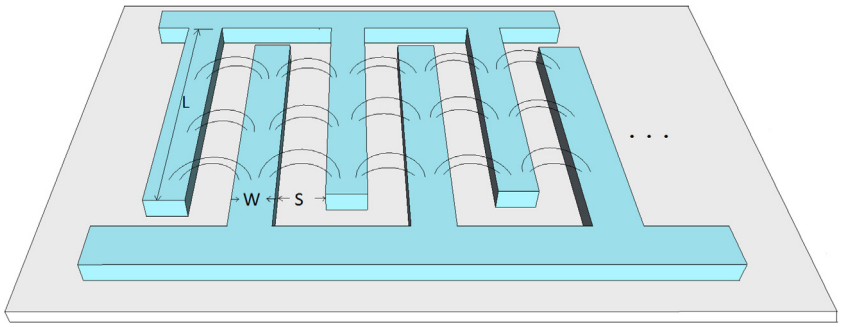

Fig. 1. An interdigitated array and the electric field between electrodes. S is the spacing, W is the width of electrodes and L is the mutual length of each two neighbor electrodes.

sive and highly time-consuming preparation. The fiber optic can be broken due to mechanical treatment. Some advantages of DEA are one-sided measurement, high sensitivity, the availability of cheap disposable sensors or re-usable sensors, with the possibility to integrate a thermocouple inside the sensors. The major disadvantage of interdigital sensors is getting a short cut by embedding in conductive fabrics, e.g. carbon. This problem is solved in our novel approach be having thin metal oxide insulation layer, which expands the field of applications for dielectric analysis.

For thermoset resins, thermal analysis is important. There are three different phases of formulation (which type of resin, hardener and modifier), processing (heat control and cure monitoring) and structural health monitoring of the product (structures and properties). The **processing** of the composite consists of homogeneity, viscosity, flow front, reactivity and degree of cure. With the exception of homogeneity that can be guaranteed by good mixing, all other properties can be monitored and proven by DEA.

This study presents design, fabrication and embedding of an interdigitated sensor array into the glass fiber laminate. The sensor provides a platform to measure the ion viscosity of resin based on DEA analysis and monitor the degree of resin cure.

When the curing of the resin is finished; the embedded sensor cannot be taken out from the composite anymore. The used reference sensor for DEA measurements is a commercial one by Netzsch-Gerätebau. It is a printed pattern on a PCB (Poly Carbonate or 150 μm thick polyimide film, 115 μm lines-spacing of electrodes), which has a sensing area of 200 mm^2 and a thickness of (≥) 150 μm. Therefore, they can only get used in laboratory experiments. This big embedded sensor will degrade the mechanical properties of the laminate part drastically and behaves like a wound in a uniform laminate. If they stay in a part of composite, which is going to be used under static or dynamic loads, delamination is unavoidable. There are also types of commercial detachable interdigital sensors (Netzsch-Gerätebau GmbH) that stay in the mold or on the surface of the laminate. These sensors can be used over and over, but they just provide localized information about the state of resin at the surface they attached and not about the internal state of the laminate. This caused a problem when the laminate is thick (e.g. blades of wind turbines) and there is no information about the state of resin far above the sensor.

We designed and fabricated microscaled sensors which are thin, flexible and having small porous sensing area. The sensor cannot be made infinitely small, since its sensitivity depends on its size (number and length of electrodes).

Our microscale sensor is fabricated on a flexible and porous foil with a thickness of 5 μm and a sensing area of 18 mm^2. The thickness of the sensor is comparable to the diameter of glass/carbon filaments. In [12–15] the effect of the embedded micro sensors on the mechanical properties of the fiber laminate composite is investigated, showing that the impact of embedding a **thin**, **flexible** and **porous** inlay on the composite properties can be neglected in comparison to an embedded rigid inlay.

There is a high demand for real-time sensor system that can recognize the state of resin cure at different points in the composite without downgrading properties of the finished product. Our new microscale interdigital capacitive sensors determine the cure monitoring of the resin based on the most common and reliable method of DEA. Likewise, the porosity of a thin substrate lets the resin go through it and reinforce the sensor in a laminate. These are promising points that our microscale sensors can be used in a real composite production to ensure the complete curing of the resin and remain in the laminate without degrading its mechanical properties.

1.1. Background of dielectric cure monitoring

Dielectric cure monitoring of the resin in fiber composite materials is a common way to ensure the completeness of resin curing and the product quality. Planar interdigitated sensors for DEA analysis have been widely used to achieve this goal [7,17–21]. Dielectric analysis is in accordance with a thermoanalytical method standard of ASTM 2038. An AC excitation generates an electric field between interdigitated electrodes (Fig. 1). As a result of this field, dipole polarization and ion migration occur in the material. The ion migration can only be measured by DEA. Polarization of the dipole and ion migration due to the external field frequency (ω) causes energy loss (resistive) and energy storage (capacitive) behavior [22].

In DEA technique, the loss factor (resistive behavior) of the fiber composite material is measured, which is in correlation to the degree of cure of the resin (Fig. 2).

In this paper, we study the solidification (curing) behavior of a thermoset resin by means of real-time measurement of the ion viscosity. The two main categories of resins are thermosets and thermoplastics. The former form a three-dimensional cross-link networks after polymerization; the latter form linear polymers with weak chemical bonds. The difference between these two types of the resin and their properties and applications are discussed in [19]. Thermosets are materials with an irreversible cure reaction. After curing, they cannot be melted or reshaped. Single monomers connect to form a network and form a final polymer, which is a long molecule. The ion viscosity of the resin demonstrates the state of cure and formation of networks. DEA cure monitoring, shows thermoset viscosity, rigidity and a curing degree of the resin. For all thermoset resins, the polymerization starts by reduction of a viscosity. After the point of minimum viscosity (the slope is zero) the ion viscosity increases steeply. This part is due to cross-linking of the resin. When the ion viscosity reaches the maximum value and stays constant the polymerization is finished and the resin is fully cured (Fig. 3). This maximum value of the ion viscosity depends on the type of thermoset resin [17,19]. The overall trend of the changes