Challenges of complex monitoring of the curing parameters in coupons for LRI manufacturing

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Abstract: *In the aerospace industry, Liquid Resin Infusion (LRI) is gaining more and more importance as an out-of-autoclave alternative manufacturing technique to traditional pre-impregnated (prepreg) fabrics. The research in this field has been focused on understanding the cure and the process parameters of these materials, aiming to optimize the manufacturing process and reduce costs. A major problem derived from these technologies is the distortions induced by LRI process, affecting to composite parts due to non-uniform distribution of residual stresses. Such distortions can lead to non-uniform parts with shape distortions, which is a critical issue when trying to assembly with other parts due to mismatches in shape, leading to the rejection of such components. In this context, ELADINE project aims to understand and quantify the key manufacturing parameters that cause shape distortions on composite coupons (such as spring-in of curved parts) using an integrated numerical-experimental approach. The manufacturing process will be accurately monitored through Fiber Optic Sensors (FOS) and Dielectric sensors (DC) to understand how the process variables affect the distortion phenomena. The monitored data will feed a simulation tool for spring-in prediction for large integral composite wing structures. This article covers the preliminary results of cure monitoring and process parameters of thermoset composites implementing monitoring strategies for manufactured coupons by LRI.*

Key Words: *cure monitoring, shape distortions, liquid resin infusion, FBG sensors, dielectric sensors, spring-in*

1. INTRODUCTION

In recent decades, aerospace material industry has changed drastically shifting from metal-based parts to carbon fiber composite structures. Autoclave pre-impregnated "prepreg" material has been the predominant materials for structural composite components. However, this equipment presents certain limitations such as the high investment, high maintenance and operational cost, especially for large parts in which not only size is a limitation but also autoclave curing of these parts can present a bottleneck in production. Traditionally used carbon fiber prepreg fabric has proven to have outstanding mechanical properties for structural components. Still the short out-life and shelf-life, the necessity of refrigerated storage and its high price are some of the drawbacks of these kind of materials [1], [2]. For these reasons, Out-of-Autoclave liquid resin infusion methods has been gaining interest as a cost-effective process technology. Lower operational cost derived from less waste of resin and lower energy consumption, as the thermoset cure takes place in an oven, make liquid resin infusion methods attractive for the industry. Nevertheless, it also has limitations comparing to prepreg, such as lower fiber content or non-uniform resin distribution on the part as a consequence of dry spots or resin rich areas. Shape distortions are a common problem in the manufacturing of composite parts. Residual stresses produced during manufacturing results in parts with different shape than the tool (warpage of flat parts and spring-in of curve parts). These differences may result in parts that do not meet the tight tolerances required in the assembly of structures leading to scrap parts. Thus, understanding the origin of these residual stresses is key to optimize the manufacturing parameters and the quality of the final part. The main mechanisms causing shape distortions are: thermal anisotropy, resin polymerization shrinkage, tool-part interaction, resin flow and compaction and temperature gradients [3], [4]. ELADINE (Evaluation of LAminate composite Distortion by an Integrated Numerical-Experimental approach) project aims to study the phenomena causing shape distortions in composite parts manufactured by liquid resin infusion. A numerical-experimental approach is used for this purpose, in which monitoring process parameters will feed a numerical simulation tool designed to predict shape distortions. ELADINE focuses on two parts contained in the wing box structure of a plane.

2. PROCESS MONITORING IN LRI MANUFACTURING OF CARBON FIBER COMPOSITES

Fiber optic sensors (FOS) and dielectric sensors (DCS) are capable of measuring manufacturing process parameters such as temperature, resin flow, cure degree and strain. In ELADINE project, FBGs (Fiber Bragg Grating sensor, strain: SFBG and temperature: TFBG, (see Table 1) are integrated in the composite coupons together with DC sensors (temperature and resistivity to ion mobility) to monitor and understand the mechanisms of thermal anisotropy, resin polymerization shrinkage, tool-part interaction, resin flow and compaction and temperature gradients.

These mechanisms are influenced by the composite manufacturing process and directly affect the spring-in phenomena.

Table 1 - Process parameters monitored by the sensors used in ELADINE project

Sensor	Parameters	Abbrev.	Measurements
Optic fibre sensors (FOS)	Strain	SFBG	Indirect measurement (strain-T decoupling)
	Temperature	TFBG	measurement
Dielectric fibre sensors (DCS)	Resin Flow Temperature	DFS	Indirect measurements
	Resistance Temperature Cure degree (α) Tg	DCS	Resistance and T: direct measurement α, Tg: to be calibrated for each system

- **Fiber Bragg Grating sensors (FBGs)**

The great characteristics of fiber optic sensors (FOS) such as small size, lightweight, and robustness to withstand harsh environments, make them a great component to integrated in composite materials. In terms of structural health monitoring, Fiber Bragg Gratings (FBG) stands out due to the fact that is a mature, cost-effective FOS technology tested in a wide range of monitoring applications [5], [6]. In the field of composite materials, FBG sensors offer significant potential to measure residual strains within composite materials [7] and for real-time SHM (Structural Health Monitoring) monitoring [8]. Likewise, they have been used to monitor the spring-in phenomenon in composite parts [8], [9], (Figure 1).

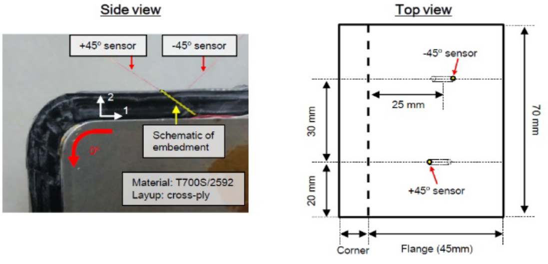

Figure 1. Picture (left) and scheme (right) of the L-shaped specimen [8]

A FBG sensor is a reflector, obtained through periodical morphological change in the optical fiber's core (<5mm). It reflects a portion of the incoming light at a specific wavelength, called Bragg wavelength, leaving the rest of the incoming light pass without altering its properties, as shown in Figure 2. The Bragg wavelength (λ_B) is defined as the center wavelength of the reflected narrow-band optical signal, being a function of the effective refractive index (n_{eff}) and of the grating period (Λ):

$$\lambda_B = 2\, n_{eff} \Lambda \tag{1}$$

As can be seen by equation (1), a shift of the Bragg wavelength can be observed when either the effective refractive index or the grating period change. As a result, whenever the fiber is affected by external changes (such as temperature, strain, deformation, vibration etc.), the Bragg wavelength will also change. Consequently, by monitoring the Bragg wavelength variations, several parameters can be monitored. Therefore, strain and temperature variations will modify the properties of the grating, leading to a wavelength shift. Two different FBG sensors must be used to measure each parameter, one of them with different thermal sensitivity, in order to unambiguously differentiate each parameter's influence. Encapsulation of the FBG in a steel or silica capillary tube is used for the temperature sensor to ensure different thermal sensitivity of one of the FBG sensors, [10].

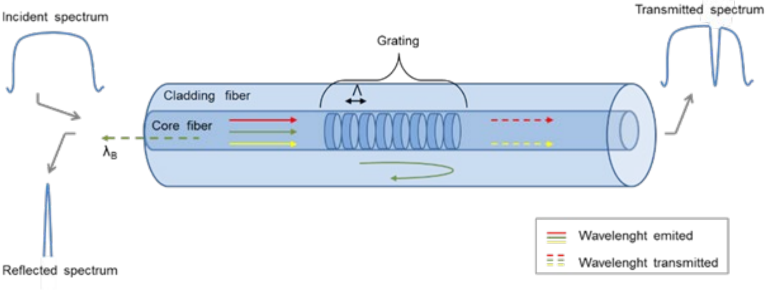

Figure 2. Working principle of Fiber Bragg Gratings

- **DC sensors**

The principle of dielectric analysis is based on the electrical properties of thermoset polymers. Before curing, the resin is mainly formed by monomers, which contains dipoles that in presence of an electric field are oriented in the same direction as this electric field.
Thus, the resistivity of a resin is related to the mobility of its ions. DC sensors measures cure evolution through the ionic viscosity of the resin.

During a resin curing process (Figure 3), two phenomena take place: polymerization and cross-linking.

At the beginning of the curing reaction, the resin is in liquid state, the viscosity is minimum and, as its monomers can move freely, the electrical resistance is also at its lowest point. As polymerization occurs, the monomers start forming long branched chains.

The movement of monomers is hindered and the electrical resistance increases. At the gel point, the polymeric chains are bonded in a complex network and from this point, the reaction is controlled by crosslinking mechanisms.

At this point, the polymeric chains movements are very limited, which is registered by the DC sensor as a drastic change in electrical resistance [11], [12].

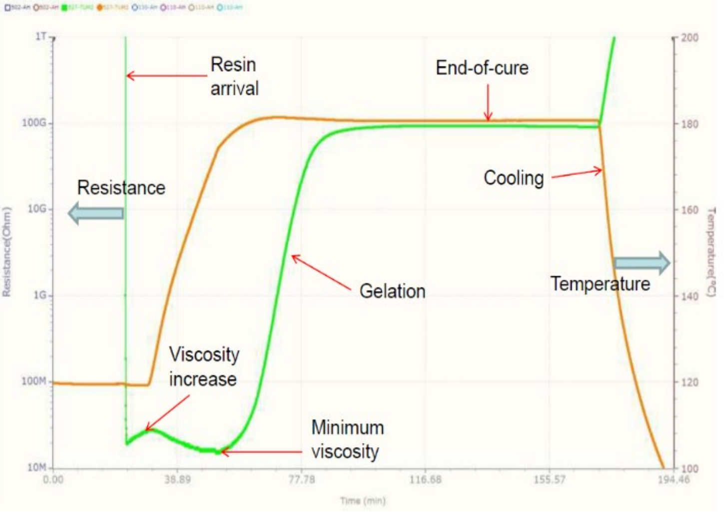

Figure 3. Real time monitoring of RTM6 resin by DC disposable sensor using Synthesites OptiMold. Different events occurring during the curing of the resin can be identified as shown. (y axis: orange (temperature), green (resistance) and x axis: time)

Film-based disposable sensors can be used for cure monitoring of manufactured parts by vacuum infusion as they are capable of detect resin arrival, minimum viscosity, gelation and end-of-cure (as shown above in Figure 3). Synthesites technology has been used for this purpose.

The sensor consists in two parallel flat electrodes covered by polyamide films with a total size of 22x12x1.2 mm and 0.6 mm thickness.

The monitoring unit (OptiMold) allows in-situ and real-time monitoring of electrical resistance (for values up to 100 TOhm).

Advantages of these sensors are the flexibility of measuring in any position of the composite part and in harsh conditions (high temperatures needed for resin curing) and their capability of measuring in carbon fiber reinforced materials without any extra protection.

3. MONITORING STRATEGIES FOR PARTS MANUFACTURED BY LIQUID RESIN INFUSION: ELADINE PROJECT CASE STUDY

Small scale composite coupons (200×300 mm) with a constant radius of curvature and different thicknesses were manufactured by LRI. A sensor scheme distribution has been designed to incorporate a set of FBG and DC sensors within the coupon that is able to monitor the processing parameters described in Table 1. This information is being compile and analyzed to further feed the simulation tool for shape distortion insights.

- **Materials and methods**

Carbon fiber preforms manufactured with HiTape dry fiber (Hexcel) by AFP were used as reinforcement material. Hexflow RTM6 (Hexcel) epoxy resin mixed and degassed according to manufacturer indications was used as polymer matrix. The parts were manufactured using vacuum assisted resin transfer moulding (VARTM), the curing cycle used was provided by the Topic Manager of ELADINE project.

The main objective of this report is to explain the fundamentals of process monitoring of composite parts manufactured by OoA LRI. For such purpose, an example case is used and the results from a few sensors are explained. Near the inlet region, two FBG sensors (one for temperature monitoring and another one registering strain) and a DC sensor were place on top of the coupon (on the surface of the coupon in contact with the vacuum bag). At the end of the part, near the vacuum port region, the same type and number of sensors were used but placed below the coupon, so they are in contact with the tool surface. This sensor configuration was set up before the injection of the resin. The vacuum bag was sealed and a vacuum test was performed before infusion.

- **Results**

Figure 4 shows the results obtained from temperature and electrical resistance monitoring with FBG and DC sensors. The full cycle is shown on graph A, in which the left axis corresponds to temperature in ºC values while the right axis refers to the resistance values in ohms. Three main phases can be distinguished on the curing cycle: injection, dwelling and cooling. During injection (Figure 4, graph B) resin arrival is detected by DC sensors as a drop in resistivity, because at that moment the viscosity of the resin is minimum and, therefore, the electrical resistivity reaches its minimum value.

In case of FBG sensors, resin arrival is seen as a decrease in temperature since the tool (and thus the sensor making contact with it) is at a different temperature at which the resin in injected. At the dwelling phase (Figure 4, graph C) both DC and FBG sensors detect the exothermic effect of the resin cure reaction. There are temperature differences between sensors placed above and below the coupon.

Regarding resistance, as temperature increased, the resistance increased rapidly until it reaches a point in which the growing trend is less pronounced. This means that the reaction slows down, and the gel point is detected. After that the resistance stabilizes around a value, which indicates that the part is close to full cure. From this point the reaction would be controlled by diffusion mechanisms.

During the cooling phase (Figure 4, graph D) the temperature decreased on both FBG and DC sensors following similar cooling rates. Conversely, the resistance increases at the same pace until the sensor saturates. This increment in resistance would be explained because there is a complex cross-linked network formed that hinders the mobility of the polymeric chains and, thus, the conductivity.

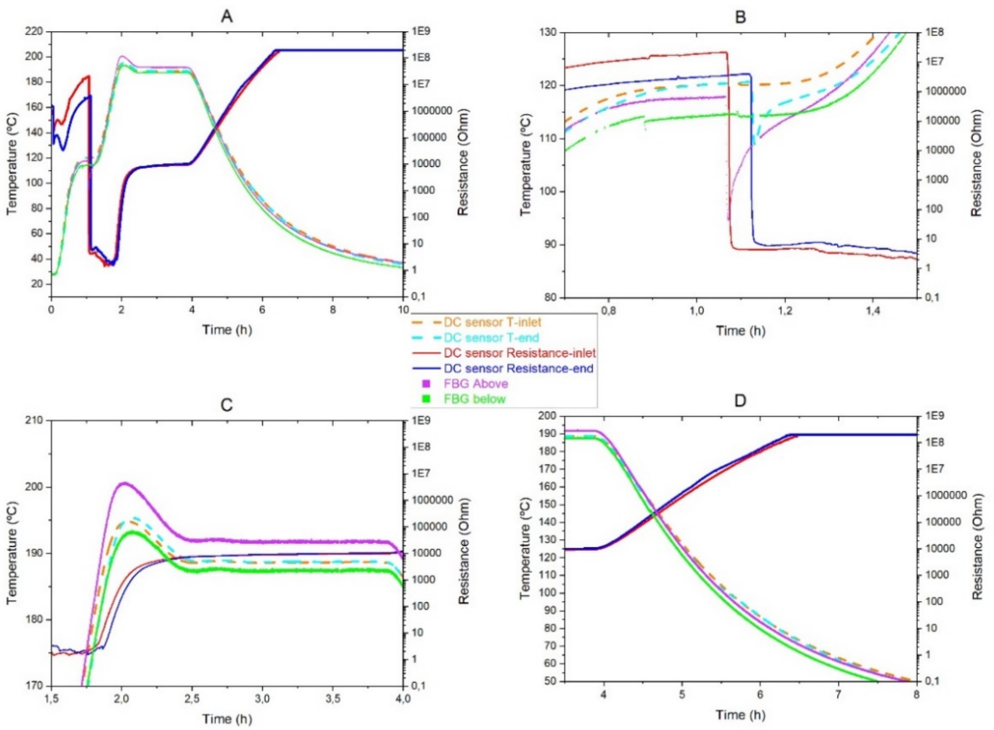

Figure 4. Temperature monitoring (green and violet lines: dashed: FBG; solid: DC) and Cure monitoring (red and blue solid lines, DC sensor). A: full manufacturing cycle, B: injection phase, C: dwelling phase, D: cooling phase

Figure shows the result from strain variations monitoring. Largest strain variations are observed during the cooling phase of the cycle, negative values of strain mean that part is under compression. Moreover, the sensor placed below the coupon registered lower values of strain variation implying that the compression on that area is higher than the one registered by the sensor in contact with the vacuum bag.

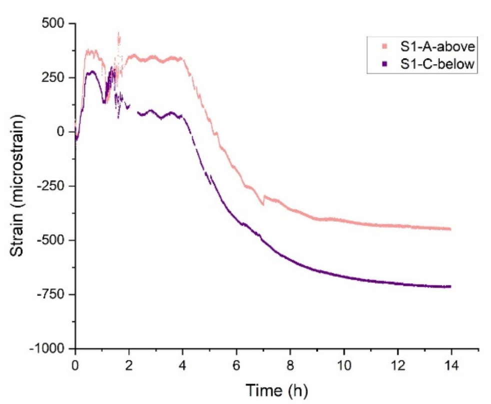

Figure 5. Strain variation monitoring with FBG sensors placed on top of the coupon (pink line) and under the coupon (purple line)

Although the results presented are only from a few sensors, a great amount of information can be extracted from the curing cycle. With an evenly distributed sensor scheme the whole coupon geometry could be monitored in order to study differences between surfaces of the coupon. Figure 3 and Figure 4 shows temperature and strain maps of the surface of the coupon at the beginning of the dwelling phase. These results were obtained with data measurements from 30 sensors (15 FBG for temperature monitoring and 15 for strain monitoring). These results could be used to obtain the residual stresses on the part after manufacturing and served as an explanation for shape distortions phenomena such as spring-in.

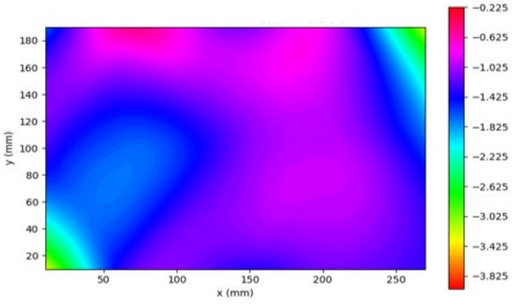

Figure 6. FBG strain monitoring, strain distribution on the surface of the coupon at the beginning of the dwelling phase

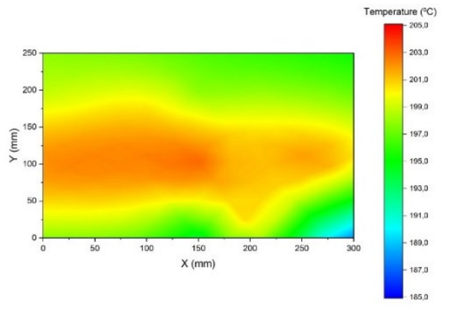

Figure 7. FBG temperature monitoring, temperature distribution on the surface of the coupon at the beginning of the dwelling phase

4. CONCLUSIONS

Monitoring of process parameters during LRI has been presented in this paper as a strategy to predict shape distortions on composite parts during manufacturing. Two type of sensors have been used: FBG sensors for monitoring temperature and strain, and DC sensors for indirect measurement of cure evolution. The manufacturing of carbon fiber composite parts has been monitored. The results obtained show that FBG sensors are capable of effectively monitor strain and temperature during manufacturing and post-manufacturing. Events such as resin arrival on different zones of the coupon, temperature variations on the coupon, tool and oven zones are detected during manufacturing. Strain variations are detected on different parts of the coupons. DC sensors are also capable of detecting different events of the curing cycle (resin injection, resin arrival and resin cure and T) through measurements of electrical properties of the resin. Therefore, process monitoring of curing parameters is a useful tool to understand the mechanisms originating the post-manufacturing deformations of composite parts.

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DISCLAIMER

The results, opinions, conclusions, etc. presented in this work are those of the author(s) only and do not necessarily represent the position of the JU; the JU is not responsible for any use made of the information contained herein.

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