Monitoring the Liquid Resin Infusion (LRI) manufacturing process under industrial environment using distributed sensors

P.WANG¹, J.MOLIMARD¹, S.DRAPIER¹, A.VAUTRIN¹, P.HENRAT² ¹ Mechanics and Materials Processing Dep., Structures and Materials Science Division and Laboratory for Tribology and Systems Dynamics, UMR CNRS 5513 École des Mines de Saint Etienne 42023 Saint-Étienne Cedex 02, France ² Hexcel Corporation SAS, 38630 Les Avenières, France wang@emse.fr, molimard@emse.fr, drapier@emse.fr, vautrin@emse.fr, Patrick.HENRAT@hexcel.com

SUMMARY

In the present paper, a novel direct approach to detect the resin flow front during the LRI process under industrial environment is proposed. To detect the resin flow more accurately and verify the results, which are deduced from indirect micro-thermocouples measurements, optical fibre sensors based on Fresnel reflection are utilized.

Keywords: Resin flow front; LRI; Micro-thermocouple; Optical fibre sensor

INTRODUCTION

Weight saving is still a key issue for aerospace industry. For instance 50% in weight of the B787 and A350 aircraft structures is made of Carbon Fibre Reinforced Polymers (CFRP). To reach a weight ratio of 60% of high performance composites in the forthcoming aircraft programmes, it is necessary to make lighter thick and complex parts. Direct processes called Liquid Composite Molding (LCM), such as Resin Transfer Moulding (RTM) or Resin Infusion Process (LRI, RFI), where liquid resin infuses fibrous preforms, are developed as interesting alternative processes to prepregs. At the present time, around 5 to 10% of the parts are manufactured by direct processes and the current trend is clearly to go ahead. RTM process consists in injecting resin into fibrous preforms placed between rigid moulds. Therefore, dimensional tolerances and porosity fraction can be kept under control and high quality parts produced. The industrialisation of this process is on going but it still needs improved models. On the contrary, the resin infusion process can be utilized in flexible conditions, such as in low cost open moulds with vacuum bags in nylon or silicone. This type of process only requires low resin pressure and the tooling is less expensive than RTM rigid moulds. Therefore LRI and RFI processes are particularity suitable for small and medium size companies because the investments are rather low compared to other manufacturing process. Figure 1 gives a schematic view of a typical LRI set-up.



Fig.1. Principle of LRI process.

In LRI-like processes, resin infusion is performed through a highly permeable draining fabric placed on the top of the fibres preform. A differential pressure is created by a vacuum at the vent of the system, it leads to the impregnation of the compressible perform in the transverse direction. The LRI process leads to final part quality improvement since resin flow and cure are distinct. However, the thickness and the fibre volume fraction of the final piece are not completely controlled because of the use of a vacuum bag instead of a rigid mould and of the large variations of the perform volume when vacuum and pressure are applied. To optimize the design and the manufacturing parameters and monitor the LRI process, a new numerical isothermal model has been recently developed [1-4]. It is postulated that the resin firstly fills in a draining fabric with high permeability, and secondly infuses the preform gradually through the transverse direction. To validate the model and to improve the knowledge of the LRI manufacturing process, we want to monitor accurately the important data along LRI process, for example, the resin flow. Experimental techniques dealing with flow front characterization can mainly be found in the literature, such as the determination of the fluid flow front advancement in the RTM process [6-8], in vacuum infusion process [9,10] and in resin infusion process [11-13].

To overcome the drawbacks of existing techniques used in our industrial conditions. In the present paper, during the whole LRI process, two types of sensors have been applied to monitoring the resin flow position, the filling time, the curing time, and the preform's temperature, which are key parameters controlling the part quality in the our infusion case carried out by LRI process.

2. NUMERICAL MODEL OF THE RESIN INFUSION PROCESSES

Recently, a complete model for the study of a reactive fluid flow through highly compressible porous media such as fibrous preforms has been proposed by Celle et al. [1-3]. This approach opens the way to the development of refine numerical simulations of real infusion process, accounting for large variations of the preform thickness during the process. The model has shown its ability to deal with the non-linear deformations of fibrous preforms during the compaction step. The infusion stage has to be assessed to completely validate this numerical tool.

In such infusion-based process, liquid resin is supposed to flow across the compressed preform thickness, as a result of the difference between the permeability of the flow enhancement fabric and the perform, usually of the order of 10^{10} . From the modeling point of view, problems of this multi-physical analysis are two fold. Firstly, one faces ill-posed boundary conditions regarding the coupling of liquid regions, where a Stokes flow prevails, with the fibrous preform regions modeled as porous media governed by

the Darcy's law and a non-linear mechanical response. Secondly, the interaction phenomena due to the resin flow in the highly compressible preform are not classical.

Using this numerical model, the simulations can bring a series of important process parameters values, like the filling time, resin mass, preform thickness etc. Example of such simulation can be found in [5]. So using this information, we can attempt to combine the numerical and the physical models.

3. EXPERIMENTAL APPROACH

3.1 Principe

3.1.1 Thermocouples

The micro-thermocouples of type-K have been chosen in our experimental study because they are most commonly used in industry. This type suites well to harsh environment and presents a wide range of temperature, between -75 °C and 250 °C. One thermocouple is composed of 2 wires in Chromel / Alumel with a diameter of 79 µm. This will induce very little disruption during the infusion process. Data is acquired through an acquisition unit Agilent 34970A, a multi-acquisition system with 20 channels for measuring electric current, voltage and resistance. The frequency acquisition meets our requirements (maximum frequency of 20 values/sec), and its resolution is 0.1°C in temperature measurement. The calibration of the measurement system has been achieved in an oven during 7 isothermal conditions and compared with a platinum probe (PT100). The resolution of this calibration is closed to 0.1°C.

3.1.2 Optic fiber sensors

To determine the resin flow front and degree of cure, the idea is to monitor the refractive index of the optical fibre during the filling and curing stage. In fact, the reflection coefficient R between two media of refractive index n_1 and n_2 can be calculated using Fresnel laws [14], it is expressed as:

$$R = \left(\frac{n_1 - n_2}{n_1 + n_2}\right)^2 \tag{1}$$

Moreover, the refractive index is sensitive to density variations. Indeed, the Lorentz-Lorenz law establishes a relation between refractive index and density of a liquid medium (Equation 2). Here, R_M is the molar refractivity and M is the molar mass.

$$\frac{n^2 - 1}{n^2 + 2} = \frac{R_M}{M} \rho$$
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As the reflected intensity at the end of optical fiber depends on the external reflection coefficient, we can monitor the resin front and degree of cure by observing the variations of the reflected intensity according to the equation 3. In this equation, A_0 is a transmission coefficient and B_f represents the coefficient of the mounting noise, the two constants must be determined at the beginning of each experiment. Figure 2 gives a schematic view of the measurement system.

$$I = A_0 R + B_f$$



Fig. 2 Principle of the measurement system by using micro-thermocouples and Fresnel's reflection with optical fibre sensors.

3.2 Experimental Set-up

Experiments were conducted with 48 plies composite plates $[0_6 \ 90_6 \ 90_6 \ 0_6]$ s, made up of "UD fabric" reference G1157 E01 produced by Hexcel Corp. These carbon fabrics are plain weave with 96% of weight in the warp direction and 4% of weight in the weft direction. The preform dimensions are 335 mm × 335 mm × 20 mm. For the resin, the experimental LRI tests have been performed using an epoxy resin (HexFlow© RTM-6). Before injection, the resin is preheated to 80°C in a heating chamber. A heating plate located below the semi-rigid mold heats the preform. As for the external pressure prescribed over the stacking, it is uniform and equal to the local atmospheric pressure, induced by the vacuum ensured in the sealed system. Figure 3 shows un example of infusion of a plate carried out by LRI process, the resin inlet and outlet are indicated in this figure. The lid helps us to obtain a homogenous temperature field in the filling and curing stage: in classical tests, the filling temperature is 120°C and the curing temperature is 180°C maintained for a period of two hours.



Fig. 3 Infusion of a plate carried out by LRI process.

The experiments are perfomed on the specimen described previously. For the purpose of detection of the resin flow and monitor the curing stage, the test consists in infusing thick plates instrumented with one optical fibre sensor and six micro-thermocouples. All of the sensors' positions are shown in Figure 4: five thermocouples are across the thickness of perform, thermocouple 3, 4 and one optical fibre sensor are embed on center of the mean plan, 1 cm between each sensor, thermocouple 1 is placed in the entrance of the system for monitoring the temperature of the resin inlet.



Fig.4 Micro-thermocouples (TC) and optical fibre sensor (OF) location in the preform stacking.

3.3 Resin flow detection

The change in the temperature of resin inlet is presented in figure 5. The temperature falls at 10 s, which can be considered as the time of test beginning. Due to the effect of resin flow, thermocouple 1 sticks on the warmer inlet pipe, then its signal gives the temperature of both resin and inlet pipe.



Fig.5 Change in time of the signal of the thermocouple placed in the inlet pipe of the infusion system.

The five curves in Figure 6 have similar features generally. Time 0 corresponds to the beginning of the infusion process. There are temperature differences about 10°C at the beginning of infusion stage and about 5° C in the end. As we have proposed in previous publications [12, 16], the temperatures tend to decrease when the test begins and the resin is left free to fill in the preform. This phenomenon confirms the presence of the resin that tends to cool down the preform, because the measurements of microthermocouple 1 show that the temperature of the resin inlet is always below 100°C during the filling stage (see figure 5). The temperature signals of thermocouples decrease more and more when the resin front flows getting close to the thermocouples. Once the resin front arrives in the vicinity of the thermocouple, a minimum temperature is got. The time of resin arrival predicted by each thermocouple is figured out. After this minimum is reached, the signals of the thermocouples increase because the resin is heated by the heating plate while it flows in the preform. When the resin reaches the mould in the bottom, it is very rapidly heated by conduction (see the signals of TC 5 and TC 6). Eventually the saturated preforms reach a stabilized temperature at about 1300s, which is considered as the filling duration of the infusion stage.



Fig.6 Change in time of the signal of the thermocouples placed across of the preform.

Figure 7 shows the light intensity reflected by the end of optical fibre and the temperatures nearby this fibre versus time along filling stage. The signal reflected by the fibre / air interface is perfectly stable initially. When the resin reaches and touches the end of the optical fibre, the light intensity falls due to the immediately changed refractive index. In 5 seconds, the optical fibre signal returns to the initial level and then falls again, it may reveal that there is some air mixed in the resin flow. When the end of optical fibre is fully surrounded by resin, its signal becomes stable and we can consider that the resin front reaches the center of the mid-ply at 350s. In the other hand, the responses of the thermocouples 3 and 4 exhibit a minimum at about 320s (315s for

thermocouple 3 and 325s for thermocouple 4), which presents 8.6% difference between the two measurement techniques. In order to fix the position of each thermocouple, a little quantity of resin (the same resin used in the infusion test) has been used to stick the head of the thermocouple. Maybe due to this, the detection of thermocouples is advanced, but this difference is acceptable regarding the characteristic of infusion duration.



Fig.7 Optical fibre and the thermocouple 7 and 8 signals versus time.

3.4 Curing stage monitoring

Figure 8 gives the temperature and optical signal of the sensors located at the center of the mid-ply during the filling, curing and cooling stages in LRI process, it can be split in 5 parts. Firstly, the resin arriving time and the minimum temperature can be observed at zone 1, which stands for filling stage. After 350 s, as the temperature increases, the refractive index and the reflected light intensity decrease. Then, in the temperature augmenting-zone (zone 2), the optical fibre signal is supposed to decease, but our optical fibre signal continues decreasing a little and does not evolve, since we have some problems in our oscilloscope during about 10 minutes. Fortunately, the oscilloscope can be functional before the curing stage. It only occurs in this test.

Zone 3 corresponds to the curing stage. In this stage, the liquid resin is transformed into solid one. In addition, the reflected light intensity is augmented during this transformation [15]. In fact, it involves an increase of resin density in the curing stage, so the refractive index of the optical fibre augments according to the equation 2. Once the reflected light intensity no longer changes, it can be considered that the curing phase

is finished and the resin is fully cured at 6000s. In other words, the curing time is about 52min. During the cooling stage in the 4th zone, the resin volume is decreased, therefore its density is increased and the signal of optical fibre is also increased relatively. At the end of LRI process, the temperature and the reflected light intensity stabilize in zone 5.



Fig.8 Change in time of the signal of the optical fibre sensor and the thermocouple placed in the centre of the mid-ply.

4. COMPARISON OF EXPERIMENTAL RESULTS WITH NUMERICAL PREDICTION

A comparison can be made between experiments and simulations of the filling stage for this case studied here. Simulations have been realized with 1426 triangle mixed velocity-pressure elements. We have considered that the resin viscosity is a constant and represented as precisely as possible the process boundary conditions. Starting from the initial thickness measured, 20 mm, computations yield a thickness after compaction of 12.7 mm by vacuum bag while measurements give 13 mm. After the filling stage, numerical simulations show that the filling time of the preform is 1095 s while the experiment notices 1180 s (\approx 120 s to infuse the distribution layer). Regarding the mass of resin received in the process, it is 750 g numerically for the plate and 655 g experimentally for the system (normally, it used 100 g for the distribution layer). In our simulations, the resin mass depends strongly on the variation of the preform thickness during the infusion stage. Here, for this infusion case with a close lid, we have not found a better way to get the information about the preform thickness variation. So this difference can be related to the problems in the simulations, such as the mesh dependency, filling algorithm, macroscopical approach... To sum up, only a global numerical framework associated with proper experiments permit to get a deep insight into the mechanisms controlling the filling stage of composite preforms.

5. CONCLUSION

The paper referring to new advances in process numerical modelling emphasises the need for advanced experimental characterisation to serve as references to benchmark new simulation tools. Firstly, as a novel direct approach, optical fibre sensor based on Fresnel reflection is used to detect the resin flow front during a plate infusion process under industrial environment. It will be feasible to monitor the whole LRI process, especially detect the resin flow position and advancement of the curing stage. Micro-thermocouples could follow the temperatures inside the preform during the process. As an indirect method, micro-thermocouples could not detect the resin flow in more accurately than optical fibre sensor, but the results obtained in particular prove that they can be used to have first information on the resin flow in the preform. So the thermocouples with low intrusively and friendly-user will be a promising technique to monitor the fabrication process of the composite materiel. Secondly, the first confrontation between experimental and numerical simulation results has let to the two following points: complexity of the phenomenon infusion and correlation encouraging for the filling time and the mass of resin received by the preform in the filling stage.

In the future work, in order to obtain the evolution of the resin front flow, it is necessary to proposed several optical fibre sensors in the different measurement points in the preform. These results can verify further the measurements of the micro-thermocouples. Moreover, the monitoring of the curing degree will be also carried out in different plies by optical fibre sensors.

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