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Anomaly Detection during Thermoplastic Composite Infusion: Monitoring Strategy Through Thermal Sensors

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Abstract. Vacuum assisted resin infusion (VARI) processes are widely used for the fabrication of large composite parts. For some years now, wind blades are designed with thermoset composite materials, however, recyclability is often the missing brick. The recent advent of thermoplastic materials such as ELIUM©, developed by ARKEMA, shows the possibility to consider the manufacture of very large parts such as wind turbine blades with recyclable constituents. The French institute IRT Jules Verne is leading the research project ZEBRA, with a consortium of industrials, focusing on different thematics of the manufacturing of wind turbine blades, including process monitoring. In the objective of evaluating the possibility for anomaly detection during the ELIUM© infusion process, an infusion test bench was developed, equipped with different sensors including infrared camera, thermocouples and heat flow sensors. In this work, the thermal sensors are evaluated by the study of several infusion scenarios of plates fabricated under different process conditions and material parameters. Artificial anomalies are imposed into the process to evaluate the potential of these sensors to detect the induced disturbances. A numerical process modelling is developed, compared to the sensor outputs and used to achieve a better understanding of some effects observed during the process.

Introduction

Wind energy, both onshore and offshore, plays a critical role in the world's transition to carbon-free energy sources. With a product lifespan of 30 years and a wind turbine recyclability rate of 85% to 90%, the wind power industry is now looking to close the remaining gap by designing and manufacturing the first 100% recyclable wind turbine blade**.** The ZEBRA (Zero wastE Blade ReseArch) project, driven by IRT Jules Verne, now brings together industrial companies and technological centers – IRT Jules Verne, Arkema, CANOE, ENGIE, Suez, LM Wind Power, Owens Corning – to tackle with this new challenge, among all technical challenges.

Fig. 1: One of the three wind turbine blades (LM107) the now flies in Rotterdam with the employees that participated in the blade design and fabrication

Infusion Process - Basics

During resin infusion, dry and environmental fabrics are stacked on a mold and form a dry preform. This dry preform is then compacted against the mold by using a vacuum bag or a semi-rigid counter-mold (elastomer type). Once the stack has been compacted, the resin is infused into the reinforcement and then penetrates the thickness and impregnates the dry fabrics. After filling the dry preform, the resin polymerises under the vacuum bag and it can be accelerated by direct heating of the mold or in an oven. Since resin infusion requires little investment and allows the production of parts in small series, this technology is widely used in small companies, or for the manufacture of parts whose dimensions do not allow the use of autoclaves. Infusion is often found in the manufacture of boat hulls or wind turbines, but also in aeronautical and naval structures. However, the family of resin infusions is very broad and the classification can be refined by distinguishing the pressures and compaction cycles applied, the used consumables, tooling to create the vacuum or the types of resins used. Among the large families of infusions, we find for example the patented Boeing CAPRI (Controlled Atmospheric Pressure Resin Infusion) [1], or SCRIMP (Seemann Composites Resin Infusion Molding Process) [2] technologies in which a distribution medium is added to ease the resin flow. This technology allows the use of different matrices: unsaturated polyester, vinylester and epoxy, and more recently liquid thermoplastics.
Infusion process has many advantages such as a high fiber content, therefore a better strength-to-weight ratio, the realization of large parts in "one shot", a large possibilities of shapes, a good protection of operators from emissions of solvents such as styrene, a relatively low investments given that the pressure to which tooling is subjected remains low and relatively simple tooling. On the other hand, some limitations subsists such as the possibility to have only one smooth side, a low production rates, and high use of consumables and also extensive labor need for draping and bagging creation. Moreover, the process is often handmade and reproducibility is questionable. That is why it is necessary to monitore the process to ensure the good quality of the process during filling and then curing (or polymerization), which is not user-friendly for high size configurations.
The materials often used for large composite parts processed by infusion ar thermosets, due to very low viscosity. The recyclability of thermoplastics gives them a real competitive advantage over thermosets. In order to be used, the thermoplastic must have a very low viscosity (traditionally under 300cP). Reactive processes are one of the solutions considered industrially.

Material Description

Thermoplastic Elium® 191 XO/SA is a clear, yellow, two-components pre-accelerated thermoplastic acrylic resin. It is designed, manufactured and supplied by Arkema. The polymerisation principle (radical polymerisation of methyl methacrylate (MMA) to acrylic copolymers) is described in Fig. 2. The polymerisation is initiated by the addition of a catalyst: Butanox® M-50, which contains methyl ethyl ketone peroxide. This peroxide cleaves at the O-O bond at ambiant temperature. These oxygen atoms become radicals (they have an unpaired electron). This characteristic makes the peroxide products very reactive. These attach to the methacrylate function of the MMA (this step is

called priming). The product of priming also contains a free radical to which an MMA is attached (propagation). The propagation is repeated until the radical site is deactivated (termination) or reacts with another copolymer (chain transfer).

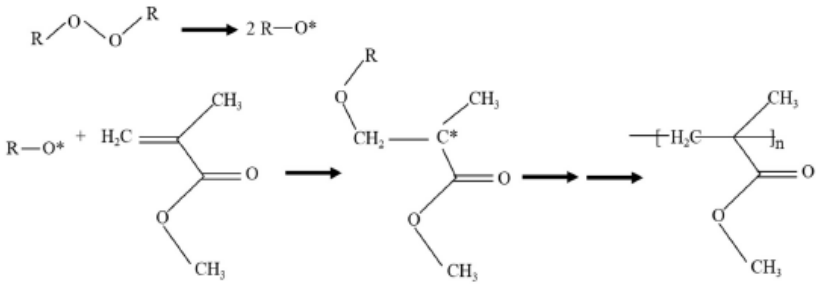

Fig. 2: Polymerization MAM->PMMA

Infusion Process Monitoring

Monitoring the infusion process usually means evaluating in real time the position of the material front, the progress of the cure reaction and the appearance of defects. This is particularly interesting from an economic point of view as soon as the part is (almost) unitary and with high added value. There is a fairly large number of sensors available that can measure some of these quantities. The interpretation of the signals is rarely easy, and the scientific literature is quite abundant in this sector. The Table 1 from *Konstantopoulos et al.* [3] shows the different types of sensors used in the infusion process, and their associated physics. The authors present a most exhaustive list of monitoring technologies and literature review concerning composite infusion.

Table 1: Properties detected by different sensor types and process elements deduced from an infusion process [3]

		Flow front	Curing degree	Void content	Delaminations
Electromagnetic properties	Dielectric analysis (DEA)	✓	✓	✓	✓
	Direct Current (DC) analysis	✓	✓		
	Electrical time domain reflectometry (ETDR)	✓	✓		
Mechanical properties	Optical fiber interferometers (OFI)	✓	✓		
	Ultrasonic transducers	✓	✓	✓	✓
Optical properties	Optical fiber refractometers (OFR)	✓	✓		
	Spectrometers	✓	✓		
Thermo-dynamical properties	Thermometers	✓	✓		✓*
	Pressure transducers	✓			

In the present work, some heat transfer sensors (thermocouples, infrared camera and heat flux sensors) have been chosen to monitor the process, and to detect some process deviations or anomalies.

Experimental Trials

Experimental test bench. A 5-ply plate (300mm x 500mm) tool is placed on a measurement test bench (Figure 3). The vacuum is provided by a vacuum pump and a downstream pressurising machine is used to control the progress of the resin (Figure 4). The structure of the test bench allows for the installation of an infrared camera, a visible light camera, and also allows for the integration of the necessary support for thermocouples and heat flux sensors instrumentation.

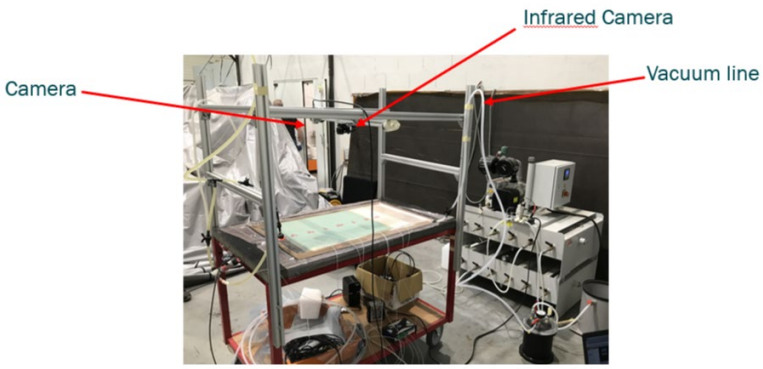

Figure 3: Experimental test bench

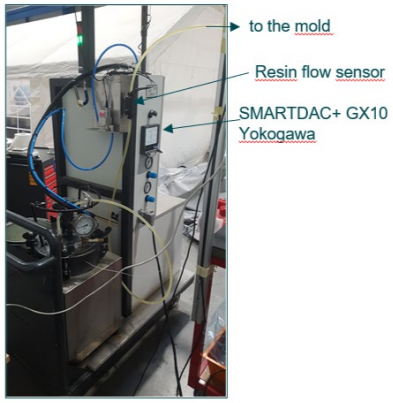

Figure 4: Dedicated LRI machine

Experimental set-up. The part is made by frontal infusion on a flat half-mould. Figure 5 shows the structure of the impregnation bench and the associated measurement chains. Before the infusion, the circuit is put under vacuum (R1, R2, R3 open, R4 closed). A vacuum loss test is carried out by connecting a manometer to the position of R3. This test is used to check that the amount of leakage present does not allow too much air to enter the preform (vacuum loss objective < 10mbar / 10 minutes). The main stages of the infusion are then:

- Filling the upstream resin circuit (R1, R2, R4 open, R3 closed)
- Start the acquisition
- Infusion (R1,R3,R4 open, R2 closed)
- End of acquisition. Closing R4

Every step is under time control by an in-house control software.

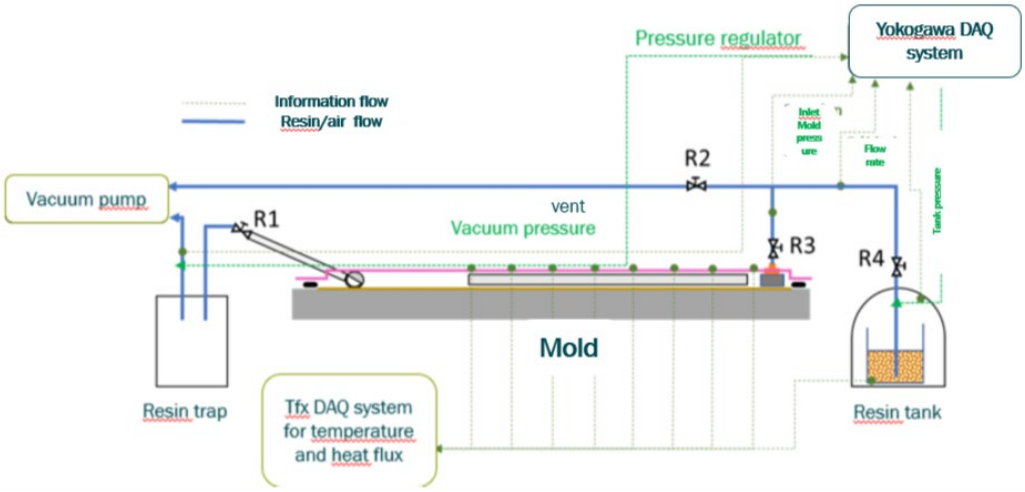

Figure 5: LRI test bench scheme and associated data acquisition systems

Heat flux sensor. Resin infusion in reinforcement induces thermal changes which can be detected thanks to the high sensitivity of heat flux sensors. Heat flux sensors takes advantage of heat transfer by diffusion and convection for monitoring thermal changes inside products without direct contact. It is therefore possible to monitor material temperature throughout the complete polymerization cycle from the outside of vacuum layers. Sensors are simply taped on the top of vacuum bag, on the area of interest (Figure 6).

Infusion process of large parts involves almost isothermal filling of performs and if so, should not be followed by standard temperature or heat flux sensors. By adding a heat source near a heat flux sensor, it is possible to enhance the sensitivity of the sensor to resin flow front. The main effect

detected is the temperature change induced by resin convection. Prior tests [4] have shown that the heat flux level after the flow front is also linked to the local flow rate of the resin beneath the sensor.

Measuring local resin flow rates: The calibration of such an active heat flux sensor as resin flow is however difficult due to an inherent sensitivity to thermal changes of ambient and sometimes existing thermal regulation. A simple method consists in measuring the transient of the signal when the resin flows under the sensor. The ratio of sensor size by the oblique-step shape duration provides a direct measurement of the flow rate in mm/s in the sensor area.

Following curing reaction: Most reactive resins start to polymerize during infusion, generating heat which increases the resin temperature. In this case it is not necessary to activate the heat source of the sensor in order to detect flow front, and the heat flux sensor is useful for estimating the temperature of the resin beneath the vacuum bag. After infusion, classical calorimetric methods can be applied like for DSC (differential scanning calorimetry) in order to identify a base line, curing rate and relative cure level by integration from the heat flux signal [5].

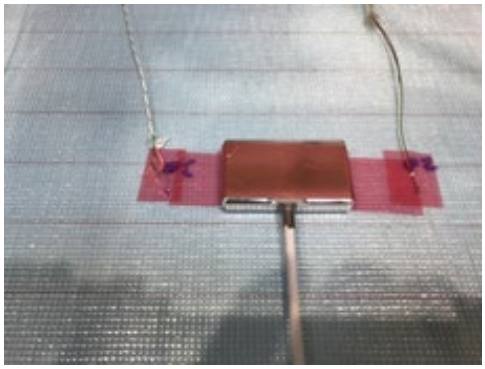

Figure 6: Tfx© Heat flux sensor on vacuum bag

The Figure 7 shows the implementation of data acquisition system including heat flux sensors, 7 thermocouples and infrared camera OPTRIS on the test bench. The sensors are placed as Implementation of sensors shows.

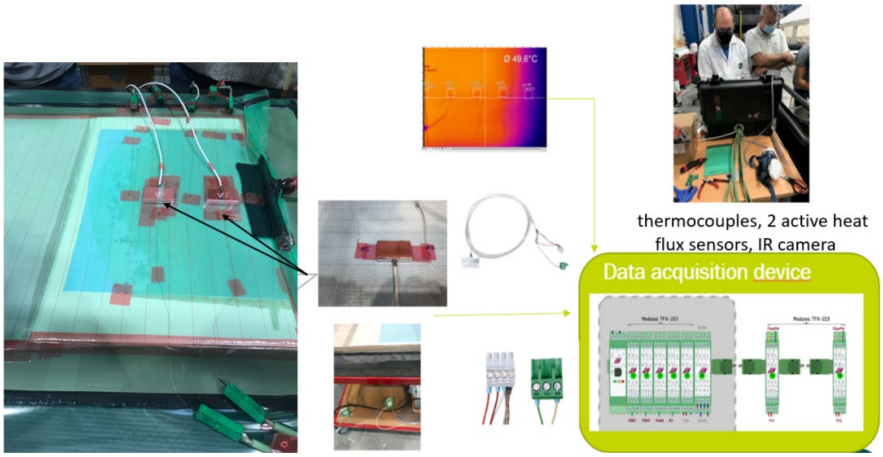

Figure 1: General view of the data acquisition devices : 7 thermocouples, 2 heat flux sensors (Tfx), 1 infrared camera, stored by Tfx device linked with Yokogawa system

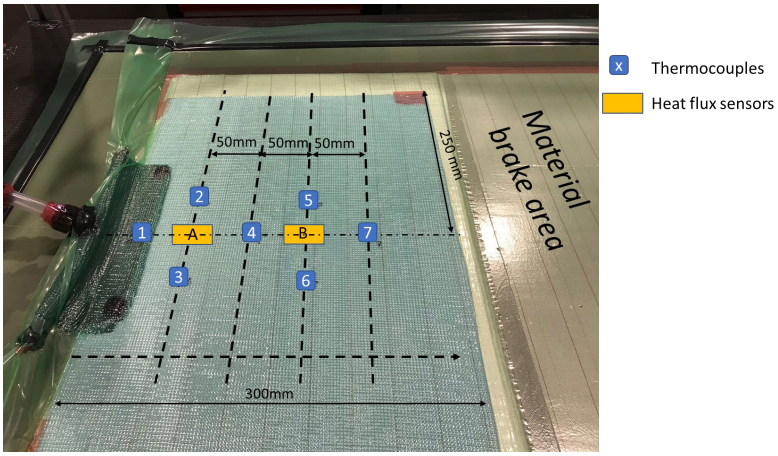

Figure 2: Implementation of sensors

Experimental Results: Reference Case

Results Description. The heat flux evolution with time shows clearly filling phase, and then polymerization phase (Figure 9). During filling phase (Figure 10), the resin is colder than the heat flux sensor. That is why the heat flow sensor detects a positive heat flow which means the heat is brought into the resin from the sensor. On the other hand, during the exothermic polymerization, the resin brings heat into the sensor, and the heat flux is negative. Note that this is only conventional approach. Due to thermal contact resistance between the sensors and the vacuum bag, the accuracy of heat flux value is not very high. Nevertheless, this value during filling is significantly higher for the sensor near the inlet. Indeed, the sensor is very sensitive to the speed of the flow (and associated convection), and the front velocity is higher on sensor A than on sensor B. Moreover, there is a saturated front near the sensor A, while there is an unsaturated front then saturated front near sensor B. The end of the filling phase seems to stop after about 10 minutes. During polymerization, the heat flux becomes significantly negative, reaching a peak after about 4h after filling phase. It is interesting to note that the polymerization seems to begin very slowly just after filling, since the heat flux evolution shows it decreases slowly during 3 hours before accelerating the exothermic reaction.

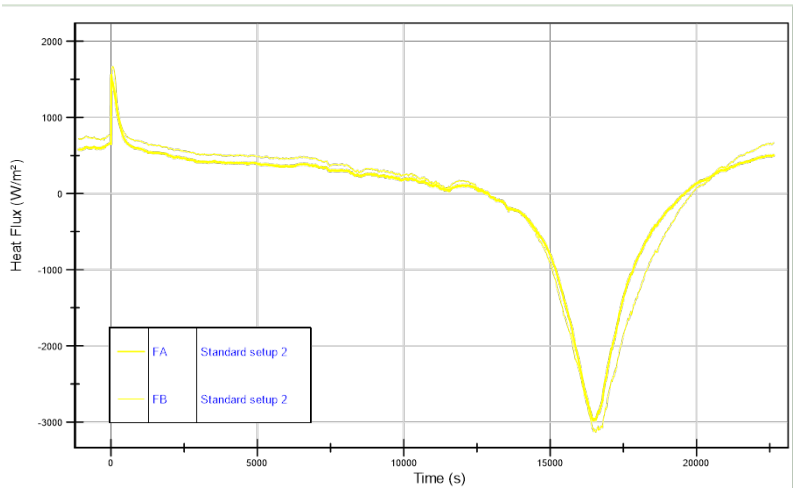

Figure 9: Heat flux in position A & B – Filling and polymerization

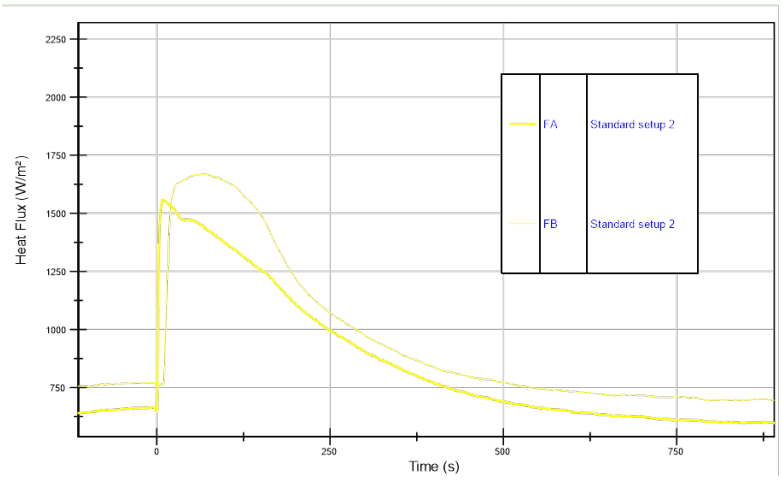

Figure 10: Heat flux in position A & B – focus on filling phase

Comprehension by Process modeling and simulation. In order to understand the process and the signals obtained by the sensors, a simulation has been studied. The infusion process combines several physical phenomena: fluid mechanics represented in resin flow, heat transfer and resin kinetics of reaction. The potential deformation of the medium because of using flexible tooling is not considered in this study, the focus is drawn to resin flow, heat transfer and resin polymerization.

Flow in porous media : The flow of the incompressible fluid is governed by the simplified form of the mass balance equation (Eq. 1)

$$\nabla.\left(\vec{U}\right) = 0 \tag{1}$$

Where $\vec{U}$ is the velocity vector. The flow of Newtonian fluids through porous media is described by Darcy's law, which relates the velocity vector the permeability tensor $\boldsymbol{K}$, the resin viscosity μ and the pressure gradient ∇P (Eq. 2)

$$\vec{U} = \frac{-\boldsymbol{K}}{\mu}\nabla P \tag{2}$$

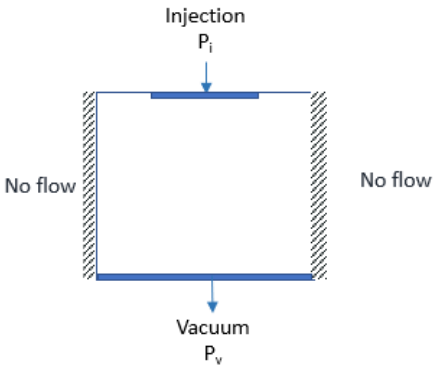

Figure 11: Boundary conditions of the flow problem

The problem is modeled as a two-dimensional (rectangular) geometry. As shown by Figure 11, part of the mold upper-side is prescribed as the flow inlet (line injection), with a constant pressure value equal to the atmospheric pressure ($P_i = P_{Atm}$). The lower-side line of the mold is prescribed as the flow outlet or the vacuum pressure. The no-slip boundary condition is applied to the other two sides of the mold.

Heat transfer: The heat transfer problem is governed by the energy balance equation. The terms of the energy balance equation in the left-hand side are respectively from the left to the right: the transient and advection terms. In the right-hand side of the equation, the first part of the equation is the conduction term. The internal heat generation in the system is represented by r (Eq. 3)

$$\rho\, c_p \frac{\partial T}{\partial t} + \rho_r c_{p,r} \vec{U}.\nabla T = -\nabla.(\lambda.\nabla T) + r \quad (3)$$

where T is the temperature, ρ and ρ_r are respectively the composite and the resin density. c_p and $c_{p,r}$ are the composite and the resin specific heat capacity. The subscript r denotes the resin. λ is the thermal conductivity of the composite material.

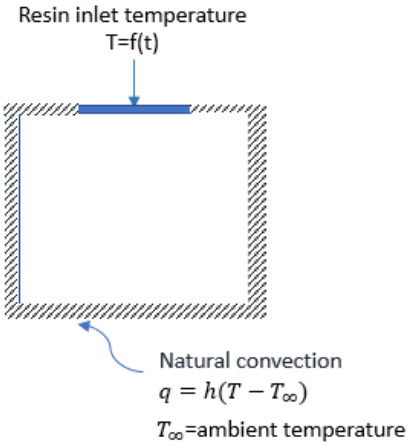

Figure 12: Boundary conditions of the heat transfer problem

The heat transfer problem is controlled by the initial temperature of the resin and its evolution with time at the preform inlet as well as the advancement of the reaction kinetics of the resin. All the other surfaces are subjected to natural convection boundary condition, governed by Fourier's law for homogeneous media:

$$\vec{q} = h(T - T_\infty) \quad (4)$$

$\vec{q}$ is the heat flux density, h is the convective heat transfer coefficient and T_∞ denotes the surrounding temperature, i.e. the ambient temperature.

Resin polymerization: The term r in the energy equation is the volumetric heat generation related to the resin polymerization. It is given by Eq. 5:

$$r = \rho_r \Delta H \frac{\partial \alpha}{\partial t} \quad (5)$$

Where ΔH is the total enthalpy of reaction and α is the degree of cure. It is essential to know the kinetics of cross-linking of the resin in order to determine the amount of heat generated during the transformation. The kinetics is the relationship that expresses the reaction speed as a function of the rate of cross-linking α (advancement of reaction) and the temperature T (Eq. 6):

$$\frac{\partial \alpha}{\partial t} = f(T, \alpha) \quad (6)$$

Several models were proposed for modeling the polymerization kinetics of polymers. Few researchers have investigated the polymerization kinetics of Elium® resin ([6-7]). Palmeiri et al [8] studied the curing kinetics of Elium®188 resin through differential scanning calorimetry isothermal and dynamic DSC tests. Results are fitted to Kamal-Sourour model (Eq. 7-8):

$$\frac{d\alpha}{dt} = (k_1 + k_2 \alpha^m)(\alpha_{max} - \alpha)^n \quad (7)$$

$$k_i(T) = A_i e^{-\frac{E_i}{RT}} \quad (8)$$

The parameters of Kamal-Sourour's semi-empirical model are: E_1 and E_2 the activation energy (4.34x104 and 1.5x104 J/mol), A1 and A2 the pre-exponential factors are respectively 1.8x103 and 1.99 s-1, R is the gas constant 8.314 J/mol.K, and the parameters m and n are respectively 1.3 and 1.75.

It will probably necessary to modify this model in order to take into account the very slow evolution of α at the end of the filling phase. Nevertheless, we study here only filling phase, and we don't need accurate evolution of the polymerization phase.

The simulation is realized with the commercial software PAM-RTM©. The software is dedicated to modeling the resin infusion process with continuous fibers. It permits coupling the physical phenomena involved in the infusion process. The geometry is modeled as the real dimensions of the part, meshed and the boundary conditions are applied as shown by Figure 13. Seven sensors are introduced at the top of the stacking. An absolute pressure of 0.8 bar is applied at the inlet and a vacuum pressure of 0 bar is applied at the vent line.

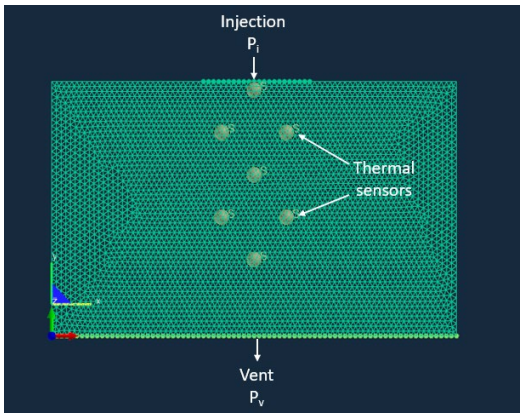

Figure 13: Geometry, meshing and flow boundary conditions

The simulation has shown a good agreement in filling time with the experiment (418 s for the simulation versus 420 s obtained experimentally). Figure 14 shows the pressure distribution and the flow velocity profile at the end of infusion.

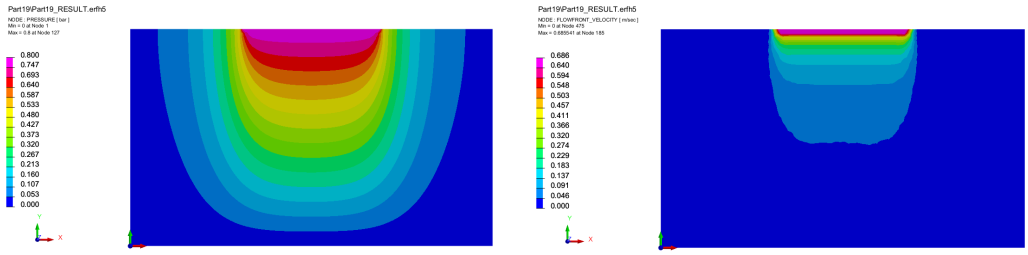

Figure 14: Pressure distribution at the end of filling and the flow front velocity

The simulation and experimental results are compared in term of resin passage time at sensors. As seen in Figure 15, the flow velocity is very high at the beginning of infusion, hence, the resin enters the medium and arrives instantly at sensor 1, then reaches the sensors 2 and 3, and sensor 4 at a Δt of 0.5 s. Then the time difference is widen after as the resin advances towards the end of the mold. The time evolution of temperature depends on the applied boundary conditions as well as the advancement of reaction kinetics. Before the part filling, the preform is assumed to have a constant temperature. As the resin enters the mold, the filled regions start to attain the resin temperature which evolves with time. Higher increase in resin temperature is registered in the injection pot, however, when the resin crosses the connections of almost 2.2 m long, its temperature drops at the part entrance. During part filling, only slight temperature increase is observed. The Figure 15 below shows the time evolution of temperature along the part at t*= 0.1, 0.25, 0.5, 0.75 and 1 (t* is the normalized filling time, t*=t/total filling time).

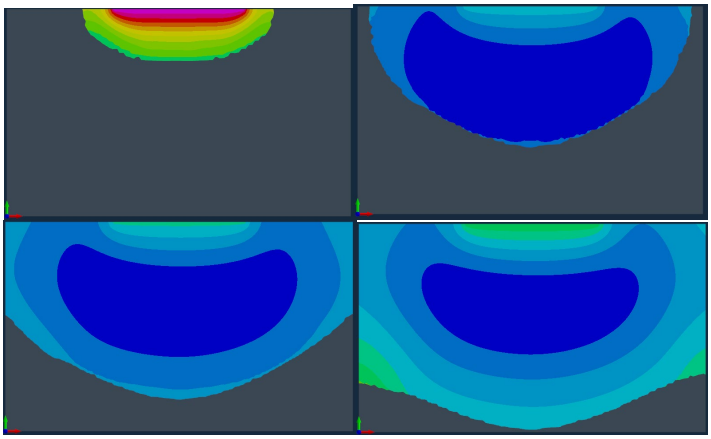

Figure 15: Time evolution of temperature at t=0.1, 0.25, 0.5, and 0.75 during the filling phase*

The beginning of infusion (t*=0.25) the resin temperature is higher than the fibrous medium. By the end of infusion, the temperature at part edges drops as a consequence of natural convection between borders and the surrounding medium. Very slight heat increase is numerically observed during infusion because the exothermic reaction takes place long time after.

Experimental Results: Repeatability and anomaly detection.

Process repeatability. 5 parts were produced under similar conditions, with identical process parameters. A software tool was specifically developed to time and control each manual step of the process. The preforms were stored in the same way, with mixing carried out under controlled conditions. The aim of this study is to evaluate the reproducibility of the process and its associated measurement.

On the heat flow signals (Figure 16), a certain apparent dispersion of the experimental results can be observed. Over the whole cycle, the polymerization peak times are quite different for the five parts studied, ranging from 15000s to 17500s, i.e. over 40 minutes. The shapes of the curves are very similar in term of slopes and minimum values. That means that the kinetic of polymerization is quite similar. During filling phase (Figure 17), the reproducibility seems good, since peaks, slopes, and characteristic times are similar.

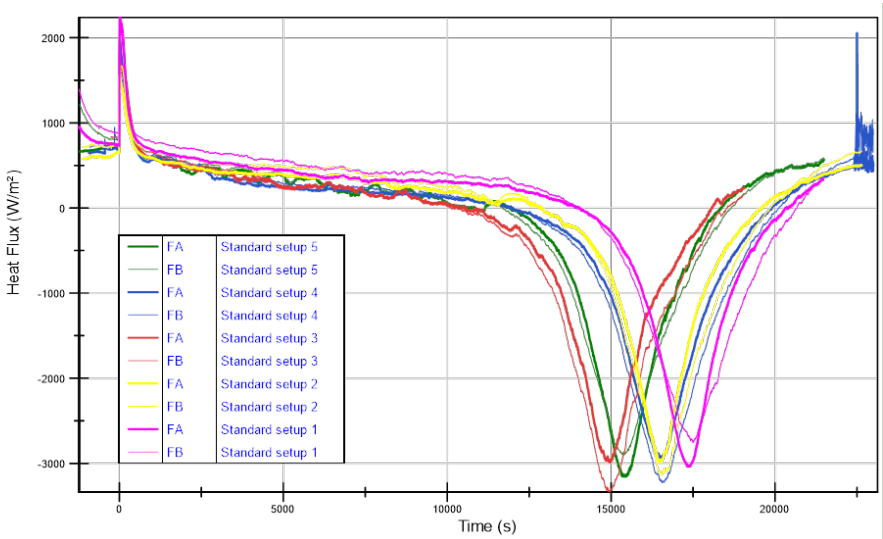

Figure 16: Data acquisition of 5 similar parts under same conditions

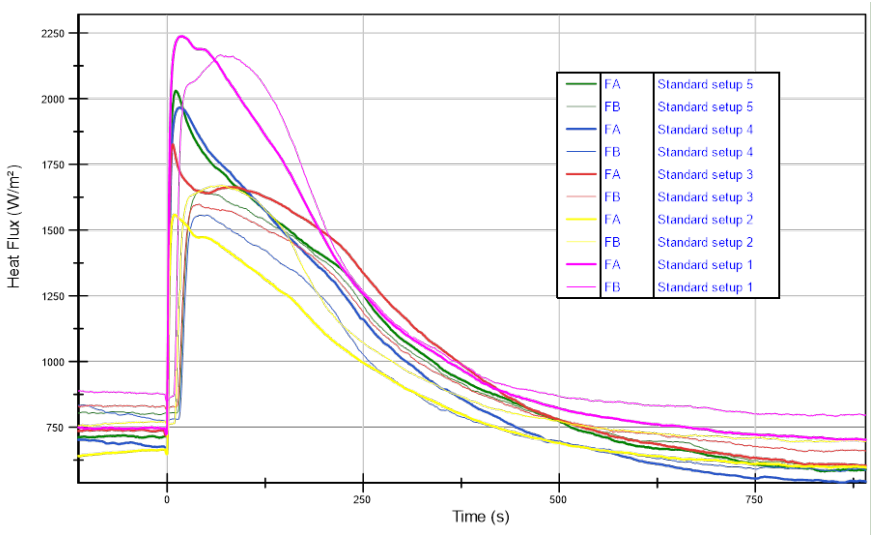

Figure 17: Data acquisition of 5 similar parts under same conditions – Zoom on filling phase

Detection of process anomalies. A FMEA (Failure Mode Effects Analysis) focusing on the risk of producing no-conformal plates by ELIUM infusion was used to select and rank the process parameters (classified according to the 5Ms : Method, Man, Material, Machine, Measurement) that can contribute to poor quality. The challenge of this study is to evaluate the detectability of this non-quality created by the non-control of these parameters by the thermal sensors embedded in the process. Three parameters have been chosen:

Parameter 1: Method-Fixative spray for dry lay-up applied with too much quantity: The simulation of a bad application of spray between plies was carried out. We deliberately applied a spray twice as close and for a longer time to join the plies. This should decrease the final mechanical properties of the part after process. The results are summarized on the Figure 18. Two similar tests were carried out. The two tests give relatively different heat flux measurements, although the peak exotherm and endotherm values are significantly lower than in the reference cases for both. Polymerization also appears to be significantly slower, although it remains within the range of the reference cases. This can be identified as a process signature of excessive glue applied during preforming step. During the filling phase (Figure 19), the flows are significantly lower than in the reference cases, and the signal appears later. This corresponds to a slower front speed and is also detectable by thermal flux sensors.

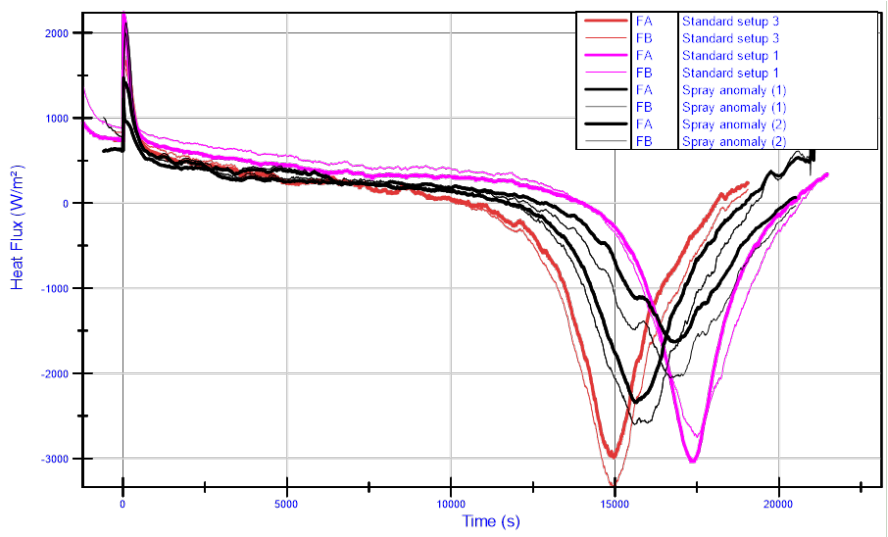

Figure 18: Heat flux evolution for high quantity of spray, and comparison with references

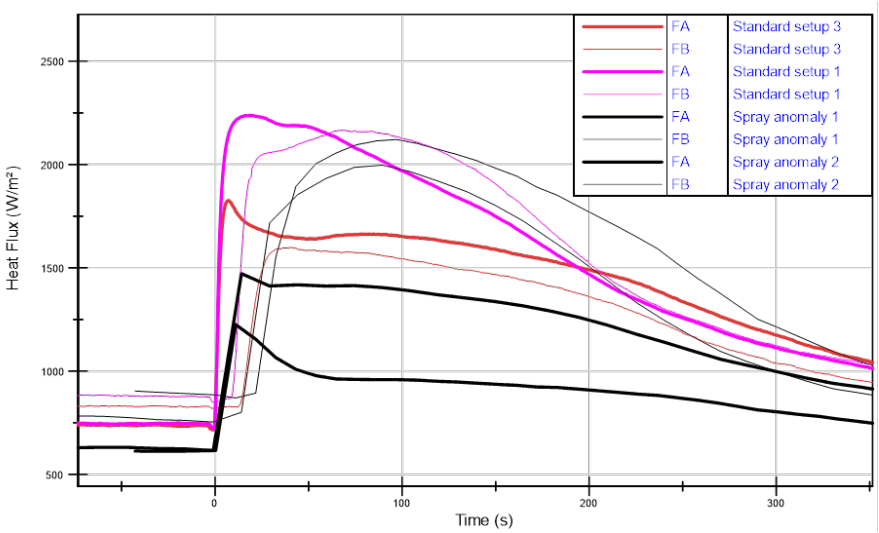

Figure 19: Heat flux evolution for high quantity of spray, and comparison with references – Focus on filling phase

Parameter 2: Wrong content of initiator in bulk resin: The simulation of an invalid concentration of initiator (catalyst) prior to resin infusion. This error may occur due to mixing head fault when such system is used. In this simulation, the initiator volume is added manually approximatively 20 minutes before the start of infusion. On the one hand, the infusion of resin with a higher content of initiator is quite representative of standard setup measurement with very close amplitude of polymerization heat peak occurring in the range of time observed with the five standard infusions (Figure 20). On the other hand, the downstream fluxmeter (FB) shows a rapid increase 26 seconds after the upstream one (FA) (figure 21). As a matter of fact, resin flows slowly in comparison with reference or little initiator content test. The infusion with a low initiator content doesn't show a significant deviation from reference during filling phase. Nonetheless, the polymerization phase is slower and less energetic than the reference's one.

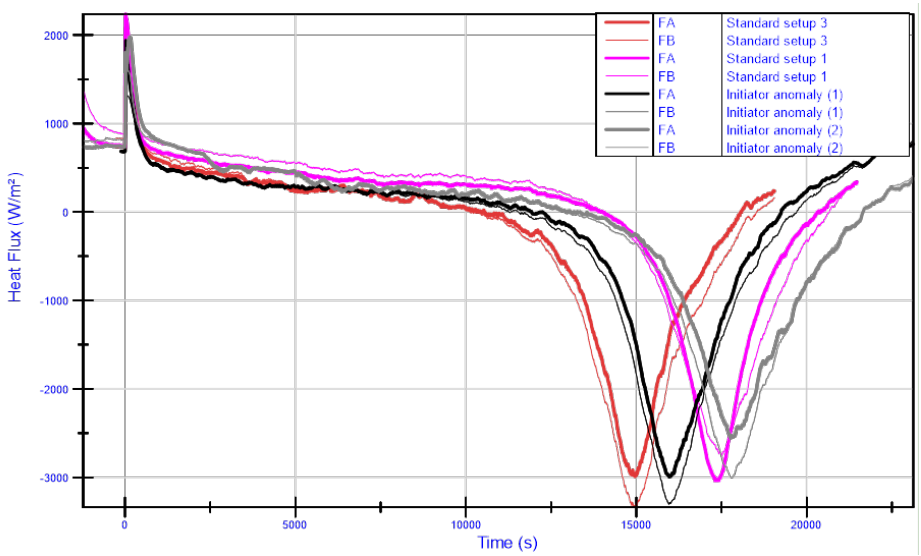

Figure 3: Heat flux evolution for more initiator content (+33%, 1) and less (-33%, 2), and comparison with references

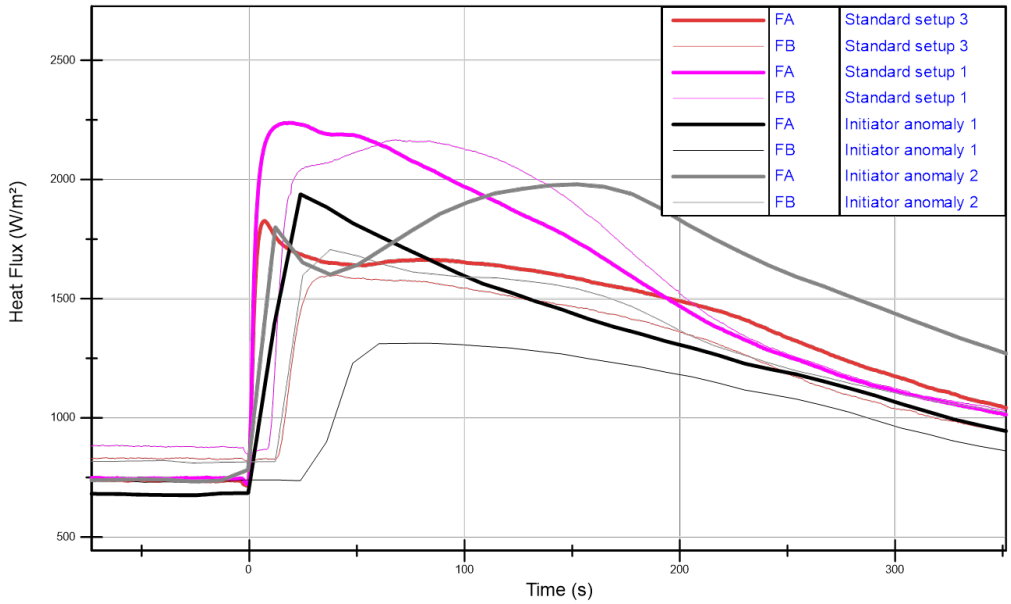

Figure 21: Heat flux evolution for more initiator content (+33%, 1) and less (-33%, 2), and comparison with references - Focus on filling phase

Conclusion and Perspectives

This work highlights the value of thermal instrumentation during the infusion process of thermoplastic composites. It showed that the process is sensitive and that control of the surrounding parameters is necessary. Furthermore, the Tfx© sensors chosen, which can be implemented industrially, allow certain process anomalies to be detected. In the future, it will be interesting to couple these sensors with other types of measurements, to increase the accuracy of detection, and to classify the anomalies.

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