Practical and Theoretic Analysis of Resin Flow in Vacuum Assisted Resin Transfer Molding Processes

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Summary: In this study, the non-isothermal resin infiltration during the VARTM process has been analyzed and characterized.
In particular, the resin flow through the fibers has been described by the Darcy law and introducing the degree of saturation, the percent of void effectively occupied by the resin within a control volume, as variable in order to account for the moving boundary nature of the problem.
Some preliminary tests to measure the advancement of a reference substance, pure water, between the reinforcement fibers with embedded optic fibers sensors, show the capability of a new sensorial technology, based on use of non intrusive fiber optic COTS components, assembled into an original way. These sensors will be used for real time monitoring of the resin flow during the experimental infusion tests and for in-situ demonstration of the capability and the reliability of the model, in the aim to assure a "process repeatability".
Due the effectiveness lack of repeatability of VARTM, the Authors propose an approach to extract a parameter during the process, meaning a "Deviation" of modelized output (MOD Modelized Output Deviation). This parameter, obtained by use of optic fibers technology, may improve the standard quality of the production, analyzing and correcting with feed-back capability the efficiency of the moulded manufactured devices.

Keywords: fiber optic sensor; infiltration; manufacturing; parameters; refraction index; smart process; VARTM

Introduction

Liquid Molding processes are promising and attractive low cost techniques for high performance polymer based composite materials manufacturing. The processing cycle involves the impregnation of a dry fiber math from a thermoset polymer resin. In general, the process is known as Resin Transfer Molding when the resin is injected into a closed mold, while as Vacuum Assisted Resin Transfer Molding (VARTM) process when the infiltration is driven by

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the vacuum and only one tool side is used being the other a polymeric flexible bag. Thus, the conventional RTM technique is a successful technology for small composite products in large series. On the other hand, the VARTM is interesting and more economical for the production of large size parts [1]. After complete infiltration, the resin reacts to form a cross-linked polymer network (cure reaction) giving the composite consolidation.

Due to the complex nature of the process, the processing modeling and characterization coupled to a proper control should aid the design of the processing parameters and enable the manufacturing of high performance parts. For this reason, to optimize the processing cycle not only by trial and error procedures, some authors have developed numerical simulations able to model the resin infusion process [2-3]. Theoretically, accurate information about the processing evolution could be provided by modelling both the resin flow, the heat transfer, the chemo-rheological resin behavior as in the case of RTM and compaction too [4]. Further, to ensure an high quality production, one of the most important issues is not only to describe correctly the fiber impregnation, but also to control the fiber wetting that affects strongly the final mechanical properties. Therefore, in last years many researches are developing innovative and non-intrusive techniques, such as fiber optic sensors to characterize the resin advancement. Fiber optic sensors offer the significant advantage to identify in-situ the flow properties during the real manufacturing process performed with the effective flowing resin system. This technique has a great potential providing experimental flow data in different locations being capable to be multiplexed. In addition, due to the small size (100-140μm), the optical fibers are minimally intrusive in the composite structures. Currently some authors performed experimental flow tests to monitor the resin advancement by fiber optic sensor during the RTM [5, 6, 7] and VARTM [8] stating the low cost and the flexibility of this technique to monitor the fiber wetting in-situ. In particular, the resin flow front position was recorded by measuring the light intensity at the end of the optic fiber which was embedded through the reinforcement after removing short segment of the cladding layer [5,7]. In fact, when the flowing resin reaches the fiber core, the light intensity drops significantly. In addition, by using a three-dimensional sensor grid embedded into glass fiber preforms having different fiber volume fraction, the permeability fiber optic measurements showed good agreement with flow visualization data [5].

Main focus of this study, partially developed inside the SMARTCOMP Project funded by the Italian Ministry of Research, is the modeling and experimental characterization of the resin flow during the VARTM process, in the aim to define a procedure where in-situ parameters can improve the repeatability of the system. In particular, the flow of a reference substance through a dry carbon reinforcement has been experimentally measured using an optical technique that take account the evidence of variations within a certain range of "normality". For this, COTS optic fibers have been used in an optic scheme [14] to highlight the walk-through of resin in front of the cut part of a single fiber. Some repeated tests show the lack of repeated behavior of flux, even if the boundary conditions were the same.

Infiltration Modeling

The resin flow during liquid molding processes is a typical moving boundary problem, characterized by a moving flow front that divides different phases: dry fibers and the wet fibers/resin system. In general, many authors have developed theoretical models of this kind of process all based on the Darcy's law [9 - 11]. In fact, the most common governing equation for the pressure distribution is:

$$\underline{\nabla} \cdot \left(\frac{\underline{\underline{k}}}{\mu} \cdot \underline{\nabla} p \right) = 0 \tag{1}$$

that is based on the substitution of the Darcy law into the continuity equation.

$\underline{\underline{k}}$ is the permeability tensor, p is the pressure and μ is the resin viscosity. The boundary conditions for eq. 1) are:

- fixed pressure or flow rate at the injection;
- atmospheric pressure along the flow front;
- no slippage at the mold surface.

Once the pressure distribution is known by solving the equation 1), the relationship between the filling time and the wet length can be obtained by integrating the Darcy law. In the case of one-dimensional flow (e.g.: x direction), the eq. 1) has an analytical solution, in particular the pressure field and the impregnation time t_{fill} are respectively:

Constant pressure p_0 — Constant volumetric flow rate Q_0

$$\begin{cases} p(x) = p_0 \cdot \left(1 - \frac{x}{L}\right) \\ t_{fill} = \frac{\mu \cdot L^2}{2 p_0 K_x} \end{cases} \qquad \begin{cases} p(x) = \frac{\mu \cdot Q_0}{A \cdot K_x} \cdot (L - x) \\ t_{fill} = \frac{A \cdot L}{Q_0} \end{cases} \tag{2}$$

where A is the cross sectional area of the gate and L is the total length that the resin has to impregnate.

Infiltration Problems

- High pressure injection processes are characterized by strength inertial forces, that produce movements or deformations of the preform during the moulding filling phase;
- The permeability of the mat is difficult to be estimated and it is affected by the stacking sequence;
- Track race.

These reasons make necessary the permeability measurement to characterize the reinforcing material.
It is important to create, too, an instrument to measure infiltration phase parameters (by the resin) of the carbon fibers layers.

Available Techniques

- CCD Cameras
- Metallic contacts
- Optic Fibers

Considerations:

- a CCD Camera can measures only on a plane, not in different plies, but in this work we have not examined this case;
- for use of CCD we need to use a transparent window, but this case occurs very rarely for the ordinary processes;
- metallic contacts are much intrusive and not reliable.

Using optic fibers sensors we may assess an innovative measuring technique having the following properties:

- Monitoring capability along whole thickness, not only on a single ply;
- Minimally intrusive;
- Useful as multi-parameter sensor, infiltration, cure degree and temperature measurement;
- May be multipoint;
- No sensible to EMI.

A possible configuration to set-up a sensorial system using optic fibers as sensors, is showed in Fig. 1, where some layers of carbon fibers prepreg are overlapped in a vacuum bag.

The reflection coefficient of the embedded Optic Fibers dramatically decreases when a fiber end is reached by the resin.

Traditional thermocouples are used as temperature sensors.

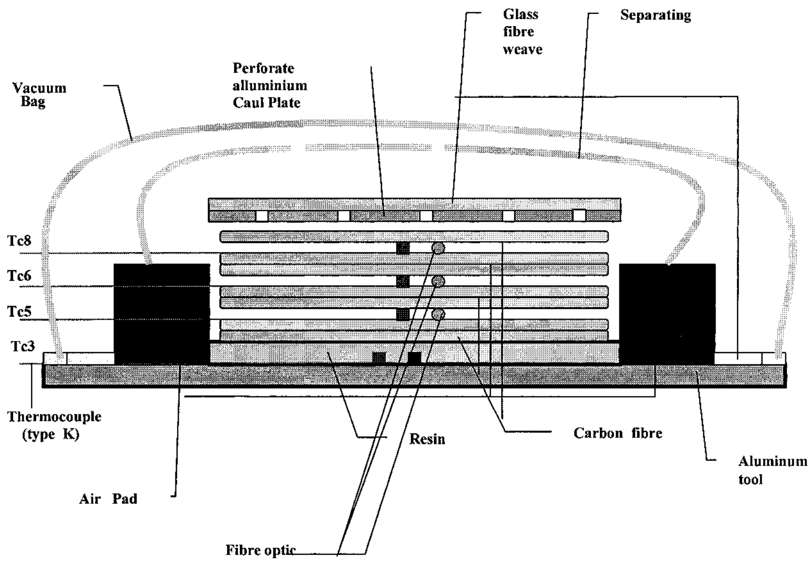
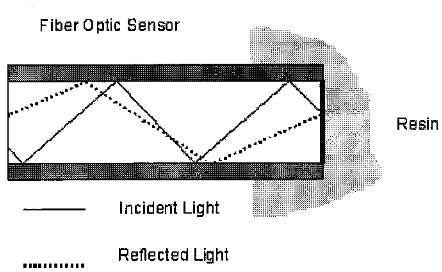


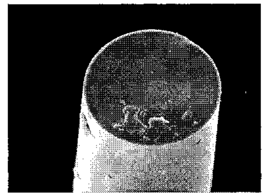
Figure 1. Fiber Optic are used to detect the preform wetting at the resin passage. The reflection coefficient dramatically decreases when fiber end is reached by the resin.

Description of the Optical Technique

COTS fiber optic components used in tele-communication [12] were experimentally tested with respect to a major category of defect: infiltration measure in a simulated environment.

Development tests were carried out using a preliminary optic scheme configuration, a "Refractometer", designed by CIRA/SMAS and CNR/IMCB, based on measure of the refraction index differences between the glass of the cut fiber and the surrounding environment [Fig. 2].





Fiber optic
sensing surface

Figure 2. Commercial Fiber Optic used as sensors.

The sensor is based on the Fresnel reflection principle: the reflection coefficient Γ is related to the difference between the resin refractive index n_m, the fiber optic refractive index n_f and the incidence angle.

In the case of a monomode fiber the reflection coefficient Γ at fiber end-resin interface can be expressed:

$$\Gamma = \left(\frac{n_f - n_m}{n_f + n_m} \right)^2 \tag{3}$$

Non intrusive fiber optic COTS assembled components, are reported in Fig. 3.

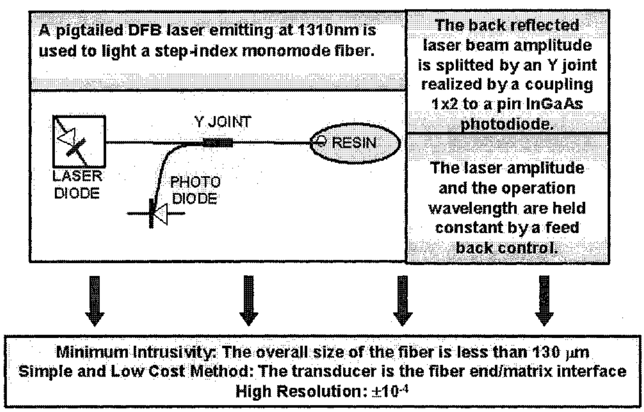


Figure 3. Working scheme of the sensor system.

The experimental set up involved in refractive index monitoring comprises:

1) A light source as a Pigtailed Laser Diode emitting at 1310 nm, 0.3mW power;

2) A step index monomode fiber SMR type;

3) A 3 dB Y joint realized by a coupling 1x2 fibers;

4) A photodiode.

The laser amplitude and the operation wavelength are held constant by a feedback control.
The laser is the light source for the system. The back reflected laser beam amplitude, splitted by the Y joint, impinges the photodiode. The reflection of the laser beam is due to the normal cut edge of the fiber embedded within the host.
In order to increase the signal to noise ratio, synchronous detection has been implemented. For this, the laser beam is externally amplitude modulated (1KHz) and the signal from the photodiode is filtered by the combination of a mixer and a low pass filter.
Usually, this function is implemented by using a Lock In Amplifier. Lock-in amplifier output voltage is proportional to the laser intensity reflected at the sensor interface that, in turn, is a function of the refractive index variation only. As a consequence the sensor output can be expressed as:

$$V = K\,P(\Gamma)\,R \tag{4}$$

where P is the incident laser power as a function of Γ the reflection coefficient at the fiber/host interface, K accounts for the lock-in amplifier gain, fiber loss and coupling coefficients, R is the current/light ratio, representing the responsivity of the photodiode.
In Fig. 4 is reported the logical scheme, including the necessary devices and a picture of the early sensor system [Fig. 5]:

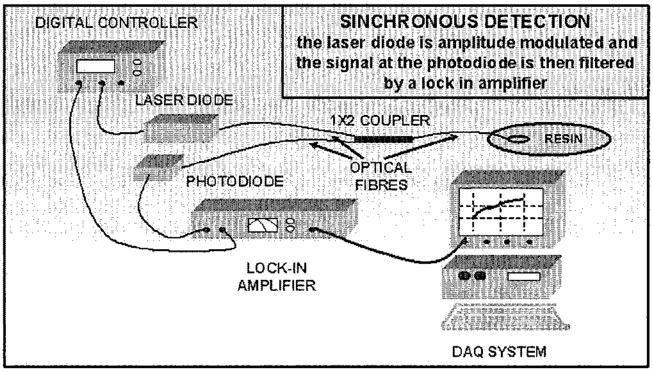


Figure 4. Logical devices scheme.

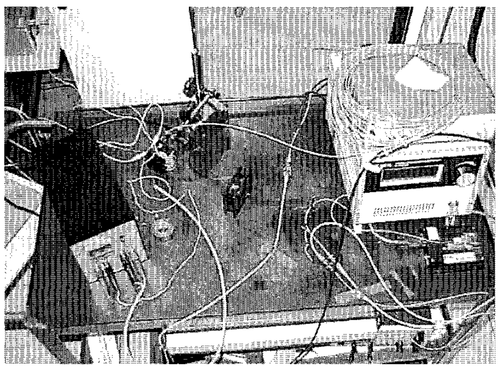


Figure 5. Early sensor system.

Starting by these experimental working set-up, we need to increase the number of measurement points with respect to a major category of defect: infiltration measure in a real environment.
To this aim, a multipoint optoelectronic system has been developed in CIRA/SMAS laboratories using an Optical Switch, where a single point of measure may be increased in more channels.
The principle of operation is similar to a simple Level Meter for a fuel tank: one by one, the sensors will be interrogated using the laser source. At the beginning, every sensor reads "on air" so that a big mismatching appears. So, an high reflection between the fiber and environment, produces a voltage Vr at the photodiode, represents the reference measure for that

specific sensor. When the sensor is covered by a substance having a refraction index similar to the fiber, a better matching between fiber and environment will result, so that the reflection effect falls down and so the voltage Vr does. If a threshold is fixed, the voltages before and after the occurrence, can be compared through a visual alarm on PC that will represent the passage of the substance on the face of the sensor.

A logical scheme of the "multipoint refractometer" is reported in Fig. 6.

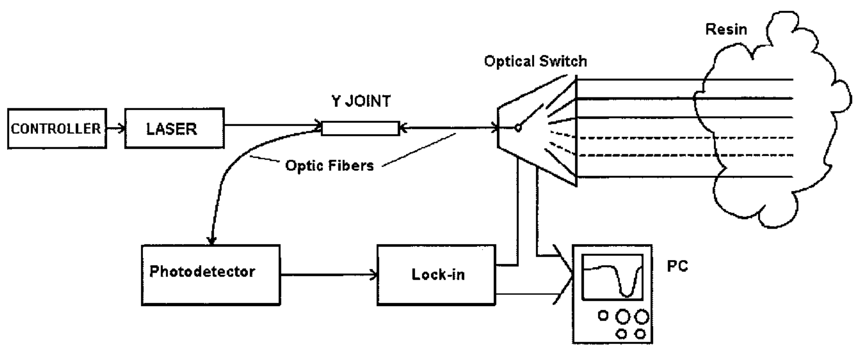


Figure 6. Multipoint Refractometer Fiber Optic Sensing System, based on a refractometer and an Optical Switch, with a specialized PC based Software.

Experimental Work

To monitor the resin infiltration in a mould during a VARTM process, was set-up and tested a sensor system, namely a "multipoint refractometer".

The light source for the tests was an E-TEK Laser controller and a MRV Communication 1310nm wave length laser source, directly monomode fiber coupled, an O/E Land Y fiber joint 50/50, a Thorlab DET410 photodiode, an Optical Switch LightTech LT1100 1x16 channels, a professional Cutting Machine and a "stripper" instrument for Optic Fibers.

Nine layers of EXCEL G1157 math with dry carbon fiber HTA5131 Tenax were overlapped on a metal plate, using a GS General Sealents Inc. rubber sealing to delimit a test area of 300 x 160mm. The polyester bag was placed on a metal frame to offer a certain repeatability to the tests. The vacuum was obtained with a vacuum pump.

The tests were oriented to provide indications on the design of the necessary set-up scheme, involving Hardware, Software and Experiences to solve some technology problems.

Nine optic fibers were lied on the metal plate [Fig. 7 and 11] of the simple mould.

Was built a software Plug-in in LabView environment capable to automatically acquire the tension Vr from the photodetector and at the same time to give the command pulse to the Optical Switch.

A fundamental step was the treatment and placing of the sensors on the mould, Fig. 8. The sensors consist of monomode optic fibers supplied from one side with an FC/APC connector, for the Optical Switch, and with another side having the nude fiber directly placed in test environment.

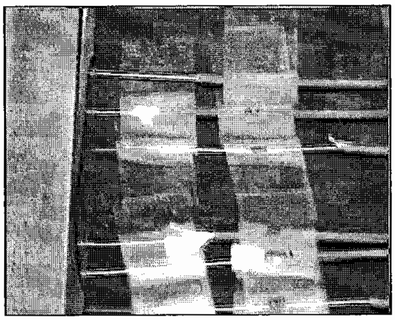


Figure 7. Placing optic fibers.

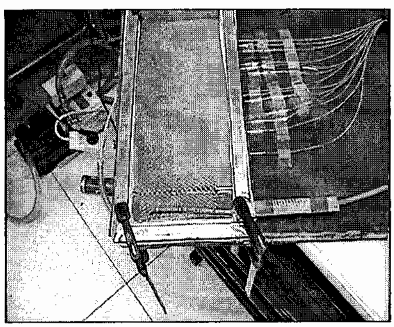


Figure 8. Experimental tests at Cira Labs; Fiber Optic sensors embedded in the mould.

Of course, if we cut the fiber, along glass crystalline lattice the reflection effect will be caused only from the difference between the index refraction of substance vs. fiber core, not from false reflections due to wrinkled surface, that can exhibit systematic errors during the measurement. The fiber core is the circular central zone of the fiber.

Another element on which focus our attention is the commutation speed of Optical Switch, Fig. 9, from a successive channel to another.

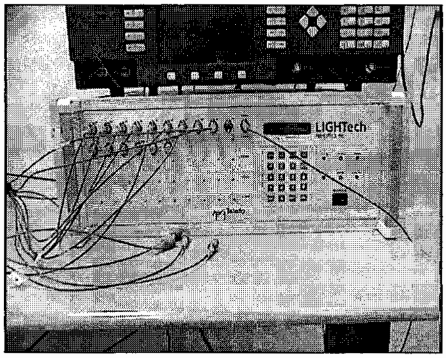


Figure 9. Optical Switch.

The switch time declared by the manufacturer was 30ms for successive channels, and 16 x 300ms for a commutation at a previous channel. It must take in account of all of these delays during the realization of management software. We can conclude that critical element of the chain is the Optical Switch because we have to wait that the MEMS (Micro Electro Mechanical Systems that are the core devices of Optical Switch) have the right rotation. We'll take in account of this delay in the Software.

Anyway, if this time is Δt, the minimum distance from a sensor to another must be at least

$$\Delta x \geq v_f \cdot \Delta t \tag{5}$$

where v_f is the advancement speed of the liquid forehead under observation.

Regarding the management software, the considerations made before suggest the requirement for the successful realization, due the fact that the software at the same time will collects and records data and it will control the switch commutation from a channel to another.

Depending from these considerations, the Software can be implemented in two different approaches:

1) SEQUENTIAL APPROACH. The control logic read the first channel and after an arrangement delay can records this data considering them as starter values. After this, the same channel may be interrogate until the current/recorded value ratio is less than the threshold value. At this moment is activated a LED to indicate the flux passage so that successive sensor interrogation is activated. Iterating all the operations, the cycle can terminates.

In this kind of solution the Software can't turn-off measurement LEDs, it can only turn-on them. So the working sequence may be only in one "direction". The order of all sensors must be sequential. If we have 1000 sensors we could know at which input correspond the fiber.

The starting value is recorded only when we interrogate the sensor and some variations will be take in account just during the lecture;

2) CYCLIC APPROACH. We can interrogate sensors cyclically one by one. During the first scanning we record the starting values and during next passages we control the actual and recorded values ratio vs. the threshold level.

In this kind of approach we don't take importance of the sensors order in the mould.

At every cycle of control would be necessary to re-define or adjust the starting values of each sensor, to avoid, as stated before, fluctuations that will create false readings.

In this case will be possible to use also the turning-off of the sensors, using a different threshold control, creating an histeresy revelator.

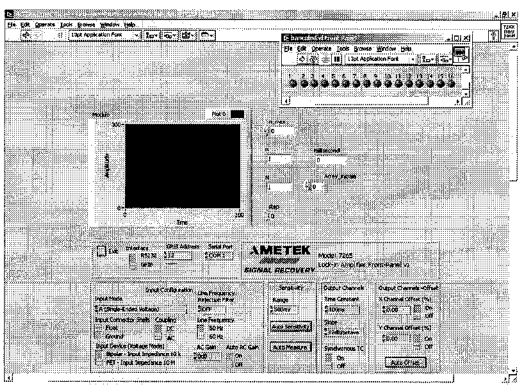


Figure 10. Screen Shot of Lab View Plug-in.

We choose the first approach, sequential interrogation, because of major measure time precision. In fact, for cyclic approach there is an uncertainty of 4 seconds, while in sequential approach the uncertainty is 10ms.

We made five repeated tests, placing the sensors in group of three. We considered a starting single point, as reference, placed on front of flux inlet, on the left side of Fig. 11.

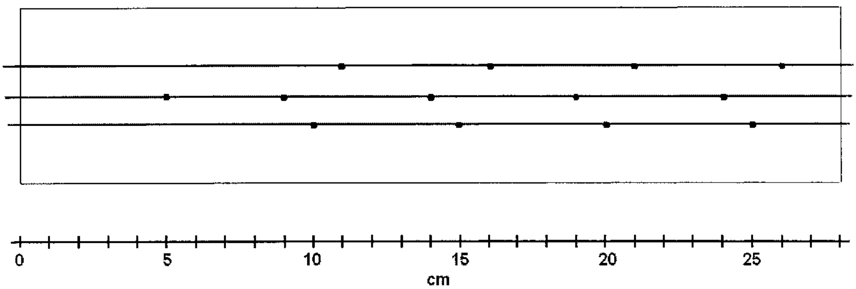


Figure 11. Sensors position scheme in the mould bottom.

We placed the carbon fibres plies normally to the mould, instead of optic fibres "sensors" that were placed longitudinally, so to exhibit the maximum time for the test; this involves changes on permeability variation.

Since we want to validate the procedure, we need to execute some repeated tests in the same repeated conditions. Using normal resin we could have to substitute the bag and the sealant in every test, cleaning perfectly the mould surfaces. Using pure water instead of resin we can have the possibility to use the same system: for this, we assume fluidity of resin and pure water are the same at a certain temperature.

In table 1 we summarize the infiltration time data measured by the sensors using pure water at 25°C.

Table 1. Infiltration time vs. sensors position.

Time (sec.) / X axes sensors pos. (cm.)	TEST 1	TEST 2	TEST 3	TEST 4	TEST 5
5	0	0	0	0	0
9	3,393	2,415	1,361	3,249	3,960
11	4,917	4,393	2,774		
14		6,668		9,382	9,406
16		8,690	5,770		
19		11,715	6,941		
21		13,668	10,472		
24		16,770			
26		19,696			

Some measures were missing because of oxidation of fiber termination, causing the not-activation of that sensor.

Diagramming tests 2 and 3, we have the curves in Fig. 12, where was made a polynomial interpolation.

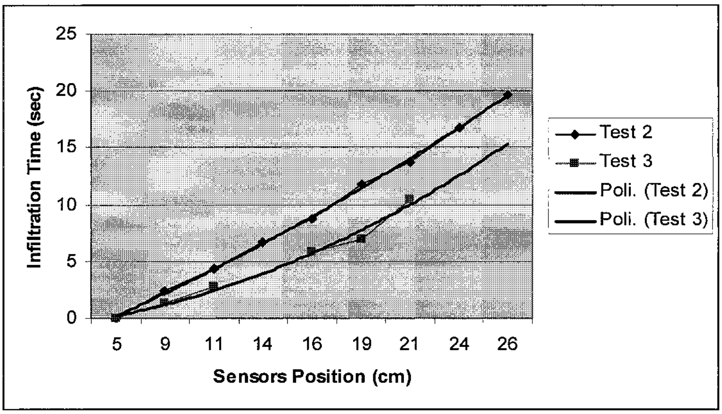


Figure 12. Tests 2 and 3 experimental data, with 2nd order polynomial interpolation.

As we can see, repeatability it is not present. This happen principally for the lack of repeated test conditions, e.g. manual manufacturing of preform, guides missing on the upper frame.

Inserting the results of test n. 2 in Matlab Statistics Toolbox, we have a fitting curve using "polytool" function, Fig. 13.

We interpolated data with two basic polynomial: one of the second order and another of third order, with a global confidence interval not-simultaneous of 95%.

Using a third order polynomial the total uncertainty on all coefficient increases, so the polynomial that better interpolates is that of second order. Coefficients of the curve are here reported for Test n. 2:

Quadratic term coefficient	Linear term coefficient
0.0144	0.6294

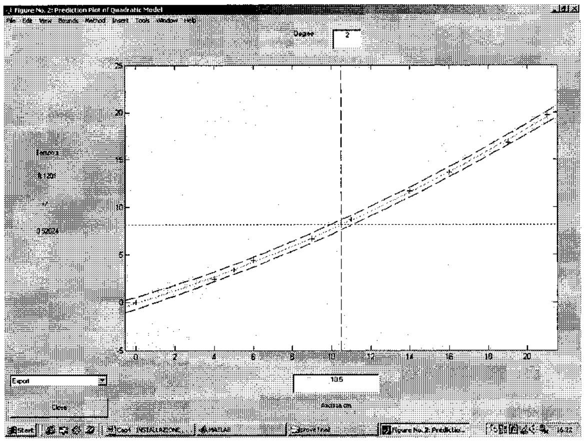


Figure 13. Fitting curve for test n. 2 (it is a boundary of 550ms uncertainty wrap).

We obtain that linear term is one order of magnitude higher than quadratic ones, thus preponderant. It means that the analytical model to validate, in these conditions, is not valid. Repeating the fitting curve operation for test n. 3, we have a preponderant quadratic term. This means that analytical model must have a linear term, too.

Conclusions

Part of the the results were obtained using Thermoanalytical Techniques. In fact Isothermal data output from a Fiber Optic Thermoset Cure monitoring Sensor [14] have been compared with calorimetric analysis of an epoxy based resin, showing correlation between sensor output and conversion advancement. The in-situ identification of the material status (conversion, temperature) during manufacturing is the best way to control the process operations with feedback capability.

In the present work a sensor system, aimed to monitor the resin infiltration in a mould during a VARTM process, was set-up and tested. The tests were oriented to provide indications on the design of the necessary set-up scheme, involving Hardware, Software and Experiences to solve some technology problems. After tests, a fitting curve was assessed for particular boundary conditions, maining the uncertainty time-range on infiltration time.

In VARTM we have to consider an high process parameters variability. We pointed out on a linear term makes evidence of these facts. Furthermore, this term exhibits variations so high as 100% ÷ 200%. These effects could be minimized but not eliminated. Some specific manufactured devices may suffer of lack of similar boundary conditions, e.g. an aircraft fuselage where square meters of carbon fibres are hand-placed or aided-machined-placed and cut. For these reasons we need to manage the infiltration process dynamically.

Our solution for this problem is to use the Multipoint Refractometer to individuate, since at an early stage, characteristic parameters of an infiltration process so that, consequently, may be adjusted the vacuum percentage and other critical process parameters. To this aim, could be possible to extract another parameter during the process, meaning a "Deviation" of modelized output (MOD Modelized Output Deviation). This parameter, obtained by use of the described Optical Technique, may improve the standard quality of the Production, analyzing and correcting in real time the manufacturing process and the efficiency of the moulded manufacturer devices.

In fact, the knowledge of the higher and lower extremes of the MOD Parameter variations, can suggest the opportunity to actuate principal parameters of the VARTM, pressure, temperature and capacity of the flow. In this way should be possible to obtain the best results in Composites Manufacturing, Fig. 14.

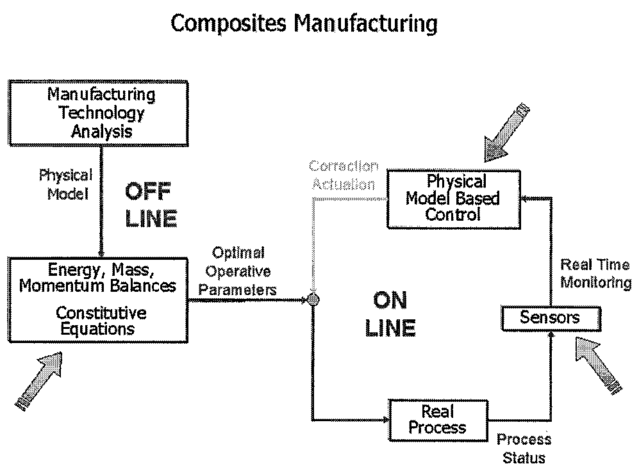


Figure 14. On-Line Composites Manufacturing block diagram.

Future Work

The demonstrated technique is very powerful, highly precise, but still full of challenges to study.

Numerical simulations have to be performed by a Finite Element software in which it is possible to insert different kind of models able to describe the behavior of the most important process variables (degree of cure, temperature, pressure, viscosity) and to investigate the control strategy to improve the efficiency of process manufacturing.

A preliminary version was developed and conceived at CNR-IMBC and CIRA labs as an open simulation platform, will be an object of study of a further work.

As further development, may be possible to deploy a self-learning system for the Control Logic, having fitness functions, so that the system may learn from the "history" some new situations without operator or process engineer participation. These are so-called "Smart Systems", able to take decisions also for never happened cases.

Many objectives are still to be achieved in order to put this technology to build a new class of instruments on the higher levels of the measuring systems. To handle this aim, the ultimate capacity of **FBG** (Fiber Bragg Grating) sensors in responding and measuring local temperature and high frequency dynamic deformation are currently studied. Going ahead in applying deformations at higher frequency, studying the linearity of these sensors, and modifying/updating the interrogating system, are considered among the most important issues to look after.

Furtherly, the characterization of the fiber reinforcements and advancing of matrix cure degree will be assessed through refraction index measurements approach.

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