THERMOSETTING MATRIX COMPOSITE CHARACTERIZATION AND CURE CONTROL VIA A SCALE MODEL MOULD

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SUMMARY: The main restriction for the widespread use of composites in many practical applications remains their prohibitive manufacturing costs. However, the development of new liquid composite moulding (LCM) processes brought an opportunity to significantly reduce these manufacturing costs. In order to successfully control composite manufacturing, an accurate material characterization and a close curing control of the composite are required. However, traditional characterization tools such as differential scanning calorimeter (DSC) didn't succeed really well in generating a macroscopic modeling that reflects the real resin-reinforcement behaviour, in an industrial context of LCM manufacturing. Moreover, this equipment is usually too costly and out of reach for small businesses that only wish to characterize their resin batches before production and to monitor their ageing. Thus this article described a low-cost characterization and control tool for the curing of composites which is robust and non-intrusive. This tool, using thermal heat flow sensors, is able to emulate, on a reduced scale model, the thermal phenomena which take place during the curing stage of composites, inside a real industrial mould. Furthermore, this characterization tool can provide good insights on resin and composite thermo-physical and thermo-kinetic properties. First of all, a description of the experimental set-up employed will be carried out. Then, the selected characterization and temperature control strategies will be detailed. Finally, a numerical validation of these strategies, using the finite volume method, will be presented.

KEYWORDS: Liquid Composite Moulding (LCM), cure control, PID, heat flow sensor, resin characterization, Differential Scanning Calorimetry (DSC)

INTRODUCTION

At the very beginning, thermosetting matrix composite materials were predominantly used in the military industry. Nowadays, they have an increasing contribution in many industrial fields such as aerospace, automotive, transportation, medical and recreational industries. Their good mechanical performances such as specific rigidity, high fatigue and corrosion resistance as well as their good acoustic and electrical insulation properties have contributed to their popular dissemination. The main restriction for their widespread use in many practical applications remains their prohibitive manufacturing costs. However, the development of new liquid composite moulding (LCM) processes brought an opportunity to significantly reduce manufacturing costs of composite materials in comparison with more traditional manufacturing processes such as autoclave curing for instance [1].

In order to successfully control composite manufacturing by LCM processes, a good initial knowledge of the resin properties and its thermo-kinetic modeling as well as an efficient temperature and polymerization control of the composite are absolutely necessary [2]. Unfortunately, traditional characterization tools such as differential scanning calorimeter (*DSC*) didn't succeed really well in generating a macroscopic modeling that reflects the real resin-reinforcement behaviour, in an industrial context of LCM manufacturing. Moreover, this equipment is usually too much expensive for small and medium-size companies that only wish to characterize their resin batches before manufacturing and to keep track of theirs thermo-physical properties.

Thus this article proposes an affordable characterization and control tool for the reticulation of composites which is robust and non-intrusive. This tool, based on the use of thermal heat flow sensors, succeeds in reproducing, on a reduced scale model, the thermal phenomena which take place during the curing stage of composites, at a larger scale. First of all, a description of the experimental setup employed will be carried out. Then, the selected characterization and temperature control strategies will be detailed. Finally, "pseudo-experimental" results of these strategies, using the finite volume method, will be presented.

EXPERIMENTAL SETUP

This low-cost characterization mould looks like a RTM mould. Firstly, it is made up of a kapton insulated flexible heater[1] from *Omega* (as illustrated in Fig.1). It is an etched foil design heater which is able to sustain up to 392°F (200°C). This 0.010" thick electrical heater can deliver 2.5, 5 or 10 Watts/inch^2 from a 115 Volts or a 28 Volts electrical supply.

Moreover, this characterization tool is provided of one or two rigid heat conductive flow sensors (HCFS) from *Thermoflux*[2] (see Fig.2). These heat flow/temperature combined sensors are designed for autoclaves, furnaces, ovens and flat tools. The typical industrial applications for this technology are *RTM* and resin infusion processes as well as autoclaves in the aircraft and food

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[1] http://www.omega.com/Heaters/pdf/Intro_flexheaters.pdf
[2] http://www.thermoflux.ch/fr/

industries. They can be placed inside a mould cavity, inside the mould wall itself (*RTM* light) or stick on a vacuum bag. These 900mm^2 heat flow sensors can sustain up to 446°F (230°C) and they are installed at 0.5mm from the wall, on both cavity sides (if it is necessary).

McMaster high temperature fibreglass pipe insulation and silicone backed laminated sheet aluminium are used to insulate thermally the radial mould walls, top and base. Accordingly, this insulation prevents lateral heat losses and keeps heat flow predominantly unidirectional. Also, machined 6061-T6 aluminum blocks are integrated in this mould. The resin receiver (reactor cavity) is machined directly in one of those aluminium blocks. Furthermore, a *McMaster* direct-acting solenoid valve is employed to evacuate the surplus heat from the cavity.
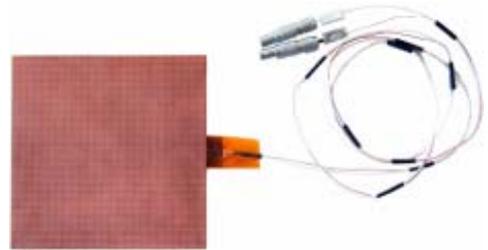

Fig.1 *Omega* Insulated Flexible Heater.

Fig.2 Tfx-161 Rigid *Thermoflux* Sensor.

This reduced scale mould offers two configuration possibilities for characterization as illustrated in Fig. 3 which depends on characterization tests performed by users.
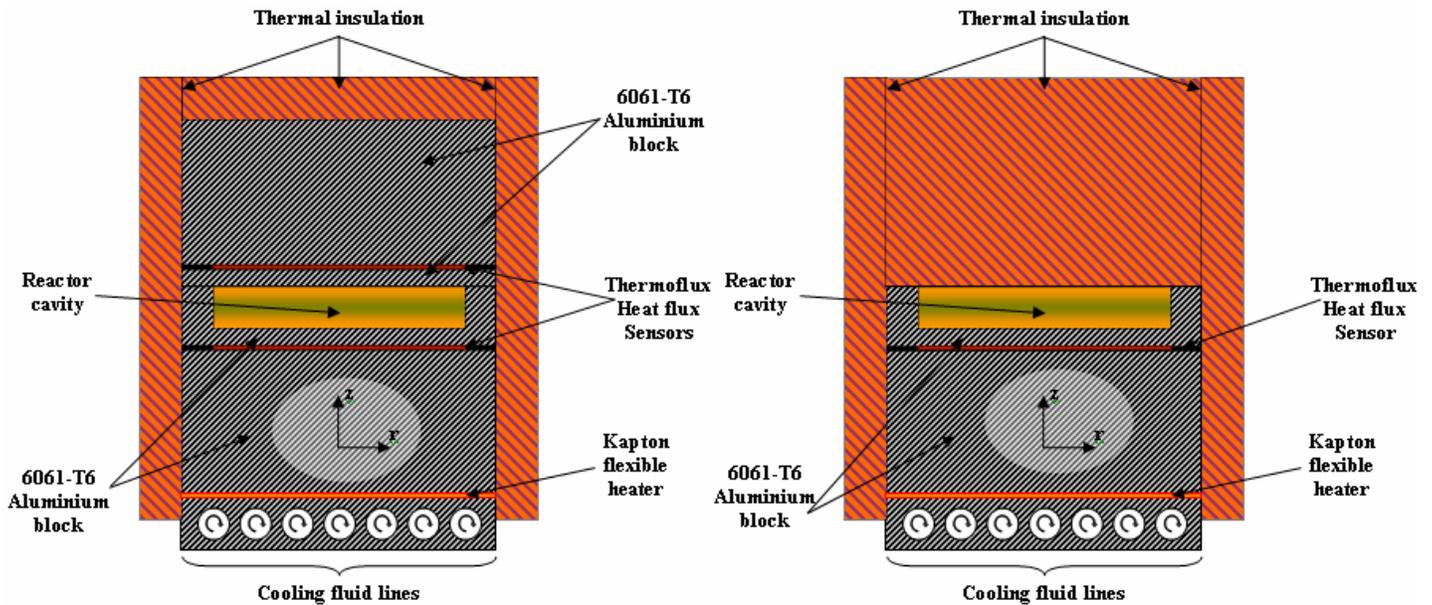

Fig. 3 2D cut schematics of bidirectional and unidirectional characterization mould.

CHARACTERIZATION STRATEGIES

Thermal Conductivity

The two heat flow sensors configuration (see Fig. 3) allows users to carry out thermal conductivity measurements on non reactive resin and composite as with a conductivity meter. This thermo-physical property is a really important parameter to perform full scale mould design as well as non isothermal filling and curing simulations. Some metrology standards such as ASTM E1225-04 already specify the critical dimensions (thickness, width, etc.) for the analyzed samples. In this configuration, user imposes a constant heat flow with the electrical heater. Afterwards, when steady-state is reached inside the cavity, heat flow and temperature measurements on each side of this cavity are recorded. With these values at equilibrium, an average heat flow and an average temperature gradient are computed. Therefore, the material thermal conductivity can be approximated quite well with the Fourier's law (1) and a harmonic mean formulation of thermal conductivities.

$$\underbrace{\rho c_p \frac{\partial T}{\partial t}}_{\equiv 0} = \vec{\nabla} \bullet \vec{q} + \underbrace{\dot{r}}_{\equiv 0}$$

$$\vec{q} = -\lambda \ \overrightarrow{\nabla T} \rightarrow \lambda_{approx} \approx \left. {}^{-q_{avg}} \middle/ \left(\partial T / \partial z \right)_{avg} \right. \tag{1}$$

$$q_{avg} = \frac{q_{upstream} + q_{downstream}}{2}; \ \ \partial T / \partial z \Big|_{avg} = \frac{T_{upstream} + T_{downstream}}{\Delta z_{sensor \ gap}}$$

Kinetic and Enthalpy of Reaction

The one heat flow sensor configuration (see Fig.3) allows users to realize resin kinetic measurements on reactive resin as with a reaction calorimeter. In this configuration, user sets a desired temperature at the lower mould cavity wall. Later on, when resin cross-links in the cavity, heat generated by this exothermic reaction is read by the heat flow sensor, smoothed by a numerical filter and recorded in a database. Then, an averaged bell-shaped curve of these heat flow values can be plot and numerically integrated to get the enthalpy heat of reaction and an averaged degree of cure trend of the monitored resin.

CONTROL STRATEGIES

An accurate resin kinetic characterization needs a close control of the mould temperature. To do so, computer-assisted control strategies, using readings the *Thermoflux* sensors, were investigated. They were all implemented in C++ object-oriented language and validated on an Intel® Xeon™ PC workstation (3.20 GHz CPU). Among many strategies, three of them were selected to control mould heating and cooling with an electrical heater and a low-cost solenoid valve respectively. These algorithms are the Proportional-Integral-Derivative (*PID*) and the fuzzy logic controllers for the heater and the hysteresis controller for the valve.

PID Temperature Controller

A traditional *PID* controller with an integrated reset anti-windup has been implemented [3-5]. The error integral component has been computed with a conventional Simpson rule and the error derivative term was approximated with a backward first-order finite difference. At first, it was highly recommended to just use proportional and integral term for temperature control because noisy signals such as heat flow and temperature measurements could be seriously amplified by the derivative term and then generate a really noisy command signal for the electrical heater.

Fuzzy Logic Temperature Controller

A fuzzy logic controller was implemented to maintain the temperature of the lower mould cavity wall at a specify setpoint [2, 6-9]. This fuzzy logic controller is using two distinctive inputs to compute a new heat power command sent to the electrical heater. These two inputs are the current error between measured temperature and desired temperature setpoint and the backward numerical first derivative of this error. This fuzzy logic controller was using:
- classical triangle membership functions;
- detail decision rule base (2D fuzzy associative matrix);
- centroid defuzzification method (COG);
- max-min inference engine;
- Union Rule Configuration method (URC) to prevent the well-known "combinatorial explosion problem" [10].

Hysteresis Controller

Concerning control of the cooling system, the algorithm selection was quite straightforward due to the binary mechanism of the on-off solenoid valve. A simple bang-bang controller with a hysteresis band was chosen.

NUMERICAL VALIDATION WITH FINITE VOLUME METHOD

A traditional finite volume model [11, 12] of the characterization mould has been developed to predict and emulate its thermal response and to numerically validate the previous selected control algorithms. This 30-nodes model was solved using the Crank-Nicolson method. This numerical model is one-dimensional because of the perfect radial insulation hypothesis. Moreover, resin and fiber thermo-physical properties are considered independent of the temperature and the degree of cure. Also, the epoxy resin degree of cure evolution, modeled by the Kamal-Sourour kinetic model [13, 14], is updated with a classical fourth-order Runge-Kutta method. Finally, thermal contact resistances (steady-state) and impedances (transient) at material interfaces are neglected in first approximation.

For the sake of technical realism, 0.25% additive white Gaussian noise (AWGN) was added to the simulation result values obtained at virtual sensor locations. Furthermore, these noisy numerical data were truncated according to the binary resolution of analog-digital converter used (ADC) which is 8 bits. From these data, heat flow and temperature were computed at the sensor

locations and more sophisticated calculations were performed such as heat balance over the reactor cavity, resin kinetic modeling, thermal conductivity and volumetric heat capacity approximations and so on.

The results of epoxy resin thermal conductivity characterization are given in Fig. 4. The thermal conductivity has been computed thanks to the harmonic mean formulation [11], knowing already the geometry of the cavity, the distance between the two sensors and the thermal conductivity of aluminum.
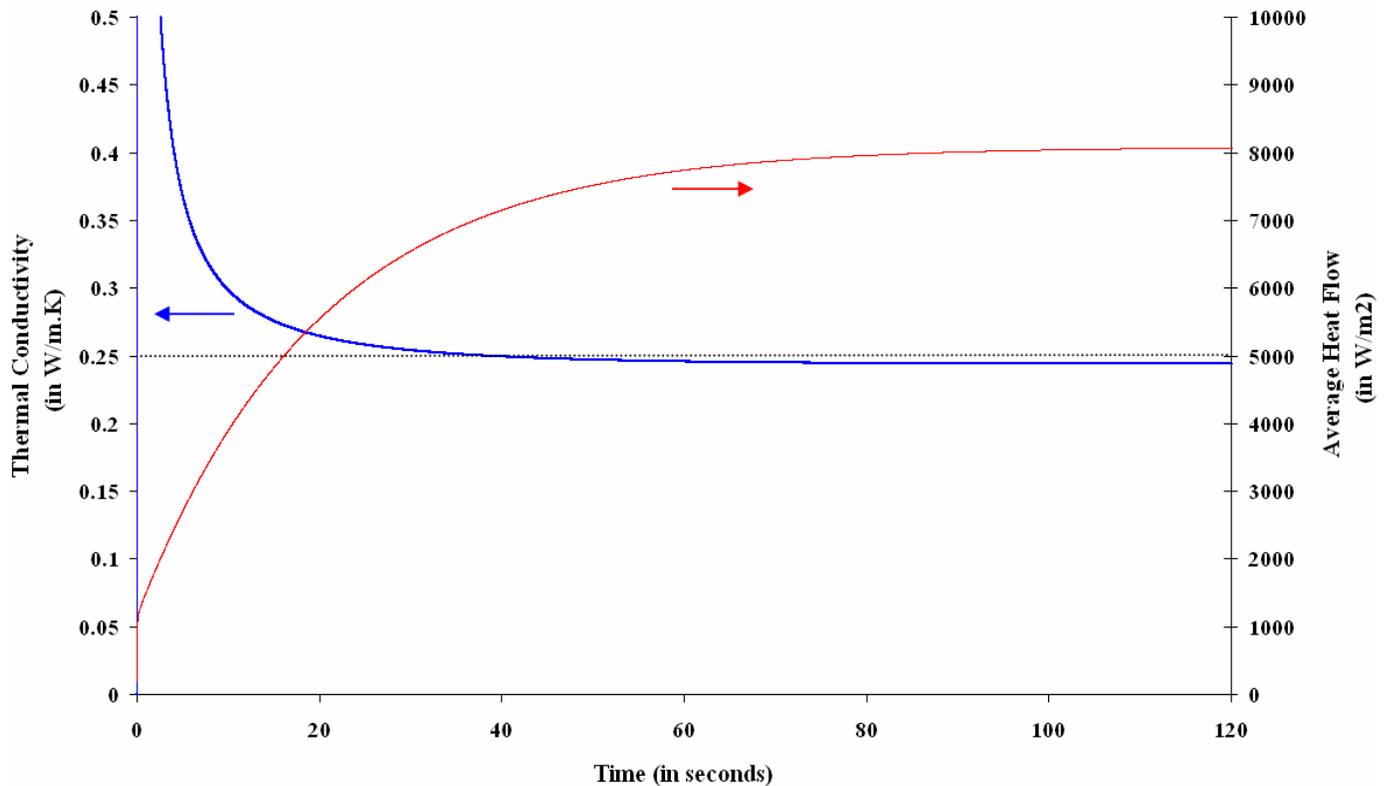

Fig. 4 Resin thermal conductivity virtual characterization.

Fig. 5 shows the results of the enthalpy of reaction characterized with a *PID* controller. This polymerization heat analysis has been performed after a 371 degrees Kelvin heat set. The lower cavity wall of the mould was maintained at a 373 degrees Kelvin isotherm throughout the exothermic reaction monitoring. The general trend of this enthalpy of reaction curve is good (polynomial fitting) and is approaching the averaged reaction heat evolution inside the cavity (See Fig.5). However, this heat flow signal is discontinuous and highly disrupted by the cooling action of the on-off valve. So, replacing this on-off valve by a proportional one that would be operated by a *PID* controller should be a better cooling solution and still a low-cost improvement. A step further on temperature control would be operating this characterization mould like industrial one. For instance, a coolant/heating medium (water, oil, etc.) could be handled by a heat exchanger to control the temperature of this scale model mould.
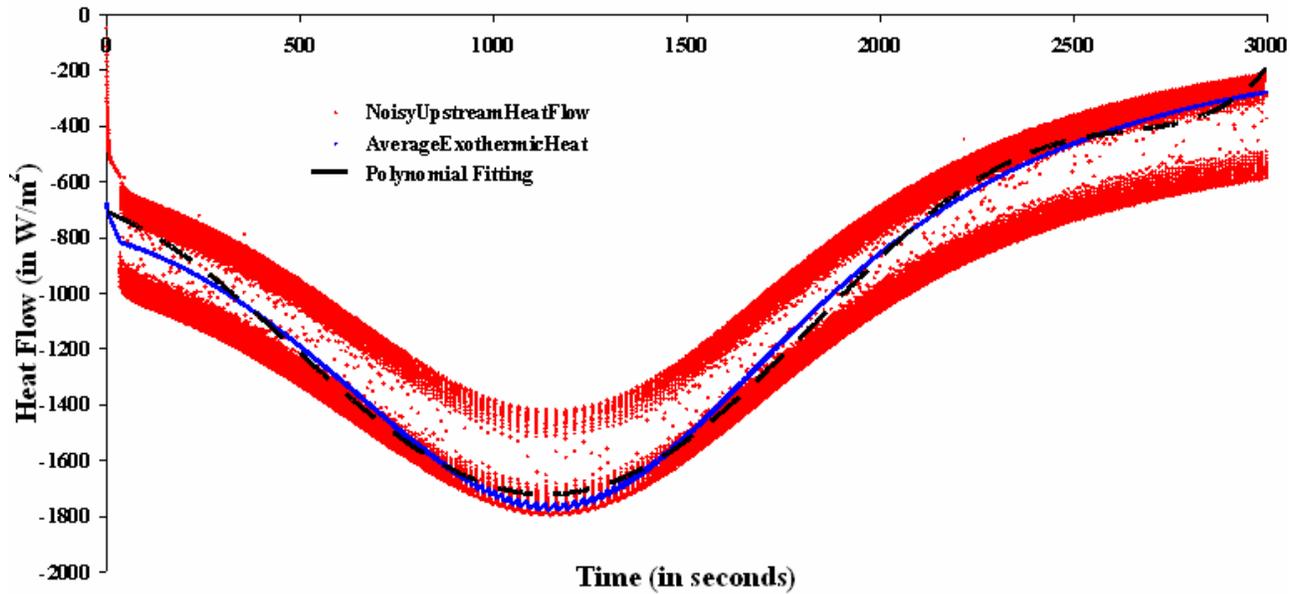
Fig. 5 Enthalpy heat of reaction virtual characterization.

CONCLUSIONS

This low-cost characterization tool based on heat flow sensors, simple cooling/heating system, *PID* and fuzzy logic control algorithms seems to be promising. This reduced scale mould can provide to users a good insight on the thermal phenomena that take place inside an industrial mould as well as help them to perform mould design optimization, process parameter improvements and control strategy adjustments. Moreover, kinetic modeling and thermo-physical properties characterization can inform operators about the resin ageing evolution. However, there are still some possible upgrades to apply on this mould and on the numerical model such as:

- using a proportional solenoid valve rather than a on-off valve as a cooling system;
- optimizing *PID* tuning parameters and fuzzy decision rule base
- filtering data with a more appropriate numerical filter
- modeling thermal contact resistances or impedances with respect to the situation.

In the next months, characterization strategies for thermal diffusivity and specific heat will be elaborated with this reduced scale mould. Furthermore, numerical validations will be gradually substituted by experimental and manufacturing validations. Then the real performance of this low-cost tool will be evaluated.

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