CURE PROCESS MODELING AND CHARACTERIZATION OF COMPOSITES USING IN-SITU DIELECTRIC AND FIBER OPTIC SENSOR MONITORING

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ABSTRACT

Liquid Composite Molding (LCM) techniques including the Resin Transfer Molding (RTM) and Vacuum Assisted Resin Transfer Molding (VARTM) are gaining significant importance for fabricating aerospace and automotive composite parts, owing to the low investment costs. During the curing process, the resin undergoes a property change due to cross-linking of polymers, where it transitions from the liquid state to the solid state. Further, during the cooling process, there is a change in the glass transition temperature, resulting in residual stress and strains. The residual strain and deformations accumulated during the curing of the resin at high temperatures result in significant challenges to the final part shape and performance of the composite structure. This research presents a thermo-chemo-mechanical curing model for liquid composite molding processes, which is validated with in-situ sensor monitoring data including viscosity, temperature, and degree of cure using dielectric sensors, and the distribution of induced strains during the curing process using distributed optical sensors. The viscoelastic curing model developed in ABAQUS constitutes of the resin cure kinetics, viscoelastic resin properties, and thermal and stress analysis components. A case study is performed for an angle bracket, where the resulting cure-induced stress deformation is observed and validated, and the spring-in angle of the bracket is predicted.

Keywords: Liquid Composite Molding (LCM), Vacuum Assisted Resin Transfer Molding (VARTM), Curing Model, Dielectric Sensors, Fiber Optic Sensors

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INTRODUCTION

As an alternative to the traditional high-cost autoclave manufacturing process, many manufacturers use the liquid composite molding (LCM), which includes the Resin Transfer Molding (RTM) and Vacuum Assisted Resin Transfer Molding (VARTM) processes. It offers manufacturing complex and high-quality parts in shorter time with low operating costs. There has been tremendous amount of research work done on LCM for the past 3 decades, however, it is not uncommon for manufacturers to observe issues in manufacturing, such as dry spots, resin rich and resin poor regions, as well as poor curing in the composite structure. As these LCM processes involve the liquid resin entering the dry preform material, it is important to understand both the fabric structural properties such as permeability, draping behavior and tow properties, as well as resin properties such as viscosity, and temperature and wetting process conditions.

There are different types of real-time process monitoring sensors, available for different manufacturing processes. For the traditional autoclave processes, thermocouples are used to monitor and control the cure cycle. For out-of-autoclave processes such as RTM, optical fiber sensors, dielectric sensors, and pressure and flow rate monitoring devices are used monitor temperature, strain, degree of cure, and flow speed of resin. Optical fiber sensors include fiber Bragg grating to provide continuous measurements of strain and temperature, and 3D-printed resistive carbon fiber sensors and carbon coated piezoresistive fiber sensors for monitoring resin frontal flows.

IR-based thermographic vision-camera systems for monitoring temperature are often used during AFP manufacturing process, and the sensor data can be used to model the physical phenomena in the layup during manufacturing. In the case of AFP, previous research has demonstrated that there is up to a 25% difference in temperature between the top and the bottom ply, when the plies are laid one above the other. As in additive manufacturing, since the process itself is highly digital in nature, having more sensor data aids in process modeling. Hence, with the use of all these sensors, and the real-time monitoring data collected to form a digital thread, the manufacturing process models can be utilized as digital twins to have real-time information to make accurate predictions on the composite part.

Among the various methods for in-situ monitoring of the manufacturing process that exist, dielectric cure monitoring is the most mature, where a sinusoidal (AC) or constant (DC) voltage, in the present case, is applied to two electrodes, which combined act as a single sensor. The material of interest is placed in contact between these two electrodes through the sensor, and dielectric properties such as electrical resistance are measured, which provide further information on the material state [1]. It has been demonstrated that the electrical resistance of a resin correlates to its viscosity and Glass Transition Temperature (Tg). Synthesites [2] has developed a proprietary technology for the online estimation of the development of the Tg and/or the degree of cure based on this observation and experimental data of several resins. This technology is valid not only in the laboratory but also during composites production. This method has already been successfully applied to thermoset-based composites manufacturing in automotive, wind turbine blades, and aerospace applications, but it has only been employed in lab-scale trials with reactive thermoplastics. The resistivity of a resin was directly related to its viscosity, while the Tg can be determined online, according to a well-established hypothesis that has been thoroughly confirmed through the Online Resin State (ORS) module [1,3,4].

Another well-known in-situ monitoring measurement technique is fiber optic sensing, where the transmission wavelength is separated into measurement and reference paths by a tunable laser. The input wavelength, travels into a measurement path and is subsequently received back from the reflection. The temperature or strain is calculated based on the change in the wavelength of the transmitted and reflected signals in the fiber optic sensors [5,6].

CURE PROCESS MODELING

A multi-physics model was developed that covers resin cure kinetics, chemical shrinkage, and thermal loading as part of the curing model. The initial stage in the model is a heat transfer analysis, which determines the temperature and cure history inside the composite based on the cure cycle and internal heat generation from cross-linking of the resin. The temperature and cure history are subsequently used in the second stage of the model to map the cure-dependent constitutive relations in the stress analysis. In addition, an algorithm for calculating the spring-in angle for an L-Beam composite part was created. This modeling approach is further explained in detail in other publications [7–11].

During the curing process, heat is applied to the surface of the composite to accelerate the cross-linking process, which in turn generates additional heat. The temperature history can be solved using the heat transfer governing equation including the heat source term based on the resin cure kinetics as

$$\rho c_P \frac{\partial T}{\partial t} = \sum_{n=1}^{3} \frac{\partial}{\partial x_n}\left(k_n \frac{\partial T}{\partial x_n}\right) + \rho_m\left(1 - V_f\right) H_r \frac{d\varphi}{dt} \tag{1}$$

The reaction rate $d\phi/dt$ is a function of temperature and Degree of Cure (DOC). For an epoxy resin such as EPON 862/W, the cure kinetics relation can be expressed using the Kamal-Sourour autocatalytic model as [12]

$$\frac{d\varphi}{dt} = \left(A_1\, exp\left(-\frac{E_1}{RT}\right) + A_2\, exp\left(-\frac{E_2}{RT}\right)\varphi^m\right)(1-\varphi)^n \tag{2}$$

The cure kinetics parameters are determined by measuring the resin heat generation response under various temperatures using a Differential Scanning Calorimeter (DSC) [16].

Following the heat transfer analysis, the stress analysis is carried out to obtain the resulting stress–deformation response based on the temperature and curing history. Since the resin shows a thermo-viscoelastic response during curing, the composite cure-dependent constitutive relation becomes

$$\sigma(t) = \int_0^t C_r\,(t-s)\frac{d}{ds}\left(\varepsilon - \varepsilon^{\mathrm{th}} - \varepsilon^{\mathrm{ch}}\right)ds \tag{3}$$

The stress analysis is simplified by assuming that the composite relaxation modulus E can be decomposed into instantaneous and time-dependent parts as [14]

$$E(t) = E_a + E_m e^{-t/\tau} \tag{4}$$

When the resin is in the rubbery phase, the relaxation time is small, and the relaxation modulus can be approximated as

$$E_R = E_a \tag{5}$$

When the resin is in the glassy phase, the relaxation time is large, and the relaxation modulus can be approximated as

$$E_G = E_a + E_m \tag{6}$$

As a result, the cure-dependent composite constitutive relation can be simplified as

$$\sigma(t) = \int_0^t C_i \frac{\partial}{\partial t}\left(\varepsilon - \varepsilon^{\mathrm{th}} - \varepsilon^{\mathrm{ch}}\right) dt \tag{7}$$

$$\Delta\sigma = C_i(\Delta\varepsilon - \Delta\varepsilon^{\mathrm{th}} - \Delta\varepsilon^{\mathrm{ch}}) \tag{8}$$

where $i = R, G$ represent the composite properties at the rubbery and glassy phases, respectively.

Since the composite response is dependent on the curing state of the resin, micromechanics analysis must be employed to determine the effective thermo-chemo-mechanical properties of the composite. To efficiently model the curing response of a composite structure, the micromechanics model should be coupled with a homogenized macroscale model through a multi-scale modeling approach as discussed further.

A multi-scale modeling approach was formulated, as schematically shown in Figure 1 to characterize the effect of resin curing response on the resulting residual stress development in the composite. When the multi-scale modeling framework is synthesized in an FE setting, the composite part is homogenized as an orthotropic solid at the macroscale. The effective composite constitutive relation is implemented at each integration point of the macroscale model, which is determined through a mesoscale model that is represented as a Representative Volume Element (RVE) of stacked unidirectional or textile laminae. The RVE thermo-chemo-mechanical properties can be determined either through analytical approaches [15] or FE analysis [16]. The constitutive relations of a unidirectional lamina in a multi-directional laminate or a single fiber tow in a textile ply are further determined through a subscale micromechanics model at the individual fiber and matrix level. It is worth mentioning that the proposed modeling framework can be applied to both textile and multidirectional laminates. The mesoscale model provides the possibility to model several stacked layers together, which can save significant computational costs for modeling thick composite structures. The mesoscale model is used to determine the constitutive relation of a plain weave textile ply by modeling it as a cross-ply laminate.

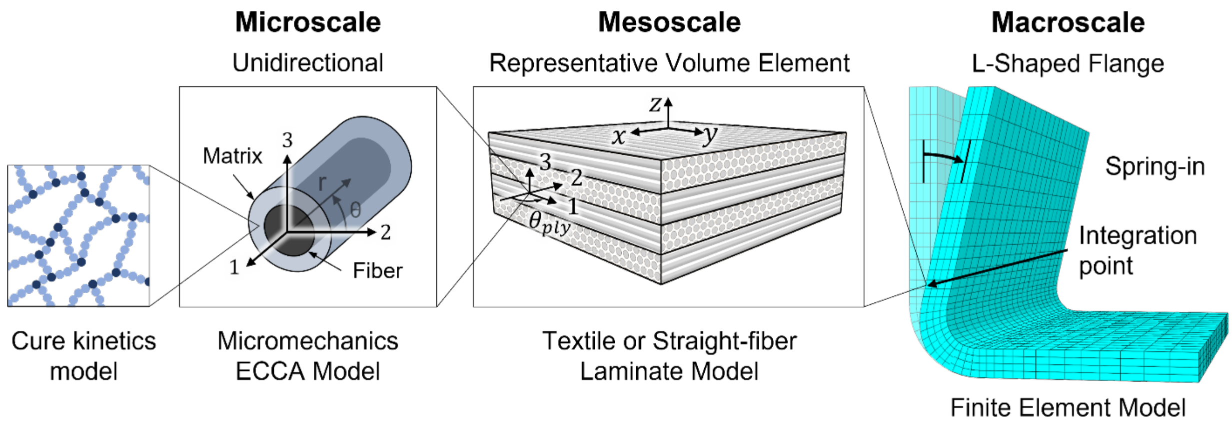

Figure 1. Illustration of the macro-meso-micro-scale model used in this study.

In the heat transfer analysis, the resin-specific heat and thermal conductivity are dependent on the DOC and temperature. It is assumed that the two thermal properties are linearly proportional to the temperature. Thus, based on the cure kinetics model defined in Eq. (2), the shear modulus of resin at a given temperature and DOC can be interpolated as, respectively,

$$G_r(t,T,\phi) = G_\infty(\phi) + \sum G_i(\phi) exp\,(-\frac{t}{\tau_i(\phi)/\alpha_T}) \tag{1}$$

Experimental results for the specific heat of epoxy during isothermal cure are given in [17] As the resin cures, it transitions from a liquid to the solid state under an elevated temperature, and the glass transition temperature grows monotonically with the DOC, which can be described using the DiBenedetto Equation [18]

$$\frac{T_g(\phi) - T_g^0}{T_g^\infty - T_g^0} = \frac{\lambda\phi}{1-(1-\lambda)\phi} \tag{10}$$

Detailed discussion regarding the experimental characterization of the cure-dependent glass transition temperature of the EPON 862/W epoxy resin system is given in [13]

For a thermoset resin, the curing process can be divided into three regions, as illustrated in Figure 2. Gelation occurs at a defined point in the curing reaction, after which the resin can carry loads. Before this point (Region A), the resin is in the liquid phase and stress accumulation is negligible. After gelation, the resin transitions to the solid phase, and the thermal and chemical strains should be considered in the constitutive relations. When the curing temperature is above the resin glass transition temperature (Region B), the resin is at a rubbery state and it is assumed that the resin modulus is close to zero, and its Poisson's ratio is close to 0.5 since a gel can be approximated as an incompressible material. Based on the DiBenedetto Equation, the glass transition temperature of the resin grows monotonically with the DOC. Once the glass transition temperature of the resin exceeds the curing temperature, vitrification occurs and the resin transitions from the rubbery state to the glassy state (Region C), followed by an increase in the elastic modulus and decrease in the Poisson's ratio.

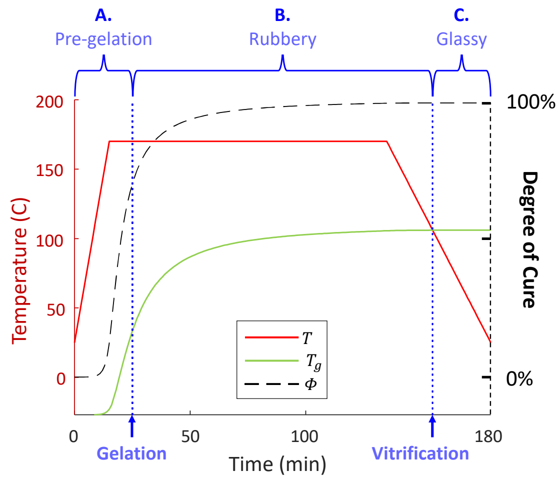

Figure 2: Temperature and cure diagram.

The goal of the micromechanics model is to determine the effective lamina (or fiber tow) thermo-chemo-mechanical properties based on the constituent fiber and resin properties. These effective properties will be used as inputs to the mesoscale model. An efficient analytical method based on the Extended Concentric Cylinder Assemblage (ECCA) micromechanics models [19] is adopted to compute the effective lamina properties.

The effective moduli of the unidirectional lamina are computed based on the fiber and resin properties through the ECCA model, in which the unidirectional composite is represented as a fiber–matrix concentric pair. Closed-form solutions can be obtained for the five independent constants used to characterize the transversely isotropic lamina, including the longitudinal Young's modulus, Poisson's ratio, shear modulus, transverse shear modulus, and bulk modulus. These effective properties are derived by combining the classical Concentric Cylinder Assemblage (CCA) model and the Generalized Self-Consistent Method (GSCM). In this study, the ECCA micromechanics model is also employed to compute the effective CTEs and coefficients of chemical shrinkage of the lamina by solving the elasticity problem of a fiber–matrix concentric pair subjected to temperature change and resin shrinkage.

Proceeding further a mesoscale model is developed to determine the effective laminate RVE thermo-chemo-mechanical properties based on the lamina properties. The unidirectional properties are passed up to the mesoscale. For the laminate composite, the unidirectional properties are transformed to the RVE coordinates based on the orientation of each ply, then the stacked plies are homogenized by an extended Classical Laminated Plate Theory (CLPT)-based approach to obtain the full 3D properties. This approach is particularly useful for modeling textile composites, which can be homogenized as a multi-directional laminate.

IN-SITU SENSOR MONITORING

In this research work, plain weave dry woven fabric carbon fiber T300 is used with EPON 862/W resin system. The infusion is carried out at 121°C and curing is performed at 177°C for 2 hours as per the manufacturer recommended cure cycle. 8 Plies of fabric were used, and two separate experiments were conducted, individually for the dielectric cure process monitoring and the distributed optical fiber sensing.

Dielectric Process Monitoring

The typical VARTM setup for dielectric process monitoring is shown in Figure 3. Four Resin Arrival and Temperature sensors, also called flow sensors, are connected to the OptiFlow equipment, and the two Cure sensors are connected to the two OptiMold units which are provided by Synthesites [2]. Through this experiment, the resin arrival is monitored at the sensor locations and the online estimation of Tg is obtained from the resistance and temperature measurements.

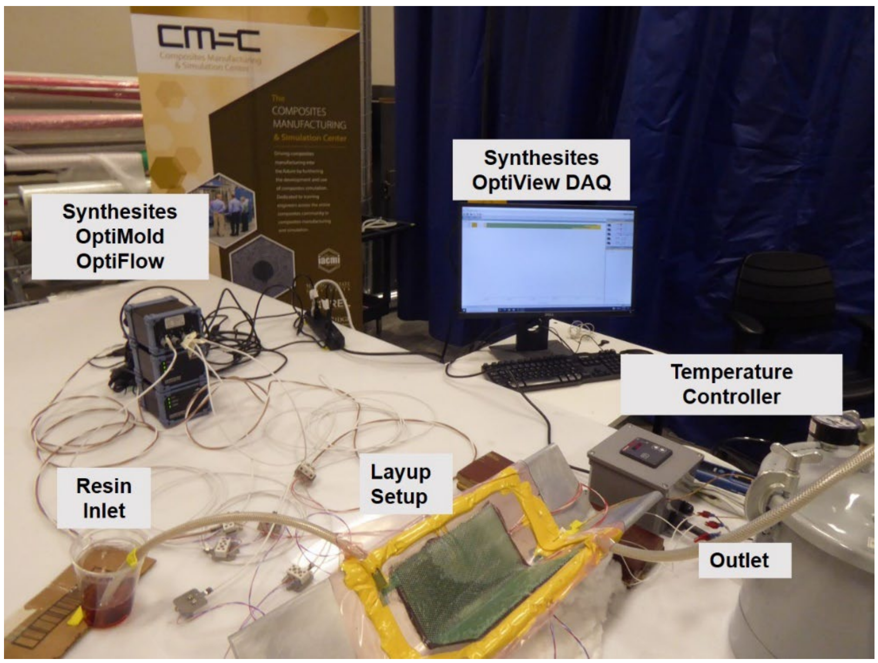

Figure 3: VARTM Experimental Setup for Dielectric Cure Process Monitoring

In this research experiment, the sensors are placed outside of the top and bottom peel-plies to facilitate removal after the part is manufactured and eliminate interference with the composite structure being manufactured. On both the top and bottom of the 8-layer fabric layup, peel ply is placed. Flow sensor F1, and Cure sensor C1 are placed on the bottom layer, and flow sensor F2 is placed on the top layer, at the resin inlet end. At the other end where the vacuum outlet exists, the flow sensor F3 is placed on the bottom layer, and another flow sensor F4 and cure sensor C2 are placed on the top layer. With this placement of the dielectric cure monitoring sensors, the flow and cure are monitored at critical locations, and the setup is shown in Figure 4.

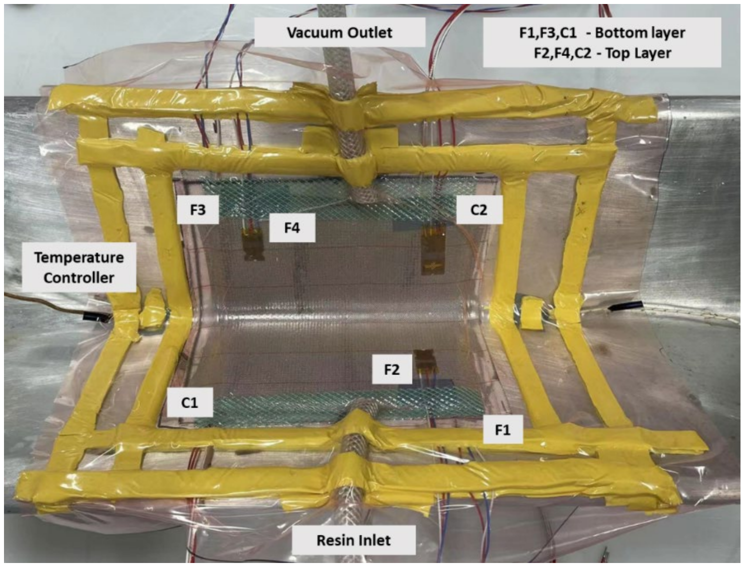

Figure 4: Flow (F) and Cure (C) Dielectric Sensor Placement during VARTM

Distributed Optical Fiber – Strain Sensing

As discussed earlier, distributed fiber optic sensing has been used in composites manufacturing and structural health monitoring. In this research experiment, the High-Definition strain sensors provided by LUNA Innovations Inc were used, with the ODISI-B Interrogator equipment using the OFDR. The fiber optic sensors were placed in each layer of the composite structure as shown in Figure 5. The segment A-B was placed between the 2nd and 3rd ply, the segment C-D was placed between the 3rd and the 4th ply, and similarly, the final segment of K-L was placed between the 7th and 8th ply. The data acquisition rate was set to 23.8 Hz with a gauge length of 1.3mm. The fiber optic sensors need to be handled very carefully during the layup and the bagging for the VARTM process, to avoid any sensor breaks and data failure.

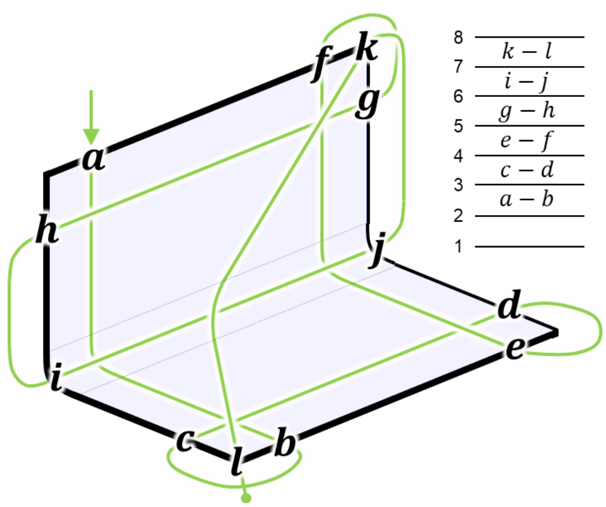

Figure 5: Schematic of the Fiber Optic Sensor Layup

RESULTS AND DISCUSSION

Curing Model

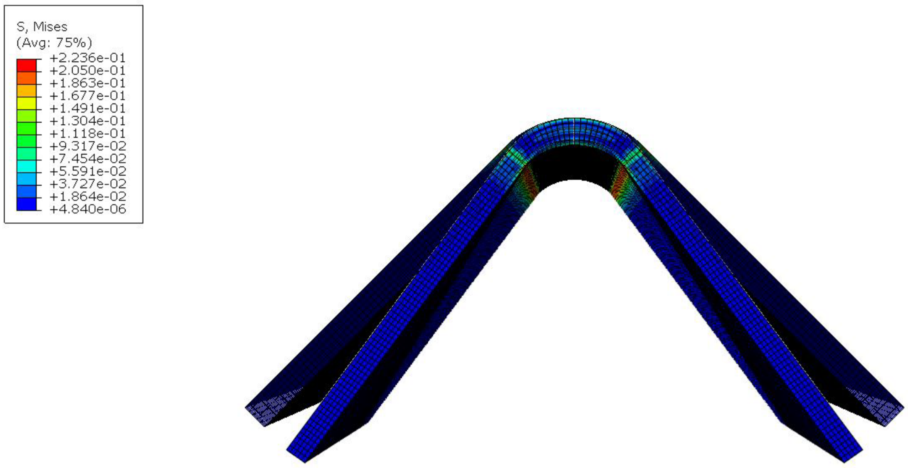

Figure 6: Spring-In of L-Beam after curing

The multi-scale modeling framework presented in the preceding sections is implemented in the commercial finite element software ABAQUS, via the user subroutines UMATHT and UMAT for the heat transfer and stress analyses, respectively. The sequential coupling of the multi-physics workflow offers a performance advantage over a fully coupled analysis and is feasible because the temperature field can be found without knowledge of the stress/deformation response. Once the residual stresses and deformation are obtained from the multi-scale curing model, the spring-in angle of the L-beam is calculated using a Python script. The spring-in obtained from the curing model is presented in Figure 6, and Figure 7 shows the spring-in response over time obtained by the curing model.

Table 1. Comparison of Experimental vs. Simulation Spring-In

Spring In of L-Beam	
Experimental Value	2.3°
Curing Model	2.21°

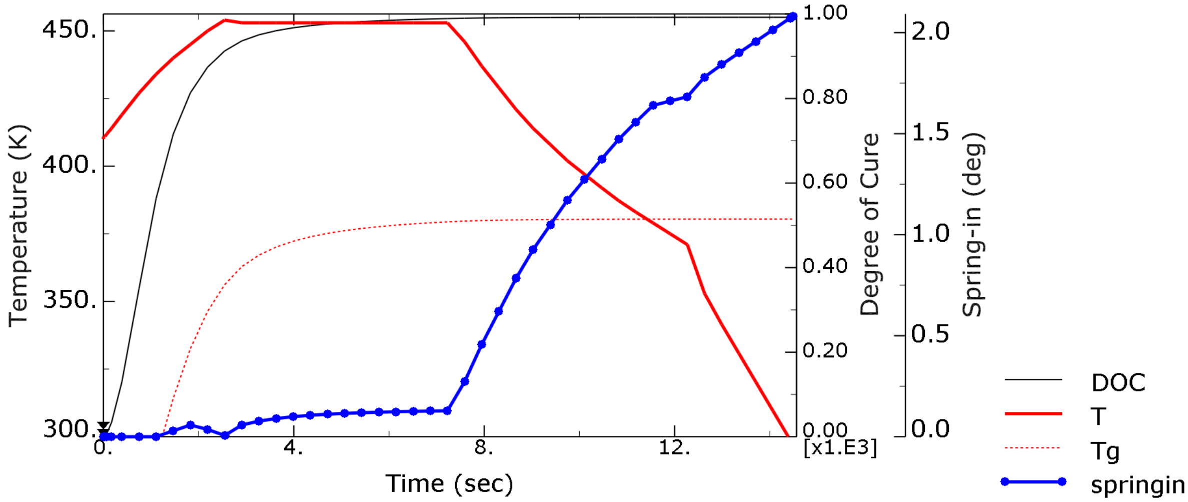

Figure 7. Spring-In response over time

Dielectric Cure Monitoring

Resin Flow Front

During the infusion process which happens at 121°C, the flow sensors, record the resin arrival time, as the sensor gets filled. The Cure sensors also record the resin arrival, which is recorded by the change in the resistance values. By using the data acquisition software, OptiView, provided by Synthesites, the flow information is obtained and presented in Figure 8.

Figure 8: Resin Arrival measured by Flow Sensors

Based on the results from the Flow Arrival Sensors, it is observed that the resin reached F2 first, which is located on the top layer, at the inlet location followed by F1, which is located at the bottom layer of the inlet location. After this, the Cure Sensor, C1 is filled, from the drop of the resistance curve. After that, F3, located at the bottom layer near the outlet location, is filled, which indicates that the resin traveled faster in the bottom layer than in the top layer. This is due to the higher temperature in the bottom layer of the composite than the top layer, and hence, due to the lower viscosity, the resin travels faster. Finally, the resin arrival is recorded in the F4 and C2 sensors, located in the top layer close to the vacuum outlet. From this experiment, the resin arrival is monitored in the composite structure layer by layer, hence by proper placement of sensors at critical locations of complex composite parts, the resin filling is tracked and the time needed to close the inlet, for bleeding. Figure 9 shows the temperature data collected by the 4 flow sensors. It can be observed that there is a temperature gradient through the thickness of the layup.

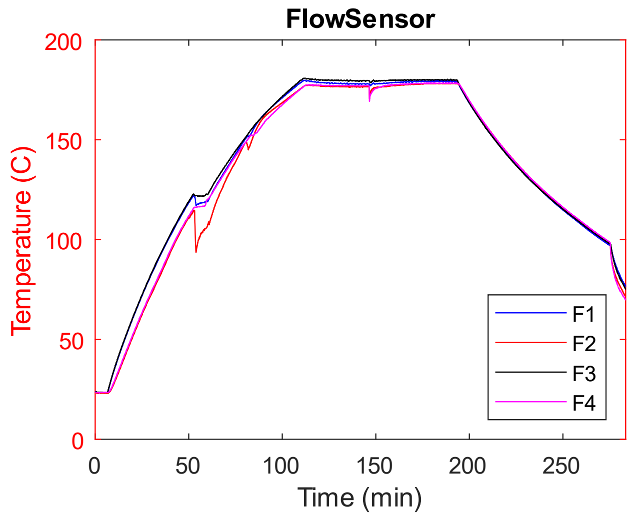

Figure 9: Temperature data from the Flow Sensors

Curing of Resin and Tg Estimation

The cure sensors measure the resistance and temperature values of the resin while using the Online Resin State (ORS) system, the information on the glass transition temperature Tg, Degree of cure, viscosity, and other information can be obtained online. As shown in Figure 10, there is a temperature difference between the cure sensor C1 and C2, which is expected as the base mold is heated and the entire setup is covered with an insulation shield.

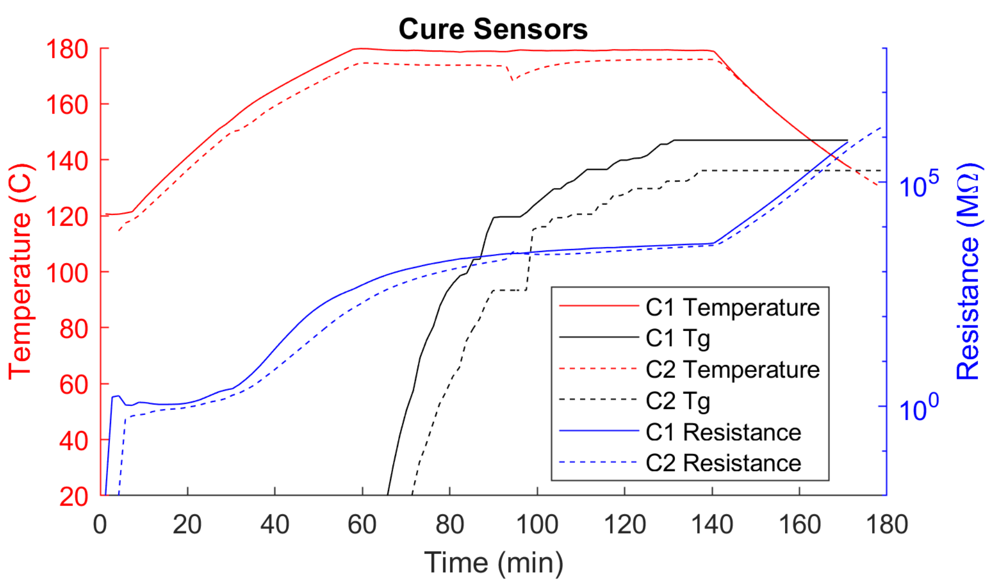

Figure 10: Resistance and Tg from Cure Sensors

From the ORS, the Tg value is obtained. It is evident from the duration of the temperature hold is only 87 mins, instead of the 120 mins of cure time recommended by the manufacturer. This is because, by this time, the required Tg is achieved at both the cure sensor locations, and hence no further curing is required. By this, the cure is monitored at critical locations, and the manufacturing process is optimized to save time and costs, without compromising the part quality.

Distributed Optical Sensing

The global average strain among all the different segments located in each layer of the composite structure is shown in Figure 11, and the strain obtained by the fiber optic sensors in each segment are shown in Figure 12. Based on the results and comparing with the temperature data, it is very clear that during the cooling period, when the cure reaches gelation, there is chemical shrinkage strain, i.e negative strain being induced due to the curing, which contributes to the spring-in. As a monitoring and prediction methodology, these curing induced strain data are used to develop and validate the curing process models developed.

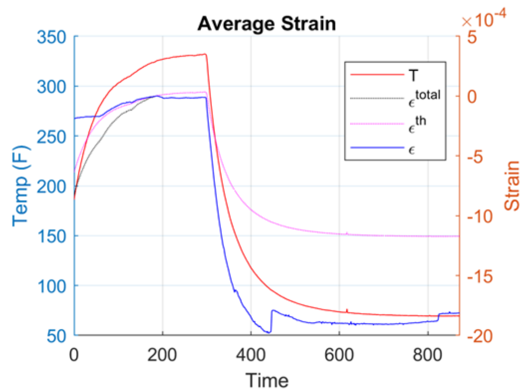

Figure 11: Average Strain measured by Fiber Optic Sensing

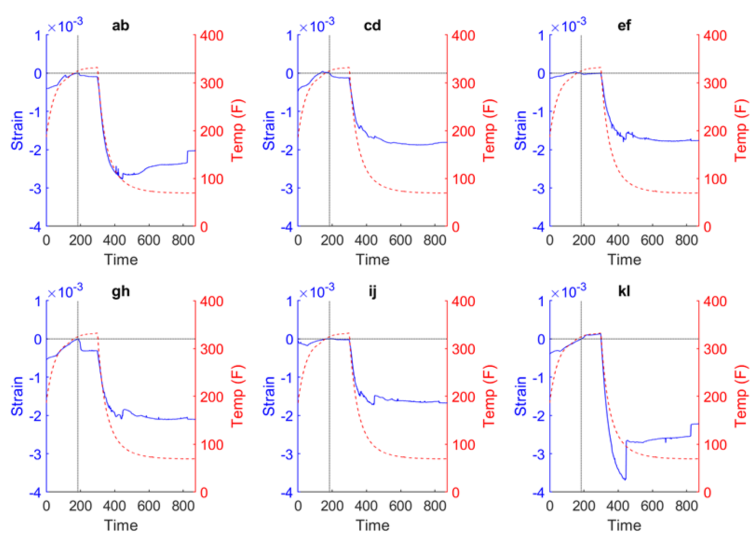

Figure 12: Strain in each segment using Fiber Optic Sensors

CONCLUSIONS AND FUTURE WORK

This work demonstrated the use of in-situ process monitoring methods such as dielectric monitoring and fiber optic sensing, for the purpose of developing cure processing models. The dielectric sensors are used to obtain the resin arrival and temperature data, as well as measure the cure quality of the resin, and this data is used to inform the infusion and curing processing model. The distributed fiber optic sensing system is used to measure the strains developed during manufacturing and validated against the processing models.

In the future, further analysis on these monitoring capabilities will be explored, including establishing a real-time cure monitoring with cure process modeling, so the experimental and physics-based models can be combined to make accurate real-time predictions on the structure being manufactured. Also, the in-situ sensor systems are used to enhance control and automation in the manufacturing processes, to achieve and accelerate the sustainable manufacturing of composite materials.

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