

ANALYSIS OF CURE BEHAVIOUR UNCERTAINTIES IN THERMOSET COMPOSITE PARTS USING PARTICLE FILTER

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Summary: *Process-induced deformations result from internal residual stress caused by the anisotropic properties of thermoset composite parts. The study's focus is diagnosing the polymerization process, or curing, and considering how uncertainties in boundary conditions affect cure kinetics. This is achieved through a Particle Filter approach, utilizing a Bayesian framework. This framework recursively estimates the evolving cure state's posterior distribution based on observed measurements from Differential Calorimetry Scanning tests and thermocouples. The algorithm simultaneously estimates the cure state parameters and predicts part temperature and process-induced deformations, which are closely tied to cure behaviour. This is accomplished using diffusion cure kinetics and analytical deformation models. Furthermore, it introduces an augmented cure state formulation to address uncertainties in cure boundary conditions, which conventional models overlook. The developed stochastic approach adeptly captures uncertainties related to cure evolution while providing comparable deformation predictions with minimal computational costs and memory usage. Experimental measurements of process-induced deformations in C-shaped thermosetting parts, made of unidirectional 8552/AS4 fibres and cured following the Manufacturing Recommended Curing Cycle, are validated using the developed algorithm. After validation, the proposed model is employed to predict outcomes, which are then utilized to determine the optimal curing cycle using a Genetic Algorithm.*

1 INTRODUCTION

Thermoset composite materials require iterative analyses before an efficient manufacturing process can be established. This is due to the Process-Induced Deformations (PIDs) observed during the manufacturing stage, [1]. Composite parts attain a deformed shape along with internal residual stresses after being processed within the mould during manufacturing phase, [2]. The mechanism behind the development of such PIDs is influenced by various factors at different mechanical levels, [3, 4]. Due to the polymerization reaction, the thermosetting resin undergoes transitions from a viscous to a rubbery and,

finally, to a glassy state. Throughout these transitions, the composite material undergoes internal residual stresses due to the thermal and chemical behaviour of the resin, [5]. Such deformations are constrained from developing freely due to contact with mould when cured and this results in the development of internal residual stresses at micro-scale level. Looking at the macro-scale perspective, factors such as anisotropic properties, stacking sequence, part thickness, mould material, mould-part interaction etc., are found to have significant impact on PIDs, [6, 7, 8]. Thermally induced residual stress field are caused by the discrepancy between coefficients of thermal expansion of the resin and fibre, [9]. These thermal residual stresses are tensile and compressive in nature for the resin and fibre respectively, since the coefficient of thermal expansion of resin is much higher than that of the fibre. Another source of the residual stresses is because of chemical shrinkage associated with the coefficients of chemical shrinkage, causing the reduction of volume of resin during cure process. The resin molecules are pulled inwards throughout the end linking reactions at higher temperatures, leading to transition between different states. This causes adding pressure on the fibre in form of internal residual stress distributed uniformly across the entire composite laminate. Upon de-moulding, the stresses on free surface are released to give deformed shapes to the manufactured parts.

Analytical approaches, [10] have been introduced to predict the thermoelastic and non-thermoelastic components associated with the PIDs. These components are observed to be dependent on the cure state variables and mechanical properties, as studied in a previous work, [11]. In recent times, numerous studies have addressed uncertainties in composite manufacturing, [20]. The state-of-the-art Finite Element (FE) based constitutive model, [24] considers cure kinetics parameters to be deterministic in each time step, thus overlooking the variability in expected cure times. The uncertainty in cure behaviour related to different temperature rates is apparent from experimental characterizations, [17]. Cure-kinetics parameters significantly influence the actual part temperature, [15]. Consequently, there is a need for stochastic simulations and analyses to quantify the uncertainties linked to cure kinetics models and cure temperature cycles in thermosetting composite manufacturing.

We intend to address these limitations in the study by presenting a novel approach. The demand for active manufacturing and cost-efficient methods drives the adoption of the Particle Filter (PF) framework. The relationship between the cure state of a thermosetting part and the measurements from Differential Scanning Calorimetry (DSC) exhibits variations due to uncertainties in manufacturing conditions and autoclave tolerances. In this work, the PF Sequential Importance Resampling algorithm is employed to filter the uncertainty associated with the evolution of the cure state, as described by the diffusion cure-kinetics model, combined with the inherent randomness. The filtered information is then utilized to numerically update the Probability Density Function (PDF) of the future cure state, part temperature, and PIDs. The model parameters associated with the Degree of Cure are included in the state vector, and their corresponding PDF is sequentially updated based on thermocouples measurements. The filtered information is then employed to numerically update the PDF of future cure states. The parameters that are stochastically updated at discrete time steps to describe cure evolution are utilized

for predicting future part temperature overshoots and PIDs. The prognosis of part temperature overshoot and PIDs, as a function of future cure states, could be crucial when uncertainties in manufacturing boundary conditions exist. Furthermore, a Genetic Algorithm is integrated with the converged PF prognosis models to determine optimal curing cycles.

2 THEORY

2.1 Cure induced expansional strains and PIDs

The Degree of Cure, denoted as X , characterizes the system's condition during the curing process. It signifies the proportion of heat released, H_t , at a specific time in comparison to the total heat released throughout the entire curing cycle at temperature. Upon entering the glassy state during the curing process, there is a significant decrease in molecular mobility due to the reduction in resin volume. This phenomenon is quantified by another parameter called the glass transition temperature, T_g . The transition from the rubbery state to the glassy state is understood to occur when the cure temperature, T , aligns with T_g , which is known to be dependent on X .

The actual part temperature, denoted as T_{part} , the anisotropic thermo-chemical and mechanical properties represented by Σ_{Mech} , the thermoelastic strain component denoted as ε_{th} , and the non-thermoelastic strain component denoted as ε_{ch} are highly dependent on the cure state. The thermal and analytical models for predicting the actual part temperature and PIDs in curved thermosetting parts, as mentioned in studies [23] and [14] respectively, are defined as functions of the cure state.

$$T_{\text{part}} = f(X, k, C_p, T, b, \text{Mould}) \quad (1)$$

$$\Delta\theta = g(\Sigma_{\text{Mech}}(X, T_g), T_{\text{part}}, b, \text{Mould}) \quad (2)$$

In this context, k represents the thermal conductivity of the material, C_p indicates the specific heat capacity, and b stands for the total thickness of the part. Therefore, comprehending the impact of uncertainties in the cure state on the part temperature, which in turn influences the PIDs during the manufacturing process, is of paramount importance for addressing an efficient manufacturing process.

2.2 Non-linear Bayesian tracking problem

The Bayesian tracking operates on this probabilistic state-space formulation and updates the state variables with incoming new measurement data at the next time step. The estimate of state, \mathbf{x}_i is performed up to time step i based on available set of measurement datasets. The primary idea is to recursively estimate different values of the state \mathbf{x}_i at a time step i , given the available data $\mathbf{z}_{1:i}$, resulting in the formulation of PDF $p(\mathbf{x}_i|\mathbf{z}_{1:i})$. It is assumed that the initial PDF, $p(\mathbf{x}_0|\mathbf{z}_0)$ is available, which allows the PDFs $p(\mathbf{x}_i|\mathbf{z}_{1:i})$ to be obtained through prediction and update stages. During the prediction stage, the prior PDF is obtained at time step i , assuming that the PDF $p(\mathbf{x}_{i-1}|\mathbf{z}_{1:i-1})$ at time step $i-1$ is given by,

$$p(\mathbf{x}_i|\mathbf{z}_{1:i-1}) = \int p(\mathbf{x}_i|\mathbf{x}_{i-1})p(\mathbf{x}_{i-1}|\mathbf{z}_{1:i-1})d\mathbf{x}_{i-1} \quad (3)$$

The state evolution, $p(\mathbf{x}_i|\mathbf{x}_{i-1})$ is described by the process model equation. At time step i , the measurement \mathbf{z}_i becomes available, and consequently, the prior PDF is updated using Bayes' rule,

$$p(\mathbf{x}_i|\mathbf{z}_{1:i}) = \frac{p(\mathbf{z}_i|\mathbf{x}_i)p(\mathbf{x}_i|\mathbf{z}_{1:i-1})}{p(\mathbf{z}_i|\mathbf{z}_{1:i-1})} \quad (4)$$

where $p(\mathbf{z}_i|\mathbf{z}_{1:i-1})$ is the normalizing constant that depends on the likelihood PDF, and $p(\mathbf{z}_i|\mathbf{x}_i)$ is described by the measurement model. In the updating stage, the prior density is adjusted using experimental measurements to derive the required posterior PDF at a given time step. The set of above equations (3-4) mentioned above establishes the foundation for an optimal Bayesian solution, which computes the precise PDF of a system state with uncertainties.

2.3 Particle Filters

The estimation of the posterior density is a conceptual and complex task to implement analytically. For this purpose, PF are utilized to approximate Bayesian solutions. PF falls under the category of non-linear Bayesian filters, and the idea behind PF is to recursively implement a Bayesian filter using the Monte-Carlo sampling technique, [18]. In this approach, the Probability Density Function (PDF) is represented by a random sample of N_s particles, each associated with weights. The estimation is ultimately based on the available set of samples and their corresponding weights. The prediction stage employs the process model to predict the PDFs, accounting for random noise from one measurement to the next. In the update stage, using Bayes' rule, the new measurement adjusts the predicted PDF. The algorithmic flow of the PF methodology is outlined in Pseudocode (1).

2.4 Particle Filter framework for the curing process

Consequently, the need arises to regulate the cure rate on the basis of thermocouple measurements. Using discrete time steps, the evolution of the cure kinetics state, [19] is discretized, and the value of X in the subsequent time step i is given by,

$$X_i = X_{i-1} + e^{\nu_i} \left[\frac{KX^m(1-X)^n}{1 + e^{C(X-X_C)}} \right] \quad (5)$$

where $K = Ae^{-\Delta E/RT}$ and $X_C = \kappa_0 - (\kappa_t * T)$

where R represents the Gas constant (J/mol/K), A is the pre-exponential cure coefficient (Sec^{-1}), E stands for the activation energy (J/gmol), m and n denote the exponential parameters, C signifies the diffusion constant, κ_0 is the critical degree of cure at the beginning (K^{-1}), and κ_t is the constant accounting for the increasing significance of the variable X as the temperature T changes. The Gaussian noise, $\nu_i \sim \mathcal{N}\left(-\frac{\sigma^2}{2}, \frac{\sigma^2}{4}\right)$, is introduced to add intrinsic stochastic characteristics to the cure evolution. The primary concept is

Algorithm 1 Algorithmic flow for implementation of Particle Filter algorithm

- 1: **for** time step i **do**
 - 2: Let $\{\mathbf{x}_{0:i}^k, w_i^k\}_{k=1}^{N_s}$ be the random measure where $\{\mathbf{x}_{0:i}^k\}$ is a set of state variables with associated weights, w_i^k describing posterior PDFs.
 - 3: Draw N_s particles $\mathbf{x}_{0:i}^k = \{x^j, j = 0, 1, 2, \dots, k\}$ from the prior PDF, $p(\mathbf{x}_i | \mathbf{x}_{i-1})$
 - 4: Compute the modified weight, w_i^k as a function of the likelihood, $p(\mathbf{z}_i | \mathbf{x}_i^k)$ which is in a Gaussian form, $\prod_{j=1}^s \frac{1}{(2\pi\sigma_{\omega,i}^2)^{0.5}} e^{-\frac{(\mathbf{g}^j(\mathbf{x}_i) - \mathbf{z}_i^j)^2}{2\sigma_{\omega,i}^2}}$ where s are the total observations within i time steps, $\mathbf{g}^j(\mathbf{x}_i)$ and \mathbf{z}_i^j are the j^{th} element of the response function and observations respectively.
 - 5: Compute the posterior density with the new modified weight and resampling of $\mathbf{x}_{0:i}^k = \{x^j, j = 0, 1, 2, \dots, k\}$ with updated weights.
 - 6: **end for**
 - 7: Prognosis of the system states in future time step, $i + l$, $p(\mathbf{x}_{i+l} | \mathbf{z}_{1:i}) = \sum_{k=1}^{N_s} w_{i+l-1}^k p(\mathbf{x}_{i+l} | \mathbf{x}_{i+l-1}^k)$ where $p(\mathbf{x}_{i+l} | \mathbf{x}_{i+l-1}^k)$ is the PF estimates at i^{th} time step. w_{i+l-1}^k is the computed weight of k^{th} particle in future time step and is assumed to be equal to $(1/N_s)$ after resampling at every time step.
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to formulate a model that treats the specified constants stochastically, updating them at each time step to establish the process equation in the form,

$$\begin{bmatrix} \Omega_i \\ X_i \end{bmatrix} = \begin{bmatrix} \sqrt{1 - h^2} \Omega_{i-1} + (1 - \sqrt{1 - h^2}) \tilde{\Omega}_i + \tau_i \\ X_{i-1} + e^{\nu_i} \left[\frac{KX^m(1-X)^n}{1 + e^{C(X-X_C)}} \right] \end{bmatrix}, \quad T_{\text{part}_i} = g(X_i) + \omega_i \quad (6)$$

$$\text{where } \Omega = [A \ E \ m \ n \ C \ \kappa_t \ \kappa_0]^T \quad (7)$$

$\tilde{\Omega}_i$ represents the mean of samples for the cure-kinetics parameters, and $h \in [0, 1]$ serves as the kernel smoothing constant. Upon estimation of $\tilde{\Omega}_i$ with thermocouple measurements, T_{part_i} , the PF framework is employed to predict future cure states. Utilizing this information, the PDFs of T_g , are obtained to forecast state transitions during the manufacturing of thermosetting parts. Ultimately, the part temperature overshoot and PIDs, as a function of the cure state, are predicted post-manufacturing. Five DSC experiments corresponding to temperature cycles at three different rates, specifically 1.5°C/min, 0.55°C/min, and 0.5°C/min, along with isothermal dwells at 180°C, 175°C, and 185°C, including some with partial curing, are carried out for the 8552/AS4 material. The correlation between T_g and X is established based on the DSC tests, following the relation given by,

$$T_g = (39.98X^2) + (222.37X) - 32.17 \quad (8)$$

The initial cure state distribution is defined using the dataset of DSC tests. This is accomplished by expressing the diffusion cure-kinetics constants in terms of a joint PDFs with the mean and covariance matrices defined as:

$$\mathbf{E}[\boldsymbol{\Omega}_{i=0}] = \begin{bmatrix} 6.72 \times 10^4 \\ 6.33 \times 10^4 \\ 4.92 \times 10^{-1} \\ 1.76 \times 10^0 \\ 3.01 \times 10^1 \\ 5.26 \times 10^{-3} \\ -15.55 \times 10^{-1} \end{bmatrix} \quad (9)$$

$$\Sigma[\boldsymbol{\Omega}_{i=0}] = \begin{bmatrix} 1.96 \times 10^3 & 0 & 0 & 0 & 0 & 0 & 0 \\ \vdots & 1.21 \times 10^3 & 0 & 0 & 0 & 0 & 0 \\ \vdots & \vdots & 5.80 \times 10^{-2} & 0 & 0 & 0 & 0 \\ \vdots & \vdots & \vdots & 5.00 \times 10^{-3} & 0 & 0 & 0 \\ \vdots & \vdots & \vdots & \vdots & 1.79 \times 10^{-1} & 0 & 0 \\ \vdots & \vdots & \vdots & \vdots & \vdots & 6.39 \times 10^{-5} & 0 \\ \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & 2.79 \times 10^{-2} \end{bmatrix} \quad (10)$$

The PDF assumes that the different diffusion cure-kinetics parameters are inherently independent. In simpler terms, the covariance between these parameters is set to 0 due to the unavailability of data. A random multiplicative term is integrated into the process equation to depict the evolution of the cure state within the range of $[0, 1]$, while introducing perturbations to encompass its uncertainty. At each time step i , the distributions of process noise are sampled and allocated to the particles. The initial range for X is considered to be within the range of $[1 \times 10^{-4}, 1 \times 10^{-3}]$. The process noise is modelled as a Gaussian distribution with parameters $\mathcal{N}\left(\frac{-(1.15)^2}{2}, \frac{-(1.15)^2}{4}\right)$, while the measurement noise is assumed to follow a zero-mean Gaussian distribution with a standard deviation of $\sigma_\omega = 1$ °C. The number of particles, denoted as N_s , is set to 16000, and the kernel smoothing parameter, h , is assigned a value of 0.2.

3 DISCUSSION AND RESULTS

3.1 Case study: Curing of C-Shape part with Uni-directional plies

The methodology discussed in Section 2.4 is implemented for the case study of unidirectional AS4/8552 prepregs produced by Hexcel. A C-shaped part which is 4 mm thick is composed of a stacking sequence, $[90^\circ]_{16}$. The Manufacturer's Recommended Cure Cycle (MRCC) is employed, involving an initial temperature increase of 2°C/min up to 120°C, followed by a 60-minute isothermal hold. Subsequently, a second temperature increase of 2°C/min up to 180°C is carried out, followed by another 120-minute isothermal hold. Finally, the temperature is gradually brought back to room temperature at a rate of 2°C/min. A pressure of 7 bars is maintained throughout the curing cycle. To mitigate thermal expansion in the circumferential direction, the C-part with 270° segments is cured within a carbon-epoxy tube mould. The convective heat transfer oven boundary condition is taken as 60 W/m²K. Thermo-chemical and mechanical properties are taken from the studies, [21, 22, 23].

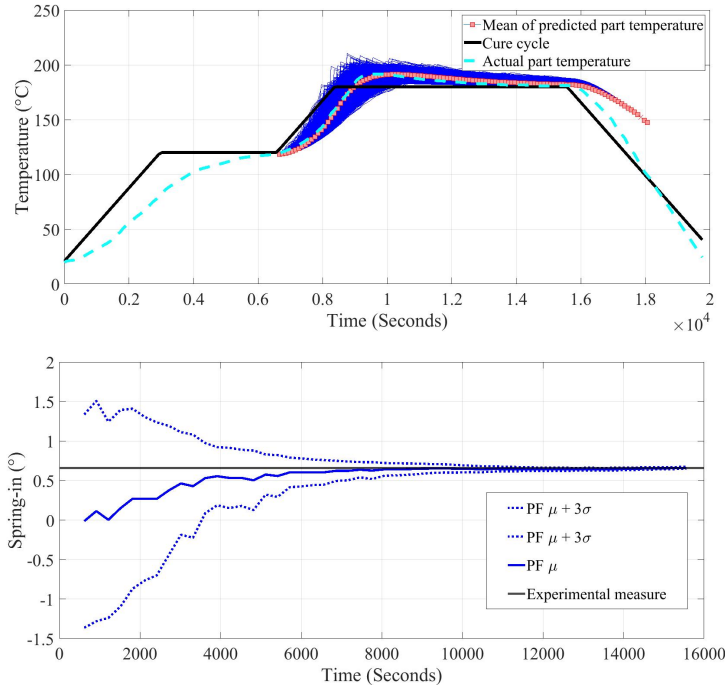


Figure 1: (a) Actual part temperature predictions after onset of gelation (b) PIDs (spring-in angle) predictions with estimated model parameters at discrete time steps

The prior joint distribution naively assumes the diffusion cure-kinetics parameters to be uncorrelated initially. However, after a few state updates, a smooth emergence of correlation between the parameters governing the PF algorithm becomes evident. The PF algorithm is capable of providing meaningful estimates of the cure state, because of the updated likelihood variance. The expected mean upon updating, $\mathbf{E}[\boldsymbol{\Omega}]$ shifts as the process model adjusts to the collected thermocouple data points along with the influence of measurement noise. The process noise, introduces some blurring effect to the relationship between the process model and the model parameters. This ensures the retention of the inherent uncertainty linked to the cure state of the part. Furthermore, at time, $t = 6560$ seconds, the associated uncertainty with the sequential state update decreases. The successful prediction of the future cure state until the culmination of the manufacturing process is attributed to the estimated parameters as the transition to the rubbery state takes place. Figure 1 illustrates two key predictions resulting from the stochastic analysis: the actual part temperature predictions after gelation point ($X > 0.3$ and $t = 6560$ seconds) and the predictions of PIDs (in terms of spring-in angle) along with a 95 % confidence interval. The analysis shows a significant alignment with both the thermocouple measurements and the final experimental measures of PIDs, [25]. The narrowing of the confidence intervals in the case of PIDs reflects the updating of state evolution model parameters, thereby providing well-informed estimates and improved prognostic capabilities. The fundamental concept here is to uphold the stochastic nature of the model parameters while converging towards accurate values, factoring in the temperature changes throughout manufacturing. It is observed that after the conclusion of the first isothermal dwell at 120°C, the projected trajectories of the particles tend to correlate with

the measured part temperature.

3.2 Optimization of optimal cure cycle profile

In the context of curing boundary conditions, the objective is to determine optimal parameter values for the two dwell cure cycles. This is aimed at achieving complete curing of the thermoset composite while minimizing PIDs, part temperature overshoot and total cure time.

Table 1: Design parameters range

Curing cycle characteristics	Two-dwell curing cycle
Δt_1	60 to 120 (Minutes)
r_2	0.5 to 3.5 ($^{\circ}\text{C}/\text{Min}$)
T_2	170 to 200 ($^{\circ}\text{C}$)
Δt_2	60 to 120 (Minutes)
r_3	-3.5 to -0.5 ($^{\circ}\text{C}/\text{Min}$)

After passing the gelation point, the two isothermal dwells are characterized by the duration of the first and second dwells (Δt_1 and Δt_2), the heating rate r_2 , the cooling rate r_3 , and the temperature of the second dwell T_2 . The range of these parameters used for the optimization procedure is outlined in Table 1. The incorporation of the GA optimization facilitates the concurrent minimization of multiple objectives within the PF framework.

Table 2: GA-PF optimization parameters

Parameters	GA parameters
Optimization individuals	5
Maximum number of generations	200
Population size	100
Cross-over probability	8×10^{-1}
Tolerance (best fitness function value)	1×10^{-4}

In each iteration, the GA generates a set of 5 cure cycle parameters for analysis. The GA parameters employed in the optimization procedure are detailed in Table 2. The PF prognosis model, having converged after the gelation point, utilizes the estimated diffusion cure-kinetics state parameters to guide the selection of new inputs or control actions that are optimal within the updated test conditions. Upon reaching the threshold of X at 93 %, the interface formulates the three objective functions in terms of the expected part temperature, PIDs, and the total cure time. The GA explores various combinations of cure cycle characteristics to discover a set that collectively minimizes all three objectives. The outcome of this optimization would yield a set of input cure cycle characteristics that

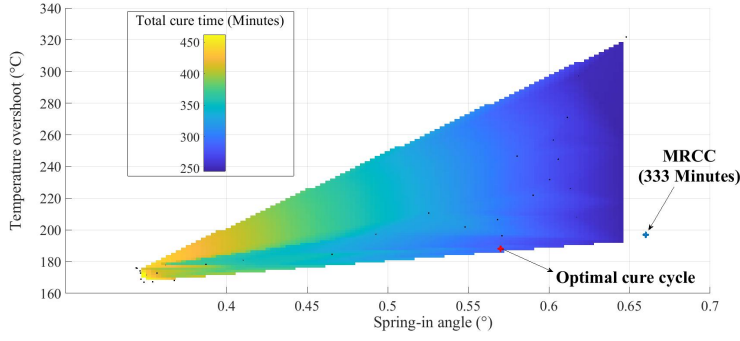


Figure 2: 3D Pareto front representing the MRCC and the optimal cure cycle

simultaneously minimize PIDs, temperature overshoot, and total curing time. This set of cure cycle characteristics represents the optimal trade-off among the diverse objectives and contributes to a more efficient and energy-conserving thermoset curing process. The interface between GA and PF requires approximately 1.2 hours for optimizing the cure cycle characteristics, leading to the generation of a 3D Pareto front depicted in Figure 2. This approach surpasses exhaustive searches involving the complete cure cycle and is more effective compared to searches conducted after the gelation point.

An optimal cure cycle is devised based on the 3D Pareto front. The optimized cycle is 33 minutes shorter, and the temperature overshoot of the part is reduced by 10.2°C . Furthermore, the optimized cycle is validated against the MRCC using the FE based constitutive model, [24] to analyse residual stress/strain fields. By adopting the optimized cycle, the part undergoes an extended duration during the first isothermal dwell, effectively spending more time in the rubbery state. Consequently, the chemical shrinkage counterbalances thermal expansion, leading to a decrease in maximum residual tensile stress field. This effect is demonstrated in Figure 3. Consequently, a more efficient cure cycle with minimized PIDs can be developed with reduced computational costs.

4 CONCLUSIONS

The study incorporates a sequential Monte-Carlo model in the form of a Particle Filters within the realm of manufacturing thermoset composite parts. This methodology serves for estimating polymerization, and predicting the cure state under varying boundary conditions. This is achieved by advancing a set of particles through time intervals, which represent potential states of the manufacturing system, and adjusting their respective weights based on observations from thermocouples. The characterised data offer valuable insights into the behaviour of the cure state, the glass transition temperature, and related parameters. Moreover, the methodology enables the incorporation of the stochastic nature of the parameters linked to the cure state. This consideration allows us to address the variations in the peaks of cure reaction rates, which arise from uncertainties linked to boundary conditions. The methodology is integrated with the actual temperature profile of the thermoset part during the curing process, enabling accurate predictions of PIDs based on the predicted cure state. This distinction is important because the part's temperature differs from the control temperature. The converged PF prognosis capability

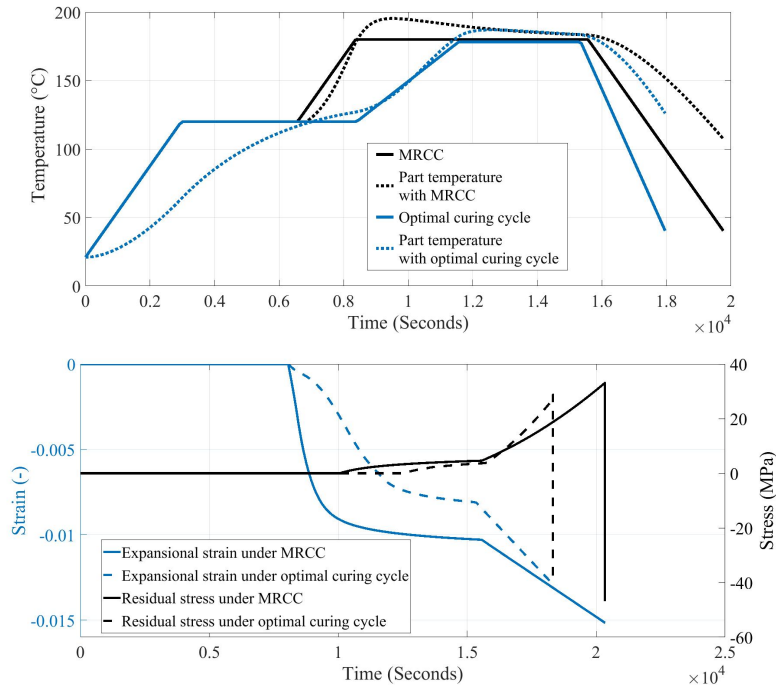


Figure 3: Part temperature, stress and strain fields comparison (in centre ply) between MRCC and optimal curing cycle

is integrated with an evolutionary Genetic Algorithm to conduct multi-objective optimization, aiming to minimize three key objective functions: total cure time, PIDs, and temperature overshoot in the part. In summary, the implementation of online diagnosis and prognosis models in the active manufacturing of thermosets contributes to enhanced process reliability, quality assurance, and cost-effectiveness.

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