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# Stochastic modelling of out-of-autoclave epoxy composite cure cycles under uncertainty

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ABSTRACT

Thermoset polymers and composites are subject to several sources of uncertainty which can produce a range of cure outcomes. Recent research into stochastically modelled thermoset cure has indicated that accounting for raw material and process uncertainty can model this range of expected output parameters. However, the uncertainty quantification methods are highly test-intensive, and the results of the simulations have been validated with limited experimental data. This study proposes a simple approach to cure kinetics uncertainty quantification that can be applied to any cure kinetics model without the need for additional testing. Stochastic cure kinetics and temperature conditions for a popular out-of-autoclave carbon fibre/epoxy prepreg were used to produce output distribution functions for key cure events, and the results were validated using data from ten cure replicates. The quantified variation expected from the cure of this prepreg resulted in processing recommendations to ensure quality metrics are met during processing.

## 1. Introduction

Thermoset composites are an attractive option for high-performance components in a variety of industries. Performance is strictly tied to quality parameters such as the degree of cure and the glass transition temperature ($T_g$) at the end of the cure cycle, which are directly related to the processing conditions [1]. A current trend in composites research is to accelerate and optimise the processing conditions, while still producing parts of a sufficient quality. Optimisation techniques include exhaustive test matrices [2] or numerical methods [3,4] including gradient based techniques [5], genetic algorithms [6], and the Evolutionary Strategy [7]. Optimisation techniques typically target minimising a specific feature such as the total process time [7], temperature gradient [8], or the exotherm temperature [9] or maximising the part quality [10] or performance [11]. Studies have also investigated the balance of multiple objective functions which can potentially have conflicting solutions [9,11,12]. The success of the optimisation activities is dependent on the accuracy of the numerical modelling tool which is used to produce the optimised parameters.

Cure simulation tools can predict cure behaviour over a range of complexities and scales. A 0-dimensional (0D) simulation provides the most fundamental view of how a thermoset polymer reacts to a given cure profile. A 1-dimensional (1D) or 2-dimensional (2D) simulation will provide insight to how the depth or spatial area of a resin responds, which encompasses heat transfer behaviour from the surrounding polymer reaction [13,14]. Finally, a 3-dimensional (3D) view provides the highest complexity with the capability of modelling specific part geometries [15–17]. However, thermoset composites display a large amount of final part property variation due to uncertainty which impacts the accuracy of these practices. Typically these systems do not account for process uncertainty, instead they rely on a deterministic cure kinetics model which has limited accuracy [16,18]. By not considering this uncertainty there is an increased risk of an optimised process resulting in a part not meeting the quality requirements.

Uncertainty in composites originates from several sources including fibre architecture, resin formulation and mixing, environmental conditions, and from the processing steps [19,20]. It is also shown that variation in resin formulations and mixing can strongly impact the viscosity and cure behaviour [21]. Varying parameter values to illustrate this uncertainty can produce a significant impact on the final part outcome [22]. The multiple origins and sources of uncertainty may also interact with one another, making it necessary to understand their impacts both independently and together. For example, an epoxy vinyl ester resin system [23] produced equivalent responses for varying cure kinetics parameters and for varying heat transfer model parameters by one standard deviation [24]. Another epoxy system, however, had a far

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**Table 1**
A summary of studies implementing stochastic modelling to capture composites processing uncertainty including details on uncertainty quantification method, sampling method, and input and output parameters.

| Source | Stochastic parameters | Uncertainty quantification | Sampling method | Material | Output parameters |
|---|---|---|---|---|---|
| [27] | Temperature, cure kinetics parameters | Assigned 1.5 %, 3 %, 5 % variance with normalised deviations | Latin hypercube | Epoxy, polyester | Cure time |
| [22] | Temperature, cure kinetics parameters | Assigned 2 %, 3.5 %, 5 %, 10 % variance | Latin hypercube | Polyester | Cure time, maximum temperature, maximum temperature difference, degree of cure |
| [25,31] | Temperature, heat transfer coefficient, cure kinetics parameters | Experimentally determined | Monte Carlo, Probabilistic Collocation | Carbon fibre/epoxy | Cure time |
| [22,31] | Preform permeability, resin viscosity, and cure kinetics parameters | Assigned 1 % probability distributions | Latin hypercube | Generalised resin transfer moulding materials | Fill time, degree of cure |
| [32] | Woven fabric preform permeability | 1D and 2D flow measurements to quantify variance | Monte Carlo | Woven fabric | Flow ending location |
| [33] | Fibre tow direction and dimensions | Image analysis to quantify probability distributions for parameters | Monte Carlo | Carbon fibre/epoxy | Wrinkling strain |
| [36] | Initial degree of prepreg impregnation | Analysis of CT scans to quantify stochastic distributions | Probabilistic collocation | Out-of-autoclave prepreg | Void content |
| [37] | Cure kinetics parameters | Experimentally determined | Monte Carlo, Probabilistic Collocation | Carbon fibre/epoxy | Maximum temperature, time at maximum temperature |

stronger influence of temperature and heat transfer coefficient boundary conditions compared with the impact of cure kinetics [25]. As each polymer is unique, it is necessary to identify the influence of uncertainty sources for each system.

Uncertainty in cure cycle designs can be modelled using stochastic methods [26–28], multiperiod formulations [29], and parametric methods [30]. Stochastic methods have characterised a number of composites aspects which display high levels of uncertainty including flow during resin infusion [31,32], wrinkling effects in woven composites [33], residual stress build-up [34], delamination onset time [35], tow impregnation [36], structural properties [28], and resin curing [12,27,37]. Stochastic methods are based on uncertainty quantification, sampling of parameters from the resultant distribution, inputting the parameters into a deterministic model, and extracting output parameters over a series of iterations to establish a converging value. A variety of rationales have been provided for quantifying uncertainty in composites processes, with a summary of the methods and their use in stochastic modelling given in Table 1.

A popular out-of-autoclave carbon fibre/epoxy prepreg, CYCOM® 5320-1 [38,39] has been evaluated in many studies. Areas of interest have included modelling of cure kinetics [40–43], viscosity [40,42,43], thermal expansion coefficient [44], residual stress development [45], and cure cycle evaluation and optimisation [46,47]. While there have been numerous cure kinetics models proposed for this resin system, it is unknown how the models respond to sources of uncertainty. This paper characterises the stochastic behaviour of 5320-1 under two sources of uncertainty: cure kinetics modelling and processing temperature. A new methodology for assessing cure kinetics parameter variance for complex

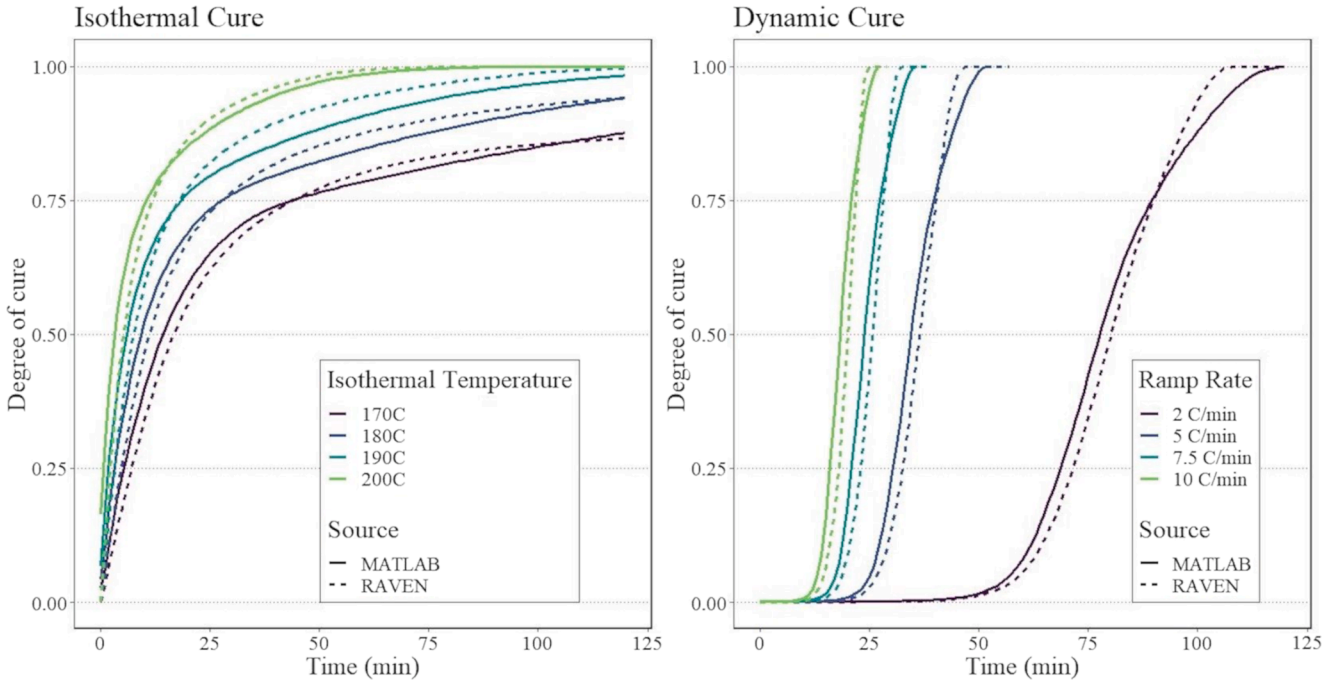


**Fig. 1.** Comparison of MATLAB generated kinetic model from [43] for a (left) isothermal cure and (right) dynamic cure rate, demonstrating a good model fitting compared with RAVEN.

cure models is proposed and compared with existing methodologies. Stochastic cure simulations for a standard ramp and dwell cure cycle are provided, with assessments on how the cure kinetics and viscosity models react to uncertainty in the temperature and kinetics. The resulting distribution of output parameters is then compared with experimental data to assess the accuracy of the simulation. Finally, suggestions are made for cure cycle considerations to ensure conforming products.

## 2. Methodologies

### 2.1. Cure kinetics and viscosity models

#### 2.1.1. CYCOM® 5320-1

The original model for CYCOM® 5320 epoxy was developed by Kratz et al [41]. The updated 5320-1 version was later developed to improve the material out-life [48], which has resulted in multiple kinetic models that apply to this system of materials. These models include a two-step kinetic equation similar to that of 5320 [42], a two-step equation with parameters designated by a lookup table based on degree of cure change [49], and a neural network model [40]. The model used in this paper was developed by Kim et al [43], which is comprised of four distinct reactions and weighted parameters to account for the impact of material out-time on the reaction rates. This model has been validated in multiple publications including to evaluate the effect of cure cycles on degree of cure [46,47]. Other available models were evaluated; however, the Kim model was determined to be the most accurate for the purposes of this study and is shown in Eqs. (1) and (2):

$$\frac{d\alpha}{dt} = \sum_{i=1,3} w_i K_i \alpha^{m_i} (1-\alpha)^{n_i} + \sum_{j=2,4} \frac{w_j K_j \alpha^{m_j} (1-\alpha)^{n_j}}{1+\exp\left(D_j\left(\alpha - \left(\alpha_{C0,j} + \alpha_{CT,j} T\right)\right)\right)} \tag{1}$$

$$K_n = A_n \exp\left(-\frac{E_{A,n}}{RT}\right) \text{where } n = i, j \tag{2}$$

where the reaction rate ($\frac{d\alpha}{dt}$) is calculated as functions of the degree of cure ($\alpha$) and the temperature ($T$). In these equations $A_n$ and $E_n$ are respectively the Arrhenius coefficient and activation energies, $R$ is the gas constant, $m$ and $n$ are reaction orders. The impact of diffusion is taken into account using the Chern and Poehlein model [50] modified by [51], for which $D$ is the diffusion constant, $\alpha_{C0}$ and $\alpha_{CT}$ are the critical degree of cure at absolute zero and it's increase at the instantaneous temperature. Parameter values can be found in [43]. The weight factors ($w$) originally represented the impact of out-time on the curing kinetics, but as the out time for the prepreg used here is not precisely known the values used were $w_1 = 0.8, w_2 = 0.35, w_3 = 1.1, w_4 = 1.2$, as these values provided the best fit for the model against a known cure simulation tool, as can be seen in Fig. 1.

The model was compiled in MATLAB, which was used to generate the cure behaviour and output parameters. This model and the reported weight factors were validated using Convergent RAVEN simulation software, with the results provided in Fig. 1. A set of 0D cure profiles were evaluated using the CYCOM® 5320-1/IM7-12 K material card which is available in RAVEN. This material card makes use of a lookup table to assign kinetic parameters [40]. Isothermal cure cycles were run at 170 °C, 180 °C, 190 °C, and 200 °C. Dynamic cure cycles were run from 20 °C to 300 °C at rates of 2.0, 5.0, 7.5, and 10.0 °C per minute. The degree of cure progression during each cure cycle was exported for comparison with the MATLAB degree of cure for the same cycle. The degree of cure progression compares well for these models. The key areas of interest, the time at 88 % cured and the final degree of cure, show close fitting. The value of 88 % as defining 'fully cured' is chosen for simplicity based on the manufacturers minimum degree of cure, which is commonly represented as 88.2 % [52]. It should be noted that many end-users of this material system may select a specific degree of cure value in accordance with their design values, any of which could be applied to the methods presented in this paper.

The viscosity model for 5320-1 used for this study is also published in [43], where the parameter values can be found. The model takes the form shown in Eq. (3). In this model the viscosity ($\eta$) is calculated using two terms. The first term is solved by the Arrhenius viscosity component ($\eta_i$) given in Eq. (4), which contains the viscosity activation energy ($E_\eta$), the gas constant, and the temperature. The second term of this equation is from the Castro-Macosko model [53], which relates viscosity with the

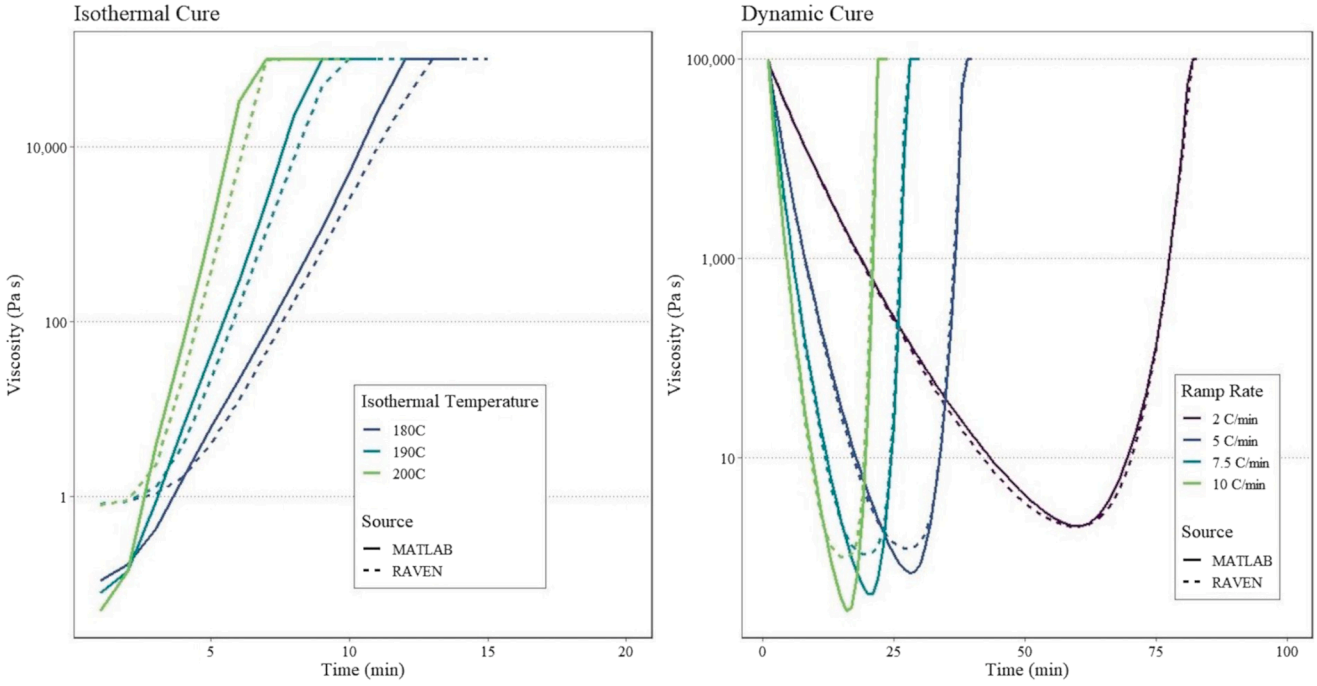


**Fig. 2.** Comparison of MATLAB generated viscosity model from [43] for a (left) isothermal cure and (right) dynamic cure rate, demonstrating a good model fitting compared with RAVEN.

degree of cure and the gel conversion point ($\alpha_{gel}$), and uses fitting constants $A$, $B$, $C$, $d$, and $e$. As the weight factors ($w$) are derived from the out-time, which is unknown, fitting parameters $w_1 = 1, w_2 = 2$, were chosen to ensure the best fit compared with RAVEN. Parameter values are published in [43].

$$\eta = w_1\eta_1 + w_2\eta_2\left(\frac{\alpha_{gel}}{\alpha_{gel}-\alpha}\right)^{A+B\alpha^d+C\alpha^e} \tag{3}$$

$$\eta_i = A_{\eta_i}\exp\left(\frac{E_{\eta_i}}{RT}\right) \text{where } i = 1,2 \tag{4}$$

The MATLAB viscosity model was written and included the cure kinetics model as detailed above. This model was also validated using Convergent RAVEN simulation software. A set of 0D cure profiles were evaluated using the CYCOM® 5320-1/IM7-12K material card, and the viscosity curve was extracted. Isothermal cure cycles were run at 180 °C, 190 °C, and 200 °C, and dynamic cure cycles were run from 20 °C to 300 °C at rates of 2.0, 5.0, 7.5, and 10.0 °C per minute. A comparison of the RAVEN output with the MATLAB output is given in Fig. 2, showing good comparison between the models. Of note is the minimum viscosity is slightly lower in the MATLAB model for both isothermal and dynamic cures. However, this study will evaluate only the time at which the minimum viscosity occurs, which is comparable for both models. The time of minimum viscosity is an important parameter for out-of-autoclave prepreg systems, as it can directly influence the temperature and dwell time during the consolidation step of the process.

#### 2.1.2. Hexcel RTM6

Kinetic modelling of Hexcel RTM6 was also completed using MATLAB for the purposes of validating the uncertainty quantification method presented in this paper. The kinetic model was originally developed in [54,55] and is given in Eq. (5), with the comprehensive set of parameter values reported in [9]:

$$\frac{d\alpha}{dt} = K_1(1-\alpha)^{n_1} + K_2\alpha^m(1-\alpha)^{n_2} \tag{5}$$

$$\frac{1}{K_i} = \frac{1}{K_d} + \frac{1}{K_c} \text{where } i = 1,2 \tag{6}$$

$$K_d = A_d\exp\left(-\frac{E_{A,d}}{RT}\right)\exp\left(-\frac{b}{0.00048(T-T_g)+0.025}\right) \tag{7}$$

where $K_1$ and $K_2$ are modified by the Rabinowitch model [56] in Eq. (6), which accounts for either the control mechanism being chemical ($c$) or diffusion ($d$) driven. For this, $K_c$ is given by Eq. (2) and $K_d$ represents the diffusion rate constant given by the Macedo and Litovitz expression [57] in Eq. (7). In this equation, $A_d$ and $E_d$ represent the coefficient and activation energy for diffusion, $b$ is a fitting constant, and $T_g$ represents the instantaneous glass transition temperature. There are multiple diffusion models which may account for the rates balance between the chemical reaction and diffusion step [50,51,58–61]. While RTM6 has been modelled using both the Macedo and Litovitz model [9,55] and the Chern and Poehlein model [62], this paper will use the Macedo/Litovitz expression for simplicity. RTM6 was unavailable for this study, so the validation of this model will be assumed from the subsequent publications from the research group given in [9,25,62,63].

#### 2.1.3. Cure cycles

The cure cycle used in this study is based on actual measurements taken during a part cure. The cure cycle is a modified version of the manufacturers recommended cure cycle [38] which has a 2 °C per minute dynamic ramp to 180 °C, followed by a 180 min isothermal dwell. The cure kinetics model used in this paper is a 0D model, meaning that it reports the cure progression of a dimensionless point in space. As the 0D kinetic model does not account for heat transfer influence on the actual temperature experienced by the laminate, the cure cycle used is a representative temperature cycle taken from the mid-plane of the IDEX2 cure from [64]. In this laminate cure, the oven temperature was set to the defined cure cycle, and the temperature profile was measured by an embedded thermocouple in the centre of the laminate. The laminate was verified to meet the manufacturers recommended cure cycle, which requires a minimum of 120 min above 171 °C [38]. This laminate achieved exactly 120 min at the cure temperature, and thus represents the threshold for complete cure.

### 2.2. Stochastic methodology

#### 2.2.1. Uncertainty quantification

Accounting for sources of uncertainty is the foundation of the stochastic approach, as the resultant variation in the manufacturing system has a very real impact on the actual process conditions that the part experiences. This study focuses on uncertainty in the cure kinetics and viscosity models and due to the applied temperature cycle. Both cure kinetics and viscosity modelling uncertainty originate from variation in raw material composition (for example, monomer content) and model fitting variation (for example, from baseline selection, equipment measurement, data reduction and fitting [59]). The main source of temperature uncertainty is due to equipment variability which can originate from the temperature control mechanism [65], temperature tolerance [66], and part location within the oven or autoclave [67].

*2.2.1.1. Kinetic model.* Previous methods have attempted to capture the actual variation of the cure kinetics values, as measured from batch-to-batch DSC testing [37]. However, as the 5320-1 model has 22 parameters this method was deemed impractical. Instead, a new approach for estimating parameter variance is proposed here, in which a coefficient of variation (COV) of 3 % was assumed for all stochastic variables based on the expected model fitting of within 3 % error [59]. This assumption of a 3 % COV is consistent with previous works [22,27] and supported by standard error expected by DSC measurements [68]. A sensitivity analysis was conducted to verify that 3 % is applicable to all parameters without distorting the cure kinetics outside of reasonable bounds. Each parameter was varied by +/- 3 % and the resulting maximum reaction rate was compared to the deterministic solution. Previous works have indicated that variation in model fitting practices can produce mode 10 % variability of the result [59]. Thus, values which yielded a greater than 10 % deviance from the maximum reaction rate were rejected, as such values would have likely changed the fitting of the original model. Any values with deviations of over 10 % were examined at reduced COVs until a value was found which kept it within the 10 % boundaries. To evaluate if the 3 % assumption allows for excessive variation, a second set of analyses were conducted using half the COV. The baseline variation was set to 1.5 %, and any parameters which required a reduced variation were also reduced by half.

To validate this approach, a comparison was made on the well-studied epoxy, RTM6, which has been evaluated for stochastic cure kinetics by Mesogitis et al. [37]. In the reported study, the cure kinetic parameter variation was determined experimentally by fitting multiple DSC curves and examining the variance of each parameter amongst the different fittings. Using the kinetic model for RTM6 indicated in Equation (5) the three stochastic parameters indicated by Mesogitis ($\alpha_0$, $E_2$, $m$) were varied according to their calculated COV. A stochastic simulation and convergence analysis was run for both a dynamic cure rate from 20 °C to 250 °C at a rate of 2 °C per minute, and for the standard cure cycle used in this study. The time to reach 88 % cured was reported as the output variable. The results of this convergence analysis were then compared to the method proposed here, of a standard 3 % variance of parameters, and a simulation using the actual COV of all parameters reported by Mesogitis. The half-variance method was also included.

**Table 2**
Test plan for comparing different methods of stochastic parameter assignment.

| Model | Output parameters | Definition |
|---|---|---|
| Cure Kinetics | Vitrification point (min) | $T = T_g$ |
| | Time at fully cured (min) | Time at degree of cure of 88 % |
| | Final degree of cure (%) | Final value of the degree of cure |
| Viscosity | Time at minimum viscosity (min) | Time at minimum viscosity |
| | Gel point (min) | Time at viscosity = 10,000 cP |

*2.2.1.2. Viscosity models.* The viscoelastic behaviour of a thermoset polymer is primarily influenced by the temperature and cross-linking of the polymer [69] and can be modelled with reasonable accuracy [70]. For this reason, the viscosity model absorbs the temperature and cure kinetics modelling uncertainty. While viscosity modelling may have additional sources of uncertainty due to measurement or fitting error, this will not be the focus of this paper. Further, viscosity magnitude has potential sources of error due to natural material variability and out-time conditions [43], which are also not considered in this paper. The viscosity modelling uncertainty will focus only on the temperature and cure kinetics modelling variance, with the aim to demonstrate the range of properties that these sources impact.

*2.2.1.3. Temperature profile.* Temperature profile uncertainty was determined based on a series of oven measurements. The oven used for this study is a fan forced convection oven with internal dimensions of 500 mm (width) by 500 mm (depth) by 550 mm (height). Thermal measurements were made using a thermocouple in air, approximately 100 mm above the part, and a thermocouple embedded in the centre of the part. The tool was placed with the rack in the middle shelving position in the oven, which places the part at approximately 150 mm below the top of the oven. The standard cure profile used in this study was measured from [64] test measurement labelled IDEX2. Ten cure profiles represented in this paper were compared to determine the actual expected variance of mid-part temperature for cures in this oven. The stochastically generated standard cure profile was varied by this percentage from the original IDEX2 temperature curve. As the small oven used for this study demonstrated a very reliable temperature profile, an additional set of analyses were done with a higher temperature variation of 5 %. This limit was chosen to account for the maximum temperature tolerance limit of 5 % which is commonly imposed on composites processing ovens and autoclaves [66].

### 2.2.2. Output parameters

The output parameters which will be evaluated in this study are given in Table 2, including their definitions for this paper.

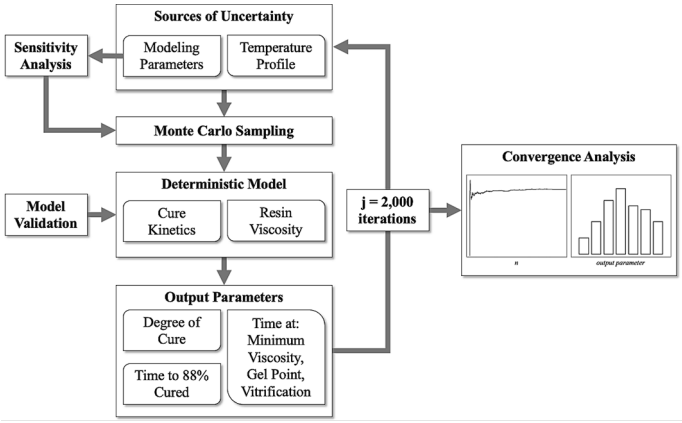


**Fig. 3.** Schematic depicting the stochastic methodology.

### 2.2.3. Stochastic methods and convergence analysis

The stochastic method involves identification and quantification of the parameters under uncertainty, sampling of the parameters, incorporating these parameters into a deterministic numerical model, and extraction of the output parameters. All of this occurs repetitively over several iterations until the output parameters converge to a resultant value. The approached used in this study is summarised in Fig. 3.

The combination of sources of uncertainty to be examined are defined in Table 3. As previously stated, all kinetic parameters are assumed to have a 3 % variance due to natural batch-to-batch fluctuations in resin composition and due to kinetic modelling error, excepting any parameters which were reduced in accordance with the sensitivity analysis in Table 6. The temperature variance is calculated based on actual measured temperature variation from oven cures. To evaluate the impact of these, cure kinetics with a half-COV and a standard 5 % of temperature were also evaluated. All varied parameters were randomly sampled using a Monte Carlo distribution method. The sampled parameters are then input to the deterministic models, Eqs. (1) and (3). The output parameters were extracted in accordance with Table 2 and added to an iterative list. The output parameters are iterated for 2000 cycles to ensure that the standard deviation converges to within 5 %. The stochastic outputs are compared with the deterministic solution, for which the MATLAB code was run with a variance of 0 % for all variables.

## 2.3. Experimental validation

The results of the convergence analysis are compared with actual measurements from 5320-1 cures meeting the requirements of the standard cure cycle definition. The cure methods and data collection techniques for the experimental tests can be found in [64]. The temperature profile data from the laminate mid-point for each part was then run through the code used for this paper, and the relevant outputs were determined in accordance with Table 2 for the standard cure.

# 3. Results and discussion

## 3.1. Uncertainty quantification

### 3.1.1. Cure kinetics

The methods employed in [37] were compared to the new method proposed in this paper, which uses a standard 3 % variance of kinetic parameters. The kinetic parameters for RTM6 were varied by ±3 % to evaluate the impact to the reaction rate maxima. The results shown in Table 4 demonstrate that all parameters excepting $E_1$ and $E_2$ provide a satisfactory outcome when varied by 3 %. $E_1$ and $E_2$ both exceeded 10 % deviation to the maximum reaction rate, indicating that the parameters are unlikely to be varied as high as 3 % while still providing a good fitting to the actual reaction rate. This is supported by the actual measured variation of each parameter being 1 % as reported by [37]. Subsequently, the COV of each parameter was reduced until the fit falls within 10 % with new COV values for $E_1$ being 2 % and $E_2$ being 1.5 %. The half-COV measurements for these were 1 % and 0.75 % respectively.

Using the determined COV values, the three parameter uncertainty methods were compared, with the results in Table 5. All stochastic

**Table 3**
Set of stochastic analyses to be evaluated in this paper detailing the sources of variation and their limits.

| Analysis name | Cure kinetics variance | Temperature variance |
|---|---|---|
| CK-3 | 3 %* | None |
| CK-Half | 1.5 %* | None |
| T | None | Actual measurement (1.5 %) |
| T-5 | None | 5 % |
| All | 3 %* | Actual measurement (1.5 %) |

* Excepting parameters with reduced variances.

**Table 4**
Results of varying RTM6 kinetic parameters by ± 3 % on the maximum reaction rate. Also indicated are the parameters reported actual COV and an * identifying the stochastic variables from [37].

| Parameter | Reported COV | Reaction rate deviation, −3% | Reaction rate deviation, +3% | Notes |
|---|---|---|---|---|
| $\alpha_0$* | 19 % | −0.1 % | 0.1 % | |
| $A_1$ | 3.5 % | −0.9 % | 1.0 % | |
| $E_1$ | 1 % | ***13.5 %*** | ***−15.1 %*** | Rejected, > 10 % |
| | | 9.0 % | −9.4 % | Updated COV = ± 2 % |
| $n_1$ | 9 % | 0.0 % | 0.0 % | |
| $A_2$ | 2.6 % | 1.1 % | −0.6 % | |
| $E_2$* | 1 % | ***−20.0 %*** | ***16.4 %*** | Rejected, > 10 % |
| | | −9.5 % | 8.6 % | Updated COV = ± 1.5 % |
| $m$* | 7 % | 1.5 % | −1.0 % | |
| $n_2$ | 6 % | −2.7 % | 2.6 % | |
| $A_d$ | 4 % | 0.0 % | 0.0 % | |
| $E_d$ | 2 % | 0.0 % | 0.0 % | |
| $b$ | 11 % | 0.0 % | 0.0 % | |
| $w$ | 9 % | 0.0 % | 0.0 % | |
| $g$ | 19 % | 0.0 % | 0.0 % | |

**Table 5**
Comparison of three methods of determining parameters variance on predicting the average value and variance of the time for RTM6 to reach 88% cured.

| Cure cycle | Method | Average (min) | Standard deviation (min) | COV (%) |
|---|---|---|---|---|
| Dynamic | Deterministic solution | 94 | – | – |
| | COV from [37], 3 stochastic parameters | 93.85 | 1.26 | **1.34** |
| | COV from [37], all parameters are stochastic | 93.85 | 1.40 | **1.49** |
| | 3 % COV | 93.87 | 1.69 | **1.80** |
| | Half COV (1.5 %) | 93.84 | 0.90 | **0.96** |
| Standard Cure Cycle | Deterministic solution | 156 | – | – |
| | COV from [37], 3 stochastic parameters | 156.07 | 4.74 | **3.03** |
| | COV from [37], all parameters are stochastic | 156.08 | 5.27 | **3.38** |
| | 3 % COV | 156.38 | 6.48 | **4.14** |
| | Half COV (1.5 %) | 155.98 | 3.27 | **2.09** |

simulations converged to the deterministic solution, which confirms that the parameters variance doesn't distort the simulation results. The method from [37] resulted in a variance approximately halfway between the 3 % COV and half-COV evaluations shown here. This indicates that the method from [37] may align best to a variation of near 2.25 %. While the variance for the 3 % method is slightly higher than the method reported by [37], it still produces a satisfactory result.

As the 3 % variance method has been shown to be comparable to previous methods for the RTM6 resin, the same sensitivity analysis was performed on 5320-1. The results of this are in Table 6. Of note are the results for $E_1$, $E_3$, and $E_4$, which all produced variances which exceeded the 10 % threshold. As noted in the table, these parameter variances have been reduced to 2.5 %, 0.8 %, and 2 % respectively, with the half variances at 1.25 %, 0.4 %, and 1 %.

### 3.1.2. Temperature

Firstly, the range of temperatures measured during a standard cure profile for 5320-1 are shown in Fig. 4. The average variation between the runs was 1.5 %, which will be used as the temperature COV for this

**Table 6**
Results of varying 5320-1 kinetic parameters by ± 3 % on the maximum reaction rate.

| Parameter | Reaction rate deviation, −3% | Reaction rate deviation, +3% | Notes |
|---|---|---|---|
| $A_1$ | −0.6 % | 0.7 % | |
| $E_1$ | ***−16.6 %*** | ***10.3 %*** | Rejected, > 10 % |
| | −4.8 % | 6.0 % | Updated COV = ±2.5 % |
| $m_1$ | 1.0 % | −0.9 % | |
| $n_1$ | −0.9 % | 0.8 % | |
| $A_2$ | 0.1 % | −0.1 % | |
| $E_2$ | −2.2 % | 1.3 % | |
| $m_2$ | −0.1 % | 0.1 % | |
| $n_2$ | 0.0 % | 0.0 % | |
| $D_2$ | 0.0 % | 0.0 % | |
| $\alpha_{C0,2}$ | 0.0 % | 0.0 % | |
| $\alpha_{CT,2}$ | 0.0 % | 0.0 % | |
| $A_3$ | 1.8 % | −1.8 % | |
| $E_3$ | ***−45.9 %*** | ***26.3 %*** | Rejected, >10 % |
| | −9.9 % | 8.5 % | Updated COV = ±0.8 % |
| $m_3$ | −6.8 % | 6.2 % | |
| $n_3$ | −3.9 % | 3.6 % | |
| $A_4$ | 0.7 % | −0.7 % | |
| $E_4$ | ***−12.7 %*** | ***8.1 %*** | Rejected, >10 % |
| | −7.8 % | 5.8 % | Updated COV = ±2 % |
| $m_4$ | −2.6 % | 2.3 % | |
| $n_4$ | −0.3 % | 0.3 % | |
| $D_4$ | 0.0 % | 0.0 % | |
| $\alpha_{C0,4}$ | 0.0 % | 0.0 % | |
| $\alpha_{CT,4}$ | 0.0 % | 0.0 % | |

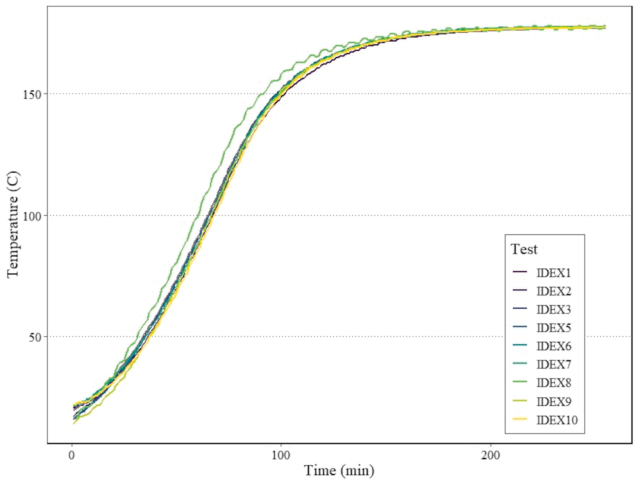


**Fig. 4.** Actual measured temperature profiles at the mid-plane of the 5320-1 IDEX panels.

study. While this variation is representative of the small oven used in this study, a 5 % variation will also be used to demonstrate the variation which is possible in larger ovens. IDEX8 demonstrates a slightly different temperature profile compared with the other tests and shows more fluctuation throughout the cure. At the completion of the cure, it was identified the laminate had shifted, and the part was cured under only one layer of breather material, in contrast with the other laminates which were cured under two layers. This variation resulted in a large impact on the heat transfer through the laminate and demonstrates another source of uncertainty which can impact composite cures.

## 3.2. Convergence analyses

The impact of cure kinetics and temperature uncertainty on the

**Table 7**
Results of the 5320-1 convergence analysis of viscosity modelling outputs, reflecting the impact of stochastic parameters.

| Stochastic parameter | Time at minimum viscosity | | | Time at gel point | | |
|---|---|---|---|---|---|---|
| | Avg | Std dev | COV (%) | Avg | Std dev | COV (%) |
| Deterministic Solution | 85 | – | – | 110 | – | – |
| CK-3 | 82.40 | 1.95 | **2.37** | 110.00 | 2.89 | **2.63** |
| CK-Half | 82.83 | 1.47 | **1.77** | 110.07 | 1.42 | **1.29** |
| T | 82.83 | 1.46 | **1.76** | 109.98 | 1.10 | **1.00** |
| T-5 | 82.40 | 1.97 | **2.38** | 110.19 | 3.76 | **3.42** |
| All | 82.40 | 1.99 | **2.42** | 109.98 | 3.12 | **2.84** |

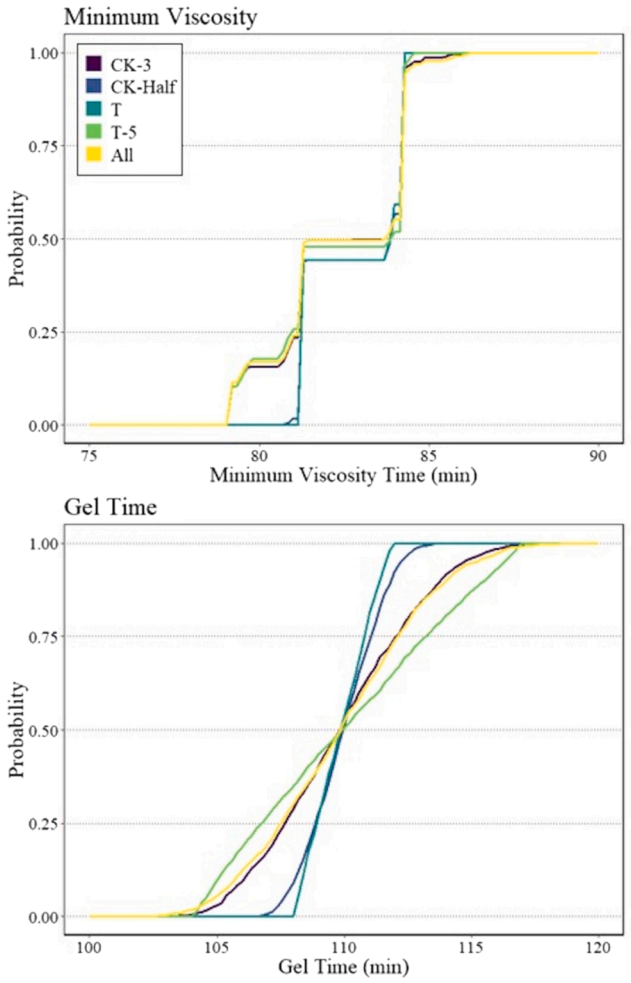


**Fig. 5.** Probability distributions for the stochastic cases for 5320-1 gel point (left) and minimum viscosity (right).

viscosity modelling outcomes is given explicitly in Table 7 and portrayed graphically in Fig. 5. All stochastic evaluations converge approximately to the deterministic solution, which confirms that the parameter variance is not drastically impacting the simulation.

The variance for all scenarios is low, indicating that the viscosity of this material system has a low sensitivity to cure kinetics and temperature variation. The highest variance is for the 5 % temperature COV on the gel point. For this case, the standard deviation of less than 4 min shows that the gel point of 5320-1 is very stable. It is also evident that the cure kinetics variation has a stronger impact on the output variance compared to the temperature. The 3 % variance in cure kinetics results in 2.63 % variance in gel point, where the 1.5 % variance in temperature only results in 1.00 % variance in gel point. When these two scenarios are combined (All) the variance is 2.84 %, which is largely dominated by the influence of cure kinetics uncertainty rather than the influence being additive. The magnitude of temperature variation influences the results, with the T-5 resulting in a 3.4 % variation on the gel point time, indicating that a higher temperature variation would likely contribute a proportionally stronger influence if coupled with the cure kinetics variation.

The results of the cure kinetics stochastic simulation are given in Table 8, with the probability distributions shown in Fig. 6. These output parameters follow similar trends to that of the viscosity outputs: cure kinetics and high temperature variations have a strong influence, low temperature variation has minimal influence, and cure kinetics influence dominates when combined with a low temperature variance. However, beyond this there are several items of note.

Firstly, the final degree of cure for all scenarios showed a low variance, indicating that the final degree of cure is stable for 5320-1. However, for CK-3, T-5, and All there are a number of parts which do not achieve the necessary degree of cure. Visually, the 88 % threshold is shown in Fig. 6 for the number of parts which are below 88 % in the probability distribution. The number of under cured parts is also detailed in Table 9, including the percentage of the total parts for this set. The most extreme scenario, which includes both cure kinetics and temperature variance, results in 5.8 % of parts being under cured.

Secondly, the variation of the time to fully cured is quite high for CK-3, T-5, and All, with variances of more than 10 %. While most parts met the minimum cure threshold for this study, the high COV indicates that there is a strong likelihood of under cure if an aggressive cure cycle were to be used. It should be noted that the output parameter of Time to Fully Cured only includes the parts which have achieved 88 % cured. This is reflected in the probability distribution in Fig. 6, which shows a final probability of less than 1 for several of the cases. The gap here is due to the under cured parts, which are quantified in Table 9.

The results of the probability distributions support the following recommendations for processing considerations for 5320-1:

1. Temperature variation for any given manufacturing conditions should be accurately determined and minimised where possible. Common equipment requirements allow for a 5 % variation of temperature within the oven or autoclave, with larger heating chambers and parts potentially having larger variations. If this translates to a 5 % variation of temperature within the part itself, a

**Table 8**
Results of the convergence analysis of 5320-1 kinetic modelling outputs, reflecting the impact of stochastic parameters.

| Stochastic parameter | Vitrification point | | | Time to fully cured | | | Final DOC | | |
|---|---|---|---|---|---|---|---|---|---|
| | Avg | Std dev | COV (%) | Avg | Std dev | COV (%) | Avg | Std dev | COV (%) |
| Deterministic Solution | 163 | – | – | 218 | – | – | 93.33 | – | – |
| CK-3 | 164.71 | 10.99 | **6.67** | 221.50 | 24.31 | **10.97** | 92.99 | 2.74 | **2.95** |
| CK-Half | 164.29 | 5.48 | **3.33** | 220.18 | 12.67 | **5.75** | 93.16 | 1.46 | **1.57** |
| T | 164.01 | 1.53 | **0.93** | 220.00 | 7.16 | **3.25** | 93.22 | 0.89 | **0.96** |
| T-5 | 164.15 | 5.12 | **3.12** | 221.49 | 23.32 | **10.53** | 93.08 | 2.90 | **3.12** |
| All | 164.47 | 11.25 | **6.84** | 219.67 | 23.61 | **10.75** | 93.11 | 2.91 | **3.13** |

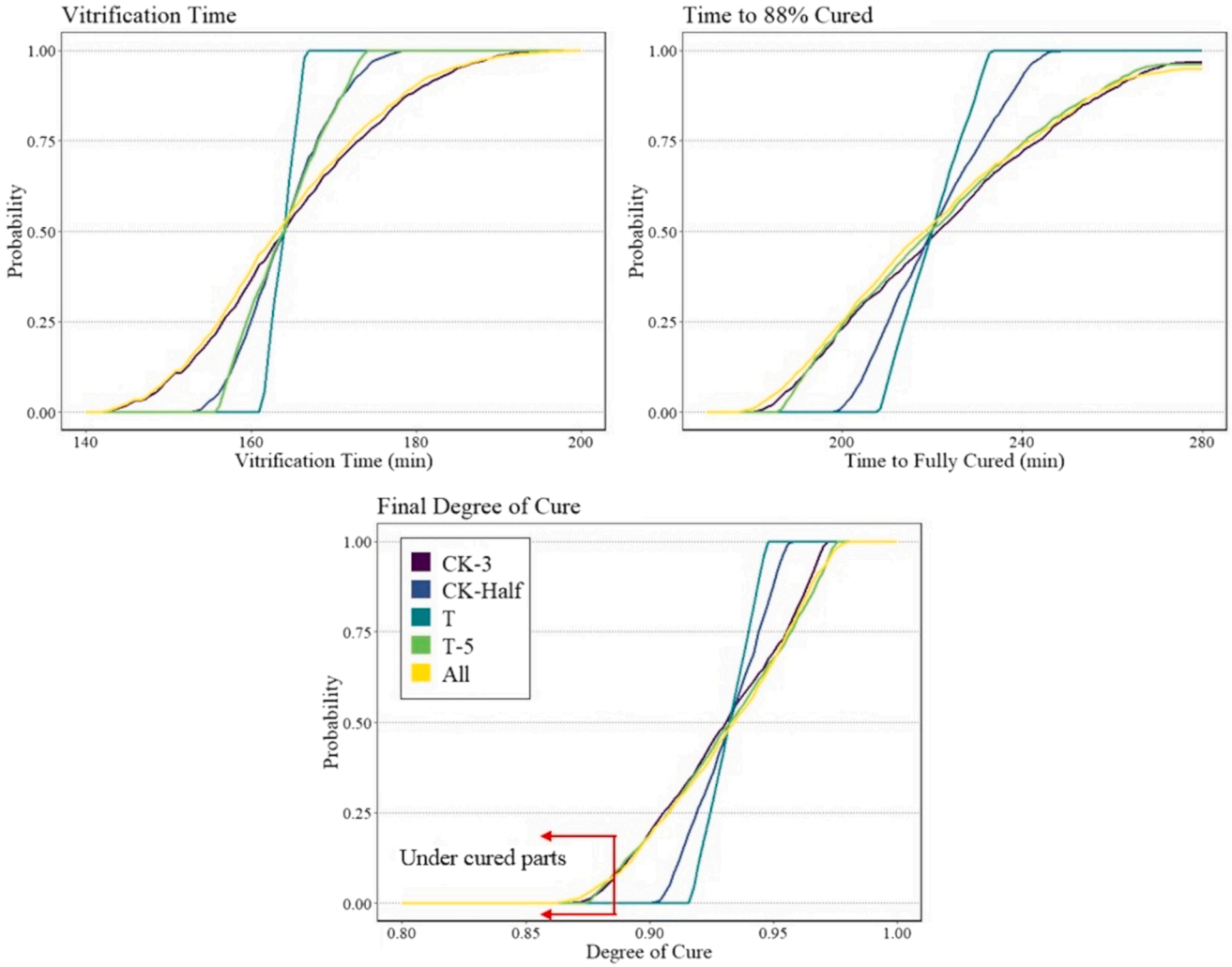


**Fig. 6.** Probability distributions for the stochastic cases for 5320-1 vitrification point (top left), time to fully cured (top right), and final degree of cure (bottom).

**Table 9**
Under cured parts (below 88% final degree of cure) for each stochastic scenario of 5320-1.

| Stochastic parameters | # Parts under cured (of 2000) | Percent parts under cured (%) |
|---|---|---|
| CK-3 | 55 | 2.75 |
| CK-Half | 0 | 0 |
| T | 0 | 0 |
| T-5 | 70 | 3.50 |
| All | 116 | 5.80 |

potentially large variation of final cure properties can result. As can be seen in Table 8, the final degree of cure for T-5 only varies by 3 %, however, the time to fully cured varies by over 10 %. While a longer cure may guarantee a satisfactory part, a shorter or optimised cure may be at risk of not meeting quality requirements.

2. The point of minimum viscosity and the gel point have low output variation, indicating that the material system is a robust choice for out of autoclave processing. In the worst-case scenario, there is a 15-minute window between the minimum viscosity and the gel. During this time the prepreg can achieve satisfactory volatile release, resin flow, and ply compaction prior to the gel event. However, if the early stages of cure are accelerated too quickly, this window may shorten and the resin may achieve gel prior to achieving sufficient consolidation, resulting in a part with high porosity which must be rejected. It is thus recommended that the compaction stage of the composite cure be accelerated with caution.
3. The time to fully cured displays a high amount of variation, and the process times should be treated conservatively. Shortening of process times may result in parts which are under cured, unless a direct cure monitoring method is used to evaluate the degree of cure progression [64]. Without directly monitoring the degree of cure it is possible that under cured parts are fabricated, despite complying with an approved cure cycle.

### 3.3. Experimental validation

The results from the cure tests of the $[0,90]_s$ laminates are given in Table 10, with the values being calculated in the same manner as the deterministic values provided in this paper.

As only one batch of prepreg was tested for this study, the cure kinetics variation will be disregarded. The source of variation which will be investigated here is oven temperature variance. The 1.5 % COV value will be used as this is representative of the actual variance measured for this oven. The results of the temperature convergence analysis compared with the experimental results is shown in Fig. 7.

All the values measured in Table 10 are consistent with the

**Table 10**
Results of the 5320-1 IDEX test temperature profiles, as calculated using the MATLAB code methodology in this paper.

| Part | Minimum viscosity (min) | Gel point (min) | Vitrification point (min) | Time at fully cured (min) | Final degree of cure (%) |
|---|---|---|---|---|---|
| IDEX1 | 86.2 | 113.2 | 168.6 | 223.2 | 93.3 |
| IDEX2 | 84.0 | 110.0 | 164.0 | 219.8 | 93.2 |
| IDEX3 | 82.2 | 110.2 | 165.0 | 217.8 | 93.6 |
| IDEX4 | 83.2 | 110.2 | 164.8 | 218.6 | 93.5 |
| IDEX5 | 84.2 | 110.6 | 165.2 | 218.6 | 93.3 |
| IDEX6 | 83.4 | 110.8 | 165.0 | 217.8 | 93.5 |
| IDEX7 | 83.2 | 111.4 | 165.2 | 217.2 | 93.7 |
| IDEX8 | 77.2 | 103.4 | 158.8 | 210.4 | 93.9 |
| IDEX9 | 82.2 | 111.4 | 165.4 | 219.0 | 93.5 |
| IDEX10 | 86.2 | 112.8 | 166.6 | 220.2 | 93.3 |

probability distribution predicted by this study. The minimum viscosity, gel time, and vitrification time values span the probability distribution ranges generated by the stochastic simulation. The final degree of cure and the time to cure are also aligned with the predicted values, however the actual range appears to follow a slightly tighter distribution than predicted. This indicates that the results of the stochastic model may be slightly more conservative than the experimental results. Additionally, the convergence analyses were conducted as a 0D simulation of the epoxy cure only, not in the presence of carbon fibres. The experimental validation was completed with 5320-1 prepreg, which has a fibre volume content of approximately 67 % [38]. The presence of the carbon fibres can influence the heat transfer in the epoxy and is a potential source of deviation between the experimental results and the convergence analyses. Additionally, IDEX8 appeared to be a slight outlier for some metrics, however it is noted in Fig. 4 that the temperature profile appears to be deviated from the rest of the test replicates. This has been attributed to the shift of the breather material during vacuum bagging and cure. This further demonstrates how slight variations in the cure configuration can impact the final part properties for identical oven programs.

## 4. Conclusion

CYCOM® 5320-1 epoxy/carbon fibre prepreg was evaluated using stochastic methodologies to capture the resulting variance due to cure kinetics and temperature uncertainty. A new proposed method for estimating parameters uncertainty provided a satisfactory result compared with methods which require extensive testing. This method is applicable to any known cure kinetics model, regardless of the model type or complexity. Further, these methods could be integrated into a finite element analysis scheme using a custom script, with the objective of evaluating the influence of cure kinetics uncertainty on complex 3-dimensional structures.

The impact of uncertainty on the resin viscosity and cure kinetics were demonstrated by a series of convergence analyses. For this material system, the impact of strongly varied cure kinetics or temperature conditions resulted in the highest amount of output variation. When compounded with a consistently low-variation oven the cure kinetics effect dominated, with the temperature effect only contributing slightly. Thus, it is important to capture the actual temperature variation expected for a given manufacturing scenario. The convergence analysis was compared with results from 10 cure cycles and confirmed that 1.5 % temperature uncertainty accurately represented the distribution of the given output parameters.

Cure cycle limitations for 5320-1 have been proposed, including recommendations on utilising direct-cure monitoring methods to ensure compliant parts are produced. Overall, 5320-1 displays robust viscosity behaviour which is suitable for an out-of-autoclave prepreg. However, optimisation of the cure process should be viewed with caution to minimise the chance for poorly compacted or under cured parts. Further, equipment temperature control should be well characterised so that large temperature variations are avoided, thus avoiding unintended product variability.

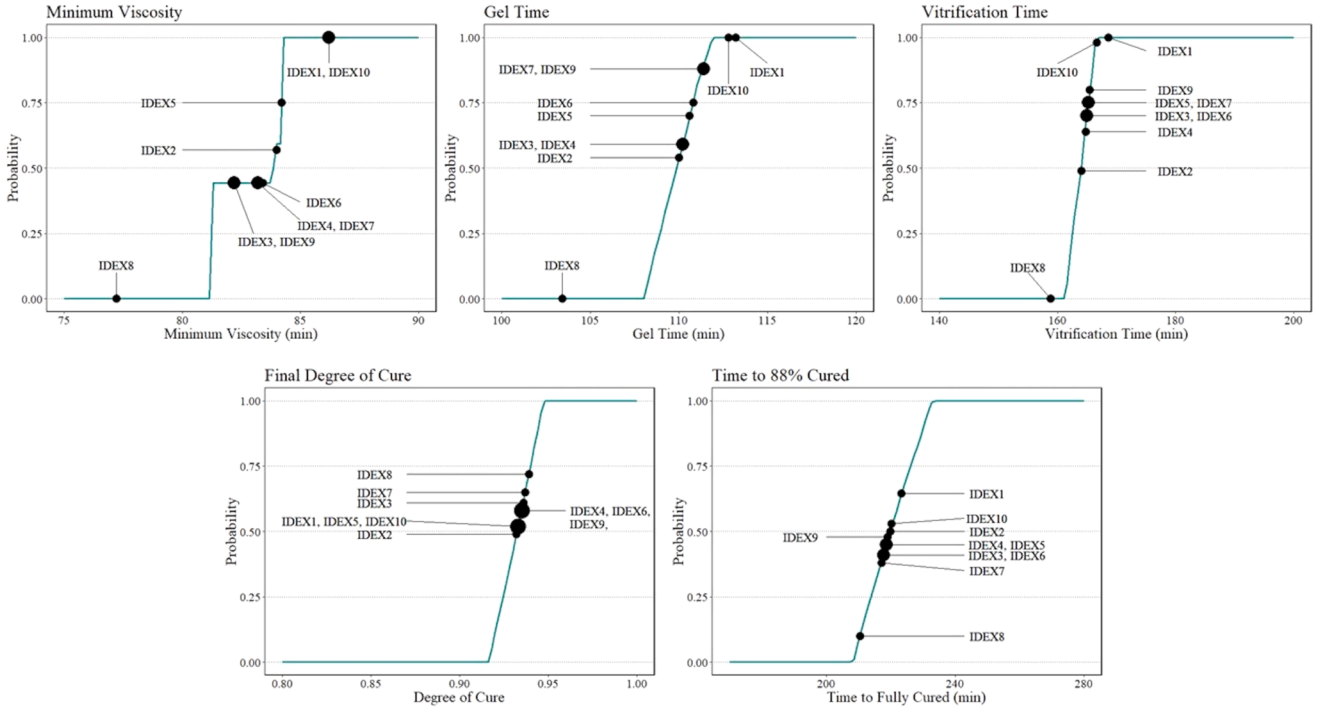


**Fig. 7.** A comparison of the distribution functions of the output parameters with actual measured values from 5320-1 IDEX panels from the results detailed in Table 10.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

Data will be made available on request.

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