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Composite Structures in Autoclave Processing**

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**Model Based Control Optimization for Curing the Shell-like  
Composite Structures in Autoclave Processing**

**ABSTRACT**

The paper studies a problem of optimal control synthesis for curing the shell-like composite structure using open die molding in an autoclave. The necessary control should eliminate early hard skin formation, emergence of resin-rich or resin-dry areas, insufficient consolidation, and uneven cure. This purpose can be achieved through providing uniform distribution of temperature and degree of cure within the cured part volume. The used approach, including the cure problem formulation, its finite element implementation where the correct timing of heating-up and isothermal hold should be found by the optimization procedure, is illustrated using the example of the shell-like composite part manufactured by means of two-stage curing in an autoclave. The control synthesis problem is formulated as a multi-objective optimization problem where minimization objectives are deviations of temperature and degree of cure within a cured part considering constraints imposed by thermal-kinetic properties of prepreg and manufacturing requirements. The Pareto-based optimization procedure and means of its results visualization allow estimating the best achievable quality indicators of manufactured composite parts, finding satisfactory parameters of the control law, and decision-making considering all imposed constraints.

Keywords: composite technology, multiobjective optimization, model-based optimal control, finite element analysis.

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## Introduction

The landscape of composite parts manufacturing has been spreading rapidly over the recent years. The growth in use of composites is the most intensive in aircraft and rotorcraft industries due to increased specific and fatigue strength or stiffness and better weight saving of these materials that sufficiently exceed the same properties of metal lightweight alloys. Most typical polymeric composites reinforced with glass/carbon fibers with the thermoset resin matrix are used in manufacturing of such parts as wing and tail skins, spars, stringers, ribs, fuselage sections, helicopters main and tail rotor blades, pressure vessels, radomes, cowlings, etc.

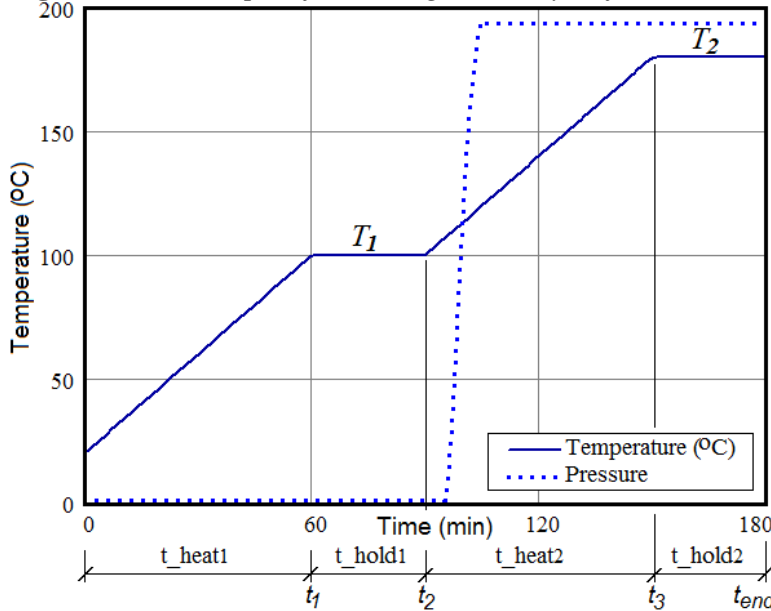
Most components based on long fibers, tows, yarn, or fabrics are manufactured using some kind of laminating procedure. In this process, sheets of reinforcement precoated with resin (prepreg) or freshly applied resin are forced against the surface of a mold under required conditions of pressure, temperature, and time. Many different technologies have been developed to produce aircraft components of different shape, dimensions, carrying loads, and wall thickness. A wide variety of components such as wing-body fairings, engine pylon fairings, radomes, and engine cowlings with open-shell geometry are widespread in aircraft structures [Baker].

The open-mold forming is the most suitable for components with such geometry. According to this technology involving the use of only one mold surface, over which the layers of fiber or fabric are tightly placed, a quasi-isotropic laminate is most often formed on the part surface. Such quasi-isotropic balanced laminate, which has equal in-plane mechanical properties, can be achieved using  $[0^\circ, +60^\circ, -60^\circ]$ ,  $[0^\circ, +45^\circ, -45^\circ]$ ,  $[0^\circ, +45^\circ, -45^\circ, 90^\circ]$ , or other scheme of laying-up [Mallik]. Then a mold packed with a raw material is placed into a vacuum bag, which is a flexible plastic film embracing the surface of the lay-up, and a vacuum-tight bag together with a mold is disposed in an autoclave.

The autoclave system includes a vessel containing a bag mold with laid-up prepreg, sources for heating the gas stream and circulating it uniformly inside the vessel, a subsystem for pressurizing the gas stream, a subsystem for applying vacuum to a part covered with a vacuum bag, and a subsystem for controlling operating parameters. Gas circulation inside an autoclave is essential to provide mass flow for temperature uniformity and heat transfer to the part load. The vacuum is applied to remove air and volatiles from the polymerized prepreg, while the pressure is required to consolidate all layers together into a laminate. At the first stage of heating the prepreg, resin viscosity in prepreg plies decreases, attains a minimum, and then increases rapidly (gelation stage) as the curing (cross-linking) reaction begins and proceeds toward completion. The temperature required to cure the thermoset resin is most often applied to the open mold using hot-air blowers and electric elements installed in an autoclave [Baker; Akovali].

Temperatures up to 180°C – 200°C may be required to complete the polymerization process of epoxy resin systems [Baker; Akovali]. Figure 1 shows a typical two-stage cure cycle for a glass / carbon fiber–epoxy prepreg.

**Figure 1.** An Example of Two-Stage Cure Cycle for a Glass Fiber–Epoxy Prepreg



The first stage of this cure cycle consists of increasing the temperature at a controlled rate up to the start of resin fluidization and dwelling at this temperature until the minimum resin viscosity is reached. Such an isothermal hold is applied at the stages of resin fluidization as well as at the process completion to allow the temperature distribution to become more uniform, especially in large components with thickness variations, to prolong the time allowed for volatiles removal, and to allow pre-reactions and consolidation of the resin [Mallik]. At the end of cure cycle after resin vitrification, the temperature is slowly reduced while the laminate is still under pressure. The ready laminate is removed from the mold and, in some cases, postcured at an elevated temperature.

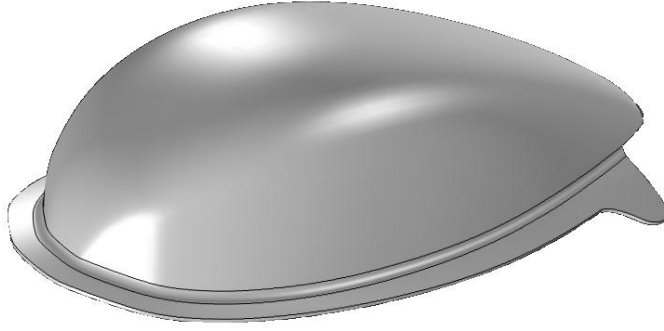
In order to eliminate such problems as overheating, early hard skin formation that prevents the release of gases, porosity, resin-rich areas, resin-dry areas, poor surface finish, insufficient consolidation, and uneven cure, the correct timing of application of temperature and pressure should be used. All means of process monitoring that support traditional autoclave curing can provide information on temperature, pressure, and sometimes resin viscosity (with DEA sensors placed in some points of the cured part). Unfortunately, these means cannot indicate the state of cure throughout the cured component. Due to this circumstance, the composite technology uses the trial-and-error approach to the tooling design and process development that causes the high cost of advanced composites. It is therefore very important to develop and use the methods of process modeling or simulation that can be used to predict a

desired tooling and process design window, within which a reduced number of trials can be conducted.

A good process model should correctly describe a number of coupled physical and chemical phenomena, including heat transfer, thermal, kinetic and rheological properties of material transformation at all stages of cure, and should take into account different constraints imposed by the process equipment, material properties, etc. This requires preliminary resin testing using DSC, DMA, DEA techniques to identify all resin properties during the full cure cycle. Due to the complex mathematical description of the problem and complexity of the components geometry, the finite element method is the most efficient and has been used recently by many authors to model a variety of polymeric composite cure processes [Park; Melnik; Slusar; Jiazhong; Liu; Oh]. Such finite element implementation of the cure process allows formulating an optimization problem where the degree of cure at the final stage of the curing process and variations of the degree of cure and temperature within the cured body during the whole process can be accepted as objects, whereas time dependencies of applied heat flow, temperature and pressure are design variables. Such an approach, which has been implemented in some recent works [Saad; Jahromi; Shevtsov; Koorevaar], allows synthesizing the most appropriate process control that provides the best quality of the cured part for the acceptable processing time as well as determining some process parameters, which are related to the state of the controlled plant and can be effectively monitored, e.g., temperature at some accessible points on the mold or cured part surface. The multi-objective approach to the problem optimization of such a system is the most efficient in practice when mapping of the area in the design space to the objective space is considered, and a pseudo optimal solution is selected, taking into account various constraints, including trade-offs between the full processing time, maximum allowable temperature gradient and curing uniformity.

Such a multi-objective optimization model based approach to the synthesis of the pseudo optimal process control is illustrated in this paper using an example of the cure process of glass fiber composite radar domes installed on the airframe (see Figure 2). The part with open-shell geometry and wall thickness varied in the range of 4 – 6.5 mm is manufactured by means of the open mold curing technology. The governing system of coupled heat transfer and thermokinetic equations for the prepreg is similar to that used in [Shevtsov] but it has different boundary conditions and dependencies of thermal and rheological properties on the temperature and degree of cure numerically identified by preliminary DSC, DEA testing of the studied material. Assuming a two-stage cure cycle, we optimize the duration of the first and second heating up and two dwelling sections to provide minimum variations of the degree of cure and temperature gradient within the cured part, while maximum duration of each cure stage and the whole cure cycle and temperatures at the first and second isothermal holds are constrained. We also study a possibility to improve the heating-up rate by varying thermal properties of the mold in order to verify the advice given by monograph [Baker].

**Figure 2.** A Modeled Composite Cowling with Open-Shell Geometry



### Modeling of Controlled Cure Process

The full problem statement includes the heat transfer equation

$$\rho_{c/m} C_{c/m} \partial T / \partial t + \nabla \cdot (-k_{c/m} \nabla T) = \begin{cases} Q_{exo} \\ 0 \end{cases}, \quad (1)$$

coupled with the kinetic equation, which is accepted for the considered prepreg in Kamal's form [Koorevaar; Kamal]

$$\partial \alpha / \partial t = A_2 \exp(-E_2 / RT) \cdot \alpha^m \cdot (1 - \alpha)^n. \quad (2)$$

Indices  $c$  and  $m$  near materials density  $\rho_{c/m}$ , specific heat capacitances  $C_{c/m}$ , and thermal conductivities  $k_{c/m}$  in equation (1) indicate the composite prepreg and mold respectively (see Fig. 3). Heat sources intensity  $Q_{exo}$  is the specific exothermal heat, which acts inside the composite body, while there are no any internal heat sources inside the mold. In kinetic equation (2) for the degree of cure  $\alpha$ , which is defined as the ratio of heat energy released during the partial reaction to the total enthalpy of the reaction  $Q_{tot}$ ,  $A_2$  is a pre-exponential factor,  $E_2$  is the activation energy,  $R$  is the universal gas constant,  $m, n$  are reaction orders. Equation (2) shows how the local value of the degree of cure depends on the temperature  $T$ , whose distribution is described by heat transfer equation (1). In turn, coefficients  $C_c, k_c$  and internal heat sources intensity  $Q_{exo}$  in equation (1) depend on the state of prepreg materials, which is a function of the degree of cure [Kamal].

$$Q_{exo} = Q_{tot} \cdot \partial \alpha / \partial t. \quad (3)$$

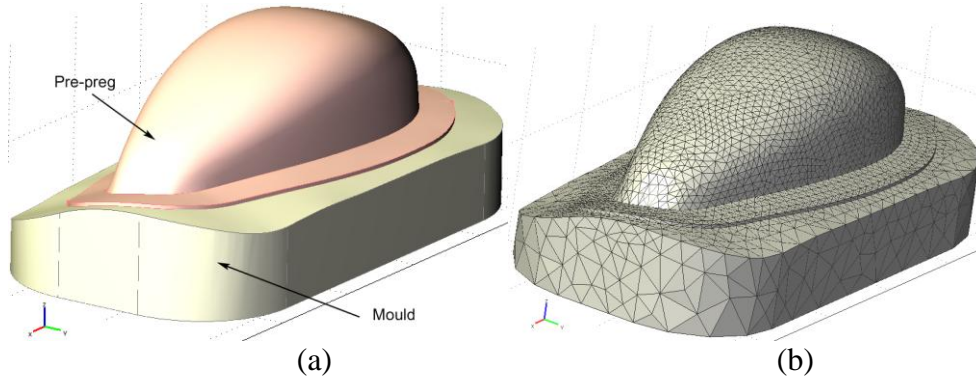
The dependencies  $C_c(\alpha), k_c(\alpha)$  have been determined by similar empirical formulas

$$P_c(\alpha) = P_c^{raw} - (P_c^{fpol} - P_c^{raw}) \cdot H(\alpha^{gel}, \delta \alpha), \quad (4)$$

where each thermal property of the cured material  $C_c(\alpha)$  or  $k_c(\alpha)$  depends on its value in the initial state before polymerization (index *raw*) and the value for the fully polymerized material (index *fpol*), and the first argument of the smoothed Heaviside function  $H$  is the degree of cure  $\alpha^{gel}$  at the start of

gelation. All these parameters describing the material thermal properties during cure have been identified using results of experimental studies.

**Figure 3.** *Geometry (a) and FE Meshing (b) of the Modeled System*



The initial condition for temperature  $T$  was accepted equal to  $20^{\circ}\text{C}$  everywhere in modeled domains. As equation (2) can only evolve from the initial value of  $\alpha(0) > 0$ , this value has been determined from the DSC study after numerical identification of  $\alpha(0)$  together with  $A_2, E_2, m, n$ . The boundary conditions that are common for the autoclave processing differ from those that are usual for the matched die technology [Jiazhong; Shevtsov]. All external surfaces, except the thermally insulated bottom surface of the mold, are exposed to the autoclave temperature that is controlled according to the law presented graphically in Fig. 1. In order to simplify the finite element implementation of the heat transfer problem we have neglected the thermal resistance of the vacuum bag thin film. The change of the prepreg volume  $V$  after applying pressure was neglected due to its insignificance.

Two temperatures  $T_1, T_2$  of dwelling sections have been assigned using DSC and DEA data, whereas four timings  $t_{heat_1}, t_{hold_1}, t_{heat_2}, t_{hold_2}$  (see Figure 1) are design variables that should be varied using the optimization procedure.

A significant difference in thickness between the shell-like cured part and the mold requires a very dense mesh (see Figure 3a). It makes the computational cost of the forward problem very high. To overcome this difficulty, we used the distributed solver of the Comsol Multiphysics FE package.

### Identification of Cured Prepreg Parameters

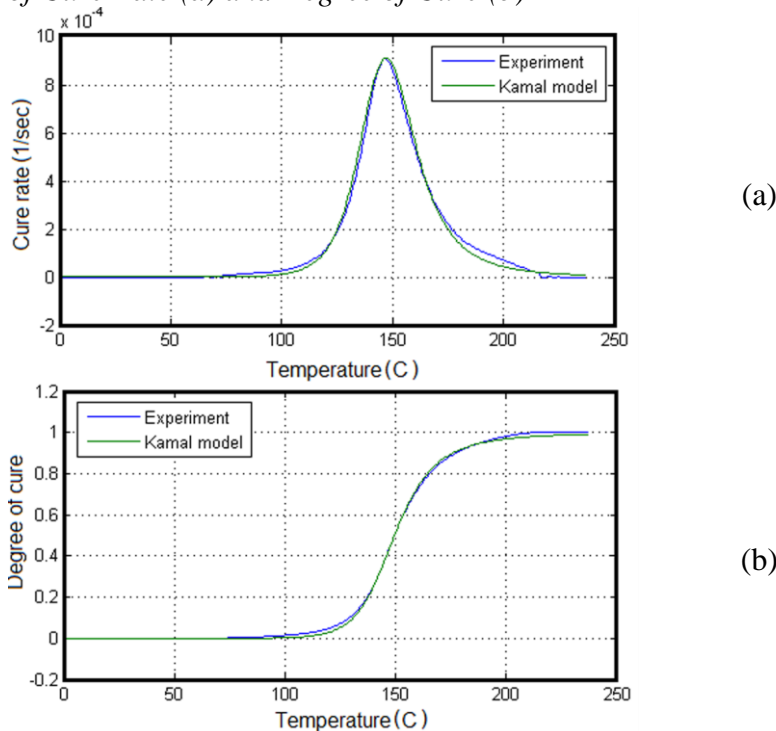
In order to determine four parameters of kinetic equation (2), an initial value of conversion  $\alpha(0)$ , total isothermal heat evolved during cure  $Q_{tot}$ , and heat capacitances of a prepreg with the minimum viscosity and solid state, the DSC experiments have been performed using the equipment NETZSCH DSC

200 F3 Maia. The studied samples of the prepreg were subjected to heating from  $-20^{\circ}\text{C}$  to  $250^{\circ}\text{C}$  with rates of 1, 1.5 and 2 K/min. After the second heating the DSC curves didn't have any peaks. That means the polymerization was completed at the first heating stage, i.e. the resin reached value 1 of the degree of cure. The dependencies of differential heat flow on temperature and time were exported and numerically processed to exclude the contribution of specific heat capacity to the total heat flow. To identify all five parameters of kinetic equation (2), we have used Genetic Algorithm Toolbox MATLAB<sup>®</sup> (see Figure 4), which minimizes the objective function according to the method presented in some studies [Hardis; Wu].

$$\min_{A_2, E_2, m, n, \alpha_0} \Phi = \int_0^{t_{end}} \left| \dot{\alpha}_{exp} - \dot{\alpha}_{mod}(A_2, E_2, m, n, \alpha_0, t) \right| dt, \quad (5)$$

where  $\dot{\alpha}_{exp}, \dot{\alpha}_{mod}$  are values of the cure rate experimentally observed and calculated from equation (2) respectively,  $t_{end}$  is the end time of DSC monitoring. For the studied composite material, the values of  $A_2, E_2, m, n, \alpha_0$  are present in Table 1.

**Figure 4.** Experimentally Observed and Modeled Temperature Dependencies of Cure Rate (a) and Degree of Cure (b)



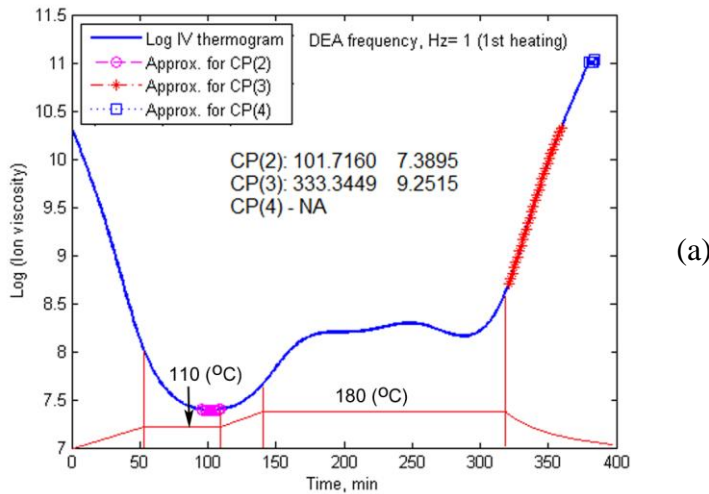


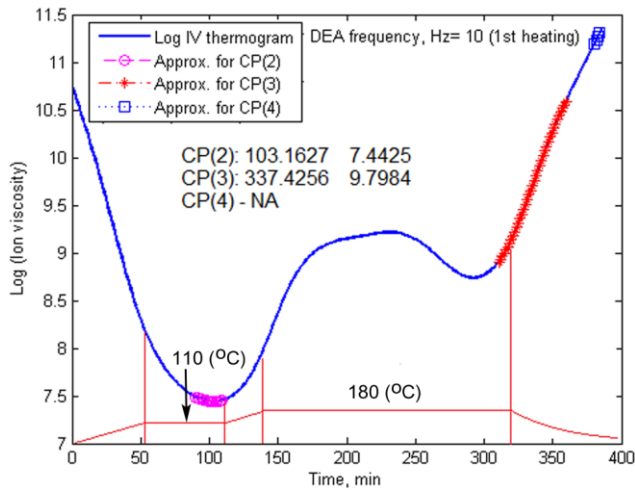
**Table 1.** Thermal-Kinetic Parameters of the Prepreg Identified using DSC Monitoring Results

$A_2$	$E_2$ (J/kg)	$m$	$n$	$\alpha(0)$	$Q_{tot}$ (J/kg)
9.66e-3	2.49e6	1.035	1.374	1.01e-6	1.106e5

To study the rheological transformation of the prepreg during cure, we have performed the dielectric monitoring using NETZSCH DEA 230/2 Epsilon analyzer with built-in software Proteus® that allows smoothing the high frequency noise generated by external electro-magnetic fields. The specimens were exposed to two-stage heating with varied heating rates on excitation frequencies of 1 and 10 Hz, which provided the best repeatability and resolution. Dependencies for the logarithm of ion viscosity after smoothing have been numerically processed to identify critical points of thermoset prepreg cure that determine the time instants when resin undergoes a phase transition [Lee], i.e. moments and temperatures at which the material changes its specific heat capacitances  $C_{c/m}$ , and thermal conductivities  $k_{c/m}$  as well as the point in time at which we need to apply the shaping pressure in an autoclave (see Figure 5). These points are: CP(2) is an ion viscosity minimum, which also corresponds to the mechanical viscosity minimum. This critical point indicates the moment at which the accelerating crosslinking reaction starts dominating the behavior of the system. CP(3) is an inflection point, which identifies when the crosslinking reaction begins to slow, and it is used as an indicator that is associated with gelation. CP(4) is a user defined slope predicting the end of cure.

**Figure 5.** Dependencies of Prepreg’s Ion Viscosity on Time at the DEA Scanning with the AC Frequencies 1 Hz (a) and 10 Hz (b)





(b)

The change of thermal conductivity during the transition from the viscous to the solid state of resin contained in the prepreg has been determined using the light flash apparatus NETZSCH LFA 467. This transition of thermal conductivity is characterized by the initial value 0.2 [W/(m×K)] (viscous state), by the value that matches the fully polymerized state 0.08 [W/(m×K)], and by the temperature range ~110 – 150°C.

All found thermal, kinetic and rheological parameters of the prepreg should be inserted in equations (1) – (4) and inequalities that determine the technological constraints imposed on the curing process schedule.

### Optimization of the Curing Process Control

The optimization procedure was organized as follows. The FE model of part cure was converted and included into the MATLAB script, which calls Comsol's binary file of the model for each set of design variables. The operating algorithm fills the area in the design space with points, mapping them to the area in the objective space. The calculation results were saved in a text file, which then was numerically analyzed to satisfy all imposed constraints and reconstruct the optimum areas. Four timings of the two-stage cure cycle (4D design space) were constrained by the inequalities  $t_{heat_1}, t_{heat_2} \leq 60$  min, and the full cycle duration was limited by the value 180 min due to manufacturing conditions

$$t_{end} = t_{heat_1} + t_{hold_1} + t_{heat_2} + t_{hold_2} \leq 180 \text{ min.} \quad (6)$$

Conversion at the end of cure cycle, which were calculated by integration and averaging over the cured composite volume  $\Omega$  was assumed to be no less than 0.98

$$\langle \alpha \rangle = \int_{\Omega} \alpha(\mathbf{r}, t_{end}) \cdot dv / V \leq 0.98 \quad (7)$$

Cure process objectives included two variables that should be minimized.

First objective is the maximum of the mean deviation of conversion during the cure cycle

$$\langle \Delta \alpha \rangle = \max_{t \in [0; t_{end}]} \int_{\Omega} |\alpha(\mathbf{r}, t) - \langle \alpha \rangle| \cdot dv / V. \quad (8)$$

The second objective is the mean deviation of the temperature at the first and second stages of heating-up. This objective, which allows to estimate the temperature uniformity within the cured prepreg, was calculated according to the following relationships

$$\langle \Delta T \rangle_{1,2} = \max_{t \in \left\{ \begin{array}{l} [0; t_1] \\ [t_2; t_3] \end{array} \right\}} \int_{\Omega} |T(\mathbf{r}, t) - \langle T(t) \rangle_{1,2}| \cdot dv / V, \quad (9)$$

where mean temperature at the heating stages of cure cycle

$$\langle T(t) \rangle_{1,2} = \int_{\Omega} T(\mathbf{r}, t) \cdot dv / V; \quad \text{at } t \in \left\{ \begin{array}{l} [0; t_1] \\ [t_2; t_3] \end{array} \right\}. \quad (10)$$

These values of both objectives calculated at the different control parameters are the points in the objective space that were analyzed to find the reasonable minimum of criteria (9) and (10).

The concept of Pareto optimality used here is widely applied in many industries to aid designers in their decision-making process [Tarantola]. However, the Pareto set and Pareto frontier for more than three objectives cannot be readily visualized using traditional 2D and 3D graphical means. Both objective and design space for the studied problem are multidimensional. All multidimensional visualization techniques attempt to transform a multidimensional problem or dataset so that it can be mapped to a 2D or 3D visual space. The three broad categories, which include the best-known methods, are [Agraval]:

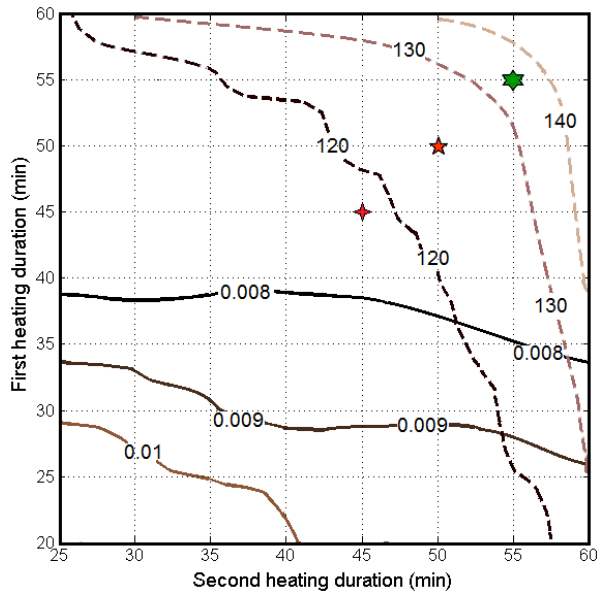
- Techniques based on 2D displays as well as multiple views of 2D displays.
- Techniques based on multivariate displays where colors and symbols are often used as a key to the representation.
- Techniques using time as an animation parameter.

In the considered case when a control law is parameterized by four parameters whose values completely characterize the thermal action on the cure system, two objective functions, and three constraints, the first approach is the most appropriate for providing insight into complex cure phenomena. We use some projections of the subset in the objective space to the 2D design subspace where these projections are drawn as level lines.

Some optimization results are present in Figures 6 – 10. Four, five, and six-pointed stars denote the temperature control laws, parameters of which are present in Table 2.

**Table 2.** Parameters of Three Studied Control Schedules

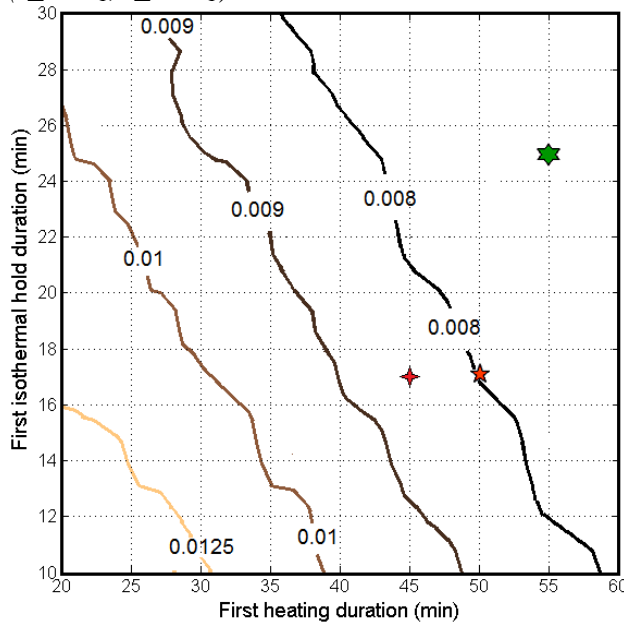
Schedule	Marker	Timings, min (see Figure 1)			
		$t_{heat_1}$	$t_{hold_1}$	$t_{heat_2}$	$t_{hold_2}$
1 <sup>st</sup>	★	45	17	45	13
2 <sup>nd</sup>	★	50	17	50	13
3 <sup>rd</sup>	★	55	25	55	15

**Figure 6.** Level Lines (Solid) for the Maximum Variation of Conversion within the Cured Part during the Whole Cure Cycle Combined with Level Lines (Dashed) for the Cure Cycle Duration (min). The Projections on the Plane ( $t_{heat_2}$ ,  $t_{heat_1}$ )


The level lines in Figure 6 have some irregularities due to finite number of forward problem simulations, which are about 5,000. The plots demonstrate that the heating rate at the first stage of heating-up has a much greater effect on the uniformity of conversion  $\langle \Delta \alpha \rangle$  than the heating rate at the second stage. It is a very important result, which proves that the precise temperature control is necessary even at the start of the cure cycle despite the fact that polymerization proceeds mainly at a later stage of the cure cycle.

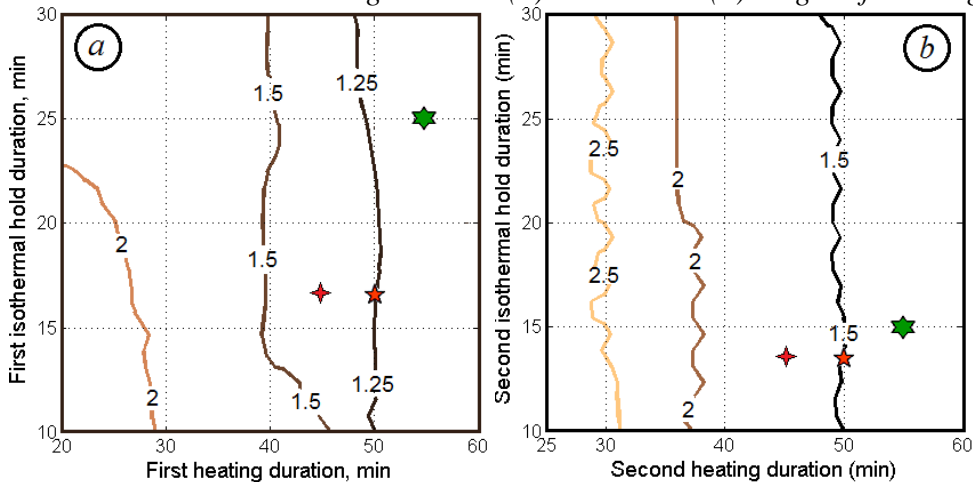
Plot for the deviation of conversion projected on the coordinates of control parameters ( $t_{heat_1}$ ,  $t_{hold_1}$ ) demonstrates a significant effect of these parameters on the homogeneity of spatial distribution of  $\alpha$ .

**Figure 7.** Level Lines for the Maximum Variation of Conversion within the Cured Part during the Whole Cure Cycle. The Projections on the Plane ( $t_{heat1}$ ,  $t_{hold1}$ )



The maximum deviation of temperature within the cured part at the first and second stages of heating has simpler dependence on timings (see Figure 8 a, b).

**Figure 8.** Level Lines for the Maximum Deviation of Temperature  $\langle \Delta T \rangle_{1,2}$  within the Cured Part during the First (a) and Second (b) Stages of Heating



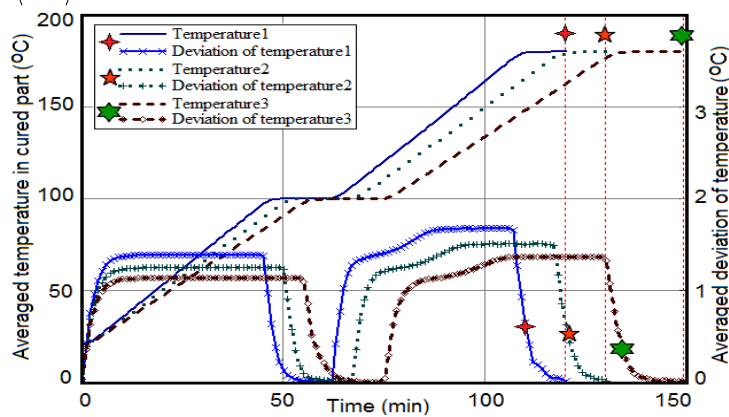
The contour plots with corresponding time histories for the average temperature and its deviation within the cured part (see Figure 9) demonstrate very weak dependence of temperature non-uniformity on the duration of isothermal holds. When the controlled heating-up is stopped, the temperature of

the prepreg is quickly and uniformly distributed due to high thermal conductivity of the aluminum mold. Figure 10 illustrates that the spatial variation of degree of cure undergoes a peak at the end of first heating when growing of  $\alpha$  is fastest. It confirms an importance of correct choice the first heating rate.

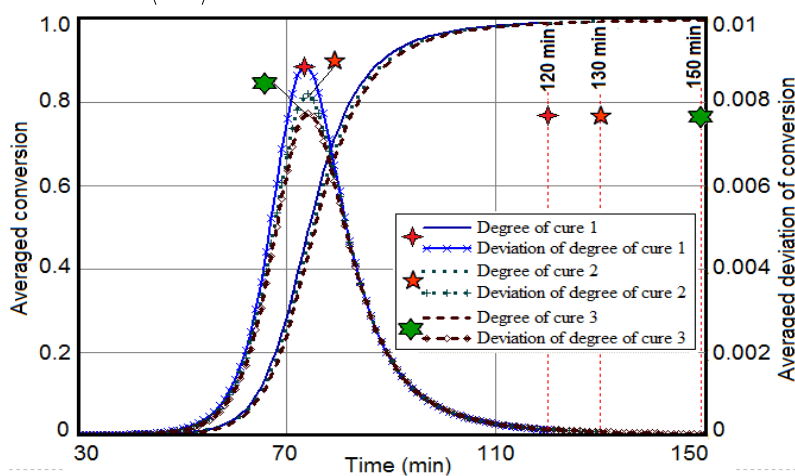
Our supplementary study of the curing process with a mold made of hard wood with sufficiently low thermal conductivity demonstrates significantly worse performance of the cure process, especially in terms of temperature uniformity. This result confirms the findings of the abovementioned study [Baker] and provides a quantitative assessment of the expected quality of the process. The plots in Figures 6 – 10 allow making a decision about the best choice of timing which is defined by the control law of the cure process.

We shall note that all contour plots in Figures 6 - 8 and time dependencies in Figures 9, 10 are valid for the accepted geometry and material properties of the mold. So, aforementioned considerations cannot be directly extended on other prepregs, parts geometries, and mould properties.

**Figure 9.** Time Histories for the Average Temperature  $\langle T \rangle$  and its Deviation  $\langle \Delta T \rangle$  within the Cured Part



**Figure 10.** Time Histories for the Average Degree of Cure  $\langle \alpha \rangle$  and its Deviation  $\langle \Delta \alpha \rangle$  within the Cured Part



## Conclusions

The problem of the optimal two-stage cure process control for the shell-like composite part, which is cured in a vacuum bag placed into an autoclave, has been formulated and numerically solved as a multi-objective optimization problem to provide the best performance of processing objectives, including minimum deviations of the temperature and degree of cure within the cured part considering constraints imposed by thermal-kinetic properties of the prepreg and manufacturing requirements.

The forward problem uses the system geometry exported from its CAD model. It describes the thermal-kinetic phenomena in the glass-fiber epoxy prepreg during the full cure cycle, and it is implemented as the FE model, whose input parameters represent the points in the design space. The outputs of the transient analysis of the model, which spreads over the full cure cycle duration, are the set of process objectives calculated by numerical integration at each time step over the volume of the cured composite part.

All thermal, kinetic and rheological properties of the cured material that appear in the model equations have been preliminary determined using data of DSC, DEA, DMA experimental techniques and procedures of numerical identification.

The Pareto-based optimization procedure was controlled by the MATLAB script calling the binary file of the forward problem for each set of design variables. This algorithm performs mapping of the area in the design space to the area in the objective space. For the clarity of the control law optimization results, we used the multiple views of 2D plots, which are projections of the objective subset to the 2D design subspaces where these projections are drawn as the level lines.

Our study of the effect of the mold thermal properties on the cure process quality and performance has found that the best controls can be obtained at the maximum thermal conductivity of the mold materials. It is especially true for the open die molding.

The present modeling and optimization approach to the cure process control of the prepreg with thermosetting resin as well as the means of optimization results visualization allow providing insight into complex curing phenomena, estimating the best achievable quality indicators of manufactured composite parts, finding satisfactory parameters of the control law, and making a decision taking into account all manufacturing constraints.

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