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Semi-analytical study of thick polymer composites behavior during curing process

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ABSTRACT

Composite materials show different mechanical properties in different directions which leads to complications in the prediction of their behavior. In addition to being inhomogeneous, behavior of composite materials is highly dependent on the curing process and many studies have tried to address these issues. Residual stress can induce high errors in prediction of composite material properties and leads to a difference between theoretical and experimental results. It has been observed that the humidity of environment and the chemical properties of composite materials are also important. Fewer studies have addressed thick layer composites and no damage theory has been developed for these materials. In this paper residual stress in thick layer composites have been studied. Using numerical analysis, the curing process has been modeled and mechanical properties of a thick layer composite are obtained and the induced residual stress in these composites has been assessed. Unlike thin layer composites, the stress in each layer is not specific and a tensile and compressive stress field can exist simultaneously in thick layer composites.

1. Introduction

Residual stress is the stress experienced without the presence of external or thermal loading (Callister & Rethwisch, 2007). Residual stresses may be created through the manufacturing process or working conditions (Ghasemi & Shokrieh, 2010). Residual stresses lead to matrix cracking, structural delamination, fiber tear and twist of asymmetrical multilayers and are highly effective in decreasing the structure strength (Kim & Mai, 1998). Shokrieh and Ghanei (2010) used a circular single-fibered element for investigating the residual thermal stresses in their Finite element modeling. Inter-phase properties were modeled as polynomial, periodic, cubic along radius direction. They extended the model to multi-fiber and concluded that the results are compatible with proper boundary conditions. Composites are divided into thin and thick layered ones. This classification is due to the different effects caused by residual stress. In fairly thick composites (up to 2.54 cm thickness), the composite behavior is similar to thin composites, but in thick composites (7.5 cm thickness) due to difference in temperature during curing level in different layers, results are very different with thin composites. Lateral residual stress distribution in uni-lateral composites is such that in middle sections stress is tensile and in outer sections is compressive. The stress profile changes twice in the curing process. Residual stress in thin layer composites is mainly derived using elastic models which are only dependent on cooling after curing (Bogetti & Gillespie, 1992).

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Bogetti and Gillespie (1992) studied the residual stress in thick layer composites for the first time. They used one-dimensional elastic model for simulating the curing. The effects of curing history, layer thickness and resin expansion has been considered in residual stress prediction. Mechanical properties were considered to be dependent on the curing process. They showed that the residual stress during curing may be high such that it causes the failure of composite. This method is not applicable to thermoset composites during curing. Bermejo et al. (2006) studied the effect of residual stress on the strength, toughness and fracture of layered ceramics with different thickness ratios. Based on threshold strength and energy absorption capability of crack burification mechanism at fracture, the optimum layered design was discussed in their study. Shokrieh and Safarabadi (2012) found the micromechanic residual stress in thin layer polymeric composites by considering the properties of middle phase averagely and get the total residual stress by gathering these stresses and macromechanic residual stresses. Shokrieh et al. (2012) first considered a single fiber element in a finite element simulation to assess the thermal residual stress. The interphase was considered to be exponential, periodic and third degree in the radial direction. This model was generalized for multiple fibers. They concluded that the answers are correct when considering correct boundary conditions and not considering the interphase leads to great errors. Nielsen et al. (2013) showed the main factors of distortions caused during the process are large cure gradients. They used a three dimensional thermomechanical model in ABAQUS and investigated different modelling approaches such as cure hardening instantaneous linear elastic, viscoelasic and path-dependent approaches. Safarabadi and Shokrieh (2014) presented a new method for combining macro-mechanical and micromechanical residual stresses caused by curing. They compared the results from this study with the results of a drilling test carried out previously. The results show that neglecting micromechanical residual stresses can lead to high errors in predicting the residual stress of each layer. Telford et al. (2014) studied unsymmetrical composite laminates by analyzing through-thickness residual stresses at dry and saturation states and proposed a combined experimental–numerical analysis to study the effect of moisture ingress. Their study shows that moisture ingress can affect through-thickness residual stresses and thus the shapes of unsymmetric laminates. A three-dimensional thermo-viscoelastic model is used by Ding et al. (2015) to simulate the residual stress in composite laminates during curing. They used a differential constitutive law. They found that the aluminum skins have negligible effects on the cure of composite laminates and significant effects on the residual stress development. Hu et al. (2018) experimentally investigated non-uniform gelation with characterization of necessary material properties. They demonstrated that the high temperature layer gelled earlier and showed more chemical shrinkage.

In this study residual stresses are caused by composite processing. A two-dimensional thermo mechanical analysis is presents an implicit finite difference method in order to predict thick polymer composite residual. The equations are coupled. A MATLAB program is used to solve these equations. Afterwards the elastic modulus of the composite through the process is calculated. After finding the stiffness matrix and using superposition for the strains, the stress is calculated. Results show two exothermic reactions during curing. Residual stress obtained in this study differs only slightly from Bogetti's solution.

2. Micromechanical residual stress prediction in thick layer composites

In this section, the factors leading to residual stress in thick layer composites are discussed. Thickness over 2.54 cm is considered thick. A thermo-chemical 2D implicit finite element model is used to predict the temperature and curing degree during the process. In this paper, the residual stresses in unidirectional composites as shown schematically in Fig. 1 are discussed. In these composites, residual stress is only dependent on layer thickness. If single layer composite is cured uniformly and curing degree and mechanical properties are uniform, no residual stress would be observed. If the curing is not uniform, residual stresses are observed.

2.1 Thermo-chemical model

A thermo-chemical model is used to specify temperature and curing degree inside a part. The Fourier equations in Cartesian space are as follows (Bogetti & Gillespie, 1992):

$$\rho C_p \frac{\partial T}{\partial t} = K_1 \frac{\partial^2 T}{\partial x_1^2} + K_2 \frac{\partial^2 T}{\partial x_2^2} + K_3 \frac{\partial^2 T}{\partial x_3^2} + \dot{q}, \tag{1}$$

where $\dot{q}$ is the heat created inside the composite due to chemically pyrogenic reactions inside the epoxy resin. C_p, ρ and K_i are the specific thermal capacity, density and heat conduction factor of composite which are assumed to be constant. Their values in this study are shown in Table 1 (Bogetti & Gillespie, 1992).

Table 1. Thermo-chemical properties of epoxy (Bogetti & Gillespie, 1992)

Property	*Value*
$\rho\left({}^{Kg}/_{m^3}\right)$	**1578**
$C_p\left({}^{J}/_{Kg.K}\right)$	**862**
$H_T\left({}^{J}/_{g}\right)$	**199**
$K_2 = K_3\left({}^{W}/_{m.k}\right)$	$\mathbf{414 \times 10^{-3}}$

If the length in the direction of x_1 is large, the heat transfer in this direction is negligible and the energy balance equation is two-dimensional and is shown as Eq. (2) (Bogetti & Gillespie, 1992).

$$\rho C_p \frac{\partial T}{\partial t} = K_2 \frac{\partial^2 T}{\partial x_2^2} + K_3 \frac{\partial^2 T}{\partial x_3^2} + \dot{q} \tag{2}$$

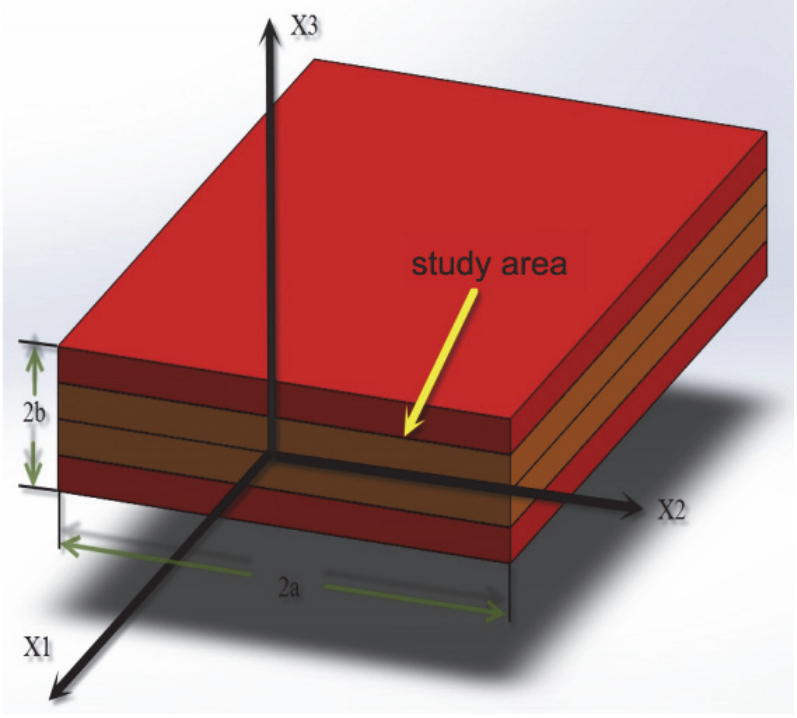

Fig. 1. Schematic of multilayer composite

The internal heat generated is a function of curing degree (Bogetti & Gillespie, 1992).

$$\dot{q} = \rho H_R \frac{d\alpha}{dt}, \tag{3}$$

$$H_R = \int_0^{t_f} \frac{dQ}{dt} dt, \tag{4}$$

$$H = \int_0^{t} \frac{dQ}{dt} dt, \tag{5}$$

where H_R is the heat of the reaction until time t. $d\alpha/dt$ is the curing degree in each instant. The curing degree is a function of H_R and H (Chamis et al., 1973).

$$\alpha(t) = H/H_R = \left(\frac{dQ}{dt}\right)/H_R = \int_0^t \frac{d\alpha}{dt} dt. \tag{6}$$

The experiments of Lee and Springer (1982) led to $H_R = 473.6 \pm 5.4\, J/g$. The reaction heat is equal to the area between heat flow and base line which equals to $\int_0^t \left[dH/dt\right] dt$ (Chamis et al., 1973).

The relationship between the cure rate and reaction heat is a nonlinear function of heat, history and resin properties. The instantaneous cure rate is usually presented as a function of cure rate and temperature (Chamis et al. , 1973).

$$\frac{d\alpha}{dt} = f(\alpha, T), \tag{7}$$

in which α is the cure rate and T is temperature. In this study, the model presented by Lee and Springer (1982) is used.

$$\begin{aligned} \frac{d\alpha}{dt} &= (K_1 + K_2\alpha)(1-\alpha)(B-\alpha) \quad for\ \alpha \le 0.3 \\ \frac{d\alpha}{dt} &= K_3(1-\alpha) \qquad\qquad\qquad for\ \ge 0.3 \end{aligned} \tag{8}$$

K_1, K_2 and K_3 are found using

$$K_i = A_i exp\left(\frac{-\Delta E_i}{g_c T}\right) \qquad i = 1,2,3 \tag{9}$$

g_c is the gas constant. A_i and ΔE_i (activation energy) for carbon/epoxy composite is shown in Table 2. T is the absolute temperature. B is a constant found to be 0.47 and independent of temperature based on experiments on cure rate (Lee and Springer, 1982).

Table 2. Cure kinetic constant for carbon/epoxy composite (Lee and Springer (1982))

major equation constant	***value***
$A_1\ (min^{-1})$	$\mathbf{2.1 \times 10^9}$
$A_2\ (min^{-1})$	$\mathbf{-2.1 \times 10^9}$
$A_3\ (min^{-1})$	$\mathbf{1.96 \times 10^5}$
$\Delta E_1\ (J.mol^{-1})$	$\mathbf{8.07 \times 10^4}$
$\Delta E_2\ (J.mol^{-1})$	$\mathbf{7.78 \times 10^4}$
$\Delta E_3\ (J.mol^{-1})$	$\mathbf{5.66 \times 10^4}$

Integrating Eq. (8), leads to:

$$t = \frac{a}{K_2}\ln\left(1+\frac{K_2}{K_1}\alpha\right) - b\ln(1-\alpha) - c\ln\left(1-\frac{\alpha}{B}\right), \tag{10}$$

$$t = -\frac{1}{K_3}\ln\left(\frac{1-\alpha}{0.7}\right) + t_c, \tag{11}$$

in which:

$$\begin{aligned} a &= K_2^2(B-1)/d, \\ b &= \frac{K_1 + K_2 B}{d}, \\ c &= -\frac{K_1 + K_2}{d}, \\ d &= (K_1+K_2)\{K_1 B^2 + (K_1 - K_2)B - K_1\}. \end{aligned} \tag{12}$$

In Eq. (11), t_c is calculated for $\alpha = 0.3$ in equation 10. Eqs. (2-5) and Eq. (8) are a set of differential equations for heat energy flow through curing. Four boundary conditions for temperature and two initial conditions (for temperature and cure rate) are required. The surface temperature throughout the cycle is known:

$$T(a, x_3, t) = T_s(t), \tag{13}$$

$$T(x_2, b, t) = T_s(t), \tag{14}$$

in which $T_s(t)$ is surface temperature in t. using symmetry conditions, only a quarter of the model is used. The two temperature boundary conditions are:

$$\frac{\partial T(0, x_3, t)}{\partial x_2} = 0\,, \tag{15}$$

$$\frac{\partial T(x_2, 0, t)}{\partial x_3} = 0. \tag{16}$$

Initial temperature conditions and curing degree are found using geometry:

$$T(x_2, x_3, 0) = T_0, \tag{17}$$

$$\alpha(x_2, x_3, 0) = \alpha_0, \tag{18}$$

where T_0 and α_0 are the temperature and initial curing degree. These equations have no analytical solution and a recursive finite difference method is used. Fava (2005) developed this method. This method uses a finite difference method with a time step of $\Delta t/2$. The first equation is solved implicitly in the direction of x_2 :

$$\frac{T_{ij}^* - T_{ij}^n}{\Delta t/2} = \delta_{x_2}^2 T_{ij}^* + \delta_{x_3}^2 T_{ij}^n + \dot{q} \tag{19}$$

in which T_{ij}^* is the temperature between $t = t_n + \Delta t/2$ and T_{ij}^n in the instant $t = t_n$ and Δt is the time. The second equation in the direction of x_3 similarly equals:

$$\frac{T_{ij}^{n+1} - T_{ij}^*}{\Delta t/2} = \delta_{x_2}^2 T_{ij}^* + \delta_{x_3}^2 T_{ij}^{n+1} + \dot{q} \tag{20}$$

$$\delta_{x_2}^2 T_{ij} = \frac{T_{i-1,j} - 2T_{ij} + T_{i+1,j}}{(\Delta a)^2} \tag{21}$$

$$\delta_{x_3}^2 T_{ij} = \frac{T_{i,j-1} - 2T_{ij} + T_{i,j+1}}{(\Delta b)^2} \tag{22}$$

In order to find T_{ij}^*, Eq. (20) is solved and replaced into equation 20. T_{ij}^{n+1} Can be found at the end of Δt.When only considering heat transfer in the direction of x_3 Eq. (2) yields:

$$\rho C_p \frac{\partial T}{\partial t} = K_3 \frac{\partial^2 T}{\partial x_3^2} + \dot{q} \tag{23}$$

The curing process and temperature T_s is shown in Fig. 2 based on producers' advice. The part is held initially in $116^{\circ}C$ for an hour and then the temperature increases stepwise by $2.5^{\circ}C$ degrees per minute and after reaching $177^{\circ}C$ the temperature is considered constant for two hours. The initial temperature is $T_0 = 25^{\circ}C$ and the part is initially raw so $\alpha_0 = 0$.

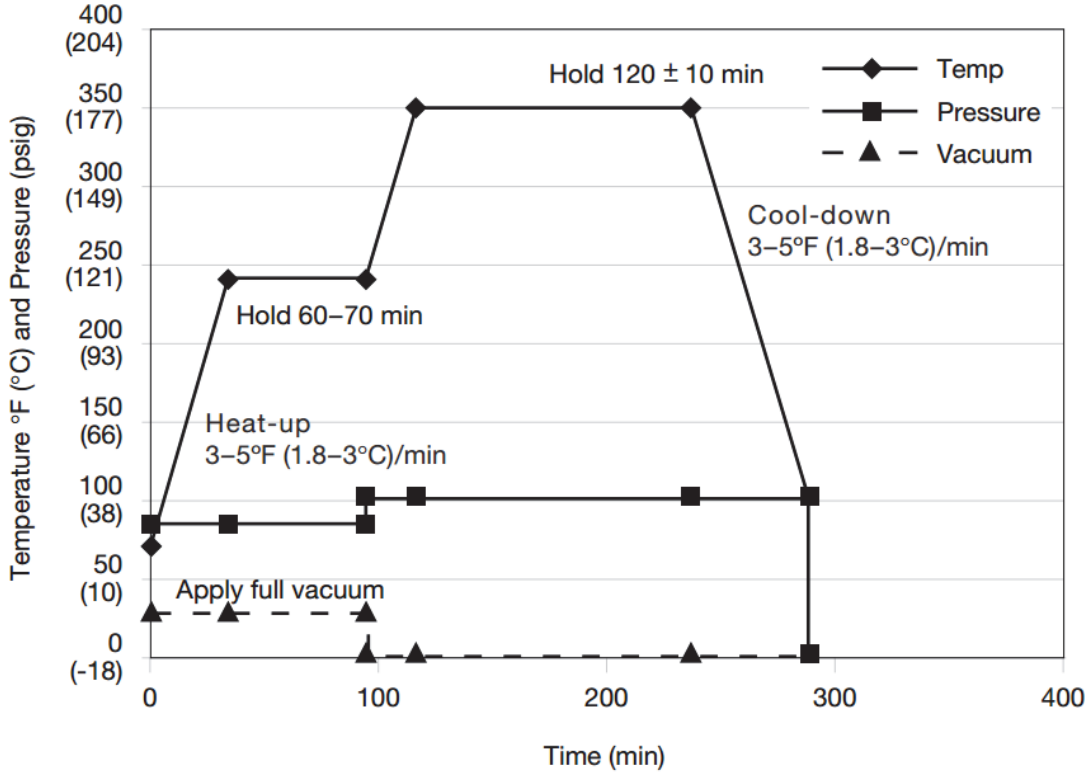

Fig. 2. The producers plan for curing carbon/epoxy composite (Almeida, 2005)

Based on the mentioned points for solving Eq. (20), an implicit equation comprised of an ODE is solved by:

1. Solving Eq. (8) and finding the curing degree based on time and temperature
2. Finding $\frac{d\alpha}{dt}$ only based on only time and temperature
3. Calculating the integral in Eq. (4)
4. Calculating Eq. (3) to find $\dot{q}$ based on time and temperature
5. Solving Eq. (2) and finding temperature based on time

2.2 The dependence of composite module to curing temperature

The composite module through the curing process is an indicator of mechanical properties. Thick layer composite modules are highly dependent on cure degree, which is effected by viscoelastic properties and reaction kinetics. The implicit equation is as follows (Bogetti & Gillespie, 1992):

$$E_m = (1-\alpha_{mod})E_m^0 + \alpha_{mod}E_m^\infty + \gamma\alpha_{mod}(1-\alpha_{mod})(E_m^\infty - E_m^o) \tag{24}$$

in which:

$$\alpha_{mod} = \frac{\alpha - \alpha_{gel}^{mod}}{\alpha_{diff}^{mod} - \alpha_{gel}^{mod}} \qquad (-1 < \gamma < 1) \tag{25}$$

E_m^0 and E_m^∞ are raw resin module and fully cured resin module, which are considered to be constant (given in Table 3). γ Is the coefficient of the effect of each stress release factor and resin chemical hardness. It is assumed that $\alpha_{diff}^{mod} = 1$ and $\alpha_{gel}^{mod} = 0$ (Bogetti & Gillespie, 1992).

Table 3. Resin properties during curing (Bogetti & Gillespie, 1992)

Mechanical property	***Resin(epoxy)***
$\boldsymbol{E_m^0(MPa)}$	*3.447*
$\boldsymbol{E_m^\infty}$	*3.447*×10^3
$\boldsymbol{V_{sh}^T}$(%)	*1-3*

2.3 Resin chemical volumetric expansion model

The resin undergoes chemical strain during curing which leads to internal stresses. These chemical stresses are solved using the following equation (Bogetti & Gillespie, 1992).

$$\Delta\varepsilon^{sh} = \left(\sqrt[3]{1+\Delta V_r}\right) - 1 \tag{26}$$

$$\Delta V_r = \Delta\alpha \times V_{sh}^T \tag{27}$$

Self-compatible micromechanical equations are widely used to calculate unidirectional composite mechanical properties (Mulle et al., 2009). In this study, a method similar to that of Garstka et al. (2007) is used in which N is the number of layers and the mechanical properties of the N^{th} layer is found. Superposition is used to find the composites properties.

$$[C_{eff}] = \sum_{m=1}^{N} (f_m [T]_m^T [C_m][T]_m) \tag{28}$$

where C_{eff} is the effective stiffness of each cell, f_m is the percentage of fiber in the N^{th} layer of composite and $[T]_m$ is transformation matrix from the local coordinate system for the N^{th}layer to the general coordinate.

2.4 Effective thermal expansion coefficient and composite chemical expansion

The strain due to thermal expansion and chemical contraction is dependent on fiber and resin physical properties and fiber volume percentage. A simple model is used to calculate the effective thermal expansion and chemical contraction of the composite (Leng & Asundi, 2002):

$$\mathcal{E}_1 = \frac{\mathcal{E}_1 E_{1f} + \mathcal{E}_1 E_{1r}(1-f)}{E_r f + E_{1r}(1-f)} \tag{29}$$

$$\mathcal{E}_2 = \mathcal{E}_3 = \left(\mathcal{E}_{2f} + \nu_{12f}\mathcal{E}_{1f}\right)f + (\mathcal{E}_{2r} + \nu_{12r}\mathcal{E}_{1r})(1-f) - \left[\nu_{12f} f + \nu_{12r}(1-f)\left[\frac{\mathcal{E}_{1f}E_{1f}f + \mathcal{E}_{1r}E_{1r}(1-f)}{E_{1f}f + E_{1r}(1-f)}\right]\right] \tag{30}$$

In this study the fiber chemical expansion is considered to be zero.

5.2 Predicting residual stress

In this study thermal stress, stress due to chemical contraction and external load stress are obtained. By finding the strain due to the mentioned factors, stress can be calculated (Bogetti & Gillespie, 1992).

$$\Delta\varepsilon = \Delta\varepsilon^m + \Delta\varepsilon^{th} + \Delta\varepsilon^{sh} \tag{31}$$

where $\mathcal{E}^m$ is the strain due to external forces. $\Delta\varepsilon^{th}$ Is the thermal strain.

$$\Delta\varepsilon^{th} = \alpha \times \Delta T \tag{32}$$

The stress in the composite is found using the following equation:

$$[\Delta\sigma] = \left[C_{eff}\right]\{[\Delta\varepsilon^m] + [\Delta\varepsilon^{th}] + [\Delta\varepsilon^{sh}]\} \tag{33}$$

3. Results and Discussion

In the following, the results of thick composite curing degree, multilayer temperature, mechanical properties and residual stresses have been shown.

3.1 Curing degree

In Fig. 3, the curing degree rate based on temperature and curing degree, which are found from equation 8, are shown. For $\alpha > 0.3$ the curing degree rate stays constant with temperature. The curing degree rate decreases with increasing temperature and curing degree.

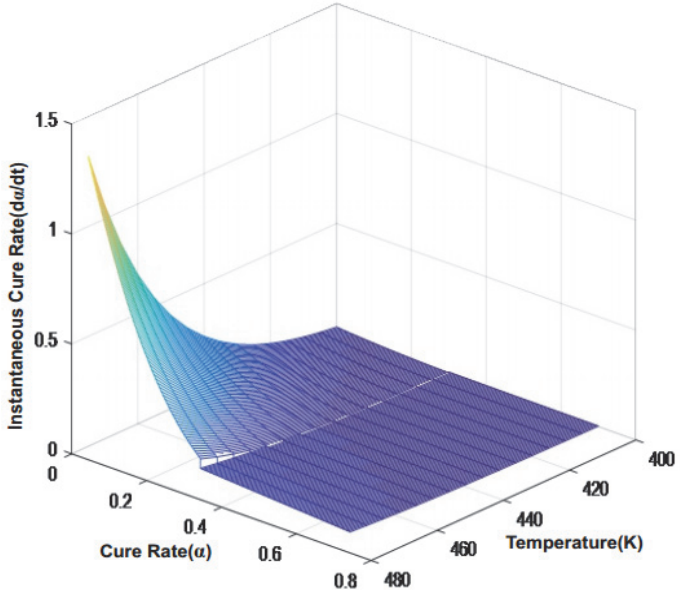

Fig. 3. Three-dimensional plot for temperature, curing degree and curing degree rate

By solving the differential equation for curing degree, based on time, Fig. 4 is obtained. The curing degree increases from 0 to 1 with time. The rate decreases towards the end.

The curing degree rate, which has in relation with the heat produced during polymerization, is shown in Fig. 5 based on time. Fig. 6 shows the heat generated through the process. This figure has two maximums, which shows two reactions occurring which release heat and leads to a temperature higher than the oven, which is seen in Fig. 7. The second reaction releases almost twice as much heat.

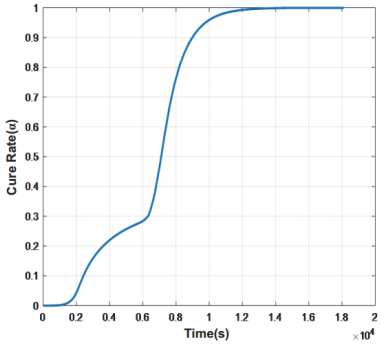

Fig. 4. Composite curing degree based on time

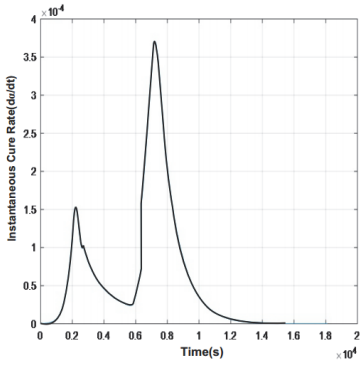

Fig. 5. Curing degree rate based on time

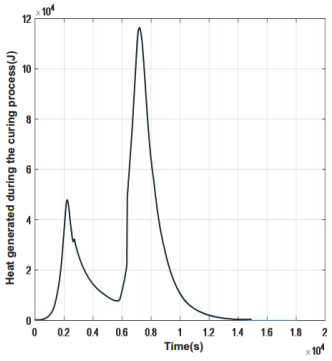

Fig. 6. Heat generated in the composite based on time

3.2 Multilayer temperature

Due to the two reactions (Fig. 6) goes above the oven temperature in two points. As is expected, the center of the multilayer is the last point to get cold (Fig. 7). The reason the residual stress is compressive is thus found; the center is still hot while the outside is becoming solid, and thus the center is under pressure and leads to residual stress in the center of the part.

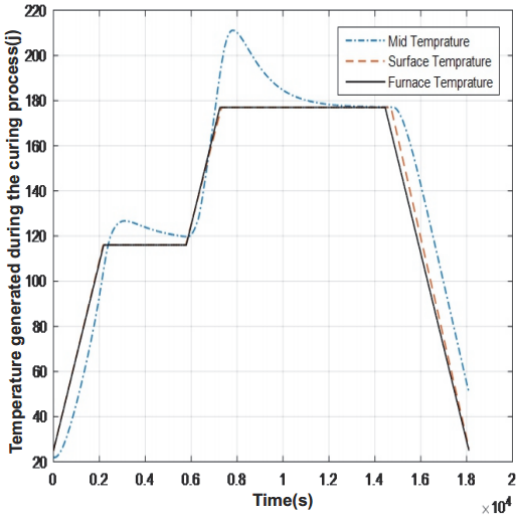

Fig. 7. Temperature based on time in the center of composite layer and its comparison with the surface using MATLAB

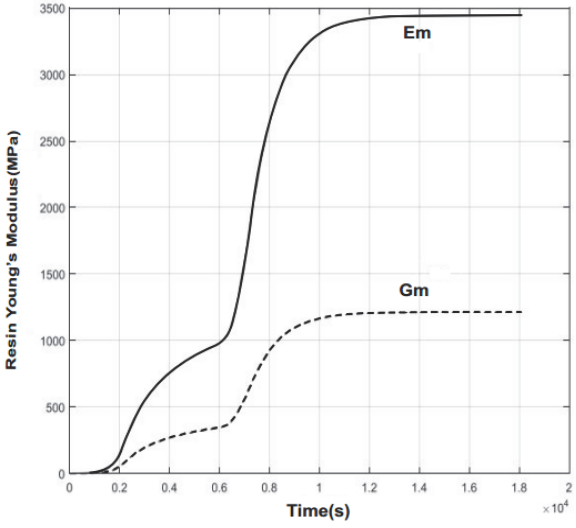

Fig. 8. Resin shear and Yong modulus

3.3 Resin elastic modulus and thermal residual stress due to chemical contraction

Fig. 8 shows the composite elastic modulus which changes through the process. The modulus increases from zero to its maximum. The final stress through thickness has been found from equation 33

and is shown in Fig. 9. This figure depicts the results for the thick layer composite with a thickness of 2.54 cm. Compressive stress is -7.93 MPa (pressuring stress) at the center of composite (z=0) and increases symmetrically with distance from center to surface and the stress will be zero at the thickness of z=0.723. The stress is 12.07 MPa (tensile stress) in the surface of the composite (z=1.27 cm). The maximum error in calculations when using Bogetti's solution is 6.24%.

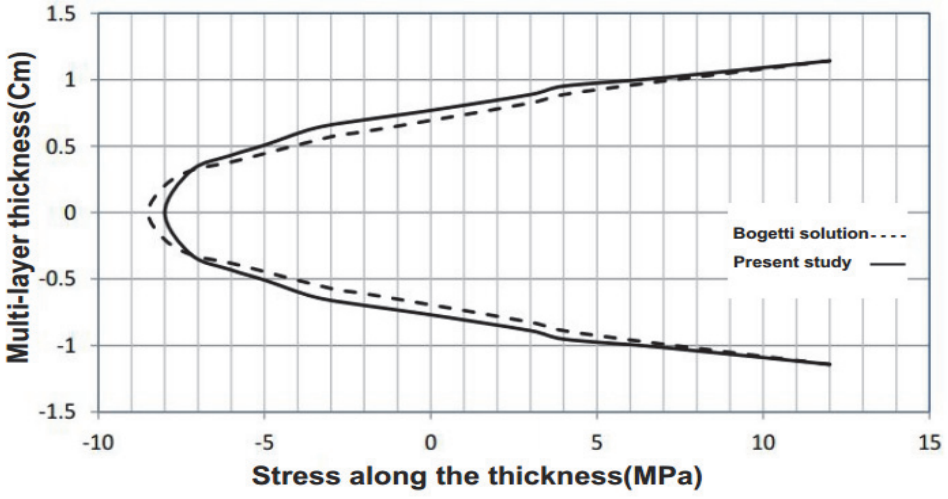

Fig. 9. Stress along the thickness of composite (2.54 cm thickness)

4. Conclusion

In this study a chemical and heat transfer approach was adopted. The curing process and change in mechanical properties through the process were assessed. Using superposition, the residual stresses were found in the direction of thickness.

The main achievements of this study are:

1. Assessing the theory and equations of thick layer composites

2. Numerically solving the asymmetrical implicit heat transfer equations

3. Calculating the temperature and chemical factors producing heat which effect the curing process

4. Calculating composite mechanical properties which change dynamically through the curing process

5. Calculating the residual stress in the direction of composite thickness

Based on the results of the analysis of curing of thick layer composites it was found that composite curing is ascending and in parts of the process, the speed decreases that is due to resin chemical properties and is dependent on material. During curing due to resin chemical reactions two gravure reaction take place, which increases the temperature and speeds up the curing. The resin and composite mechanical properties are calculated and residual stress in thick layer composites is found. Since composite fibers are all in one direction, the stress in the direction of thickness is such that in the middle of the composite, it is compressive and in the sides tensile which may lead to delamination.

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