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MULTIPHYSICS PREDICTION MODEL OF MICROWAVE CURING FOR THICK
POLYMER COMPOSITES

by

SIVA SAI KRISHNA DASARI

A THESIS

Presented to the Faculty of the Graduate School of the
MISSOURI UNIVERSITY OF SCIENCE AND TECHNOLOGY

In Partial Fulfillment of the Requirements for the Degree
MASTER OF SCIENCE IN MECHANICAL ENGINEERING

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Approved by:

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PUBLICATION DISSERTATION OPTION

This thesis consists of the following one article, formatted in the style used by the Missouri University of Science and Technology:

Paper: Multiphysics prediction model of microwave curing for thick polymer composites (pages 4-29) have been submitted to the proceedings of the CAMX Conference, Anaheim, CA, September 23-26, 2019.

ABSTRACT

Microwave curing technologies have many advantages over the traditional thermal curing methods for the manufacturing of fiber reinforced polymers, especially the processing speed and energy efficiency. Energy can be instantaneously transferred through applied electromagnetic fields and heat is generated based on dipolar rotational interactions. Microwave curing processes have been used for glass fiber composites but there are significant challenges associated with microwave curing of carbon fiber composites. Efficient heating may be difficult due to high dielectric loss associated with carbon fibers. Laminate quality will be highly dependent on the uniformity of the electromagnetic field in the material. In this work, a multiphysics three-dimensional model was developed to study the composite curing behavior and temperature distribution of the laminate in the presence of microwave radiation. Microwave heating depends on the thermal conductivity, convective heat transfer, surrounding temperature, intensity of the electromagnetic field and the geometry of the sample. The anisotropic properties of a composite were incorporated into the simulation model. This model can be used to optimize process parameters to cure thick and complex shaped composite parts. A cure cycle optimized to the microwave energy was developed and compared to the traditional thermal cure cycle.

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Last, but certainly not least, I would like to thank my parents, Dr. Mani Kumar Dasari, Dr. Sujatha Tadikonda, and my sister, Dr. Utheja Dasari, for their constant support throughout my pursuit of a master's degree despite the long distance.

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1. INTRODUCTION

Composite material, particularly carbon fiber-reinforced polymer composites, being widely used in aerospace and automotive industries due to their unique properties such as high specific strength, corrosion, improved fatigue life and wear resistance. The limitations of composite materials are the cost of the raw materials and intense time and energy requirements during their processing. The cost of the raw materials depends on the demand-supply in the market. The latter limitation can be controlled by development of alternative curing processes such as microwave curing. Current manufacturing of composite components relies on energy-intensive and time-consuming conduction heating methods. Autoclave manufacturing is the dominant process used for producing high-performance composites, especially for the aerospace industry. The temperature and pressure requirements are very high for autoclave manufacturing. The heat is generated using electric heaters, and the heat travels to the surface of the composite part from the gas medium (inert atmosphere like nitrogen which has a typical thermal conductivity of 0.025W/m.K) surrounding the part in the oven chamber. The thermal conductivity of the polymer matrix is low. Therefore, it takes a relatively long time for the part to attain thermal equilibrium. As a result, it takes relatively longer time and energy to cure thick composites.

Microwaves (MW) are electromagnetic waves which lie in 300 MHz to 300 GHz in the electromagnetic spectrum [1] (Figure 1.1). MWs have electric field E and magnetic field H orthogonal to each other. The intensity of the electric and magnetic field of microwaves at a position is given by the sine and cosine functions of time. Both E and H

travel in the same direction. Heat is generated when material interacts with the microwave irradiation. The heating of material depends on the dielectric and magnetic properties of the material. Microwave heating can be explained by the polarization of charged particles in an alternating external MW electric field. The external field causes the formation of an electric moment in the entire volume of the dielectric material leading to heating of the material. In the case of heterogeneous materials such as fiber reinforced polymer composites, interfacial polarization is the main type of heating mechanism.

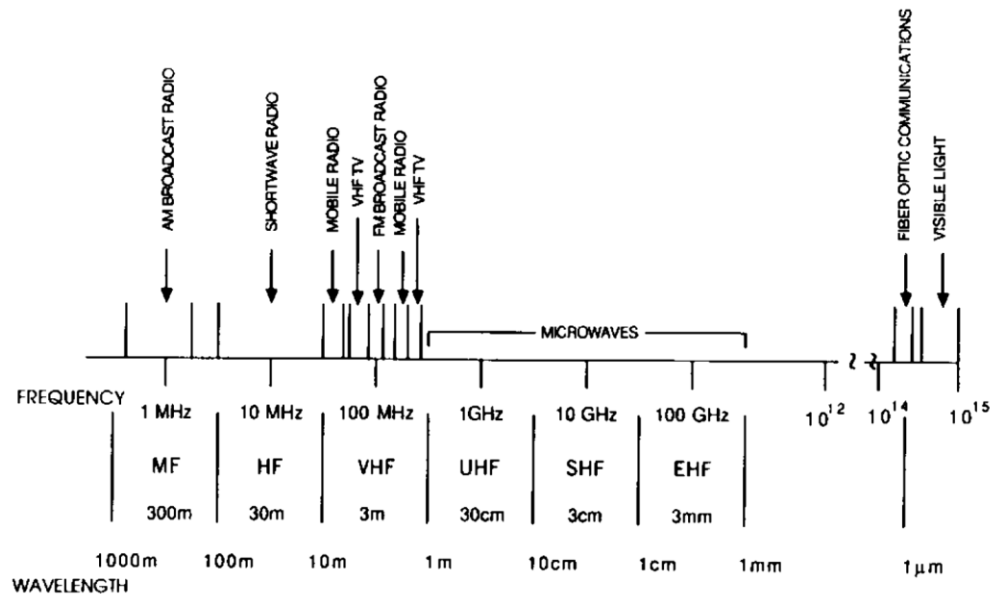


Figure 1.1. The electromagnetic spectrum [2].

In regard to microwave – material interactions, materials can be classified into three classes. Reflective materials such as metals reflect the microwaves without heating the material. Transparent materials transmit the microwaves through the material with a negligible amount of heating. Whereas absorptive materials will volumetrically heat by

dielectric polarization or ionic polarization or other mechanisms. When curing a composite part, only the part is heated in microwaves curing process. The mold and the surrounding air medium is not heated. Therefore, the energy and time required for heating mold and the air is reduced. In certain materials such as epoxies, the dielectric loss properties reduces with the increase of the degree of cure. As a result, the heat generating capacity of the epoxies will decrease with the increase in the degree of cure. This will be very beneficial when curing thick composites by preventing the overheating or thermal runaway of resin material.

A major challenge of microwave curing is uneven energy distribution. This is the major barrier for curing composites using microwaves at an industrial scale. Microwaves have fixed wavelength and it can be very difficult to obtain a MW field which will produce homogeneous heating and curing. The biggest obstacle when heating carbon fiber reinforced polymers is arcing and uneven energy distribution. Arcing at the tips of the carbon fibers will most likely lead to combustion, damaging the material [3, 4]. Another important challenge in using microwave curing technology is the current tooling materials. The tooling materials are generally made up of metals which are considered to reflect and absorb the microwaves leading to distortion of microwaves as well as unequal heating near the part-tool interface. Distorted microwaves further lead to uneven distribution of microwaves.

The aim of the present work is to model the microwave curing process to determine the degree of cure and power required for curing of the carbon-epoxy composites. To understand the influence of different heating rate, a parametric study was performed.

PAPER

I. MULTIPHYSICS PREDICTION MODEL OF MICROWAVE CURING FOR THICK POLYMER COMPOSITES

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ABSTRACT

Microwave curing technologies have many advantages over the traditional thermal curing methods for the manufacturing of fiber reinforced polymers, especially the processing speed and energy efficiency. Energy can be instantaneously transferred through applied electromagnetic fields and heat is generated based on dipolar rotational interactions. Microwave curing processes have been used for glass fiber composites but there are significant challenges associated with microwave curing of carbon fiber composites. Efficient heating may be difficult due to high dielectric loss associated with carbon fibers. Laminate quality will be highly dependent on the uniformity of the electromagnetic field in the material. In this work, a multiphysics three-dimensional model was developed to study the composite curing behavior and temperature distribution of the laminate in the presence of microwave radiation. Microwave heating depends on the thermal conductivity, convective heat transfer, surrounding temperature, intensity of the electromagnetic field and the geometry of the sample. Required parameters are determined using experiments. The anisotropic properties of a composite

were incorporated into the simulation model. This model can be used to optimize process parameters to cure thick and complex shaped composite parts. A cure cycle optimized to the microwave energy was developed and compared to the traditional thermal cure cycle.

1. INTRODUCTION

Advanced manufacturing techniques provide a gateway to tailor the material properties in order to achieve high performance, and environmentally friendly engineered structures. Advanced manufacturing techniques reduce fuel consumption and emissions and help combat climate change [1]. Fiber reinforced polymers (FRP) are lightweight materials with high strength-to-weight ratio. FRP can be tailored for modern high performance structural applications allowing for efficient engineering solutions to severe and varying operating conditions like dynamic and impact events on composite aircraft [2-3].

Autoclave manufacturing is the dominant process used for producing high performance composites, especially for the aerospace industry. However, the autoclave curing process is time consuming and expensive. The temperature and pressure requirements add demand on tooling materials. Moreover, growing demand for polymer composites in critical structures has given rise to an urgent need for a reliable and rapid composite repair as well as fastener-less joining such as adhesive bonding. This is especially true in the maintenance, repair and overhaul (MRO) sector in aerospace. The quality of adhesively bonded structures is strongly dependent on the variation produced by process parameters such as temperature, curing duration and rate [4]. The term curing

in thermosetting polymers refers to the transition of liquid resin and hardener components to a solid material. Curing is initiated when the components are stoichiometrically blended together and heated. Polyester, phenolic and epoxy resins are among the most widely used thermoset polymers with epoxy being popular for use in high performance composite structures. The curing of thermosetting polymer systems can be expressed mathematically through cure kinetic equations. Several researchers have worked on the mathematical modeling and simulation of composite curing kinetics [5-7]. The cure kinetics equations can be used to evaluate temperature distribution during cure of thermoset resin systems [8]. Initial research on cure simulation of composites used one or two-dimensional finite difference analysis and was applicable for simple geometries.

Microwave curing is an advanced alternative curing method, which can result in reduced cure times and reduced energy consumptions. In microwave curing, only the laminate is heated and parts like the mold and breather materials remain at lower temperatures. In the case of thick composite laminates higher heating rates can be achieved using microwaves whereas a typical heating rate using conventional oven was observed to be less than 1 °C per minute. Certain materials exhibit a reduction in dielectric loss properties with increasing degree of cure. As a result, materials such as epoxy lose their ability to absorb microwave radiation and generate heat at higher degrees of cure [9]. Due to the self-limiting nature of these reactions, microwave heating can be adopted for the materials that degrade at higher temperature.

Microwave curing processes have been used for glass fiber composites but there are significant challenges associated with microwave curing of carbon fiber composites. Laminate quality is highly dependent on the uniformity of the electromagnetic field in the

material. Microwave field homogeneity is a huge concern, particularly when using fixed frequency systems. Efficient heating may be difficult due to high dielectric loss associated with carbon fibers. Another major challenge is related to arcing of carbon fiber bundles, which can result in very high localized temperatures and damage to surrounding materials [10]. Many of the aforementioned studies found little temperature control was available during experimentation. Power input to the microwave is not controlled as a function of time or temperature in order to achieve a constant heat rate or fixed dwell temperature [11].

In this work, a finite element model was developed to study the microwave curing process in a cost-effective manner. The model can be used to estimate the power input of the microwave required to obtain uniform heating rates and constant holding temperatures. The model developed can also be used to predict the degree of cure. This model can optimize process parameters to cure thick and complex shaped composite parts.

1.1. MATERIALS

1.1.1. Carbon-Epoxy Composite Material. In this study, Cycom 5320-1 unidirectional prepreg system (Cytec Industries) was used. Cycom 5320-1 is a well characterized toughened epoxy resin system with a fiber areal weight of 145 gsm. The unidirectional prepreg contains 33 % resin by weight and is highly suitable for out-of-autoclave curing. The cure kinetics of common out-of-autoclave (OOA) systems, Cycom 5320 and MTM 45-1, were characterized by Kratz et al [12].

1.1.2. Consumables. Consumable materials such as vacuum bag, breather, and release film were modeled as pure insulators using the properties listed in Table 1. The consumables were modeled as a single layer with equivalent properties derived using the rule of mixtures.

Table 1. Material properties of consumables

Material	Thickness (mm)	Thermal conductivity (W/m·K)	Specific heat capacity (J/g·K)	Density (kg/m ³)
Release film	0.05	0.4	1.05	2200
Vacuum bag	0.05	0.23	1.67	1140
Breather	2.54	0.06	1.35	260
Combined layer	2.64	0.079	0.119	314.2

2. EXPERIMENTATION

2.1. METHODOLOGY

The matrix in a composite laminate is a thermosetting resin which is polymerized by application of heat. When the laminate is at curing temperature the polymerization of the epoxy resin is prompted and energy is released. The curing of Cycom 5320-1 is an exothermic reaction. The rate of reaction is dependent on the temperature history of the system and exotherm. Multiphysics models were built corresponding to the manufacturing layup and the results of the simulation were studied. Relevant process

parameters such as microwave energy were optimized through extensive simulation. The multiphysics model contains cure kinetics and heat transfer modules.

2.2. CURE KINETICS MODELING

Differential Scanning Calorimetry (DSC) is a widely used technique to monitor and obtain cure kinetic parameters of exothermic reactions. DSC monitors the heat flow out of a sample during the curing process. For an exothermic reaction the degree of cure, α , at a certain time, t , is defined as the ratio of heat evolved from a sample during curing until t to the total heat of reaction of the cured sample. In order to calculate the degree of cure of a sample Equation 1 can be used.

$$\alpha = \frac{\Delta H_t}{\Delta H_U} \quad (1)$$

where, ΔH_U is the total heat of reaction (total exotherm) and ΔH_t is the heat of reaction at a time, t . Using multiple isothermal DSC experiments, the cure kinetic equation can be calculated by integrating the measured heat flow curve at the peak. For the carbon/epoxy prepreg system used in the current study, the cure kinetic equation is defined in Equation 2a and Equation 2b. Since the process is not an isothermal curing process, Arrhenius law was used. The equation accounts for the interplay between kinetics-controlled and diffusion-controlled reaction mechanisms [13].

$$\frac{d\alpha}{dt} = \sum_{i=1,3} K_i \alpha^{m_i} (1 - \alpha)^{n_i} + \sum_{j=2,4} \frac{K_j \alpha^{m_j} (1 - \alpha)^{n_j}}{1 + e^{D_j \{\alpha - (\alpha_{c0,j} + \alpha_{cT,j} T)\}}} \quad (2a)$$

$$K_l = A_l e^{(-E_{A,l}/RT)}, l = i, j \quad (2b)$$

where A_n is the Arrhenius constant (value), $E_{A,n}$ is the activation energy for the reaction, R is the universal gas constant, m_i and n_i are reaction order-based fitting constants, D_j is the diffusion constant, T is the temperature, a_{c0} is the critical degree of cure at absolute zero, and a_{cT} accounts for the increase in critical degree of cure with temperature. The cure kinetics equation which accounts for the effects of prepreg out-time was developed for Cycom 5320–1 OOA prepreg system [10]. Values of the parameters are given in Table 2.

Table 2. Cure kinetic parameter

$A_1 (s^{-1})$	1.48×10^7	$A_3 (s^{-1})$	6.39×10^7	$A_2 (s^{-1})$	8.3×10^4	$A_4 (s^{-1})$	9.8×10^4
$\frac{E_{A,1}}{R} (K)$	1.02×10^4	$\frac{E_{A,3}}{R} (K)$	8.94×10^3	$\frac{E_{A,2}}{R} (K)$	8.54×10^3	$\frac{E_{A,4}}{R} (K)$	7.1×10^3
m_1	0.17	m_3	1.65	m_2	0.7	m_4	1.66
n_1	19.3	n_3	16.6	n_2	0.87	n_4	3.9
D_2	97.4	D_4	63.3	$a_{c0,2}$	-1.6	$a_{c0,4}$	-0.6

2.3. THERMAL MODEL

The thermal portion of the model was deduced from the first law of thermodynamics. The thermal equilibrium equation (Equation 3) was used to solve for energy balance in the laminate portion. The heat generation is dependent on both time and position within the laminate. There are two sources of thermal energy in the system,

the heat generated within the system by external microwave radiation and the heat generated by the exothermic chemical reaction.

$$\rho_c C_c \frac{\partial T}{\partial t} = \frac{\partial}{\partial x} \left(k_x \frac{\partial T}{\partial x} \right) + \frac{\partial}{\partial y} \left(k_y \frac{\partial T}{\partial y} \right) + \frac{\partial}{\partial z} \left(k_z \frac{\partial T}{\partial z} \right) + v_m \rho_m H_u \frac{\partial \alpha}{\partial t} + 2\pi f \varepsilon_o \varepsilon'' E_{rms}^2 \quad (3)$$

where ρ_c is the density of composite, C_c is the specific heat capacity of composite, T is temperature, t is time, k_x , k_y and k_z are the thermal conductivity of composite in x, y and z directions, v_m is the resin volume fraction, ρ_m is the resin density, H_u is ultimate heat of reaction of the system and α is the degree of cure, f is the frequency of 2.45 GHz for microwave radiation, ε_o is dielectric constant of composite, ε'' is dielectric loss factor, E_{rms} is root mean square power of the microwaves. The material properties of the tool and prepreg materials were extracted from [4] and Cycom 5320-1 datasheet as shown in Tables 3 and 4. Thermal conductivity along thickness direction k_{zz} is calculated using Springer-Tsai model [14]. In the simulation, the microwave generator was placed above the laminate. The effect of degree of cure on thermal conductivity for epoxy resins was studied by Struzziero et.al [15]. The thermal conductivity of epoxy increases as the degree of cure increases but in this work it was assumed that the specific heat capacity and thermal conductivity are constant during the curing process, and their dependence on the degree of cure was not incorporated into the multiphysics model.

Table 3. Material parameters of Cycom 5320-1

ρ_c (kg/m ³)	ρ_r (kg/m ³)	ρ_f (kg/m ³)	v_r %	v_f %
1591.6	1310	1780	40.09	59.91
k_r (W/m·K)	k_f (W/m·K)	k_x (W/m·K)	k_y (W/m·K)	C_c
0.167	5.4	3.3021	0.5067	1260

Table 4. Thermal properties used in the model

	Density (kg/m ³)	Specific heat (J/kg·K)	Thermal conductivity (W/m·K)		
Laminate (prepreg)	1591.6	1260	x	y	z
			3.302	3.302	0.506
Mold	2700	505	0.5		
Caul plate	2700	505	0.5		
Consumable	314.2	0.119	0.079		

The key parameter for heating materials effectively using microwaves is the microwave penetration depth into the material. The penetration depth of the material is a function of microwave frequency, magnetic permeability, and electric conductivity. Penetration depth is used to determine whether the material will heat evenly through the thickness. The penetration depth of the material is given by Equation 4.

$$D_p = \sqrt{\frac{1}{\pi f \mu' \sigma_{ec}}} \quad (4)$$

where f is the frequency (Hz), μ' is the magnetic permeability (H/m) and σ_{ec} is the electrical conductivity (S/m).

2.4. MULTIPHYSICS CURE MODELING

A thermo-chemical model was built in Comsol Multiphysics software (Figure 1). The model is built to simulate the curing of composite parts. The partial differential equations module was used in the Comsol Multiphysics software to address the cure kinetics process in the laminate. The thermal model was incorporated using the heat

transfer module. The cross section of the multiphysics model used is shown in Figure 2.

The model reflects the actual manufacturing layup used to cure composite components.

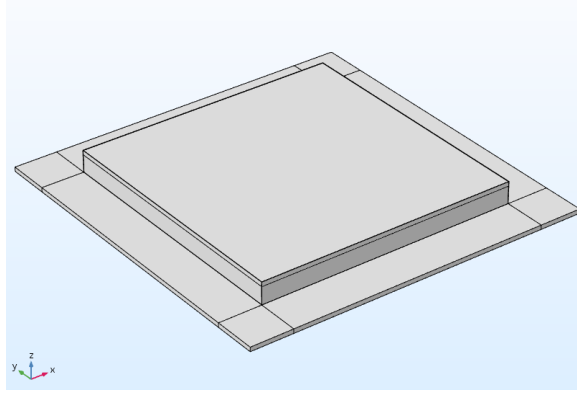


Figure 1. Three-dimensional model

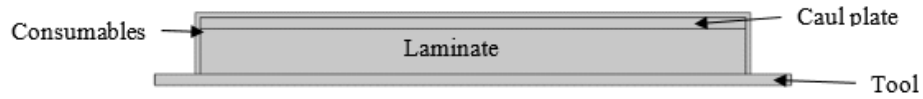


Figure 2. Cross section of the model

A heat flux boundary condition was applied on all outer surfaces according to Equation 5. This corresponds to the cure cycle for composite manufacturing.

$$Q = h(T_{ext} - T_{boundary}) \quad (5)$$

Where Q is the heat generated, h is the convection heat transfer coefficient, T_{ext} is the temperature of the autoclave and $T_{boundary}$ is the temperature at the external surface of the layup. The convective heat transfer coefficient was set to 150 W/m²K. The multiphysics model used illustrated in Figure 3.

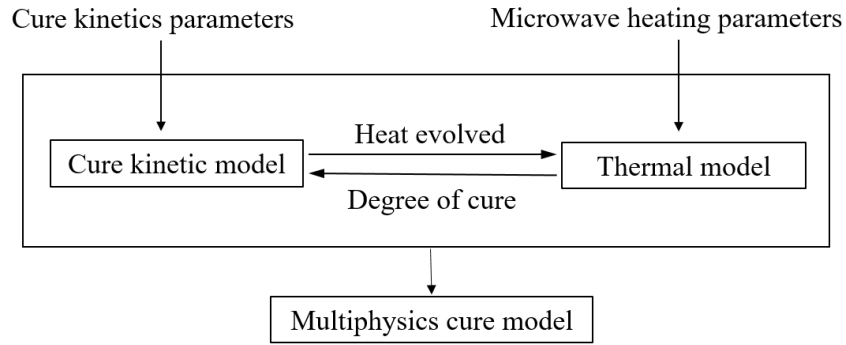


Figure 3. Multiphysics modeling of microwave composite curing

A Square laminate of size 300 mm with a thickness of 20 mm was used in the simulation. Four locations were chosen to study and monitor the temperature within the laminate. Probe 1 and probe 2 were located near the top of the surface, probes, probe 3 was located at the middle of the thickness and probe 4 was located at the corner of bottom surface. During the simulations, the temperature at probes, Probes 1 and 2, were used to dynamically control the microwave power input to the laminate. The locations of four probe used in the simulations are tabulated (Table 4).

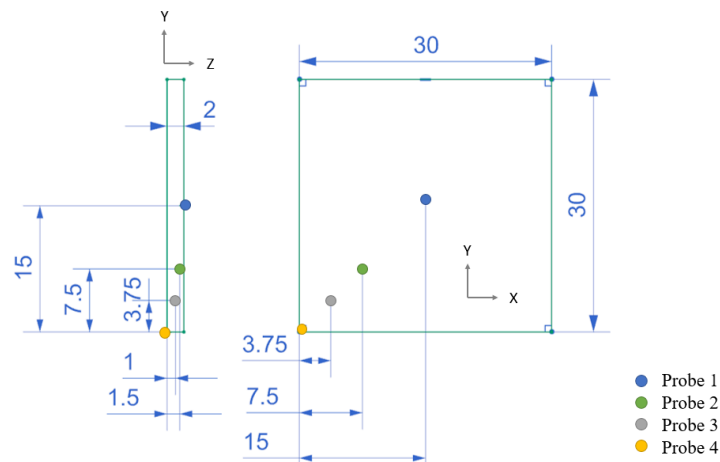


Figure 4. Probe locations (Units in centimeters)

Table 5. Probe locations

	X position (cm)	Y position (cm)	Z position (cm)
Probe 1	15	15	2
Probe 2	7.5	7.5	1.5
Probe 3	3.75	3.75	1
Probe 4	0	0	0

2.5. TEMPERATURE PROFILES FOR CURING PROCESS

A baseline study was conducted to understand the difference between conventional curing process and microwave curing process. For the modeling of conventional curing process, the heat flux boundary condition in the model was replaced by a temperature boundary condition. The temperature boundary condition is a function of time. The manufacturer thermal cure cycle was used as the temperature boundary condition. A microwave power input cycle was generated for modeling of microwave curing process to achieve temperature profile similar to that of the conventional curing cycle.

Different types of temperature profiles were chosen to study curing behavior for the microwave curing process. The variables used in the simulation were the heating rate and holding temperature of dwell. Two profiles were used to study the effect of heating rate and two profiles to study the effect of maximum dwell temperature on the degree of cure and theoretical power input. The temperature profiles chosen are tabulated (Table 6).

Table 6. Temperature profiles

Temperature profile	Heating rate (°C/min)	Holding temperature (°C)	Holding time (min)
A	3	150	90
B	3	180	90
C	6	180	90

3. RESULTS

To examine the influence of microwave heating on cure behavior and temperature distribution of a thick laminate, a 20mm thick laminate was modeled using Comsol Multiphysics for Numerical Investigation. As a baseline study, a simulation using the proposed model was performed using the traditional thermal cure cycle recommended by material manufacturer. To illustrate the influence of microwave heating on cure behavior, the same composite model following the manufacturer cure cycle was subjected to microwave heating. The cure cycle chosen for the simulation was a ramp of 1.1 °C/min to 60 °C followed by dwell at the same temperature for two hours. Then a 0.5 °C/min ramp to 120 °C followed by a two hour soaking period. The manufacturer recommended cure cycle for Cycom 5320-1 is shown in Figure 5.

Figure 6 shows the temperature profile at the part center for both microwave heating and autoclave heating during curing. It was observed that there was a thermal lag at the part center for autoclave cured model when compared to the part surface (The manufacturer thermal cure cycle was applied as a boundary condition at the surface of laminate for autoclave heating). When the laminate was cured using microwave heating

there was no significant temperature lag at the part center. There was a temperature overshoot in the laminate during the soak period for autoclave heating cycle, this was not observed using the microwave heating process. Direct heating using the microwave results in the reduced thermal lag and the thermal overshoot during the soak period. In a conventional curing process, very slow heating rates were required to reduce the thermal lag. Figure 7 compares the degree of cure at the part center for both models. The onset of cure occurs earlier in the microwave heating process than the traditional thermal curing process. Both curing models achieved 0.61 degree of cure at the end of manufacturer recommended cure cycle. To illustrate the potential reduction in cure cycle times that microwaves can offer, the cure cycle for conventional processing was modified presented in Section 3.1.

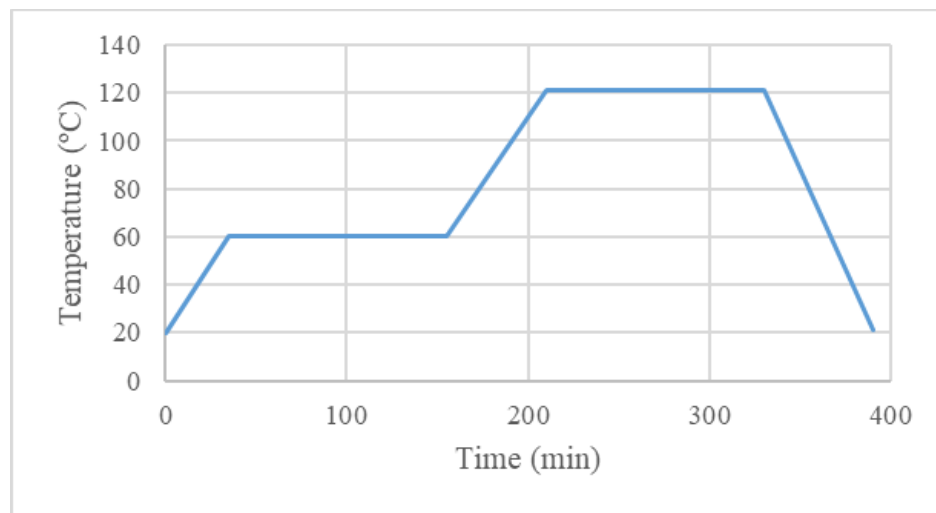


Figure 5. Manufacturer recommended cure cycle

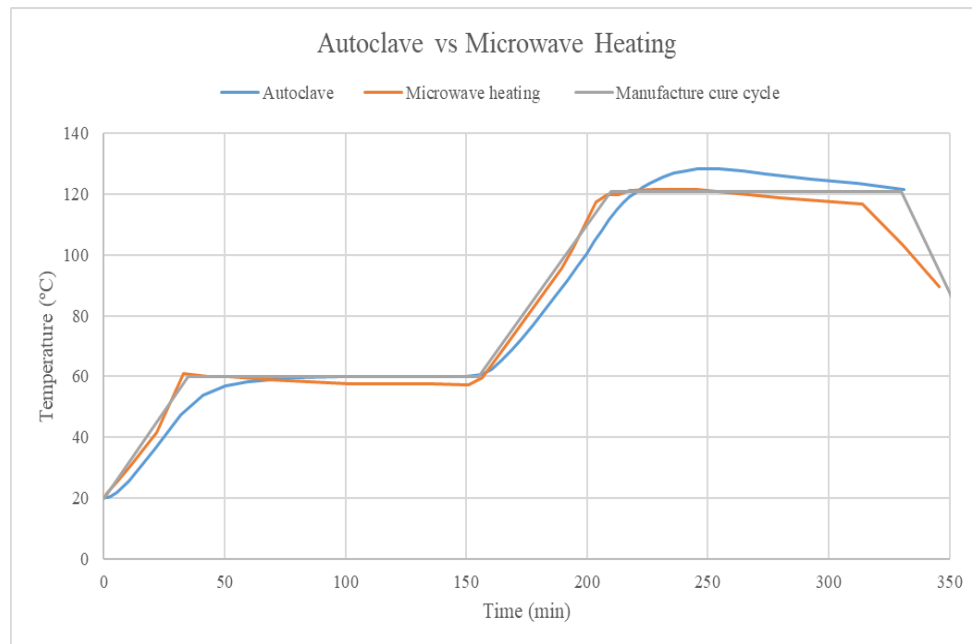


Figure 6. Temperature profile for autoclave heating and microwave heating process

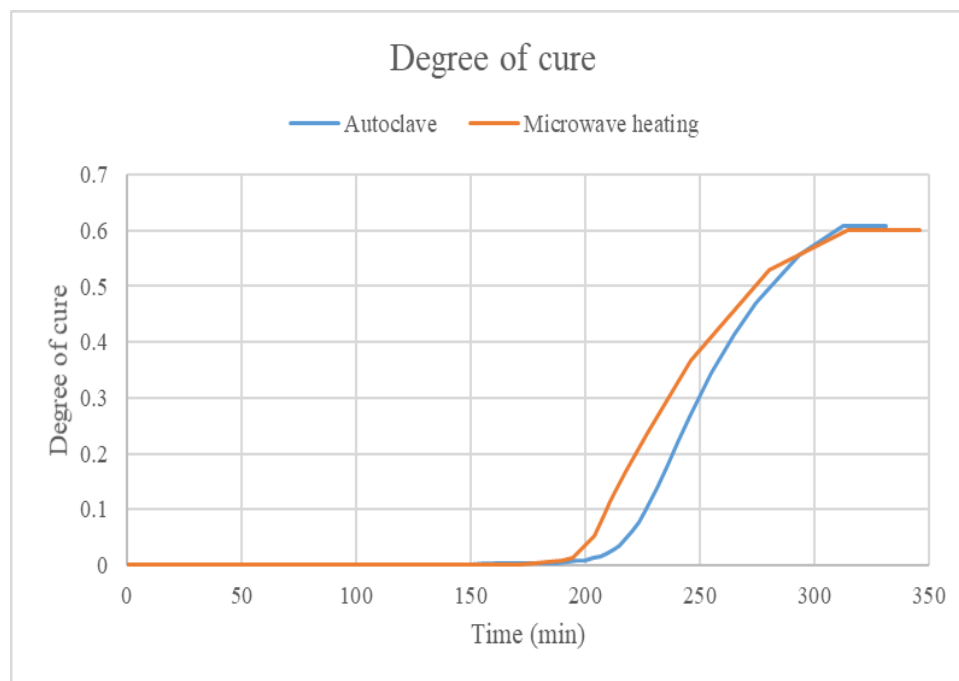


Figure 7. Degree of cure profile for autoclave heating and microwave heating process

3.1. MICROWAVE TEMPERATURE PROFILES

Different temperature profiles were employed to simulate the curing behavior of the laminate. The temperatures and degree of cure were recorded at different locations during the cure cycle. The temperature profile, A, with a heating rate of 3 °C/min at a holding temperature to 150 °C and a soak period of 90 minutes was used in the model. The temperature history and degree of cure were recorded at four probe locations. Figure 8 represents the temperature profile at four probe locations. During the dwell period, a constant temperature gradient of 50 °C was observed between the top and the bottom corner of the laminate. Because the laminate surface was exposed to air in the microwave chamber, free convection between air and the laminate surface occurs. This results in thermal gradient between the top and bottom surface of the laminate when using microwave cure model. Figure 9 shows the degree of cure over time. In the simulation probe 1 and probe 2 were used as a dynamic feedback device and used to control the microwave power. From the plot at the end soak period, the laminate had the highest degree of cure of 0.843 near probe 2, while probe 1 recorded 0.834 degree of cure. Probe 3 and probe 4 reported a maximum degree of cure of 0.803 and 0.0213 respectively. On the top surface where probe 1 was located, free convection between air and the surface occurs. As a consequence, there was a heat loss from the surface of the laminate to the microwave chamber. The probe 3 was located in the middle of the laminate. Due to the effect of penetration depth, heat was not directly generated near probe 3 or probe 4 but was transferred through carbon fiber. Hence, thermal lag exists near these probes.

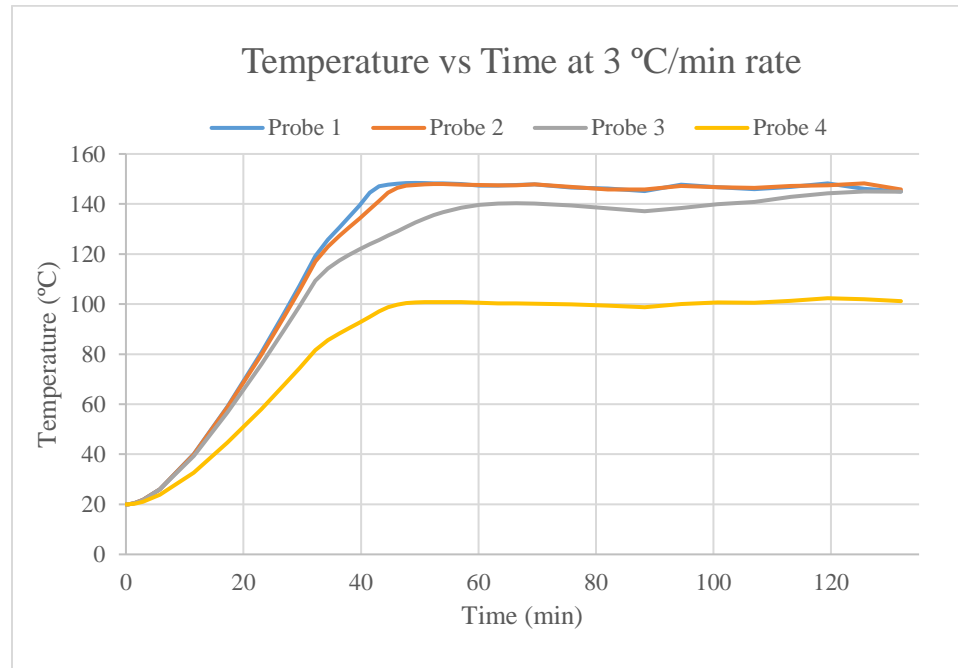


Figure 8. Temperature profile for cure cycle A for 20 mm thick laminate

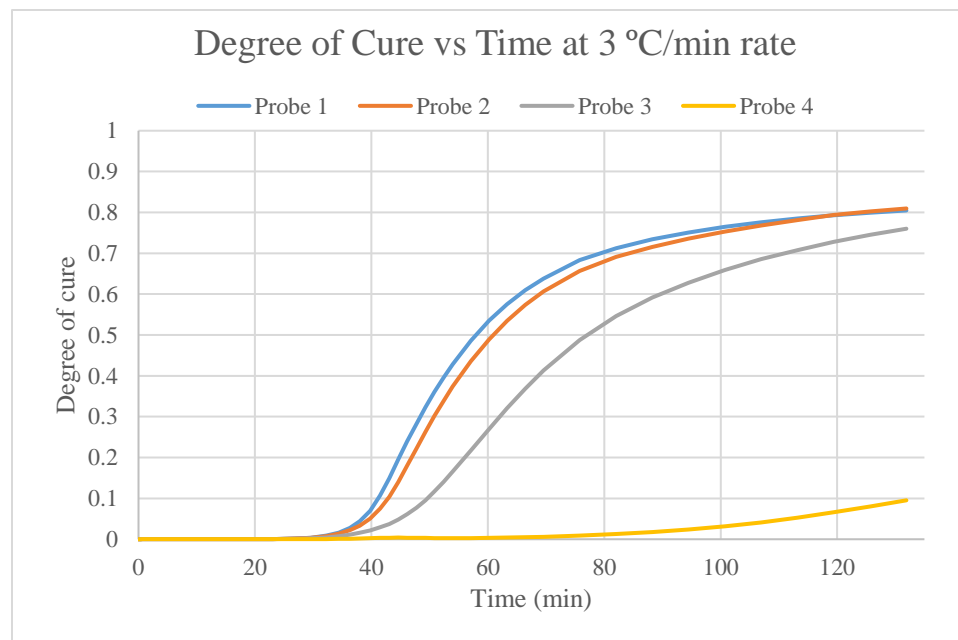


Figure 9. Degree of cure profile for cure cycle A for 20 mm thick laminate

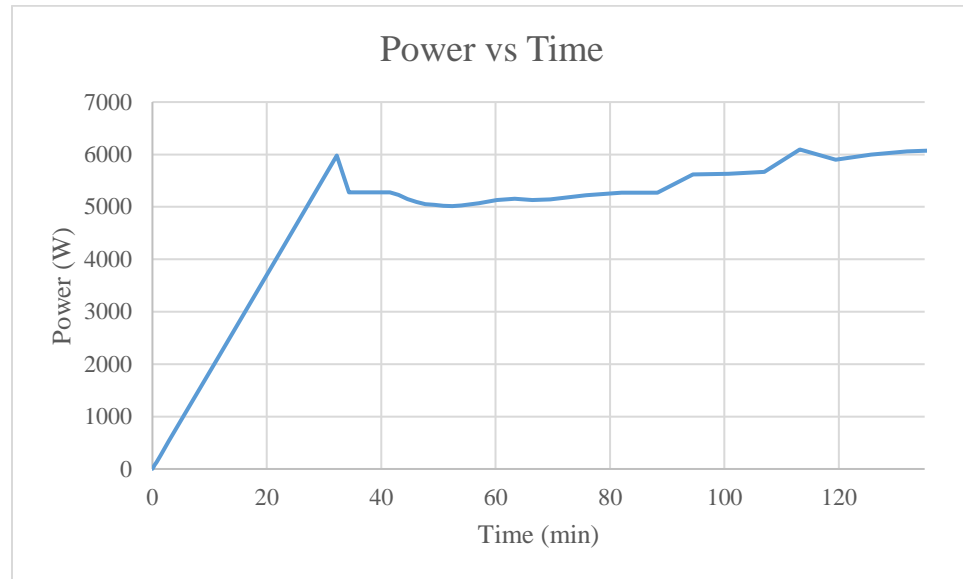


Figure 10. Power input for cure cycle A

The theoretical power required to obtain the above cure cycle was shown in Figure 10. The plot can be classified into three regions. In the ramp up region the power input to the microwave increases. This ramp up region continues till the dwell region. The maximum power required was obtained in this region. Following this region there was a fall in the power used by microwave. The third region was the dwell region. In this region, the power input required by microwave to maintain a constant temperature during laminate curing remains constant. Figure 11 shows the degree of cure of the laminate at the end of the cycle. The edges of the laminate have the least degree of cure because of the heat loss to the microwave chamber.

The temperature profile, B, was designed to study the effect of holding temperature on the degree of cure at different thicknesses in the laminate. The heating profile was a ramp of 3 °C/min to 180 °C (recommended post cure temperature) followed by 90 minutes of dwell.

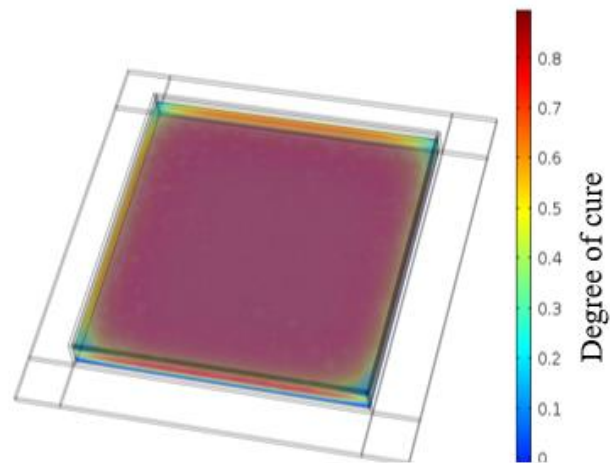


Figure 11. 3D degree of cure at time 135 min for cycle A

Initial ramp up from 20 °C to 180 °C was achieved in 53 minutes for probe 1 and probe 2. It took an additional 4 minutes for probe 3 to reach 180 °C. The probe 4 reached a maximum temperature of 137 °C. Figure 12 shows the temperature profile at four different probes.

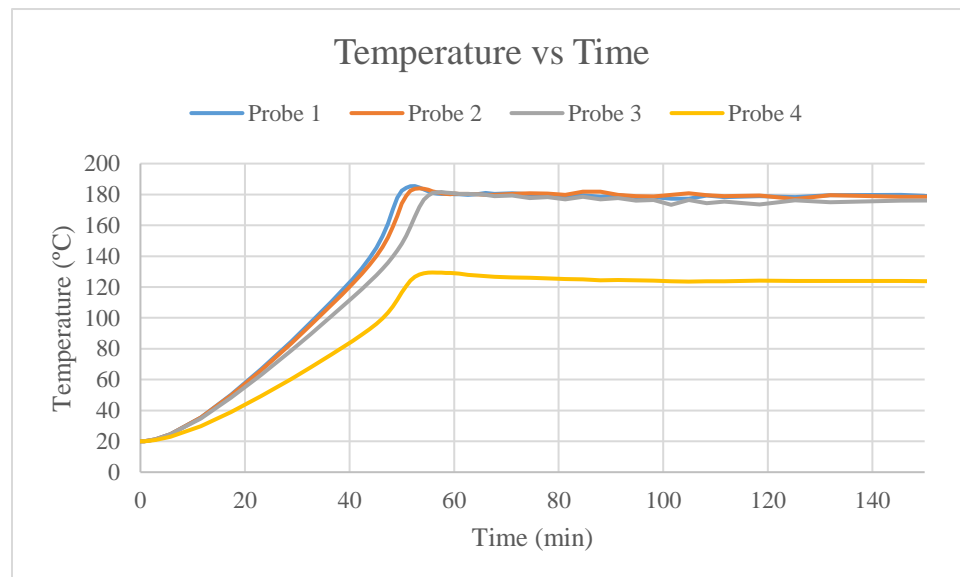


Figure 12. Temperature profile for cure cycle B for 20 mm thick laminate

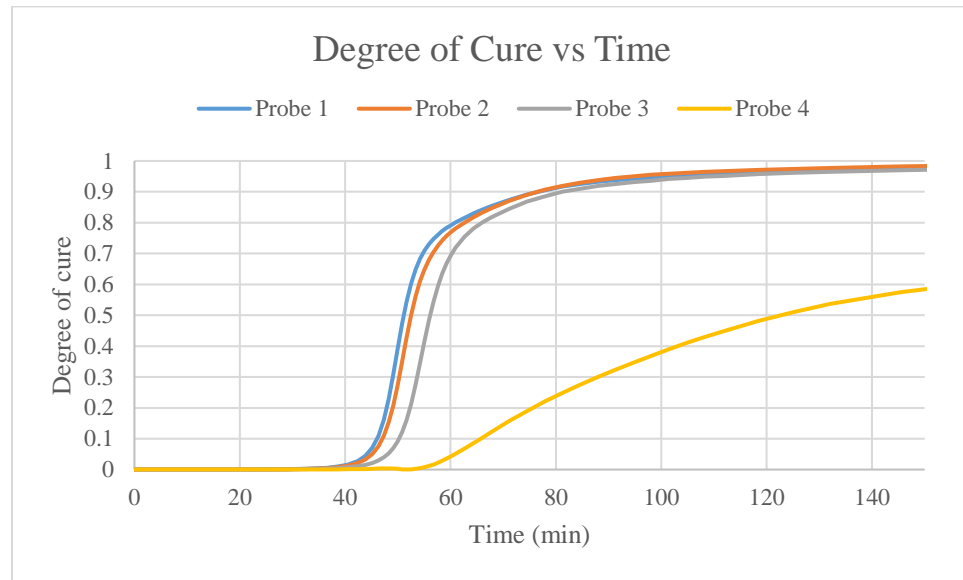


Figure 13. Degree of cure profile for cure cycle B for 20 mm thick laminate

The effect of penetration depth was clearly observed at higher depths in temperature versus time plot (Figure 12) plot for probe 3 and probe 4. Observation from the degree of cure versus time plot shows the curing in microwaves was an inside-out process. When comparing figure 9 and Figure 13 it was noticed that the slope of the cure curve was higher in later, i.e. at the start of dwell period the amount of degree of cure near probe 1 region was more than the probe 2. Whereas at the end of dwell period the degree of cure near probe 2 was more than probe 1. The maximum degree of cure at the end of the cycle was observed to be 0.98. From Figure 14, the maximum theoretical power required in the cycle was 6.4 kW. During the dwell period to maintain a constant temperature, 60% (4 kW) of the maximum power was required.

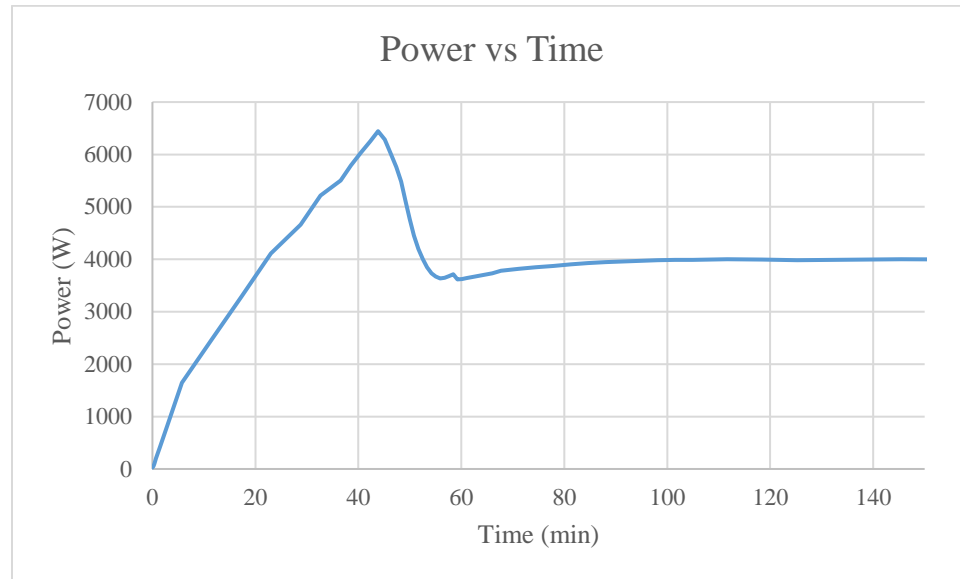


Figure 14. Power input for cure cycle B

The influence of higher cure rate was studied in temperature profile C. Profile C has a heating rate of $6\text{ }^{\circ}\text{C}/\text{min}$ for ~ 26 minutes followed by the dwell for 90 min. Figure 15 shows the temperature profile at the four different probes. From the plot, the temperature profile at different thicknesses was almost constant except the region near probe 4. It was also observed from the degree of cure to time plot (Figure 16) that onset of cure near probe 3 region was slightly delayed. Three probes (1, 2 and 3) had achieved 90% degree of cure by ~ 53 minutes. More uniform curing through thickness was observed at higher heat rates.

Figures 17 show the power required by the microwave to perform the required thermal profile. Maximum power required was observed to 9.1 kW. During the holding period, 50% of maximum power was necessary to maintain $180\text{ }^{\circ}\text{C}$. Figure 18 show degree of cure through thickness for thermal profile C. The lowest degree of curing was at the bottom surface.

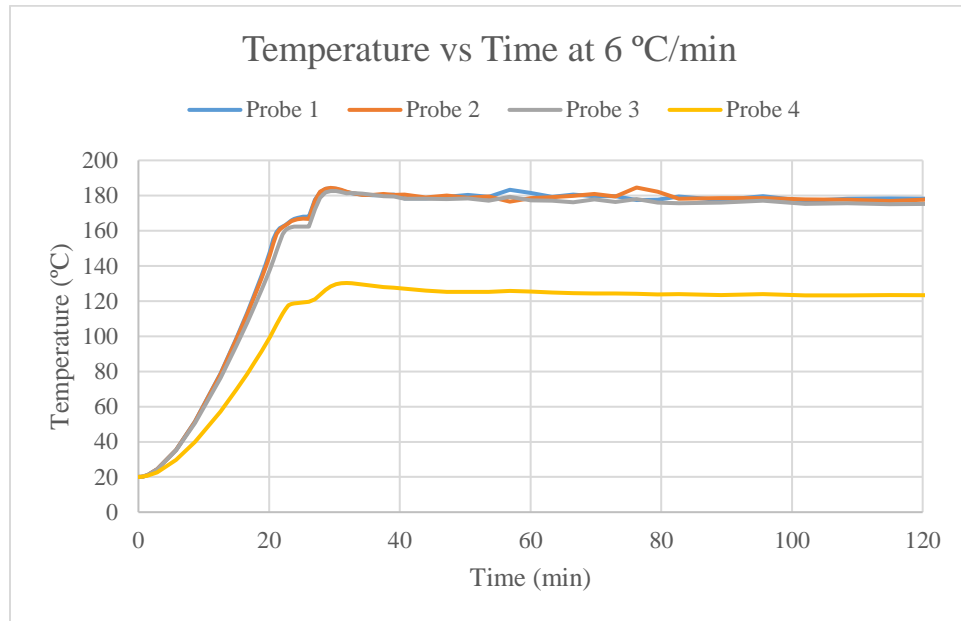


Figure 15. Temperature profile for cure cycle C for 20 mm thick laminate

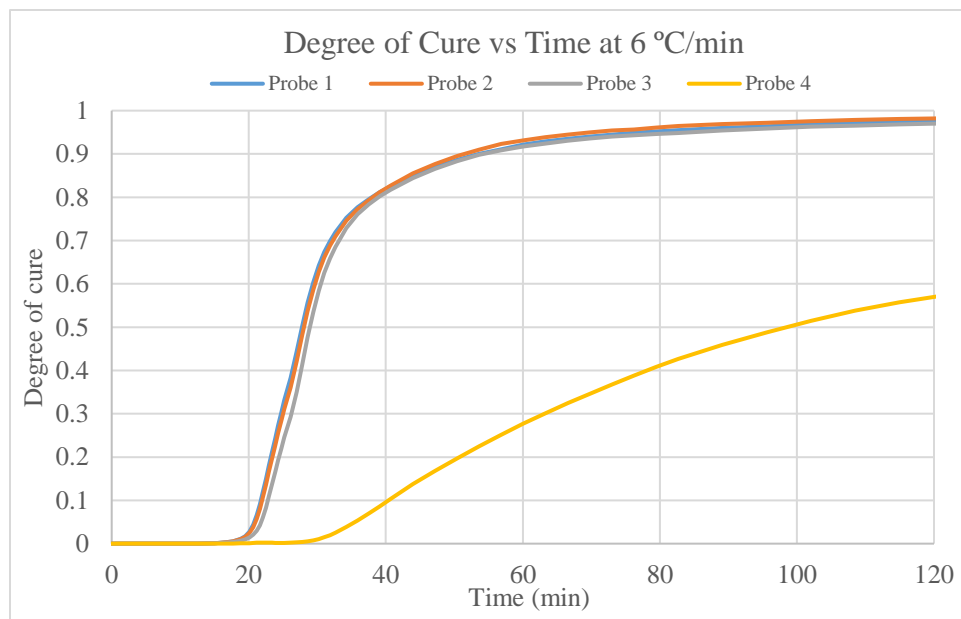


Figure 16. Degree of cure profile for cure cycle C for 20 mm thick laminate

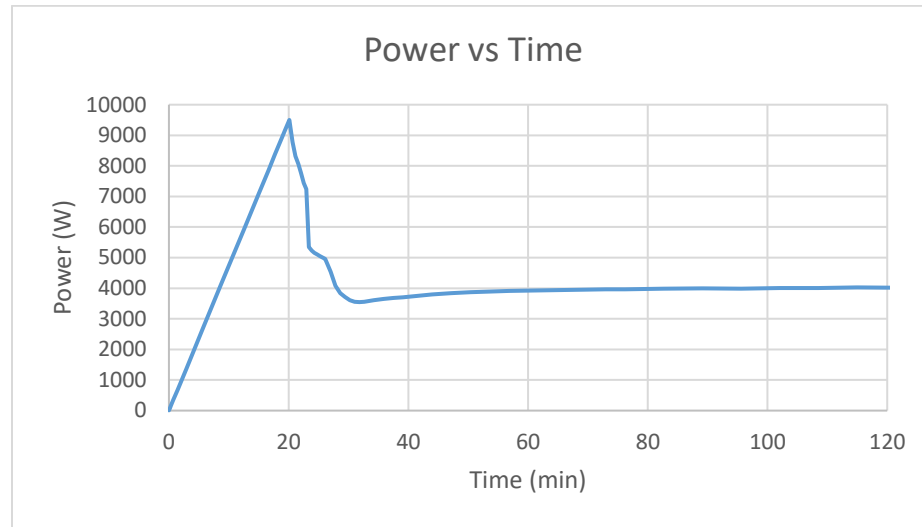


Figure 17. Power input for cure cycle C

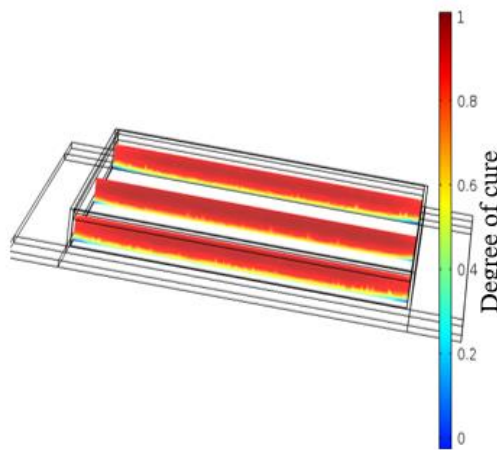


Figure 18. 3D cross sectional degree of cure profile at time =120 min for cure cycle C

4. CONCLUSIONS

In this study, a new multiphysics simulation model was designed to evaluate the microwave curing process of carbon fiber reinforced thermoset composite laminates. Anisotropic properties of the composite and asymmetric microwave distribution in the

laminate was incorporated into the model. The microwave processing model developed was used as a tool to investigate the cure behavior of thick composite laminate.

Comparison between conventional process model and microwave processing model was performed on a 300 mm square laminate with 20 mm thickness. Both the models use the same curing cycle. Thermal lag and temperature overshoot were observed in conventional processing model. The cure cycle was modified over the traditional thermal cure cycle to study the effect of heating rate and holding temperature. Higher heating rates of 3 °C/min and 6 °C/min were studied. Higher heating rates produced more uniform cure than the lower heating rate. The maximum power required to achieve a higher heating rate is more. Two holding temperature, 150 °C and 180 °C, were also studied. The simulation predicts the degree of cure, the temperature distribution in the laminate and theoretical power required by the microwave.

REFERENCES

1. The Paris Agreement: International Action on Climate Change, United Nations Framework Convention on Climate Change; Paris 2015.
2. H. Y. Nezhad, F. Merwick, R.M. Frizzell and C.T. McCarthy, "Numerical analysis of low-velocity rigid-body impact response of composite panels," *International Journal of Crashworthiness*, Vol 20, pp. 27-43, 2015.
3. H. Koerber and P.P. Camanho "High strain rate characterization of unidirectional carbon-epoxy IM7-8552 in longitudinal compression," *Composite: Part A*, Vol 42, pp. 1004-1019, 2011.
4. C.O. Mgbemena, D. Li, M. Li, P. Liddel, L. Daniel, K. Katnam, V. Thakur and H. Nezhad, "Accelerated microwave curing of fiber-reinforced thermoset polymer composites for structural applications: A review of scientific challenges," *Composites: Part A*, Vol 115, pp. 88-103, 2018.

5. L. Khoun, T. Centea and P. Humert, "Characterization methodology of thermoset resins for the processing of composite materials - case study: Cycom 890RTM epoxy resin," *Journal of Composite Materials*, Vol 44, pp. 1397-1415, 2010.
6. J. Balvers, H. Bersee, A. Beukers and K. Jansen, "Determination of cure dependent properties for curing simulation of thick-walled composites," *Proceedings of 49th AIAA/ASME/ASCE/AHS/ASC Structures*, pp. 1–12, Schaumburg, IL, 7–10 April 2008.
7. P. Karkanis, I. Partridge and D. Attwood, "Modeling the cure of a commercial epoxy system for applications in resin transfer molding," *Polymer International*, Vol 41, pp. 183-191, 1996.
8. S. Anandan, G.S. Dhaliwal, Z. Huo, K. Chandrashekhara, N. Apetre and N. Iyyer, "Curing of thick thermoset composite laminates: Multiphysics modeling and experiments," *Applied Composite Materials*, Vol 25, pp. 1155-1168, 2018.
9. L. Zong, S. Zhou, N. Sgriecchia and M.C. Hawley, "Dielectric properties of an epoxy-amine system at a high microwave frequency," *Polymer Engineering and Science*, Vol 45, pp. 1576-1580, 2005.
10. R. Mishra and A.K. Sharma, "Microwave–material interaction phenomena: Heating mechanisms, challenges and opportunities in material processing," *Composites: Part A*, Vol 81, pp. 78-97, 2016.
11. M. Kwak, P. Robinson, A. Bismarck and R. Wise, "Curing of composite materials using the recently developed HEPHAISTOS microwave," *Proceedings of 18th International conference on composite materials*, Jeju Island, Korea, 2011.
12. J. Kratz, K. Hisao, G. Fernlund and P. Hubert, "Thermal models for MTM45-1 and Cycom 5320 out-of-autoclave prepreg resins," *Journal of Composite Materials*, Vol 47, pp. 341-352, 2013.
13. D. Kim, T. Centea and S. Nutt, "Out-time effects on cure kinetics and viscosity for an out-of-autoclave (OOA) prepreg: modeling and monitoring," *Composite Science and Technology*, Vol 100, pp. 63-69, 2014.
14. G. Springer and S. Tsai, "Thermal conductivities of unidirectional materials," *Journal of Composite Materials*, vol. 1, pp. 166-173, 1967.
15. G. Struzziero, B. Remy and A.A. Skordos, "Measurement of thermal conductivity of epoxy resins during cure," *Journal of Applied Polymer Science*, DOI: 10.1002/app.47015, 2018.

SECTION

2. CONCLUSIONS

Research at Missouri S&T based upon developing a finite element multiphysics method determined the appropriate degree of cure and power cycle for microwave curing of fiber reinforced polymers. Comparison between conventional process model and microwave processing model was performed. Thermal lag and temperature overshoot were observed in conventional processing model. Anisotropic properties of the composite and asymmetric microwave distribution in the laminate were included in the model. The microwave processing model developed was used to investigate the cure behavior of thick composite laminate. A parametric study was performed to understand the effect of heating rates on curing. Higher heating rates produced a more uniform cure than the lower heating rate. Power cycle required for the curing process was numerically simulated.

BIBLIOGRAPHY

- [1] R. Benitez, A. Fuentes and K. Lozano, "Effects of microwave assisted heating of carbon nanofiber reinforced high density polyethylene," *Journal of Materials Processing Technology*, Vol. 190, pp. 324-331, 2007.
- [2] Allan W. Scott, "Understanding Microwaves," *John Wiley & Sons, Inc.* 1993.
- [3] Bada J, "Encyclopedia of Astrobiology," *Springer Berlin Heidelberg*, 2011.
- [4] J. E. Green, B. Nuhiju, K. Zivtins, M. P. Bower, R.V. Grainger, R. J. Day and R. J. Scaife, "Internal Model Control of a Domestic Microwave for Carbon Composite Curing," *IEEE Transactions on Microwave Theory and Techniques*, vol. 65, pp. 4335-4346, 2017.

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