Kinetics and temperature evolution during the bulk polymerization of methyl methacrylate for vacuum-assisted resin transfer molding

Yasuhito Suzuki a,b,⇑, Dylan Cousins b, Jerred Wassgren b, Branden B. Kappes a, John Dorgan b, Aaron P. Stebner a

a Mechanical Engineering Department, Colorado School of Mines, Golden, CO 80401, USA
b Chemical and Biological Engineering Department, Colorado School of Mines, Golden, CO 80401, USA

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Curing reactions of methyl methacrylate (MMA) comprise an induction time of gradual temperature change over tens of minutes, followed by a sudden temperature rise within tens of seconds because of auto-acceleration known as the Trommsdorff effect. These curing effects were investigated as initial initiator and polymer concentrations were varied. A mathematical model combining the reaction kinetics with heat transfer was developed and verified in its ability to simulate the processing kinetics and temperature evolutions throughout thick MMA-based parts. It was further demonstrated that the processing conditions at specific points within a part during manufacture could be actively controlled via the Trommsdorff effect by locally varying the initial concentration of poly(methyl methacrylate) (PMMA) solution. Together, these advancements provide an enhanced ability to design and optimize the manufacture of thick, large-scale PMMA materials by taking advantage of auto-acceleration instead of avoiding it.

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1. Introduction

The use of thermoplastic resin systems instead of thermosets can improve the recyclability of composites, shorten the production cycle time, and reduce manufacturing costs. Although the concept has long been proposed, it is still challenging to fabricate thick, large-scale thermoplastic-based fiber-reinforced composites (FRCs) [1]. Vacuum-assisted resin transfer molding (VARTM) is most commonly used to fabricate large FRCs like wind turbine blades [2] and ships [3]. With this method, fibers are placed on a mold and shielded with a vacuum bag and vacuum tape. A low-viscosity thermoset resin and hardener are infused with the aid of a vacuum. The resin is then cured at a high temperature [4,5]. There are two approaches to using thermoplastic resin: melt infusion and reactive processing. Because the viscosities of polymer melts are too high at a reasonable temperature in a vacuum, reactive processing is a practical approach. Promising systems include ring-opening polymerization of nylon [6–10] and free-radical polymerization of acrylics [1]. In this study, we focus on the poly (methyl methacrylate) (PMMA)-based system.

Acrylics are widely used in many applications, including window glass substitutes and FRCs. An acrylic polymer such as PMMA is cost-efficient and provides comparable tensile modulus to epoxy. PMMA can be synthesized via free-radical bulk polymerization of MMA [11,12]. The PMMA reaction can be initiated by benzoyl peroxide in the presence of an amine at room temperature. In other words, a separate heating device is not required for the initiation of the reaction. A key issue to be addressed is the thermal management during the curing reaction. The heat of polymerization of MMA to PMMA is 57.8 kJ/mol [13], which is around three times greater than a typical epoxy resin. In addition, it is known that the bulk free-radical polymerization reaction will auto-accelerate due to the gel (Trommsdorff) effect [14]. The phenomenon originates from the sudden drop of the termination rate due to the increased viscosity of the resin [15,16]. After an induction period, the temperature increases dramatically. To make high-quality parts, thermal runaway has to be avoided; the temperature should not exceed the boiling point of the monomer, which is approximately 100°C at ambient pressure [13].

Thus, to design the manufacturing of PMMA parts, it is desirable to predict the induction time, the maximum temperature, and the total reaction time. Toward this goal, we proceed to study the curing kinetics of free-radical polymerization of MMA with various initial initiator and polymer concentrations. We then develop a
model that can be used to design the manufacture of parts. Bulk polymerization curing kinetics of PMMA have been extensively investigated in the past [15–20]. Although most models still require some assumptions or artificial functions to capture the gel effect, they can be reasonably calibrated to predict the curing kinetics [21]. In this study, we advance these models by combining a curing model [19] and a one-dimensional heat transfer equation to simultaneously predict the temperature evolution. This combined model is desired to design the manufacturing process for large, thick parts. For thermostat resins, a simple model or even just differential scanning calorimetry (DSC) data can be used to design an infusion process, but the auto-acceleration nature of bulk free-radical polymerization is very sensitive to the initial conditions and boundary conditions, namely, the temperature evolution during the cure. Lastly, we draw upon the Trommsdorff effect to propose and demonstrate a method for further active temperature control throughout the part by locally grading the viscosity of the resin through variations in the initial PMMA concentrations to achieve different curing kinetics and thermodynamics at different material points within the parts.

2. Methods

(a) Materials

Methyl methacrylate (MMA), N,N-dimethyl-p-toluidine (DMT) and benzoyl peroxide (BPO: Luperox® AFR40) were purchased from Sigma-Aldrich. The MMA inhibitor was removed using pre-packed column inhibitor removers (Sigma-Aldrich). Poly(methyl methacrylate) (PMMA) (M_w = 99 kg/mol, PDI = 1.2) was obtained from commercial sources and used as received. For all but one experiment, the resin was formulated with 800 g of MMA, 200 g of PMMA and 5.3 g of N,N-dimethyl-p-toluidine. For the experiment studying the effect of pre-dissolved PMMA (Fig. 6), the molar ratio of MMA, N,N-dimethyl-p-toluidine, and BPO (269:1:1) were kept constant while the amount of pre-dissolved PMMA was varied. In this study, “cure” refers to the in situ polymerization of resin.

(b) Temperature measurement using thermocouples

Temperature as a function of time was measured using thermocouples and a data logger from National Instruments. J-Type thermocouples were connected to a 4-Ch thermocouple input (NI 9211), and data were collected with a USB data logger (NI cDAQ-9171). For the small-scale experiments, resin and initiator with a total weight of 6.0 g were well mixed in a 20 ml scintillation vial. To avoid evaporation, a hole was drilled in a lid with an aluminum backing, and a thermocouple was inserted through the hole. In addition, the gap between the lid and the thermocouple was sealed using a vacuum bag sealant tape. The scintillation vial with the sample was placed in a constant temperature stirred oil bath.

(c) Temperature measurement during the VARTM with an IR camera

An IR camera (FLIR A325sc) was used to record the temperature evolution during the cure of a panel fabricated via the VARTM process. A VARTM setup was developed to infuse a 30 cm by 30 cm panel. Glass fibers, peel ply, and infusion flow media (purchased from Fibre Glast) were placed on a glass mold and sealed with vacuum tape and a vacuum bag. The MMA-based resin was then infused. The IR camera was positioned at a height of about 70 cm on a tripod set at a 90° angle capture the heat signature of the infusion and subsequent cure.

(d) Simulation technique

The coupled equations of curing kinetics and heat transfer that we develop in Section 3 were numerically solved using Wolfram Mathematica (version 11.0). As shown in the supporting information (Fig. S1), typical radical concentration was on the order of 10^{-16} lower than the initial monomer concentration; thus, the computation requires high working precision. Hence, 24 digits working precision was used. An accuracy goal, which specifies the effective digits of accuracy in the final result, was set to 18 digits for this computation, which showed convergence and stability relative to a choice of 10 digits (Fig. S1).

For the simulations that accounted for spatial geometry (Figs. 7 and 8), the model parameters were simplified to speed up the computations (a single simulation of the full kinetic model had not completed in 2 weeks on a 32-core, 256 GB RAM workstation). The kinetic parameters are treated as constants, using their values at 323 K. In addition, thermal conductivity (0.12 W/(m K)), density of the matrix (2.0 kg/m³), and the heat capacity (1.4 kJ/(kg K)) are held constant using the averaged values of PMMA and glass fibers.

3. Results and discussion

3.1. Temperature profile during the polymerization reaction

Fig. 1 shows an example of the temperature profile during the curing reaction of the MMA-based resin. The resin contained 20 wt% pre-dissolved PMMA and exhibited a comparable viscosity to epoxy resin. The reaction was initiated by benzoyl peroxide in the presence of an amine (N,N-dimethyl-p-toluidine). The redox reaction of benzoyl peroxide by the amine generates radicals, even at room temperature [29,30]. The temperature gradually increased for the first 35 min. During this time, the termination rate of the growing macroradicals was governed by the ability of the radical chain ends to diffuse and orient to each other. This phenomenon is known as segmental diffusion. Translational diffusion (movement of the entire macroradical through the solution) is not hindered at the beginning of the reaction because the solution is dilute. Once the solution becomes concentrated enough, termination is further limited by translational diffusion. During this period the temperature of the solution begins to display a noticeable increase, though it is not extremely rapid. Translational diffusion was observed from 35 to 40 min in Fig. 1. After this initial induction time through the first two phases, the temperature of the resin...
begins to increase dramatically. Within 3 min, the temperature changed from 35°C to 70°C. This auto-acceleration of the reaction kinetics is known as the Trommsdorff effect. This effect arises due to both the entire macroradical and its chain ends being trapped in the viscous and entangled solution. At this point, the rate of termination drops dramatically, but the monomer units are still able to diffuse through the viscous liquid. Therefore, the overall reaction rate increases dramatically.

The increased viscosity affects kinetic parameters; in particular, the rapid drop in the termination rate impacts the total kinetics. The individual radicals are trapped due to the high viscosity of the matrix, and the chance to terminate with another radical decreases. As a result, more and more radicals continue reacting, and more heat is generated per a unit time. The generated heat increases the temperature and further speeds up the reaction; hence, the system has an auto-acceleration nature. For the application purpose to FRCs, the induction time corresponds to the pot life of the resin. The total reaction time determines the cycle time. In addition, controlling the maximum temperature below the boiling point of the monomer is critically important to avoid the bubble formation.

In this study, the temperature evolution was tracked using thermocouples, as shown in Fig. 2 (left). A total of 6 g of the resin and the initiator were mixed in the scintillation vial. The scintillation vial was placed in a constant temperature oil bath. A hole was
drilled in the lid, and a thermocouple was inserted through the hole. To minimize the evaporation, the gap between the thermocouple and the lid was sealed with a yellow vacuum tape. Fig. 2 (right) plots the temperature profile during the cure for different amounts of the initiator. The temperature profiles strongly depend on the initiator concentrations. While the induction time was $t_\text{ind} \approx 60$ min with 0.5 wt% initiator concentration, it shortened to $t_\text{ind} \approx 15$ min with 5.0 wt% initiator concentration. Furthermore, the maximum temperature tends to become higher with a higher initiator concentration. The temperature profile also depends on the heat dissipation by the heat transfer; thus, the volume-to-surface ratio of the container, the materials of the container, and the surrounding environment affect the temperature profile.

### 3.2. One-dimensional heat transfer model

As mentioned in the introduction, predicting the pot life, the production cycle time, and the maximum temperature during the cure enables better applications of the resin. The temperature profile is a result of the competition between the heat generated by the reaction and the heat dissipated by the heat transfer. Assuming that the heat generated is proportional to the monomer consumed by chemical reaction, and heat transfer can be approximated by a simple one-dimensional term, temperature ($T$) change as a function of time ($t$) by chemical reaction and a simple one-dimensional heat transfer can be written as follows:

$$\frac{dT}{dt} = \frac{\Delta H_{\text{PMMA}}}{m \cdot c_p} \cdot v \cdot \left( -\frac{dM_{\text{MMA}}}{dt} \right) - \frac{h \cdot A}{m \cdot c_p} (T(t) - T_\text{cold})$$  \hspace{1cm} (1)

In Eq. (1), $\Delta H_{\text{PMMA}}$ is the heat of polymerization of PMMA, $m$ is the total mass, $v$ is the total volume, $c_p$ is the heat capacity, $M_{\text{MMA}}$ is the mass of the monomer (MMA), $h$ is the heat transfer coefficient and $T_\text{cold}$ is the temperature of the surroundings. In Eq. (1), the first term is the heat generation by the polymerization reaction and the second term denotes the heat dissipation by the heat transfer to the surroundings.

To determine the first term in Eq. (1), a model of the reaction kinetics is needed. After the research by Trommsdorff, a number of studies have attempted to model the kinetics of bulk free-radical polymerization. The difficulty in developing a kinetic model is that all the kinetic parameters are functions of both conversion and temperature in a complex manner. As conversion increases, the solution changes from dilute regime to semi-dilute regime and, eventually, concentrated regime [22–24]. Depending on the concentration and molecular weight of the polymer in the resin, the polymer starts to be entangled at some point of the reaction. The property of the resin affects translational diffusion, rotational diffusion, and segmental dynamics of polymer chains. To take all
the factors into account, models require some assumptions or empirical statements. Nevertheless, some of the models are successful in predicting the curing kinetics.

Achilias et al. demonstrated a mathematical model for an acrylic resin initiated with BPO and an amine for application in the field of dentistry [18] based on a previously developed mathematical framework [17]. Recently, Zoller et al. reported a mathematical model of MMA polymerization initiated by BPO in the presence of N,N-dimethyl-p-toluidine [19]. In the current study, we followed these two approaches. The model used herein is based on the free-volume ideas. It assumes that the free-volume change due to the reaction governs the diffusion controlled initiation and propagation. The effect of free-volume on the initiation was modeled by introducing the efficiency parameter of the initiation reaction (Table S3, Eq. (S6)). The efficiency parameter is an empirical equation and was adjusted to capture the experimental result (Table S2). For the propagation, the free-volume change was related to the diffusion coefficient of the monomer as described in Eq. (2).

\[ D_m(t) = D_{m,0} \cdot \exp \left( -\frac{\gamma_m}{V_f[t]} \right) \]  

(2)

Here, \( V_f[t] \), \( D_{m,0} \), \( D_m[t] \) and \( \gamma_m \) denote the free volume, the initial diffusion coefficient, the diffusion coefficient, and the overlapping factor, respectively. The diffusion coefficient of monomer affects the effective kinetic parameters, as shown in Eq. (3).

\[ \frac{1}{k_{\text{eff}}[t]} = \frac{1}{k_0} + \frac{1}{4\pi N_A r_p D_m[t]} \]  

(3)

Here, \( k_{\text{eff}}[t] \), \( k_0 \), \( N_A \), and \( r_p \) express the effective kinetic parameter, the initial kinetic parameter, Avogadro’s constant, and the reaction radius, respectively. In Eq. (3), the first term \( (k_0) \) is kinetically controlled, while the second term \( (4\pi N_A r_p D_m[t]) \) is controlled by the diffusion [21]. At a higher conversion, the second term dominates the effective kinetic parameter. The same equation structure was used for the transfer coefficients as well.

The kinetic scheme for the polymerization initiated by BPO in the presence of an amine is presented in Scheme 1. In addition to the main reactions of decomposition, initiation, propagation, and termination, side reactions by chain transfer reactions are also considered. Based on Scheme 1, balance equations can be derived, as follows:

**Decomposition:**

\[ I + A \stackrel{k_{\text{decomp}}}{\rightarrow} I^* + A^* \]

**Initiation:**

\[ I^*/A^* + M \rightarrow P_n^* \]

**Propagation:**

\[ P_n^* + M \rightarrow P_{n+1}^* \]

**Termination:**

\[ P_n^* + P_n \rightarrow P_{n+m} \]

\[ P_n^* + P_n \rightarrow P_{n+m} \]

**Side Reactions:**

\[ I/A + P_n \rightarrow P_n^* \rightarrow I^*/A^* + P_n \]

\[ M + P_n^* \rightarrow M^* + P_n \]

\[ I^*/A^* + P_n^* \rightarrow I + A + P_{n+1} \]

\[ I = \text{initiator}, \]

\[ A = \text{amine}, \]

\[ P = \text{polymer}, \]

\[ M = \text{monomer}. \]

Scheme 1. Kinetic scheme for free-radical polymerization initiated by benzoyl peroxide in the presence of an amine. The superscript * indicates radicals.
was defined as follows: 20 wt% of pre-dissolved PMMA was added, the conversion (x) of the experiment and of the model agree. The obtained heat transfer coefficient so that the maximum temperatures due to the evaporation of the monomer from the surface. As the reaction proceeds, some parameters (i.e., density, heat capacity, and thermal conductivity) change because the amount of PMMA in the matrix increases. However, these changes are small, and they are treated as constants using average values in the model. One parameter that needs to be obtained from the experiment is the heat transfer coefficient. For practical applications, the heat transfer coefficient depends a lot on the material, the geometry, and the surrounding environment of the mold. It is therefore necessary to obtain or estimate a mold-specific heat transfer coefficient. Here, we used one of the experimental data and adjusted the heat transfer coefficient so that the maximum temperatures of the experiment and of the model agree. The obtained heat transfer coefficient was fixed for the rest of the simulation in this study. The simulated results are plotted in Fig. 3 (solid line). As a comparison, the corresponding experimental data are also presented in Fig. 3 (dashed line). The model reasonably captures the induction time and the maximum temperature when the initiator concentrations are changed.

The model also provides the profile of all the parameters as a function of time. Some of them are difficult to access experimentally. Fig. 4 depicts the conversion as a function of time. Because 20 wt% of pre-dissolved PMMA was added, the conversion (x(t)) was defined as follows:

$$x(t) = \frac{M(t)}{M(0)} - M(t)$$

Here, M(0), M(t), and P_0 denote the initial monomer concentration, the monomer concentration at time t, and the relative amount of pre-dissolved PMMA, respectively. Because of the definition, the conversion starts from 0.2 in Fig. 4. The final conversion decreases as the initiator concentration decreases. The model implies that the Trommsdorff effect starts at a conversion of 0.4.

3.3. The effect of pre-dissolved PMMA and an application

The previous result described in Fig. 5 allows for another way of controlling the reaction time. Because the onset of the Trommsdorff effect correlates with the viscosity of the matrix, the amount of pre-dissolved PMMA can be manipulated to control the induction time and the cycle time. Fig. 6 displays the effect of pre-dissolved PMMA. In this experiment, the molar ratio of reactive species (i.e., MMA, N,N-dimethyl-p-toluidine, and BPO) was kept constant. The induction time shortens from 45 min (0 wt% PMMA) to 8 min (30 wt% PMMA). It is noted that if it were a solvent instead of pre-dissolved PMMA, it would just dilute the concentrations of the reactive species and slow the total reaction time.

The mathematical model can simulate these phenomena. The different amounts of pre-dissolved PMMA were inserted in Eq. (4). The computed result is provided in Fig. S2. While the model qualitatively predicts the shortening of the induction time by adding the pre-dissolved PMMA, the obtained induction times are slightly different from the experiments. In addition, the model shows a decrease of the maximum temperature with addition of pre-dissolved PMMA. This trend is not observed in the experimental result. One possible explanation is that the model does not take into account the effect of molecular weight distribution. It is known that the molecular weight distribution of bulk free-radical polymerization is very broad [19]; thus, the matrix, in reality, becomes very inhomogeneous. The inhomogeneous environment may affect the reaction kinetics (i.e., the induction time and the maximum temperature).

In a practical application for VARTM processing, the desired viscosity range of the initial resin is predetermined by the vacuum setups and the mold; thus, the control of the induction time by the amount of pre-dissolved PMMA in the resin is limited. On the other hand, the phenomenon can be used to locally control the

### Balance equations:

1. Concentration of the initiator

$$\frac{d[I]}{dt} = -f \cdot k_A \cdot [I][A] - k_{A,p} \cdot [I][P^*]$$

2. Concentration of the amine

$$\frac{d[A]}{dt} = -f \cdot k_A \cdot [I][A] - k_{A,p} \cdot [A][P^*]$$

3. Concentration of the initiator radicals

$$\frac{d[P^*]}{dt} = f \cdot k_A \cdot [I][A] - k_{A,p} \cdot [I][P^*] + k_{A,p} \cdot [I][P^*]$$

4. Concentration of the amine radicals

$$\frac{d[A^*]}{dt} = f \cdot k_A \cdot [I][A] - k_{A,p} \cdot [A][P^*] + k_{A,p} \cdot [A][P^*]$$

5. Concentration of the monomer

$$\frac{d[M]}{dt} = -(k_{11}[P^*] + k_{1A}[A])[M] - k_{2}' \cdot [P^*][M] - k_{2,r,M} \cdot [M][P^*]$$

6. Concentration of the polymer radicals

$$\frac{d[P]}{dt} = (k_{11}[P^*] + k_{1A}[A])[M] - (k_{2}' + k_{2,r,M}) \cdot [P^*]^2 - (k_{A,p}[I] + k_{2,1A}[A])[P^*] - k_{2,r,M} \cdot [P^*][M] - (k_{2}'[P^*] + k_{2,r}[A])[P^*]$$

### Heat transfer equation:

$$\frac{dT}{dt} = \frac{\Delta H_{PMMA}}{\rho \cdot c_p} - \frac{d[M]}{dt} \cdot \frac{h \cdot A}{m \cdot c_p} \cdot (T(t) - T_{cool})$$
reaction kinetics by a local viscosification. The concept is demonstrated in Fig. 6. A highly concentrated solution of PMMA in MMA (40 wt%) was placed on the four spots of glass fiber sheet as indicated by black circles. One more glass fiber sheet was placed on top to avoid the direct contact of the high-concentration PMMA solution to the peel ply. Then, a VARTM infusion was conducted as usual using a resin containing 20 wt% pre-dissolved PMMA. The temperature profile during the cure was captured using an IR camera. The IR camera image shows that the onset of the Trommsdorff effect started at the same position where a high-concentration PMMA solution was pasted. The infused resin was locally viscosified at the spots, so the kinetic parameters change at these positions, accelerating the onset of the Trommsdorff effect. To quantify this effect, temperature profiles at two different spots are extracted and plotted in Fig. S3. Instead of a high-concentration PMMA solution, a thin film of PMMA can be placed for the same effect. Since the phenomenon originates from the local viscosification, another polymer that is miscible with MMA can also be used. One important parameter is the dissolution kinetics to the resin. The dissolution must be completed in a reasonable time range to control the phenomenon. For a practical application, a further engineering is needed, including determining how to place the reaction spots. The method opens a possibility to cure the resin without any extra heating device. Because the reaction is highly exothermic, the active control of the onset of the Trommsdorff effect enables regulation of the temperature within a desired range.

3.4. Two-dimensional model

The developed model can be expanded taking into account spatial geometry. Here, the model shown in Fig. 7(a) is considered. The resin and the glass fibers are placed in between two walls with fixed temperatures. The temperature profile can be written as follows:

\[
\frac{dT}{dt} = \frac{\Delta H_{\text{PMMA}}}{\rho \cdot \alpha_{\text{r}}} \left( -\frac{dM_{\text{MMA}}}{dt} - \frac{\kappa}{\rho \cdot \alpha_{\text{r}}} \left( \frac{\partial^2 T}{\partial x^2} \right) \right)
\]  

(5)

The differences from Eq. (1) are the \( \kappa \) and \( x \) parameters, which denote thermal conductivity and the distance between the walls, respectively. Eq. (5) can be coupled with the balance equations in Scheme 2. A computational challenge is that the differential equations are very stiff. Combined with the huge dynamic range of the concentration of reactive species, the computation becomes expensive. For a qualitative understanding with a reasonable computation time, we fixed kinetic parameters at 323 K. A computational result with a 30 cm thick wall is shown in Fig. 7(b). Because of the simplified kinetics, the induction time and the maximum temperature are not precise; however, the computation captures the qualitative temperature profiles as functions of time and position.

Using the extended model, the dependence on wall thickness was studied. Fig. 8 shows the temperature profile as a function of time and position at different thicknesses. The temperature is scaled from 27 °C to 100 °C. The important outcome is that the maximum temperature at the center position strongly depends on the thickness. The thicker the panel, the more difficult it is to dissipate heat within a given time frame. In this example, while the temperature only reached 40 °C at a thickness of 20 cm, it exceeded the boiling point of the monomer at a thickness of 40 cm. These results imply that for a given chemical formulation of the resin and the heat transfer from the mold, there exists a maximum wall thickness that can be used to avoid the boiling of the monomer.

4. Conclusion

Reaction kinetics and temperature evolution during the polymerization of MMA were investigated. Because of the Trommsdorff effect, the temperature increases rapidly after an induction time. The important parameters for VARTM (i.e., pot life, cycle time, and the maximum temperature) depend on the chemical formulation and the heat transfer. A mathematical model based on the reaction kinetics and heat transfer equation was developed to simulate these behaviors. Furthermore, it was demonstrated that the addition of pre-dissolved PMMA increases the viscosity of the resin and changes the kinetic parameters locally. We specifically showed that the induction time shortens from 45 min for 0 wt% PMMA to 8 min for 30 wt% PMMA. This phenomenon can be used intentionally to control the position where the Trommsdorff effect starts. The concept was demonstrated by placing 40 wt% PMMA solution at four spots during the VARTM cure. Lastly, it was demonstrated that the mathematical model can be used to calculate the maximum thickness allowed to cure the resin without reaching the boiling point of the monomer with fixed chemical formulation and thermal conductivity. These results will be particularly advantageous for designing the manufacture of thick fiber-reinforced materials with PMMA matrix, especially as geometries become complex, such as geometries of variable thicknesses.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.compositesa.2017.10.022.

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