JOURNAL OF Applied Polymer SCIENCE

Compensation of Microwave-Source Heating Attenuation into Epoxy-Glass Composites: Experimental Results

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ABSTRACT: The final goal of this study was to manufacture an epoxy–glass leaf spring by microwave processing. The physical properties of the final part to be manufactured, in particular, the mechanical properties, were directly related at the repartition of microwave-source heating during the treatment. The major problem in microwave processing, however, is the attenuation of the microwave source. Here, we propose a dielectric effect of attenuation inversion of the electromagnetic waves as a new method for the uniform treatment of epoxy–glass by microwave energy. This solution used the dielectric properties of the mold to control the microwave-heat-source attenuation into the composite to be treated. Many experiments were carried out to validate the proposed solution. The results show that the microwave-source heating attenuation could controlled and inversed. We demonstrated the uniform treatment of epoxy–glass parts about 100 cm long by means of the compensation of microwave-source attenuation. © 2013 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2014**, *131*, 39908.

KEYWORDS: composites; crosslinking; dielectric properties; manufacturing

Received 8 November 2012; accepted 25 August 2013
DOI: 10.1002/app.39908

INTRODUCTION

The production of auto parts with polymer-based composite materials requires physicochemical transformation of the components of the matrix. The application of this type of material for large-scale and continuous industrial production of auto parts is often difficult because of the excessive duration of the manufacturing cycle at lower temperatures. Indeed, to shorten this time, it is necessary to raise the temperature of the materials to accelerate the physicochemical transformation. On the other hand, it is well known that polymeric materials are usually low in thermal conductivity and high in thermal degradability. These characteristics inevitably limit the possible increase in temperature and hence the possible reduction of the production time.

The application of internal thermal sources created by the dielectric hysteresis phenomenon offers an alternative[1] because it makes it possible for them to act quickly on materials with poor thermal conductivity in several minutes[2] without the risk of overheating the material surface. The use of microwaves in the manufacturing of polyurethane in car seats was first reported by Morey in 1963.[3] In 1974, Kawasa and Hayakawa[4] reported the preparation of composite materials by the microwave crosslinking of unsaturated polyester resins charged with glass balls. In the following year, the same method was successfully applied to the reticulation of polycondensation resins, such as polyamides, polyimide, and polyester resins, phenolic or epoxydic.[5] Wilson and Salerno[6] studied the reaction kinetics of the curing process of an epoxy resin with an amino hardener in a microwave field. The reticulation of epoxy–glass, carbon–epoxy, resin polyesters, polyurethane, and other varieties of composite materials with microwave heating was studied extensively during the 1980 s.[7–12]

Research results reported by Jullien and coworkers[13,14] showed that compared to conventional heating, the reticulation of composite materials with microwaves could not only reduce the processing time by a factor of up to 5and save energy consumption by a factor of up to 10 but also improve the mechanical performance of the final products. Other results[15] showed that the reticulation of pure epoxy resin (diglycidyl ether of bisphenol A with diamino-4,4′-dimethyl-3 (DGEBA)-3′-dimethyldicyclohexylmethane (3DCM)) with microwaves the as energy source improved its mechanical properties compared to those

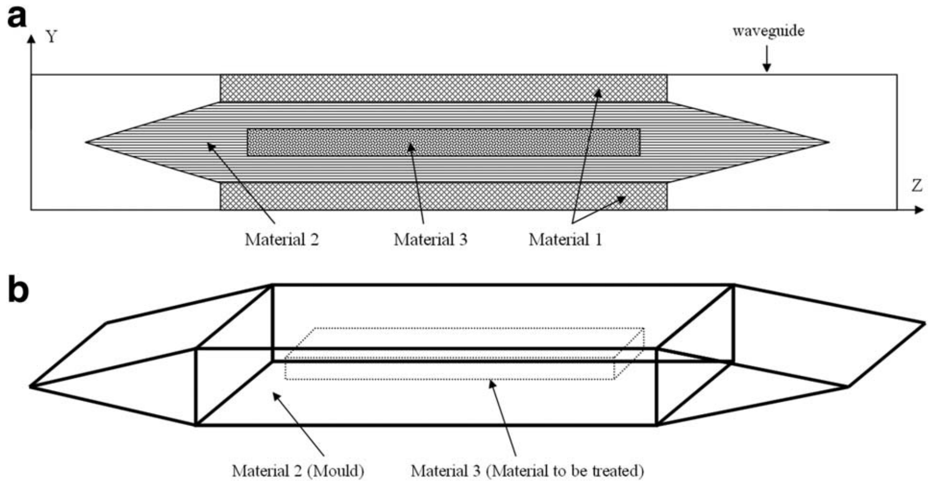

Figure 1. Without DEAI case: (a) waveguide filled by three materials and (b) the mold and the treated material.

obtained by a conventional process. These authors further studied the curing of epoxy resin and polyurethane in pulsed microwave fields.[16–18] According to their results, the use of microwaves in pulsed mode reduced the treatment time and, at the same time, had no influence on the physicochemical structure of the prepared material.

Polymerization of the thermosetting matrices by microwave heating has instigated great research interest.[19–27] Hord[28] studied the transmission of waves at a frequency of 8.2 GHz from one media to another media with a higher permittivity (ε') to find a solution to minimize wave reflection at the interface. Moreno[29] and Outifa[30] experimentally optimized the bevel shape for the best interface effect at frequencies of 8.2 and 2.45 GHz, respectively. Jules[31] carried out thermal modeling of microwave heating on a short sample (<20 cm in length) made of epoxy–glass composite at a frequency of 2.45 GHz. Hug[32] compared mechanical responses under a high-speed loading of carbon–epoxy laminates prepared by a conventional thermal curing process and by an alternative process based on microwave heating. Hug used the alternative process to compensate for the attenuation in the short sample (<20 cm in length). Only a few research studies, however, focused their attention on the issues of attenuation and repartition of microwave in long composite parts, the length of which are typically no less than 20 cm.

Douadji and Delmotte[33–35] proposed that uniform microwave heating treatment could be achieved by the control of the repartition of the electric field into composite materials. They carried out a theoretical study on electromagnetics and thermal properties to demonstrate the possibility of using the dielectric effect of attenuation inversion of electromagnetic waves (DEAI) for the uniform treatment of long and thick parts made of materials with strong electromagnetic absorption, such as epoxy–glass composites.[36–39] To achieve uniform thermal transformation of a composite material by dielectric hysteresis, it is crucial to ensure a uniform spatial distribution of the electric field. This distribution depends directly the increase in the temperature.[40,41] The propagation of an electromagnetic wave in an empty parallelepiped waveguide in fundamental mode (TE01) leads to the most uniform spatial distribution of the electric field. Propagation in any other mode creates a not-so-uniform spatial distribution of the electric field.

In the case of a waveguide filled symmetrically and completely by three different dielectrics, as shown in Figure 1(a), the spatial distribution of the electric field in the waveguide depends on both the dielectric properties of the filling materials and their dimensions. It is possible to maintain uniformity (TE01) in a waveguide filled by three different dielectrics through respect of the conditions relating to these dielectric characteristics.[42,43] As a matter of fact, in the direction *X* of the space, which is also the direction of the electric field vector, the field amplitude is constant. In direction *Y*, the amplitude of the electric field follows either a sinusoidal law with a maximum or an approximate law with a widened maximum plateau; this depends on the properties of the filling dielectric materials. In direction *Z*, the field amplitude is attenuated along the direction of wave propagation because of energy absorption by material 3, which is the treated material and placed in the middle, and the weaker absorption by material 2, which represents the dielectric mold [Figure 1(b)].

The results of electromagnetic modeling show that the longitudinal distribution of the electric field in material 3 strongly depends on the thickness of material 2. Therefore, it is reasonable to believe that attenuation of the wave intensity in material 3 can be essentially eliminated[42] by the proper variation of the thickness of the dielectric mold (material 2) along the direction of the electromagnetic wave propagation, as illustrated in Figure 2(a,b). For specific values of the dimensions of material 2, we observed attenuation in the direction opposite to the electromagnetic propagation.[37] This is the so-called DEAI. In Figure 2(a), R_{ent} and R_{exi} represent the ratios of the thickness of material 2 to that of material 3 at the entrance and exit of material 3, respectively.

The experimental results reported in this article of the microwave treatment of epoxy–glass matched our earlier theoretical prediction and further suggested that DEAI is a way to ensure

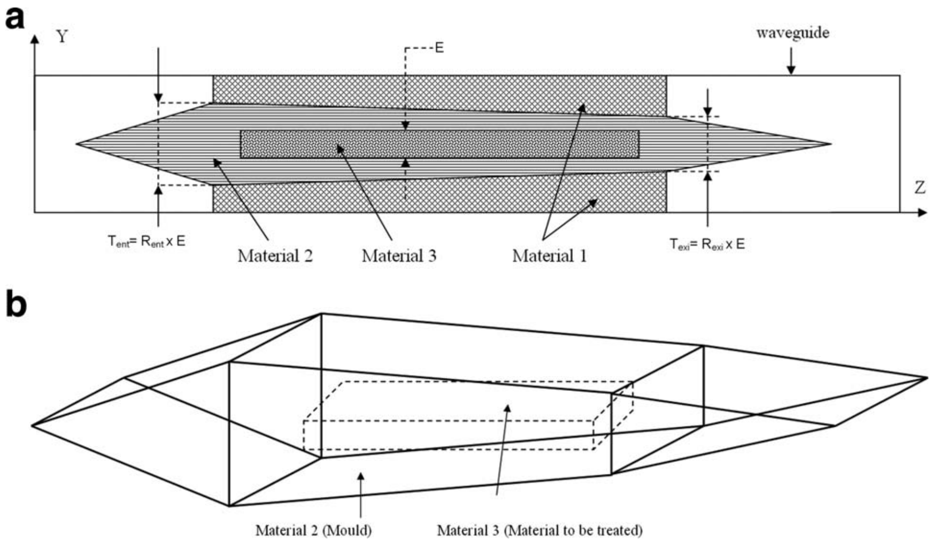

Figure 2. With DEAI case: (a) waveguide filled by three materials and (b) the mold and the treated material.

the uniform treatment of long composite parts in a microwave field.

EXPERIMENTAL

Microwaves Process

The process microwaves required a parallelepiped waveguide where the wave was propagated in TE01 filled by three dielectrics, in which one (material 2) represented the mold of the part to treated. The applicator or waveguide had a parallelepiped form ($0.12 \times 0.25 \times 2$ m^3) in aluminum (Figure 3). The fundamentals of electromagnetic theory, dielectric response, and applications of microwave heating to materials processing, especially fiber composites, were reviewed by Thostenson and Chou.[44]

Materials

For all of the experiments, we used two completely different materials on the dielectric and thermal plans. The first material (material 1) used was polyethylene (PE) with a honeycomb structure. The origin of this material was NIDAPLAST Composites Co., and the material was 10 mm thick. Because of these dielectric properties, this material interacted little with the waves, offering little reflection to the waves and little electromagnetic energy absorption. This material had an ε' close to 1 ($\varepsilon' = 1.10$) and a dielectric loss (ε'') of about 0.001. This material thus played the role of support to maintain the mold (material 2) and the part (material 1) in the median plane of the applicator (waveguide).

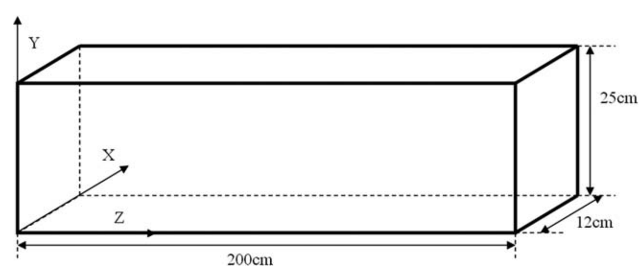

Figure 3. Waveguide in aluminum and their dimensions.

The second material (material 2) was the silicon glass composite. The origin of this material was Isotac Isolant du Sud-Est Co. This material represented the mold. Because of these dielectric properties, this material surrounded the part to obtain an effect of concentration of the waves in the median plane of the applicator. This phenomenon was due to its dielectric properties, which were much higher than those of the PE honeycomb. It had an ε' of about 5 and a characteristic of absorption of about 0.03, that is, 30 times greater than that of the honeycomb PE.

The treated material was an epoxy–glass composite. This material was preimpregnated Vicotex M10/46%/664-120 cm. The long fibers glass were oriented in one direction and were impregnated in the epoxy matrix. This matrix was named DGEBA–dicyandiamide(DDA). The composite was of industrial origin and was presented under the long band aspect in the roller, whose layers were protected by an intercalated sheet from PE to prevent the pasting of the sheets between them.

The values of the dielectric properties (look Table I) of the PE and silicone glass composite were taken from the literature.[29,31] However, the epoxy–glass composite was characterized in the Materials Engineering Laboratory by a conventional method. The method used to obtain the dielectric properties of epoxy–glass was called small disturbances of a cavity resonator.

This method was developed in the Materials Engineering Laboratory in 1988 by Ollivon et al.,[2] and it has been applied since

Table I. Dielectric Characteristics of the Three Materials

Material	ε'	ε''
Material 1: Epoxy-glass	5.50	0.30
Material 2: Silicone glass	4.50	0.03
Material 3: Honeycomb PE	1.10	0.001

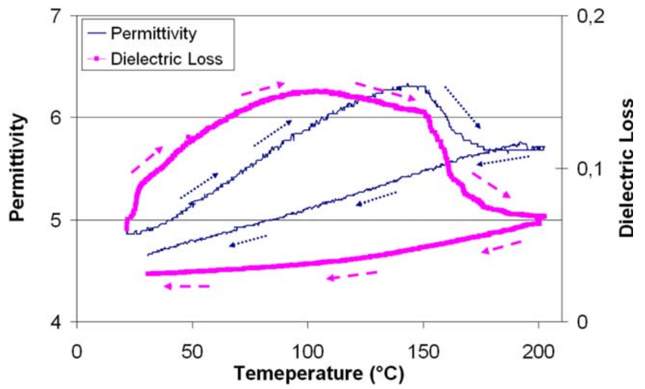

Figure 4. Variation of the dielectric characteristics at 915 MHz of the epoxy–glass according to the temperature (the evolution follows the arrows direction). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

then and remains one of the simplest and most precise methods. This method allows the measurement of ε' and ε'' simultaneously. The principle involves the placement of a small amount of material into a rectangular resonant cavity TE_{13} single-mode type. Dielectrics characterization was performed at a frequency of 2.45 GHz on a sample of 0.12 g, and the results were extrapolated to a frequency of 915 MHz of this measurement according to the methods reported in the literature.[45–47] The dielectric properties of the epoxy–glass composite were obtained by the imposition of the thermal cycle in three stages. The first stage consisted of an increase in the temperature from room temperature to 200°C for 10 min. The second stage was the maintenance of the temperature of 200°C for 2 min, and in the third phase, we allowed the sample to cool down to room temperature during a 10-min time period. For details of the characterization, refer to the results in ref. 40.

The dielectric properties of these materials have important significance in obtaining a uniform repartition of the electromagnetic field in the materials to be treated. In our case, the dielectric properties (Figure 4) of these materials explained the idea of the solution used for the compensation of microwave-source heating attenuation. The role of silicon glass (mold of the part) was to obtain a concentration effect of the waves in the median plan of the applicator, where the part was placed and situated [see Figures 1(a) and 2(a)] and conserved mode TE01. However, the PE played a support role in maintaining the mold (material 2) and the part (material 3) in the median plane of the applicator (material 1 had a characteristic of absorption ε'' of about 0.001).

Experimental Equipment

The part of the epoxy–glass or the treated material was prepared by the stacking of the strips cut from the roller of the preim-pregnated epoxy–glass. After we obtained our wished thickness of the part to be treated, the part was put into the mold and the waveguide according to Figure 5. After this step, the probes were inputted to center plan, where the part was situated and where, according to electromagnetic and thermal modeling, the maximum of the electric field and the temperature were located. The part was installed within the mold in the waveguide for the treatment for 1800 s with a 915-MHz frequency and a power of 1200 W.

In this study, we were interested on the compensation of microwave-source heating attenuation into the epoxy–glass composite. So, the temperature was an important parameter of the process and depended the kinetic aspects related to the matrix transformation. Therefore, it was thus necessary to take a precise measurement of the temperature that would require precautions on the material of measurement to be used. Most of the temperature sensors existing were connected electrically to the system processing the data. These electric connections played the role of antennas. However, in general, the use of these techniques in microwave processes is thus impossible because of the problems of electromagnetic compatibility. The heating of the thermocouple itself can considerably distort the measures.[48] However, in special cases, a particular orientation of the probes compared to the direction of the electric field can limit the interferences but cannot to remove them. According to results,[49] these harmful effects are moderated in the case of low power or in the case of strongly dissipative materials.

On the other hand, there are compatible measurement techniques in electromagnetism; they are used to eliminate the previous measure problems. The conducting connections in this case are replaced by optical fibers, which do not interfere with electromagnetic fields. The thermometer used for the experiments in this study was a fluoroptic thermometer from Nortech. It had four connections to receive four simultaneous temperature measurements. Then, it was possible to simultaneously follow the change of the temperature in four different points of the object to be treated. Moreover, it had a screen of posting, which allowed a follow-up of the evolution of these temperatures.

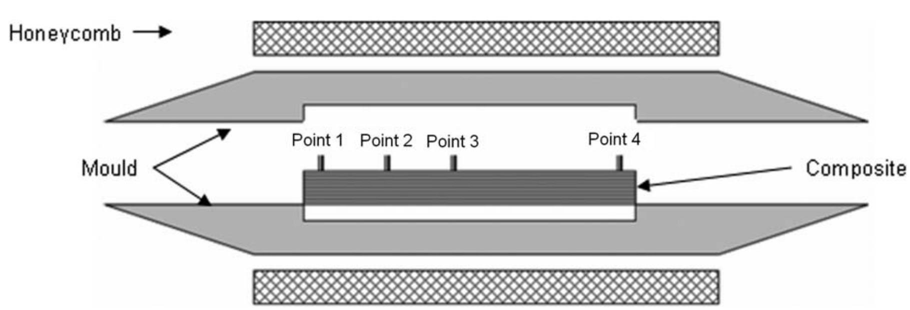

Figure 5. Mold and the composite to be treated before assembling.

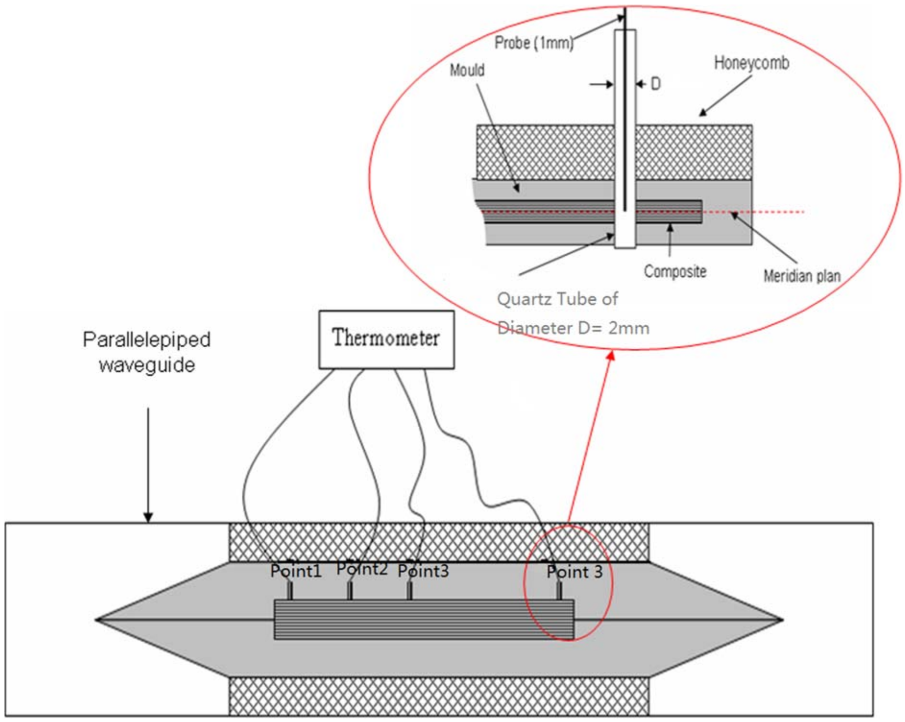

Figure 6. Mold and the composite to be treated after assembling. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The probes had an optical fiber that did not interfere with the electric field.[50] Each optical fiber was finished by a detector in fluorogermanate of magnesium with dimensions on the order of a millimeter. When this detector was excited by UV light, it emitted a light wave of fluorescence whose intensity was a function of temperature.[51–53] The signal was then treated with photodiodes; this made it possible to determine the temperature with a precision of 0.1°. To prevent disturbances from the light signals of excitation and emission by ambient light, the glass fiber that conveyed these signals was covered with pigment in Teflon. The pigment was also used to protect the fiber from this environment.

The probes were introduced to the four points of the composite to the median plane, as shown in Figure 6. The preliminary precautions and measures conducted for the probe heads are on this plan. The role of the tubes was to protect the probes during and after the matrix crosslinking reaction.

Many series of thermal measurements were carried out on some 10 and 15 mm thick parts in four points with a power of 1200 W and a frequency of 915 MHz by probes that did not interfere electrically and functioned on the principle described previously. The four points chosen started from the interface of entry of the part at $Z_1 = 2$ cm, $Z_2 = 20$ cm, $Z_3 = 38$ cm, and $Z_4 = 74$ cm. (Z_1, Z_2, Z_3 and Z_4 are the coordinates of the four measurements points). The dimensions of the molds and the composite to be treated are summered in Table II.

RESULTS AND DISCUSSION

Results of Thermal Measurements without DEAI

The first results were obtained on a part with a 10 mm thickness and a mold with 7x the parts thickness. The curves of Figure 7 represent the evolution of the temperature according to the time obtained by the thermal measurements of the treated material without DEAI (the case of uniform thickness). The general shape of the temperature curves according the time, obtained during the crosslinking of the epoxy resin DGEBA, was observed also by Gourdenne and Le Van.[10] According to the time, we noticed that the curves of Figure 7 showed an evolution in four phases. In phase 1, the temperature changed almost linearly. This was for a period corresponding to a temperature rise due to heating by microwaves to about 120°C. This phase began at the same time throughout the entire part, but it did not end up at the same time for all points of the part. Phase 2 began with another growth with a steeper slope than in phase 1, and it ended with a maximum or a plateau. This growing phase was the sign of crosslinking. In Figure 7, the points of change in the slope between phases 1 and 2

Table II. Dimensions of the Molds and the Treated Materials

Thickness of Material 1 (mm)	Thickness of Material 2 (mold) without DEAI (mm)	Dimensions of Material 2 (first mold) with DEAI (mm)	Dimensions of Material 2 (second mold) with DEAI (mm)
E=10	70	T_{ent}=190 T_{exi}=19.95	T_{ent}=170 T_{exi}=19.95
E=15	105	T_{ent}=126.66 T_{exi}=13.3	T_{ent} =113.3 T_{exi} = 13.3

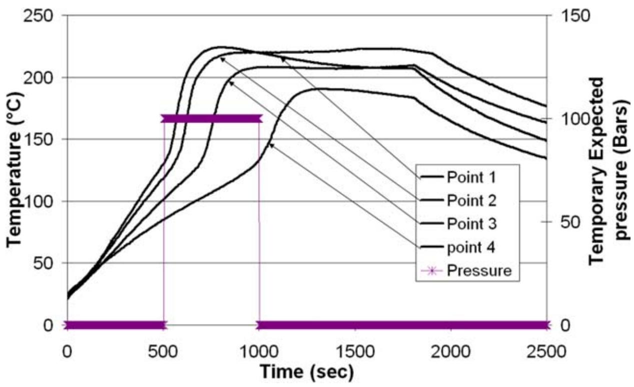

Figure 7. Temperature according the time in different points of the epoxy–glass material without DEAI. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

occurred for the same temperature but at different times for each measurement point. They corresponded to the beginning of the crosslinking reaction. Because of the electromagnetic wave source attenuation along *Z*, the crosslinking reaction started from the input interface; then, the crosslinking reaction followed the other points. Phase 3 began after the end of the crosslinking reaction. This change in the temperature was caused by heating due to only microwave energy and cooling by heat exchange with the environment. We noted that in this phase, the temperature variations versus time at the four points were not the same; this was also due to heat transfer within the part and exchange between the part and mold and their environment. Phase 4 was the last phase and started after the third phase. They corresponded to the calling phase after the end of heating by microwave energy.

First Results of Thermal Measurement with DEAI

We relied on the results of electromagnetic modeling (Figure 8) to design our mold, represented in Figure 9, and to test the compensation of the electromagnetic heat source attenuation.

Figure 8 represents the electrical field repartition into the waveguide filled by three dielectric materials. However, to obtain these results, we solved the equation of electromagnetic propagation into this waveguide filled by three dielectric materials, where one of these materials represented the materials to be treated. The modeling was realized by FEMLAB software, version 3.2. More information about the modeling can be found in the results in refs. 34 and 36.

The results were obtained on a part with a thickness of 10 mm and with the dimensions of the mold shown in Table II. The first results of the thermal measurements with the dielectric effect inversion of the electromagnetic phenomenon are shown in Figure 10.

We noted from the curves of Figure 10 that in the temperature evolution according to time, there were always four phases, as in the previous case without EDAI (Figure 7). The

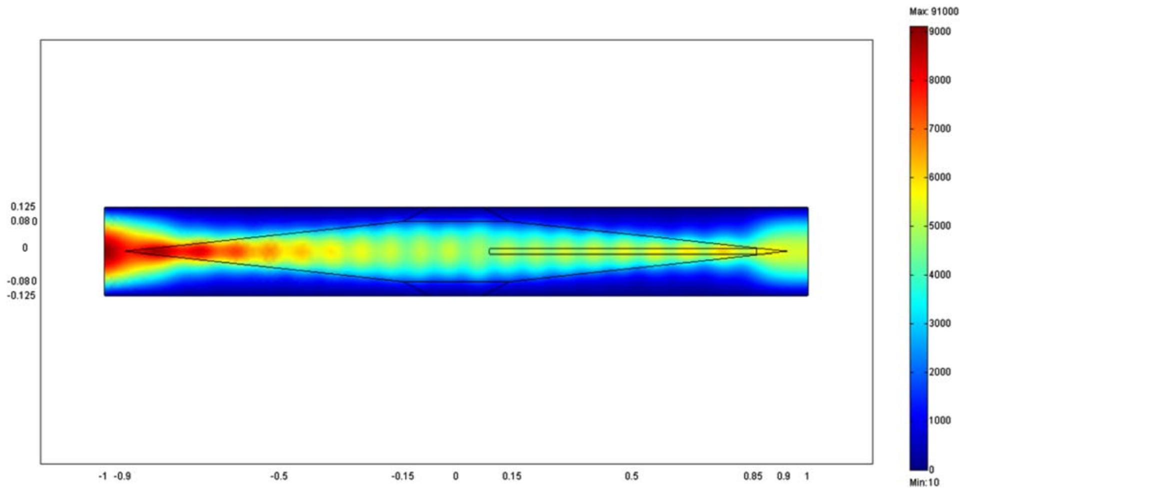

Figure 8. Electromagnetic modeling results of the electrical field repartition according the length of the treated composite. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

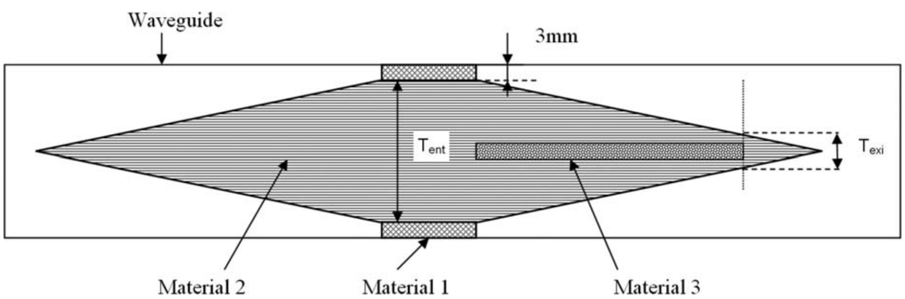

Figure 9. Geometry and dimensions of the experimental mold of the first experience with DEAI.

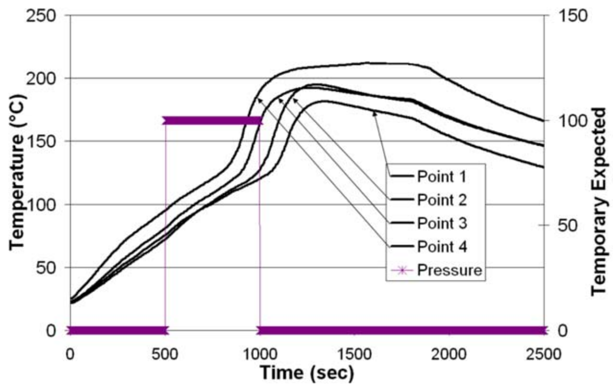

Figure 10. Temperature according the time in different points of the epoxy–glass material: first result. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

interpretation and details of these phases were the same as in the previous case.

However, in contrast to the previous case, we noticed that this time, point 4 had the highest temperature, although the left-side switch was exposed to the greatest intensity of electromagnetic power conveyed by the wave. This was because the attenuation of heat sources of electromagnetic origin was inversed by the use of the dielectric effect phenomenon of inversion of the attenuation.

Second Results of Thermal Measurement with DEIA

The results were obtained on a part 10 mm thick and are presented in Figure 11. As shown in Figure 11, as in the previous two cases, the temperature change versus time exhibited all four phases. The interpretations and analyses of the four phases were similar to those of the first two cases. In contrast to the previous cases (Figures 7 and 10), we observed that in phase 1, the first three points (1, 2, and 3) had the same temperature profile. However, when the chemical reaction began around 120°C, the temperature profiles for these three points separated but remained relatively close. These temperature profiles showed that there was a compensation of the heat electromagnetic source attenuation. Indeed, the strong rise in temperature at

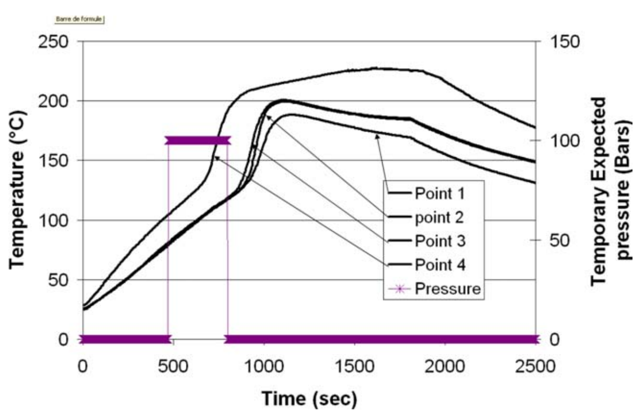

Figure 11. Temperature according the time in different points of the epoxy–glass material: second result with DEAI. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

point 4 depended also on the heat capacity of the mold at this point, which was lower than that along the part. The portion of the part that was located in the vicinity of the output interface of the wave was surrounded by a layer of silicone glass only equal to 1.325 times the thickness of the part.

According to the Figures 7, 10, and 11, because of the DEAI effect, the crosslinking reaction of the epoxy–glass composite did not start at the same time. For example, in Figure 7, the crosslinking reaction began at about 600 s at the first point. However, in Figures 10 and 11, at the same point, we noticed that the crosslinking reaction started at about 1000 s. We explained and attributed this observation to the repartition of microwave energy.

The uniform repartition of microwave energy, in the cases of Figure 11, along the epoxy–glass part, helped to start the crosslinking reaction at the same time as the that of the composite to be treated. However, in the case of Figure 7, the nonuniform repartition of microwave energy along the composite started the crosslinking reaction at nonsimultaneous times with the composite, and the reaction started by point 1. The case of Figure 10 was the opposite as the case of Figure 7 because the crosslinking reaction started by point 4.

A uniform distribution of microwave energy into the epoxy–glass part led to a uniform distribution crosslinking reaction of the matrix along the part, and the crosslinking of the matrix started simultaneously at all points. In other way, the DEAI had an effect on the microwave energy repartition, and the energy repartition had an effect on the starting time of the crosslinking reaction of the matrix along the part. In addition, the marks of the temperature on the parts realized by different molds represented in Figure 12 are the proof of attenuation inversion of microwave energy into the treated material.

Attenuation

The temperature measurements that we performed allowed us to compare the new and different electromagnetic behaviors of the three molds that we used. The electromagnetic behavior could be accessed by the intensity of the heat sources of electromagnetic origin. The intensity of these heat sources could be measured from the derivative with the time of the temperature at any point of the specimen and at the initial instant embodiment of the dielectric hysteresis phenomenon, whereas the temperature moment of the specimen and the temperature of its surroundings were equal and for which chemical reactions had not yet started.

The heat equation applied to a sample heated by microwaves or by any internal heat source was expressed by the following equation:

$$\rho c_p \times \frac{dT}{dt} = div(\text{Flow of heat transfer by conduction}) + (\text{Heat source}) \text{ with } dT/dt \text{ is variation of the temperature with the time} \quad (1)$$

At the initial moment of heating of the specimen by heat sources, the change from the time of the temperature corresponding to this heating condition was independent of the heat transfer

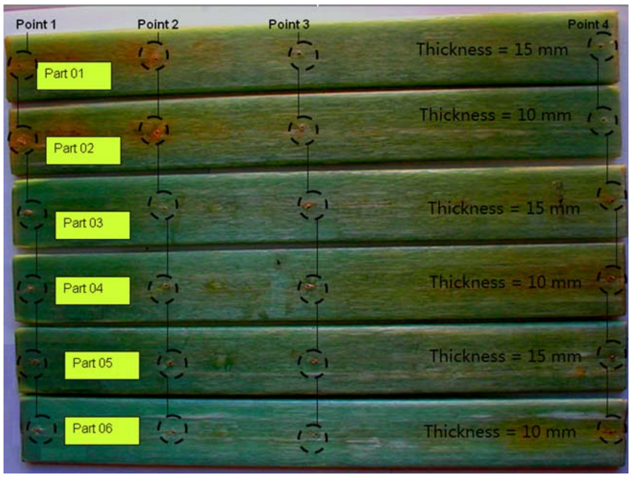

Figure 12. Realized parts with different molds: (parts 01 and 02) produced without DAEI, (parts 03 and 04) produced with DAEI (first mold), and (parts 05 and 06) produced with DAEI (second mold). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

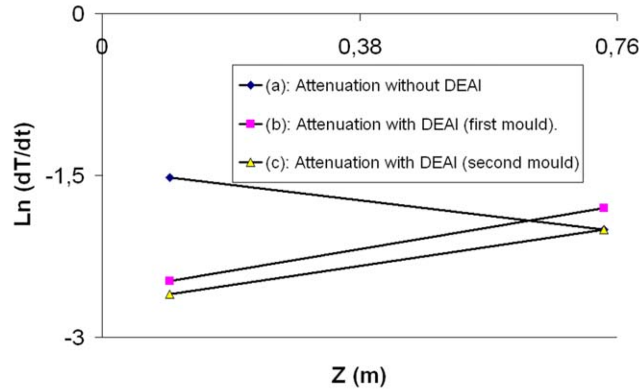

Figure 13. Results of attenuation with different molds. (a): attenuation without DEAI, (b) attenuation with DEAI (first mold), and (c) attenuation with DEAI (second mold). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

between the part and its environment and was directly dependent on the intensity of the heat sources. When we observed that this variation with respect to time was constant for a given time interval, we could neglect the losses by heat transfer between the part and its environment and could considered it the beginning of heating, and the heat equation was reduced:

$$\rho c_p \times \frac{dT}{dt} = \text{Heat source} \quad (2)$$

In eq. (2), the intensity of the heat source is a function that depends on the space variable (z) and the attenuation of the source (α; m^{-1}) of the electromagnetic field at the initial time of the heating. Thus, we obtained the following equation:[54]

$$\rho c_p \times \frac{dT}{dt} = q_0 e^{-2\alpha z} \quad (3)$$

where q_0 is initial source (1200 W) and z is the longitudinal dimension. We therefore expressed the Neperian logarithm of the temperature evolution versus time according to z:

$$\text{Ln}\left(\frac{dT}{dt}\right) = (-2\alpha)z + \text{Ln}\left(q_0/\rho c_p\right) \quad (4)$$

where ρ is the density of the composite (1900 kg/m^3) and c_p is the specific heat of the composite (1500 J kg^{-1} K^{-1}).

By plotting the curve Ln(dT/dt) function of z, we obtained a straight line of the form:

$$Y = -Az - B \quad (5)$$

A and B are constantes. By comparing the two eqs. (4) and (5), we obtained the following equalities $2\alpha = -A$ and $B = \text{Ln}(q_0/\rho c_p)$, as shown in the curves of Figure 13 and the summary in Table III.

Calculations of the attenuation of the heat source of the electromagnetic origin between measurement points 1 and 2 in the case of a uniform mold [without DEAI; Figure 13(a)] give an attenuation equal to 0.3201 m^{-1}.

Calculations of the electromagnetic heat source attenuation between measurement points 1 and 4 in the case of a nonuniform mold [Figure 13(b)] gave a negative apparent attenuation equal to −0.36601 m^{-1}, which justified the term of attenuation inversion. Calculations of the electromagnetic heat source attenuation between measurement points 1 and 2 in the case of a nonuniform mold [Figure 13(c)] gives an apparent negative attenuation equal to −0.384951 m^{-1}; this always justified the term of attenuation inversion.

The results of the attenuation calculations are summarized in Table III.

CONCLUSIONS

First, in this study, we confirmed the electromagnetic heat source attenuation in the case of a mold of uniform thickness. The results of this study also show the compensation of the source attenuation with a nonuniform thickness mold. However, we saw in the first case of the mold of nonuniform thickness a

Table III. Results of Attenuation

	Without DEIA	With DEAI (first mold)	With DEAI (second mold)
Equation of the curve	Figure 13(a): $Y = -0.64020.Z\text{-}1.805$ $A = -0.64020$	Figure 13(b): $Y = +0.73202.Z\text{-}2.4490$ $A = 0.73202$	Figure 13(c): $Y = +0.77002.Z\text{-}2.72031$ $A = -0.77002$
α (m^{-1})[a]	0.3201	−0.36601	−0.38501

[a] $\alpha = -A/2$.

strong temperature gradient along the composite in the opposite direction of wave propagation. Also, in the second experiment with the mold of nonuniform thickness, we proved, with a suitable choice of mold dimensions, that we could obtain a low heat-source attenuation in the direction of propagation and a uniform temperature accepted at point 4 at the end of the part. This fourth rise in temperature was due to the heat capacity of the mold, which was not constant along the specimen (part).

All of the experiments were carried out on a parallelepiped and the long part to prove the compensation and control of attenuation. This problem represents a major problem in microwave processing. Our results are considered a solution toward a uniform treatment by microwave energy. In the process case, to obtain a better quality composite product, we had to apply a temporary expected pressure to eliminate air bubbles (see Figures 7, 10, and 11). The moment of application of this temporary pressure was before the beginning of matrix crosslinking when the viscosity of the matrix was weak. By the way, as we mentioned in the Introduction, the results of Bai[15] showed that the reticulation of pure epoxy resin (DGEBA–3DCM) with microwaves as an energy source improved its mechanical properties compared to those obtained by a conventional process. According to the results,[16,17] with a microwave process, we obtained a reduced treatment compared to the conventional processes. Other authors[18] concluded that the use of microwaves in pulsed mode did not influence the physicochemical structure of the material obtained. However, in the composites cases, the applications of these results are limited in the parallelepiped and long part or curved part. We are currently working to generalize these results on rubber vulcanization and the complex geometry of composites.

ACKNOWLEDGMENTS

The authors thank the Commission of Science and Technology of Chongqing (contract grant number Cstc2012ggB50002), the French National Centre for Scientific Research, and Chongqing Academy of Science and Technology for their financial support.

REFERENCES

1. Metaxas, A. C.; Meredith, R. J. Industrial Microwave Heating; Peter Peregrinus, London, United Kingdom, **1983**.
2. Ollivon, M.; Quinquenet, S.; Seras, M.; Delmotte, M.; Moré, C. *Thermochim. Acta* **1988**, *125*, 141.
3. Morey, W. *U.S. Pat.* 3,244,571 (**1963**).
4. Kawasa, K.; Hayakawa, K. Jpn. Pat. 74.42522 (**1974**).
5. Ono, K.; Kitamura, M.; Shigeta, T.; Nishnura, T.; Shikuma, H. Jpn. Pat. 75.20984 (**1975**).
6. Wilson, L. K.; Salerno, J. P. Microwave Curing of Epoxy Resins; Vanderbilt University: Nashville, TN, **1978**.
7. Loos, A. C.; Springer, G. S. Curing of Graphite/Epoxy Composites; University of Michigan: Ann Arbor, MI; **1983**.
8. Springer, G. S. *J. Compos. Mater.* **1982**, *4*, 357.
9. Gourdenne, A.; Maasarini, A. M.; Monchaux, P.; Aussudre, S.; Thourel, L. *Am. Chem. Soc. Div. Polym. Chem. Polym. Prepr.* **1979**, *20*, 471.
10. Gourdenne, A.; Le Van, Q. *Am. Chem. Soc. Div. Polym. Chem. Polym. Prepr.* **1981**, *22*, 125.
11. Teffal, M.; Gourdenne, A. *Eur. Polym. J.* **1983**, *19*, 543.
12. Pinquie, L. Ph.D. Thesis, Institut National Polytechnique, **1988**.
13. Jullien, H.; Delmotte, M.; Thuillier, F. M.; Valot, H.; Boulonnais, D.; Sailleau, J.; Priou, A.; Aussudre, S. Final Contract Rapport DRET, N85002, Doc. ONERA, 3/1324 MO; **1986**.
14. Aussudre, S.; Priou, A.; Jullien, H.; Delmotte, M.; Thuillier, F. M.; Boulonnais, D.; Sailleau, *J. Recherche Aérospatiale* **1988**, *5*, 1.
15. Bai, S. L. Ph.D. Thesis, Ecole Centrale de Paris, **1993**.
16. Jullien, H.; Valot, H. *Polymer* **1983**, *24*, 810.
17. Jullien, H.; Vallot, H. *Polymer* **1985**, *26*, 506.
18. Beldjoudi, N.; Bouazizi, A.; Douibi, D.; Gourdenne, A. *Eur. Polym. J.* **1988**, *24*, 49.
19. Gay, D. Matériaux Composites; Hermès: Paris, **1989**.
20. Cours d'Électrothermie. Vol.3. Traitements thermiques par micro-ondes et radiofréquences, Publisher: **Centre français de l'électricité**, Paris 2001.
21. Lee, W. I.; Springer, G. S. *J. Compos. Mater.* **1984**, *18*, 387.
22. Williams, N. H. *J. Microwave Power* **1967**, *2*, 123.
23. Gourdenne, A.; Monchaux, P.; Le Van, Q.; Aussudre, S.; Thourel, L.; Audo, N.; Nedelec, J. Presented at the Third International Congress of Composite Materials; 1980.
24. Jean, A. O.; Roussy, G. Fr. Pat. 2,316,761 (**1977**).
25. Becheret, C. Eur. Pat. 0,347,299 (**1989**).
26. Kim, W. U.S. Pat. 4,777,707 (**1984**).
27. Yamamoto, K. Jpn. Pat. 600135230 (**1985**).
28. Hord, W. E. Ph.D. Thesis, University of Missouri, **1966**.
29. Moreno, T. Microwave Transmission Design Data; Artech House: Boston, **1989**.
30. Outifa, L. Ph.D. Thesis, University of Paris *VI*, **1992**.
31. Jao Jules, E. Ph.D. Thesis, École Nationale Supérieure d'Arts et Métiers, **2001**.
32. Hug, G. Ph.D. Thesis, École Nationale Supérieure d'Arts et Métiers, **2005**.
33. Douadji, L.; Delmotte, M. Presented at JEPO33, Saint-Etienne, France, Sept 18–23, **2005**.
34. Douadji, L.; Delmotte, M. Presented at J.S.F.A., Ouargla, Algeria, Dec 11–14, **2004**.
35. Douadji, L. *Edition Universitaire Européenne*, June **2012**.
36. Douadji, L.; Delmotte, M. *Presented at Congrĕs Internationale sur la Physique et ses Applications* **2007**; Oran, Algeria, Dec **2007**.
37. Douadji, L.; Delmotte, M. *J. Microwave Power Electromagn. Energy* **2012**, *46*, 176.

38. Douadji, L.; Delmotte, M. Modélisation de la Température Obtenue par l'Effet Diélectrique d'Inversion de l'Atténuation des Ondes Électromagnétiques pour le Traitement Thermique Uniforme des Matériaux Composites; Société France de Thermique: *Île de Ré*, France, **2006**; p 799.
39. Douadji, L.; Delmotte, M. *Chin. Pat.* ZL 2 034924.2 (**2012**).
40. Douadji, L. Ph.D. Thesis, École Nationale Supérieure d'Arts et Métiers, **2007**.
41. Jao Jules, E.; Delmotte, M.; Moré, C. *Réc. Prog. Génie Proc.* **2001**, *15*, 169.
42. Delmotte, M.; Outifa, L.; More, C. Fr. Pat. 93/10710 (**1993**).
43. Delmotte, M.; Outifa, L.; More, C.; Delaunay, D. Fr. Pat. 407008 (**1994**).
44. Thostenson, E. T.; Chou, T. W. *Compos. A* **1999**, *30*, 1055.
45. Debye, P. Polar Molecules; Dover: New York, **1945**.
46. Zong, L.; Kempel, C.; Hawley, C. M. *Polymer* **2005**, *46*, 2638.
47. Cole, K. S.; Cole, R. H. *J. Chem. Phys.* **1941**, *9*, 341.
48. Bowman, R. R. *IEEE Trans. MTT* **1976**, *24*, 43.
49. Larsen, L.; Moore, R. A.; Jacobi, J. H.; Halgas, F. A.; Brown, P. V. *IEEE Trans. Microwave Theory Tech.* **1979**, *27*, 673.
50. Rozzell, T. C.; Johnson, C. C.; Durney, C. H.; Lords, J. L.; Olson, R. G. *J. Microwave Power* **1994**, *9*, 241.
51. Wichersheim, K. X.; Leffèvre, H. C. Presented at Photonetics, Inc., Nov **1994**; p 6.
52. Urbach, F. *U.S. Pat.* 2,551,650 (**1951**).
53. Roussy, G.; Oberlin, C.; Rochas, J. F. "Chauffage diélectrique", Principes et spécificités, Techniques de l'ingénieur, D, vol. 5940, 2003, p. 1–11.
54. Sacadura, J. F. Initiation aux Transferts Thermiques—Technique et Documentation; Lavoisier: Paris, **1982**; p 446.