



Comparison by thermal analysis of Joule-cured versus oven-cured composites

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Abstract

The current technology for curing high-performance composites, such as those used in industries like such as aeronautics and the automotive industry, is based on the use of autoclaves, where the material is cured by external heating, in large ovens. This type of curing requires enormous amounts of energy, of which only a small part is invested in the actual curing of the material, and the rest is mainly used for heating and maintaining the temperature of the autoclave. An alternative method that entails a lower energy cost compared to the traditional methodology is curing through the Joule effect, in which an electric current is passed through the material, so that it acquires temperature from the inside due to the passage of current through the carbon fibres, triggering and accelerating the curing process of the composite. While Joule curing may provide a much more efficient and faster curing, a control technology is needed to ensure that temperatures all throughout the composite match the temperature programme. In this work, a procedure has been developed to control the Joule effect curing of carbon fibre/epoxy composites in order to compare, by means of differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA), the curing obtained by this method with that obtained by the traditional oven curing method.

Keywords Carbon fibre · Composites · Joule curing · DSC · DMA

Introduction

Fibre-reinforced materials, including those composed of an epoxy resin matrix and carbon reinforcements—either nanotubes or continuous fibres—have been used in various applications that require both low density and good mechanical properties. These applications include, but are not limited to, the energy (for wind turbine manufacturing), aerospace, military and automotive sectors. Over the years, this type of composite materials has recorded annual growth rates of approximately 13% [1], which is linked to an increase in the demand for these materials. Among the main reasons for this growth are the growing concern for the environment, as well as the desire to significantly reduce fuel consumption.

The success of this type of materials is largely due to the excellent balance between their mechanical properties,

such as high strength and stiffness, and their low density. They have low densities and specific mechanical properties superior to those of other materials commonly used in the same sectors. Carbon fibre (CF) composites also provide additional advantages such as corrosion resistance, oxidation resistance and reduced effective cost compared to metals. In addition to all these advantages, their electrical and thermal properties are also interesting as they allow them to function as heating elements through the conversion of electrical into thermal energy [1–4].

Most parts and products made of carbon fibre-reinforced materials (CFRM) are obtained from prepregs in which the matrix is a thermosetting resin. The term prepreg is a shortened form of preimpregnated fibres and refers to a thin sheet of fibre polymer composite. These laminates are manufactured by impregnating reinforcing fibres or textiles with custom formulated, pre-catalysed resin systems, establishing close contact between the fibre and the matrix. The use of prepregs allows the fibre content to be controlled more efficiently than with other manual techniques. In these prepregs, the resin is in an intermediate stage of curing. This partial degree of curing provides a set of viscoelastic properties that

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make the prepregs very convenient for transport, storage and conformation of the desired parts [1, 2, 5, 6].

The curing process during the manufacture of parts using this type of material is a defining point in terms of manufacturing time, property performance and total cost. This is usually carried out using autoclaves or ovens. The autoclave process is used primarily for small number batches, up to 100, of high-performance composite structures. This method provides very good mechanical properties to the part, thanks to the high temperature and pressure curing conditions inside the vessel. However, in spite of the good results, this technique has a few but relevant problems. First, initial equipment investment is extremely costly, which contributes to the high production costs of components cured by this technique. Besides, this process is inefficient from the energetic point of view, since it is necessary to heat the whole autoclave with the piece to be cured inside, so that a significant percentage of the energy is not used for curing but is consumed in the heating of the autoclave. In addition, the parts have to remain in the oven for long periods of time to achieve uniform temperature and complete curing. Since heat inside the vessel is transferred mainly by convection, temperature gradients inevitably appear along the thickness of the part, and dwell times are required to reach the same temperature throughout the part. Such dwell time results in an additional increase in the cost of the curing process. In addition, the size of the parts to be cured is limited by the dimensions of the autoclave chamber [6–9].

In view of all these limitations, it would be very interesting to find another approach to manufacturing processes that would allow us to dispense with equipment such as autoclaves or ovens for curing advanced composites. The ideal characteristics of the proposed methods should avoid convective heating and would produce heating from the inside of the piece. They must also reduce the accumulation of stresses inside the composite and avoid uncontrollable exothermic reactions. Some of the new proposed technologies are based on heating through the Joule effect using electric current in different ways. This method, in contrast to oven curing, allows the power supplied to the part to be cut instantaneously, which leads to better control over the curing process, in addition to protecting against overheating caused by unwanted exothermic reactions during the curing process, and facilitating predictive control over the process. In addition to all of the above, one of the most significant benefits of these new curing methods is, due to the direct control of the power applied to the part, the low energy consumption compared to traditional methods. This is because with these methods all the energy supplied to the curing system is used only in heating the part itself that is being cured, without the need to raise the temperature of the surrounding environment. This would also significantly reduce costs, which are a limiting factor in any manufacturing process, and brings

the advantage of improving the sustainability of existing processes [10, 11].

New technologies developed for curing composites by electrical curing include the work of Bayerl et al. on electromagnetic induction curing [12], the microwave curing of Joshi et al. [13], and the work of Maguire et al. on vacuum bag curing [14]. This study focuses on another type of curing also based on electrical curing: Joule curing. This type of curing can be found as well in the literature as self-resistive curing (SRC), or direct electrical curing [8]. This technology is based on taking advantage of the low electrical resistivity of the carbon fibre to use it as a heating element for the composites in which it is present by means of the Joule effect. Some advantages of using this novel curing technology are the uniform temperature distribution across the part, the fast heating and cooling rates, the high energy efficiency achieved and the simplicity of the curing equipment compared to oven-based heating technologies. Importantly, curing time and energy consumption can be significantly reduced compared to oven processes, with Joule curing being faster and more energy efficient [9].

The work presented by Fukuda et al. was one of the first studies using this curing technique, providing insight into the limitations of the method, such as the large amount of current required to achieve consistent values in the mechanical properties of the composites [15]. That work constitutes a basis for the development of the technique and justifies that, overcoming the limitations, it is applicable to the curing of carbon-reinforced composites. Hayes et al. [7], taking the previous work as a reference, among others, tested different configurations for the contacts and managed to achieve higher degrees of curing than even those obtained by oven, confirming the results obtained previously. Fosbury et al. studied this technique using different interlayer contact configurations, such as perpendicular directions of the fibres, in order to study the current conduction through the different layers of the samples in order to use the composite as a heating element [11]. Mas et al. proposed using this technique for the repair of parts and welding, rather than for the fabrication of parts itself, incorporating carbon nanotubes as a reinforcement [16].

In the present study, a system developed ad hoc for the curing of epoxy resin and carbon fibre prepregs is used in order to compare by means of thermal analysis techniques the degree of curing of samples cured by the developed system with that of samples cured by a traditional oven method. Thus, the comparison of typical parameter values, such as those of T_g or E' obtained from samples cured by the Joule effect, with those obtained by conventional oven heating will easily demonstrate whether or not it is possible to obtain results comparable to those obtained by oven curing, despite the fact that the former method uses a much shorter time. In addition, using DMA data, attention will be paid to the shape

of the relaxation peaks corresponding to the glass transition and their possible significance in terms of the degree of cure obtained by each of the methods. A thorough understanding of how different qualities can be achieved with each method will allow a choice to be made that is appropriate to the requirements of the various applications that may arise.

Experimental

Materials and equipment

To carry out the study, laminates were manually prepared from three layers of unidirectional carbon-epoxy prepreg MTC 510 UD 300 supplied by Castro Composites (Spain). The layers were arranged in the 90-0-90 directions. The laminates were cured by the two proposed methods, here referred to as oven and Joule. The prepreg has an epoxy resin content of 33% by mass and a thickness of approximately 0.2 mm. The epoxy resin of the prepreg is based on diglycidyl ether of bisphenol A (DGEBA). Carbon/epoxy prepreps are generally used for the manufacture of composites. As they contain carbon fibres, these prepreps are suitable for the study of Joule curing. The recommended curing cycle for this material in the oven is used as a reference for this study. It consists of an initial temperature ramp at no more than $3\text{ }^{\circ}\text{C min}^{-1}$, from room temperature to $100\text{ }^{\circ}\text{C}$, followed by an isothermal stage at $100\text{ }^{\circ}\text{C}$ for 4 h. Then, the material is allowed to cool by itself at room temperature. If high T_g values were sought, the manufacturer also recommends a post-cure stage at $120\text{ }^{\circ}\text{C}$ for 1 h, reaching that temperature following the same temperature ramp considerations as before—in this work, that post-cure stage was not performed, as it was wanted to observe the curing process for standard T_g values.

To prepare the samples, plies about 250 mm long by 50 mm wide were cut. The top and bottom layers are placed in the 90° direction and are about 20 mm shorter than the central layer, while the middle layer is in the 0° direction. The plies are placed in a centred position so that about 10 mm of the middle ply is exposed towards the ends to allow electrical connections to be made, as this is the ply that will be used to produce Joule heating. The laminates are connected through the free ends of the central layer to two electrodes, each of them formed by two steel plates of $80 \times 35\text{ mm}$ and $60 \times 25\text{ mm}$, joined by means of screws that allow tightening to ensure the electrical connection. The connection of these electrodes with the power supply and the rest of the elements involved was made by means of copper cables, whose ends were adapted to facilitate the connections both with the power supply and the laminate sample, as well as with the rest of the elements.

The sample was connected to a 150 V, 40 A maximum power supply through the electrodes consisting of steel plates described above. The power supplied by this source is controlled by an Arduino Leonardo board, which is connected both to the source and to the computer running the control program, which provides the power source with instructions to tune the power delivered to the sample. This circuit is closed by a solid state relay (SSR, Relequick, Spain) located between the sample and the source and which is controlled by a control card for this type of device (Tinkerforge, Germany), which, in turn, is connected to the computer. From a comparison of the sample surface temperature with the programmed temperature, the control card will send the SSR the instruction to open or close the electric circuit to adjust the temperature accordingly. A schematic of the general configuration is shown in Fig. 1.

In addition to the above items, a Master Brick control board (Tinkerforge, Germany), which drives the control computer and bundles the relay board connections, and a thermal imaging Tinkerforge Bricklet incorporating a Flir LWIR Lepton micro-thermal camera module have also been included in the system.

While the described setup is used to obtain the samples by Joule curing, to obtain samples cured by the traditional method a Vacuitem-TV oven (P SELECTA, Spain) is used, which allows a heating rate of $3\text{ }^{\circ}\text{C min}^{-1}$ and an accuracy of $\pm 2\text{ }^{\circ}\text{C}$.

The equipment used for thermal characterization of the samples by differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) are, respectively, a Q2000 and a DMA 2980, both from TA Instruments.

Curing programmes

A computer programme was coded in Python to control the temperature in the Joule curing system. This programme runs on a computer and interacts with the Arduino board and the Tinkerforge bricklets. Its functions include retrieving data on the temperature of the sample and the voltage applied during curing, setting the target temperature at

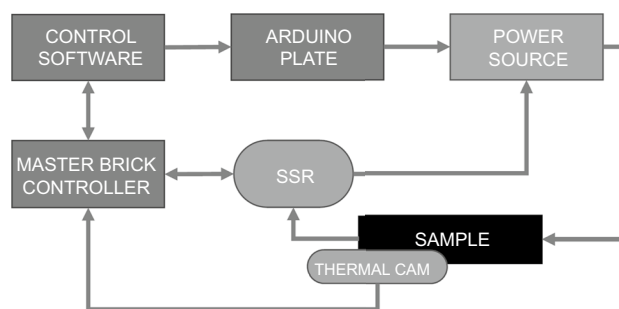


Fig. 1 Scheme of the developed system for Joule curing

all times, and controlling through the actuators the power applied to the prepreg at each instant.

The thermal imaging Bricklet module, in charge of providing the thermal image, contains a thermal microcamera and interacts with the BrickViewer control software, which allows to delimit the area of the thermal image that best suits the experimental needs, and to obtain the results of the maximum, average and minimum temperature for that area. In this case, the central area of the sample was chosen. An scheme of this is shown in Fig. 2.

In the case of oven-cured parts, taking the manufacturer's recommendations as a reference, a linear heating at $2\text{ }^{\circ}\text{C min}^{-1}$ from room temperature to $100\text{ }^{\circ}\text{C}$ is used, followed by an isotherm with duration in some cases of 3 h and in others of 4 h in order to evaluate the effect of the isotherm time.

In the case of parts cured by the Joule effect, different combinations of temperature, isotherm time and heating rate are used in order to identify possible interactions between these parameters. Table 1 summarizes the curing conditions used for each of the samples. Once the samples were oven and Joule cured, they were kept individually bagged at room temperature (between 15 and $20\text{ }^{\circ}\text{C}$) until the specimens were extracted for the DSC and DMA tests. As they were tested on the same day as the curing, it was not necessary to store them in the freezer.

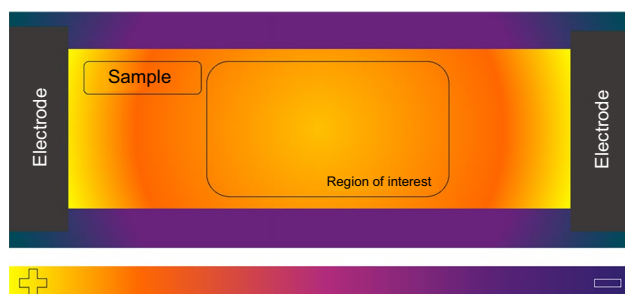


Fig. 2 Scheme of temperature distribution through the sample during Joule curing

Table 1 Curing conditions for each of the manufactured samples

Sample	Curing method	Heating rate/ $^{\circ}\text{C min}^{-1}$	Isothermal temperature/ $^{\circ}\text{C}$	Dwell time at isothermal/min
Oven A	Oven	2	100	60
Oven B	Oven	2	100	60
Joule 1	Joule curing	2	100	60
Joule 2	Joule curing	2	100	60
Joule 3	Joule curing	10	160	3
Joule 4	Joule curing	10	160	9
Joule 5	Joule curing	10	170	6 (2 cycles)

DSC and DMA testing

The parts cured by the methods described above are analysed by DSC and DMA. For the DSC tests, samples of about 15 mg were taken from the central part of the laminates. The tests consisted of a linear heating at $10\text{ }^{\circ}\text{C min}^{-1}$ from 30 to $180\text{ }^{\circ}\text{C}$. A double cantilever geometry with a free distance of 17.5 mm between clamps, on both sides of the central clamp, was used for the DMA tests. The samples consisted of strips about 15 mm wide and 1.3 mm thick that were taken from the central part of the laminate. A heating ramp of $2\text{ }^{\circ}\text{C min}^{-1}$ up to $180\text{ }^{\circ}\text{C}$ was applied, maintaining an oscillation of 1 Hz with a strain amplitude of $250\text{ }\mu\text{m}$. The values of G' , G'' and $\tan(\delta)$ were recorded.

Results and discussion

Figure 3 shows that the samples subjected to Joule curing followed the temperature programme very closely. Figure 4 shows the heat flow curves obtained from samples subjected to identical temperature programmes, two of them in furnace and the other two by Joule heating. The heating ramp was $2\text{ }^{\circ}\text{C min}^{-1}$, and they were maintained at $100\text{ }^{\circ}\text{C}$ for 1 h.

It is observed that the curves corresponding to the oven-cured samples show a significant cure exotherm, preceded by a clear step-like change corresponding to the glass transition. That change due to the glass transition appears to overlap somewhat with the residual cure. This means that the curing in these two cases has not been completed, and that it is during the DSC test that it is completed. However, the Joule-cured samples with the same temperature programme do not have such an obvious residual cure exotherm, which shows that the curing occurred more effectively than in the oven-cured samples.

Figure 5 shows the evolution of $\tan\delta$ obtained by means of DMA from the same samples as Fig. 4.

In all cases, a bell-shaped evolution, typical of the glass transition, is observed. It is noted that although the maximum $\tan\delta$ values appear at similar temperatures, there

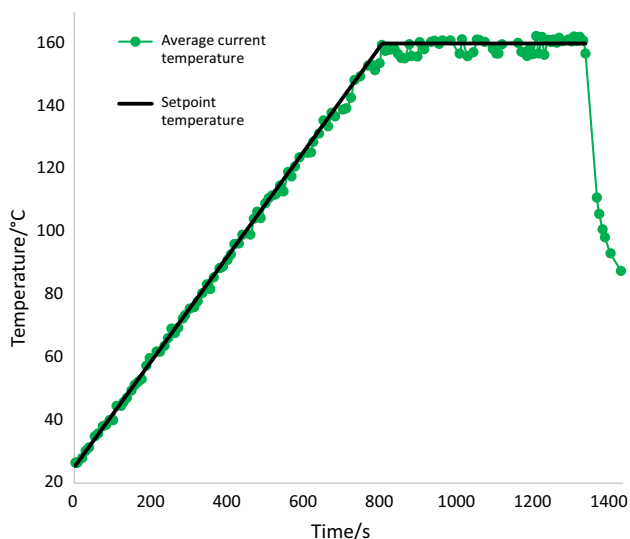


Fig. 3 Plot of the temperature programme and the measured temperature of sample Joule 4

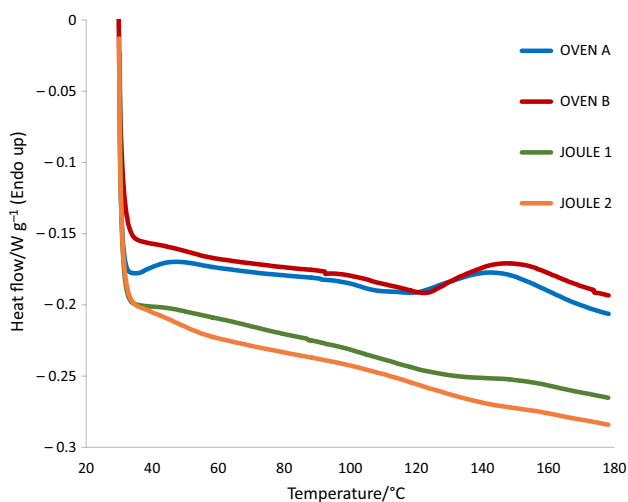


Fig. 4 DSC results for samples cured in oven, for curing temperature of 100 °C and 3 and 4 h, and Joule curing, for curing temperature of 100 °C and 1 h isothermal

are clear differences in the shapes of the curves, with the peaks obtained from the Joule-heated samples being much wider and less high than those obtained by oven curing. $\tan \delta$, which represents the ratio of loss modulus, E'' , to storage modulus, E' , is the most commonly used parameter to represent dynamic T_g , T_{gd} . However, the relaxation process associated with the glass transition typically spans a wide temperature range, which is manifest in the full $\tan \delta$ peak. Although little attention is usually paid to the shape of the peak, its height/width ratio may be of interest as a narrower peak indicates that the complete transition takes place over a narrower range of temperatures, suggesting

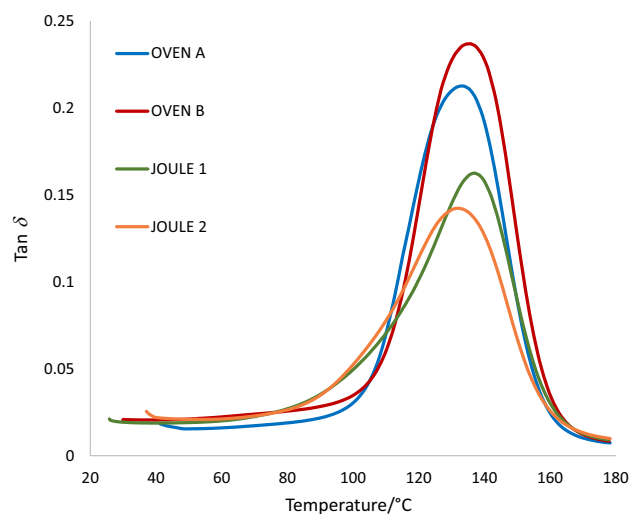


Fig. 5 DMA results obtained from samples cured in oven at 100 °C for 3 and 4 h, and by Joule curing at 100 °C for 1 h

greater structural homogeneity of the sample, at the scale of the ability of the molecular segments involved in the relaxation to move. Taking the curves for the oven samples as a reference, they are much narrower than those generated by the Joule-cured samples. Focusing on the shape of the curve, defined by the value of $\tan \delta$ for each temperature up to 180 °C, the Joule-cured samples present a relation between the curve height and the full width at half maximum (FWHM) in the curves with a much less pronounced shape than the oven samples, as well as not being as high as the previous ones. The area under the curve is larger for Joule curing than in the case of the oven-cured samples, being the peak at a temperature a little below than that of oven-cured samples. This suggests that the curing did not occur as uniformly as in the case of the reference oven-cured samples. This behaviour may be related to some material heterogeneity at the molecular scale, so that there would be chain segments of different lengths that would relax at different temperatures, hence the width of the $\tan \delta$ peak. Such material heterogeneity may easily result from inhomogeneous passage of electric current through the carbon fibres and from the varying distance of each part of the resin to the nearest fibres. To reduce heterogeneity, temperature and time can be played with in several ways. One of them consists of raising the maximum temperature, without being excessive, so that all prepreg parts reach the T_g of the fully cured material, $T_{g\infty}$, during the time necessary to complete the curing.

Taking these results into account, a curing temperature of 160 °C is applied, reducing the isothermal time to 3 min, with the premise that similar values are still obtained for T_g , and that the curve relative to tangent of δ is adjusted, reducing its area. However, despite the change applied, the results obtained were not as expected, as can be seen in Figs. 6 and 7.

With respect to the curve generated in DSC, no notable differences can be seen with respect to the samples obtained in the previous experiment with Joule, and in the same way as before, there is no second curing peak with curing by this method. However, observing the values obtained for $\tan \delta$ during the DMA test, it can be seen that the curve, although it presents a greater peak and its initial slope is greater than in the case of curing at 100 °C, still has a considerably larger area and lower slope than the results obtained for the reference samples, so it could be concluded that, even having increased the curing temperature by 60 °C with respect to the previous experiment, the 3-min isotherm at 160 °C is still insufficient to generate a complete cure. This leads to consider that it is necessary to perform the isotherm for a slightly longer period. However, after all, the shape of the curve for Joule 3 in DMA has improved with respect to the Joule 1 and 2 samples.

Figure 8 shows that extending the isotherm time at 160 °C to 9 min in Joule curing allows obtaining a relaxation curve similar to that of oven curing. However, a significant improvement can be observed in comparison with the previous tests. The curve obtained is narrower and higher than that of the previous Joule samples, resulting in a smaller area and a higher peak. The peak height is even higher than that obtained from the oven-cured samples. As in the previous case, the shape of the curve suggests that the curing was more homogeneous in this case, since the ratio between peak height and peak width is higher than in the previous cases. The results obtained suggest that the use of a higher curing temperature allows a higher proportion of the material to be cured, resulting in a more homogeneous curing of the sample. However, the left side of the peak is still wider than that obtained for the oven-cured samples. This suggests that there is still room for improvement.

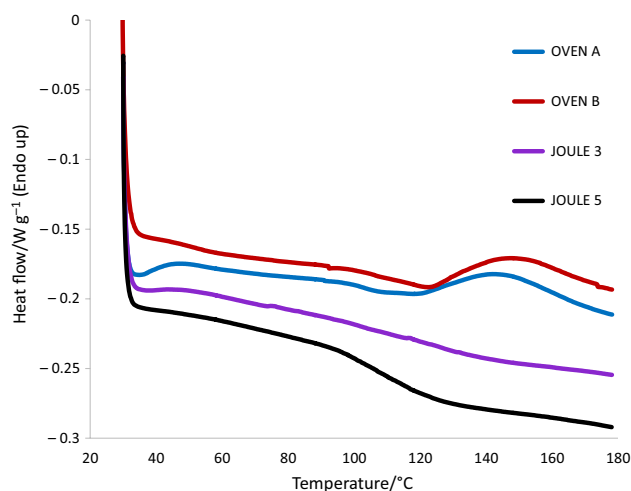


Fig. 6 Heat flow curves obtained from oven-cured and Joule-cured samples. Some curves were vertically shifted

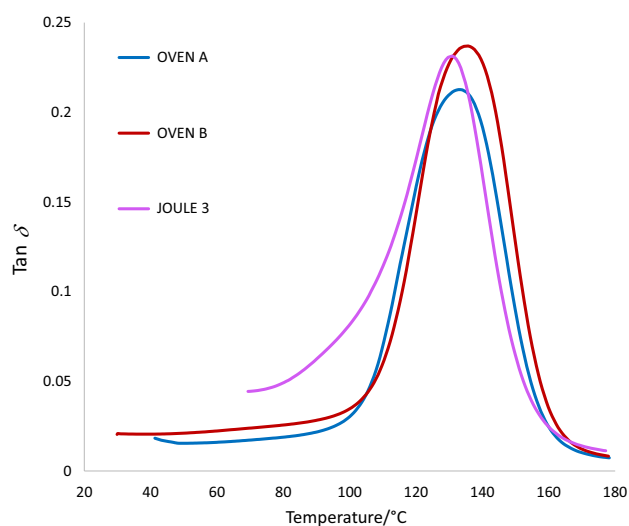


Fig. 7 DMA results obtained from samples cured in oven at 100 °C for 3 and 4 h, and by Joule curing at 160 °C for 3 min

In order to identify a possible effect of a further temperature rise together with a repetition of a Joule curing cycle, sample Joule 5 was prepared. In this case, the treatment consisted of repeating a curing cycle reaching 170 °C, as shown in Table 1.

As it can be seen in Fig. 6, there is no substantial difference between this sample, Joule 5, and Joule 3, which was cured at 160 °C for only 3 min. The different step change height at the glass transition observed between these two samples is probably a consequence of the different resin contents in the composite samples extracted from the laminates for the DSC tests. However, as mentioned above, it is convenient to look at the DMA tests to see more subtle

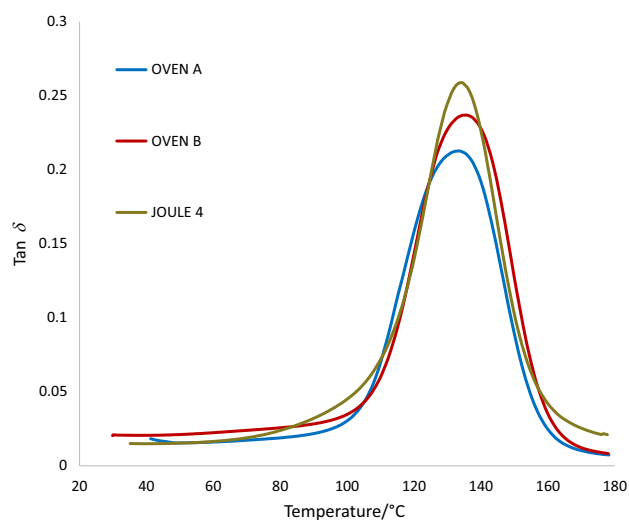


Fig. 8 DMA results obtained from samples cured in oven at 100 °C for 4 h, and by Joule curing at 160 °C for 9 min

differences. Figure 9 shows how a further increase in temperature, up to 170 °C, doubling the curing cycle, produces a small narrowing of the peak width and a small increase in peak height in addition to those already obtained with 6 min at 160 °C. In general, it is very similar in shape to the previous one, so the second curing cycle could be dispensable. Also, the Tgd obtained for both Joule 4 and Joule 5 samples is practically the same.

Considering all the results obtained in the different tests, for all the samples manufactured, it can be observed that the glass transition temperatures results are all maintained within the same order. The temperature difference between the maximum Tg obtained, for the Joule 2 sample, and the minimum, corresponding to the Joule 3 sample, is 6.16 °C, not being an enormous difference between them. Moreover, considering also that the difference between the Tg of Joule sample 2 and that of Joule samples 4 and 5 is about 3 °C, we can confirm that the curing obtained using the Joule effect is quite effective despite using a shorter time. Figure 10 shows the Tg values obtained by DSC and DMA, along with the curing temperature used in each case. The Tg values determined by DSC are around 105 °C in all cases, while those obtained by DMA are around 135 °C. It is observed that the Tg reached slightly higher values in oven curing despite the fact that the maximum temperature applied during curing was about 70 °C lower than in Joule heating. While in Joule curing the temperature of the laminate was measured at all times, in oven curing only the temperature of the oven was measured, so it cannot be ruled out that some overheating occurred due to the curing heat. Based on the data in Fig. 11, which represents the Tg values obtained by DMA versus the curing time applied, for each sample, it was observed that

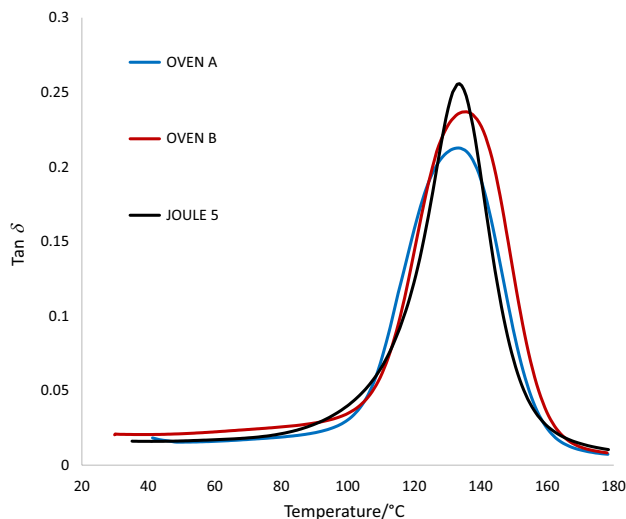


Fig. 9 DMA results obtained from samples cured in oven at 100 °C for 4 h, and by two cycles of Joule curing with isotherm at 170 °C for 3 min in each cycle

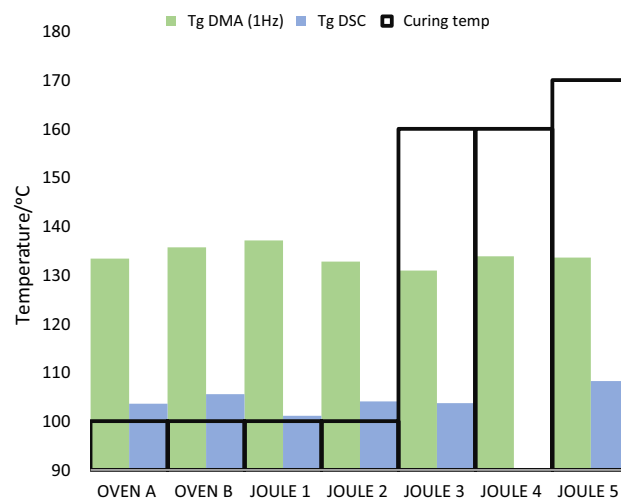


Fig. 10 Tg values obtained by DSC and DMA from the different samples. The temperature at which each sample was cured is superimposed

Joule curing provides good results in terms of curing time, since it allows obtaining similar Tg values, in a time almost ten times shorter than oven curing.

However, it is also observed that, although with a much longer time, traditional oven curing allows to obtain high Tgd values using a much lower temperature.

Figure 12 shows how, in general, the peak width reduces with increasing curing time. This indicates that a narrower peak is associated with more complete curing. It is also clearly observed how Joule curing is much more sensitive to curing time so that with small variations in curing time the width of the tan δ peak can be greatly modified. This higher sensitivity of Joule curing to the curing time is probably due to the much higher temperatures involved in this type of curing.

Based on these results, it can be observed that the use of the tan δ peak shape, together with the maximum peak value, has allowed a better comparison of the degree of cure

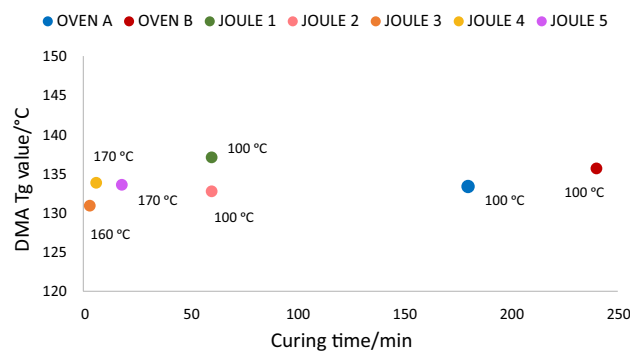


Fig. 11 Plot of the Tg values obtained by DMA versus curing time. The curing temperature is indicated next to each point

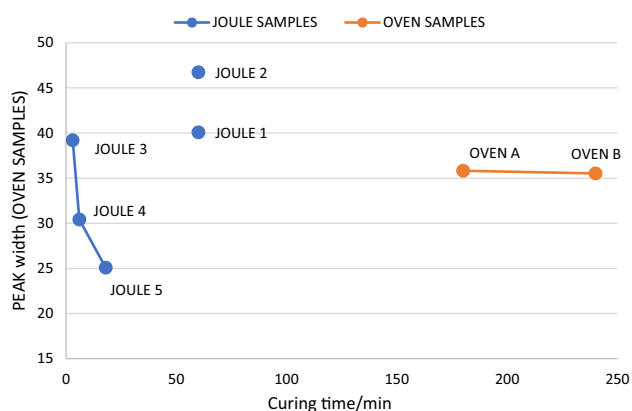


Fig. 12 Plot of peak width at half-height of $\tan \delta$ values obtained by DMA versus curing time

achieved with respect to using only the maximum value. While the peak value informs about the predominant degree of cure in the material, the peak width is related to the structural homogeneity at the scale of movement capacity of the molecular segments involved in the glass transition.

Conclusions

A method for curing carbon/epoxy composites by the Joule effect has been developed to compare the curing obtained by this method with the traditional oven curing method using DSC and DMA. The DSC measurements were useful to identify residual cures, indicative of incomplete cure of the samples in the oven, but did not allow finding clear differences between the samples cured by Joule effect. In contrast, the DMA technique allowed a clear assignment in all cases of the T_{gd} based on the maximum of the $\tan \delta$ relaxation peak corresponding to the glass transition. Values close to 135 °C were obtained, with those corresponding to oven-cured samples being slightly higher. It is very interesting that traditional oven curing allows to obtain high T_{gd} values using a much lower temperature than Joule curing, although this is at the cost of a much longer time. However, it is striking that while the T_{gd} values were very similar, the Joule curing times were one-tenth of those in the oven. Finally, taking as a reference the width of the $\tan \delta$ peak at the glass transition, it is observed that the Joule curing is much more sensitive to the curing time, which is probably a consequence of the fact that the temperatures used in this process are higher than those used in the oven process. This opens the door to modifying the curing characteristics by small modifications of the curing time using relatively high temperatures compared to oven curing. In the end, the method based on the Joule effect facilitates the uniformity of curing at two scales. Macroscopically, since the system

makes it possible to observe and control the temperature distribution on the surface of the cured part. And, at the scale of molecular segments involved in the glass transition, the DMA results show that with some Joule curing programmes it is possible to obtain glass transitions in a narrower temperature range, which means that greater homogeneity is achieved at the molecular scale.

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