Polymer Testing 32 (2013) 1487–1494

Contents lists available at ScienceDirect

Polymer Testing

journal homepage: www.elsevier.com/locate/polytest

Test method

Carbon-fibre epoxy prepreg (CFC) curing in an autoclave analogue process controlled by Dynamic Mechanical Analysis (DMA)

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ARTICLE INFO

Article history:
Received 13 August 2013
Accepted 23 September 2013

Keywords:
Prepreg
CFC
Curing
DMA
DSC
Autoclave
Gelation
Vitrification
Glass transition

ABSTRACT

Carbon fibre prepregs have found widespread application in lightweight constructions. They are based on a carbon-fibre fabric impregnated with reactive epoxy resin. DMA measurements under temperature conditions similar to an autoclave programme were carried out using commercially available prepreg material with a high glass transition temperature. The characteristic of the temperature programme was a dynamic heating segment at 1.5 K/min followed by a longer isothermal segment at 180 °C. The courses of the storage modulus E′, loss modulus E″ and tanδ were recorded. The measuring frequency was varied between 1 Hz and 33.3 Hz. Gelation and vitrification are assigned. The influence of the measuring frequency on the time to vitrification and the correlation with DSC are discussed. The reaction does not end even after 10 h curing at 180 °C, which is interpreted as the slow cessation of the reaction caused by vitrification.

1. Introduction

Glass- and carbon-fibre composite parts are based on the combination of fibres, mainly as fabric, with a polymer matrix. Traditionally, thermosetting resins are used for impregnation [1]. Typical reactive resins are epoxy, unsaturated polyester and polyurethane. For curing, a second reactive species is added as a hardener. Hardener species for epoxy resins are mainly amines or anhydrides [2]. A polyaddition reaction leads to formation of a cross-linked network. The result of cross-linking is that the material is transformed from a liquid into a glass-like solid. Knowledge about the cure state reached during the technical process is of enormous importance for the process control. For safety-relevant parts the degree of cure reached during the manufacturing process must be controlled and documented. It is known that the crack propagation rate in epoxy resin can be reduced by a factor of 10 by increasing the curing temperature by 10 K, and thus changing the degree of cure from 93 to 95% – Trappe et al. [3]. Other works also show the effect of the degree of cure on mechanical properties [4].

For production of fibre-reinforced composite parts, two main technologies are established – resin transfer moulding (RTM) and prepreg technology. At the beginning of the RTM process the fabric is placed in an open, pre-heated mould. After closing the mould and applying a vacuum as needed, liquid resin is injected. The resin is often pre-heated to reduce its viscosity and the necessary curing time. In prepreg technology, the fabric is already impregnated with the reactive resin by the prepreg producer. Since the prepreg is reactive it must be handled with care and stored at low temperatures.

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0142-9418/$ – see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.polymertesting.2013.09.014

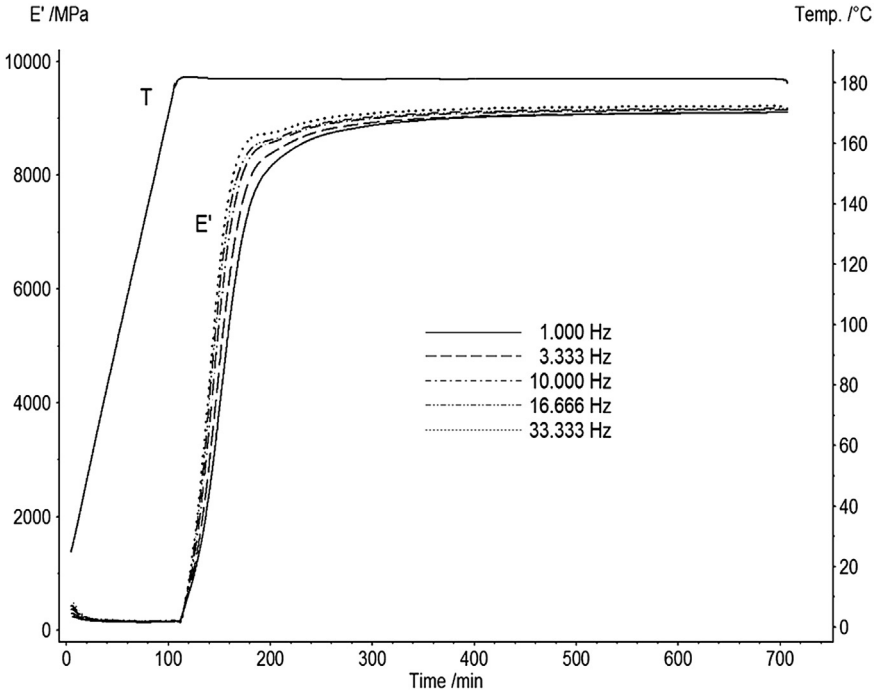

Fig. 1. Prepreg curing in an autoclave analogue process, storage modulus E′ for 5 frequencies.

To form a composite component, multiple layers are overlaid on a tool that defines the final geometry. For curing, the fibre layers are covered by a vacuum-tight foil which is taped to the edges. The air beneath the foil is evacuated, then the entire holder with the prepreg is placed in an autoclave. Within the autoclave, high pressure and high temperature are applied. A typical autoclave temperature programme consists of a non-isothermal, dynamic heating segment from room temperature to the curing temperature and an isothermal segment with sufficient curing time. Finally, a cooling segment follows. In the heating segment, the rate is controlled. Versions of the temperature programme involving isothermal intermediate stages to allow sufficient resin flow when viscosity is reduced at higher temperatures (additional dwell time) are also in use [5].

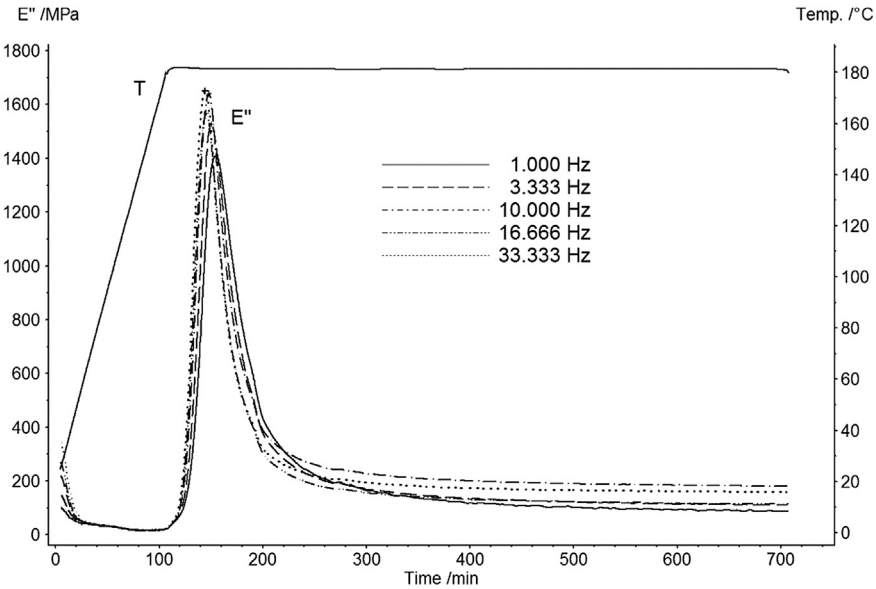

Fig. 2. Prepreg curing in an autoclave analogue process, loss modulus E″ for 5 frequencies.

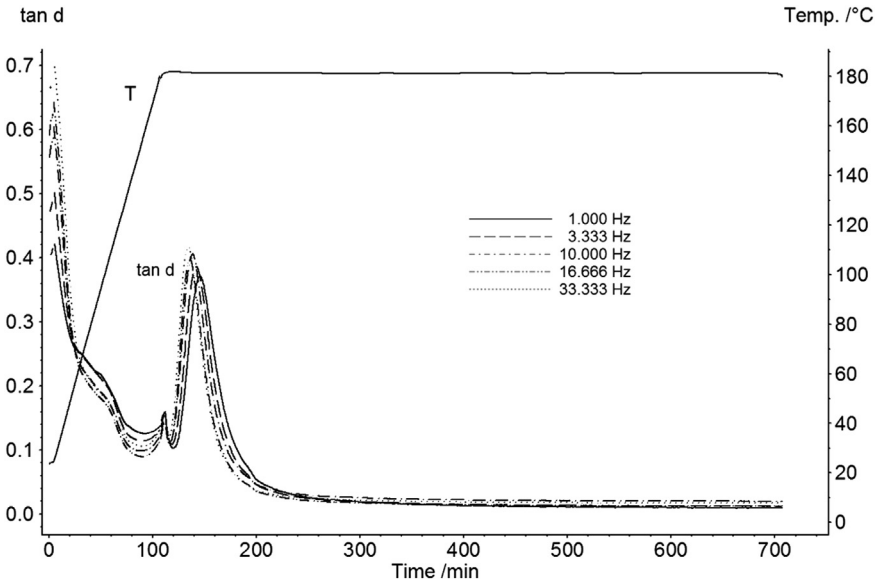

Fig. 3. Prepreg curing in an autoclave analogue process, loss tangent tanδ for 5 frequencies.

The prepreg curing process has a significant influence on the quality of the product. Therefore, knowledge about the process is of great importance to guarantee the specified properties and to optimize the manufacturing parameters. In principle, viscosity measurements can supply viable data on the curing process, therefore, an oscillating rheometer is often used [6–14]. However, the classical rheology method with a plate–plate measuring unit cannot be applied for prepregs.

Curing behaviour of epoxy resin was also investigated by classical Dynamic Mechanical Analysis (DMA) for long periods. For liquid resins, an impregnated braid [15–17] or a container on the sample holder [16] is used. The impregnated prepreg can also be clamped directly into a DMA holder [17,18]. Much of the work was done using torsional braid analysis (TBA) in a torsion pendulum arrangement – for example see [19–25].

Stark [18] demonstrated that DMA can be used to follow the entire curing process of a carbon fibre prepreg in a dynamic heating experiment with 1 or 2 K/min. Here, this work will be continued to directly control the change of the prepreg characteristic mechanical values by DMA for a typical autoclave temperature programme. DMA results are also helpful for developing and better understanding on-

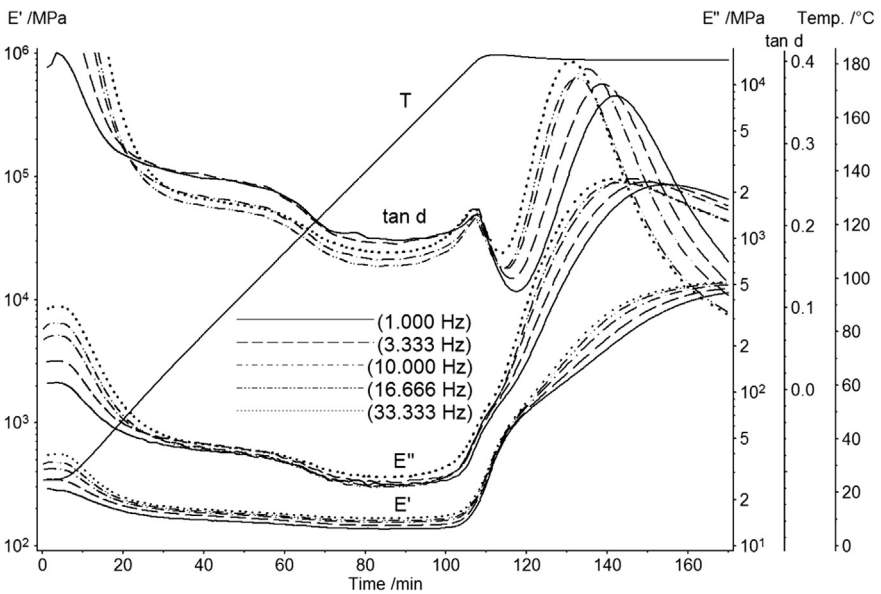

Fig. 4. First 160 min of prepreg curing in an autoclave analogue process, storage modulus E′, loss modulus E″ and loss tangent tanδ.

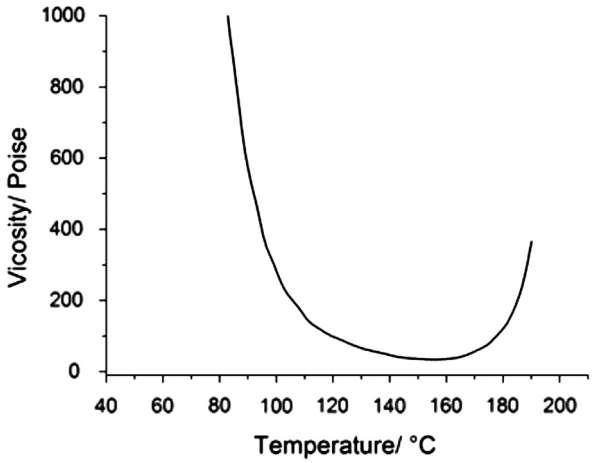

Fig. 5. Prepreg 6376 viscosity as function of temperature for neat resin, data from Hexcel data sheet [32].

line ultrasonic cure monitoring methods, which present great opportunities in the quality management of component manufacturing – [26–31].

2. Experimental

2.1. Material

The investigations were carried out with commercially available carbon fibre prepreg from Hexcel, type HexPly® 6376C (in the following denoted as 6376) [32]. HexPly® 6376 is a tough, high-performance matrix material for the fabrication of primary aircraft structures [33]. The guaranteed shell life at −18 °C is at least 6 months, at 23 °C the tack life is 10 d [32]. The material used had a film thickness of 0.275 mm. It was stored at −18 °C before use.

2.2. DMA measuring technique

The equipment used was a DMA 242C, Netzsch, Germany [34]. A single cantilever holder with a free sample length of 5 mm was used for the investigations. The measuring arrangement is drawn schematically in [18]. The measuring frequency was changed in a periodic mode in 5 steps from 1.0 Hz to 33.3 Hz. The oscillation amplitude was 30 μm. The temperature programme followed a curing process that could be typical for an autoclave curing. It is composed of a dynamic heating segment with a heating rate of 1.5 K/min and a longer isothermal segment. The heating rate was selected in the median range for industrial processes, which typically use values between 1.1 and 2.2 K/min [35]. For the isothermal segment a temperature of 180 °C was selected, which is a typical curing temperature for this prepreg type 6376 [36,37].

3. Results and discussion

A typical result of the Dynamic Mechanical Analysis is given in Figs. 1–3 for the three characteristic mechanical values: storage modulus E′, loss modulus E″ and loss tangent tanδ. Even although all parameters are connected to one another by the equation

$$\tan\delta = \frac{E''}{E'}$$

the best way to characterize prepreg materials as shown in [18] is to analyse, not only the storage modulus E′, but also the loss modulus E″ and the loss tangent tanδ.

In the dynamic heating segment, a slight decrease in storage modulus, loss modulus and a more distinct decrease in loss tangent appear. A more detailed view of this first segment is given in Fig. 4 for another measurement. At the beginning, a 5-min isothermal segment was

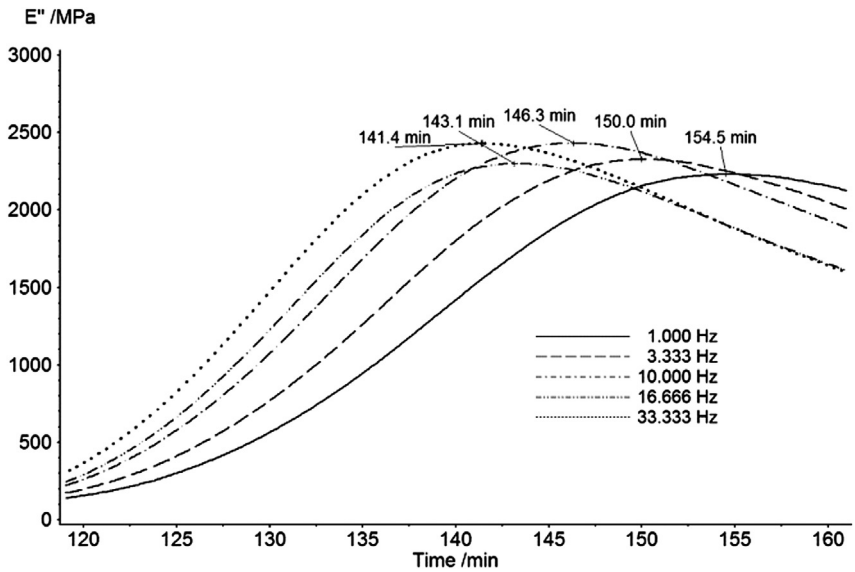

Fig. 6. Time dependence of loss modulus peak in the isothermal segment, parameter: measurement frequency.

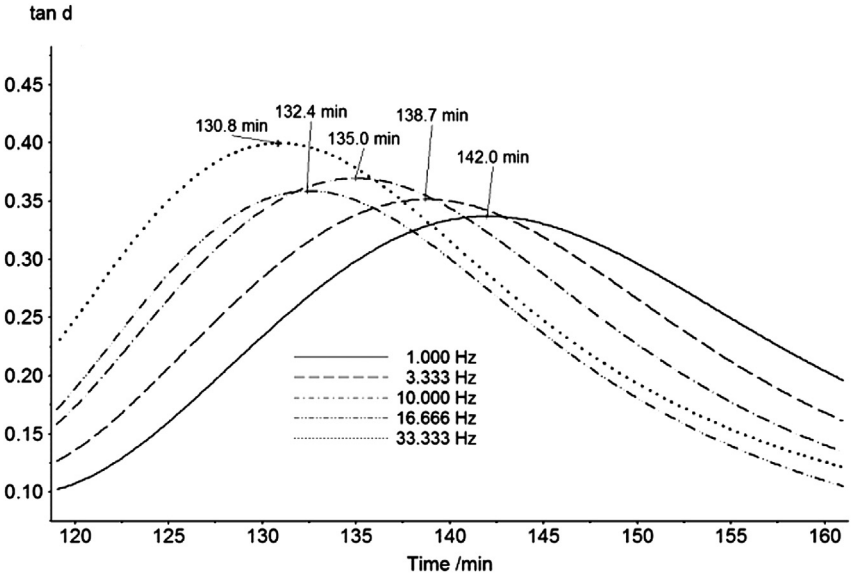

Fig. 7. Time dependence of peak in the isothermal segment, parameter: measurement frequency.

programmed to give more definite start conditions. Therefore, in the discussion these 5 min will be subtracted.

For the storage and loss modulus, a logarithmic axis was chosen in Fig. 4. The boundary values for the axis were selected such that the curves do not overlap excessively.

3.1. Time interval 0–50 min

With increasing temperature, all three mechanical characteristic values E′, E″ and tanδ decrease. Thereby, the influence of the frequency, which is quite pronounced at the beginning, is ever more reduced. The changes in the values seen here can be interpreted by the after-effect of the glass-rubber transition of the non-cured resin, often symbolized with T_{g0}. This transition is situated at around −1 °C for DSC, and between 10 °C (at 1 Hz) and 20 °C (at 33.3 Hz) for DMA [18].

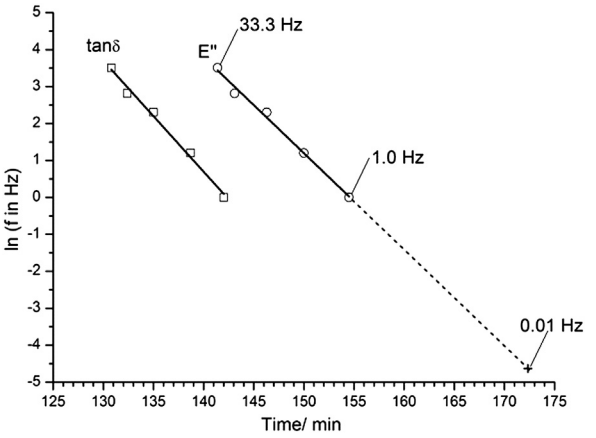

Fig. 8. Time for the peak appearance at vitrification for different measuring frequencies, 0.01 Hz added as an equivalent frequency for 10 K/min DSC measurement.

3.2. Time interval 50–90 min

At ca. 55 min (110 °C) an additional decrease in tanδ and E″ appears but is finished after approximately 90 min (160 °C). This behaviour can be interpreted by the temperature function of the resin viscosity. A temperature dependence of viscosity for 6376 resin is given in the Hexcel data sheet [32]. In Fig. 5, a plot of the scanned Hexcel data drawn here with a linear axis is given. Near 110 °C, the viscosity has ceased its sharp decrease and approaches its lowest value at 160 °C.

3.3. Time interval $t > 90$ min

First indications of a rise in all three characteristic mechanical values is seen from ca. 95 min (170 °C). This is also the temperature for a viscosity increase in Fig. 5. The deviations between the frequencies are ever more reduced until a new distinct influence of the frequency emerges at about 110 min. The curves belonging to the higher frequencies show this effect first. E′ passes through a step during which a change in the rise appears. E″ also shows a kind of a step and then a pronounced peak. It is interesting that the course of the tanδ curves after reaching a maximum decreases with progressing time, whereby the curves for all frequencies lie on top of each other. After a minimum a peak follows – for the highest frequency first.

Such behaviour as in Fig. 4 is typical for a process where the curing temperature is high enough that the degree of cure will exceed the value necessary for gelation, but still low enough that vitrification can appear. Vitrification means that the curing temperature is distinctly below the

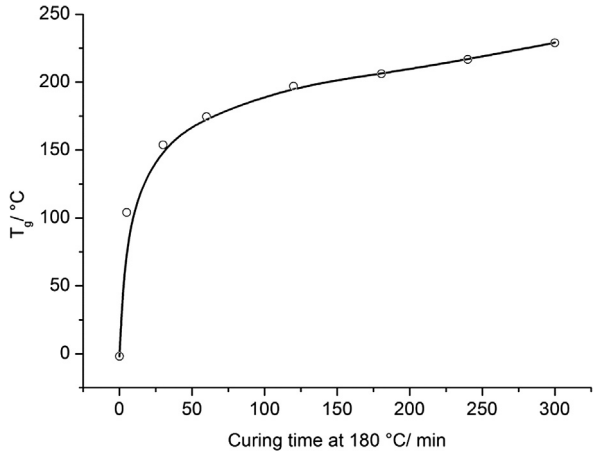

Fig. 9. T_{gDSC} as function of curing time, curing temperature 180 °C – results taken from [49].

maximum glass transition temperature of the fully cured resin [21,38–43].

For the selected autoclave curing programme, gelation followed by vitrification is expected. In the literature, the first tanδ peak and, especially, the loss of frequency influence are associated with gelation [10,11,44–47].

Gelation appears at approximately 103 to 105 min. The second tanδ peak and the E″-peak are a typical indication that the actual glass transition temperature ($T_{gactual}$) reaches the curing temperature and causes vitrification [9,12,14,23,48].

The influence of frequency on the glass transition leads to the phenomenon that the higher the frequency, the less time (lower degree of cure) is necessary to reach the maximum in E″ and tanδ, respectively. The details showing the peak location for the different frequencies are given in Figs. 6 and 7.

The correlation between the time when the peak occurs and the logarithm of the measurement frequency is sketched in Fig. 8. The measurement points can be connected by a straight line. In [18] a correlation between the frequency and the E″ peak glass transition temperature was found. An extrapolation to the glass transition temperature measured by DSC with a heating rate of 10 K/min gave an equivalent frequency for DSC as approximately 0.01 Hz. Therefore, a frequency of 0.01 Hz is additionally marked by a "+" in Fig. 8, denoting the time at which vitrification would be expected to occur in an isothermal DSC experiment.

Taking into account that the curing in the autoclave programme starts at about 95 min, it was possible to estimate an extrapolated time at which a DSC glass transition temperature (T_{gDSC}) during curing at 180 °C would occur. This time is 77 min (172 min from Fig. 8 minus 95 min). This estimated value is compared with measurements of T_{gDSC} as a function of the 180 °C curing time determined by thermo-modulated DSC [49] in Fig. 9. For 77 min curing time, T_{gDSC} does indeed come close to the curing temperature 180 °C and vitrification can occur.

In Fig. 10 E′ and E″, details for the longer tail of the curing time are shown. From the earlier discussion, it is clear that the actual glass transition temperature exceeds the curing temperature. As a consequence, the system is vitrified, the reaction velocity is considerably reduced and the reaction becomes diffusion controlled [42]. The long, slow increase of $T_{gactual}$ presented in Fig. 9 indicates that the reaction rate decreases and that vitrification plays an important role. The rise in the difference between the actual glass transition temperature and the curing temperature causes the continuous rise of E′ and decrease of E″, as shown in Fig. 10. Obviously, the reaction slows but does

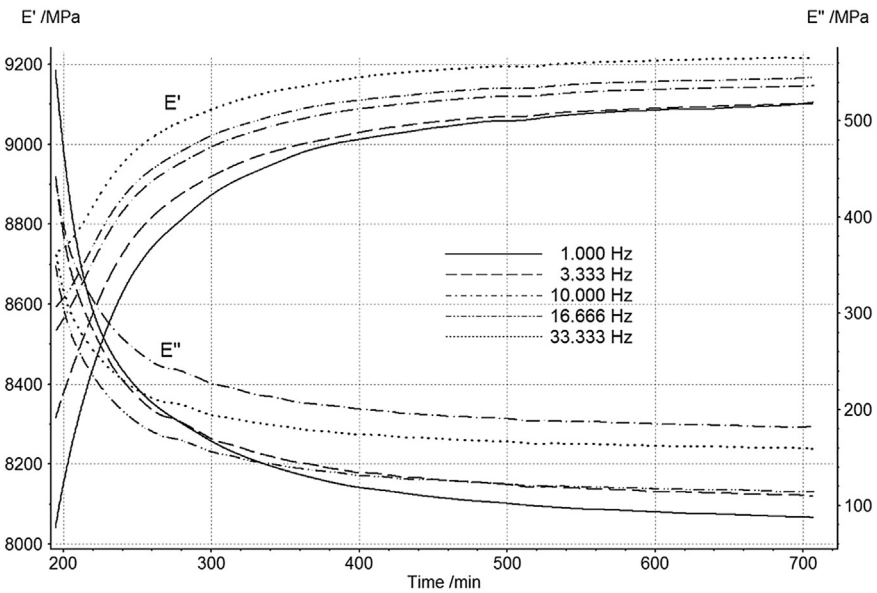

Fig. 10. Storage modulus E′ and loss modulus E″ change during the longer part of the 180 °C isothermal segment.

not come to an end in the investigated time-frame. Therefore, T_{gact} does not reach $T_{g\infty}$.

Sourour and Kamal [50] found that the glass transition temperature did approach a value more than 10 K higher than the curing temperature. Published results show that when vitrification occurs, 100% cure is not reached even for long cure times if the difference between T_{cure} and $T_{g\infty}$ is too high – [17,43,51–55].

However, vitrification is not always responsible for the long times needed to achieve 100% cure. At the end of the reaction, the concentration of the reaction partners has decreased so much that the probability of reactive partners meeting becomes very low. By Koreeda et al. [56] for example, at 10 h a 92.4% degree of cure was achieved with a curing temperature above $T_{g\infty}$, 93.5% at 12 h and 99.5% after 48 h.

4. Summary

For commercially available carbon fibre epoxy prepreg 6376, the typical autoclave curing process was traced by DMA. Typical indications were found for gelation and vitrification. The influence of the measuring frequency was demonstrated. The higher the frequency, the lower the curing times needed before vitrification becomes visible. An extrapolation to a low frequency (0.01 Hz) yielded a cure time that could be compared with T_{gDSC} measurements and provides a good correlation between DMA and DSC experiments. A major advantage from DMA is its capacity for long-term control of curing behaviour, while DSC has no chance of detecting the very small heat flow remaining. Even for cure times of 10 h at 180 °C, it is clear from DMA measurement that the end of reaction has not been reached.

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