Tg and Cure of a Composite Material

Summary

A powder-filled, epoxy-based composite material is investigated in this application note. A multi-frequency thermal scan will give information about the glass transition and cure of the material. An isothermal experiment, after raising the temperature above the cure temperature is also discussed. This latter experiment gives unusual frequency dependence information. The glass transition and the cure behavior of composite materials are of special importance due to the types of applications they are used for.

Introduction

Dynamic Mechanical Analysis (DMA) is one of the most appropriate methods to investigate relaxation events. A composite, by definition, contains more than one component. There are multiple types of composite materials used for various applications from glass fibre reinforced concrete to sophisticated aeronautical polymer composites. In these experiments, a powder filled composite of epoxy polymer proposed for battery manufacture is used.

The PerkinElmer® DMA 8000 works by applying an oscillating force to the material and the resultant displacement of the sample is measured. From this, the stiffness can be determined and tan δ can be calculated. Tan δ is the ratio of the loss component to the storage component. By measuring the phase lag in the displacement compared to the applied force it is possible to determine the damping properties of the material. Tan δ is plotted against temperature and glass transition is normally observed as a peak since the material will absorb energy as it passes through the glass transition.

Being an epoxy-based composite, the glass transition is not so simple in that there is also a cure process happening at the same time. An explanation of both the thermal scan and isothermal cure data is discussed.

Experimental

(1) Multi-frequency temperature scan of composite. The sample was mounted in the single cantilever bending clamps and run through the temperature scan at multiple frequencies.

(2) Isothermal frequency scan of composite. A fresh sample was mounted in the single cantilever bending clamps and heated quickly to 100 °C. Modulus and tan δ data were collected for 3 hours.

Equipment	Experimental Conditions	
DMA 8000	Sample:	Composite Material
1L Dewar	Geometry:	Single Cantilever Bending
	Dimensions:	9.7 (l) x 10.1 (w) x 1.7 (t) mm
	Temperature:	(1) 25 °C to 250 °C at 3 °C min^{-1}
		(2) Isothermal at 100 °C
	Frequency	0.316, 1.0, 3.16, 10.0 and 31.6 Hz

Results and conclusion

Figure 1 shows the thermal scan of the composite material. A clear frequency dependence is observed in both the modulus and the tan δ data indicating a relaxation event. As the material passes through the Tg, a cure reaction also takes place. The material gets less stiff (modulus decreases) as a result of the Tg and the increasing temperature despite the cure process going to completion. The frequency dependence is as expected with higher frequencies giving higher glass transition temperatures. The Tg at 1 Hz is approximately 92 °C.

The result from the isothermal experiment is graphed in Figure 2. 100 °C was chosen for the experiment as it was slightly higher than the Tg from the first experiment.

The peak in the tan δ shows a combination of the glass transition and the cure process. It appears that the frequency dependence is the opposite to that observed in the first experiment, with the higher frequency peak occurring first.

The isothermal experiment results are a function of time, not temperature and are shown in Figure 2. The curing of the epoxy will be the dominant process observed in these data. As the cure progresses, the relaxation time will get longer. A maximum in tan δ will be observed when the relaxation time is approximately the same as the probing frequency. Hence, the high frequency event (shortest probing frequency) will occur first as the relaxation time will be the shortest. The lowest frequency peak occurs when sufficient cure has taken place to lengthen the relaxation time appropriately.

This application note has described the ability of DMA to investigate curing and glass transition events in composite materials. The isothermal cure experiment gave data that better describes the cure than the Tg event.

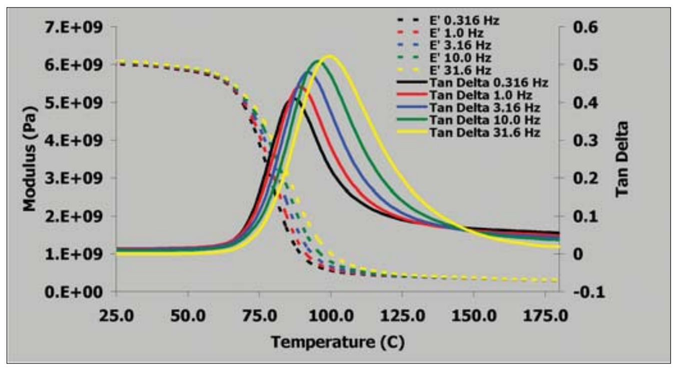

Figure 1. Thermal scan data of composite material.

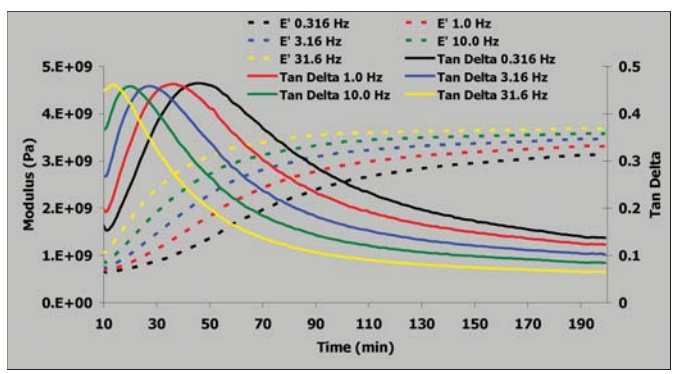

Figure 2. Isothermal experiment results.

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007771_08 Printed in USA