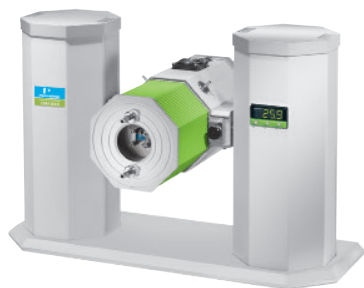




Thermal Analysis

Dynamic Mechanical Analysis (DMA) is one of the most appropriate methods to investigate relaxation events.

Get a Better Understanding of Material Properties and Cure Behavior of Composites



PerkinElmer DMA 8000

Introduction

Composites are defined as the material formed by combining two or more different materials or constituents.

The combination of different constituents in the composites provides the composite material with unique properties which are different from the individual constituents. Composites are used in a wide variety of markets, including aerospace, automotive as automobile bodies, bumpers and others, battery manufacturing as well as in architectural buildings, energy wind turbine blades, etc. They can also be found in medical applications, sports and other fields. Specifically, composites have gained popularity in the automotive industry for their lightweight and cost-effective benefits. Automotive manufacturers are using composites to help reduce vehicle emissions and improve energy efficiency.

To get a good understanding of the material properties such as glass transition and the cure behavior of composite materials are of special importance due to the types of applications they are used for.

Experimental

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.

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 fiber 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 $\tan \delta$ can be calculated. $\tan \delta$ 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. $\tan \delta$ 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.

- 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.
- Isothermal frequency scan of composite.** A fresh sample was mounted in the single cantilever bending clamps and heated quickly to 100 °C. Modulus and $\tan \delta$ data were collected for three hours.

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 $\tan \delta$ data indicating a relaxation event. As the material passes through the T_g , a cure reaction also takes place. The

Equipment	Experimental Conditions	
DMA 8000 1L Dewar	Sample:	Composite Material
	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

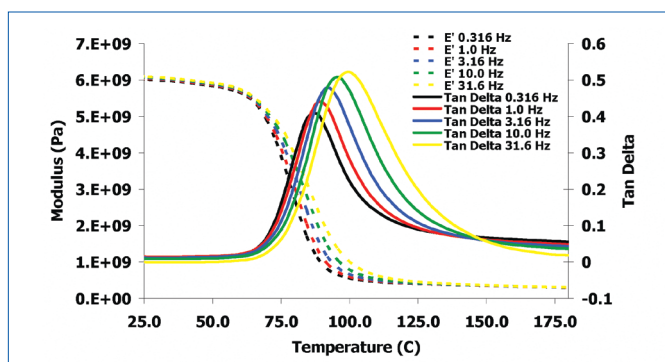


Figure 1: Thermal scan data of composite material.

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material gets less stiff (modulus decreases) as a result of the T_g 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 T_g 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 T_g from the first experiment.

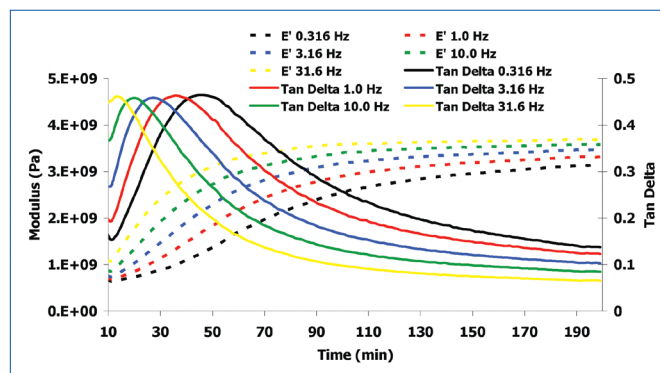


Figure 2: Isothermal experiment results.

The peak in the $\tan \delta$ 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 $\tan \delta$ 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.

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.

