SII NanoTechnology Inc.

DMA AS PROBLEM-SOLVING TOOL: EVALUATION OF THERMOSETTING RESIN

Problem

A thermal analyst is working with an uncured phenolic- epoxy thermosetting resin system and desires to have more information regarding the mechanical transformations that occur as the resin is heated. These events include: the glass transition temperature (Tg) and the subsequent softening of the resin; the point of gelation associated with the crosslinking event; and the temperature of completion of cure. These particular transitions are important in the proper processing of the resin into the final product. The uncured resin exhibits a very large change in the mechanical properties during the various transformations while heating and is thus difficult to handle.

Solution

Dynamic mechanical analysis (DMA) provides a means of assessing the viscoelastic properties of a wide range of materials, such as uncured resins. DMA yields data on the following parameters:

- Storage modulus, E', which is indicative of the stiffness of the material
- Loss modulus, E", or damping, which is related to the sample's energy absorbing properties
- tan δ, which is an index of the material's viscoelasticity (E"/E').

The Seiko Instruments DMS6100 can conveniently handle uncured thermosetting resins using a bending clamping arrangement. The instrument provides a high degree of sensitivity, through the use of Fourier transform technology, excellent subambient control and ease of use. The uncured resin is simply painted onto an inert support medium (fiberglass cloth) and this is then placed into the DMA clamping assembly. In order to prevent the resin from sticking to the clamps during curing, the inside portions of the clamps are covered with a layer of aluminum foil.

The resin/cloth sample specimen was heated at a rate of 2°C/min from -60 to 200°C to observe all of the mechanical transformations. In order to assist with the interpretation of the observed events, the sample was scanned as a function of frequency, as well as temperature. Frequencies between 0.01 and 100 Hz can be applied with the DMS 6100 and this permits the assessment of the time dependency of the observed transitions. Relaxation events, such as Tg, are frequency dependent and thus show a significant increase in the loss peak temperature with respect to increasing frequency. Chemical transformations of the sample, such as gelation or degradation, are independent of the DMA frequency. For this sample, frequencies of 0.5, 1;2, 5, 10 and 20 Hz were utilized.

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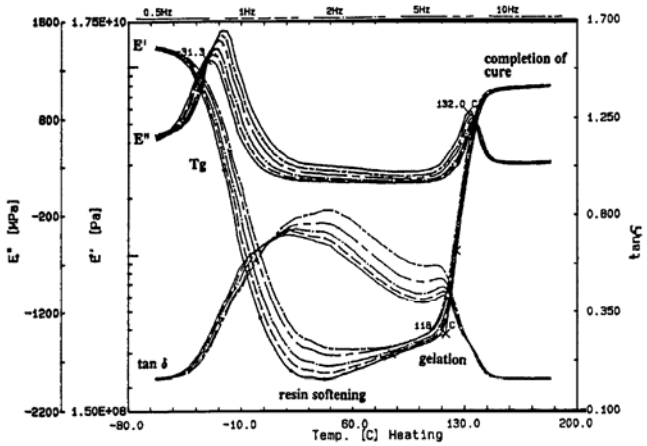

Figure 1

Displayed in Figure 1 are the results obtained from the DMA on the uncured resin specimen. The glass transition event (Tg) is observed as a series of frequency dependent peaks in E" between -32 and 0°C. The Tg, at 0.5 Hz, occurs at -31.3°C for the resin. The storage modulus exhibits a large drop at Tg as the resin softens.

At 118°C, the modulus response shows a large increase which represents the gelation of the resin. The gel point is the temperature at which the resin converts to a three dimensionally crosslinked network structure and, at this point, it can no longer be processed. The tan δ data exhibits a small peak at 118°C due to the gelation of the resin. The peaks are frequency independent since the gel point represents a chemical transformation of the material.

Upon further heating, above the gel point, the resin continues to undergo curing and eventually converts from the rubbery state to the glassy state. This is known as revitrification and is observed as a series of peaks in the loss modulus response at 132°C. Above 140°C, the modulus response is completely flat, with respect to increasing temperature, which indicates that the resin has been completely transformed from the uncured to the fully cured state.

Summary

The Seiko Instruments DMS 6100 was used to characterize the transformations of an uncured thermosetting resin. The following events can be observed using the DMA approach: the glass transition temperature or the softening of the resin, the gelation temperature, and the temperature of completion of cure.

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