Dynamic Mechanical Properties of Epoxy Resins During Polymerization

In a previous article the measurements of the electrical properties of various epoxy resins during polymerization were reported.[1] In the course of recent measurements of the dynamic mechanical properties of polymers in our laboratories, we have recorded the mechanical behavior of some epoxy resins during polymerization. It is interesting to note the comparison of electrical and dynamic mechanical measurements.

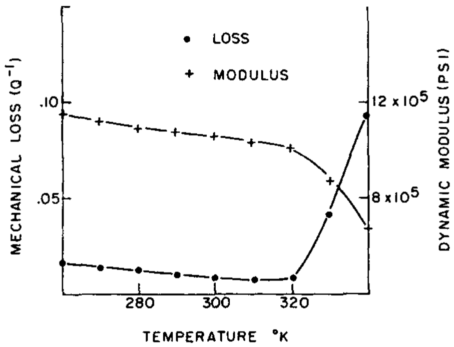

Fig. 1. Mechanical loss (●) and dynamic modulus (+) as a function of temperature for an epoxy sample nearing the second-stage cure temperature.

Experiments were conducted with a modified version of an apparatus developed by the author[2] and used in various investigations reported in the literature. Test frequencies were in the range of 700–1000 cycles/sec. Samples were prepared from a bisphenol A-based epoxy resin (Epon-828 of the Shell Chemical Co.) with *m*-phenylenediamine as a catalyst. (Epon-828 is also one of the epoxy resins cited in the work of Delmonte.[1]) These were thoroughly mixed in a ball mill near 60°C. and filled with 85 parts of fine aluminum powder (average diameter 15 microns) per 100 parts of the resin–catalyst mixture. After another mixing, specimen molds were filled and the resin allowed to room cure to the intermediate state. For practical use, this must then be second-stage cured at an elevated temperature (often near 70°C.) for times which depend upon the particular application. Much of this cure takes place in the first hour.

If one makes dynamic mechanical measurements as a function of temperature on a sample which has undergone only the room temperature cure, results are obtained as indicated in Figure 1. In this run, the temperature was raised at the rate of about 1°C./hr. The temperature rise was too rapid to permit second-stage curing before the sample collapsed because of the drop in modulus. It is noted that an increase in mechanical loss, Q^{-1}, accompanies the drop in dynamic modulus.

When the temperature of the oven surrounding the sample is stabilized to about 333°K. (60°C.) before insertion of a specimen, the mechanical loss first increases and then decreases, as shown in Figure 2. Correspondingly, the modulus decreases and then increases as the polymerization

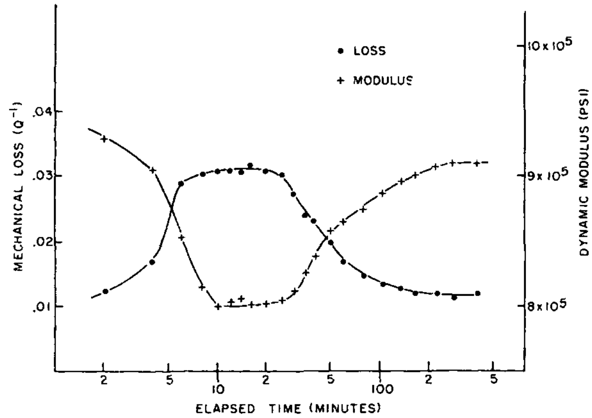

Fig. 2. Mechanical loss (●) and dynamic modulus (+) of an uncured epoxy sample as a function of elapsed time after insertion into an oven near 60°C.

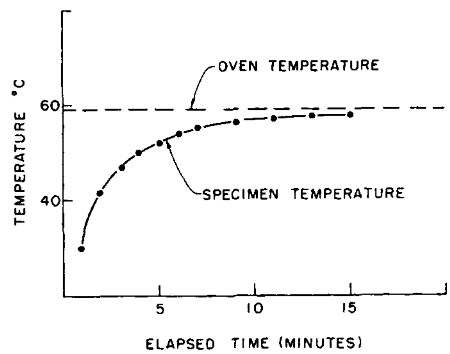

Fig. 3. Epoxy specimen temperature as a function of elapsed time after insertion into an oven near 60°C.

of the sample progresses. Both of these level off after long times. In Figure 3 the temperature behavior of an identical sample is given as a function of time elapsed after insertion. From this and the behavior depicted in Figure 1, one concludes that the effects noted in approximately the first ten minutes after insertion (Fig. 3) are largely those associated with the specimen reaching the oven temperature. The effects from this time on are a result of second-stage curing action and resemble, to some extent, the electrical results reported. Investigations of unfilled epoxy samples, rather than the aluminum-filled mixtures above, indicated the same general behavior.

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References

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