

Bridge You and Nano

Using DSC to Measure the Degree of Curing in Commercial Epoxy Resins

Among the many capabilities of the NAT Lab's Netzsch STA 449 F3 Jupiter, is the ability to determine the degree of curing for a thermoset polymer resin such as an Epoxy. Further understanding the extent of curing in the polymer system can help to ascertain the performance and reliability of the material and to identify the ideal timing for use. With poor crosslinking, epoxies risk failing to meet specifications which can lead to disastrous results for customers relying on their adhesive strength. Differential Scanning Calorimetry (DSC) operates by comparing heat flow through a sample to that of a reference. Heat absorbed or released by the sample during a transition will generate an offset correlating to the enthalpy of reaction. In the presence of a dedicated curing agent, epoxy resins will undergo such a transition where crosslinked bonds begin to form and an exothermic peak is observed in the plot of heat flow vs time.

For this experiment, epoxy was mixed using a provided corkscrew applicator tip and deposited into aluminum crucibles in preparation for analysis. Samples were allowed to cure for 7 min, 20 min, 1 hr, and 24 hr intervals prior to testing. One uncured sample (tested at < 5 min prior to the product's reported onset of curing) and one fully cured sample (heated once to 250 °C to accelerate curing, and cooled back down to room temperature prior to testing) were also included in order to demonstrate the complete thermal behavior of the epoxy before and after curing.



Figure 1. Exothermic peaks of epoxy curing for various time intervals.



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Exotherms depicting the heat flow with respect to temperature (time) were observed and the areas under the curves were used to calculate the enthalpies of transitions. Testing the uncured epoxy revealed distinct thermal peaks occurring at 43.8 °C and 114.4 °C. Using the instrument software to integrate the areas under the curve, the total enthalpy of curing was determined to be 189.79 J/g. Enthalpies were obtained for each of the curing times and compared with the total to determine a relative percent of cure (see Table 1).



Figure 2. Areas under the peaks of the uncured sample showing enthalpy of the full reaction.

Sample	Enthalpy	Degree of Cure
Uncured	189.79 J/g	00.0 %
Cured for 7 min	144.53 J/g	23.8 %
Cured for 20 min	108.60 J/g	42.8 %
Cured for 1 hr	62.33 J/g	67.2 %
Cured for 24 hr	48.52 J/g	74.4 %
Fully Cured	0.00 J/g	100.0 %

Table 1 Enthalpies of Reaction and Associated Percentages of Curing for Epoxy Resins

By comparing the percentages of curing, it can be known that approximately 42.8% of the cure has occurred by the 20 minute mark at which time the product label claims the epoxy is ready for use, and only 74.4% cure has occurred at the 24 hour mark when the product is said to be "fully cured." This observation is consistent with expected degrees of curing for two part room temperature cured epoxy that typically rely on longer time or heating to achieve a complete cure.