White Paper

Evaluation: Degree of Cure of EpoTek 353ND Adhesive with iCure Thermal Spot Curing System Versus Conventional Oven

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Overview

A series of tests have been carried out to compare two curing processes for a heat curing adhesive, namely conventional oven curing and the iCure AS200 infrared thermal spot curing system from IRphotonics.
The results obtained on Epotek 353ND adhesive show that tested samples are equally cured with both methods but the curing time is reduced from 1-2 hours to 30sec with the iCure.
Similar results have also been obtained with other types of thermal adhesives.

DSC Evaluation of degree of cure

Differential Scanning Calorimetry (DSC) is a recognized technique to evaluate the degree of cure of adhesives. The DSC instrument can detect any change in heat flow or enthalpy of test samples with a high degree of accuracy. It can also be used as a conventional oven to cure adhesives.
An automatic pressure-time dispenser from EFD was used to dispense the exact amount of adhesive and samples were sealed in aluminum pans as per recommended procedures. DSC experiments were performed with a Perkin Elmer DSC-7, at a heating rate of 10ºC/min.
A sample of liquid adhesive was first scanned with the DSC to determine a baseline specific enthalpy. Figure 1 is the DSC plot for the liquid adhesive. It shows the normal behavior of a sample as it is cured inside the chamber of the DSC. The corresponding specific enthalpy was measured at -496.63 J/g.

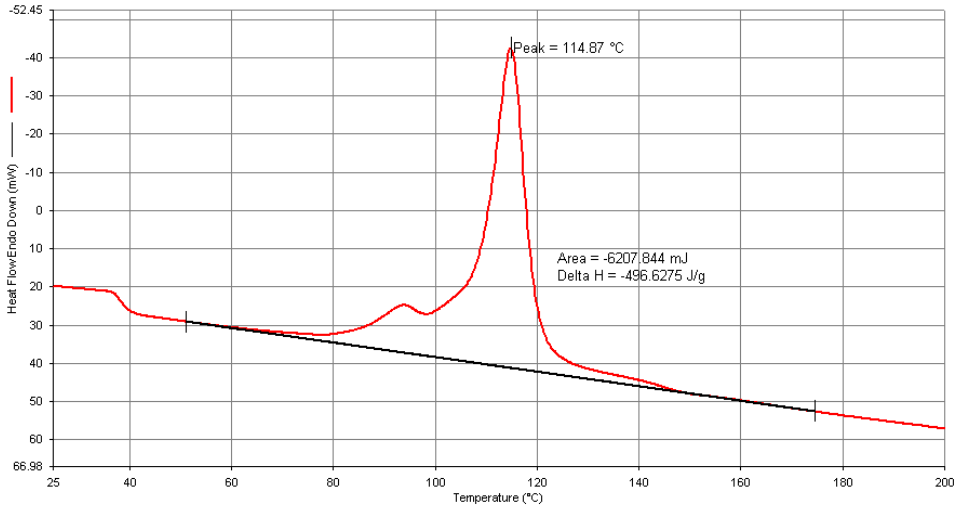

Figure 1: DSC plot of liquid 353ND adhesive

The cured sample was scanned a second time to obtain its residual enthalpy. The ratio of residual enthalpy of the cured adhesive to that of the liquid sample indicates the degree of cure of the adhesive.

Results and discussions

For the first series of tests on the 353ND adhesive, a Perkin Elmer DSC-7 calorimeter was used to simulate oven curing. To allow the highest degree of cure, the heating profiles were purposely extended longer than the minimum recommended by the epoxy manufacturer. Two 10 mg samples of liquid 353ND (labeled A1 & A2) were heated by rapidly raising the temperature to 120ºC and soaking it for a period of 1 hour and 2 hours, respectively. The cured samples were heated a second time at a constant rate of 10ºC/min to 200ºC to evaluate the residual enthalpy and the results are provided in Table 1.
Figure 2 shows that the residual enthalpy of sample A2 (120ºC during 2 hours) is -5.02 J/g and that it is 98.99% cured.

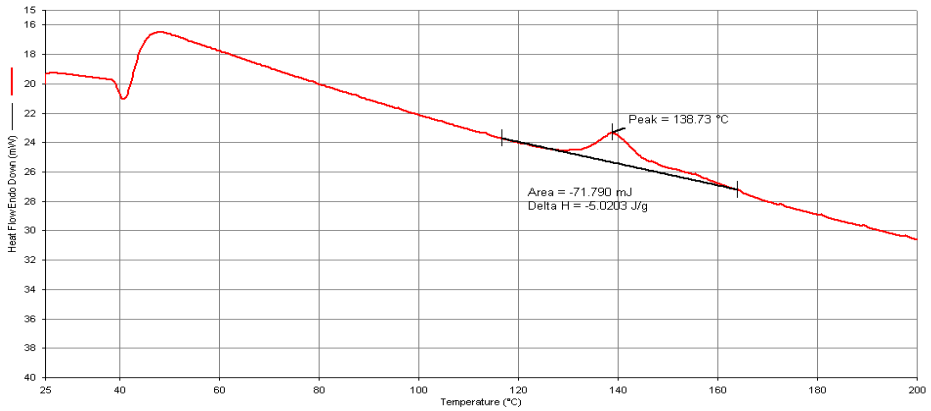

Figure 2: DSC plot of 353ND adhesive cured at 120C for 2h

Table 1 and Table 2 show experimental results obtained versus data provided by the adhesive manufacturer. Results show that even after a one hour soak at 120ºC, the 353ND is not fully cured. Data is confirmed by adhesive supplier (Table 2)

Cure Condition	Residual exotherm (J/g)	% Curing
DSC 120C 1h	-6.71	98.65
DSC 120C 2h	-5.02	98.99

Table 1: DSC curing data

Tg for A1 & A2 samples was 108ºC and 110ºC, respectively

Cure Condition	Residual Exotherm (J/g)	% Curing	Tg (°C)
Oven 120 C 1h	-8.67	98.3	110
Oven 120 C 2h	-0.8	99.8	110

Table 2: provided by EPOTEK

iCure Thermal Spot Curing

IRphotonics' thermal spot curing system, the iCure AS200 is a broadband UV-Visible-Infrared light source that provides energy in the 0.3 to 3.6 micron wavelength range. Most polymer based adhesives have a wide absorption band between 2.3 an 3.6 micron due to their hydrocarbon based chemistry. The adhesive heats by readily absorbing infrared radiation.

A second series of tests was performed using the iCure AS200. Two samples B1 & B2 were exposed to 3 Watts of power for 30 seconds. The cured samples were heated a second time in the DSC to 200°C at a constant heating rate of 10°C/min to quantify the residual enthalpy. An optical transparent substrate with a good transmission from 0.3 to 7 micron was used to ensure that the optical energy was only absorbed by the epoxy itself and that no heat was contributed to the adhesive by the substrate.

Figures 3 and 4 show the corresponding DSC curves. The virtual absence of residual enthalpy demonstrates that the samples have been fully cured with an estimated degree of cure greater than 99%.

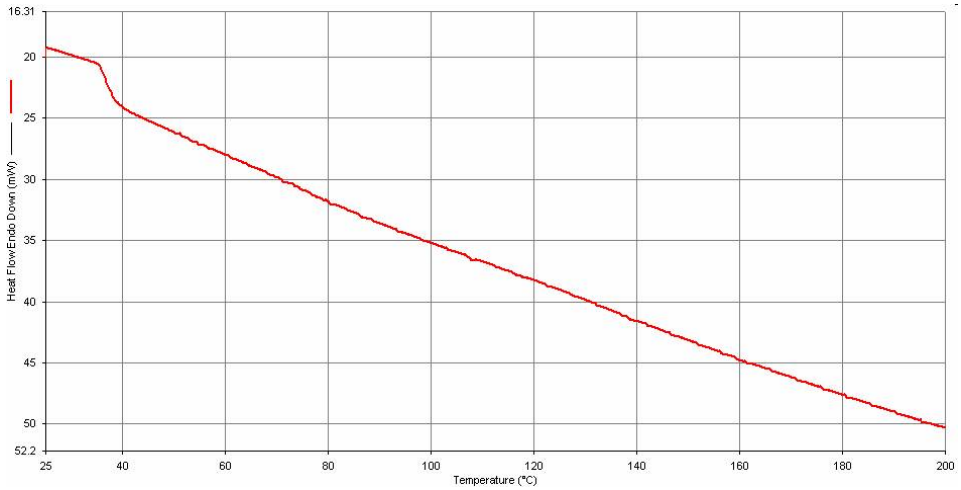

Figure 3: DSC curve of 353 ND cured with iCure 3W 30 s

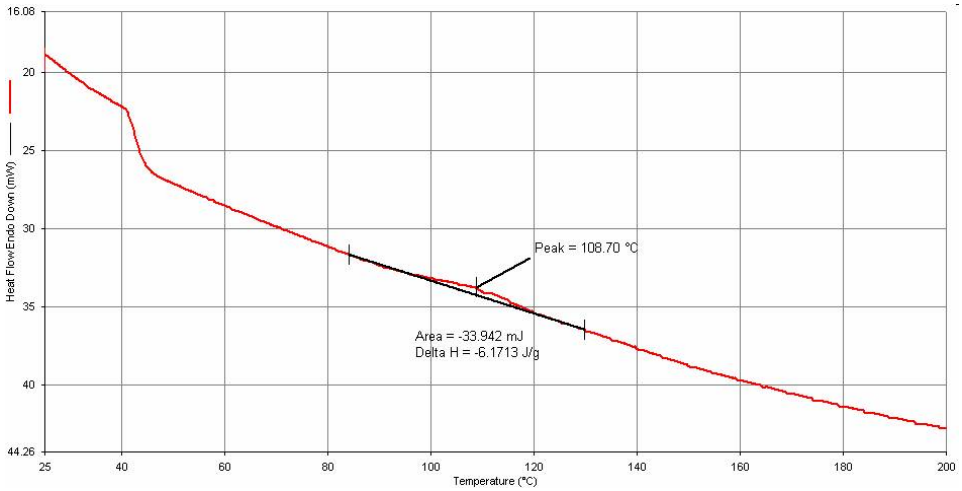

Figure 4: DSC plot 353 ND cured with iCure 3W 30 s

Table 3 shows data obtained from the samples cured with the iCure.

Sample	Power (W)	Curing Time (s)	Residual Exotherm (J/g)	Curing %
B1	3	30	-	100
B2	3	30	-6.17	98.8

Table 3: iCure curing data

When curing the 353ND with the iCure, the Tg's are typically in the 105ºC – 110ºC range which is similar to conventional oven cures as published by adhesive manufacturer.

The iCure cures in a completely different way when compared to conventional ovens. The entire adhesive mass absorbs infrared energy which causes the adhesive to heat up from within. This sets off the thermal catalysts and initiates the curing process which is then completed by the exothermic reaction. In applications outside of this experiment where the substrate also absorbs the optical energy, the curing process can be further accelerated.

Conclusion

The iCure AS200 infrared thermal spot curing system is a powerful system that delivers precise amounts of optical energy where required. Curing profiles can easily be adapted to many types of heat curing materials.
Infrared thermal spot curing offers a fast and efficient alternative to conventional ovens for thermally cured adhesives such as 353ND. Degree of cure and Tg are similar with both curing methods with IR thermal spot curing significantly reducing processing time.

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