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Application News

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DSC-60 Plus/Epoxy/Curing

Epoxy Curing Studies with Isothermal Differential Scanning Calorimetry Method

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❑ Introduction

Epoxy resins are used for many purposes, e.g. coatings, electronic materials and adhesives [1] due to their outstanding mechanical and electrical properties, chemical resistance, adhesion, and minimal shrinkage. Epoxy resin is thermoset and the curing process must be carried out properly since this affects the thermal and mechanical properties of the end-product [2]. The curing of epoxy with a curing agent occurs even at low temperature but is rapid above 100 ºC [3]. The curing reaction is exothermic, thus the reaction temperature and rate can be studied by differential scanning calorimeter (DSC). Depending on the type of epoxy resin and the application of the final product, dynamic or isothermal DSC methods can be used. This application news describes an isothermal DSC method to study the curing reaction of epoxy glue.

❑ Experimental

The epoxy glue was placed in an Aluminium pan which was then transferred to the Shimadzu DSC model DSC-60 Plus furnace. An empty Aluminium pan was used as reference. The sample was heated to 120 or 140 ºC at 10 ºC/min in Nitrogen gas atmosphere (100 mL/min) and hold for 0 min. After the sample has cooled down to room temperature, it was re-heated to 250 ºC. The same experiment was repeated with different hold (curing) times.

❑ Results and Discussion

The first heating cycle represents the exothermic curing reactions at 120 and 140 ºC (Figures 1 and 2). When the curing was carried out at 140 ºC, the third exothermic curing peak at 140 ºC is more distinct than at 120 ºC. This shows that the degree of curing was higher at 140 ºC.

The second heating cycle indicates residual exotherm i.e. the degree of uncured epoxy from the first heating cycle [4]. If no exothermic peaks are seen in the second heating cycle and glass transition is obtained, this indicates that the epoxy is completely cured during the first heating cycle.

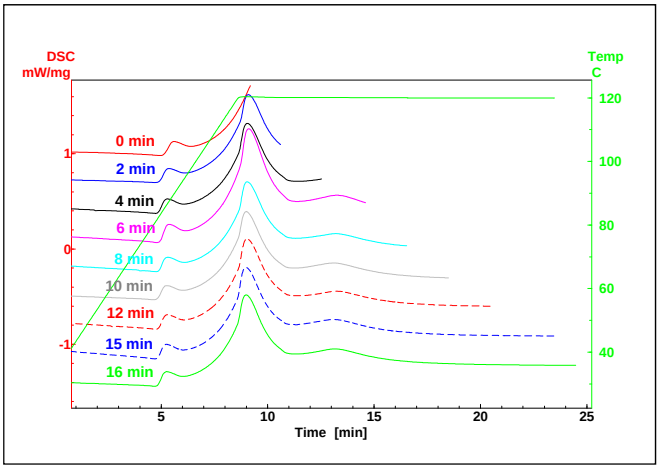

Fig. 1 First heating cycle at 120 ºC

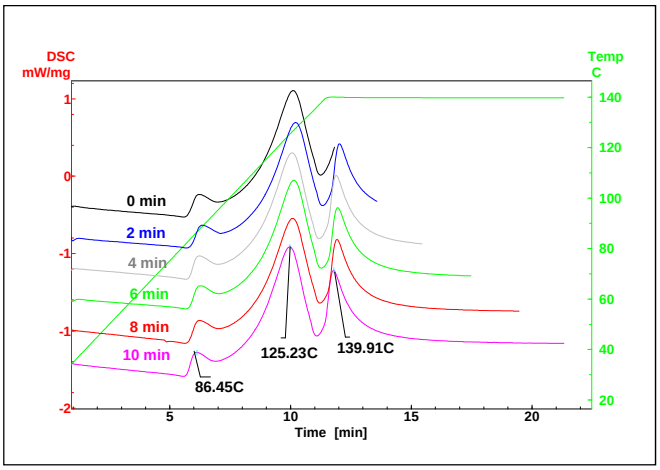

Fig. 2 First heating cycle at 140 ºC

The DSC graphs for the second heating cycle at 120 and 140 ºC are shown in Figures 3 and 4 respectively. With longer curing time, the exothermic peaks became smaller which means higher degree of curing.

When the curing reaction was carried out at 120 ºC, exothermic peaks were still present after 15 minutes, i.e. the epoxy is not yet completely cured. The percentage (degree) of curing can be calculated as in Table 1. The percentage of curing was 97.2 % after 15 minutes which also shows that the epoxy is not completely cured.

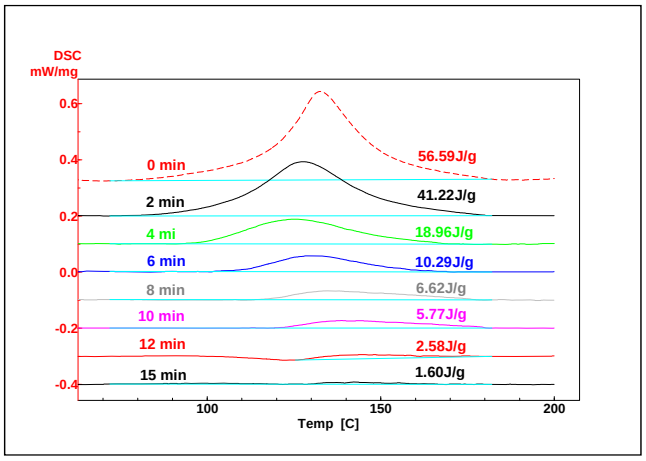

Fig. 3: Second heating cycle at 120 °C

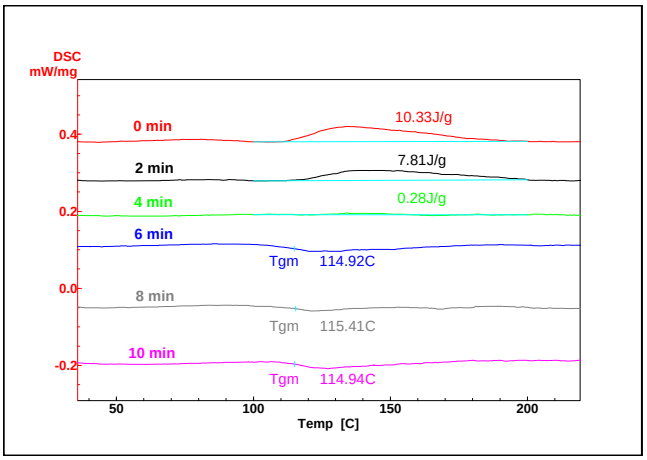

Fig. 4: Second heating cycle at 140 °C

Table 1: Percentage of curing at 120 °C

Curing Time, t (min)	Residue Enthalpy, $RH_{t\ min}$ (J/g)	Enthalpy Used for Curing (J/g)[A]	% Curing[B]
0	56.59	0.00	0.00
2	41.22	15.37	27.2
4	18.96	37.63	66.5
6	10.29	46.30	81.8
8	6.62	49.97	88.3
10	5.77	50.82	89.8
12	2.58	54.01	95.4
15	1.60	54.99	97.2

Notes [5]
A = $(RH_{0\ min})$ – $(RH_{t\ min})$
B = $(A / RH_{0\ min}) \times 100$ %

At 140 °C (Figure 4), there were no exothermic peaks after 6 minutes and glass transition at 115 °C was obtained. This shows the epoxy is completely cured. The results in Table 2 also show that the epoxy is completely cured after 6 minutes at 140 °C.

Table 2: Percentage of curing at 140 °C

Curing Time, t (min)	Residue Enthalpy, $RH_{t\ min}$ (J/g)	Enthalpy Used for Curing (J/g)	% Curing
0	10.33	0.00	0.0
2	7.81	2.52	24.4
4	0.28	10.05	97.3
6	0.00	10.33	100.0
8	0.00	10.33	100.0
10	0.00	10.33	100.0

The data in Tables 1 and 2 was used to plot the graph in Figure 5. It could be seen that the curing of epoxy was faster at 140 °C.

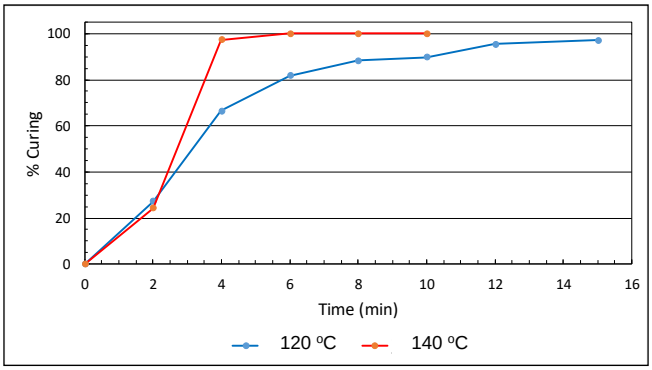

Fig. 5: Curing time at 120 and 140 °C

❑ Conclusion

Isothermal DSC method was used to study the curing of epoxy glue. The epoxy is completely cured if the calculated percentage of curing is 100 %. As the curing progresses, glass transition can also be observed.

❑ References

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