Application Brief

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DSC Measurements of Epoxy Adhesives

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1. Introduction

Epoxy adhesives are often used as adhesives and fillers for materials such as metal, glass, ceramic, and plastic. These thermosetting adhesives are mainly created from epoxy resins and curing agents. During curing, the epoxy group starts a polymerization reaction, cross-links and hardens.

Differential scanning calorimetry (DSC) can investigate various characteristics of epoxy adhesives, including glass transition temperatures before and after curing, as well as the temperature and reaction calories during the curing reaction.

This brief presents a measurement example using commercial two-component epoxy adhesives.

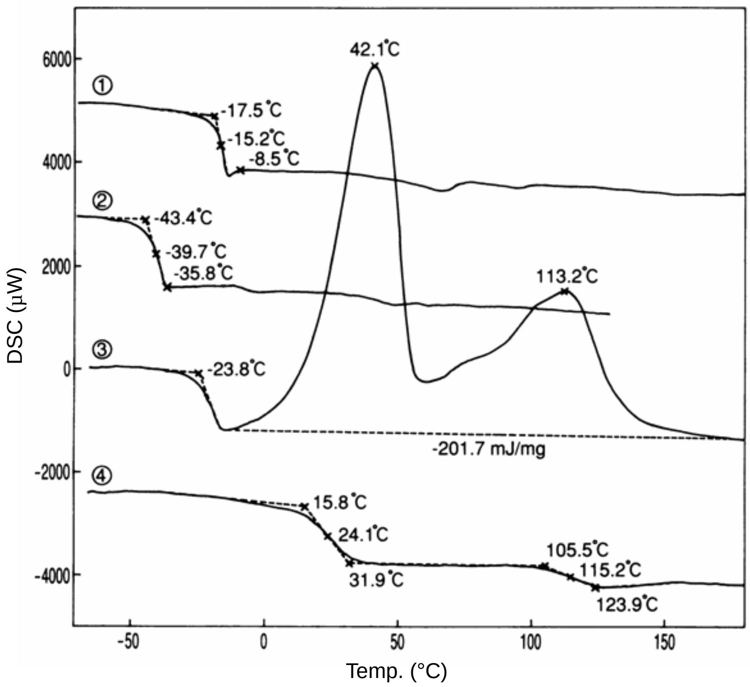

Figure 1 DSC Curves for the 5-minute type adhesive (1)
① Base compound
② Curing agent
③ Base compound and curing agent mixed and left to stand for 5 minutes
④ 2nd heating for ③ sample

2. Measurements

The measurement samples were 5-minute type and 30-minute type two-component epoxy adhesives. Both adhesives are composed of two liquids, a base compound and a curing agent. When the liquids are mixed at 20°C, the 5-minute type starts curing after 5-minutes and the 30-minute type starts curing after 30 minutes.

For the measurements, a DSC220 high sensitivity differential scanning calorimeter was connected to a SSC5200H Disk station.

The sample weight was 10mg and an Al open pan was used. The heating rate was 10°C/min and measurements were performed in a nitrogen atmosphere.

3. Results

Figure 1 shows the DSC curves for the 5-minute type adhesive. Curve ① is the measurement result for the base compound. Glass transition was observed at -17.5°C (the extrapolated glass transition start temperature). Curve ② is the measurement result for the curing agent. Glass transition was observed at -43.4°C. Neither measurement result showed an exothermic curing peak, indicating that neither agent had a curing reaction.

Curve ③ shows the results when the base compound and curing agent were mixed, left to stand for 5 minutes at room temperature and then measured. Glass transition was observed at -23.8°C. Furthermore, exothermic curing peaks were observed around 42°C and 113°C. The results show that exothermic heat started around room temperature and the curing reaction progressed even at room temperature.

Curve ④ shows the results for the 2nd heating, in which the curve ③ sample was reheated under the same conditions. In these results, there were no exothermic curing peaks. Glass transitions of the hardened epoxy resin were observed at 18.5°C and 105.5°C. The fact that glass transition was observed in two locations suggests that this epoxy resin is composed of multiple components.

Figure 2 shows more results for the 5-minute type adhesive. The base compound and curing agent were mixed, left to curing at room temperature for different lengths of time, and then measured. These results show that the longer the curing time at room temperature, the higher the glass transition temperature shifted. At the same time, the shape and temperature of the exothermic curing peaks changed along with the curing time. This likely occurred because the curing reaction progressed farther as the standing time at room temperature lengthened and this changed the degree of polymerization. Furthermore, an exothermic curing peak was seen even when the sample was left to stand for one day, showing that the sample had not completely hardened.

Figure 3 plots the relation of the curing time and glass transition temperature for the measurement results of Figure 2. These results show that the higher the degree of cure, the higher the glass transition temperature. Investigating the glass transition temperature after curing in this way makes it possible to evaluate the appropriateness of the curing time.

Figure 4 shows the 30-minute type adhesive results when the base compound and curing agent were mixed, left to curing at room temperature for different lengths of time, and then measured. As with the measurement results for the 5-minute type adhesive shown in Figure 2, different curing times created differences in the glass transition temperatures and exothermic curing peaks. Furthermore, the results show that the 30-minute type adhesive required more time to harden at room temperature than the 5-minute type adhesive.

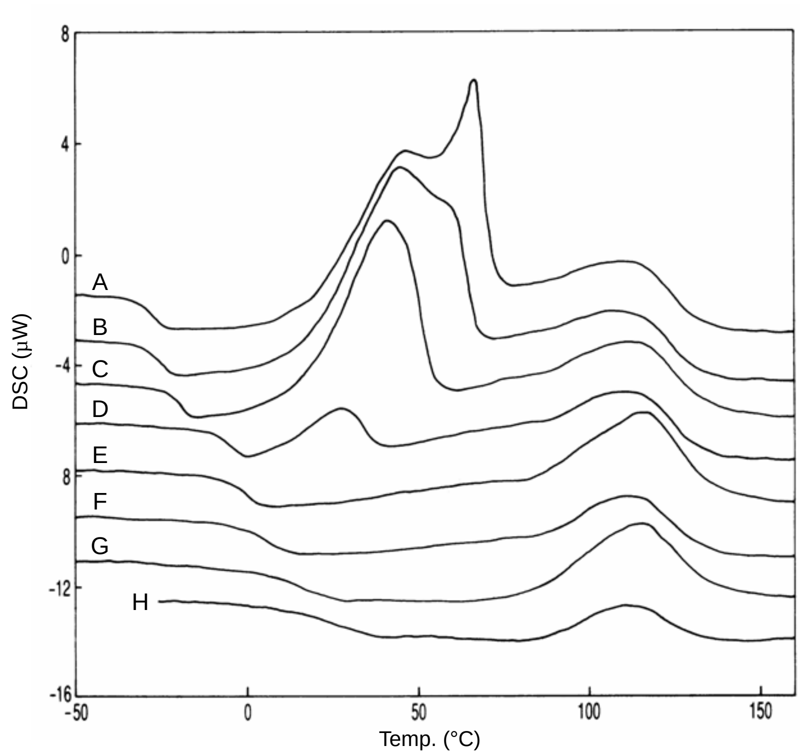

Figure 2 DSC Curves for the 5-minute type adhesive (2)
After Mixed
A : 2min, B : 3min, C : 5min, D : 10min,
E : 15min, F : 30min, G : 4hr, H : 1day

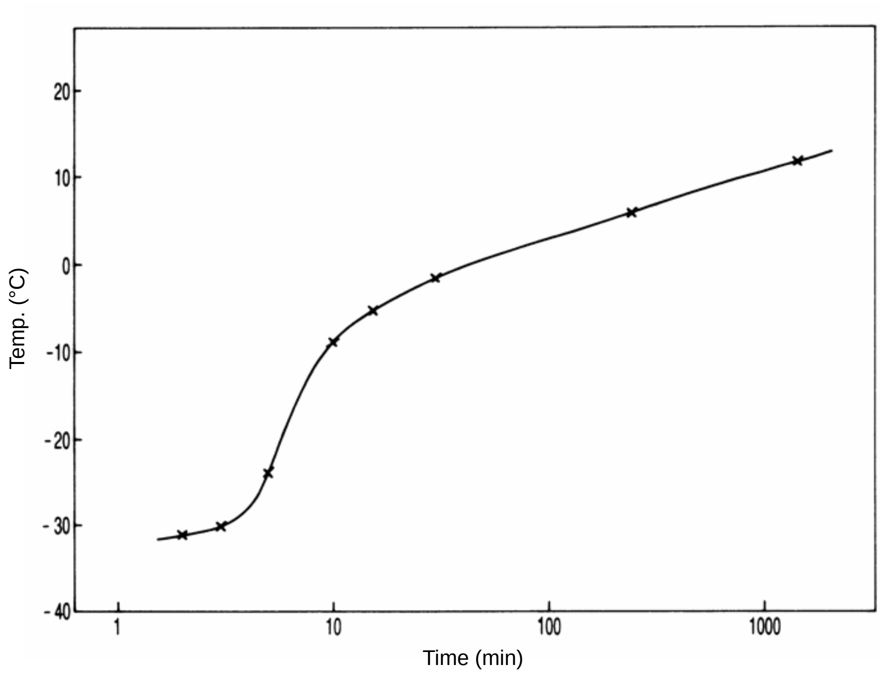

Figure 3 Curing Time and Glass Transition Temperature Relation
for the 5-minute type adhesive

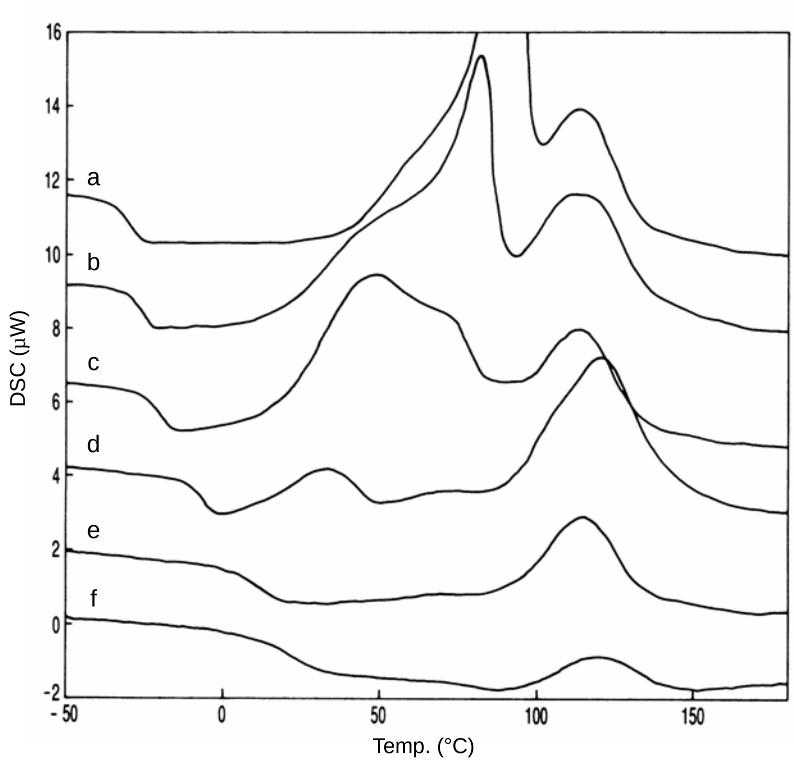

Figure 4 DSC Curves for the 30-minute type adhesive
After Mixed
a : 5min, b : 15min, c : 20min,
d : 30min, e : 1hr, f : 1day

4. Summary

In this brief, DSC was used to measure epoxy adhesive. DSC can analyze the curing process and evaluate hardened material and can also be applied to other thermosetting adhesives and paints.