

DSC Measurements of Thermosetting Resins

1. Introduction

The curing reaction heat and glass transition temperature of thermosetting resins are important to understand the characteristics of the resins. In this brief, epoxy resin and phenol resin (resol) were selected as examples of thermosetting resins and their characteristics were examined utilizing DSC measurement.

Epoxy resin and resol resin cure through the reactions shown in Figure 1. Because resol resin generates water when it cures, measurement of its curing reaction heat often becomes difficult owing to the effects of water evaporation. In order to measure the exothermic curing reactions, a hermetically sealed sample container is required which will suppress the water evaporation.

In this brief, a Hermetically Sealed Sample Container (made of Ag; withstanding up to 50atm.) was used to measure the curing reaction heat of resol resin without effects caused by the evaporation of water.

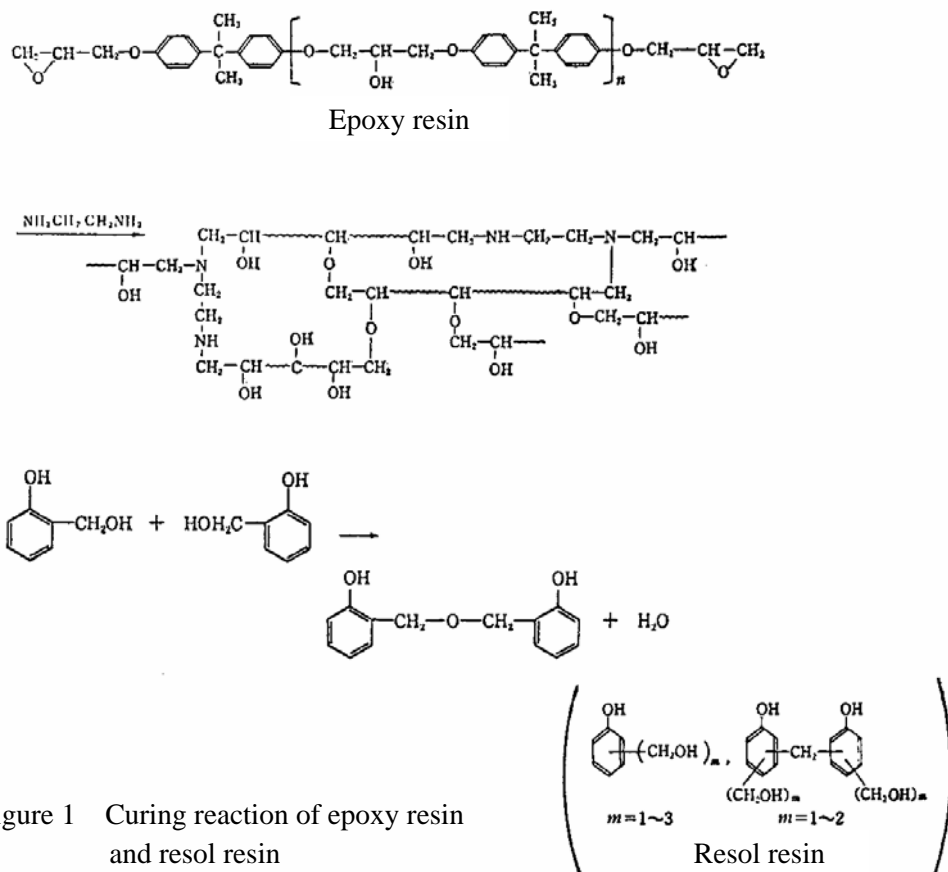


Figure 1 Curing reaction of epoxy resin and resol resin

2. Methods and Data

Figure 2 shows the shifting of the baseline to the endothermic side due to glass transition at 60°C and the exothermic peak due to the curing reaction within the temperature range of 120 to 245°C.

Figure 3 shows glass transition at 90°C, which indicates that the glass transition temperature shifted to the high temperature side due to curing. Contrary to Figure 2, which shows an endothermic peak at glass transition, Figure 3 shows no such peak. This suggests that there is a difference in thermal hysteresis between the two samples. The fact that Figure 3 shows no curing reaction exothermic peak indicates that the sample was completely cured by the heating process shown in Figure 2.

In Figure 4, the curing reaction of the resin is measured displaying a complicated DSC curve. This is because the broad exothermic peak due to the curing reaction was overlapped by the sharp endothermic peak from the evaporation of generated water.

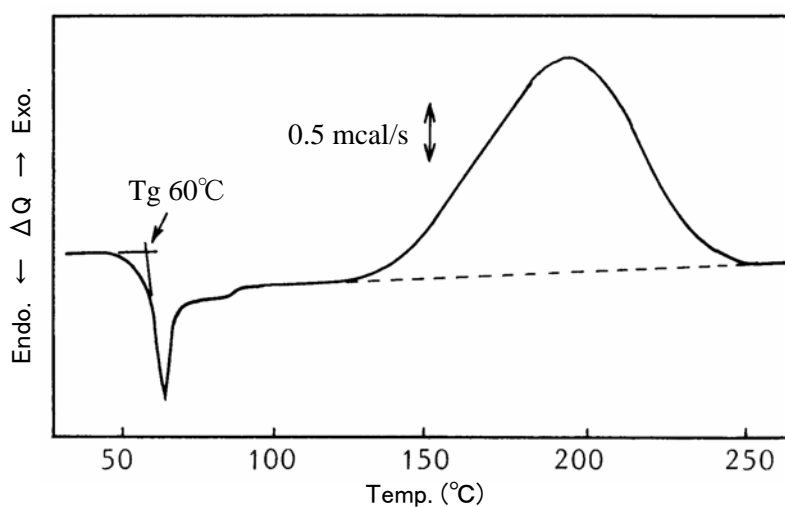


Figure 2 Uncured epoxy resin
Sample weight : 10mg
Heating rate : 20°C/min

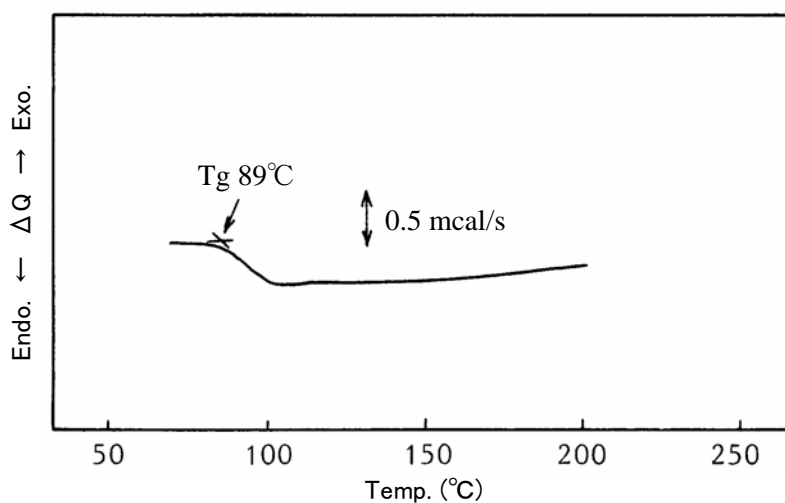


Figure 3 Cured epoxy resin
Sample weight : 10mg
Heating rate : 20°C/min

Figure 5 shows the DSC curve on the resol resin measurement using a sealed sample container. Through the use of a hermetically sealed container, water evaporation is suppressed and the curing reaction heat of resol resin is shown as a single peak within the temperature range of 100 to 240°C.

In Figure 5, an endothermic peak is observed which starts near 250°C. Since water has a vapor pressure of about 50atm. in the vicinity of 250°C, this endothermic peak is considered to have been caused by evaporation of the water after exceeding the sealing limit of the sample container.

3. Conclusion

As shown above, the DSC measurement of thermosetting resins is useful in determining the characteristics of resins. The Hermetically Sealed Sample Container has also made it possible to measure the high temperature curing reaction heat of resol resin, which generates water during the curing reaction.

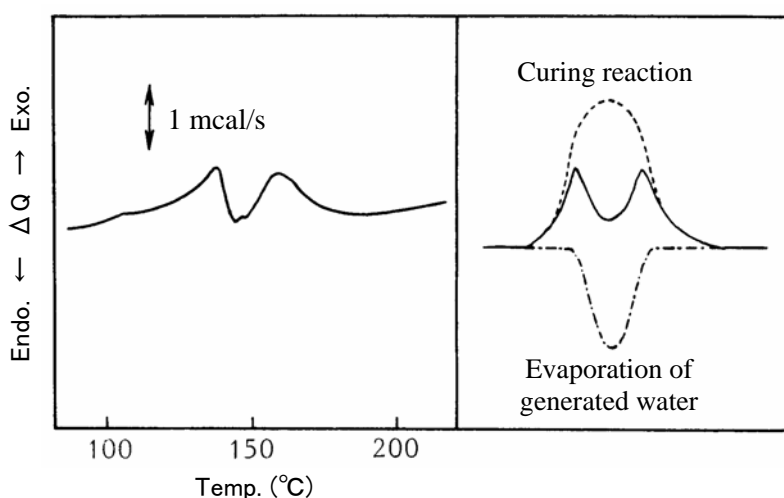


Figure 4 Resol resin (open sample pan)
 Sample weight : 15mg
 Heating rate : 20°C/min

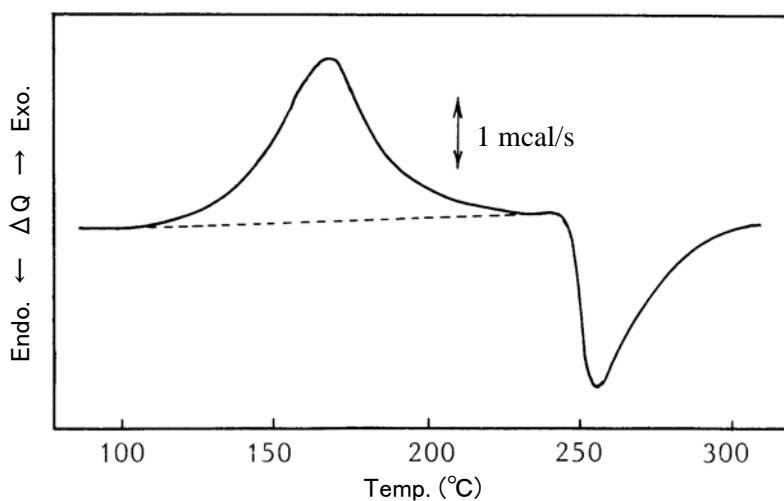


Figure 5 Resol resin (hermetically sealed sample container)
 Sample weight : 15mg
 Heating rate : 20°C/min