Application Brief

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Measurement of Curing Reaction Heat of UV Curing Adhesives

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1. Introduction

Short-time curing type ultraviolet adhesives are used in wide number of fields such as general electronics, optical electronics, medical fields, glass arts, and architecture.

Curing reaction heat when the UV is irradiated can be measured real-time by using Differential Scanning Calorimeter (DSC) and the UV Irradiation Unit. The condition which is necessary for curing can be considered by checking the relationship between irradiation intensity and irradiation time.

Measured sample is the curing type ultraviolet adhesives which is high-viscosity and transparent brown liquid. It is good for bonding plastics. Thus it is used for fixing and sealing of LCD panel, PCB, flexible wiring. Also, this is suitable for the adhesion, the sealing, and the coating of metals and glass parts. It is a single-component adhesive. It is cured by UV and heat.

In this brief, DSC and UV irradiation unit are used to evaluate the necessary heating temperature and the irradiation intensity.

2. Measurements

The sample is a single-component anaerobic 10-second curing type UV adhesive.

2mg sample is spread out on the plate type aluminum sample container as the surface curing is measured. Sample thickness is around 0.3mm.

Measurement tool is DSC7020, high sensitivity differential scanning calorimeter and PDC-7 UV irradiation unit.

Wave length 365nm is chosen using interference wavelength-selective filter. Irradiation time is 10 seconds. Preset temperature is 30°C. Nitrogen atmosphere is prepared.

In case of the heating measurement the followings are set: sample weight 2mg in the aluminum open pan, heating rate is 10°C/min, and N_2 atmosphere.

3. Results

Figure1, 2, and 3 shows 1st and 2nd measurement results with the irradiation intensity 1, 2, and 10mW/cm^2. UV is irradiated for 10seconds from one minute after measurement start. Fluctuation of DCS baseline by irradiation heat is deleted by subtracting the data which is taken again from the enough cured sample.

DSC peak height and amount of exothermic heat of each figure shows the one of 1st irradiation. It shows when the irradiation intensity goes high, the amount of exothermic heat increases. Even exothermic peak at second irradiation at 1, and 2mW/cm^2 can be confirmed; however, the peak at 10mW/cm^2 cannot be observed.

These results show the higher the irradiation intensity goes, the more curing reaction amount increases and the shorter the sample is cured. In order to cure enough with 10 seconds irradiation, the irradiation intensity greater or equal to 10mW/cm^2 is required.

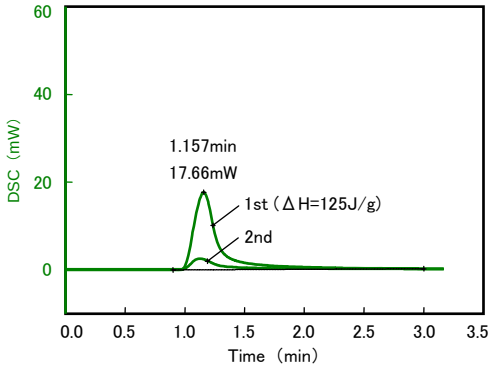

Figure 1 1st and 2nd irradiation measurement results
Irradiation intensity: 1mW/cm^2

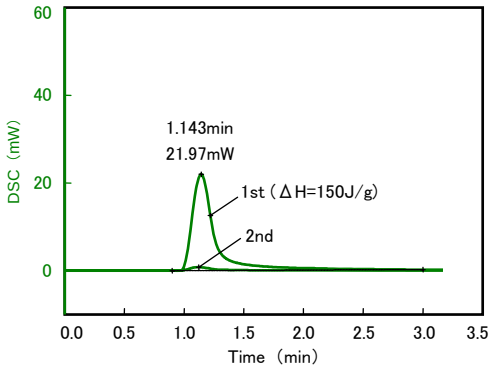

Figure 2 1st and 2nd irradiation measurement results
Irradiation intensity: 2mW/cm^2

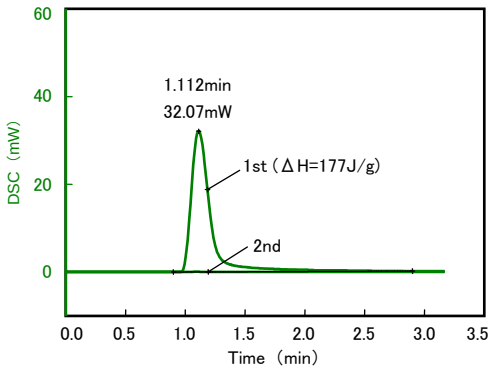

Figure 3 1st and 2nd irradiation measurement results
Irradiation intensity: 10mW/cm^2

Figure 4 shows first irradiation measurement results in case of irradiation intensity 1, 2, 10, 20, 100, and 500mW/cm^2. As the irradiation intensity increases, amount of exothermic heat also tends to increase and curing reaction amount is increased. As the exothermic peak reaches to peak top quick, it shows curing reaction speed is fast.

Figure 5 shows Figure 4 integral curves of exothermic peaks. It shows the sharp reaction rate rises as the irradiation intensity increases.

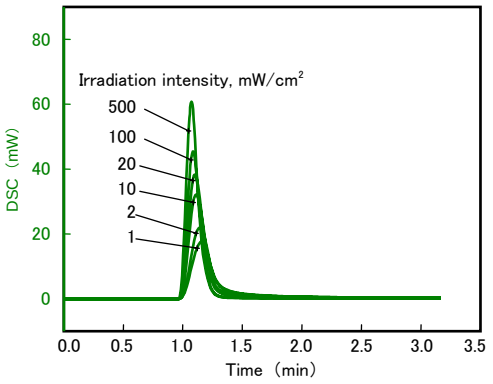

Figure 4 1st irradiation measurement results
Irradiation intensity: 1, 2, 10, 20, 100, and 500mW/cm^2

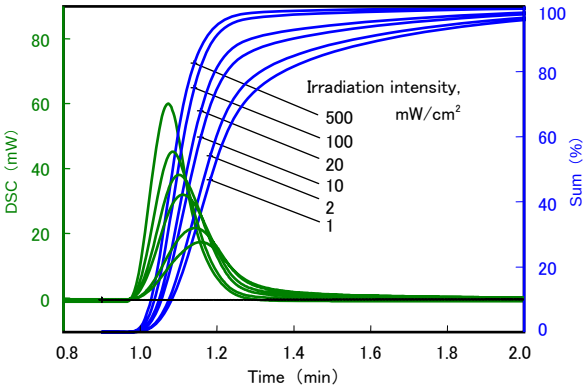

Figure 5 Integral curves of Figure 4 exothermic peaks

Figure 6 shows the heating measurement results after the unirradiated sample and irradiated sample crimped in a aluminum open pan.

The unirradiated sample has a possibility of heat by heat curing reaction at exothermic peak between 119°C and 174°C.

In case of the irradiated sample with irradiation intensity 1mW/cm^2, exothermic peak is shown at 114°C. The amount of exothermic heat of this peak is shown less than 1/6 of unirradiated sample. This indicates uncured component curing reaction by heating.

This adhesive has a possibility that even uncured component remains by the weak UV irradiation; however, further heating can make it curing.

On the other hand at the exothermic peak of high temperature side, the peak becomes smaller a bit by higher intensity irradiation. It is a different reaction than the curing reaction by UV irradiation.

Above shows it is necessary to make the low temperature side exothermic reaction happen for the curing by heat. It is around 120°C.

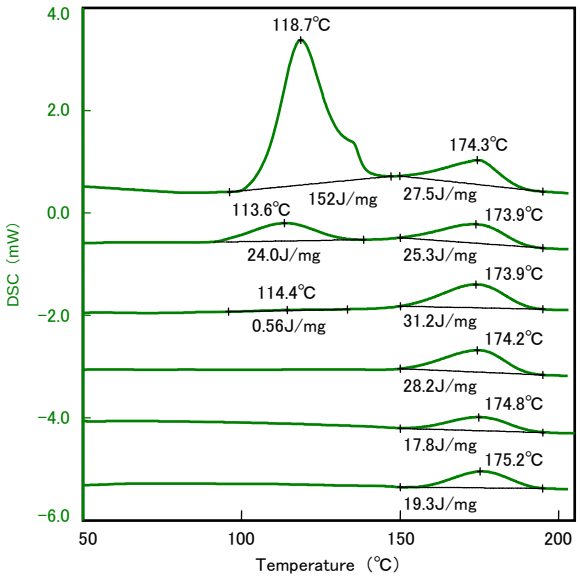

Figure 6 Heating measurement results

4. Summary

The measurement is performed using the DSC7020 high sensitivity differential scanning calorimeter and PDC-7 UV irradiation unit for curing reaction heat of UV curing type adhesives. It can make it clear of curing level by changing UV irradiation intensity.

Also, curing reaction heat can be measured as well as the temperature which is necessary for heat curing.

In conclusion, the combination of the DSC and the UV irradiation unit is very useful for measuring two types of curing reaction by the UV irradiation and the heating.