Application Note B-TA1039

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B-TA1039 - Measurement of glass transition of PMMA by dynamic DSC

Introduction

DSC is one of the analytical methods often used to investigate the glass transition temperature of polymeric materials. Since a glass transition is a change in specific heat capacity, it appears as a baseline shift rather than a peak in the DSC results. This baseline shift can be identified when the glass transition appears alone. In some cases, the endothermic peak due to enthalpy relaxation, the endothermic peak from dehydration, or the exothermic peak from crystallization immediately after the glass transition can overlap, making it difficult to identify the shift due to the glass transition. For these reasons, in normal constant-rate heating DSC measurements, cycle measurements are sometimes performed, and the glass transition is confirmed from the reheating results. In this case, the glass transition was confirmed by dynamic DSC measurements of a PMMA sample.

Measurements and results

The dynamic DSC measurement results of PMMA are shown in Figure 1. The dynamic DSC measurement was performed with a sample weight of 10 mg, a heating rate of 5°C/min, a cycle of 45 s, and an amplitude of 1°C. Figure 2 shows the results of the analysis of the dynamic DSC, which are separated into a total DSC curve, a reversing DSC curve, and a non-reversing DSC curve.

The Total DSC curve in Figure 2 corresponds to the results measured with a constant heating rate. The Total DSC curve shows endothermic peaks at 84°C and 117°C, after which the DSC curve changes in the endothermic direction. This shows that it is difficult to read the baseline shift due to the glass transition even if the sample is measured with a constant heating rate.

Next, the reversing DSC curve in Figure 2 is a component that follows the amplitude in the dynamic DSC results, and is a curve that represents the change in the heat capacity of the sample. Since the glass transition is a change in specific heat capacity, it appears in the reversing DSC curve. In this result, a baseline shift is clearly observed at 111°C, and it is clear that the glass transition of PMMA occurs at 111°C.

The non-reversing DSC curve in Figure 2 is a component that does not follow the amplitude in the dynamic DSC results. Endothermic peaks due to dehydration and enthalpy relaxation appear in this non-reversing DSC curve. This result shows endothermic peaks are observed at 84°C and 115°C with additional changes in the endothermic direction occurring afterward.

When measuring at a constant heating rate, the reversing DSC curve and non-reversing DSC curve in Figure 2 are observed together, making it difficult to confirm the glass transition. However, when performing a dynamic DSC measurement, only the change in the glass transition appears in the reversing DSC curve, allowing for a clear identification of the glass transition temperature.

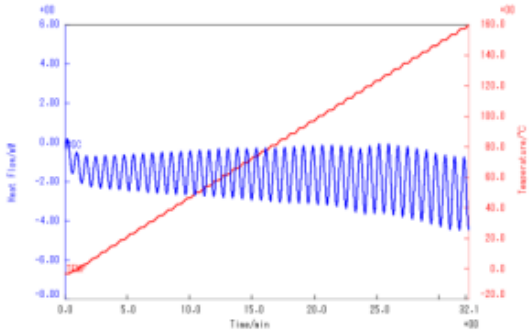

Figure 1: Dynamic DSC measurement result

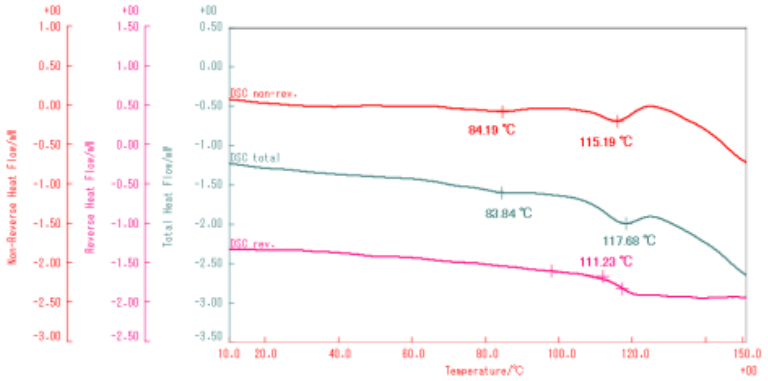

Figure 2: Dynamic DSC analysis result

Recommended equipment and software:

- DSCvesta (Refrigerated cooling unit)
- Dynamic DSC software
- Measurement and analysis software Vullios

Related products

DSCvesta

DSC is a thermal analysis technique that quantifies the amo unt of energy in a reaction.