Detecting Weak Thermal Effects by Means of Differential Scanning Calorimetry

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Introduction

Differential scanning calorimetry (DSC) is a popular method to characterize thermal effects as melting, crystallization or glass transition and many more.

The DSC signal is proportional to the specific heat capacity of the material being measured, and also to the heating rate and the sample mass. The specific heat capacity is a material size and can therefore not be changed. The glass transition is a change in specific heat capacity and can typically be detected as a small effect in the DSC curve. A possibility to magnify this or other small effects is to increase the sample mass. If the amount of material available is limited, using a sensitive DSC – a stable baseline proveided – allows for detecting small effects even with only a small sample quantity.

Measurement Condfitions

In the following, the glass transition of polystyrene with different sample masses is determined by means of DSC. Table 1 summarizes the measurement conditions.

Table 1 Measurement conditions

Device	DSC 300 *Caliris*® with H-Module			
Sample mass	10.38 mg	1.07 mg	131 µg	80 µg
Crucible	*Concavus*® (aluminum, closed with pierced lid)			
Temperature range	25°C to 180°C			
Heating rate	10 K/min			
Atmosphere	Nitrogen (20 ml/min)			

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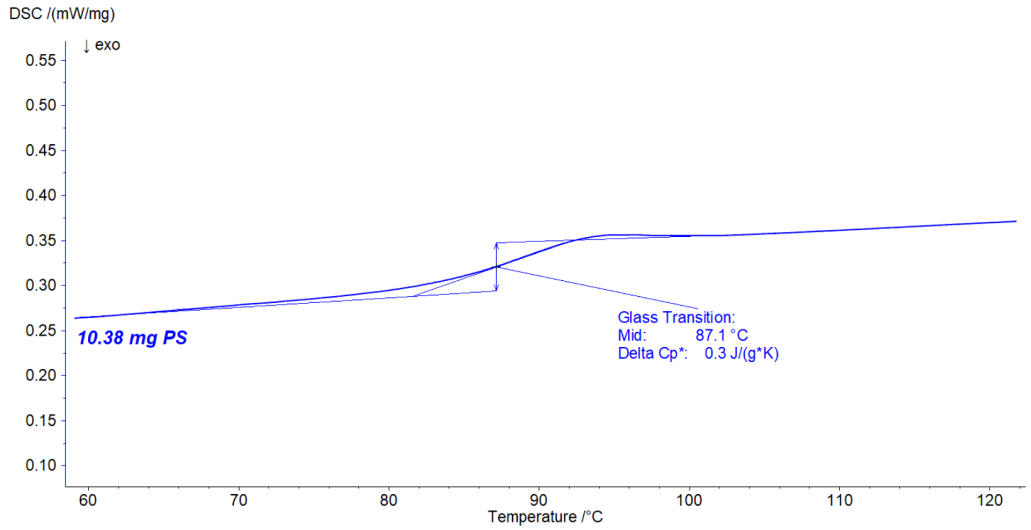
Measurement Results

Figure 1 depicts the DSC resulting curve of the 10.38-mg sample. The glass transition is detected as an endothermal step at 87.1°C (midpoint). It is related to a change in specific heat capacity of 0.3 J/(g·K).

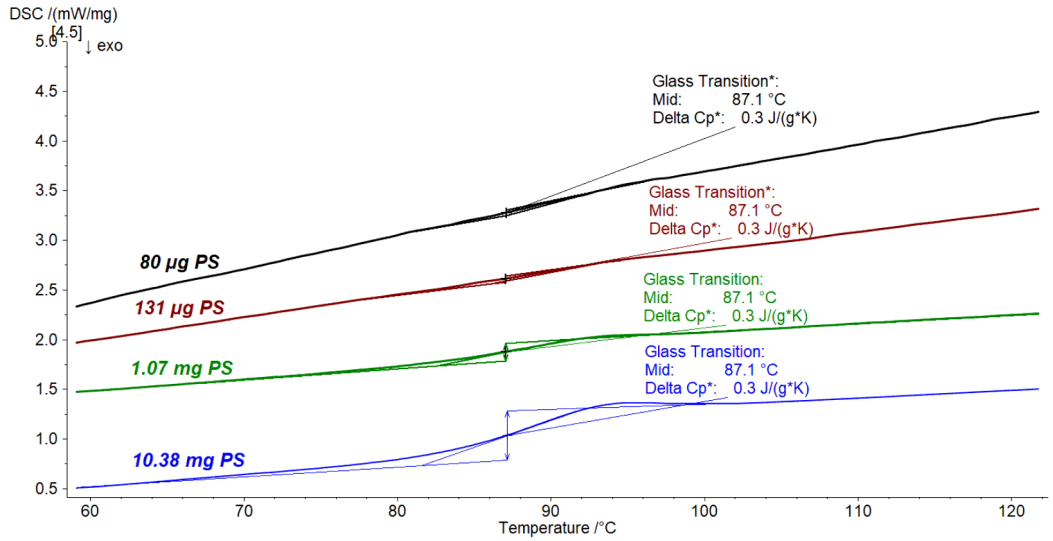
Figure 2 compares the previous measurement (blue curve) to the tests performed on lower masses. The lower the sample mass, the smaller the glass transition appears. However, it results, of course, in the same specific heat capacity change because this one is not depending on the mass.

Summary

The sensitive sensor of the DSC 300 *Caliris*® allows for detection of small effects even for small sample masses. Due to the larger sample mass, the glass transition effect in the DSC curve can be better recognized higher. This improves evaluation and precision of the glass transition temperature.



1 DSC curve during heating. The step-line change in the DSC curve represents the glass transition of the polymer.



2 Determination of the glass transition of polystyrene for different sample masses

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