

The DSC 214 *Polyma*: Ideal for Temperature-Modulated Measurements

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Introduction

Modulated DSC measurements are used to separate overlapping effects. The sample is subjected not only to linear heating rate, but also to sinusoidal temperature variations. This method leads to separation of the so-called reversing and non-reversing part of the heat flow. The reversing effects are a function of temperature and oscillate with temperature variations. The non-reversing processes are a function of time and are calculated as the difference between the total heat flow and the reversing heat flow.

A modulated measurement contains three parameters to be chosen by the user:

- the underlying heating rate
- the amplitude (in K)
- the period of oscillation (in s)

An appropriate heating rate and a sufficient frequency are necessary to ensure that the effects to be separated contain enough oscillations for an improved separation of the effects. This is a required condition for achieving good separation of the reversing and non-reversing processes. Because it is difficult for a heat-flow DSC to follow fast heating rates along with short oscillations, modulated measurements are usually carried out at heating rates of less than or equal to 5 K/min.

Thanks to the low thermal mass of the furnace, the heat-flow DSC 214 *Polyma* is able to modulate at heating rates of 10 K/min in combination with short periods and high amplitudes for results that are both quickly achieved and accurate.

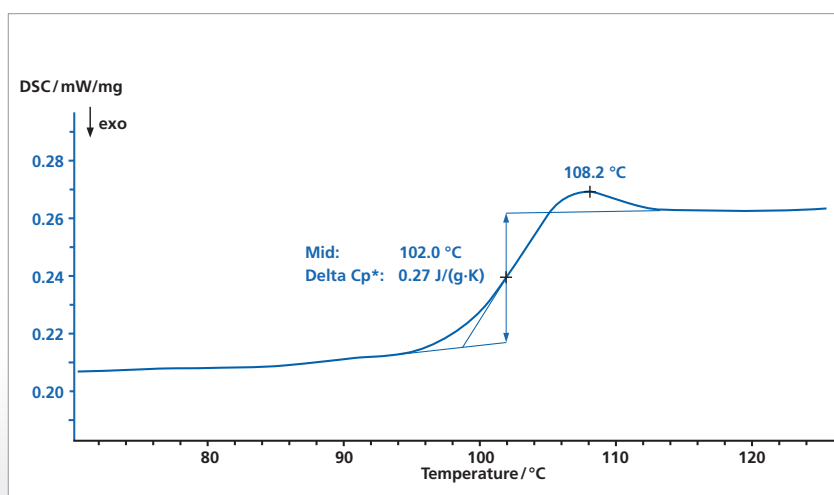
Test Conditions

A polystyrene sample was prepared in a *Concavus* pan and measured with the DSC 214 *Polyma*. This polymer was heated to 150°C at 10 K/min. Oscillations with a 20 s period and 1 K amplitude were used as modulation parameters.

Only a small amount of the polymer (2.36 mg) was used, in order to ensure a homogeneous temperature distribution within the sample despite the fast oscillations and high amplitude.

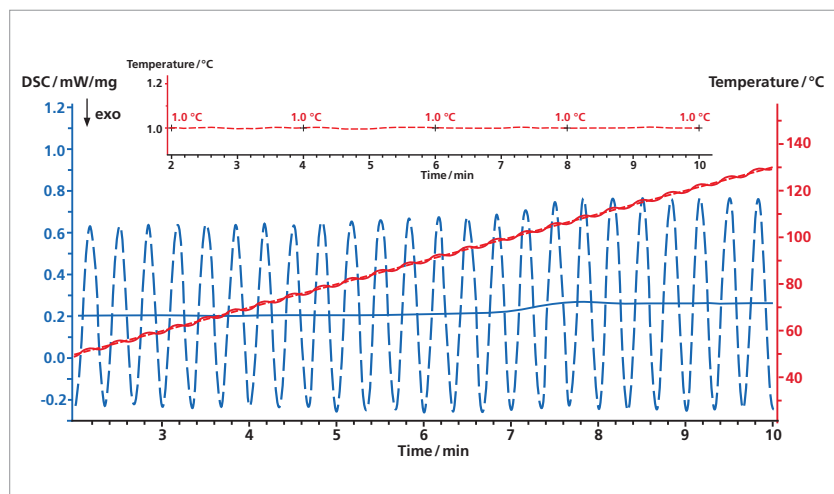
Test Results

The total measured heat flow (which conforms to a conventional DSC curve) is displayed in figure 1. The endothermic step detected at 102°C (midpoint) is due to the glass transition of polystyrene. It is overlapped with a relaxation peak at 108°C resulting from the release of mechanical tension within the sample. The two effects can only be

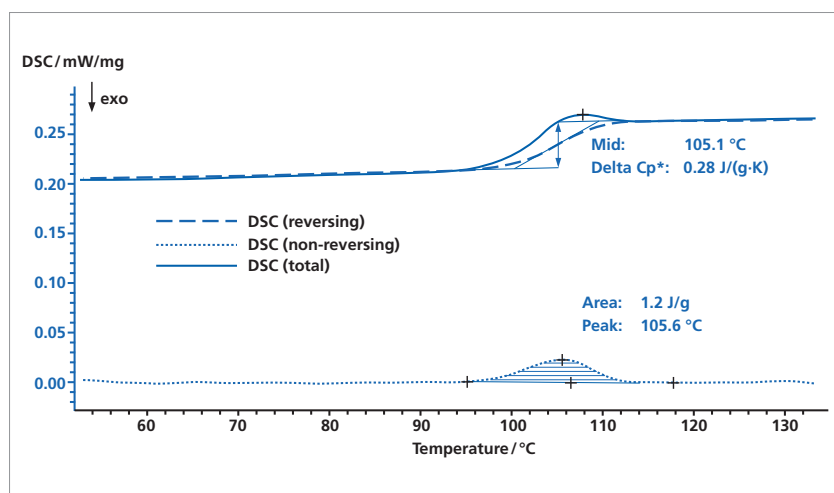


1 Total heat-flow DSC signal

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2 Raw (dashed lines) and average (continuous) signals of temperature and DSC during modulated measurement. The insert shows the excellent stability of the temperature amplitude over the entire temperature range.



3 Separation of the entire heat flow into the reversing and non-reversing signals

evaluated if they are separated. This can be achieved using temperature modulation.

Figure 2 shows that the temperature is perfectly controlled during the modulated measurement: the underlying heating rate of 10 K/min as well as the amplitude of 1 K are both maintained without any difficulty.

Separation of the total heat flow into reversing and non-reversing signals is shown in figure 3. The glass transition occurs in the reversing part of the heat flow whereas the irreversible relaxation peak is a typical non-reversing effect. Both effects can now be correctly evaluated: the

glass transition was detected at 105.1°C (midpoint) and the relaxation peak at 105.6°C (peak temperature) with an enthalpy of 1.2 J/g.

Conclusion

Thanks to modulation, only a few minutes are required to accurately evaluate the glass transition of polystyrene. The DSC 214 *Polyma* combines the robustness of a heat-flow DSC and the advantages of a fast, well-controlled furnace even allowing for temperature-modulated DSC measurements at high heating rates.