

APPLICATION SHEET

Polymers – DSC 214 Polyma

High Sensitivity or High Resolution? – The Choice of the Heating Rate

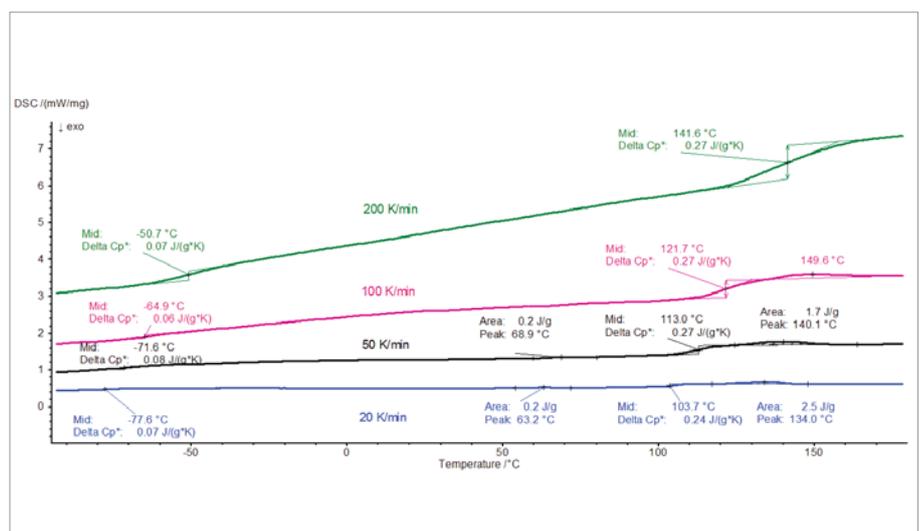
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Introduction

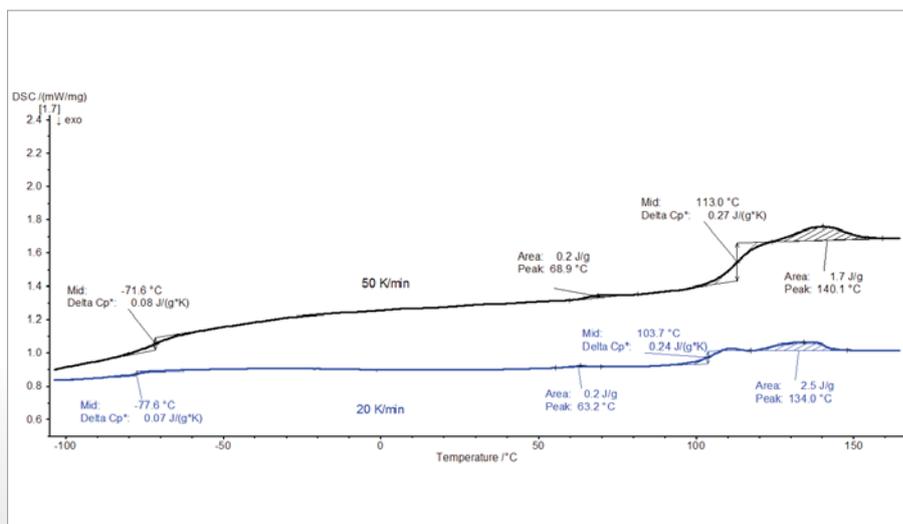
ABS is an amorphous polymer made of acrylonitrile, butadiene and styrene. Its properties, especially its impact resistance, are a function of the proportions and distribution of butadiene used in relation with acrylonitrile and styrene.

Test Conditions

An ABS sample (mass: 11.54 mg) was heated from -170°C up to 200°C at different heating rates (20, 50, 100 and 200 K/min). Between the



1 DSC curves of ABS during heating at 20, 50, 100 and 200 K/min



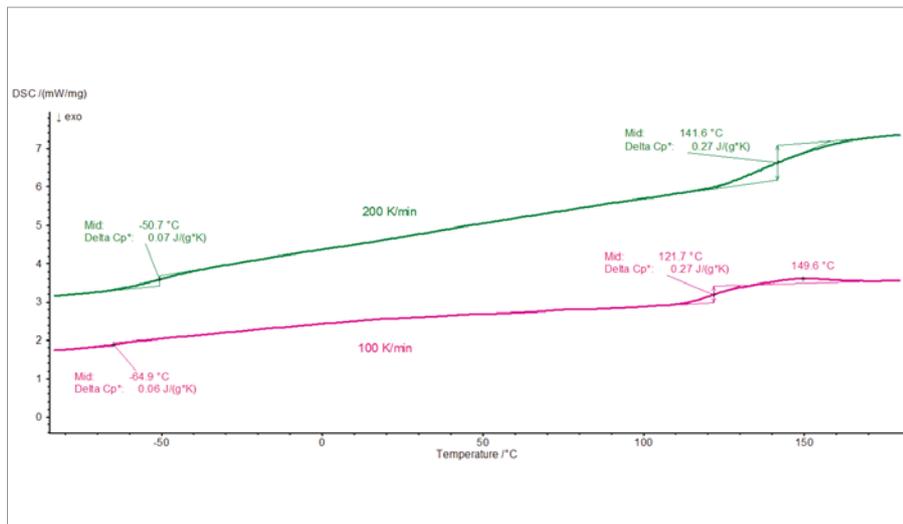
2 DSC curves of ABS during heating at 20 and 50 K/min

heating runs, it was controlled at 20 K/min, so that the polymer had exactly the same thermal history prior to each heating segment. This was to ensure that the differences detected in the heating segments would be attributable only to the heating rates.

Test Results

The DSC curves of the four heating segments are displayed in figure 1. Figures 2 and 3 show the heating runs at 20 and 50 K/min and those at 100 and 200 K/min, respectively.

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3 DSC curves of ABS during heating at 100 and 200 K/min

The endothermic step detected at -77.6°C (mid-point) during the heating at 20 K/min (blue curve) is typical for the glass transition of polybutadiene. A second glass transition at 103.7°C (midpoint) most probably results from an SAN part. Furthermore, the two peaks detected at 63.2°C and 134.0°C (peak temperature) are due to the melting of an additive. The temperature of this second peak is typical for the melting of HDPE, which is often used as a master batch in ABS products.

The heating rate has a great influence on the glass transition temperature: it is shifted higher as heating rates increase, e.g., from -77.6°C at 20 K/min to -50.7°C at 200 K/min for the glass transition of the polybutadiene part.

Furthermore, an increase in the heating rate leads to amplification of the thermal effect: the glass transition step looks larger. This is due to the fact that the heat flow is a function of the heating rate:

$$Q = m \cdot c_p \cdot \text{HR}$$

(Q: heat flow, m: sample mass,
 c_p : specific heat capacity,
 HR: heating rate)

However, it can also be seen from the curves that the peaks found in the measurements at 20 and 50 K/min were no longer detected in the heating segments at 100 and 200 K/min. The heating rate influences the resolution: lower heating rates improve the separation of neighboring thermal effects.

Conclusion

Typical heating rates used in polymer characterization are between 10 and 20 K/min. When investigating amorphous polymers, an increase in the heating rate improves the detection of their glass transition. It should be kept in mind, however, that other thermal effects such as small melting peaks from, for example, master batches or other additives can fade away. Slow heating rates can be used to improve the resolution; high heating rates to improve the sensitivity.