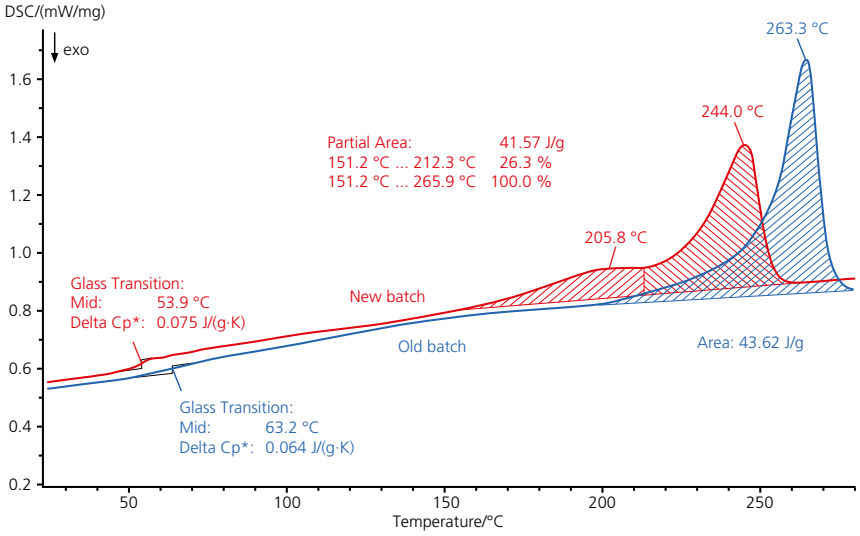
Applications

THERMOPLASTICS/RECYCLING

Incoming Goods Inspection

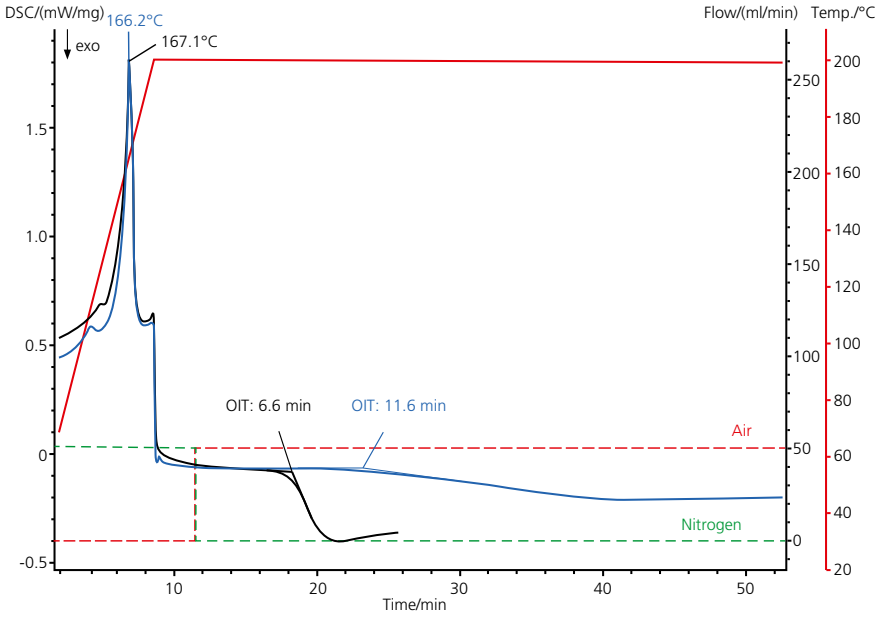
The plot shows the DSC results for two seemingly identical granulate batches, specified as Polyamide 66, which were delivered at different times (2nd heating after controlled cooling at 20 K/min). The blue curve (old batch) shows the glass transition at 63°C (mid-point) and the melting peak at 263°C, which are both typical for PA66. The new batch (red curve), however, exhibits a double peak with peak temperatures at 206°C and 244°C. This indicates that the new granulate most probably contains a second polymer which blends with PA66.



Comparison of two PA66 batches. Sample masses: 11.96 mg (blue) and 11.85 mg (red); heating to 330°C at 20 K/min after cooling at 20 K/min, dynamic N_2 atmosphere.

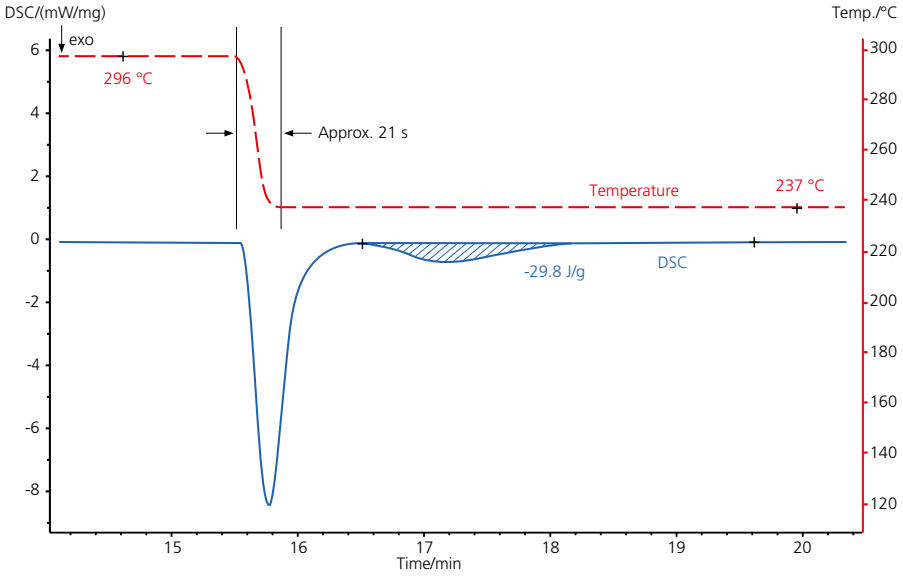
Oxidative Stability

OIT tests (oxidative-induction time) are well-known for evaluating the resistance to oxygen of polymers, in particular polyolefins. In this example, two PP samples were heated to 200°C under a dynamic nitrogen atmosphere. The endothermic peaks detected during heating illustrate the melting of the polypropylene. After 3 minutes at 200°C, the gas was switched to air. The resulting exothermic effects indicate the polymer degradation. In the present case, oxidation occurs earlier for sample A than for sample B (OIT 6.6 min vs. 11.6 min).



OIT test on PP. Sample masses: 9.48 mg (sample A) and 9.55 mg (sample B); heating to 200°C at 20 K/min under N_2 (50 ml/min), 3 min isothermal under N_2, isothermal under air (50 ml/min) until degradation.

DSC 214 Polyma –
Ideal for Quality Control of Polymers



Isothermal crystallization of a semi-crystalline thermoplastic. 11.4 mg PA66 GF30 in a dynamic nitrogen atmosphere, intracooler for the temperature range -70°C to 600°C. The temperature curve is marked in red; the DSC curve in blue. The total crystallization enthalpy at 237°C amounts to approx. 30 J/g. Important for isothermal crystallization experiments is to avoid any temperature-undershoot while changing over from cooling to the isothermal phase.

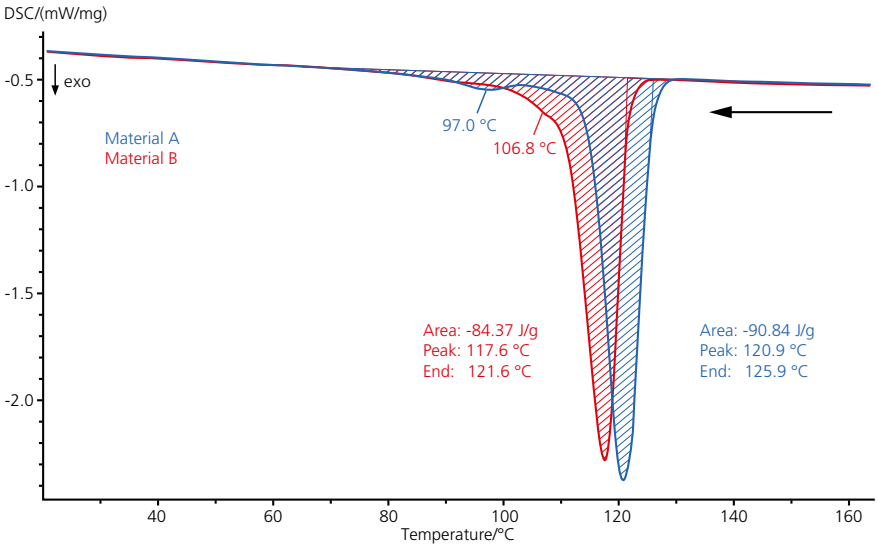
Isothermal Crystallization of a Semi-Crystalline Thermoplastic

Isothermal crystallization tests are often used to simulate the rapid cooling of polymer parts during production (e.g., injection molding). The graph on the left depicts an isothermal crystallization experiment on PA66 GF30 (containing 30 wt% glass fiber) using the DSC 214 *Polyma* in combination with the IC70 intracooler. The low thermal mass of the *Arena*® furnace allows for a temperature interval of almost 60 K to be bridged within seconds. Based on this, it is possible to separate solidification of PA66 from the starting phase of the isothermal segment. This clearly demonstrates the superior cooling performance of the heat-flux DSC 214 *Polyma*.

Failure Analysis – Influence of Recycled Material

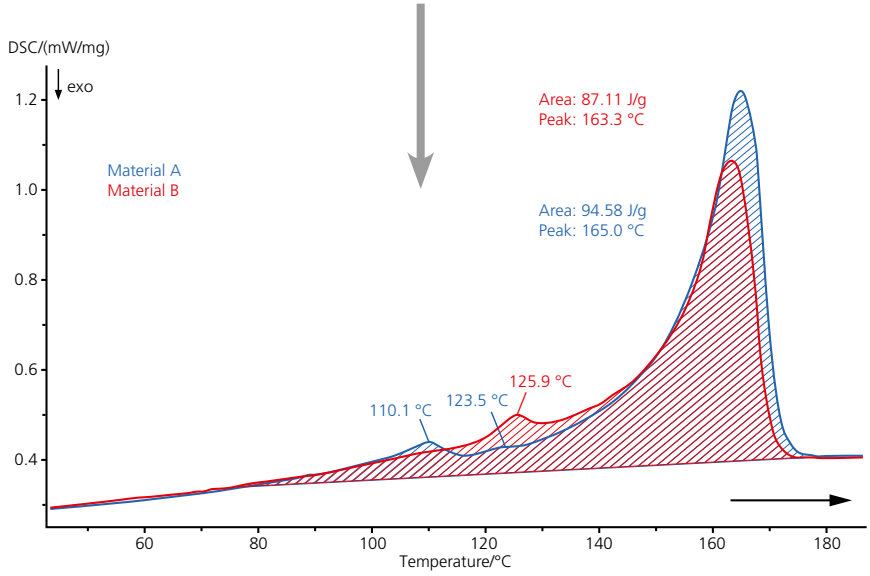
In this example, two recycled polypropylenes were being used for injection molding. Material A was completely crystallized after the molding process whereas material B was still molten. To discover the reason for the differing behavior, DSC measurements were performed.

The exothermic peaks appearing during cooling can be attributed to crystallization of the polymer. Recycled material A starts to crystallize at a higher temperature (endset temperature at 126°C, blue curve) than the second material (endset temperature at only 122°C, red curve). Furthermore, in addition to the peaks at 121°C (blue curve) and 118°C (red curve), a peak at 97°C (blue curve) and a shoulder at 107°C (red curve) occur – clear indications for the presence of a second component. The additional components in material A cause earlier nucleation.



Different solidification of two recycled PP samples. Sample mass: approx. 13 mg; cooling at 10 K/min after heating to 200°C; dynamic N_2 atmosphere.

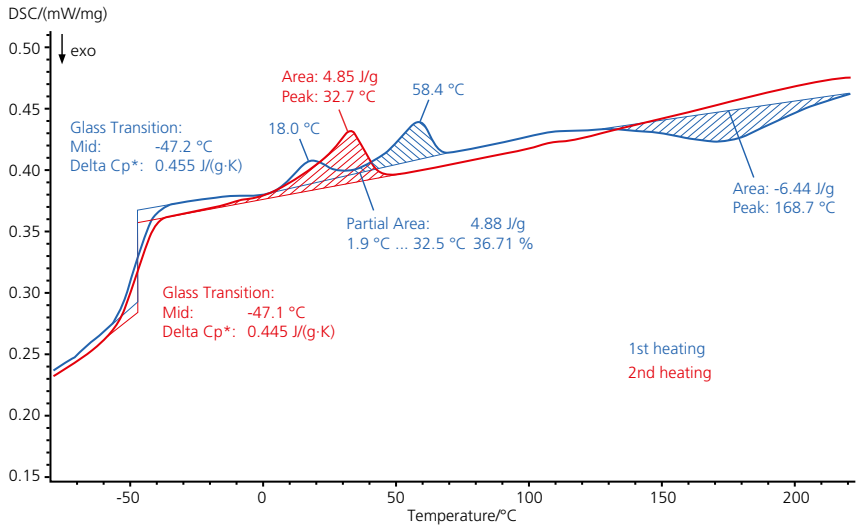
The 2nd heating reveals further information. Besides the peaks at 165°C and 163°C, which are typical for the melting of polypropylene, the blue curve exhibits two additional peaks at 110°C and 124°C, indicating the existence of additional LDPE, LLDPE or HDPE. In contrast with this, material B has only one further peak at 126°C.



Melting of recycled PP with different PE contamination.
Sample mass: approx. 13 mg; heating to 200°C at 10 K/min after cooling at 10 K/min; dynamic N_2 atmosphere.

An Essential Aid for Process Optimization

RUBBER



Thermal behavior of SBR rubber. Sample mass: 15.41 mg; heating from -100°C to 220°C at 10 K/min, twice; dynamic N_2 atmosphere.

Low-Temperature Performance of Rubber

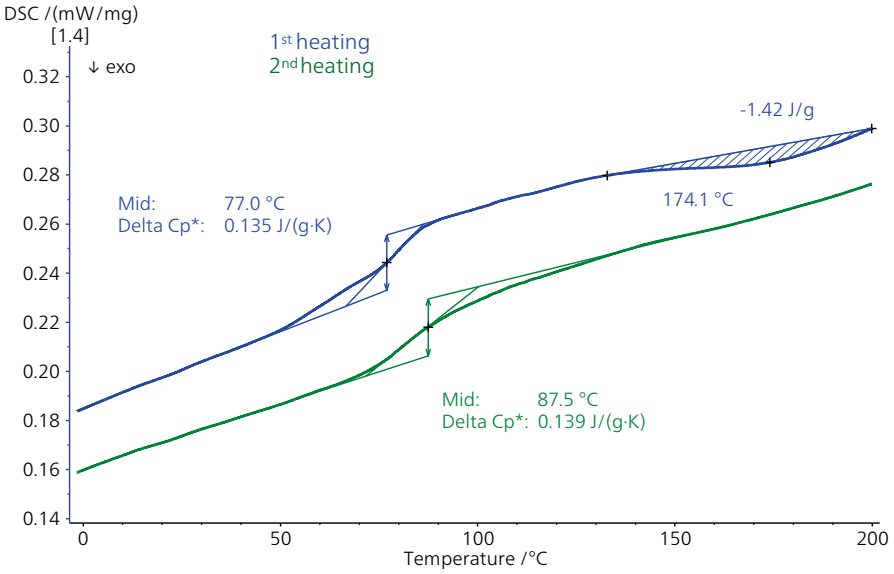
DSC measurements are important for rubbers used in tires because their service temperature range is limited by the glass transition temperature. In this example, an SBR sample was measured twice between -100°C and 220°C. The endothermic step detected at -47°C (mid-point) in both heating sequences is associated with the glass transition of SBR. Between 0°C and 70°C, endothermic effects are detected. They are most probably caused by the melting of additives. The exothermic peak at 169°C (peak temperature), exhibited only in the 1st heating, is due to post-vulcanization of the elastomer.

THERMOSETS

Epoxy Resin (EP)

As an amorphous polymer, this epoxy resin exhibits a glass transition at 77°C (midpoint) with a specific heat capacity of 0.14 J/(g·K) in the 1st heating (blue) followed by an exothermal effect (peak temperature 174°C), due to post-curing of the resin. As a result of the post-curing, the glass transition temperature in the 2nd heating (green) is shifted to 88°C (midpoint). The step height remains nearly the same. Since no further exothermal effect occurs, it can be assumed that the epoxy resin was entirely cured during the 1st heating.

Both the exothermal effect and the position (and shift) of the glass transition temperature to higher values can be interpreted as an evidence for the degree of curing of the material.



Epoxy resins (EP) undergo a polyaddition cross-linking reaction. The properties of the resin are strongly dependent on the structure, the degree of cross linking, type and amount of the reinforcement material and the processing procedure.

Sophisticated Measurement and Analysis