Simulation of the Crystallization Behavior During Injection Molding by Means of Heat-Flux DSC: Isothermal Crystallization Tests

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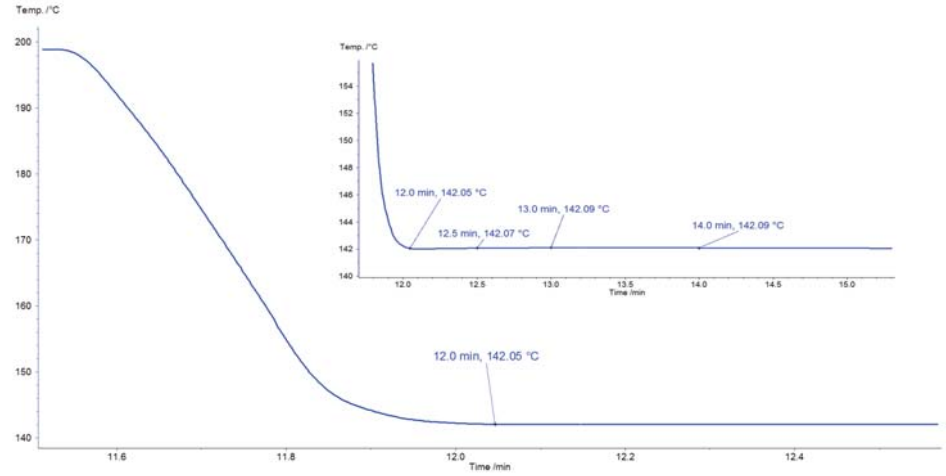
Introduction

Injection molding is the major process in the polymer industry to produce parts of a defined shape. The molten polymer is injected into a relatively cold mold cavity where it is rapidly cooled. The temperature of the mold directly influences the properties of the final product, so it has to be perfectly defined. To this end, the use of a DSC for isothermal crystallization tests, where the behavior of a polymer in the mold is simulated, is a real gain in time.



1 Temperature profile of the cooling to 142°C

Fast Cooling and Stabilization

For isothermal crystallization tests, a DSC must fulfill two requirements. The sample must be cooled very quickly to prevent crystallization from starting during cooling. In addition, the temperature must be stabilized at the specified crystallization temperature without any under- or overshooting. Particularly a temperature undershot can lead to a premature start of crystallization. Some polymers such as polyolefins crystallize very fast. Only a few seconds at a temperature slightly below the target temperature can start crystallization unintentionally.

Until now, it was only possible to achieve the high cooling rates required for isothermal crystallization measurements by means of power-compensated DSCs, because of their small furnaces. The NETZSCH DSC 214 *Polyma* is the first heat-flux DSC that can achieve very fast heating and cooling rates as well as excellent temperature control during isothermal segments, thanks to the low thermal mass of its *Arena* furnace.

Isothermal Crystallization of Polypropylene

In this example, isothermal crystallization tests were carried out on a polypropylene sample with the NETZSCH DSC 214 *Polyma*. Proper regulation parameters were used in order to optimize the transition from the fast cooling to the isothermal segment.

The 6.57-mg sample was heated at 20 K/min to above the melting temperature. After a 3-minute isotherm, the sample was cooled to 142°C, 140°C and 138°C at programmed cooling rates of 200 K/min. The entire temperature program was carried out in a nitrogen atmosphere.

The temperature profile of the cooling to 142°C (figure 1) shows the excellent stability of the temperature during the isothermal segment after the targeted crystallization temperature was reached. The temperature remains constant with a deviation of only 0.1 K.

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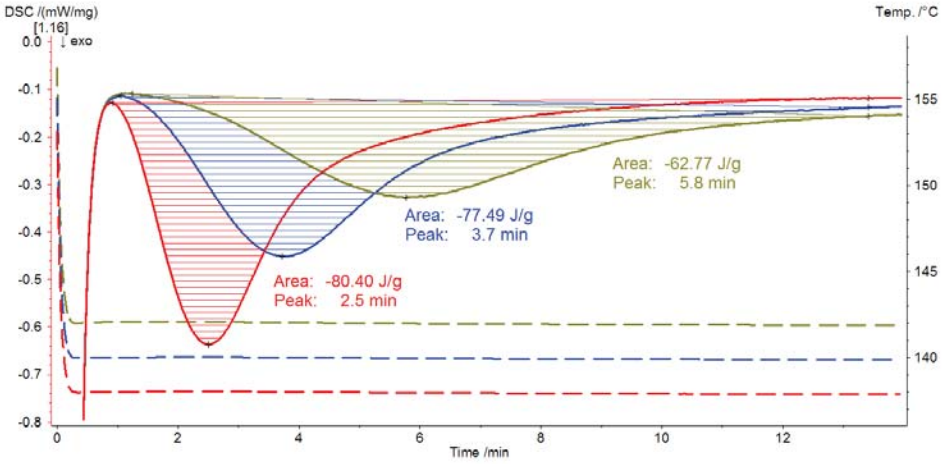

2 Isothermal crystallization of polypropylene at 3 different temperatures with the DSC 214 *Polyma* in a nitrogen atmosphere

The resulting DSC curves for the isothermal segments at 138°C, 140°C and 142°C are presented in figure 2.

Due to the fast stabilization of the temperature at the specified value, the initial effect on the DSC curve caused by the segment change from cooling to isothermal is short enough to allow separation from the thermal effects occurring at its beginning.

The exothermal peak detected during the isothermal segment of the three measurements can be attributed to the crystallization of polypropylene. As expected, the crystallization enthalpy (peak area) increases as the temperature of the isothermal segment decreases, indicating a higher degree of crystallinity in the final product. Also, the slope of the peak is steeper with decreasing isothermal temperature, so the peak minimum is reached faster. This signifies a faster crystallization.

DSC Tests Accompanying Production Save Time and Money

Isothermal crystallization tests can be carried out with the NETZSCH DSC 214 *Polyma* on polypropylene – a polyolefin known for its fast crystallization. DSC tests are easy to carry out and require only a small sample mass. In particular, isothermal crystallization measurements help to determine appropriate processing conditions such as mold temperature and cooling time so that the parts have all required properties.

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