
DSC AS PROBLEM-SOLVING TOOL: ISOTHERMAL CRYSTALLIZATION OF NYLON 6,6 FIBERS

Problem

A manager of a thermal analysis characterization laboratory has a need to perform accurate and reproducible isothermal crystallization measurements of nylon 6,6 polymers, particularly fibers. The isothermal crystallization data is important as it will be used for quality assurance purposes and to optimize the processing conditions used to manufacture the nylon 6,6 fibers. The technique and DSC instrument must be easy to use as numerous operators will be attempting to characterize the materials.

Solution

The Seiko Instruments DSC220C and the RDC (Robotic DSC) combine both ease of use and a high level of performance to make them suitable for quality assurance applications as well as for research purposes. This may be demonstrated in the analysis of the isothermal crystallization behavior of nylon 6,6 polymers.

Isothermal crystallization behaviors, as studied using differential scanning calorimetry (DSC), provides a sensitive probe for the characterization of semi-crystalline polymers such as PET or nylon. The test is performed by melting a polymer and holding under conditions which destroy the existing crystalline phase without significantly degrading the material. The molten polymer is then rapidly

cooled to a temperature which is between the glass transition temperature (T_g) and the melting temperature (T_m). The resulting crystallization of the polymer is monitored as a function of time under isothermal conditions. The time to reach the exothermic peak maximum represents the maximum rate of crystallization of the resin and this provides a very sensitive quality assurance parameter. For more advanced research purposes, the shape of the crystallization exotherm can be used to mathematically model the polymer's crystallization kinetics.

Nylon 6,6 polymers present a particular challenge as the resin crystallizes relatively quickly as compared to other polymers, such as polyethylene terephthalate (PET). The DSC instrument must be capable of cooling rapidly from the melt to the desired isothermal temperature and also be able to achieve thermal equilibration quickly.

In this study, the isothermal crystallization behaviors of nylon 6,6 fibers were characterized. Approximately 6 mg of sample was placed into a crimped DSC pan. The reference pan was furnished with 12 mg of extra aluminum in order to counterbalance the heat flow properties of the sample, and thus better achieve thermal equilibrium. The DSC220C was used with the following PID settings to permit rapid thermal equilibration of the DSC cell: P =40, I=20, D=0.50.

The following thermal program was utilized to melt the sample directly in the DSC and then quickly cool to the desired isothermal temperature:

- heat from 25°C to 300°C at 40°C/min
- hold at 300°C for 5 minutes
- cool from 300 to 240°C at 100°C/min
- hold at 240°C for 15 minutes.

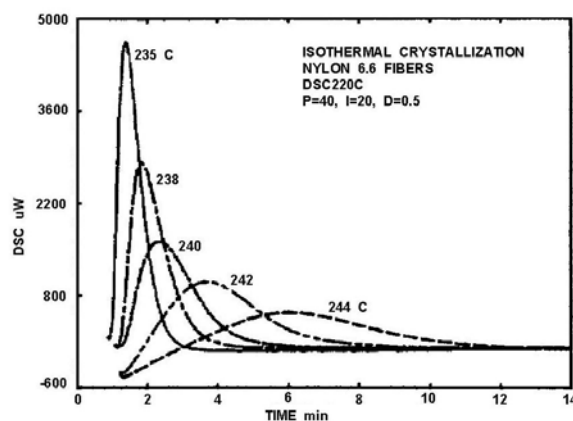


Figure 1

Figure 1 shows the effect of the isothermal crystallization temperature (235, 238, 240, 242, and 244°C) on the crystallization of the nylon 6,6 fibers. The results demonstrate that it takes

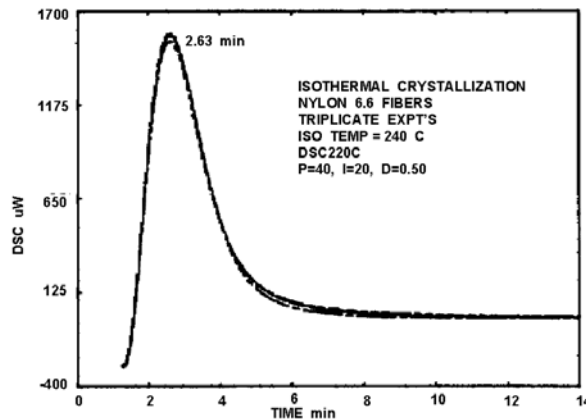


Figure 2

increasingly longer times for the polymer to crystallize as the isothermal temperature approaches the polymer's melting point (approximately 255°C). This shows that the processing temperature can have a major effect on the type and level of crystallinity achieved in the fibers during production.

Excellent reproducibility of the isothermal crystallization of the nylon 6,6 fibers is obtainable with the Seiko Instruments DSC. Three specimens of the nylon 6,6 fibers were analyzed at an isothermal temperature of 240°C. The results of the triplicate analyses are displayed in Figure 2 and an excellent level of precision is achieved.

Summary

Isothermal crystallization is a very sensitive test for the characterization of semi-crystalline polymers. The Seiko Instruments DSC offers ease of use coupled with a very high degree of reproducibility for examining the isothermal crystallization behaviors of polymers. Even rapidly crystallizable polymers, such as nylon 6,6 fibers, can be characterized with a high degree of precision with the Seiko Instruments DSC220C and Robotic DSC. The use of the isothermal PID settings (P = 40, I = 20, and D = 0.50) permits the DSC cell and the sample to quickly achieve thermal equilibrium.