

Measurement of Thermal History of Plastics by DSC

■ Introduction

It is well known that the thermal properties and mechanical properties of polymer materials change greatly depending on the thermal history associated with the sample being observed. In the case of thermoplastic resin, this is dependent on the cooling speed after melting, and because the crystalline structure of a solid changes, samples subjected to different cooling speeds may be reheated using a DSC to observe the differences in the shape of their melting peaks. This Application News introduces examples of the measurement of crystallized Nylon 66 samples that were cooled at different speeds, as well as PET (polyethylene terephthalate) and PEEK (polyetheretherketone) samples having different thermal histories.



Fig.1 DSC-60

■ DSC Measurement of Nylon 66 Samples with Different Cooling Treatment

The data in Fig.2 show the DSC curves of samples being reheated after cooling at three different speeds (-5 °C/min, -50 °C/min, quick cooling). In every sample, an endothermic peak is observed in the vicinity of 260 °C due to melting. In particular, in the sample subjected to relatively rapid cooling (-50 °C/min), an exothermic peak likely attributable to crystallization is observed just prior to melting. Moreover, in the sample

subjected to extremely rapid cooling (quick cooling), glass transition is observed at 40.7 °C, and at 62.2 °C, a large crystallization peak also appears, indicating non-crystallinity. Thus, because the DSC reheating curve is dependent on the crystallization condition in the cooling process, the peak pattern is different from that of the original DSC curve.

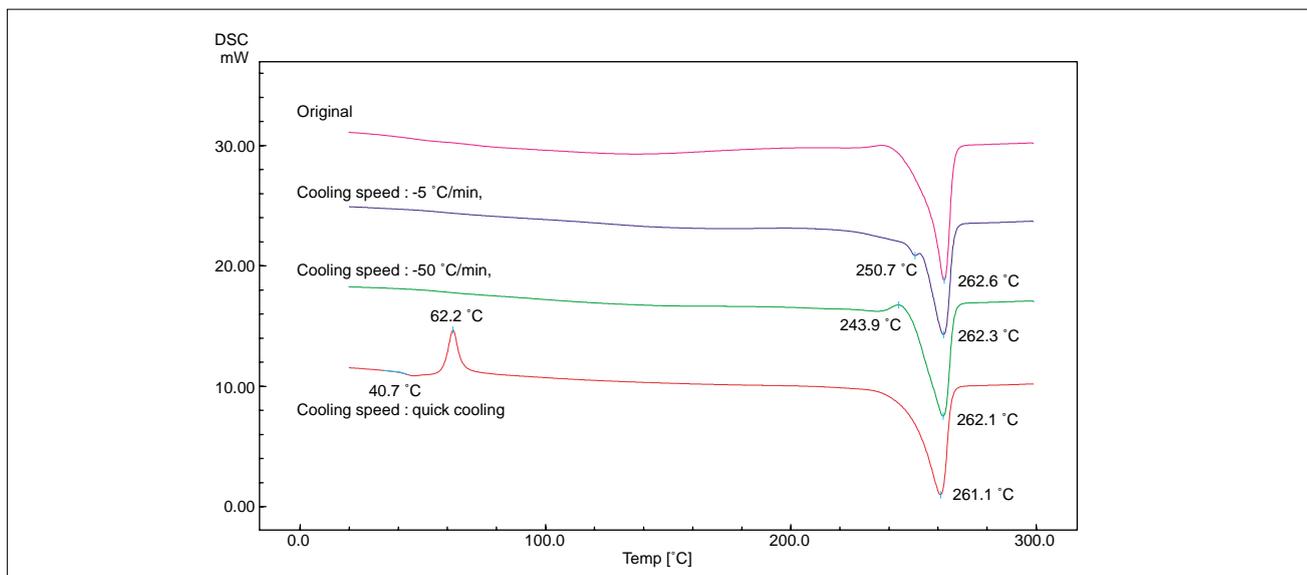


Fig.2 DSC Curves of Nylon 66 Samples with Different Cooling Treatment

■ Melting of PET Bottles for Hot and Cold Storage

Fig.3 shows the data obtained from DSC measurement of PET bottles for hot and cold storage, respectively (1st run), and during reheating following cooling (2nd run). Comparing the 1st run data for the hot and cold storage bottles, a difference in the DSC behavior is seen up to about 150 °C. The exothermic change at 100 °C in the cold storage bottle is expected

due to crystallization, and the observed absence of this in the hot storage bottle suggests that the relative degree of crystallization in the cold storage bottle is lower. Moreover, the absence of behavioral differences up to 150 °C in the 2nd run suggests that the difference seen in the 1st run is due to the difference in the thermal history during the molding process.

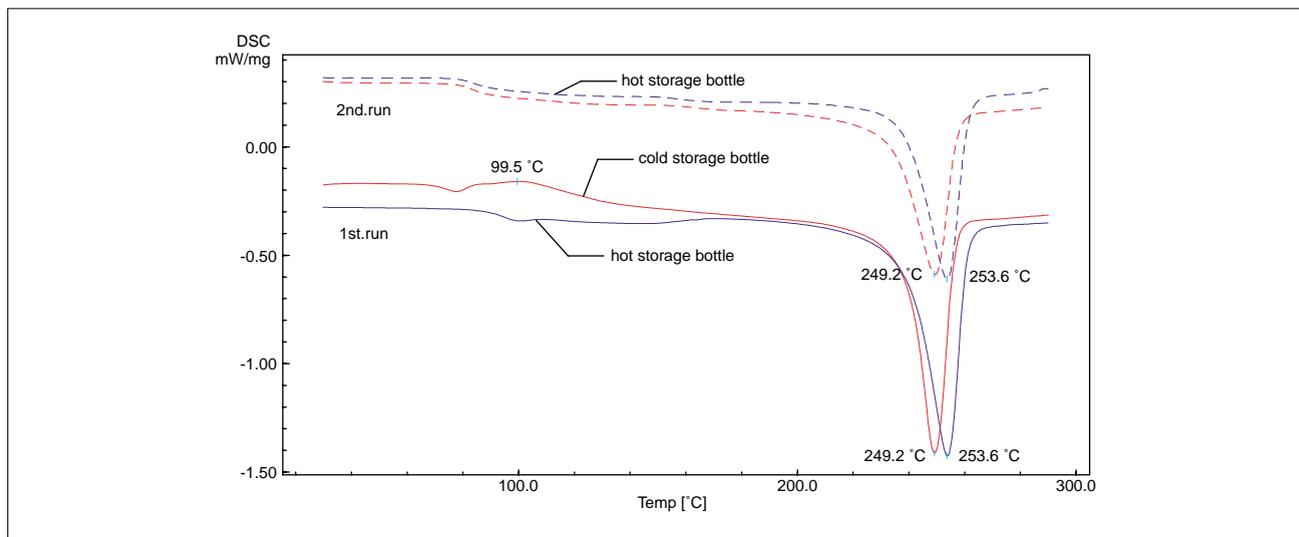


Fig.3 DSC Curves of the Melting of PET Bottles for Hot and Cold Storage

■ DSC Measurement of Film-Shaped and Block-Shaped PEEK

Fig.4 shows the data obtained from DSC measurement of film-shaped and block-shaped PEEK, respectively. In the film-shaped PEEK, because glass transition is observed at 144.6 °C and crystallization is observed at 181.4 °C, it is non-crystalline. In contrast to this, in the block-shaped PEEK, as only a melting

peak is observed at 339.2 °C, it is crystalline. This suggests that film-shaped PEEK did not become crystallized because it was cooled quickly during forming, while the block-shaped PEEK was well crystallized due to slow cooling during molding.

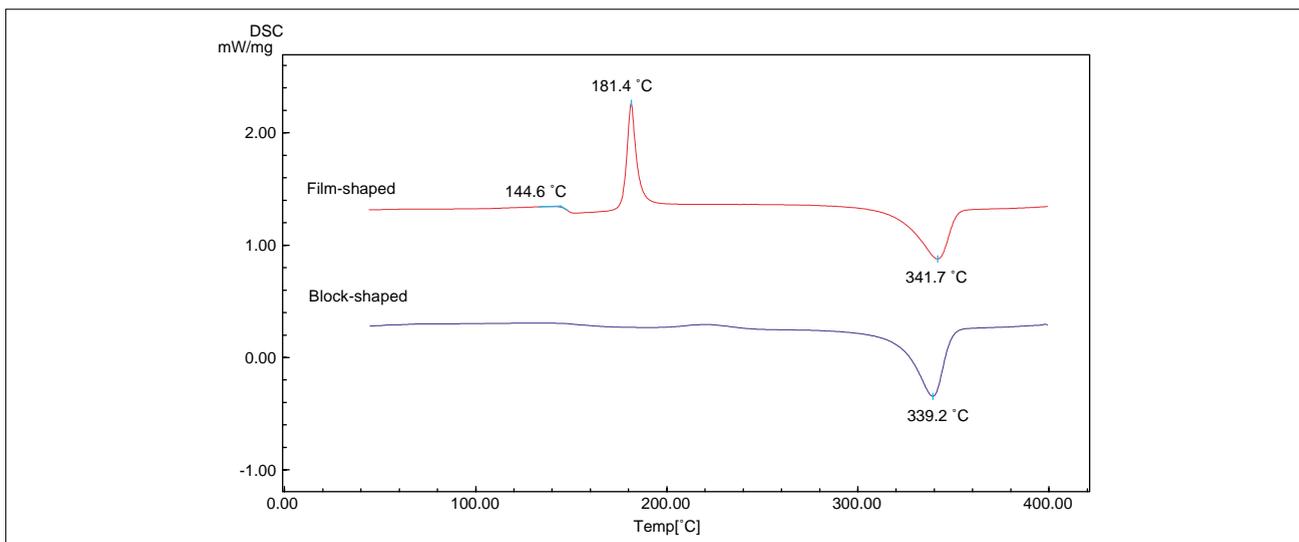


Fig.4 DSC Curves of Film-Shaped and Block-Shaped PEEK

NOTES:

*This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.



SHIMADZU CORPORATION. International Marketing Division

3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax: 81(3)3219-5710
Cable Add.:SHIMADZU TOKYO