SII NanoTechnology Inc.

DSC AS PROBLEM-SOLVING TOOL: ISOTHERMAL CRYSTALLIZATION OF BOTTLE RESINS

Problem

It is often found that bottle resin polymers (chiefly PET, polyethylene terephthalate) can yield bottles with differing properties, such as optical clarity and barrier resistance. What is needed is a straightforward analytical test which will assist in the characterization of the PET resin used to manufacture the bottles.

Solution

Differential scanning calorimetry (DSC) provides a means of easily observing the crystallization behavior of PET bottle resins. This is done by melting the polymer and holding under conditions which fully destroy the existing crystalline material. The molten polymer is then rapidly cooled to an isothermal temperature which is between the glass transition event (Tg) and the melting point (Tm). The 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 bottle resin and this provides a very sensitive quality assurance parameter.

The isothermal crystallization test provides information on the following properties of resins:

- type of nucleating agents
- concentration of nucleating agents
- average molecular weight
- molecular weight distribution
- presence of other components (e.g., diethylene glycol)

These various parameters can affect the crystallization of the resin and, therefore, the properties of the bottle produced from the resin. These properties include barrier resistance, optical clarity, creep, stiffness and brittleness.

Two PET bottle resins (A and B) were tested using the DSC isothermal crystallization test. The resins, which were supposedly the same, were held at a temperature of 290°C on a hot plate for 5 minutes to fully melt the existing crystalline domains. The molten samples were then quickly transferred to the Seiko Instruments DSC220C which was preheated to the desired isothermal temperature. The experiment was initiated when the sample was placed into the DSC cell. The resins were held under isothermal conditions for 30

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minutes to observe the crystallization. The large mass of the silver heater block of the DSC220C provides a very stable temperature environment and this yields reproducible and accurate crystallization data.

Figure 1 shows the effect of isothermal temperature on the crystallization of the PET bottle resin (resin A). The resin was crystallized at temperatures of 190, 195, 200, 205 and 210°C under isothermal conditions. The results demonstrate that it takes increasingly longer times for the polymer to crystallize as the isothermal temperature approaches the polymer's melting point (approximately 250°C). This shows that the processing temperature can have a major effect on the type and level of crystallinity achieved during the production of a bottle.

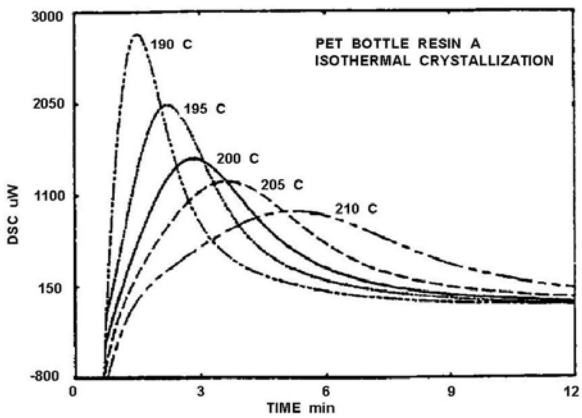

Figure 1

A direct comparison of the DSC data for resins A and B (normalized to a constant mass of 10.00 mg) is displayed in Figure 2 at an isothermal crystallization temperature of 210°C. The results show that resin A takes a significantly longer time to reach its maximum rate of crystallization (5.26 minutes) as compared to resin B (3.19 minutes). The samples were originally believed to be identical. The data indicates that the bottles produced from these two resins could be different in terms of their end use characteristics.

Summary

Isothermal crystallization is a very sensitive test for the characterization of resins used in the production of bottles. The DSC220C provides a

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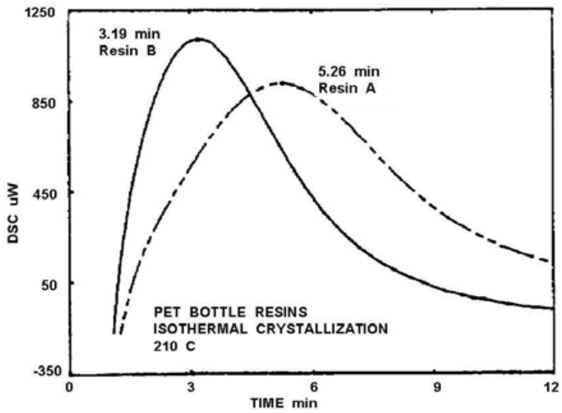

Figure 2

stable isothermal temperature environment which yields accurate and reproducible data on the crystallization behavior of the resins. The isothermal results can furnish information on the performance of the PET resins utilized in the blow-molding process and can assist in the estimation of the end use properties of the bottle, such as barrier resistance, creep and optical clarity.

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