SII NanoTechnology Inc.

DSC AS PROBLEM-SOLVING TOOL: DETERMINATION OF HEAT SET CONDITIONS OF NYLON 6 FIBERS

Problem

A thermal analyst working at a fibers R&D center has an urgent need to develop an analytical test to detect the heat set temperature or processing conditions associated with the production of nylon 6 fibers.

During the production of synthetic fibers, such as PET, nylon 6,6 or nylon 6 fibers, the material is oftentimes subjected to specific heat set conditions. The heat setting processing step entails running the spun fibers over a hot surface or a heated tunnel and this procedure stabilizes the thermal and physical properties exhibited by the fibers. Nylon 6 fibers are generally heat set using the Superba process which involves exposing the fibers to pressurized steam. The heat set temperature is somewhere well above the glass transition temperature (Tg) of the fibers, but significantly below the melting point. Both the exposure or dwell time of the fibers and the temperature/pressure of the steam in the Superba tunnel will affect the thermal/physical properties imparted to the nylon 6 fibers during production.

It is important to be able to detect the heat set conditions used to generated heat treated fibers or yarns. The measurement of the yarn heat set conditions provides the following useful information:

- quality assurance
- process optimization
- trouble-shooting for process 'upsets' such as spinline breakages
- analysis of yarn streaking defects
- characterization of competitive fibers or yarns

The detection of the yarn heat set conditions can observed using differential scanning calorimetry (DSC). DSC provides an easy to use, but yet sensitive means of detecting the small changes occurring in the fibers as a result of the heat setting conditions. For nylon 6 fibers or yarns, the structural changes taking place in the polymer as a result of heat setting are very small and can become difficult to observe or detect using standard DSC instruments. The subtle differences associated with nylon 6 yarns requires the use of a DSC instrument which provides very high sensitivity.

Thermal Analysis & Surface Solutions GmbH
Pfingstweide 21
61169 Friedberg - Germany

+49-6031-16223-1
+49-6031-16223-29
Info@thass.net
http://thass.net

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Solution

The Seiko EXSTAR DSC6100 system offers the highest sensitivity DSC instrument on the market. This advanced DSC instrument offers the following features essential for the characterization of nylon 6 fibers:

- very high sensitivity
- exceptionally stable baseline
- excellent subambient performance
- wide temperature range (-150 to 500°C)
- ease of use

With the DSC approach, a sample of the yarn (approximately 10 mg) is simply balled up and placed into a standard crimped aluminum pan. For nylon fibers, it is important to punch a small pin hole in the lid to permit the absorbed moisture to be evolved during heating. In order to best observe the heat set conditions, a heating rate of 5°C/min is recommended since this slower rate helps to better separate the heat set properties from the melting of the crystalline component. It is important, with nylon fibers, to use a nitrogen purge gas at a flow rate of 50 to 100 mL/min when conducting DSC measurements.

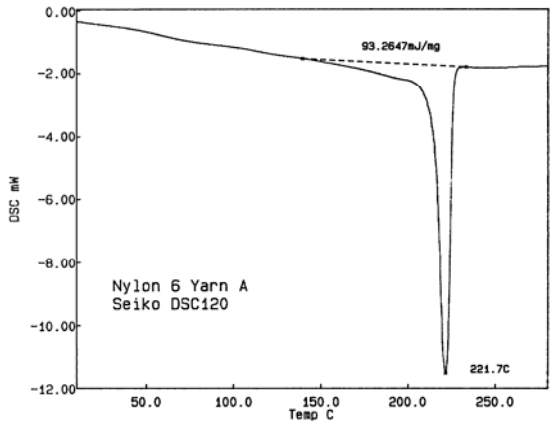

Figure 1

Displayed in Figure 1 are the results obtained from the Seiko high sensitivity DSC instrument on a sample of nylon 6 yarn under the experimental conditions described in the preceding paragraph. The melting of the crystalline component is observed as a large endotherm at 220°C.

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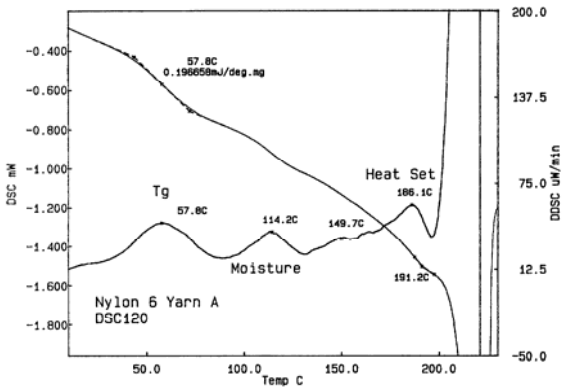

Figure 2

The heat set properties associated with the generation of the nylon 6 yarn can be observed in an enlarged view of the DSC data below the melting event and this is displayed in Figure 2 for the nylon 6 yarn. This plot shows the heat flow and the derivative trace as a function of the sample temperature. The derivative data, in particular, helps to more clearly identify the subtle heat set conditions associated with nylon 6 fibers. For yarn sample A, the glass transition temperature is observed as a small step wise change in the heat flow at 57.8°C and a peak in the derivative at 57.8°C. Another peak in the derivative trace is obtained at 114°C and this most likely reflects the evolution of a small amount of remaining moisture from the hydrophilic nylon 6 fibers. A small broad peak is observed in the derivative data at approximately 150°C and this most likely represents heat history conditions. The derivative peak detected at 186.1°C is the main heat setting peak and the temperature and magnitude of this particular peak reflects the particular heat setting conditions (dwell time and steam temperature/pressure in the tunnel). The heat setting step changes the morphology or structure of the yarn and it is believed that small, imperfect crystallites are formed as a result of heat setting. When the heat set fibers are analyzed in the DSC, the melting of these small crystallites are detected producing a change in the heat flow characteristics and generating a peak in the heat flow derivative signal.

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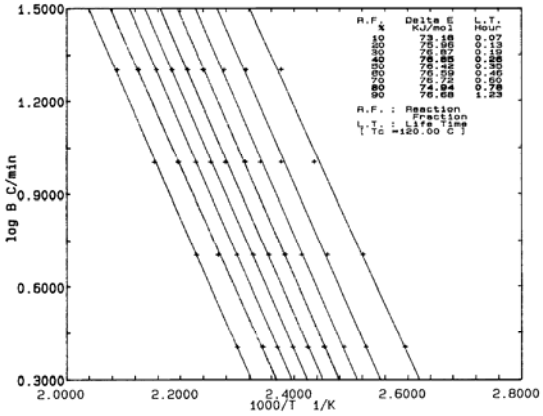

Figure 3

Displayed in Figure 3 is an overlay of the DSC derivative profiles of three nylon 6 yarn samples (A, B and C) which have been exposed to increasing rigorous processing conditions. As the tunnel temperature increases, both the temperature and magnitude of the DSC derivative peak increases. Thus, the Seiko high sensitivity DSC provides a very useful characterization parameter to ascertain the heat set conditions utilized to generate nylon 6 fibers or yarns.

Summary

DSC can be used to detect the heat set temperatures associated with the production of synthetic fibers. The characterization of nylon 6 fibers generated using the Superba heat set processing requires the use of a high sensitivity DSC instrument, such as the Seiko DSC6100.

The heat set conditions (dwell time and steam temperature and pressure) are most clearly observed in the DSC derivative data. The heat set conditions of fibers are often related to the end-use performance of the yarn in terms of properties such as shrinkage, twisting or dyeability.

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