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Detection of Heat Set Temperatures of Nylon 6 Carpet Yarns by DSC

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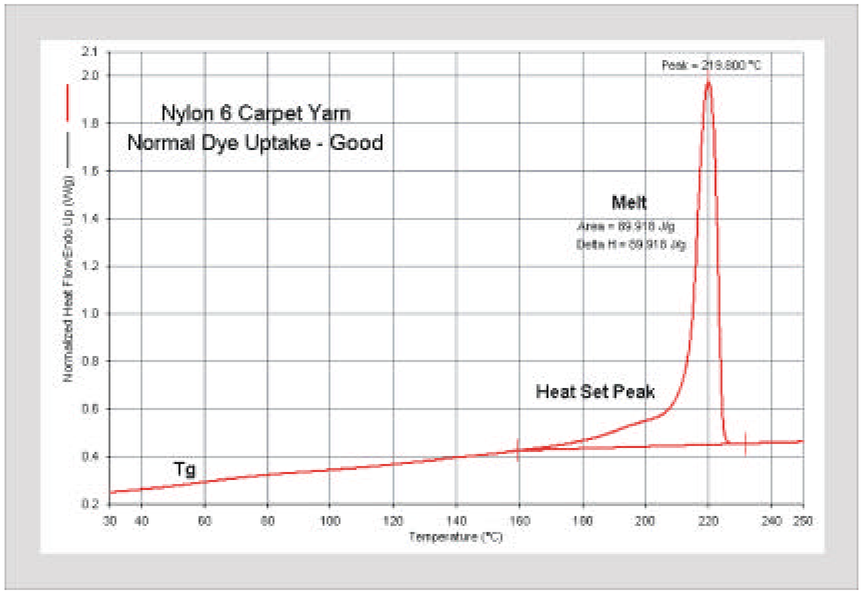
Problem

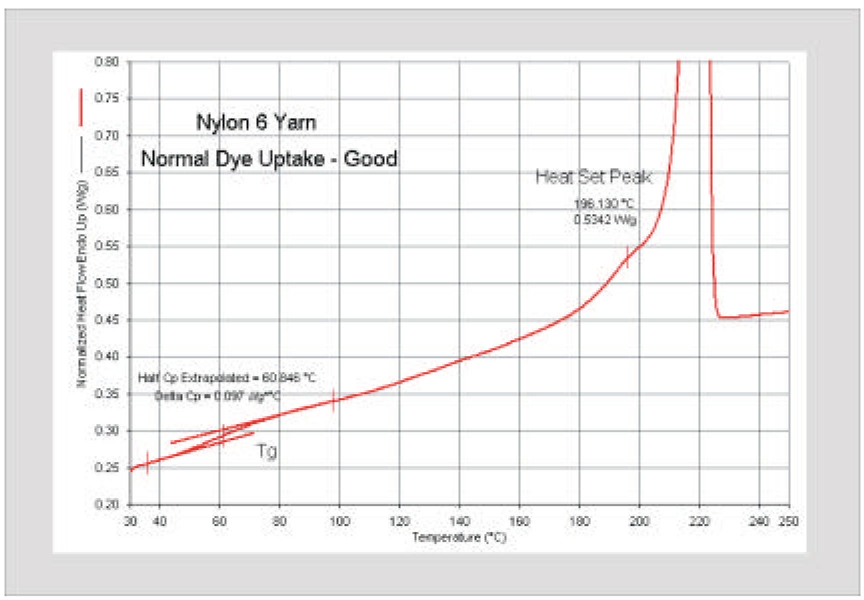
A thermal analyst working at a fibers R&D center and production facility has a need to develop an easy-to-use test to detect the heat set temperature or processing conditions associated with the production of nylon 6 fibers, used to produce carpet.

During the spinning or production of synthetic fibers, such as nylons or PET, the polymer is subjected to specific heat set or thermal treatments. The heat set processing step entails running the spun fibers over a hot surface or through a heated tunnel and this procedure stabilizes the thermal and physical properties exhibited by the fibers. Nylon 6 fibres are generally heat set using the either the Suessen or Superba treatments. The Superba process is becoming more commonplace for nylon 6 yarns and 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.

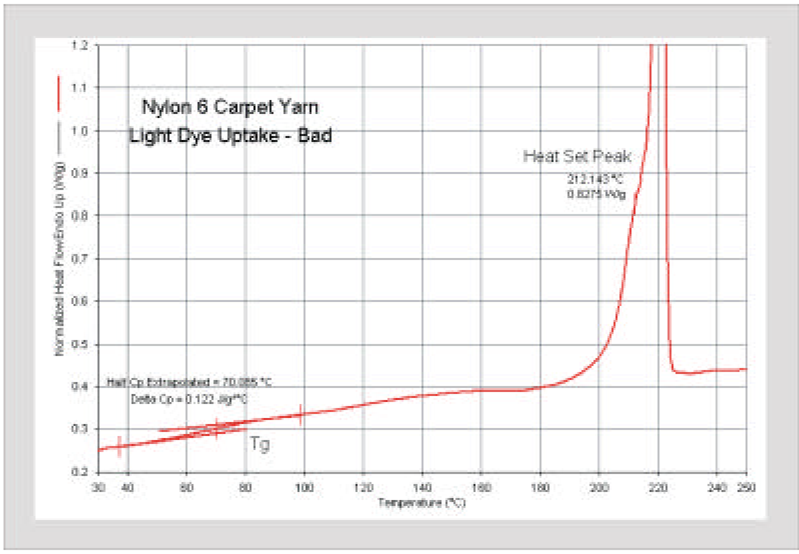
It is very important for a fiber manufacturer to be able to detect or characterize the heat set conditions used to generate the fibers or yarns. The measurement of the heat set conditions provides the following valuable information:

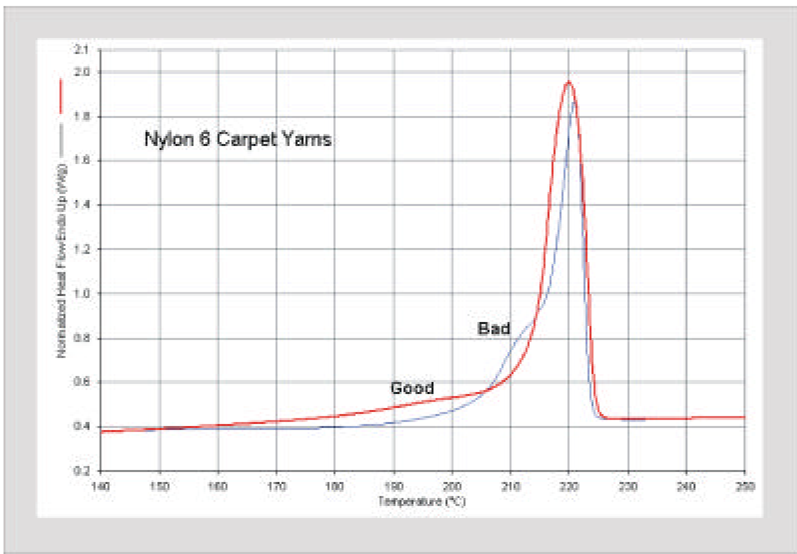
- Assessment of dye uptake performance
- Quality assurance





PerkinElmer™ instruments.





- 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 be observed using differential scanning calorimetry (DSC), which measures the heat flow into or from a sample. DSC provides an easy to-use, but yet sensitive means of detecting small changes occurring in the fibers as a direct result of the heat set processing. For nylon 6 yarns, the structural changes taking place in the polymer as a result of heat setting, especially with the Superba treatment, are very small and can be difficult to observe or detect using most DSC instruments. The subtle differences associated with Superba heat set nylon 6 yarns requires the use of a DSC instrument, which provides both high sensitivity as well as high resolution.

Solution

The PerkinElmer power compensated DSC 7 and Pyris 1 DSC provide both the necessary high sensitivity and high resolution necessary for the measurement of the heat set conditions of nylon 6 yarns. The DSC 7 and the Pyris 1 DSC offer the following features:

- Very high sensitivity
- Outstanding and unparalleled resolution
- Stable baseline performance
- Ease of use with Pyris for Windows software

The high sensitivity is required to detect the very weak heat set, pre-melting endothermic peak and the high resolution is necessary to separate the heat set peak from the main melting peak associated with the nylon 6 fibers.

With the DSC approach, a sample of the yarn (approximately 8 mg) is simply balled up and placed into a standard aluminum sample pan and crimped. The yarn is then heated at a rate of 10 C/min from 25 to 280 C. It is important to use a dry nitrogen purge (50 mL/min) to avoid oxidative degradation of the nylon fibers while heating. Because of the hydrophilic nature of nylon fibers, it is recommended to first heat the sample to a temperature of 120 C, in the DSC, and hold the specimen for about 2 minutes to drive off the absorbed moisture. The sample is then

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cooled back to 25 C and heated up through the melt.

Two samples of nylon 6 carpet yarns (produced using the Superba heat set treatment) were characterized using the DSC 7. One yarn was good in that its dye uptake properties were normal or satisfactory. The second yarn sample was bad, or defective, and was lighter in color compared to the good yarn indicative of poor dye uptake.

Displayed in the following figure are the results obtained from the PerkinElmer DSC 7 on a sample of the good nylon 6 yarn. The melting of the crystalline component is observed as a large endothermic peak at 219.8 C with a heat of melting of 89.9 J/g. The heat of melting is directly related to the percent crystallinity of the fiber.

The heat set properties associated with the production of the generation of the nylon 6 yarn can be better observed in an enlarged view of the DSC heat flow data below the melting event and this is displayed in the following figure for the good nylon 6 yarn sample.

The enlarged view shows two weak transitions which are normally very difficult to detect for Superba heat set nylon 6 fibers: the glass transition event or Tg at 61 C and the heat set endothermic peak at 196 C. The heat set peak reflects the morphological or structural changes that occurred in the fibers as a result of the heat set processing step. It is believed that small, imperfect crystallites are formed as a result of heat setting. A lower or more mild heat set conditions will produce a lower temperature pre-melting endothermic peak; whereas more severe treatments will move the heat set peak closer to the main melting peak at 220 C. When the heat set fibers are analyzed in the DSC 7, the melting of the small crystallites are detected and observed as a change in the sample's heat flow properties.

Displayed in the next figure are the DSC 7 results generated on the bad nylon 6 yarn sample. This yarn had poor dye uptake properties and was noticeably lighter in color as compared to the good yarn. The light yarn would be unacceptable for the production of a carpet as it would result in streaking or distinct color variations.

The light yarn has a higher Tg (70.1 C) as compared to the normal or good yarn (61 C) and this is indicative of exposure to higher heat set conditions. The heat set endotherm for the bad yarn is observed at 212 C, which is significantly higher than that measured for the good yarn (196 C). The higher heat set peak indicates that the light or bad fibers were subjected to much more severe conditions during the heat setting processing step.

In the following figure, the heat flow properties of the good and bad nylon 6 fibers are directly overlaid, for direct comparative purposes.

The thermal characteristics of the good and bad nylon 6 yarns are very evident in these results. The heat set endothermic peak occurs at a much lower temperature for the good or normal dyed yarn and this is consistent with good dye uptake properties.

Summary

The PerkinElmer power compensated DSC 7 and Pyris 1 DSC offer very high sensitivity and outstanding resolution. These two features are essential for the characterization of the processing conditions used to manufacture fibers or textured yarns such as nylons or PET. The PerkinElmer DSC provides outstanding data on the Superba heat-treated nylon 6 yarns, which are normally very difficult to characterize. Clear differences could be seen in the heat set peak temperatures for 'good' and 'bad' yarns, with respect to their respective dye uptake properties. More severe heat set treatments, resulting in poor dye uptake, produce a higher temperature heat set peak, as observed by DSC. The PerkinElmer DSC was able to detect the very weak Tg associated with the highly crystalline nylon 6 fibers. The measurement of both the Tg and the Superba heat set endothermic peak demonstrate the very high performance of the PerkinElmer power compensated DSC.

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