



Evaluation of Fiber Heatsetting by MDSC[™]

Els Verdonck
TA Instruments, Inc., Raketstraat 60, 1130 Brussels, Belgium
and
Valja Everaert, Myriam Vanneste and Luc Ruys
Centexbel, Yarn Engineering Unit, Technologiepark 7, 9052 Ghent, Belgium

ABSTRACT

Heatsetting is a processing step in the production of synthetic carpet yarns. Determination of the heatset temperature is important to verify the quality and homogeneity of this temperature treatment. Modulated DSC, (MDSC[™]) is shown to be the preferred tool for the determination of the heatset temperature.

INTRODUCTION

Heatsetting is an important step in the production of synthetic carpet yarns. It imposes a thermal treatment on fibers at temperatures higher than their glass transition temperature, and induces changes in fiber crystallinity, chain ordering, *etc.* These rearrangements are retained in the fiber as a ‘memory-effect’, and affect both the physical properties of the fibers and the degree of dye uptake. A higher heatset temperature (T_{HS}) typically results in a darker color.

Non-uniform heatsetting can thus induce variations in fiber morphology, leading to problems in subsequent applications, such as streaks in finished carpets. Other factors (*e.g.*, chemical differences of the raw material), however, can also lead to the formation of streaks. For both vendors and customers, an accurate diagnosis of the origin of streak formation is important. So a need exists for the detection of differences in thermal history of the yarns.

Several methods for detection of T_{HS} are proposed in the literature. Khanna proposed thermomechanical methods (TMA, DMA) are based on the different shrinkage behavior of fibers heatset at different temperatures (1). An estimate for T_{HS} from thermal methods is traditionally obtained from the premelting endothermic peak appearing in a DSC thermal curve. The latter has been related to the melting of small crystallites created by secondary crystallization during heatsetting of synthetic polymers. Detection of this premelting endothermic peak value, however, is often difficult due to the small size of this peak.

Everaert and coworkers showed that Modulated DSC, (MDSC[™]) is an excellent technique for the determination of the heatset temperature (2, 3).

EXPERIMENTAL

Polypropylene (PP) and polyamide 6 (PA6) yarns are used in this demonstration. Heatsetting was performed with the Suessen[®] technique (dry heat), or the Superba[®]

technique (pressurized steam) at different heatset temperatures. The fibers are then investigated with MDSC.

RESULTS AND DISCUSSION

Polypropylene (PP) yarns are subjected to Superba heatsetting at varying heatset temperatures (from 128 to 138 °C). The nonreversing heat flow of MDSC for these samples is shown in Figure 1a. The observed onset of the nonreversing MDSC signal is very close to the imposed T_{HS} (Figure 1b). Similar results were obtained after Suessen heatsetting. Simulation of the latter dry heatsetting process in the DSC furnace confirms this correlation (Figure 1b).

The onset of the nonreversing signal thus can be used to assess T_{HS} . Heatsetting provokes (re)crystallization of the polymer chains up to the imposed temperature. Subsequent heating in MDSC will cause (re)crystallization only from above T_{HS} . The start temperature of this effect can be detected from the onset of the nonreversing signal.

Since the temperature range for Suessen heatsetting (typically from 145 to 150 °C) and Superba heatsetting (typically from 128 to 138 °C) do not overlap, MDSC provides also an efficient method to estimate the heatsetting equipment used for the PP yarns.

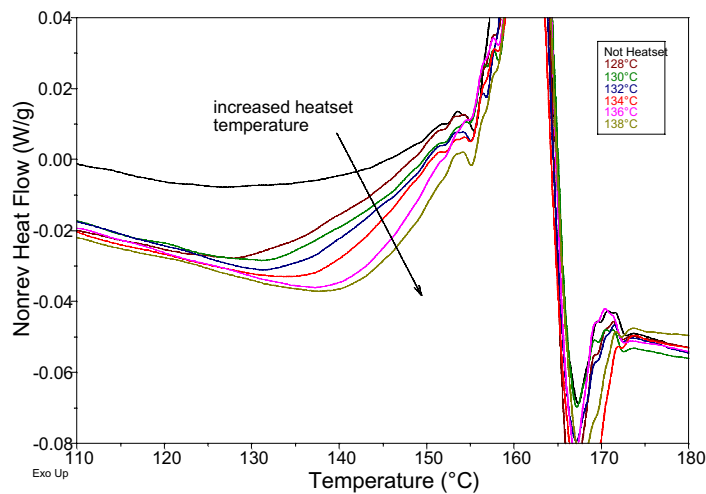


Figure 1a - MDSC Thermal Curve for Superba Heatset Polypropylene Yarns

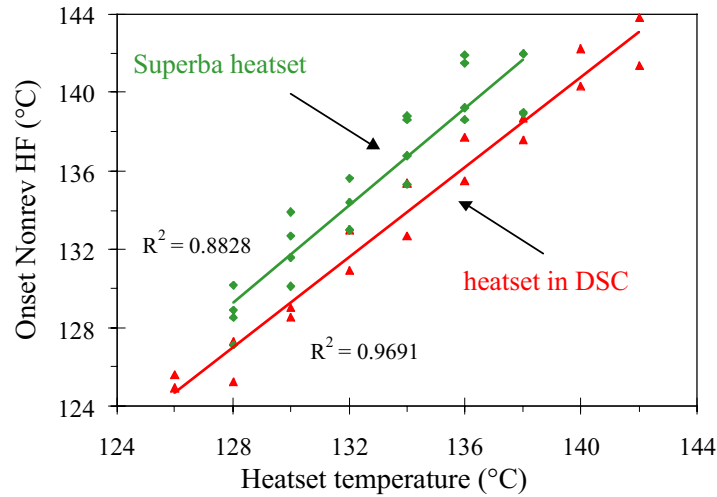


Figure 1b - Correlation Between Imposed Heatset Temperature and Onset Obtained from MDSC for Polypropylene Yarns (Superba Heatset and Simulation of Heatset in DSC)

Polyamide 6 (PA 6) Yarn

Heatsetting experiments for **polyamide 6 (PA 6) yarn** were performed both on Suessen (typically from 190 to 200 °C) and Superba (typically from 124 to 132 °C) machines. As for PP, T_{HS} coincides with the onset of the nonreversing signal for Suessen heatset PA6 yarn (Figure 2a). For Superba heatset PA6 yarns however, MDSC only provides a comparative heatset-index since the onset of the nonreversing signal is shifted to higher temperatures than T_{HS} (Figure 2b). (Re)crystallization starting at a higher temperature than T_{HS} , is a result of the high sensitivity of PA6 for moisture (present only during the Superba process). Since the nonreversing onset temperature occurs in the same temperature region for Suessen and Superba heatset fibers, it is not possible to distinction between both processing techniques for PA6.

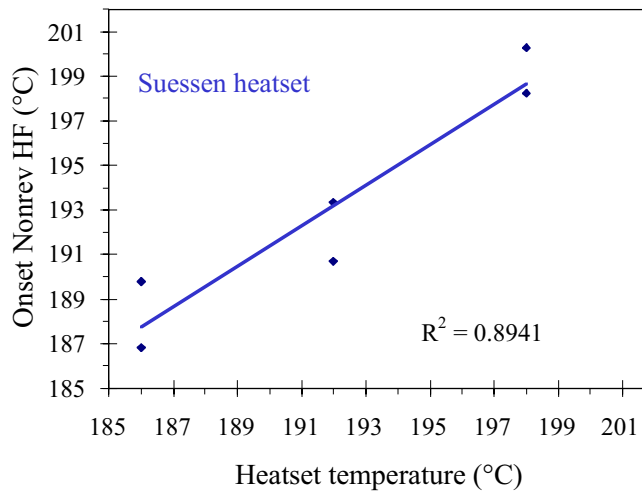


Figure 2a - Correlation Between Imposed Heatset Temperature and Onset Obtained from MDSC for Suessen Heatset Polyamide 6 Yarns

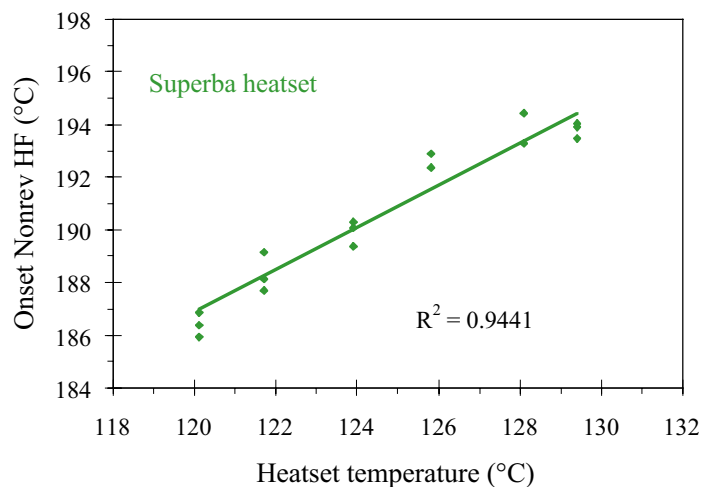


Figure 2b - Correlation Between Imposed Heatset Temperature and Onset Obtained from MDSC for Superba Heatset Polyamide 6 Yarns

CONCLUSION

For PP and PA6 yarns, an excellent correlation is found between the imposed heatset temperature and the onset of the nonreversing heat flow obtained from an MDSC experiment. Hence MDSC is an excellent tool in controlling the quality of the heatsetting of these synthetic yarns.

REFERENCES

1. Y. P. Khanna, "Evaluation of Thermal History of Polymeric Films and Fibers Using DSC/TMA/DMA Techniques", *Journal of Applied Polymer Science*, **1990**, 40, pp. 569-579.
2. V. Everaert, M. Vanneste, and L. Ruys, "Techniques for the Evaluation of Fiber Heatsetting in PP and PA Carpet Yarns", *Unitex*, **1999**, 6, pp. 33-36.
3. M. Vanneste, V. Everaert, and L. Ruys, "Evaluation of PP Fiber Heatsetting by Means of MDSC", *Chemical Fibers International*, **2000**, 50, pp. 563-564.

KEYWORDS

films/fibers, modulated differential scanning calorimetry, polyamide polymers, polyolefin polymers, relaxation