TA Instruments

Thermal Analysis & Rheology

Thermal Analysis Application Brief

Polyester Heat History Detection by DSC

Number TA-128

Summary

Differential scanning calorimetry (DSC) is a thermal analysis technique which provides heat history information about polymeric fibers. This information is useful for predicting optimum processing conditions, as well as processing and end-use properties, for the fiber.

Introduction

Heat treatments such as heat-setting, drying, autoclaving and dyeing often change the degree and type of crystalline structure (morphology) in synthetic fibers. Differential scanning calorimetry (DSC), which measures the heat flow into and out of a material with temperature, provides a convenient way to determine the crystalline properties of polymers and subsequently to predict certain processing and end-use characteristics.

Experimental

In DSC, the fiber sample (typically 5-10 milligrams) is crimped in an aluminum pan and run versus an empty reference pan. The subsequent heat flow curve obtained during heating is analyzed to obtain transition temperature and/or quantitative heats involved in the reactions. In this study, polyethylene terephthalate (PET) yarns textured at temperatures between 190°C and 225°C were evaluated while heating at 50°C/minute in a flowing 50cc/minute nitrogen atmosphere.

Results

Polyethylene terephthalate (PET) yarn samples textured at temperatures between 190°C and 225°C exhibit a small DSC endotherm at temperatures which vary with the texturing temperature, as shown in Figure 1. The inflection point or peak position of this endotherm will move to higher temperatures with increasing texturing temperature or time. Figure 1 illustrates that DSC can be used to correlate texturing temperature with the peak position of the endotherm and, therefore, DSC can be used as a rapid technique for quality assurance in the texturing process.

The pseudo-endothermic event observed at 90°C in all four thermograms is due to a volume relaxation phenomenon occuring at the glass transition temperature (Tg) of the PET. This is the result of a physical aging process which gives rise to an ordering of the amorphous molecules when the material is stored for extended periods at temperatures below Tg. The longer the aging, the larger the endothermic relaxation peak. The magnitude of the relaxation can be used to correlate with other morphological phenomena which occur as a result of phyiscal aging and which in turn may influence the subsequent processing of the material.

The large endotherms which are off-scale in Figure 1 are due to fusion of the crystalline PET. A high heating rate is used in this example to avoid annealing out the more subtle morphological changes during the DSC heat cycle.

A series of heat-treatments will often erase former peaks, although a remanent of a former peak sometimes remains as a slope change in the curve. Results have shown that if a second heat-treatment is more "severe" than the first, it predominates. Since DSC peaks are influenced by any heat treatment prior to the one being studied, the behavior of suitable contol samples should be examined when evaluating an unknown.

POLYETHYLENE TEREPHTHALATE
TEXTURING TEMPERATURE

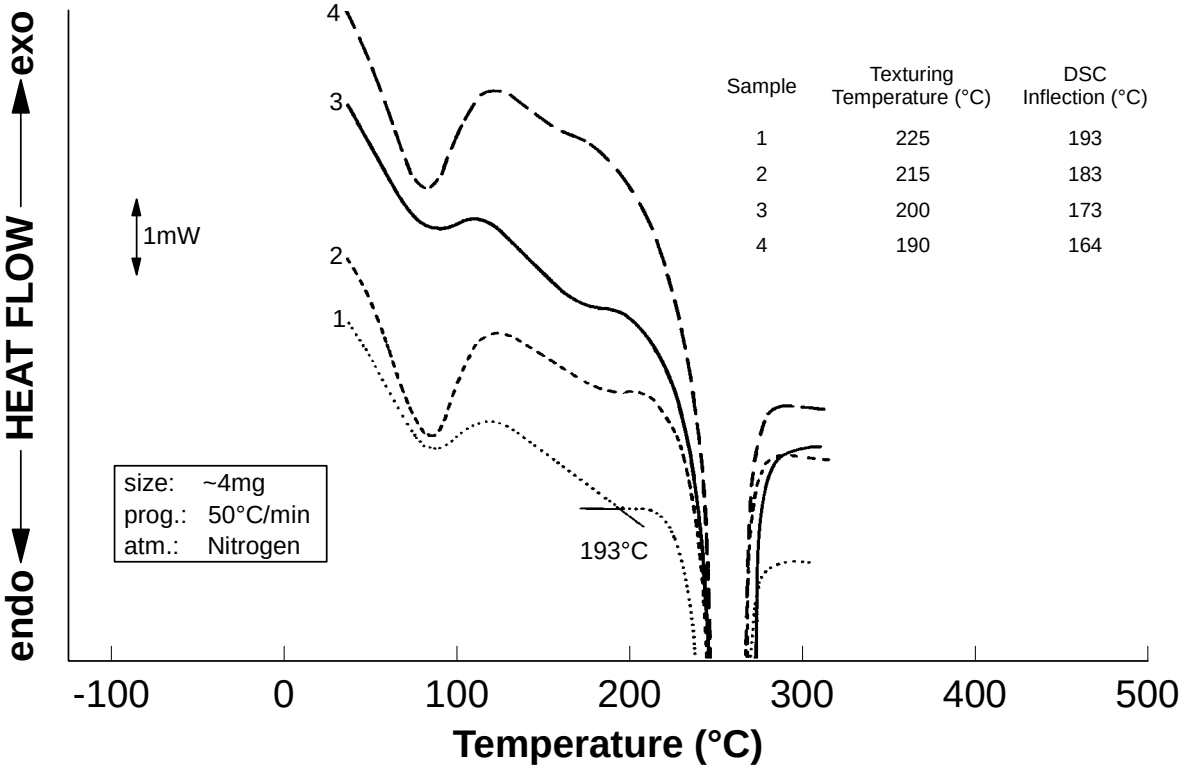

Figure 1

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