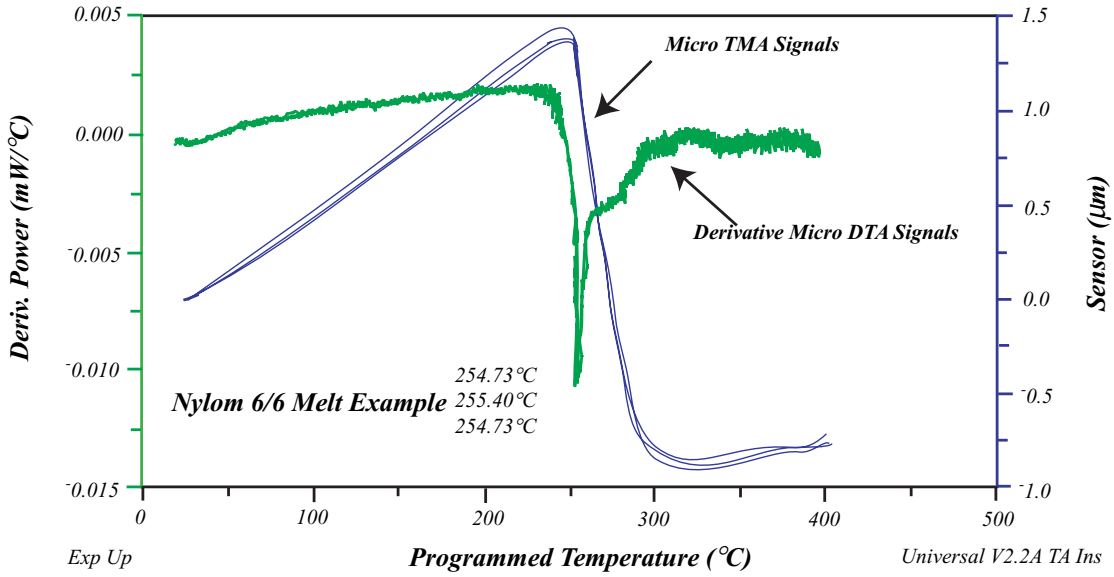
THERMAL SOLUTIONS

Polymer Melt Characterization and Reproducibility by Micro Thermal Analysis



Micro thermal analysis (µTA) uses a tiny resistive thermal probe within an atomic force microscope (AFM) to collect images related to sample topography and thermal conductivity. Using the images as guides, points can then be selected for further examination by local thermal analysis (LTA). In this technique, the probe is positioned at the selected points and the temperature is ramped from a predetermined start to a predetermined final temperature at very high ramp rates (5-25°C/s). Signals analogous to TMA and DTA are collected simultaneously in the LTA experiments. A modulated temperature signal can also be superimposed upon the base ramp to increase sensitivity.

Repeatability of µTA is demonstrated using multiple determinations. The example above highlights the use of LTA to detect the melting transition in a Nylon 6/6 polymer sample. Three separate positions were chosen and thermal scans were taken from 25-400°C at a rate of 25°C/s. The data displays the two signals generally collected: the micro thermal mechanical (µTMA) signal which is related to a macro TMA experiment, and the micro differential thermal signal (µDTA) which is related to a macro DTA experiment. Both signals are collected simultaneously and the transition is easily seen in each. The expansion of the surface prior to the melt and softening, with probe penetration, at the melt are detected in the µTMA signal. The polymer melt is detected as a peak in the derivative of the µDTA data and, analysis of the peaks indicates the reproducibility. Total time for all three experiments: ~1.5 minutes.

A peak occurs in the derivative of the DTA data, rather than the DTA data, because in a µTA experiment as the probe penetrates and heat flows through the sample, different parts of the sample melt at different times. The average of the analyzed peaks gives a temperature of ~255° C, which falls within the range for Nylon 6/6 listed in the literature as 255-265°C.