Introduction

Crime or forensic laboratories must frequently work with very small samples in order to determine the type of material and its possible manufacturer for investigatory and evidence purposes. An example would be in the characterization of single fibers found at the crime scene. Fibers are useful for forensic purposes, as they tend to cling easily and provide useful characteristics for identification purposes. The disadvantage is the fibers are very low-mass (on the order of 50 µg) which renders it difficult for thermal analysis characterization techniques.

Thermal analysis, and in particular Differential Scanning Calorimetry (DSC), is useful for characterizing polymers and fibers. Typically, the mass used for DSC experiments is at the order of 5 to 10 mg. However, a single fiber has a mass that is 100 times less than the usual weight. For this special application, a DSC instrument with a high level of sensitivity and performance is required. In particular, High Speed DSC is a very useful approach for the characterization of low-mass materials since the use of very fast heating rates (100 to 400 °C/min) provides significantly greater sensitivity. Power Compensation DSC has been successfully used for forensic studies of toners on photocopied documents.¹
PerkinElmer manufactures three types of infrared microscopy systems: the MultiScope™, the AutoIMAGE™, and the Spotlight™ 300. All systems interface with a standard FT-IR. The MultiScope is our most cost-effective microscope system. It can provide the same information as the other systems, but in a manual mode. The AutoIMAGE system is the standard for performing automated microscopy. All microscope controls are fully automated for ease of use and speed of analysis. We used the Spotlight 300 system for this work. It can quickly provide a high-quality infrared image of a small sample in at least one-tenth the time of its competitors.

PerkinElmer’s strengths in infrared microscopy are very similar to that of our double-furnace DSC. It is fast, provides a higher quality of data, and can provide information that other systems cannot because of a lack of sensitivity.

In this applications study, the thermal properties of two single fibers (approximately 1½ inches in length) were characterized using the Power Compensation DSC with the High Speed DSC approach. The objective is to demonstrate that the combination of Power Compensation DSC and the use of very fast scanning rates provide the necessary high sensitivity for obtaining forensic characterization information.

**Experimental**

The following experimental conditions were used to characterize the thermal properties of the single fiber samples. The DSC was calibrated for temperature and enthalpic responses using high purity indium metal.

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<thead>
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<th>Experimental Conditions</th>
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<tr>
<td>Instrument</td>
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<tr>
<td>Heating rate</td>
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<td>Sample mass</td>
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<td>Sample pan</td>
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<td>Initial temperature</td>
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<td>Purge gas</td>
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**Results**

Displayed in Figure 1 are the DSC results obtained on the first single fiber carpet sample. The plot shows the heat flow as a function of sample temperature and an endothermic response is oriented upwards.

The DSC results are very high quality, especially considering that the mass of the sample is only 0.05 mg.
The second single carpet fiber sample was analyzed using the High Speed DSC approach and these results are shown in Figure 3.

These DSC results show that the fiber has a melting peak at 222 °C, with a heat of melting of 262 J/g. These data indicate that this fiber is most likely nylon 6. The thermal properties below the melt are significantly different from those of the other as-received single carpet fiber (Figure 1) and this does suggest that the two different fibers, although both nylon 6, were from two different carpets. The thermal properties obtained by High Speed DSC on these two different carpet specimens are very helpful in determining the nature of the fiber and the possible source/manufacturer of the fibers.
Summary
Two single carpet fiber samples (mass of only 0.05 mg) were characterized using Power Compensation DSC and the High Speed DSC approach. The use of very fast heating rates (100 to 400 °C/min) provides greatly enhanced sensitivity for the successful characterization of very low mass materials. This is especially useful for forensic applications where the quantity of sample is usually very small. In this applications study, the analyses of two different single fiber samples were conducted using High Speed DSC. The data demonstrated that the fibers were most likely manufactured from nylon 6 polymer. The particular thermal characteristics from each of the fiber specimens provide useful “fingerprinting” information for forensic characterization purposes.

Power Compensation DSC and the High Speed DSC approach complement IR microscopy which is traditionally used for forensic purposes. PerkinElmer is the only company supplying both High Speed DSC and IR microscopy.

References