THERMAL ANALYSIS

application note

Characterization of Single Fibers for Forensic Applications Using High Speed DSC

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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 on the crime scene. Fibers are useful for forensic purposes, as they tend to easily cling and do 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 on 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].

The PerkinElmer PYRIS Diamond DSC offers the necessary high performance required for the characterization of single fibers as well as for other demanding forensic applications. The PYRIS Diamond DSC uses the unique and high performance Power Compensation DSC approach for the highest quality research-grade results. The Power Compensation DSC is able to obtain outstanding performance because of the following features:

- Use of low mass (<1 g) individual sample and reference furnaces for rapid response times
- Ability to heat and cool very quickly (up to 500 C/min) for High Speed DSC applications and greatly enhanced sensitivity
- Ability to achieve isothermal conditions rapidly
- Measurement of true heat flow rather than temperature differential for more accurate calorimetric determinations
- Use of PRT or platinum resistance thermometers, rather than thermocouples, for the most accurate and precise measurement of sample temperature
- Unparalleled resolution for separation of overlapping thermal events
- Very high sensitivity for detection of weak or low energy transitions
- StepScan DSC for the isolation of 'fast' and 'slow' thermal events (on the time scale of the DSC experiment). This provides for better data interpretation and for a clearer measurement of the glass transition event (Tg) and other thermal events.

PYRIS Diamond DSC

IR Microscopy for Forensics

The DSC thermal characterization information complements IR microscopy which is widely utilized for forensic applications. The infrared microscope has become essential in the forensic community for identification of trace materials. Single fibers, single crystals, paint chips, lipstick smears, hair-spray on hair fibers, explosives, gun shot residue, and other small particles (down to about 10 micrometers in size) can all be identified by their unique infrared spectrum. Other

PerkinElmer™ instruments.

techniques utilized for these analyses include- GC, GC-MS, SEM (though not so often), Raman (again not so often), optical microscopy, and wet chemical methods. Typically, the forensic chemist would like to have at least two tests confirm the ID of a piece of evidence. Identification is usually confirmed by comparison to a reference spectrum- either in electronic format or in the form of a book of reference data. For the most part, each organic compound provides a unique infrared spectrum.

PerkinElmer Instruments manufactures three types of infrared microscopy systems - The Multi-Scope, The AutoIMAGE, and The Spotlight 300. All systems interface with a standard FTIR. The Multi-Scope is our microscope system for people on a tight budget. It can provide the same information as the other systems but in a manual mode. The Auto-IMAGE system is the standard for performing automated microscopy. All microscope controls are fully automated for ease of use and speed of analysis. The Spotlight 300 system is our newest infrared imaging microscope. It can quickly provide a high-quality infrared image of a small sample in at least 1/10th time of its competitors. PerkinElmer Instrument's strengths in infrared microscopy are very similar to that of our PYRIS Power Compensation DSC -- It is fast, provides 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 1 ½ inches in length) were characterized using the PYRIS 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.

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.

Experimental Conditions

Instrument	PYRIS Power Comp DSC
Heating rate	200 C/min
Sample mass	Approximately 0.05 mg
Sample pan	Standard crimped aluminum pan
Initial temperature	0 C
Purge gas	nitrogen

The DSC results are very high quality especially considering that the mass of the sample is only 0.05 mg.

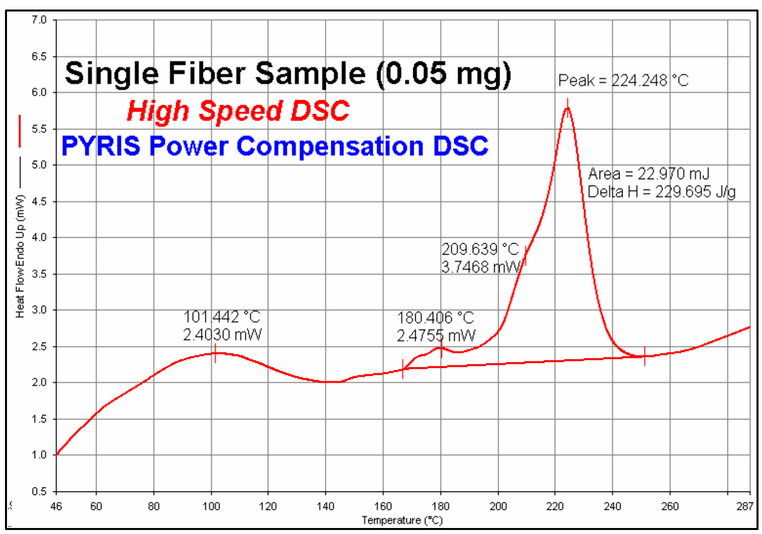

Figure 1. High Speed DSC results on as received single fiber sample

The fiber sample provides a substantial amount of characterization information with the High Speed DSC approach. There is a broad endothermic transition at 101 C and, given the temperature of this particular event, it is a strong likelihood that this is due to the evolution of water. A melting peak is obtained at 224 C with a heat of melting of 229 J/g. Some additional endothermic peaks are observed at 180 and 209 C, and these are most likely due to processing or heat-setting.

The particular thermal characteristics from this fiber specimen indicate that it is most likely nylon 6 [2]. The observed thermal properties of this single fiber are very valuable for forensic purposes since they provide a 'fingerprint' of the material which can aid in tracking down the particular manufacturer of this carpet yarn.

In order to better verify the assignment of nylon 6 for this particular single carpet fiber material, the sample was quenched cooled back to room temperature, and then reheated. The second heating results at a rate of 200 C/min are displayed in Figure 2. These reheat DSC results do confirm the assignment of nylon 6 as the polymer for this particular carpet sample. The DSC shows a glass transition (Tg) of 61 C, a cold crystallization event at 98 and a melting peak at 220 C. All of this information is consistent with the properties of nylon 6.

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.

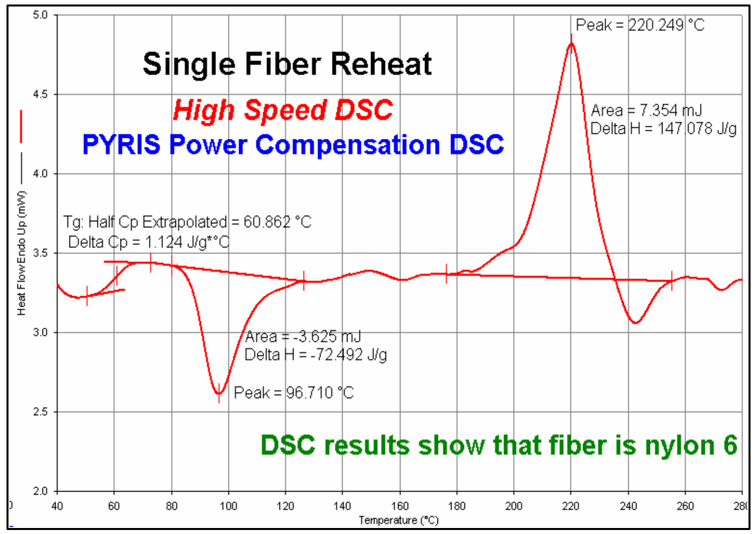

Figure 2. High Speed DSC results on quench cooled fiber sample

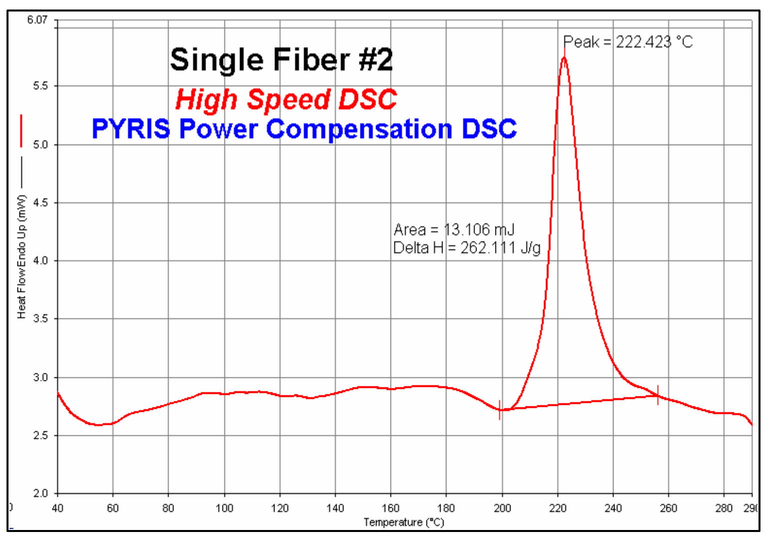

Figure 3. High Speed DSC results on second single fiber sample

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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 Instruments is the only instrument company supplying both High Speed DSC and IR microscopy.

References

[1] W.J. Sichina, PerkinElmer TEA TechNote PETech-50
[2] R.P. Chartoff, *Thermal Characterization of Polymeric Materials*, 2nd edition, Edith Turi, editor, Academic Press (1997), p. 606.

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