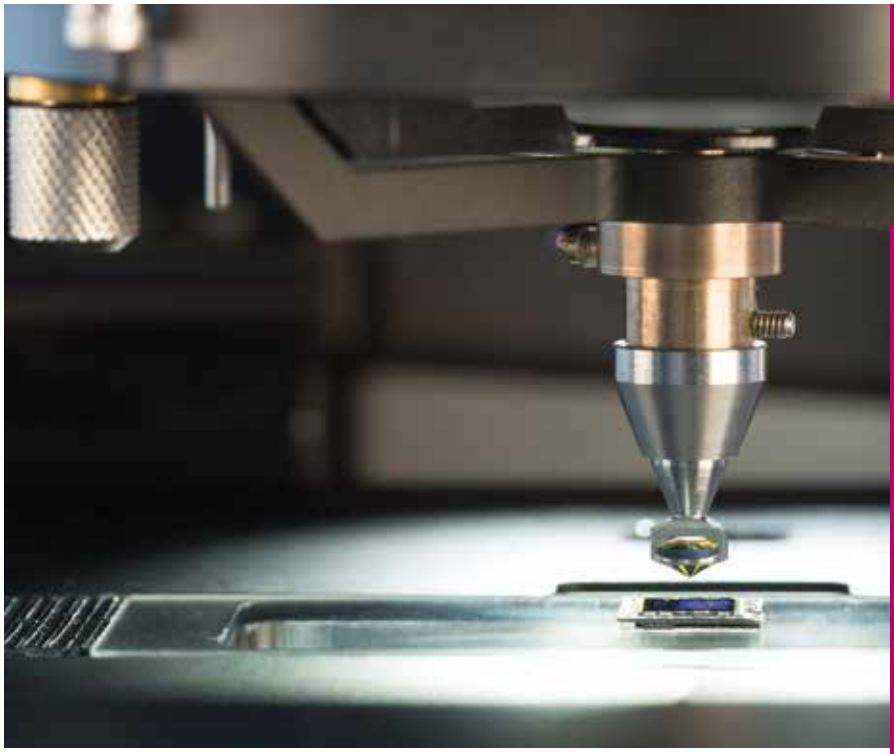

APPLICATION NOTE

Infrared, IR Microscopy

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Rapid Characterization of Multiple Regions of Interest in a Sample Using Automated IR Microscopy

Introduction

IR microscopy is a well-established analytical technique for the measurement and identification of small samples down to a few micrometers in size. It is used extensively in the polymer, pharmaceutical, chemical, food, and electronics industries, to name a few, often identifying small contaminations or foreign objects of unknown origin. In forensic applications small particles of materials such as drugs, paint chips, residues or fibers are often collected as evidence and analyzed by IR microscopy.

The type and size of the material, as well as the matrix in which the sample is contained, will dictate the type of IR microscopy sampling technique to be deployed; transmission, reflectance, or ATR.

The Spotlight™ 200i IR microscope is a fully automated system comprising:

- Automated X, Y, Z stage
- Automatic illxumination LEDs
- Autofocus
- Auto correction
- Automated switching between transmission and reflectance
- Automated dropdown ATR crystal

All of these features are controlled using the Spectrum 10 software.

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Traditionally, measurement of a sample on an IR microscope involves several manual steps to find and specify the regions of interest for the analysis and manual processing of the collected data. All of which can be very time consuming. These processes have now been fully automated within the Spectrum 10 software, using intelligent detection routines for the typical types of samples measured on an IR microscope: particles, multilayer samples, and sample inclusions.

This Application Note will demonstrate the advantages of such an automated IR microscopy platform for the characterization of particles and/or foreign objects in different types of materials.

Automated detection and analysis of microplastics extracted from a cosmetic formulation

An example of this automation is the detection and classification of microplastic particles extracted from a cosmetic formulation. Cosmetic exfoliating agents contain small microplastic particles as the abrasive material to scrub the skin. These microplastics make their way into the river systems and ultimately into the marine environment where they are serious pollutants. A commercially available product was mixed with hot water to dissolve the soluble ingredients in the formulation. The resulting solution was filtered using a 50-micrometer mesh, capturing any insoluble components greater than 50 micrometers in size. The filter was allowed to dry before transferring the residual particles onto an IR transmitting window on a microscope holder. A Visible Image Survey was collected over the area containing the majority of the particles. Selecting the "Analyze Image" icon in the Spectrum 10 software calls up the intelligent, automated routine for analyzing the image, as shown in Figure 1.

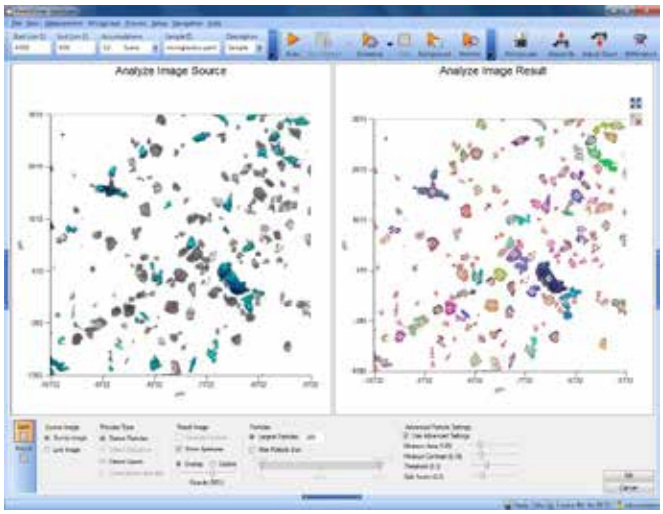

Figure 1: The "Analyze Image" function detected filtered particles deposited on a KBr window.

This routine will detect any particles present in the visible image and mark them as regions of interest. It will then calculate the maximum rectangular aperture size that can fit wholly inside each of the particles, thus minimizing signal-to-noise when the data is scanned. (In the past manual selection of the regions of interest and setting of apertures took a considerable amount of time.) Clicking "Scan Markers" will then initiate collecting transmission spectra (using equivalent apertures for the background) for each sample, displaying ratioed sample spectra in real time as they are collected. Automatic processing of the spectra, such as Search, Compare or Verify, will be performed during data collection. In the case of the analysis of the microplastics a spectral search was performed against a library of polymer spectra to give the identity of each of the particles.

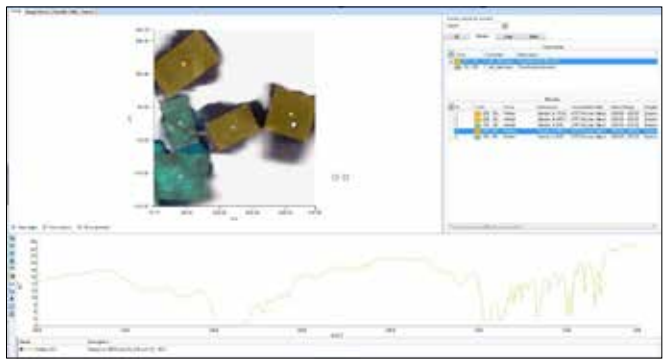

Figure 2: Results screen for detection and identification of particles.

Two different polymer types were detected in this sample, identified as polyethylene and polypropylene. The spectra are shown in Figure 3.

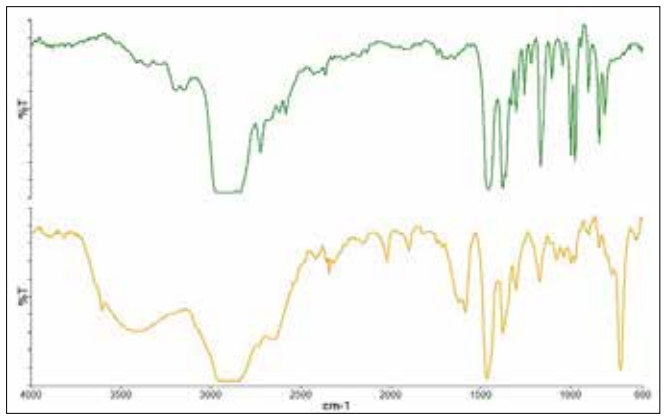

Figure 3: Spectra of the two different polymer types are shown here. Top: polypropylene, Bottom: polyethylene.

Automated detection and analysis of contaminants on an electronic contact

Electronic contacts need to be clean and free from contamination to avoid problems in operation. A sample was submitted for analysis that had visible contaminants. The sample was placed in the Spotlight 200i and a "Visible Image Survey" collected over the entire contact. The resulting image was then analyzed using the "Detect Particles" function in the Spectrum 10 software in an attempt to find any contamination. The Visible Image Survey and an expanded region showing the particles detected are shown as Figure 4.

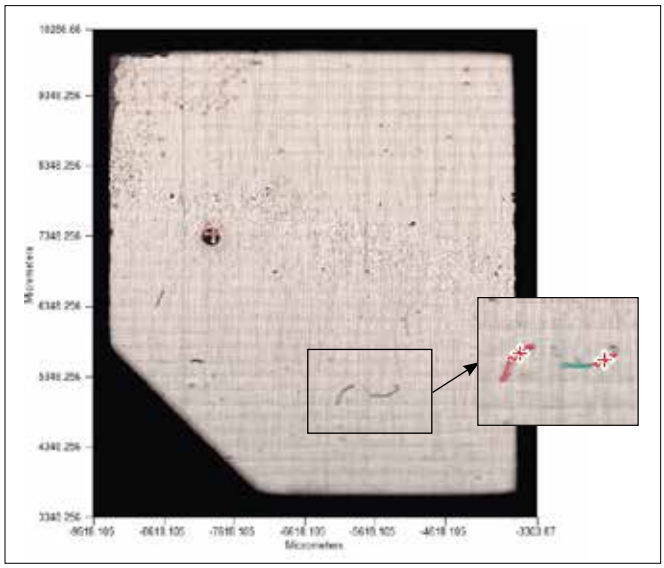

Figure 4: The Visible Image Survey and expanded region show automatic detection of contaminants.

After selecting "Scan Markers", the software automatically collected reflectance backgrounds and spectra for the particles (fibers), their spectra shown as Figure 5.

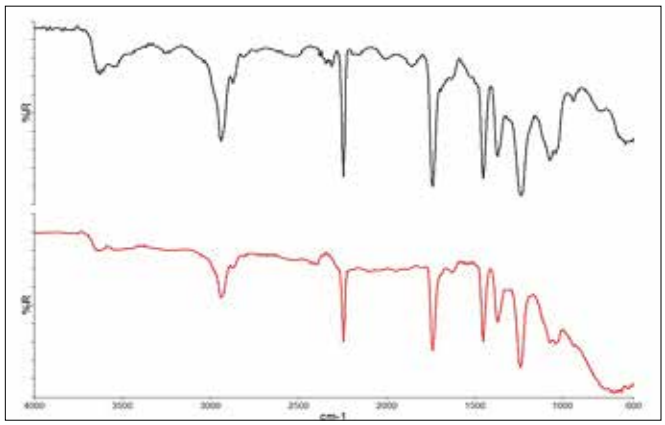

Figure 5: Reflectance spectra of the two contaminant fibers.

The spectra of these two materials are similar with the lower spectrum showing an additional broad peak centred around 700 cm-1. The top spectrum was identified as an acrylonitrile-butyl methacrylate copolymer by searching the spectrum against a spectral library of polymers and polymer additives. Since the lower spectrum clearly has another component present, it was subjected to a mixture search that also detected the presence of tin oxide in the sample.

Automated ATR analysis of layers in a polymer laminate

ATR is a convenient sampling technique requiring minimal sample preparation that has been routinely applied within the polymer industry. An automated drop-down ATR crystal on an IR microscope allows for automated measurement of polymer samples, including layers of multilayer laminates. A multilayer polymer card was clamped in a sample holder and placed on the stage of the Spotlight 200i. A "Visible Image Survey" was recorded over a 2 mm x 2 mm area of the sample and the automated "Detect Layers" function in the Spectrum 10 software was applied, as shown in Figure 6.

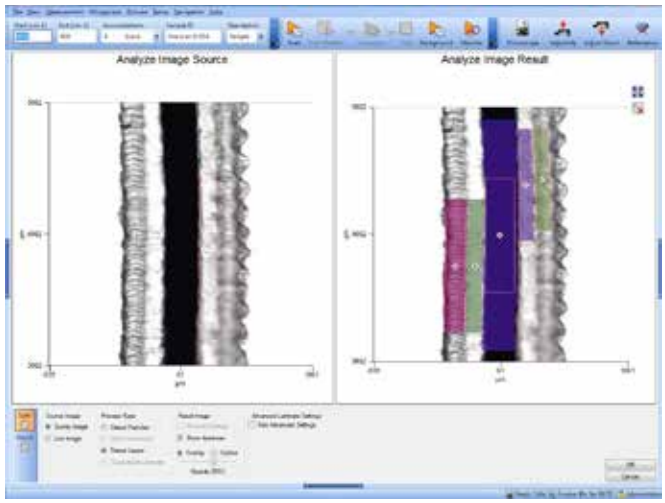

Figure 6: Automated detection feature of the Spotlight 200i show multiple layers in a polymer laminate.

Five different layers were detected in the image and markers placed in the center of each layer. Clicking "Scan Markers" will automatically collect the background scans then move to each of the markers, lower the automated ATR crystal onto the sample (Figure 7), and measure the spectra. The spectra obtained from each of the layers are shown in Figure 8 and were identified by comparison against search libraries.

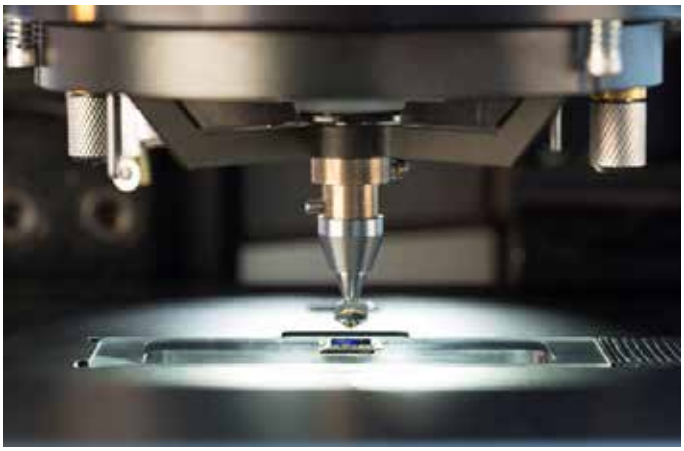

Figure 7: The automated dropdown ATR crystal.

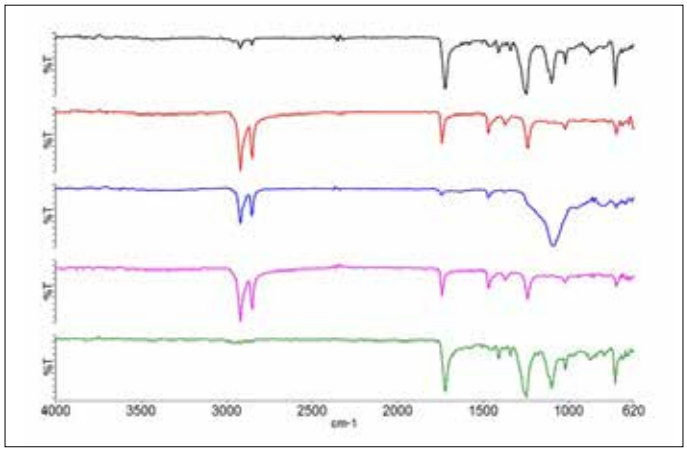

Figure 8: Spectra of the five layers.

The layers were identified from top to bottom as Polyethylene terephthalate (PET), ethylene-vinyl acetate (EVA) co-polymer, silica-loaded polyethylene, another layer of EVA and another layer of PET.

Summary

The Spotlight 200i, an intelligent automated IR microscope system, is able to simplify and dramatically speed up the process of collecting and analyzing spectra from a variety of sample types. The automation has been applied to all sampling modes: Transmission, Reflectance, and ATR, as well as a variety of different sample types: particles, fibers and multi-layers.

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