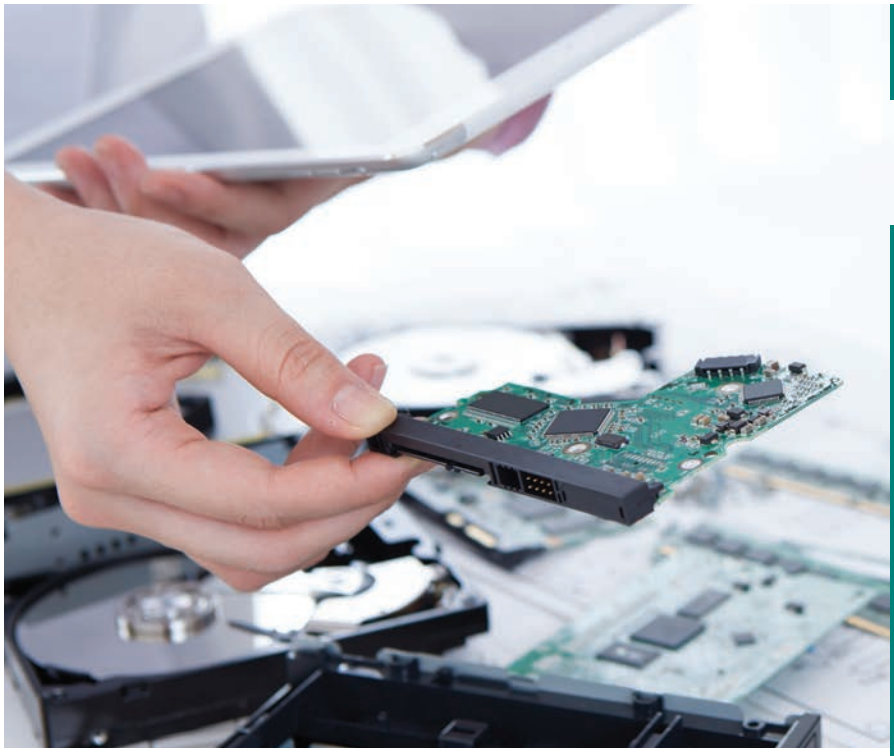

APPLICATION NOTE

Infrared, IR Microscopy

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Detection and Identification of Contaminations in the Manufacturing Process Using an IR Microscope

Introduction

Manufacturing processes are designed to create products that contain only the required components. Occasionally, foreign objects can appear within the final product as unexpected contaminations effecting product quality or even causing product failure. Investigation is required to determine what the contamination is and where it has originated. Infrared spectroscopy is one of the primary analytical techniques for the identification of materials. If the contaminant is large enough that it is visible to the naked eye then a simple (macro) IR measurement can usually be performed on the sample. In many industries, such as electronics and polymers, even micro-contaminants can cause product problems. IR microscopy allows samples as small as a few micrometers in size to be analyzed and identified and is the ideal solution for these types of problems. A range of IR microscope sampling modes (transmission, specular reflectance, and Attenuated Total Reflectance) allow spectra to be measured for contaminants in a variety of sample matrices.

This Application Note describes the use of the Spotlight™ 200i system, an automated IR microscope for the detection and measurement of different types of contaminants in a range of manufactured samples.

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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, as shown in Figure 1. The "Detect Particles" function in the Spectrum 10 software found some contaminant particles as shown in the enlarged box of Figure 1.

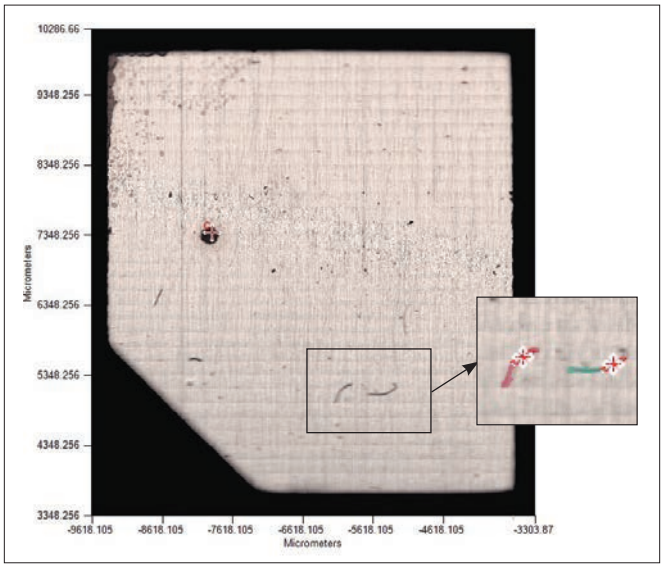

Figure 1: The Visible Image Survey and expanded region, Figure 1b, showing automatic detection of contaminants.

The software then automatically collected reflectance backgrounds and spectra for the particles (fibers), with their spectra shown as Figure 2.

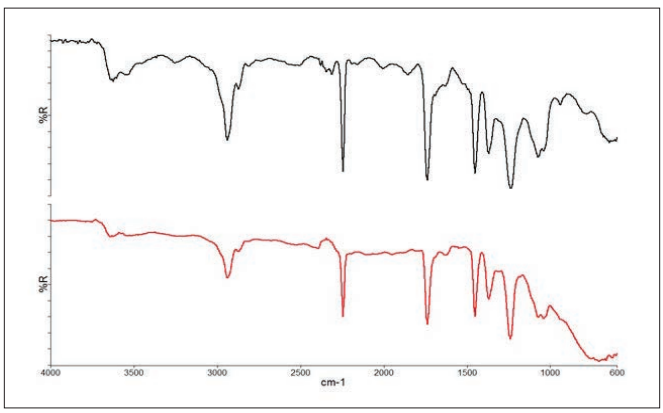

Figure 2: Reflectance spectra of two contaminant fibers.

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

Fiber Contamination on a Pharmaceutical Tablet

A pharmaceutical tablet was observed to have visible contaminants present, appearing to be on the surface. It was unclear as to whether this contamination occurred during manufacturing or at a later stage from external contamination. The sample was observed using the visible image in reflectance on the Spotlight 200i, as shown in Figure 3 and is seen to be a fiber. The image shows that a large proportion of the fiber is embedded under the surface of the tablet and hence could not have simply fallen onto the surface post-manufacturing.

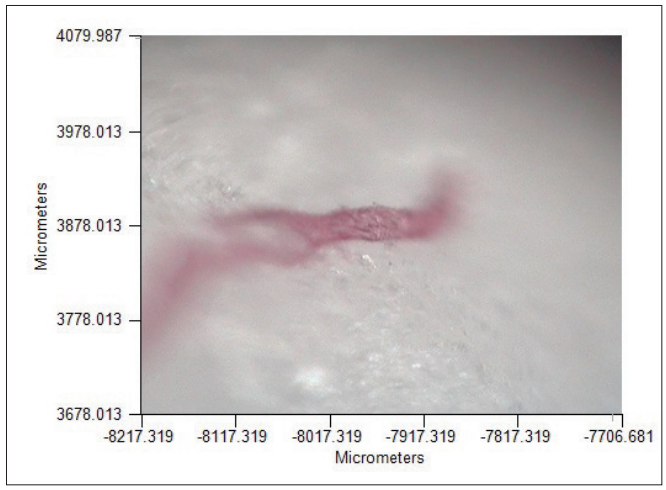

Figure 3: The visible image of a fiber embedded in pharmaceutical tablet.

Due to the fiber being embedded in the sample and covered in excipients, it was not possible to perform a direct ATR measurement. In such cases it is necessary to physically remove the fiber from the sample matrix. The sample was then placed on a KBr window, Figure 4, and the sample spectrum measured in transmission, the spectrum is shown in Figure 5.

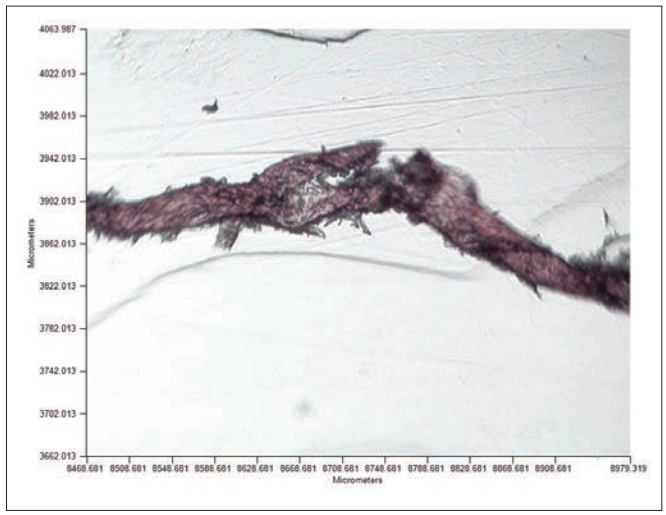

Figure 4: The visible image of the extracted fiber on KBr window.

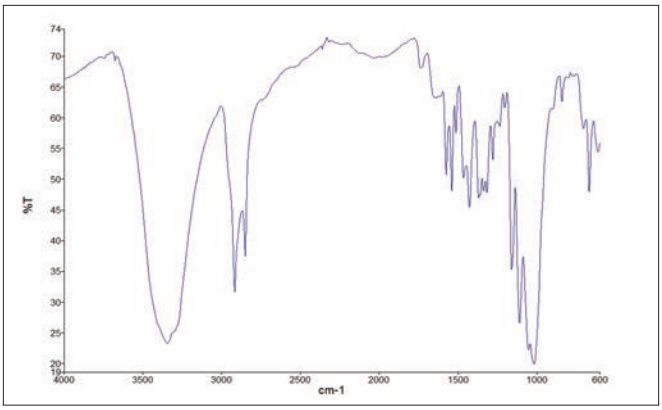

Figure 5: Transmission spectrum of fiber extracted from pharmaceutical tablet.

The spectrum obtained still contains spectral features (broad – OH in the region 3400 cm^{-1} and C-O band at ~1020 cm^{-1}) due to the microcrystalline cellulose,one of the excipients. However, performing mixture search allowed the fiber material to be identified as a chlorinated polyethylene as shown in Figure 6.

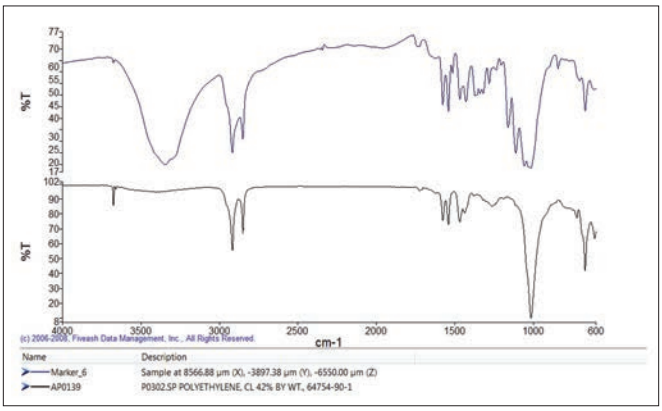

Figure 6: Transmission spectrum of fiber extracted from pharmaceutical tablet.

Summary

IR microscopy is an invaluable technique for identifying small contaminations in the manufacturing process, and we have demonstrated how an automated IR microscope is suitable for their rapid detection and measurement Contaminants can often be automatically detected, scanned and identified by the software. The range of IR microscopy sampling modes means that samples can often be measured in-situ, speeding up the process of identification. However, there are cases where the contaminant may need to be physically removed from the sample matrix in order to provide higher quality spectra free from matrix interference.

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