APPLICATION NOTE

Infrared (IR) Microscopy

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Analysis of Automobile Paint Chips Using an Automated IR Microscope

Introduction

The information obtained from paint chips involved in road traffic accidents is extremely important for piecing together evidence in criminal cases. Traces of paint can be transferred from a vehicle onto other surfaces or materials, such as victims clothing, and these can be matched to the paint type of the vehicle. This is achievable since the paint chips are multi-layered materials consisting of several coats of paint. The layer combinations are unique for an individual manufacturer, model, color, and year of a particular vehicle. Infrared (IR) spectroscopy is a standard technique used for the measurement of paint samples with ASTM method E2937 - 13 acting as a standard guide for using infrared spectroscopy in forensic paint examinations. Infrared microscopes are routinely used for measuring extremely small paint samples down to a few micrometers in size allowing spectra to be recorded for each of the layers.

This Application Note describes the use of the different sampling modes and automation features of the Spotlight™ 200i IR microscope system applied to an automobile paint chip sample retrieved from the roadside at the scene of a road traffic accident.

There are three main sampling techniques for infrared spectroscopy of solid samples: transmission, (specular) reflectance, and Attenuated Total Reflectance (ATR). All of these sampling techniques can be applied to standard (macro) IR accessories as well as IR microscopes for microsamples. Each of these techniques has been applied to this sample on the IR microscope and the relative advantages and disadvantages of each are described.

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Reflectance

Specular reflectance measurements are obtained from direct reflection from the cross-section surface of the sample. Reflectance sampling mode requires minimal sample preparation as the sample is simply secured in a small clamp device and placed on the stage of the IR microscope. The visible image from the sample in reflectance in Figure 1 shows that there are multiple layers present.

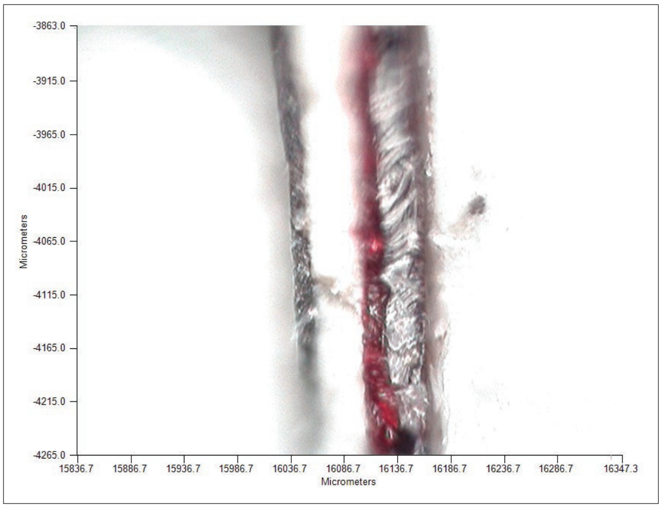

Figure 1: Visible image of cross section of a paint chip sample on IR microscope.

Sample preparation and spectral data are easy using the reflectance technique. However, it is not the optimal technique, since there are distortions in reflectance spectra due to refractive index changes and peak shifts. Therefore, reflectance spectra are generally not suitable for comparison against reference spectra that have, in the vast majority, been recorded in transmission. Reflectance spectra from the layers in this sample are typical of a reflectance spectrum from a paint sample and are shown as Figure 2. The background spectrum was recorded using a gold mirror reference.

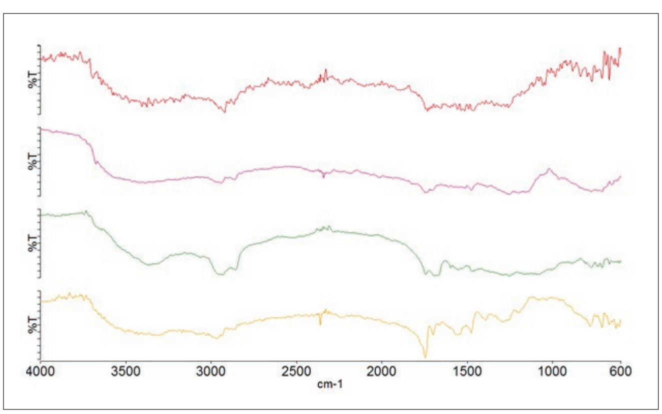

Figure 2: Specular reflectance spectra of paint layers.

Attenuated Total Reflectance (ATR)

The technique of ATR is also a surface technique with the IR radiation only penetrating 1-2 micrometers into the sample. Therefore, the sample to be measured can be a thick sample requiring less sample preparation; a simple cut of the sample to generate a cross section of the layers is sufficient. The sample for ATR can be supported in a clamp device for the microscope or embedded in a resin block and polished. Embedding provides better support and avoids distortion of the sample when pressure is applied using the ATR crystal, also aiding to preserve it, allowing repeat measurements if required. A section of the paint chip was embedded in a resin for these ATR measurements, as shown in Figure 3.

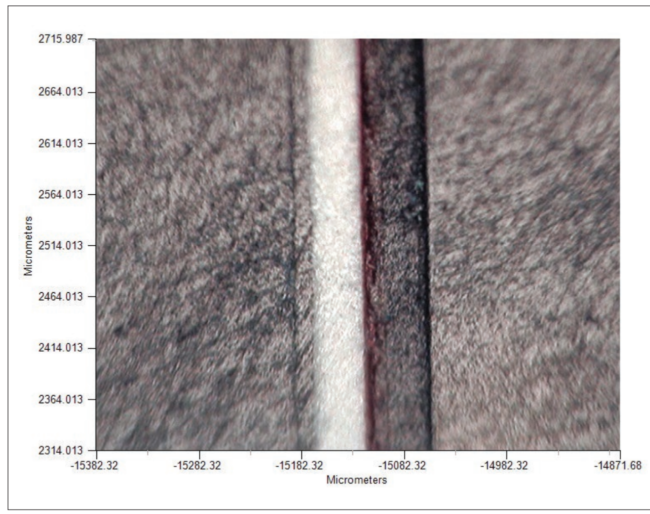

Figure 3: Visible image of a cross section of a paint chip embedded in resin for ATR measurements.

The Spotlight 200i system can be fitted with an automated drop-down ATR crystal (100 micrometer tip size) that allows measurements to be performed at discrete points on the sample, or allows linescans or maps to be collected. An alternative is to use the macro ATR accessory for the IR microscope. The crystal on this accessory is clamped across the entire sample allowing direct measurements anywhere on the sample without any crystal movement. This gives a significant improvement in the spatial resolution that can be achieved, allowing thinner layers to be measured 1. A linescan measurement was performed across the paint chip using the Macro ATR accessory with the data shown in Figure 4.

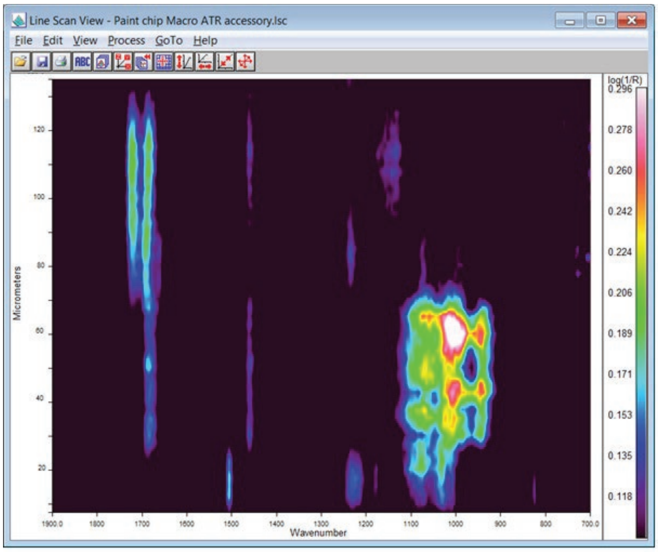

Figure 4: ATR linescan data for paint chip.

The spectra obtained for the different layers are shown in Figure 5.

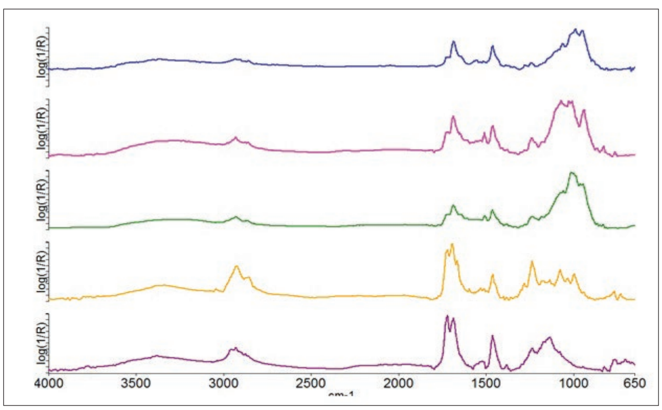

Figure 5: Spectra obtained for the different layers in the ATR experiment.

The advantage of ATR spectra over specular reflectance spectra is that ATR spectra closely resemble those of the equivalent transmission spectrum. ATR correction processing routines will correct for the wavelength-dependent intensity differences between ATR and transmission spectra, allowing ATR spectra to be compared against the extensive library databases of transmission spectra. Spectra are shown in Figure 6 comparing the ATR spectrum with the transmission sample for the same paint layer demonstrating the equivalence of the spectra using the two techniques.

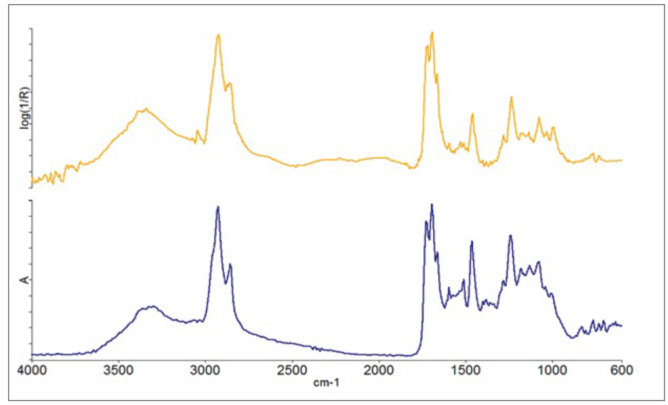

Figure 6: ATR-corrected spectrum (top) and transmission spectrum (bottom) for one of the layers in the paint chip sample.

Transmission

IR transmission measurements generally require the sample to be in the region of 10 to 20 micrometers thick in order to avoid totally absorbing bands. For paint chip samples, this requires the sample to be microtomed to an acceptable thickness prior to measurement. The paint chip sample was microtomed and placed onto the surface of a potassium bromide (KBr) window, shown in Figure 7.

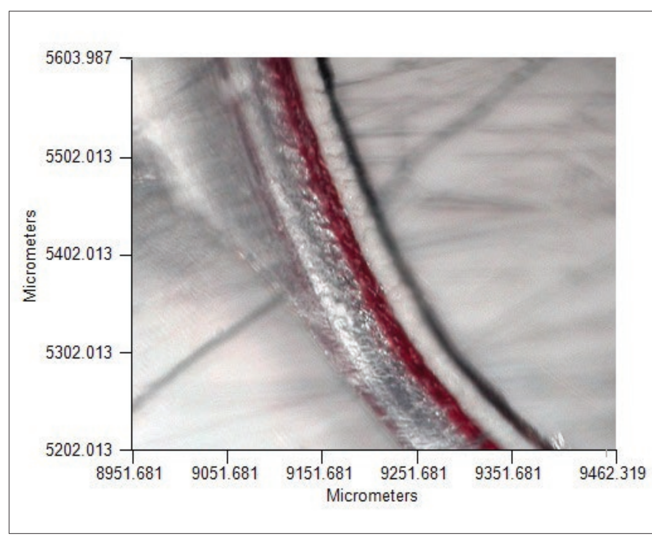

Figure 7: Microtomed paint chip sample is shown on KBr window for transmission measurement.

In order to obtain spectral information from all the layers in the sample a linescan was set up to measure spectra at five-micrometer intervals across the entire width of the paint chip. The linescan data is shown as Figure 8.

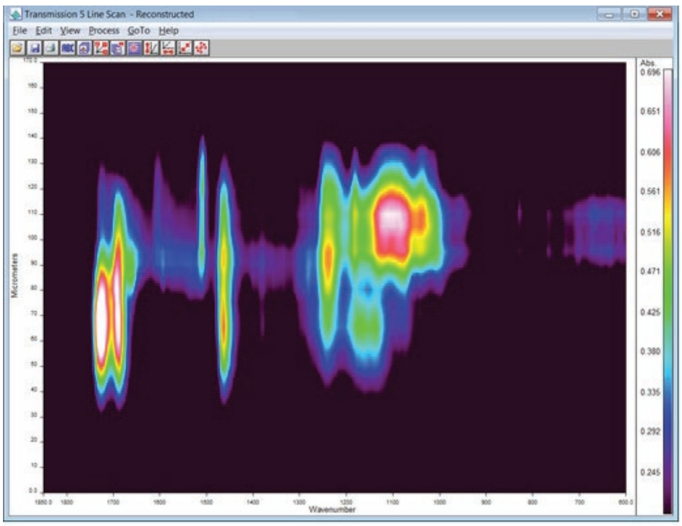

Figure 8: Transmission linescan data for paint chip sample.

The linescan data shows different spectral features in different areas of the sample, which represent the different layers. Spectra for each of the layers were extracted from this linescan data and are shown in Figure 9.

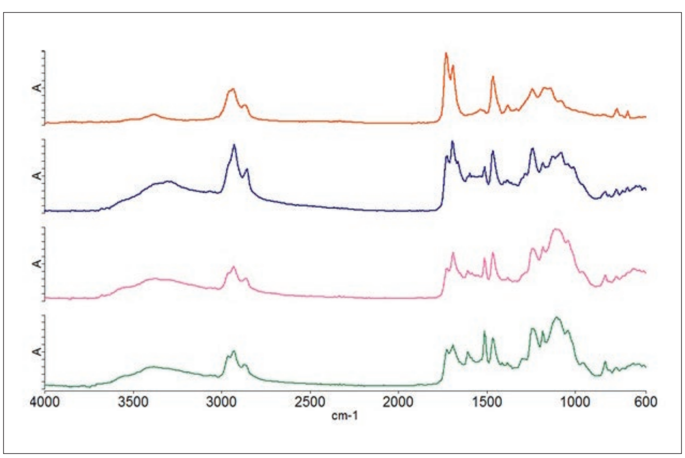

Figure 9: Shown here are transmission spectra of the layers in the paint chip.

The transmission spectra can be compared against standard libraries of paints and additives in order to identify the layers present.

Summary

IR microscopy is an invaluable technique for the measurement of the multiple layers of paint chips. The choice of sampling technique will be dependent on the sample preparation techniques available to the user and the information required from the spectrum.

Specular reflectance offers a no-sample preparation solution and will be able to show spectral differences between layers, but the spectra will contain distortions and cannot be directly identified or compared to transmission spectra libraries.

ATR also offers a no-sample preparation solution if the sample is held in a clamp device on the microscope sample stage. However, embedding the sample is advantageous. The ATR spectra are directly comparable to transmission spectra and libraries once ATR correction has been applied to adjust the relative intensities of the spectral bands. The use of the macro ATR accessory for the microscope will generate better spatial resolution, allowing thinner layers to be measured.

Transmission is the preferred method for measuring paint samples, since transmission spectra are directly comparable to existing spectral databases and reference spectra. In addition, transmission measurements do not exhibit distortions or require the mathematical corrections required for reflectance and ATR spectra. However, this measurement requires the sample to be no thicker than 15 to 20 micrometers, thus requiring some level of sample preparation of the samples to be microtomed.

References

1. PerkinElmer Technical Note 007641A-03, Spatial Resolution in ATR Imaging

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