No. 40401

SPATIAL RESOLUTION OF MICRO-ATR INFRARED SPECTROSCOPY

Figure 1. The Video Meridain™.

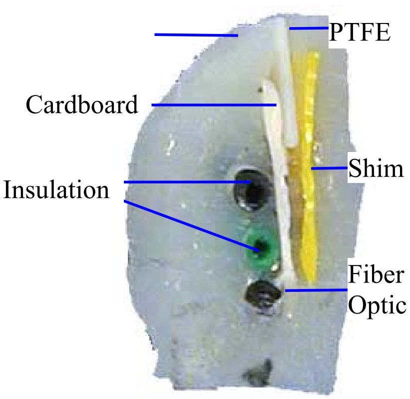

Figure 2. Epoxy Imbedding.

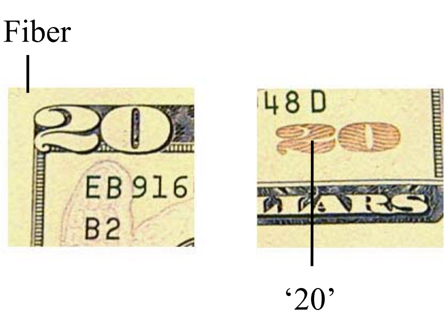

Figure 3. The two sections examined from the face-side of the new twenty-dollar bill.

INTRODUCTION

With the recent growth in single reflection ATR infrared spectroscopy, there has been a push to examine ever smaller samples or segments thereon. This method is limited by the ability to position the sample on the active sampling area of the ATR crystal and by the relative size of the sample to the sampling area. Two categories of ATR micro-samplers are currently available: microscope ATR objectives and in-compartment accessories. While the microscope ATR objectives offer higher spatial resolution, the required MCT detector restricts the spectral range. The in-compartment accessories offer greater spectral range.

This paper explores the spatial resolution of this technique, using a new micro-ATR in-compartment accessory with imaging capabilities. Several samples will be examined, including imbedded materials, inks on paper and fibers.

EXPERIMENTAL

The ATR spectra were recorded using an FT-IR spectrometer, configured for data collection at 8 cm^{-1} resolution and 32 scans. The spectrometer was equipped with Harrick Scientific's Video Meridian™ diamond ATR (Figure 1). Background spectra were collected from the clean ATR crystal and then the samples were compressed against the horizontal diamond ATR. The calibrated pressure applicator with digital read-out was used to apply 100 units of force. The spectra were then collected and the sample photographed using the built-in camera.

Several sections of two different samples were examined: an epoxy imbedding (Figure 2) and a new twenty-dollar bill (Figure 3). The imbedding was prepared by inserting several materials parallel to each other in Devcon® (ITW) 5-minute epoxy and then allowing the epoxy to dry thoroughly. The imbedding was then sliced to expose a clean surface of the materials. The materials imbedded included: 500 µm thick PTFE, 500 µm thick shim stock, cardboard (approx. 500µm thick), black and green insulation from 18 gauge multi-strand wire insulation and a fiber optic (Edmund Scientific P/N 2504).

RESULTS AND DISCUSSION

First, several samples of known thickness were examined to determined if they could be spatially separated from the background material in the embedding. For reference, the spectrum of the epoxy imbedding material is shown in Figure 4. The spectrum has increased noise in the 2100 cm^{-1} region due to the strong absorption bands of the diamond ATR crystal.

Figure 5 demonstrates the ability to resolve 500 µm thick samples from the surrounding material. No features due to the epoxy are present in either spectrum. Figure 5 shows the spectrum of PTFE, as expected, and the shim is readily identifiable as polyester. The photographs in 5 also clearly delineate the edges of the samples, reasserting the fact that the octagonal sampling area of the ATR crystal is 500 µm in diameter.

Several materials of unknown thickness and/or composition were then investigated within the imbedding. Figure 6 is the ATR spectrum of a piece of thin cardboard. It is interesting to note that the cardboard spectrum is clearly that of cellulose. There is no observable contribution from the epoxy, despite the fact that cardboard is somewhat porous.

Figure 7 shows the spectra of two different colors of wire insulation. The photograph of the green insulation shows the curved shape of the sample. The edges of this sample are also well defined, indicating that the wall thickness of this insulation is roughly that of the sampling area. Both spectra are distinct from that of the imbedding material.

Figure 8 contains spectra and photographs of a fiber optic. From this data, the black sheathing has a wall thickness of roughly 500 µm and is composed of a low density polyethylene, while the individual fibers are approx. 250µm in diameter and made from poly(methyl methacrylate).

Having determined that it is possible to spatially resolve a carefully formulated sample, a readily available sample was also examined – a new twenty-dollar bill. Figure 9 shows the spectra of two sections of the golden '2' on the face side of the bill. The photographs show that this section is actually composed of red and gold stripes, roughly 500 µm in width. The spectra of both stripes are similar, but the bands in the ATR spectrum of the golden stripe are lower in intensity. This would be expected if the golden coating is actually a thin layer of gold, since the gold would mask the spectrum of the underlying paper.

Figure 10 show the ATR spectra from two sections of the bill. One section has a noticeable blue fiber in it that, from the photograph, is approximately 65 µm in diameter. The other was more

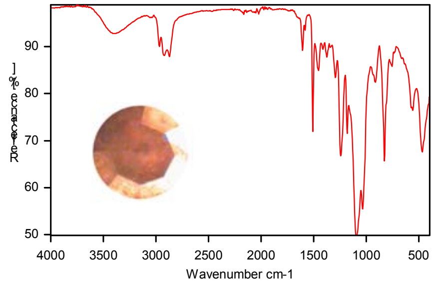

Figure 4. ATR Spectrum of Epoxy.

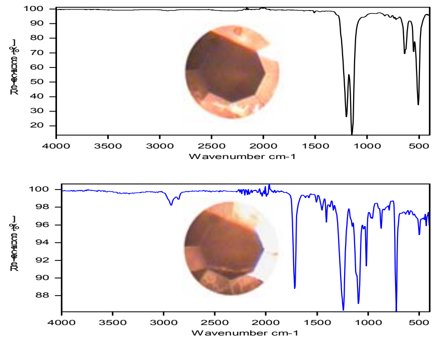

Figure 5. ATR Spectrum of 500 µm thick PTFE (upper) and Shim (lower).

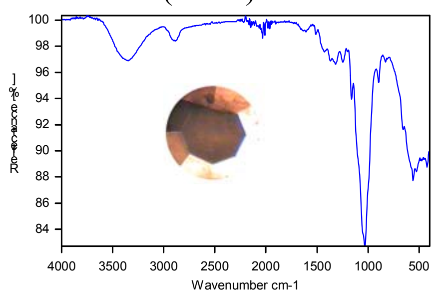

Figure 6. ATR Spectra of ~500µm thick Cardboard.

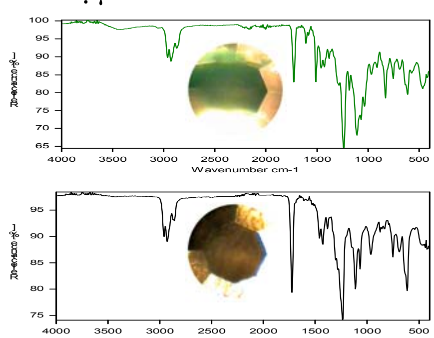

Figure 7. ATR Spectra of Green (upper) and Black (lower) Insulation.

uniform and examined as a

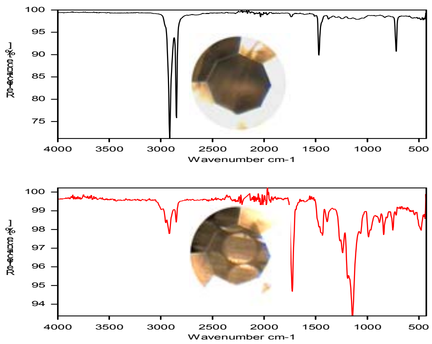

Figure 8. ATR Spectra of a Fiber Optic: Sheathing (upper) and Interior (lower).

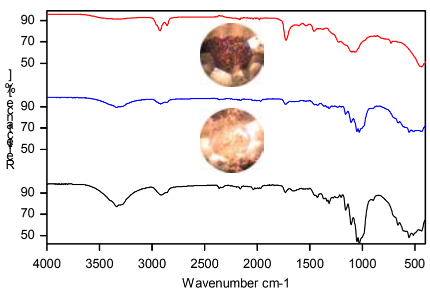

Figure 9. ATR Spectra of the shiny "20" on the face side of the bill: red section (upper), gold section (middle) and an unprinted section (lower).

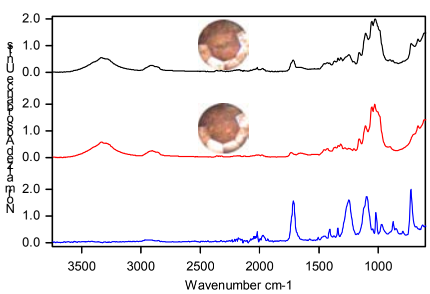

Figure 10. Normalized ATR Spectra of a blue fiber (top) on the greenback, an area near that blue fiber (middle) and the difference (bottom).

reference. The bands at 1715 cm^{-1} and 1250 cm^{-1} appear in the spectrum of the fiber but not in the spectrum of paper. Subtraction results in a spectrum that is clearly identifiable as polyester.

CONCLUSION

With the use of the Video Meridian™ micro-ATR accessory, it is straightforward to spatially resolve samples such as imbedded materials, inks on paper and fibers. In fact:

- 500µm-wide sections of materials can be readily resolved spatially from bulk substrates.
- Smaller segments can also be resolved through judicious use of spectral subtraction methods.

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