MicroLap Depth Profiling of a Paper Coating

Measuring FT-IR spectra as a function of depth is desirable for analysis of layered and gradient composition samples. PAS and ATR provide variable sampling depths but often cannot measure as deep as is desired, are susceptible to spectral interferences and do not directly provide layer-by-layer spectra. Microtoming of samples to get layer-by-layer depth information has been a useful method for avoiding these limitations but requires handling of extremely delicate slices and expensive equipment. Microtoming is also only applicable to materials that can be sliced.

The MTEC MicroLap System provides a cost effective alternative to microtoming that avoids handling of slices, provides layer-by-layer spectra with depth resolution on a micrometer scale, and is applicable to nearly all planar materials. FT-IR spectra are measured layer-by-layer prior to abrasive removal of each successive layer of material. Measurements are made by attaching the sample to a lapping puck, zeroing a micro-gage, measuring a spectrum of the top layer by PAS or ATR, removing several micrometers of material with the micro-lapper, and repeating the cycle as shown in Figure1.

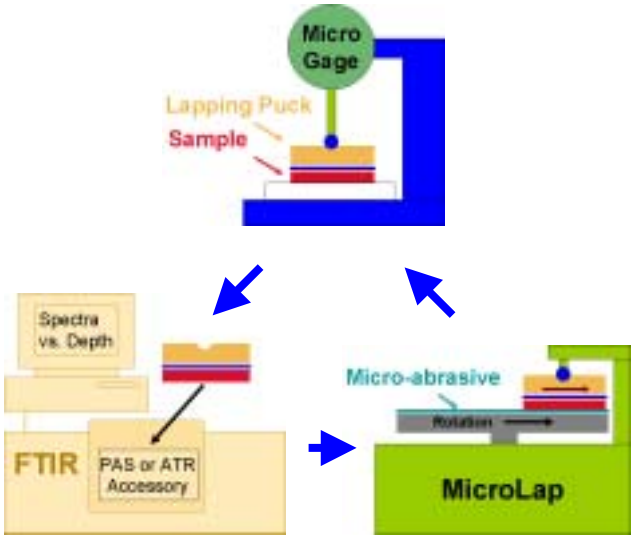

Figure 1. Schematic of the measurement cycle for measuring spectra layer-by-layer with the MicroLap system.

MicroLap can be used to depth profile materials that are difficult to microtome such as coatings on paper. Figure 2 shows the composition profiles for three paper coating components, calcium carbonate, kaolinite, and latex measured using the MicroLap device.

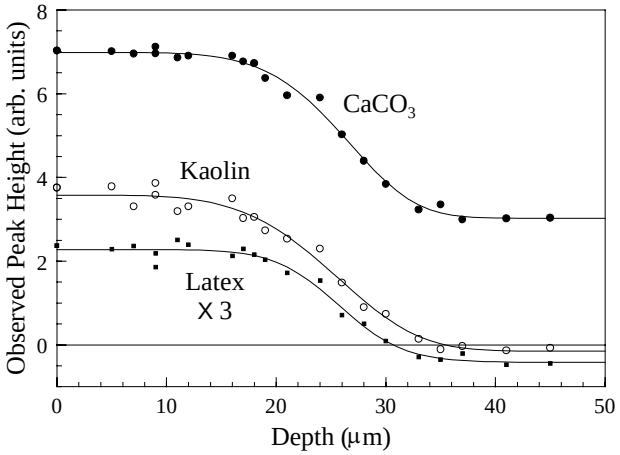

Figure 2. Depth profiles of three paper coating components.

Figure 3 shows typical MicroLap FT-IR PAS spectra of a coated paper sample used to create the profile plots shown in Figure 2. The spectra were measured at 8 cm^{-1} resolution and 20 kHz mirror velocity. The plot shows the initial spectrum (0 µm) followed by spectra from the gradient range (17 – 33 µm) where composition was found to be changing and ending at 45 µm where the coating components have minimal depth variation. The pure component spectra are also shown for reference. The points plotted in Figure 2 are the spectrum amplitude differences for the wavenumber positions as follows: calcium carbonate (875.68 cm^{-1} minus 821.67 cm^{-1}); kaolinite (3618.45 cm^{-1} minus 3599.16 cm^{-1}); and latex (the average of 702.09 and 698.23 cm^{-1} minus the average of 609.15 and 686.66cm^{-1}).

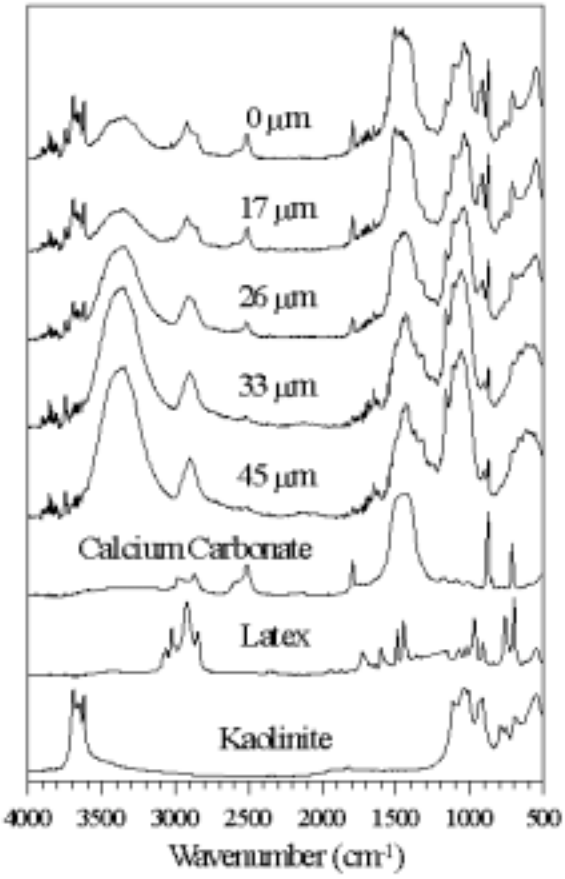

Figure 3. FT-IR PAS spectra of a paper coating as a function of depth measured with the MicroLap device. Reference spectra of the three coating components are also shown.

If the concentration profiles of Figure 2 are scaled to be equal on the ordinate axis, variations can be observed in the gradients of the three components as shown in Figure 4. The data for each of the components were fit to asymmetric sigmoid functions using SigmaPlot software to obtain the curves in Figure 4.

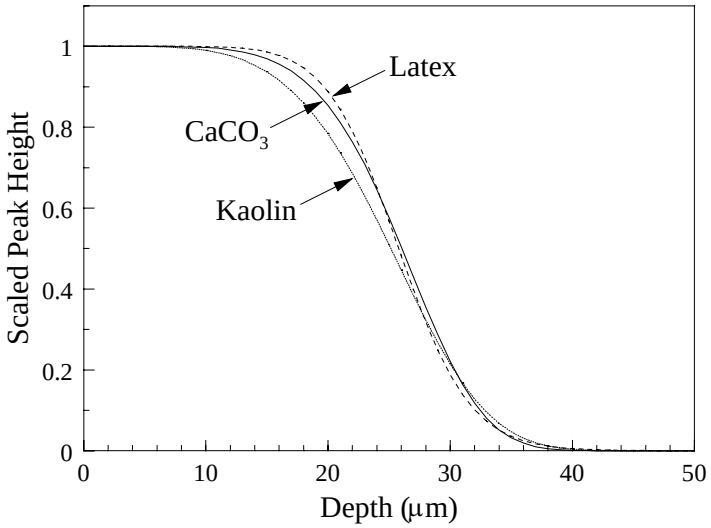

Figure 4. Concentration profiles for a three component paper coating measured with the MicroLap device.

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