

MicroLap Depth Profiling of Automobile Paint Weathering

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 Figure 1.

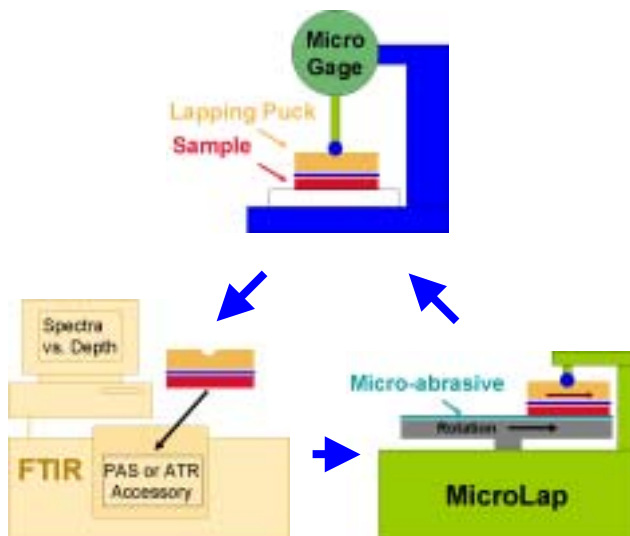


Figure 1. Schematic of the measurement cycle for measuring spectra layer-by-layer with the MicroLap system.

The outer clear-coat layer of automobile finishes is typically 50 micrometers thick and protects the pigmented and undercoat layers from weathering degradation caused by UV radiation induced radical creation followed by chemical reactions which damage the finish. Typically clear-coats are composed of a melamine cross-linked acrylic copolymer with a UV absorbing stabilizer. The acrylic clear-coat samples studied here included an unweathered retained sample and an exposed sample that was weathered for four years in Florida, USA.

Figure 2 shows the variations in PAS peak heights for exposed and retained samples with depth into the 50 micrometer clear-coat. The functional groups of Figure 2 are depleted at the surface by weathering whereas they are more concentrated for the retained sample. The groups include melamine ring C-N stretch (1558 cm^{-1}), ether cross link (1096 cm^{-1} and 1026 cm^{-1}), melamine-melamine bond (1381 cm^{-1}).

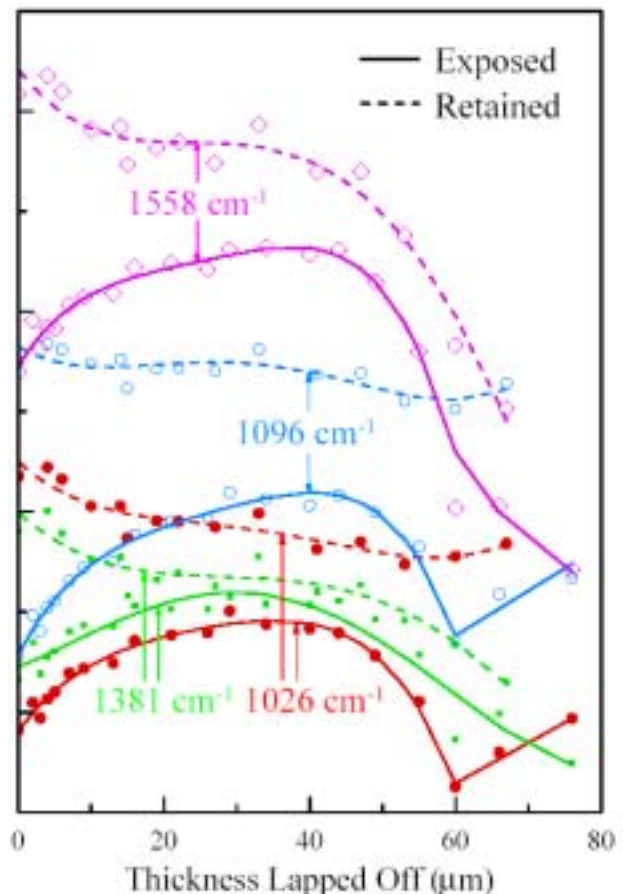


Figure 2. Profiles for clear-coat species that are reacted during weathering.

Clear-coat reaction product functional groups concentrate more at the weather exposed surface than they do at the retained surface as shown in Figure 3. The functional groups of Figure 3 include the melamine triazine ring (818 cm^{-1}), melamine methylol and amine (3364 cm^{-1}), and acrylic hydroxyl (3495 cm^{-1}).

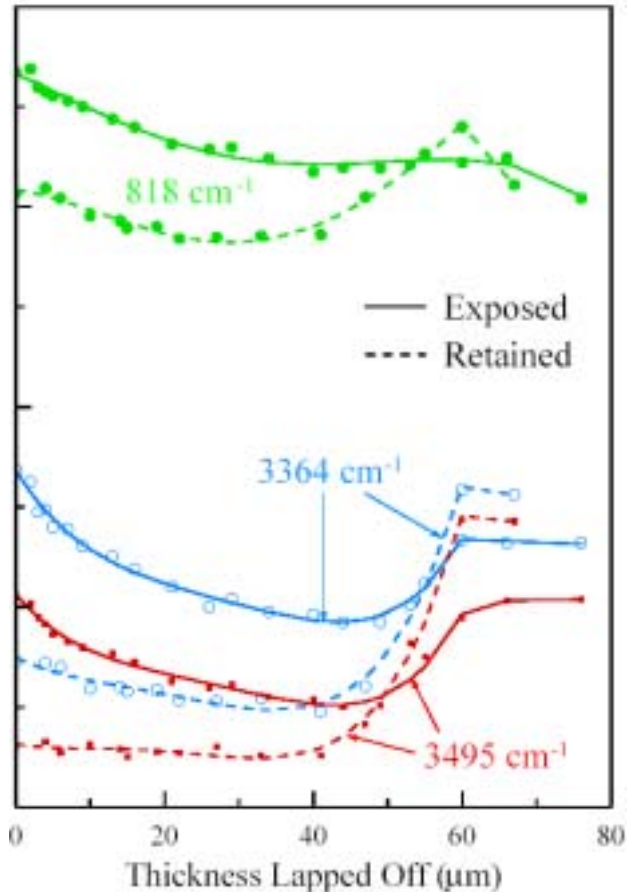


Figure 3. Clear-coat profiles of reaction product species measured with MicroLap.

The area of the acrylic ester carbonyl band is plotted in Figure 4 for the exposed and retained samples as a function of depth. The carbonyl profiles show that weather induced oxidation is highest at the surface and higher throughout the exposed clear-coat layer than for the retained sample.

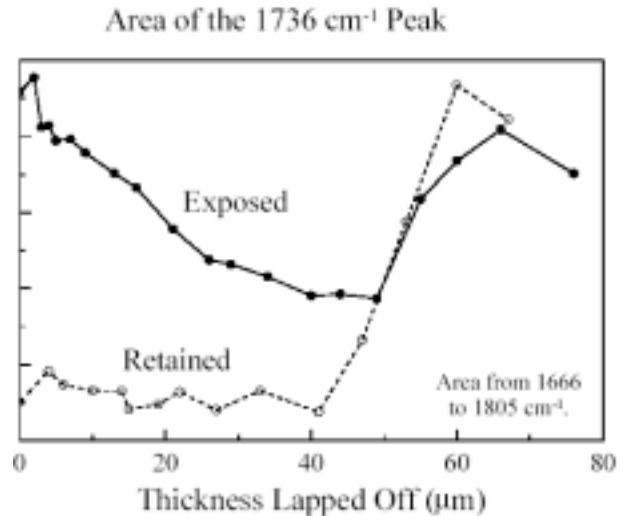


Figure 4. Area of the acrylic ester carbonyl band vs. depth for a 50 micrometer thick clear-coat coating.

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