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Comparative IR Microanalysis of Multilayer Samples Using IR Imaging and Mapping

Thomas J. Tague Jr.

Bruker Optics, Inc., 19 Fortune Drive, Billerica, MA 01821

A challenging problem for the infrared microscopist is the analysis of a thin layer located between two larger layers. This could be a paint chip, plastic wrapping, or other layered materials. The analysis is complicated when the layer of interest is thinner than about 10 microns, the diffraction limit at 1000 wavenumbers. Certainly, collecting an infrared image using a Focal Plane Array (FPA) detector provides excellent spatial resolution. A standard FPA coupled with 15x optics yields about 4 microns/pixel. However, it is not uncommon to encounter layers smaller than 4 microns.

Line mapping of the layer could also be performed, where the remote mask (aperture) would be set to the boundaries of the layer of interest or larger. The step size for doing a line map follows the Nyquist Theory and must be ½ or smaller than the layer thickness to define the layer of interest (assuming that the aperture exactly masks the layer). About 2-3 microns is as small as the aperture can be set and still have enough light for analysis.

Another important consideration for these analyses is sample thickness. The sample must be optically thin, so that the range of incident angles of light does not broaden the area of interest. In general, the sample must be at least as optically thin as the desired spatial resolution. Obviously, the numerical aperture of the objectives used must be considered.

In this investigation, a four-layer laminate was analyzed using both mapping and IR imaging. The layer of interest was a 3-micron thick unknown layer in between polyethylene (PE) and polyethylene teraphthalate (PET) layers. The sample was cryogenically microtomed to a thickness of 10 microns. The data was collected using the Bruker Optics Hyperion 3000 infrared microscope. The line map was conducted with a 3 by 100 mask and 10 seconds data collection at 4 cm^{-1} resolution. The imaging data was collected in 6 seconds at 4 cm^{-1} resolution without the use of an aperture.

Figure 1 shows the infrared and visible images of the laminate sample. Figure 2 shows the isolated 3 micron layer infrared spectrum extracted from the image (single pixel) after subtraction of the adjacent PE layer. There was no evidence of PET in the spectrum. The resultant spectrum was correctly identified as diethylyleneglycol isophthalate by searching the Hummel Polymers reference library. Some contamination from the PE layer was evident due to the optical thickness of the sample. The mapping results were very similar to the imaging results, but required subtraction of both PET and PE to isolate.

Fig 1 – The infrared image of the 4 layer polymer laminate is shown on the left and visible image is shown on the right

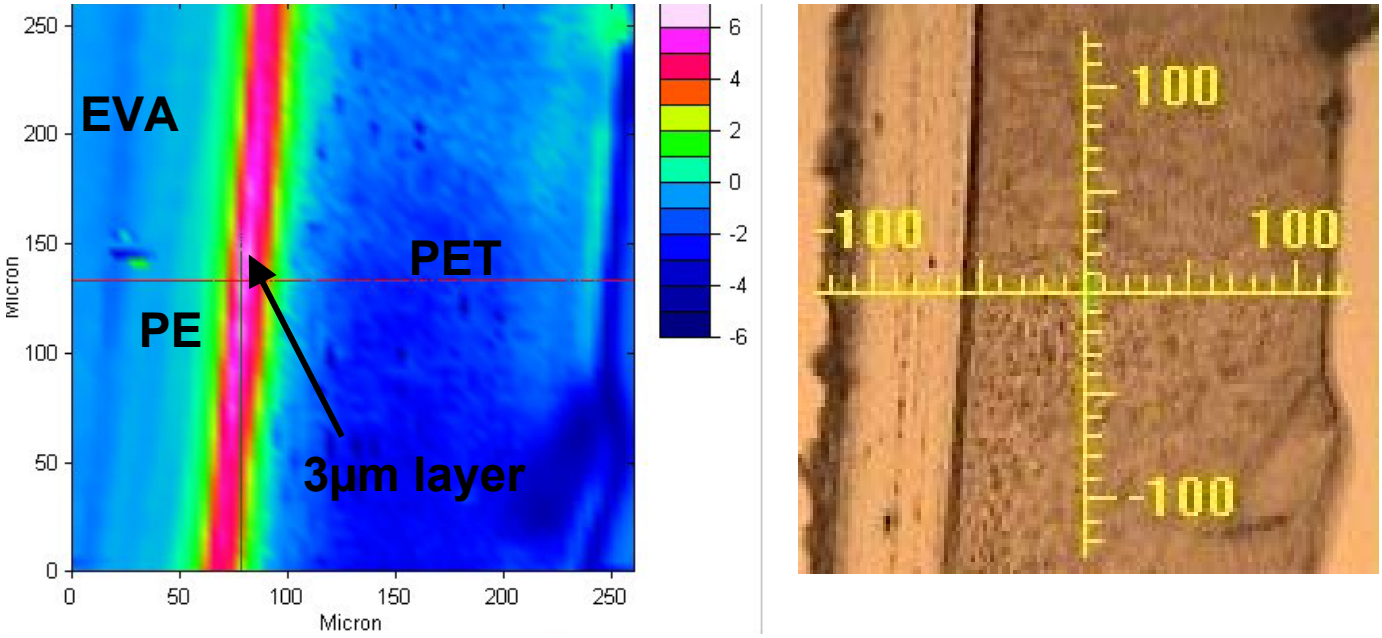
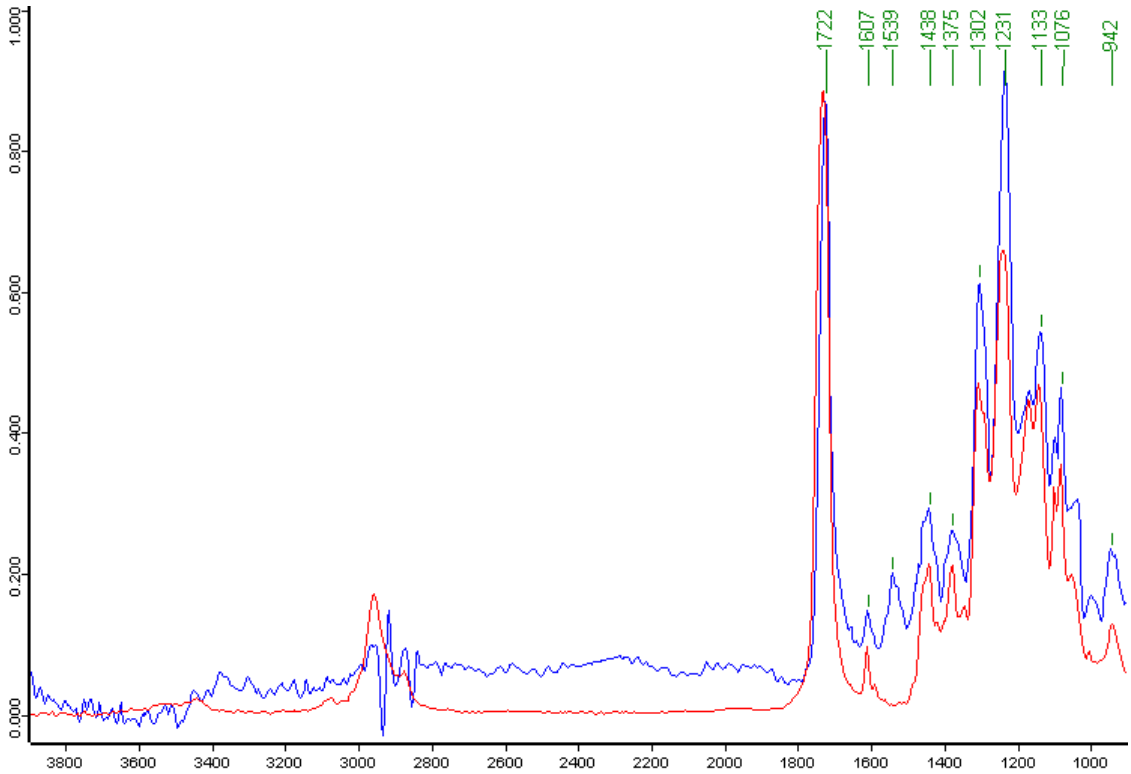


Fig 2 – Red spectrum is the reference diethylyleneglycol isophthalate from the Hummel Polymers Library. The blue spectrum is the 3 micron layer after PE subtraction.



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