

Multilayer packaging material analysis using Laser Direct Infrared (LDIR) Imaging



Introduction

Multilayer laminate packaging materials are a complex system, both structurally and chemically. These materials, despite being only a few hundred microns thick, comprise numerous distinct polymer layers that are each engineered for a specific function. Depending on its chemical identity and thickness, a layer may provide mechanical strength, permeability control, or environmental protection. Any defects or thickness errors in multilayer packaging can have a devastating impact, causing product damage and risk to consumers. Therefore, when developing and troubleshooting multilayer packaging it is critical to map each layer and precisely measure its thickness at the micrometer scale.

Agilent's 8700 Laser Direct Infrared (LDIR) Chemical Imaging System is a sophisticated chemical imaging system that can chemically identify and visualize polymer layers with high spatial resolution. With advanced visualization features available with the intuitive Agilent Clarity software, the 8700 LDIR provides a fast and effective workflow for packaging material study. This workflow reveals answers to key questions in quality assurance, failure analysis, or reverse engineering.

Key benefits and features of 8700 LDIR imaging system for laminates analysis

- The Agilent Clarity Software provides an intuitive and automated workflow from sample loading to analysis
- The Agilent sample holder allows thin specimens to be prepared in less than 5 minutes
- High magnification visible light microscope provides an overview of multilayer laminate structure with spatial resolution as high as 1 μm
- Live imaging feedback during sample contact with automated Attenuated Total Reflectance (ATR) ensures optimal contact and highest-quality spectra and images
- ATR mosaicking is intuitive and seamless with superb visible-infrared alignment, automated approach and release, and backgrounding
- Point scanning architecture measures the exact center of a layer or point defect to collect the purest spectrum possible
- Bright laser source and fast-scanning optics permits rapid spectral acquisition and imaging
- No extensive chemometrics are needed for image generation and identification of layers

Analysis Examples

One of the laminates analyzed using the 8700 LDIR is a food packaging material made up of multiple layers as small as 3 μm thick.

The Agilent laminate holder and Sample Planer (a microtome device) were used to prepare the sample. With these tools, sample preparation is fast and requires no special skills to master. The laminate is simply inserted into the laminate holder's built-in clamp. Then, both the laminate and the holder are sliced to create a flat surface. This design ensures perfect support, with no laminate bending, folding, or splitting apart during slicing or imaging. The entire process takes only a few minutes, which is immensely faster than traditional resin embedding and polishing that requires many hours.

After sample preparation, the high-magnification visible camera in the LDIR system was used to obtain an overview of the laminate structure, revealing multiple layers (Fig. 1). The total sample thickness was 117 μm .

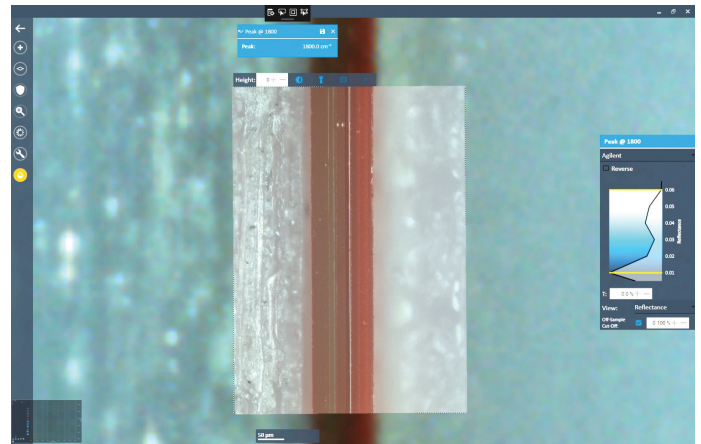


Figure 1. High-resolution visible image of the laminate (red) sandwiched between supporting plastic materials in the sample holder (white).

Then, ATR was used to acquire infrared spectra of the laminate layers and to visualize their distribution at high spatial resolution. The software performs automated ATR contact to any region of interest selected by the user. Live image feedback shows a change in contrast at the first moment of sample contact (Fig.2).

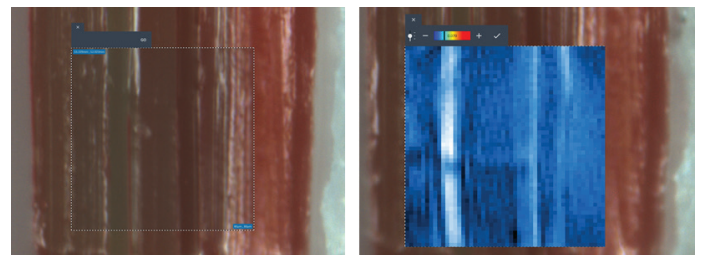


Figure 2. (Left) ATR field of view (80 x 80 μm) selected for analysis on the visual laminate image. (Right) Live ATR image after the contact with the sample.

When ATR was in full contact with the sample, a pure spectrum was obtained in less than 5 seconds by simply double-clicking (Fig.3, left). Automatic library search identified the polymer layer as polyamide.

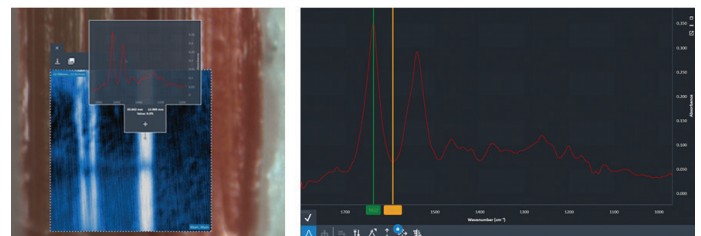


Figure 3. (Left) Spectral preview after double-clicking a point of interest. (Right) Spectrum view with the band selection for imaging.

Finally, to visualize the distribution of a chemical functional group across all layers within the ATR field of view (80 x 80 μm), a prominent band of the spectrum (Fig. 3, right) was imaged (Fig. 4, left) in less than 14 seconds at 0.2 μm pixel size. Following this intuitive process, an infrared chemical visualization of all the laminate layers was obtained using the multi-peak analysis tool that combined the chemical images taken with different spectral bands (Fig. 4, right) for each laminate layers. The ruler tool present in the software was used to measure the thickness of each layer.

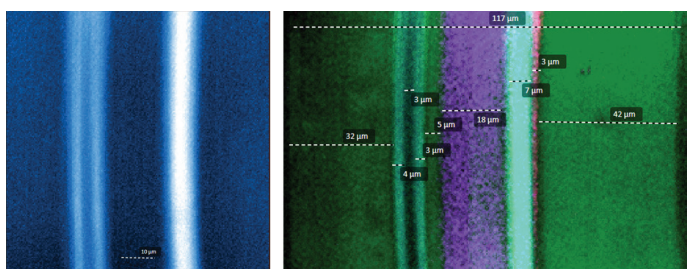


Figure 4. (Left) Distribution of polyamide spectral band. (Right) Multi-peak analysis of the laminate sample. The layers were identified as polyethylene (green), polyamide (cyan), polypropylene (purple), polyurethane (pink), and poly(ethylene) vinyl alcohol (between two cyan layers on left side).

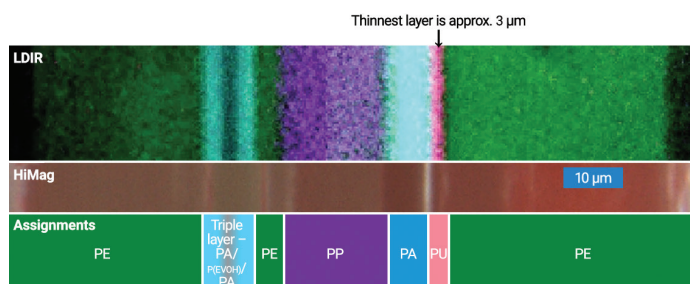


Figure 5. (Top) LDIR multi-peak analysis showing chemical layers of the laminate sample. (Middle) High magnification visible image of the laminate sample. (Bottom) Chemical assignment of polymer layers as Polyethylene (PE), Polyamide (PA), Poly(ethylene vinyl alcohol) (P(EVOH)), Polypropylene (PP), and Polyurethane (PU). The thinnest layer observed was only 2.6 μm thick.

Four pure polymer layers and one mixed-polymer layer was observed in the laminate (Fig. 5). From the mixture analysis search hits, the mixed spectrum was recognized to contain both polyamide and poly(ethylene) vinyl alcohol (EVOH). The full width of the laminate was imaged and classified by mosaicking two ATR images (Fig.5). The layers in the infrared image align perfectly with those in the visual image. The thin layer of polyurethane is highlighted in pink is only 2.6 μm thick, which could only be measured and identified with the high resolution of LDIR.

The advantage of chemical imaging with the 8700 LDIR is that it reveals all the layers with its chemical identity with the highest spatial resolution allowed by diffraction. There are two things worth noting from the food packaging laminate chemical imaging example. First, it shows that what appeared

to be a single layer in the visible microscope image (Fig. 5, center Hi-mag image at left) is in fact three layers.

Second, it shows that the layers on the right side of the laminate visible image are all the same polymer compound polyethylene, with the only difference being red dye added to it (Fig. 5). Visual inspection alone is not adequate to determine the make-up of this sample.

In a second example of laminate analysis using the 8700 LDIR (Fig. 6), six distinct layers were identified within a total thickness of 230 μm . A three-contact ATR mosaic image spanned the full width of the sample. ATR contact to the region of interest was performed seamlessly and automatically. Again, note the perfect alignment between the visible and chemical images.

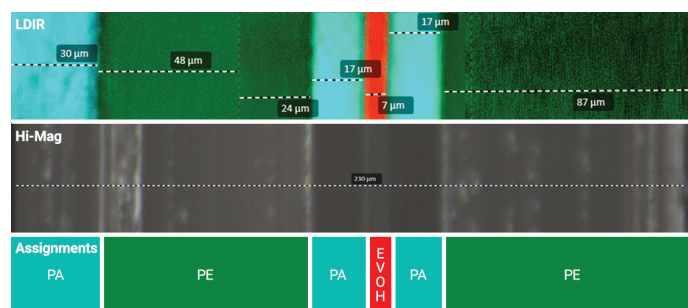


Figure 6. (Top) LDIR chemical image obtained using a multi-peak analysis of the laminate sample showing different layers and thickness. (Middle) High-magnification visible light image of the laminate. (Bottom) Identities of each layer: Polyamide (PA), Polyethylene (PE), and Ethylene Vinyl Alcohol (EVOH).

Conclusion

The Agilent 8700 LDIR Chemical Imaging System provides the ability to find and identify all the layers in a laminate sample. In the above examples, the 8700 LDIR identified layers less than 3 μm thick. The ease-of-use of sample preparation introduced by Agilent allows users to focus on understanding laminate chemistry rather than mastering sample preparation. Intuitive workflows allow chemical exploration of laminate layers in real-time. Similarly, ATR mosaicking is made seamless via perfect visible-infrared alignment, automated approach and release, and background data collection. The Agilent 8700 LDIR Chemical Imaging System provides users with a tool to analyze packaging materials at unprecedented speed with superior spectral quality and spatial resolution, revealing answers demanded by quality assurance, failure analysis, and reverse engineering.

www.agilent.com/chem/8700-ldir

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