Infrared Raman Microscope

Application News

Cross-Sectional and Depth-Profiling Analysis of Multilayer Films Using the AIRsight™ Infrared Raman Microscope

Seri Nishikura and Shoko Iwasaki

User Benefits

- Infrared Raman microscopes can perform both infrared and Raman spectroscopy without moving the sample from the sample stage.
- Depth-profiling by Raman spectroscopy does not require sample preparation, enabling faster characterization of multilayer films.
- It can quickly determine the components of a multilayer film, and it uses chemical images to show their structural distribution.

■ Introduction

One of the main functions of plastic film in food and pharmaceutical packaging is to maintain product quality. To meet the packaging requirements of the product, the film often has multiple layers. The combination of layers is tailored to provide heat resistance, impact resistance, light shielding, oxygen barrier properties, etc., so the ability to determine the composition and thickness of these layers is essential for developing new films and ensuring their quality.

The AIRsight infrared Raman microscope has a new design that incorporates a Raman spectroscopy unit within an infrared microscope (Fig. 1). So while infrared spectroscopy and Raman spectroscopy are typically performed on separate devices, AIRsight allows infrared spectroscopy and Raman spectroscopy to be performed on the same device. AIRsight can record both infrared and Raman spectra from the same target area without moving the sample, and it can also perform Raman depth-profiling analysis, which can provide a depth profile without the need for sample preparation.

This Application News describes using the AIRsight to analyze a multilayer film used in packaging.

Fig. 1 IRTracer™-100 and AIRsight™

■ Cross-Sectional Analysis: Sample Preparation and Analysis Conditions

A multilayer film used in food packaging was obtained, and then a microtome (HistoCore AUTOCUT R, Leica Microsystems) was used to prepare 10-µm thick pieces of the multilayer film for cross-sectional analysis. (The microtome featured automatic and manual cutting modes and a cutting thickness configurable between 0.5 and 600 µm.) The infrared spectroscopy and Raman spectroscopy conditions used for mapping analysis are shown in Table 1.

Table 1 Analysis Conditions

Equipment:	IRTracer™-100 and AIRsight
Infrared Spectroscopy (Cross-Sectional Measurements)	
Resolution:	8 cm^{-1}
Number of Averaged Scans:	64
Apodization Function:	SqrTriangle
Aperture Size:	10 × 10 µm
Mapping Area:	100 × 150 µm
Detector:	T2SL
Raman Spectroscopy (Cross-Sectional and Depth-Profiling Measurements)	
Number of Averaged Scans:	1
Exposure Time:	1.0 sec
Objective Lens:	50×/100×
Excitation Wavelength:	785 nm
Laser Spot Diameter:	5 µm/3 µm
Mapping Area:	(W) 90 × (H) 150 µm / (W) 140 × (H) 150 µm
Detector	CCD

■ Measuring the Thickness of Multilayer Film Layers

The AIRsight comes with AMsolution software that has a distance-measuring tool that measures the distance between user-selected points on wide-field camera images or microscope images. This tool was used to determine the thickness of the layers in a multilayer film from a cross-sectional image. The image was captured by the infrared microscope camera (Fig. 2), and then the thickness of each layer was measured from it (Table 2). Four layers were identified in the multilayer film, and the thickness of each was determined.

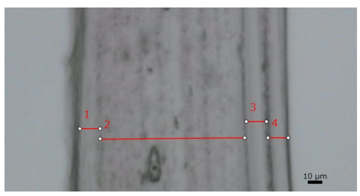

Fig. 2 Infrared Microscope Camera Image of Multilayer Film Section

Table 2 Measured Thickness of Each Layer

No.	Thickness (µm)
1	14
2	100
3	14
4	14

■ Cross-Sectional Analysis by Infrared Spectroscopy

Infrared transmission spectroscopy was used to perform mapping analysis of the multilayer film section. The aperture size was set to 10 × 10 µm and the step size to 5 µm vertically and 10 µm horizontally. The mapping area was 100 × 150 µm. Chemical images were also prepared from the mapping data (Fig. 3). These images, which visualize the distribution of constituent materials, can be created from mapping data using peak height, multivariate analysis (PCR/MCR), or the degree of spectral similarity, which is what was used in this analysis. The degree of spectral similarity is represented by a color gradient, ranging from red, signifying high spectral similarity, to blue, signifying low spectral similarity.

The chemical images showed that the first layer of the film was polyethylene terephthalate (PET). The second and fourth layers were nylon, and the third layer was polypropylene (PP). Mixed spectra were also recorded in boundary regions between the film layers. These mixed spectra were assumed to indicate the presence of very thin layers that were not readily identifiable by infrared spectroscopy.

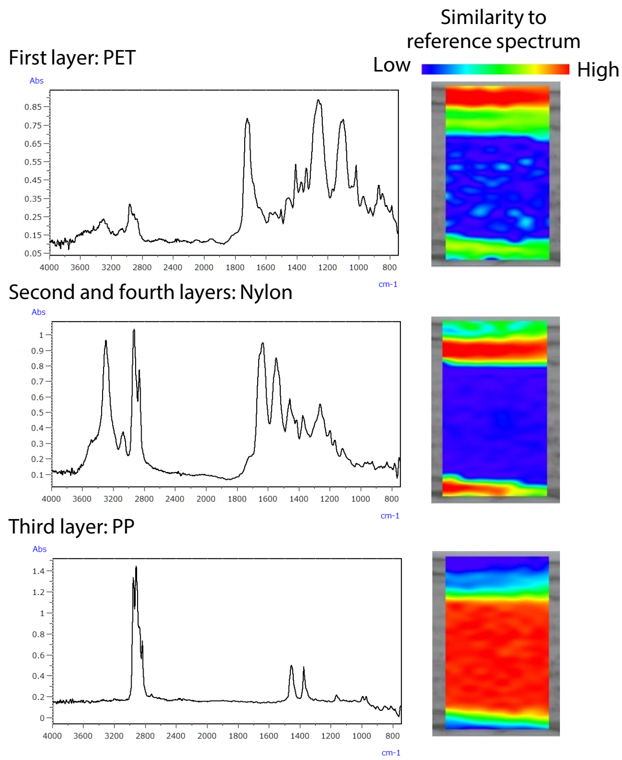

Fig. 3 Cross-Sectional Analysis by Infrared Spectroscopy:
Infrared Spectrum and Chemical Image of Each Layer

■ Cross-Sectional Analysis by Raman Spectroscopy

A 50x objective lens was used to perform Raman mapping of the multilayer film section. The laser spot size used was 5 µm in diameter, which allowed data to be recorded at a higher spatial resolution than infrared spectroscopy. The mapping area was set to 90 × 150 µm and the step size to 5 µm vertically and 10 µm horizontally.

In addition to identifying the PET, nylon, and PP layers that were identified by infrared spectroscopy, the Raman spectroscopy also recorded peaks for an alkyd resin, presumably part of an adhesive, at boundary regions between the layers (Fig. 4).[1] These results show that the higher spatial resolution of Raman spectroscopy was able to characterize very small features that were not readily identifiable by infrared spectroscopy.

The chemical images were prepared from the Raman mapping data (Fig. 5) using the same process as that of infrared spectroscopy. A chemical image for alkyd resin was also created based on the peak height at 1020–980 cm^{-1}. The data indicated that alkyd resin was present between every layer of the film.

Fig. 6 shows the structure of the laminate film, which was determined from the infrared and Raman spectroscopy. Combining data from infrared and Raman spectroscopy enables a more detailed discussion of the multilayer film. The Raman spectroscopy revealed the presence of very thin alkyd resin layers.

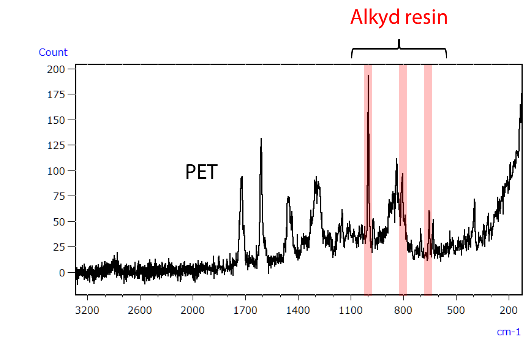

Fig. 4 Cross-Sectional Analysis by Raman Spectroscopy:
Raman Spectrum of Boundary Region between Second and Third Layers

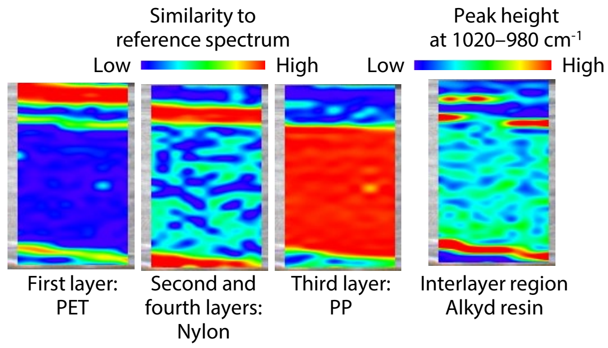

Fig. 5 Cross-Sectional Analysis by Raman Spectroscopy:
Chemical Images

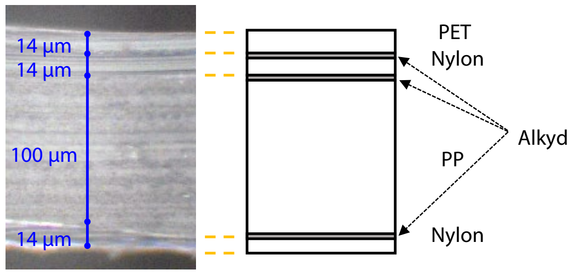

Fig. 6 Laminate Structure of Film Based on Cross-sectional Analysis
by Infrared and Raman Spectroscopy

■ Depth-Profiling by Raman Spectroscopy

Depth-profiling by Raman spectroscopy does not require the preparation of a cross-sectional sample. This eliminates the time and equipment required for sample preparation, and it allows for quicker analysis.

A 100x objective lens was used to analyze the same multilayer film in the above cross-sectional analysis. The laser spot size was 3 µm in diameter, and the depth resolution was 7.5 µm. The horizontal step size was 10 µm, and the depth step size was 3 µm. The mapping area was (W) 140 × (H) 150µm. Mapping analysis of this large area was completed in just 20 minutes.

Fig. 7 shows the chemical images created from the Raman mapping data. Depth-profiling analysis detected another PET layer. This fifth layer is believed to have been lost when the microtome was used to prepare the sample for cross-sectional analysis.

Depth-profiling analysis also detected an alkyd resin peak at around 1000 cm^{-1}, as was detected during cross-sectional analysis by Raman spectroscopy (Fig. 8).[1)] However, the Raman depth-profiling analysis only detected this peak between the first and second layers and the second and third layers. It did not detect it between other layers. This is probably because Raman depth-profiling has a higher spatial resolution in the lateral direction, rather than in the depth direction, making detection of the thin alkyd resin layer difficult. The resolution of Raman depth-profiling also decreases with increasing depth due to factors such as scattering, which also probably affected the results.

Fig. 9 shows the laminate structure of the film based on the results of depth-profiling analysis by Raman spectroscopy. Unlike cross-sectional analysis, Raman depth-profiling analysis provided these results without the need for sample preparation. Cross-sectional analysis can also be used in conjunction with depth-profiling analysis for a more detailed discussion of certain aspects of the analyzed film.

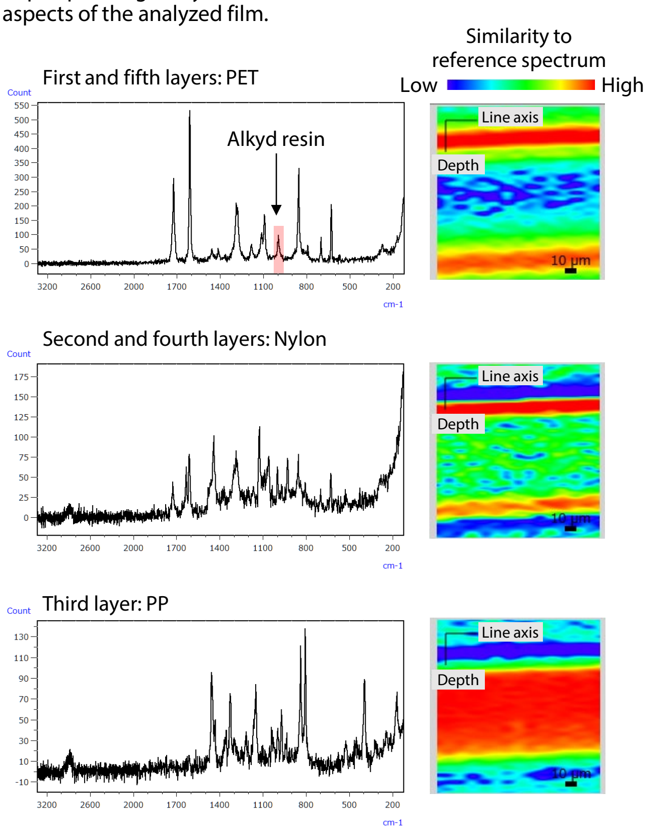

Fig. 7 Depth-Profiling by Raman Spectroscopy:
Raman Spectrum and Chemical Image of Each Layer

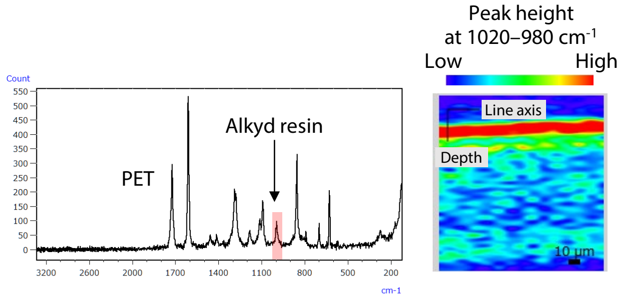

Fig. 8 Depth-Profiling by Raman Spectroscopy
Raman Spectrum and Chemical Image of Boundary Region
between First and Second Layers

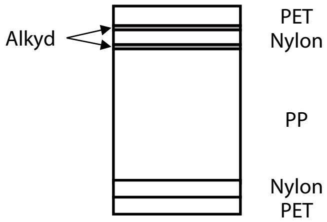

Fig. 9 Laminate Structure of Film Based on Depth-Profiling Analysis
by Raman Spectroscopy

■ Conclusions

An AIRsight infrared Raman microscope was used to perform mapping analysis of a multilayer film used in food packaging. Raman depth-profiling offered a simple and effective technique for analyzing the film without the need for sample preparation. A cross-section of the film was also prepared with a microtome, and Raman spectroscopy of it was used in conjunction with depth-profiling data for a more accurate analysis of the film material. Infrared spectroscopic analysis was also performed on the same part of the cross-sectional sample, allowing for a more multifaceted assessment of the film. Many of the plastics used in multilayer films emit fluorescent light, making infrared spectroscopy more effective than Raman spectroscopy at analyzing them.

The AIRsight can easily switch between infrared and Raman spectroscopy, and it can use readings from mapping analysis to create chemical images that show the distribution of constituent materials. The ability to quickly characterize the structure of multilayer films and assess them with different techniques will help accelerate the development of new functional materials and with the necessary quality controls.

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<References>

1) Job M Belle, David L. Stokes, and Tuan. Vo-Dinh, “Correspondence. Direct Characterization of the Phtalic Acid Isomers in Mixtures Using Surface-Enhanced Raman Scattering”, Analytical Chemistry, 62, 1349 (1990)

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1. Multilayer Film Analysis Using the AIRsight Infrared Raman Microscope Application News No. 01-00465

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