Application News

AIRsight™ Infrared Raman Microscope

Multilayer Film Analysis Using the AIRsight Infrared Raman Microscope

Karen Maruyama

User Benefits

- An infrared Raman microscope offers the ability to analyze a sample by both infrared spectroscopy and Raman spectroscopy without moving the stage.
- Raman spectroscopy is effective for analyzing multilayer films with layers 10 µm or thinner and with inorganic compound layers.
- AIRsight has a length measurement function that can measure the thickness of thin films in images from its wide-field camera and microscope camera.

■ Introduction

One of the roles of films used in food and pharmaceutical packaging is to maintain product quality. These films often have multiple layers, combining single-layer films to offer a variety of properties such as heat resistance, impact resistance, light shielding, and oxygen barrier properties, depending on the contents of the product.

Determining the material and thickness of each layer in a multilayer film is important for the development of new film materials and quality control. This analysis can also be used for comparisons with competitor products and to study competitor products.

The AIRsight infrared Raman microscope is a new microscope that incorporates a Raman unit inside an infrared microscope (Fig. 1). Analyses that previously required two separate systems can now be performed on a single unit capable of recording both infrared and Raman spectra from the same target area without needing to move the sample. Both infrared spectroscopy and Raman spectroscopy on AIRsight are controlled from the same AMsolution software, greatly simplifying the analysis process.

Infrared spectroscopy is mainly used to identify layers 10 µm or thicker, while Raman spectroscopy has the following advantages over infrared spectroscopy:
(1) Excellent spatial resolution due to the use of laser light
(2) Capable of analysis at low wavenumbers.

These advantages enable the identification of ultra-thin layers (under 10 µm) in multilayer films and the identification of layers of inorganic compounds that have characteristic peaks in low wavenumber regions.

This article describes an example application of AIRsight that uses both infrared spectroscopy and Raman spectroscopy to analyze a multilayer film packaging material.

Fig. 1 IRXross™ and AIRsight™

■ Sample Preparation and Analytical Conditions

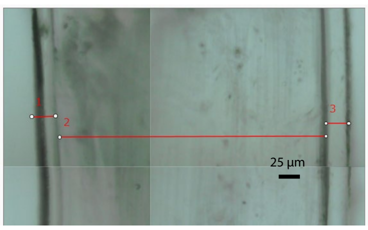
The packaging material for a hair conditioner product was analyzed. Sample sections were prepared with a microtome (HistoCore AUTOCUT R, Leica Microsystems). The microtome came with automatic and manual cutting modes and a cutting thickness configurable to between 0.5 and 600 µm. 15-µm thick sections of the packaging material were prepared and placed on a barium fluoride window for analysis. The analytical conditions used in infrared and Raman spectroscopy are shown in Table 1.

Table 1 Analytical Conditions

Instruments:	IRXross™ and AIRsight
Infrared Spectroscopy	
Resolution:	8 cm^{-1}
Number of Scans:	15
Apodization Function:	SqrTriangle
Aperture Size:	10 × 30 µm
Step Size:	2 µm
Mapping Area:	30 × 410 µm
Detector:	T2SL
Raman Spectroscopy	
Cumulative Measurements:	2
Exposure Time:	5.0 sec
Objective Lens:	50x
Excitation Wavelength:	532 nm
Step Size:	5 µm
Mapping Area:	5 µm × 85 µm
Detector:	CCD

■ Determining the Thickness of Each Multilayer Film Layer

The AMsolution control software for AIRsight comes with a length measurement function as standard that measures the distance between any two user-selected points on wide-field camera images or microscope images. The function was used to determine the thickness of each layer in the multilayer film. The image of the multilayer film section acquired using the infrared microscope camera and used for length measurement is shown in Fig. 2, together with the thickness of each layer. Three layers were identified and the thickness of each was determined as shown below.



No.	Length (µm)
1	28
2	314
3	27

Fig. 2 Length Measurements for Each Layer

■ Analysis by Infrared Spectroscopy

Infrared transmission spectroscopy was used to perform a mapping analysis of the multilayer film section. As mentioned earlier, the mapping area was set to 10 × 30 µm and mapping was performed in 2 µm steps. The mapping data was then used to prepare chemical images (Fig. 3). Chemical images can be created from mapping data using peak height, peak area, multivariate analysis (PCR/MCR), or degree of spectral similarity to visualize the distribution of constituent materials.

In this example, chemical images were created using the degree of spectral similarity. The greater the degree of spectral similarity, the more intense the color in the chemical image.

The first and third layers were identified as nylon and the second layer as polyethylene (PE), but mixed spectra recorded in boundary regions between the three layers indicated the presence of additional ultra-thin layers not readily identifiable by infrared spectroscopy.

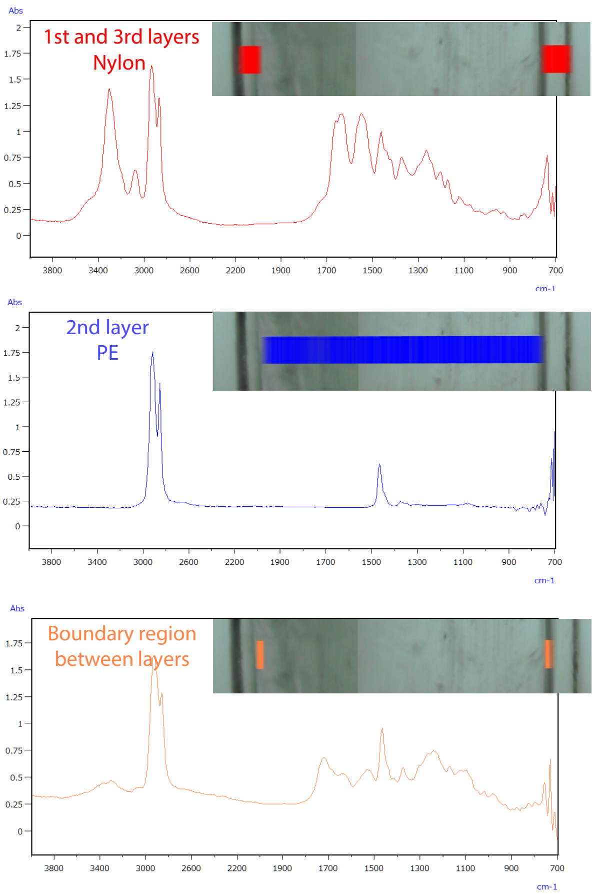


Fig. 3 Infrared Spectra and Chemical Images of Layers Identified by Infrared Spectroscopy and Boundary Regions between Layers

■ Analysis by Raman Spectroscopy

A 50x objective lens was used to perform Raman mapping of the multilayer film section. A measurement area (laser spot size) of 5 µm diameter allowed data to be recorded at a higher spatial resolution than infrared spectroscopy. Figs. 4 and 5 show Raman data mapped to the boundary region between the second and third layers. In addition to nylon and PE identified by infrared spectroscopy, Raman spectroscopy also identified peaks belonging to polyethylene terephthalate (PET) and TiO_2 in the boundary region. Because TiO_2 peaks are in a low wavenumber region, infrared spectroscopy was not able to see peaks characteristic to TiO_2 and was unable to identify the material. Conversely, Raman spectroscopy can record data from small areas and identify inorganic compounds such as TiO_2, both of which are a challenge for infrared spectroscopy.

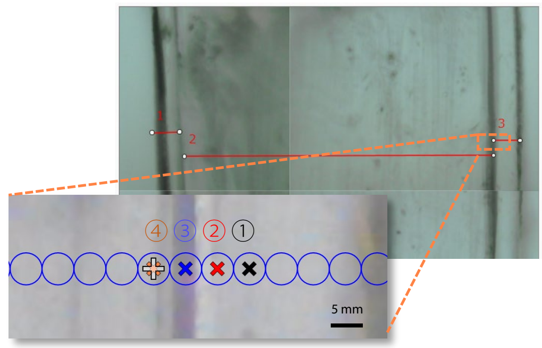


Fig. 4 Position of Boundary Region between Second and Third Layers Used for Mapping Analysis

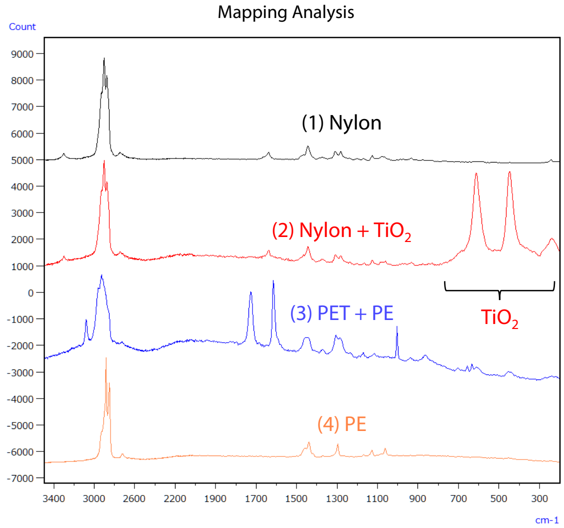


Fig. 5 Raman Spectra in Boundary Region between Second and Third Layers

■ Conclusion

An AIRsight infrared Raman microscope was used to perform mapping analysis of the multilayer packaging material of a hair conditioner product. The analysis showed that Raman spectroscopy is suited to identifying small areas under 10 µm in size and to identifying inorganic compounds, while infrared spectroscopy has trouble with both these tasks. Nevertheless, many plastics used in multilayer films emit fluorescent light and infrared spectroscopy is more effective than Raman spectroscopy at analyzing these plastics.

AIRsight switches readily between infrared spectroscopy and Raman spectroscopy through its software and visualizes distributions of constituent materials by creating chemical images from mapping measurements, thus offering a system effective for the development and quality control of multilayer films.

IRXross and AIRsight are trademarks of Shimadzu Corporation in Japan and other countries.

> Please fill out the survey

Related Products Some products may be updated to newer models.

> AIMsight
Infrared Microscope

> IRXross
Fourier Transform Infrared Spectrophotometer

Related Solutions

> Chemicals

> (Archive)Plastics

> Food and Beverages

> Food Contact Materials (FCM)

> Price Inquiry

> Product Inquiry

> Technical Service / Support Inquiry

> Other Inquiry