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Application News

AIRsight™ infrared Raman microscope spectrometer

A Comparative Study of Depth and Cross Section Measurement of Food Packaging

Dr. Aikaterini Karatzia
Shimadzu Europa GmbH

User Benefits

- The new Raman depth measurement function allows for minimum sample preparation (if any).
- No special equipment for sample preparation is needed.
- The chemical composition and the length of individual layers can be accurately determined.

■ Introduction

In recent years, the analysis of food packaging materials has gained significant attention due to its critical role in ensuring food safety and quality. Raman spectroscopy and in extend Raman microscopy have emerged as powerful tools for characterizing various aspects of foods packaging materials, offering high sensitivity and specificity in identifying chemical composition, structural properties, and potential contaminants.

The Shimadzu AIRsight infrared Raman microscope (Fig. 1) is a powerful instrument that offers a unique combination of FTIR and Raman analysis methods. This allows for a comprehensive analysis of various materials. One of its main advantages is the ability to measure the same sample without the need to change instruments, which can save a significant amount of time and effort. Furthermore, the AIRsight microscope provides the capability to accurately measure the same spot on a sample using both FTIR and Raman techniques. This is particularly valuable as it enables to obtain complementary information from the same area, enhancing the depth and accuracy of their analysis. The Raman mode of the Shimadzu AIRsight microscope allows for analysis in the depth direction, making it possible to analyze transparent or translucent samples. This is particularly beneficial when examining materials such as polymer foils or glass, where the laser used in Raman spectroscopy can penetrate the interior of the sample.

This Application News focuses on the Raman depth measurement, and its ability to deliver comparable and equally reliable results when compared to cross-sectional analysis derived from the same sample by pointing also out the non-destructive nature the method.

Fig. 1 AIRsight Infrared Raman Microscope Spectrometer

■ Sample preparation and analysis conditions

In this experiment, the same sample was utilized for both measurements, albeit prepared differently. For the cross-section measurements, a microtome (HistoCore AUTOCUT R, Leica Microsystems) was employed to cut thin cross sections of 10 µm thickness. (Fig. 2).

On the other hand, for the depth measurement, a small portion of the food packaging was cut. Subsequently, both samples were introduced into the AIRsight microscope for the actual measurement.

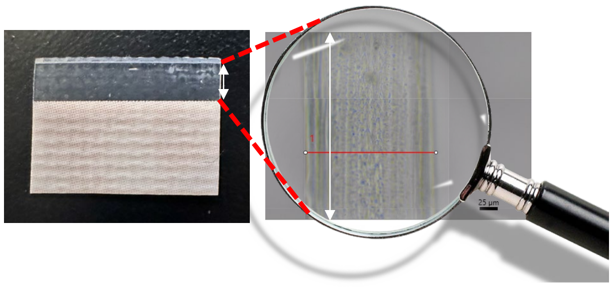

Fig. 2 Depth measurement sample (left) and cross-section (right)

Table 1 Measurement conditions – Line scan

System	: AIRsight
Number of scans	: 5
Exposure time	: 10 sec
Bleaching time	: 5 sec
Objective lens:	: 100x
Excitation wavelength:	: 785 nm
ND Filter (Laser power):	: 55 % (approximately 140 mW)
Step width	: 3 µm
Number of measurement points	: 61

■ Cross section analysis by Raman microscopy

A line scanning measurement was initially performed to identify the boundaries between the different materials/layers of the sample. A 100x objective lens was used with a laser spot diameter of 3 µm. Fig. 3 shows the distribution of the different materials which allowed the identification of the boundaries of the layers. By utilizing the new length measurement feature of AMsolutions, it was possible to measure the length of each layer. Moving from the outer sides to the inner part of the cross section, it was successfully identified that the outer layers are polyethylene terephthalate (PET) with an average thickness of 12 µm. The next layers, approximately 4 µm thick each, comprise a mixture of PET and titanium dioxide (TiO_2), which is commonly used in the food packaging industry. Next, two layers of nylon, approximately 16 µm thick each, were identified. Finally, the middle part was identified as polypropylene (PP) with a thickness of 105 µm (Fig. 4).

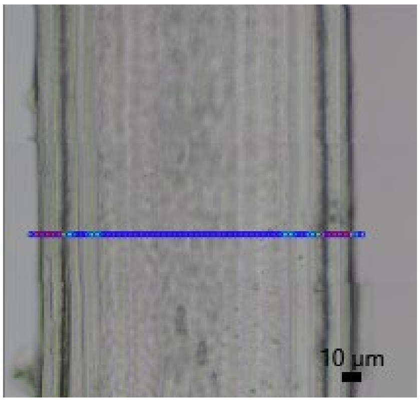

Fig. 3 Distribution of different materials according to the performed line measurement (step width 3 µm, 61 measurement points)

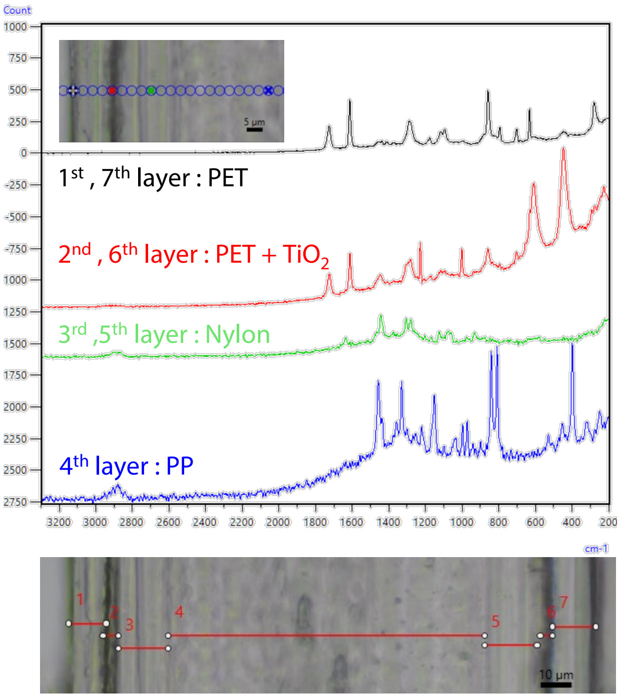

Fig. 4 Raman spectra and length measurements of the identified layers

Depth measurement analysis by Raman microscopy

The cross-section analysis was followed by a depth measurement analysis of the sample without any further preparation (Fig. 2). The same parameters as the cross-section measurements were used for the depth measurement to ensure comparable results (Table 2). A chemical image was generated from the mapping measurements. In the AMsolution mapping software, chemical images can be created using data such as peak area, peak ratio, and peak intensity ratio. In this case, the chemical image was created using the purity option, with a spectrum of the middle PP layer chosen as the reference spectrum. The areas where PP was identified are indicated in blue, while areas where other materials were identified are indicated in different colors, allowing for the identification of multiple layers/materials (Fig. 5).

Table 2 Measurement conditions – Depth measurement

System	: AIRsight
Number of scans	: 5
Exposure time	: 10 sec
Bleaching time	: 5 sec
Objective lens	: 100x
Excitation wavelength	: 785 nm
ND Filter (Laser power)	: 55 % (approximately 140 mW)
Step width	: 3 µm
Depth width	: 3 µm
Points on Line	: 14
Points in Depth	: 44

The depth measurement yielded consistent results with the cross-section analysis, confirming the presence of the same distinct polymer layers in the sample as identified during the cross-section analysis.

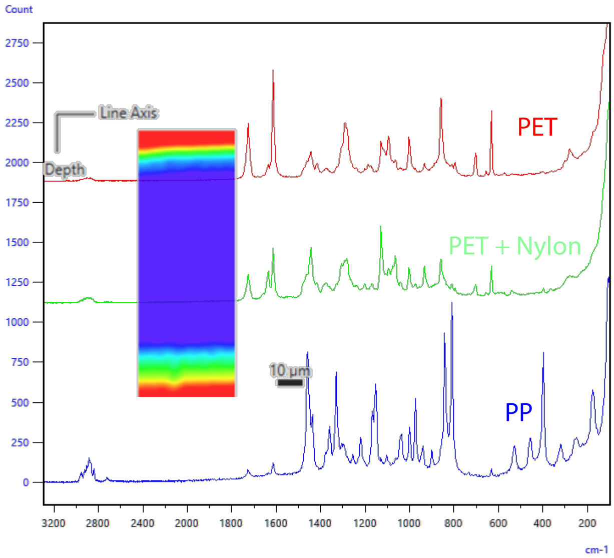

Fig. 5 Spectra and chemical image of the sample deriving from the line depth measurement.

Conclusion

In conclusion, the new depth measurement option offered by AIRsight allows for the examination of transparent samples, such as food packaging materials. It enables the identification of the distribution of multiple materials and the determination of the thickness of each layer. This feature can be particularly useful in the packaging industry for quality control purposes, especially when acquiring cross sections is not possible.

<Related Applications>

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

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