

## FT-IR Microscopy

**Author:****Kieran Evans**PerkinElmer, Inc.  
Seer Green

## Analysis of Multi-Layer Polymer Laminates with Infrared Microscopy

### Introduction

Polymer laminates are materials consisting of multiple layers of different polymers. These materials are ubiquitous in modern day life with a substantial amount of food

and pharmaceutical packaging containing laminates. A common requirement of food packaging is that the internal layer must be appropriate to be in contact with food and the external layer must be suitable for printing product information. The middle layers of the laminate can also vary depending on requirements regarding the flexibility of the packaging. Due to the large amount of variation present in the parameters of a polymer laminate, the number of combinations is innumerable making detailed analysis of these materials incredibly important.

The PerkinElmer Spotlight™ 400 Imaging System (Figure 1) with the ATR imaging attachment allows layers with thicknesses down to 2-3  $\mu\text{m}$  to be investigated.



Figure 1. Spotlight 400 IR Imaging System.

Experimental

Samples were placed vertically in a sample clamp and cut horizontally, flush to the clamp surface, to reveal a flat surface for analysis. The instrumental parameters used are shown in Table 1.

ATR imaging allows a much smaller pixel size (1.56 x 1.56 µm) to be used than conventional, non-contact imaging techniques (6.25 x 6.25 µm), providing the opportunity to image much thinner layers.

Two different polymer laminate samples were imaged as part of this study.

Table 1. Data collection parameters used for measurement of polymer laminates.

Parameter	Value
Spectral Range	4000 – 500 cm <sup>-1</sup>
Spectral Resolution	8 cm <sup>-1</sup>
Pixel size	1.56 x 1.56 µm

Results and Discussion

Sample 1

The first laminate sample comprised of five polymer layers with a total thickness of approximately 125 µm. A visible image of the laminate is shown in Figure 2.

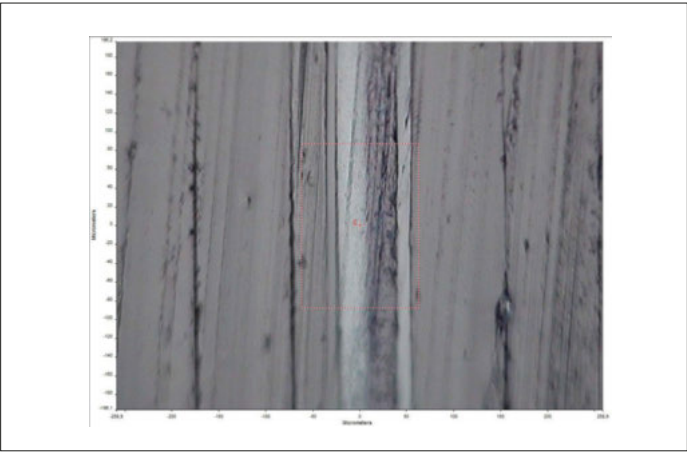


Figure 2. Visible image of polymer laminate 1.

It can be seen in Figure 2 that little information about the polymer could be deduced simply from looking at a visible image. The infrared image obtained using the Spotlight 400 system is shown in Figure 3.

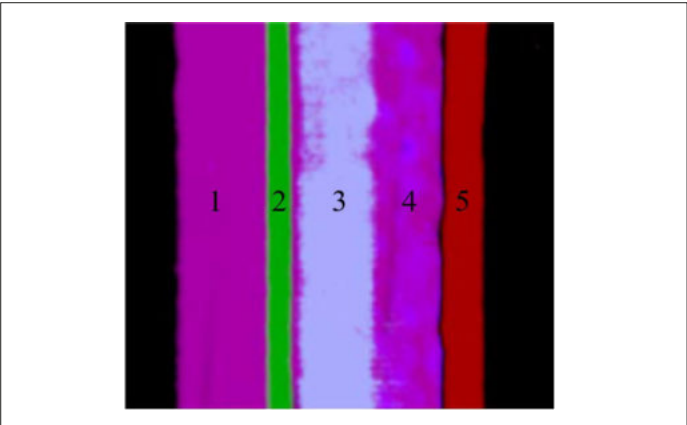


Figure 3. PCA processed ATR-image of laminate 1.

Table 2 shows the identities and thickness of each layer in laminate 1. The identities were determined by comparing the spectra obtained to a library of reference polymer spectra.

Table 2. Identities and thicknesses of the layers present in laminate 1.

Layer	Identity	Thickness (µm)
1	Polyethylene	37
2	Polyethylene + TiO <sub>2</sub>	32
3	EVOH	10
4	Polyethylene + Ultramarine pigment	30
5	PET	14

The show structure images and corresponding spectra for each individual layer of laminate 1 are shown in Appendix 1.

Sample 2

The second laminate consisted of eight layers with a total thickness of around 100 µm. The visible image of this laminate is shown in Figure 4.

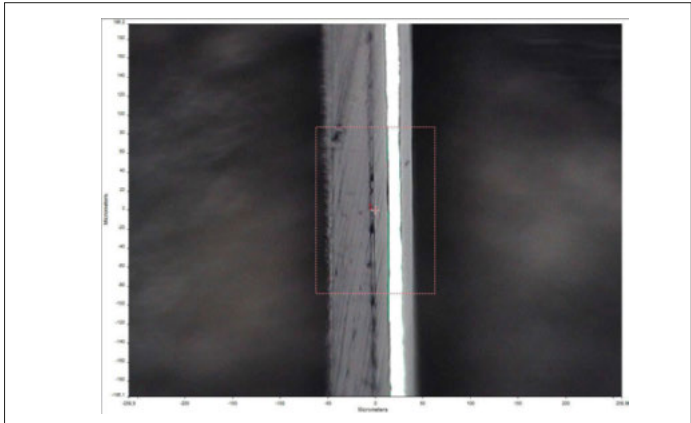


Figure 4. Visible image of laminate 2.

This visible image offers slightly more information than for the first laminate in that it could be reasonably assumed that the reflective layer is some sort of metallic foil. The infrared image is shown in Figure 5.

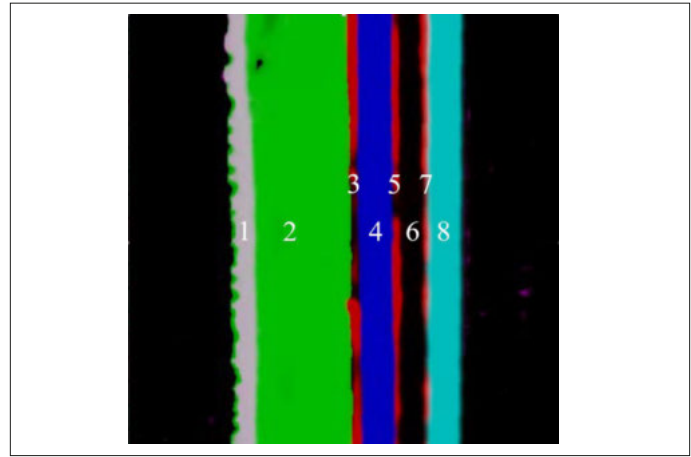


Figure 5. PCA processed ATR-image of laminate 2.

Table 3 shows the identities and thicknesses of each layer in laminate 2. This particular sample demonstrates the ability of ATR-Imaging to resolve sections of sample less than 3  $\mu\text{m}$  in thickness.

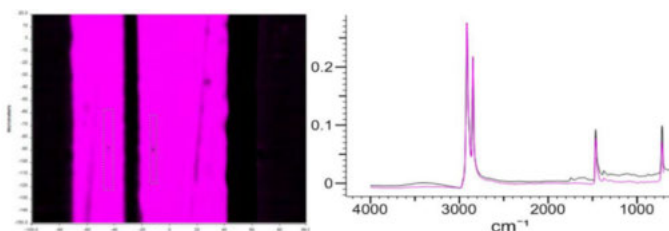
Table 3. Identities and thicknesses of layers in laminate 2.

Layer	Identity	Thickness ( $\mu\text{m}$ )
1	Polyethylene/Polypropylene Copolymer	6
2	Polypropylene	40
3	Polyurethane	3
4	Polyamide (Nylon) 6	15
5	Polyurethane	2
6	Aluminium	14
7	Polyurethane	3
8	PET	14

## Appendix 1

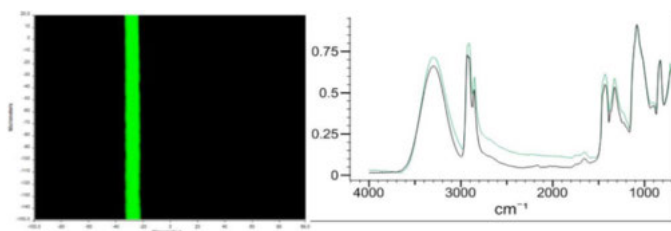
In each case, the collected spectrum is shown in the same colour as the image, while the reference spectrum is shown in black.

### Layer 1 – Polyethylene (37 $\mu\text{m}$ )

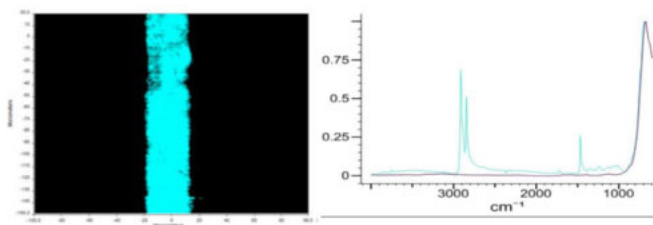


### Layer 2 – Polyethylene w/ $\text{TiO}_2$ (10 $\mu\text{m}$ )

In this layer, the reference spectrum is that of  $\text{TiO}_2$ , showing the influence the addition of this pigment has on the spectrum.



### Layer 3 – Poly(ethylene vinyl alcohol) (32 $\mu\text{m}$ )



The 'show structure' image and corresponding spectrum for each layer of laminate 2 is shown in Appendix 2.

## Conclusion

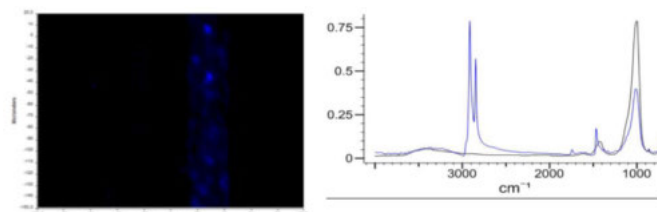
The PerkinElmer Spotlight 400 infrared imaging microscopy with ATR imaging may be used to provide detailed information about the micro-level structure of polymer laminates. SpectrumIMAGE™ software allows the user to perform data analysis with ease in order to unlock valuable information about their samples.

## Reference

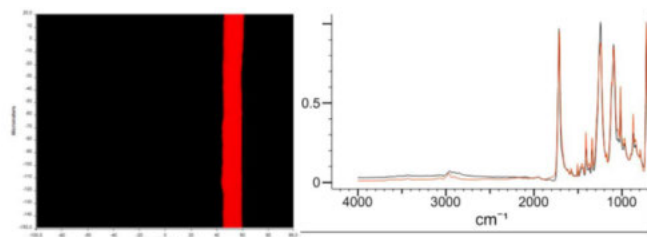
- Chanda, M., & Roy, S. K. (2008). Industrial polymers, specialty polymers, and their applications. London: CRC Press

### Layer 4 – Polyethylene w/ ultramarine pigment (30 $\mu\text{m}$ )

In this layer, the reference spectrum is that of the ultramarine pigment, showing the influence the addition of this pigment has on the spectrum.

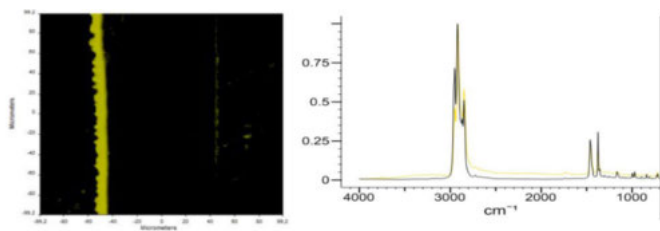


### Layer 5 – Polyethylene Terephthalate (14 $\mu\text{m}$ )

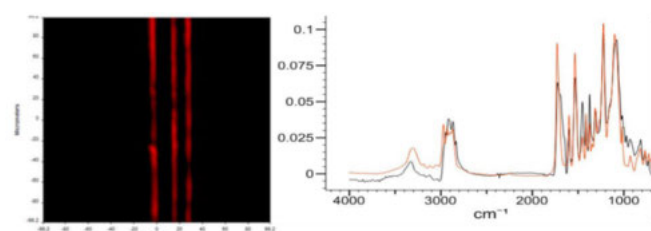


## Appendix 2

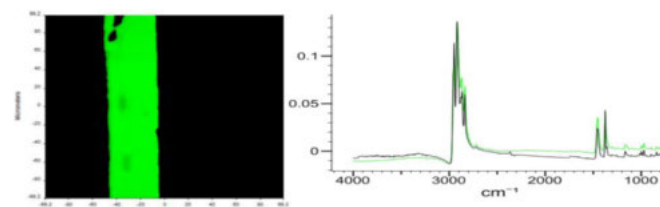
### Layer 1 – Polyethylene/Polypropylene copolymer (6 $\mu\text{m}$ )



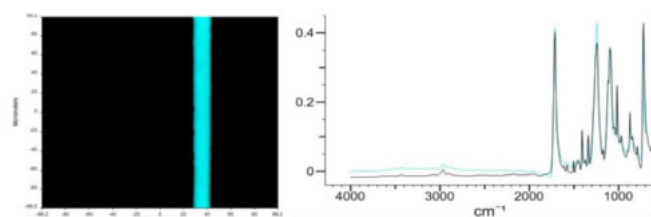
### Layers 3, 5 and 7 – Polyurethane (3, 2 and 3 $\mu\text{m}$ respectively)



### Layer 2 – Polypropylene (40 $\mu\text{m}$ )



### Layer 8 – Polyethylene Terephthalate (14 $\mu\text{m}$ )



### Layer 4 – Polyamide 6 (15 $\mu\text{m}$ )

