CASE STUDY

FT-IR Microscopy

Study of Multilayer Laminates Using the Spotlight 400 Imaging Microscope

Background

AIMPLAS is an award-winning technology centre with 30 years of experience in the plastics industry providing solutions to companies throughout the value chain, from raw material manufacturers to plastic processors and end users. One of their research areas is the development of packaging materials, usually multilayer laminates, that provide safe and effective protection to packaged materials. These packaging materials need to be fully characterized to ensure that the product is manufactured as expected. In the case of multilayer laminates, it is essential to show that all the expected layers are present, with the correct layer widths, and that the material has no defects.

AIMPLAS analyzes a wide range of multilayer laminate packaging materials. Figure 1 shows some examples.

Each of these films will consist of multiple layers of different polymer materials. Each layer has a barrier or other physical property. AIMPLAS will be able to achieve the identification of individual layers using Fourier Transform Infrared Spectroscopy (FT-IR) imaging on the PerkinElmer Spotlight™ 400 Imaging Microscope.

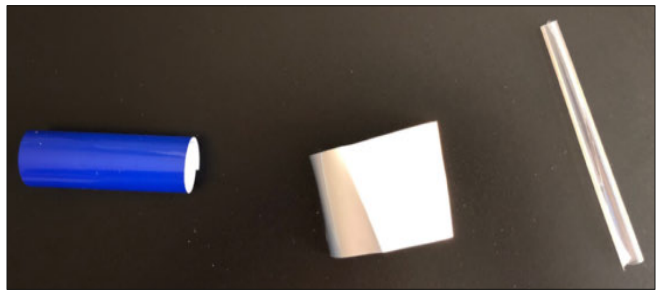

Figure 1. Cut areas of laminate films.

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Sample Preparation

Figure 2 shows the Perkin Elmer Spotlight 400 IR Imaging system.

Figure 2. The Perkin Elmer Spotlight 400 FTIR Imaging system.

The samples can be measured using the ATR Imaging Accessory, allowing layers lowering to a thickness of approximately 3 microns to be measured[1].

The preparation of the sample for the ATR imaging accessory is simple and takes only ten seconds. There is no requirement for embedding the sample in epoxy resin, the sample can simply be placed between two faces of a sample clamp. A razor blade can then be used to slice across the layers leaving a flat multilayer sample exposed. The ATR crystal can then be lowered onto the exposed sample surface until it makes contact. The ATR crystal area in contact with the sample can then be imaged. Figure 3 shows a diagram of this process.

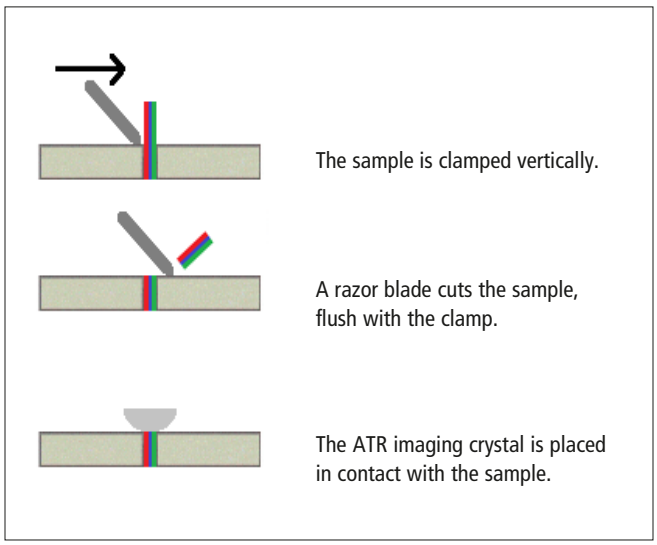

Figure 3. The sample preparation method

The first step in the analysis is to then collect the visible image of the sample to determine the area to image. The visible image of the sample is shown in Figure 4a.

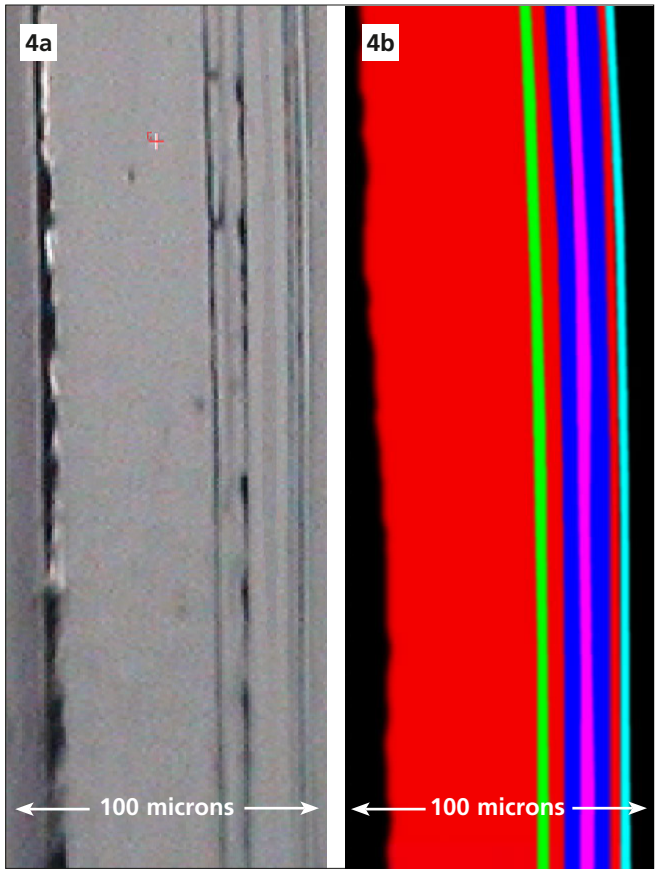

Figure 4. 4a (left) Visible Image of the sample and 4b (right) Processed IR Image.

According to this image, it is clear that there are multiple layers in this sample. The left half of the sample appears to be a thick layer of approximately 50 microns width and then there are several layers towards the right of the sample that appear to be sub 10 microns wide each. The whole area was selected for imaging as it covers the complete cross-section of the multiple layers present. The processed IR image is shown in Figure 4b. The IR ATR image is collected perfectly on the region defined from the visible image, thanks to a patented crystal centering feature. For this image containing more than 16400 high quality spectra, collection time was less than 5 minutes, including the background. The IR image was initially collected as an Average Absorbance image and then processed using the Show Structure command. This applies Principal Components Analysis (PCA) to the data and can pick out different chemical species present, represented by the different colours in the processed image. Individual component images can also be generated to show the spatial distribution of the particular polymer type and that some of the layers are composed of the same polymer type. Spectra are obtained for each layer and can be identified by comparison against polymer spectral libraries. The individual component images, their spectra, the best library match spectrum and identities are shown in Figures from 5a to 5e.

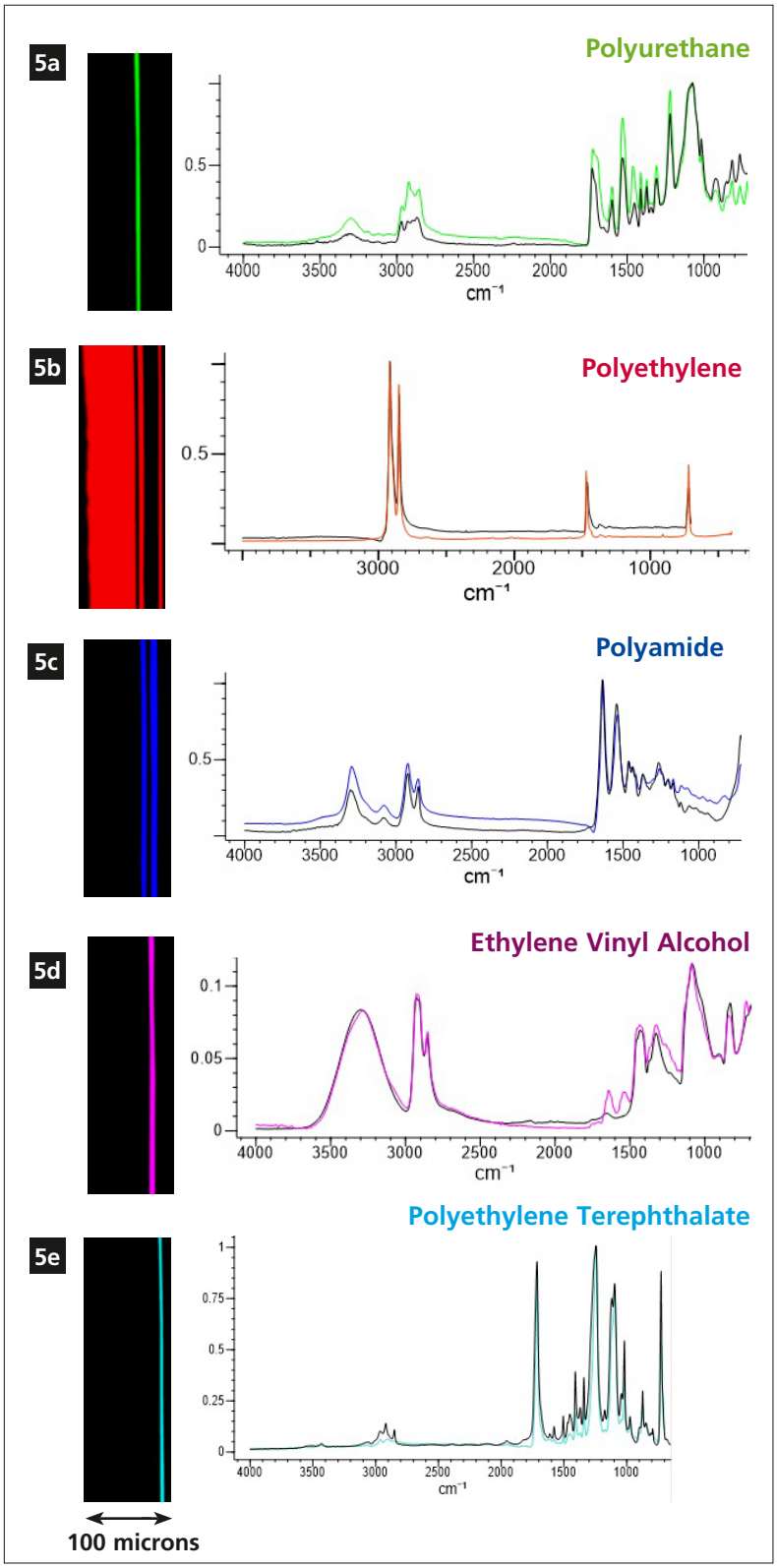

Figure 5. Individual component images (left), spectra with best library matches and their identity (right).

Table 1. Shows a summary of the layers detected, their polymer type and the thicknesses of the individual layers.

Layer Number	Identification	Layer Thickness/ Microns
1	Polyethylene	48
2	Polyurethane	4
3	Polyethylene	6
4	Polyamide	5
5	EVOH	3
6	Polyamide	5
7	Polyethylene	2
8	PET	4

Summary

The data collected by the PerkinElmer Spotlight 400 IR Imaging system provides valuable information on the composition of complex multilayer laminates. This allows AIMPLAS to provide detailed information to their customers about the quality of their packaging materials. The system could also be used for troubleshooting problems should they occur. The imaging data can be used alongside data from other analytical techniques available within AIMPLAS for complete characterisation of the materials and their properties.

References

1. 'Spatial Resolution in FT-IR ATR Imaging,' PerkinElmer Technical Note No. 007641_03 (2006).

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