Analysis of polymer laminates and coatings by automated FT-IR microscopy

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Introduction
FT-IR microscopy is an established analytical method for investigating polymers and polymer coatings on various substrates. Applications are:
- Determination of the chemical composition of laminates
- Investigation of the homogeneity of polymer coatings
- Analysis of failures in polymeric structures

To study polymeric samples by FT-IR in transmission thin sections have to be prepared by a microtome. Depending on sample properties thicknesses of 15 µm down to 2 µm are required to obtain good quality IR spectra. In contrast, FT-IR microscopy using Attenuated Total Reflection (ATR) allows the analysis of all types of polymer samples mostly without sample preparation. Though, the ATR crystals used in conventional FT-IR microscopes had to be placed without sample preparation. Though, the ATR crystals used in conventional FT-IR microscopes had to be placed and removed manually to change between IR and VIS mode. Performing the measurement procedure therefore required manual interaction of the operator and a relatively high level of method specific experience.

Bruker has now launched the first fully automated FT-IR microscope LUMOS (Fig.1).

Method
Measurements were performed with the stand-alone FT-IR microscope LUMOS using the ATR mode. Measurement positions were defined using the visual microscopic image. The complete measurement procedure was performed fully automated including the switch from transmission or reflection into the ATR mode and measurement of sample and background spectra with matching field apertures.

Data acquisition, evaluation and visualization was performed using the spectroscopic software OPUS (Bruker Optik).

Results
Polymer laminates from blister packages were analyzed by automated ATR mapping. The included polymer layers were identified by a comparison of the measured FT-IR spectra with spectral reference data. False color IR images show the signal intensities of characteristic absorption bands on the sampling area (Fig. 2b).

A polymer laminate was investigated on its top side in order to reveal if the top layer was damaged upon the removal of the cold sealing layer. An area of 3 x 3 mm was analyzed by automated ATR mapping with a lateral resolution of 100 µm. The data were evaluated by principal component analysis (PCA). The scores of the first three factors were plotted in false color in order to visualize the distribution of the top layer polymer, of the cold sealing polymer and of a contamination/clustered filler material, respectively. All three IR images were further combined in a red-green-blue (RGB) plot according to the “winner takes it all (WTA)” mode in order to reveal the full composition of the sampling area in one image (Fig. 3).

The contamination/clustered filler material was identified by a spectral comparison with a reference data set (spectral library) to be viscose.

Figure 1: Fully automated stand-alone FT-IR microscope LUMOS.

It comprises the following key innovations that provide for highest user comfort:
- Motorization and electronic coding of all movable components
- Automated ATR mode due to piezo-driven ATR crystal with 3 pressure settings for different sample hardness
- Automated switch between high numerical aperture (0.6) in IR for high sensitivity and lower numerical aperture (0.4) in VIS for high depth of focus

In this contribution, the automated mapping and imaging analysis of polymer laminates and coatings is shown.

Figure 2a: ATR line map on the cross section of a blister packaging. Colors of measurement positions and spectra are set accordingly.

Figure 2b: Chemical images calculated by integration of compound specific bands. The arrangement of the PVdC / Acrylate Copolymer (top), PU (middle) and PVC (bottom) layers in the laminate are visualized as 2D spot plots with size and color encoding.

Figure 3: Measurement of the cold sealing area of a polymer foil with a lateral resolution of 100 x 100 µm. The chemical images 1-3 show the score values of the first three factors from PCA as 2D contour plots.

Conclusion
IR-microscopic analysis of polymer samples using the ATR technique allows to determine the chemical nature of structures in the range of a few micrometers. As this technique does not require any demanding sample preparation and analysis times typically do not exceed minutes it is very suitable to be used for routine quality control and trouble-shooting. Due to the full automation of the FT-IR microscope LUMOS it was possible to establish a very intuitive workflow. Now the method is even applicable for inexperienced users.