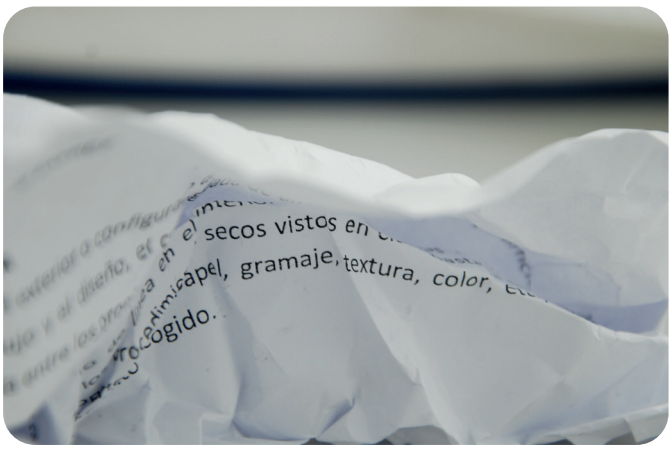

Application Note AN M137

Failure Analysis of Paper: Determination of the Chemical Identity of Impurities and Inclusions

Introduction

Modern paper consists of a multitude of different components. Besides fibers, glue, impregnating agents and auxiliary materials paper contains, depending on the type, up to 30% fillers. Typical filler materials are for instance calcium carbonate, talcum, kaolin, gypsum, and titanium dioxide. Thereby, material properties like printability, glare, or opacity can be optimized as required. In the finished product, the individual components are visually virtually indistinguishable, also defects often have only a low visual contrast and are therefore difficult to analyze.

FTIR spectroscopy is a measuring method by which the chemical identity of various organic and inorganic materials can be determined in a very rapid manner.

Since many paper types have a very inhomogeneous composition and defects are often extremely small, the analysis with macroscopic measurement methods is usually not possible. With the aid of an FTIR microscope, it is possible to measure an IR-spectrum anywhere on the sample with a high lateral resolution and thereby to determine the chemical composition of the defect. A mapping measurement allows the qualitative analysis of the components and the determination of the distribution on the measured sample area.

Keywords	Instrumentation and software
FTIR microscopy	LUMOS II FTIR microscope
Paper	OPUS spectroscopy software
Failure analysis	OPUS/SEARCH
Fillers	ATR-COMPLETE spectra library
Coatings	
Mixture analysis	

Figure 1: The LUMOS II FTIR microscope allows spatially resolved IR-spectroscopic analysis.

Instrumentation

The following examples of typical failure analysis were performed with the Bruker FTIR-microscope LUMOS II (see figure 1). The LUMOS II is a compact, stand-alone instrument with a complete automation of all device components. This results in a very intuitive, purely software-controlled operation of the microscope and allows an efficient use for routine questions. For most analytical questions, the attenuated total reflection (ATR) measurement technique is used. It allows to quickly measuring samples without, or with only minimal sample preparation. For the measurement of the IR-spectra, the LUMOS II microscope automatically contacts the user defined sample positions with the ATR-crystal measurement element. Particles, inclusions, and fibers can be analyzed within seconds and without time consuming sample preparation. Thanks to the complete automation of the ATR-measurement procedure, the LUMOS II is capable to perform grid measurements and thereby creating chemical images.

The operation is very comfortable via the dedicated OPUS video-wizard that guides the user through the whole measurement procedure and always provides the appropriate functions for the current measurement step.

Example: Analysis of a surface contamination

The first example shows the analysis of a contamination on the surface of a paper sample. Due to the low visual contrast, the contaminated area is only barely visible to the unaided eye. For the analysis and exact localization of the contamination, a grid measurement of 47x35 measurement points was performed covering an area of 2.25x1.75 mm. Hence, an area of 50x50 µm can be assigned to an individual spectrum. Figure 2 shows example spectra measured on a clean and a contaminated paper area, respectively. The spectrum of the contamination shows additional bands at 1100 cm^{-1} and 805 cm^{-1}.

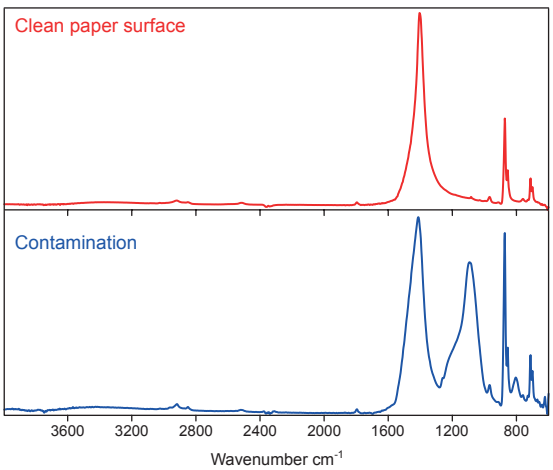

Figure 2: Spectra of the paper surface. Clean area (red) and contaminated area (blue).

The identification of the contamination was performed with the analysis software OPUS by a search in a digital library that contains reference spectra. As a result, a hit list of the best matching library spectra is shown. In case of spectra with bands of several components, a mixture analysis delivers the best fitting combination of library spectra. Figure 3 shows the identification result. In addition to the dominant component on the paper surface (calcium carbonate), a silicate was found as a further component in the region of the impurity.

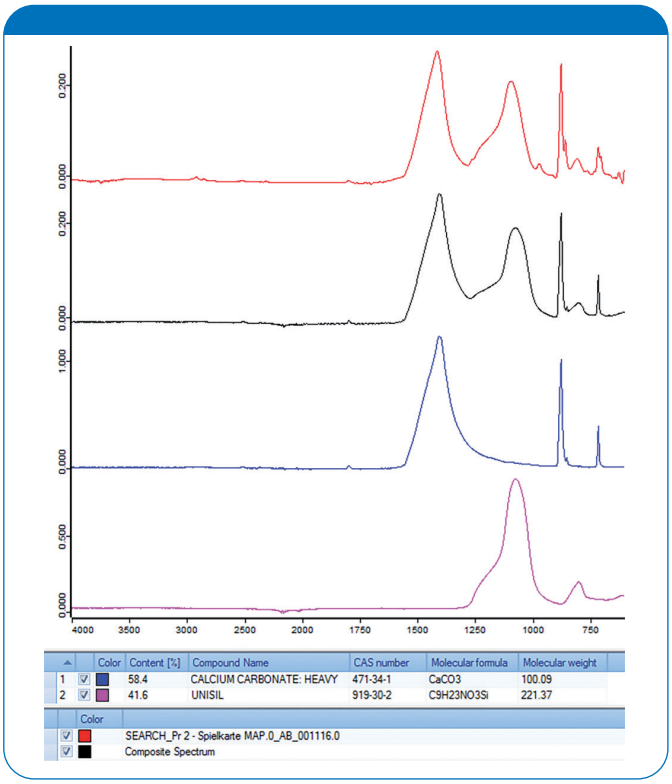

Figure 3: Result of a mixture analysis of the contamination (red). Sum spectrum is shown in black, the single components are calcium carbonate (blue), and Unisil (modified silicate, pink).

A chemical image of the distribution of the contamination can be made by the integration of the band at 1100 cm^{-1}. The resulting intensities are then shown color-coded in figure 4. They increase from black via green, blue, and red to white.

The chemical image has a much higher contrast than the visual image and allows the localization of the contamination. Besides, it is possible to draw semi-quantitative conclusions about the relative degree of coverage.

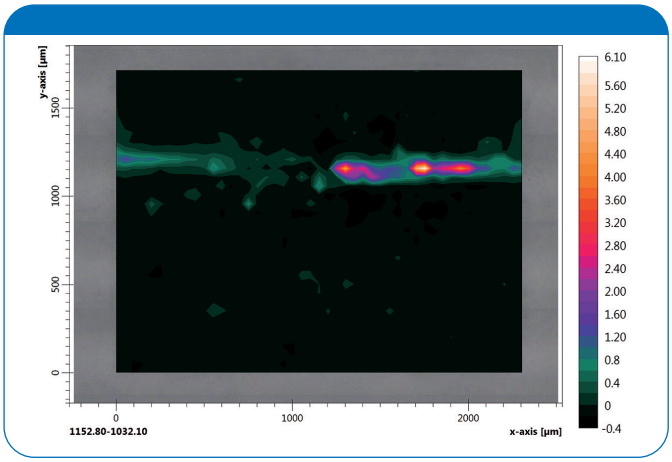

Figure 4: Chemical image that shows the distribution of the contamination.

Example: Analysis of a punctual defect

This example shows the identification of a punctual defect and the analysis of the distribution of the filler. A 2x3 mm sized sample area was analyzed by a mapping measurement in a way that a spectrum was acquired for each 100x100 µm sized area. Figure 5 shows the spectra of the defect and of the paper at two different positions. The paper-spectra show the typical bands of cellulose and a varying content of the filler calcium carbonate.

These bands are also clearly visible in the spectrum of the defect. In addition, further spectral bands are visible that cannot be explained by the bands of the cellulose or the calcium carbonate.

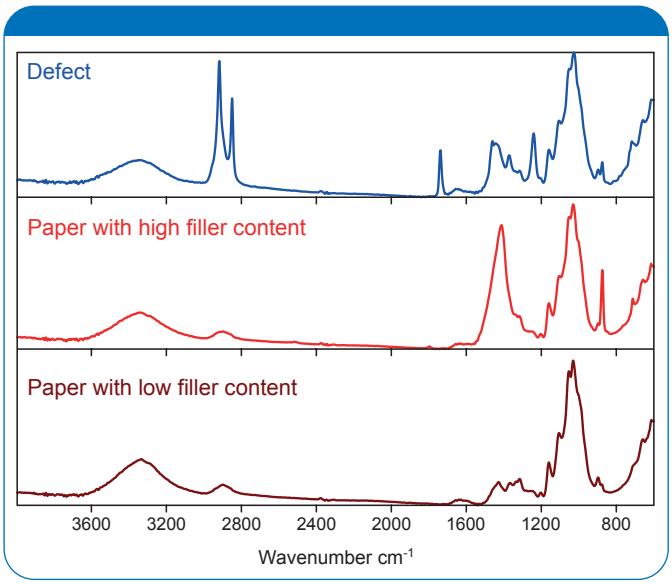

Figure 5: Representative spectra of the defect (blue), the filler (red), and the paper (brown).

The chemical composition of the defect was determined by a mixture analysis. The result (figure 6) shows the components paper, fatty acid ester ("Struktol") and the polymer ethylene-vinyl acetate ("Evatane").

For the visualization of the defect the carbonyl band around 1738 cm^{-1} was used, the chemical image is shown in figure 7. In order to show multiple components the so called WTA-color assignment ("winner takes it all") was chosen. It assigns the color of the dominating component to each individual image-pixel. In figure 8, the defect is shown in yellow and the calcium carbonate in red. At the dark areas, the paper therefore contains only a small proportion of filler.

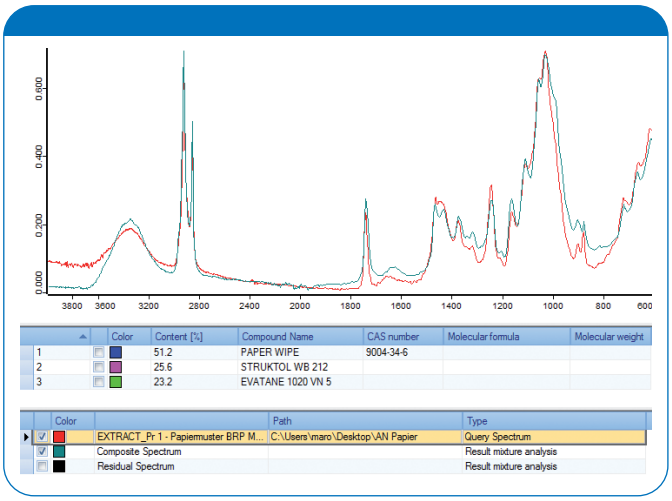

Figure 6: Result of the mixture analysis of the punctual defect. The composit spectrum calculated from the single component spectra (turquoise) almost perfectly matches the sample spectrum (red).

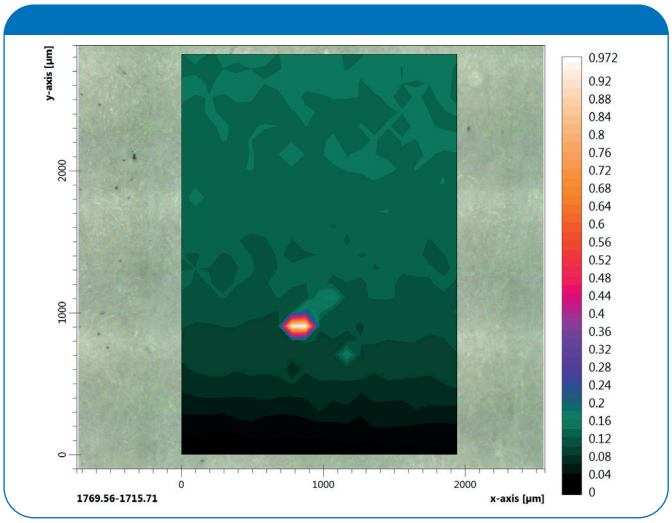

Figure 7: Chemical image of the punctual defect.

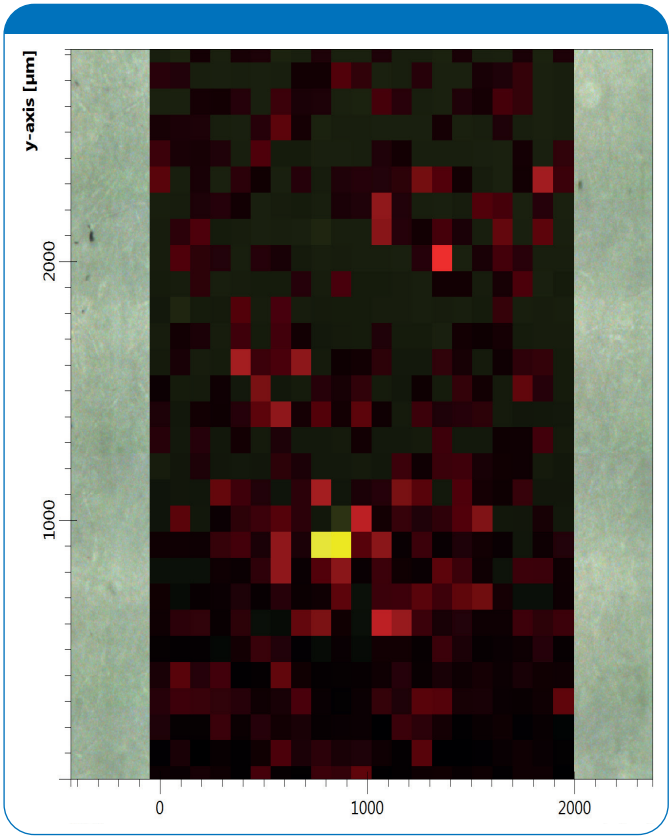

Figure 8: WTA-image that shows the distribution of the filler calcium carbonate (red) and the point defect (yellow).

Summary

The FTIR microscope LUMOS II allows to quickly localize and identify defects in paper. Thus, it is of great use in finding the cause of product defects in failure analysis.

Besides the visual analysis and point measurements, it is also possible to perform fully automated grid measurements. Thereby, chemical images are generated showing the distribution of the materials used in paper, such as fillers. Furthermore, it is possible to verify the homogeneity of coatings with a high lateral resolution.

With the aid of extensive spectral libraries and efficient functions for the spectra search and mixture analysis, it is possible to identify found components.

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