Application Note AN M130

Coating Analysis via FTIR Spectroscopy

Introduction

Coatings are applied to protect surfaces from scratches, damages and corrosion or to generally enhance the visual appearance of a product. Additionally coatings allow to modify surfaces for functional reasons such as electrical and thermal insulation or the improvement of the wettability. Applying the coatings incorrectly will result in product failure and failure and defects. For this reason, reliable quality control and appropriate analytical tools are essential.

First and foremost, it is important to determine the presence of the coating layer as well as its thickness. Furthermore the homogeneity of the layer has to be monitored. Besides using the correct coating material, absolutely clean surfaces are a crucial prerequisite for the correct application of coatings. In order to ensure technical cleanliness, it is extremely important to find the source of contaminations in case of product failures.

IR spectroscopy is a very powerful technique to obtain information about the presence and chemical identity of coatings. In addition to the verification of known coating materials, unknown substances can be clearly identified, which makes IR spectroscopy an indispensable tool in reverse engineering.

Keywords	Instruments and software
FTIR spectroscopy	ALPHA II FTIR spectrometer
FTIR microscopy	LUMOS II FTIR microscope
Identification of coating materials	OPUS spectroscopic software
Layer thickness determination	ATR-COMPLETE spectral library
Failure analysis	
Surface analysis	

Example: Analysis of coated pieces of costume jewelry

Pieces of costume jewelry are typically made of inexpensive materials like simulated gemstones which are set in nickel or brass. Often the metal parts are coated with a polymer layer to protect the surface, prevent color changes, enhance shine, and guard the human skin from allergic reactions.

The ALPHA II FTIR spectrometer is a very suitable analytical instrument to check the presence of the coating on such costume jewelry parts. Furthermore it allows to identify what kind of coating material has been used. Additionally the cleanliness of the metal surface can be verified prior to the coating process.

Figure 1 shows a piece of costume jewelry that is being analyzed with the ALPHA II FTIR spectrometer. The measurement is performed using the upward looking reflection sampling module. It allows a non-destructive analysis without the need of any sample preparation: the sample just has to be placed on the aperture hole where the measurement is performed in a few seconds. Clear instructions from the OPUS spectroscopic software interface guide the user through the complete workflow of measurement, data evaluation and reporting.

Figure 1: Exemplary piece of costume jewelry on the ALPHA II upward DRIFT.

In Figure 2 spectra are displayed that were measured on the surface of different jewelry parts. The upper spectrum results from a acrylic resin coating, the spectrum in the middle is typical for a clean metal surface (showing no absorbance bands), and the spectrum on the bottom originates from a piece of jewelry coated with polyurethane.

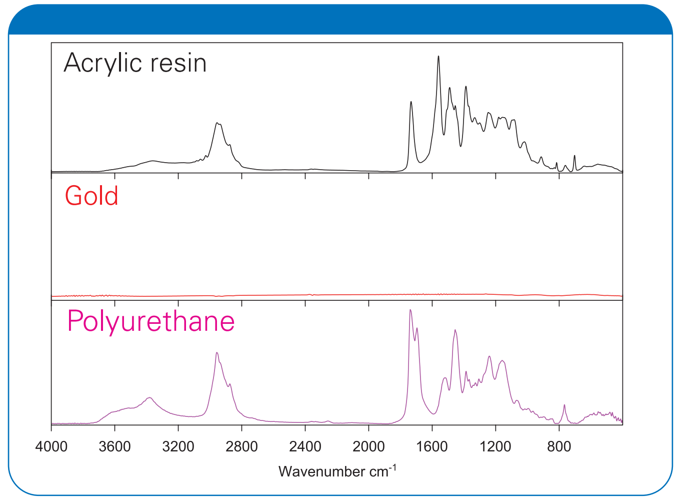

Figure 2: Example spectra taken on the surface of jewelry.

To identify an unknown coating material, its spectrum can be automatically compared against a set of reference spectra stored in a digital library. Figure 3 shows the search result for the upper spectrum in figure 2. The query spectrum (red) is displayed together with the spectrum of the first hit (blue). Measured and reference spectrum are in good agreement whereby the coating is clearly identified as poly(methyl methacrylate).

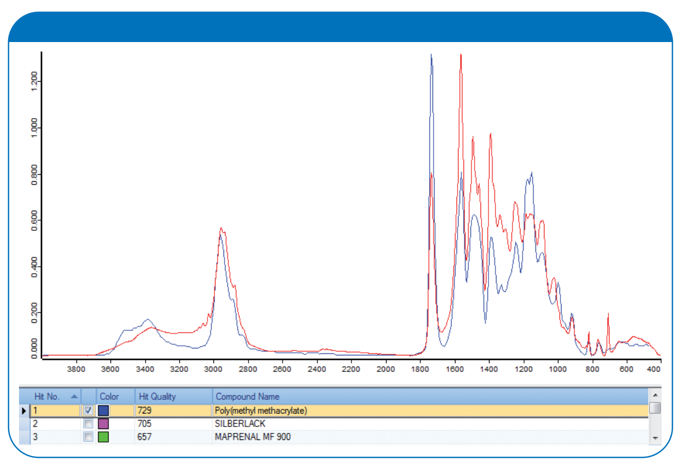

Figure 3: Identification of the upper example spectrum of figure 2 as PMMA by library search. The query spectrum is colored red.

Example: Measurement of an insulated copper wire

Enamelled copper wire is mainly used for the construction of electronic coils and has a typical diameter in the range of 0.02 - 6 mm. On modern wires usually a very thin insulation from polymer film is applied. Defects in this coating can have dramatic effects like leakage and short circuits that may ultimately result in the destruction of the whole circuit.

IR spectroscopy allows to check the presence of the polymer coating on the wire and identifies its chemical nature. Since the sample is very small, a macroscopic analysis as in the previous example is not possible. Instead, an IR microscopic approach is required. The LUMOS II FTIR microscope (Figure 4) allows the chemical analysis of samples and structures that are only a few microns in size. LUMOS II is a stand-alone system that is very easy to use thanks to its full automation and intuitive analysis software. Furthermore its design is very compact and space saving.These features make the LUMOS II the perfect choice for routine analysis.

Figure 4: FTIR microscope LUMOS II: Effective quality verification of coatings on smallest samples

In the following example, an enameled copper wire was analyzed with the LUMOS II. In order to identify the insulation material, a measurement applying the ATR (attenuated total reflection) technique was performed. For the measurement the wire was fixed on the microscopic stage and then measured without further preparation. Figure 5 shows a representative spectrum together with the result of a spectrum search that was performed for identification. The high similarity of the measured spectrum and the search result identifies the coating to be thermoplastic polyurethane (TPU).

In a separate approach the homogeneity of the coating layer thickness was analyzed by performing a line map reflection measurement on the wire surface. For this purpose a line of 15 measurement positions was measured fully automated with the LUMOS II. By using the automated knife edge apertures of the LUMOS II, the measurement area of each individual position was limited to about 60x60 µm.

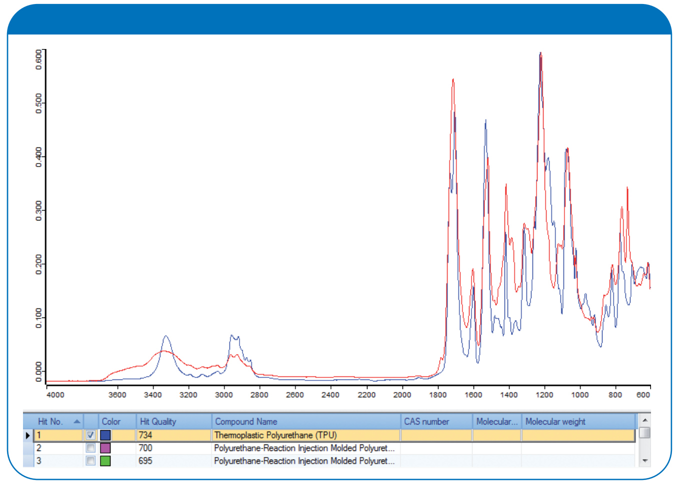

Figure 5: Spectrum search result of the insulation layer of the enamel wire. The query spectrum (red) shoes a high similarity with a polyurethane spectrum from the spectral library (blue).

Reflection measurements on optically transparent thin layers result in so called fringes in the IR spectrum. Fringes are a phenomenon based on the interference of the light that is reflected on top of the coating layer and the light that is reflected on the copper metal of the wire. The light rays reflected on the copper wire travel longer and an optical retardation is introduced. The wavelength dependent interference finally results in a sinusoidal baseline of the spectrum. As an example, figure 6 shows fringes of two different spectra.

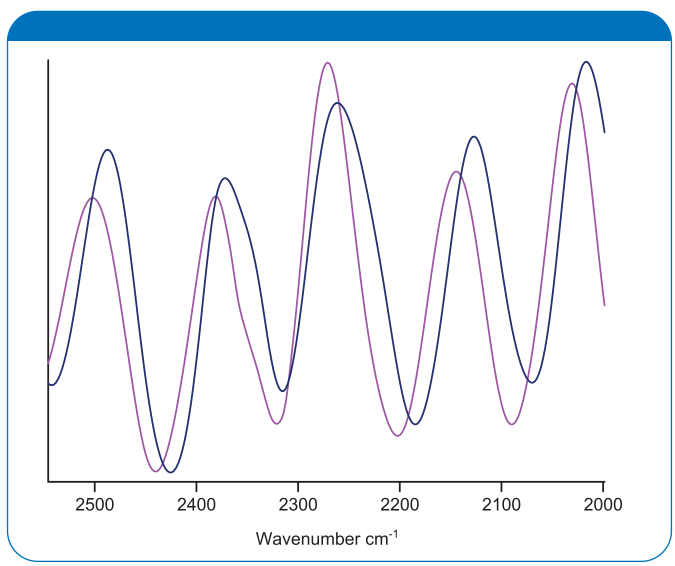

Figure 6: Spectral region with fringes resulting from a thin coating layer. The difference in the fringes is a result from slightly varying layer thickness.

The calculation of the layer thickness from the spectra was performed by the OPUS spectroscopic software. In order to determine the layer thickness correctly, the refractive index of the layer material is needed. Although the exact value is not known, a generic value of 1.5 can be assumed for transparent polymers. Figure 7 shows a mapping of the determined thickness superimposed over the visual image. A layer thickness of about 25 µm was determined for a large part of the wire. Only an area that was slightly scraped with a knife prior to the measurement shows a crack where the layer was removed and a thicker area where the excess material was moved to.

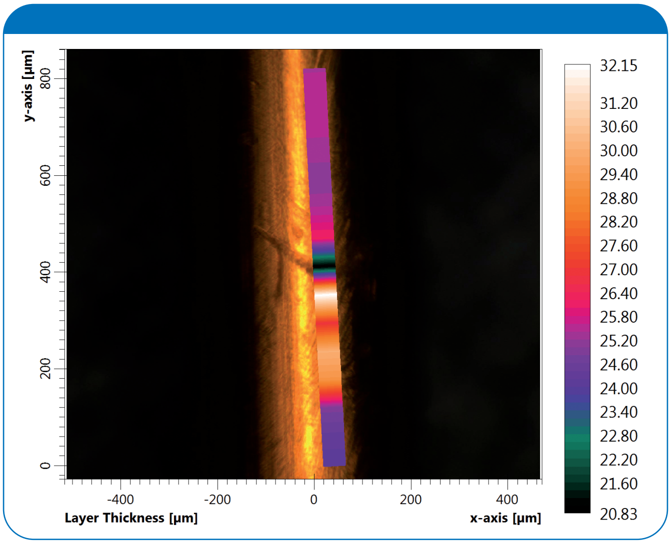

Figure 7: Layer thickness determination of an enamel wire.

The determination of the layer thickness by interferometric measurements has several major advantages for being non-destructive, very fast, contact-free and without limitations regarding the shape of the work piece. The method is also applicable for other coating materials than polymers, e.g. Diamond Like Carbon (DLC) layers on tools.

Summary

FTIR spectroscopy is a valuable method for the analysis of coatings. It allows to identify the chemical identity of the used material and to determine the thickness of coating layers. The method is nondestructive, fast, and precise.

Bruker's ALPHA II is an easy to use system for routine quality control and identification of surface coatings.

Coatings on small and unevenly shaped samples can be analyzed using the fully automated FTIR microscope LUMOS II. It allows to monitor the homogeneity of a coating with high spatial resolution and to identify small defects and contaminations for effective failure analysis.

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