

## METHODS FOR EVALUATION OF PAINT COATINGS COMPOSITION

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### Abstract

The paper describes combination of optical and electronic methods together with IR analysis for evaluation of the paint coatings. Based on these methods the fingerprint of each paint layers is identified according to requirements of ISO 12944-9. The database of numerous typical industrial painting systems is created. These data allow to identify composition of the existing systems before their renovation or the mistakes in newly applied painting systems, e.g., different type of binder, pigments, etc.

**Keywords:** Paint system analyses, IR method, electronic scanning microscopy

### 1. INTRODUCTION

Paints are very complication system and mixture of the organic and inorganic substances. This is reason for more complicated identification and clarification of the causes of defects. This is also very important in order to prevent errors and thus ensure the required long life of corrosion protection. In addition to physical methods such as measuring the thickness of the protective film, its adhesion, gloss, colour, checking the cleanliness of the surface, which can be performed in the field, it is necessary to obtain samples for laboratory evaluation. In the laboratory it is then possible to perform further analysis and microscopic examination. A paint examination always begins with a microscopic examination of the samples and reference coating used for comparison. The mutual combination of microscopic evaluation, EDX-SEM, FTIR analysis, is important for the detection of possible defects.

Since paint consists of an organic binder as well as inorganic fillers and pigments, it is need analytical techniques that can characterize a paint sample in both of these areas. FTIR spectroscopy and EDS spectroscopy are two techniques perfectly suited for the task of paint analysis. FTIR spectrum is unique for a given organic material and can be thought of as the materials "chemical fingerprint". The area of the FTIR spectrum between 1 600 – 4 000 cm<sup>-1</sup> is commonly referred to as "functional group region" (fingerprint) and is primarily composed of peaks corresponding to the stretching motions of the functional groups [1]. Both bulk and surface analysis can be performed with FTIR depending on the mode of the analysis performed [2-4]. The special holder has been developed for FTIR analysis of multilayer paint systems allowing analysis of each layer. This holder enables analyses of individual, gradually removed layers in a given specific place.

The paper gives some results of studies related to the combination of methods for clarifying defects in protective coats of car body as example of complex analysis. Modern automobile paint is applied in several layers, with a total thickness of around 100 µm (0.1 mm). This coating system is multi-layered with different composition of each layer. A basecoat is applied after the primer paint. Following this, a clearcoat of paint may be applied that forms a glossy and transparent coating. The top clearcoat layer must be able to withstand UV light.

Paint defects can have many causes. Maybe owner had an accident, or maybe the car was exposed to aggressive atmospheric conditions. It is also possible, that mistakes were made during the coating process. In industrial areas the chemicals released into atmosphere reacting with pollution in the air to damage paint coating of car body.

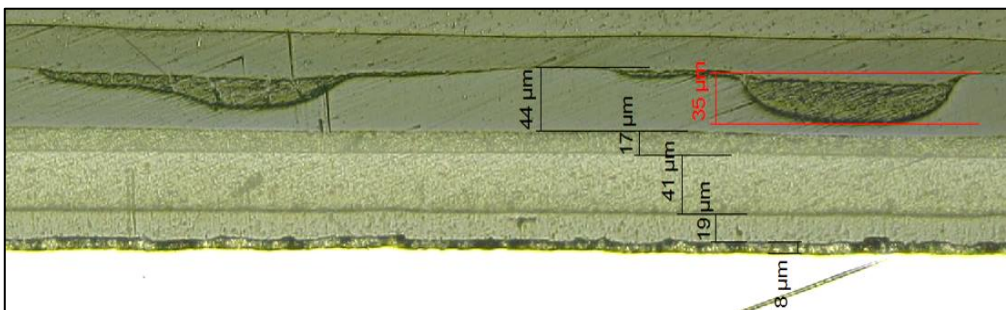
**2. MICROSCOPIC EVALUATION**

**2.1. Optical microscopy**

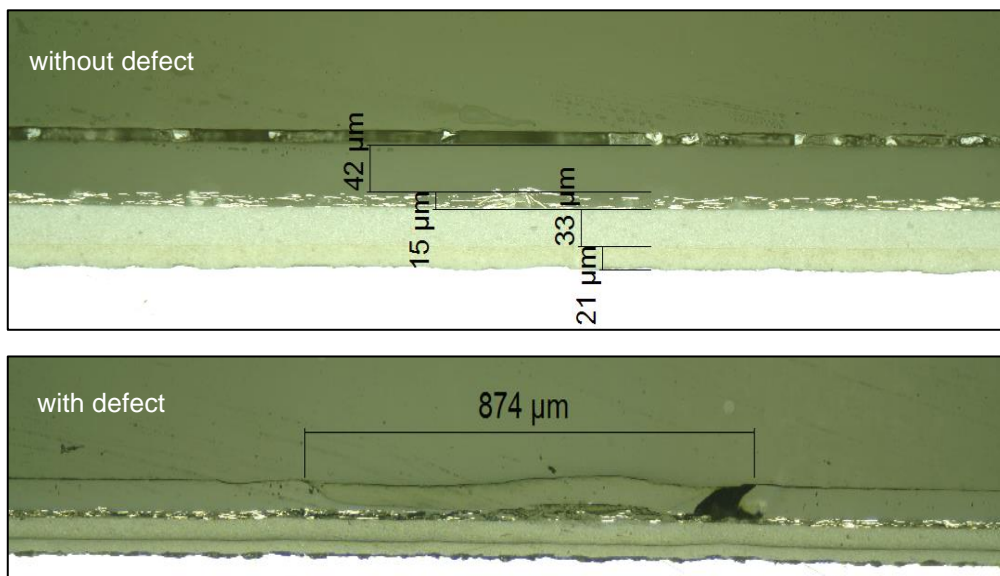
Optical microscopy evaluation was used to evaluate car body paint defects. Two types of car paint system were evaluated: white colored coating (sample 1) and anthracite metallic pigmented coating (sample 2). Both paint systems showed defects in the form of cracks, coloured spots or peeling in the clear coat (**Figure 1**). In the field the defects of coating were documented by portable microscope with 50x magnification.



**Figure 1** Defects in the car coating (left sample 1, right sample 2)



**Figure 2** Microscopic evaluation – sample 1 cross section – holes in clear lacquer layer



**Figure 3** Microscopic evaluation – sample 2 cross section – holes in clear lacquer layer, clear coat degraded – corrosion of Al pigment

Detailed evaluation was done. **Figure 2** and **Figure 3** show cross section of car body coatings, in which a significant thinning of the top clear coat is visible. Depth of the defect was different, from 10  $\mu\text{m}$  to 40  $\mu\text{m}$ , practically peeling clear coat. This evaluation was done in laboratory by light microscope Zeiss NEOPHAN 32. Light microscope cross section allows to identify the number of coating layers, the thickness of each one, and possible the structure of layer, especially the metallic pigment particles are evident – shape, size, amount, distribution, etc. In case of sample 2 the deterioration of top layer and corrosion of aluminium pigment particles is evident.

## 2.2. SEM+EDX analysis

This analysis showed a higher sulfur content, in range 2 – 6 wt. % in stain in comparison to clear surface. In this industrial area, where the deterioration of car body coating occurred, steel and zinc coupons were exposed for 1 month to estimate the atmospheric corrosivity. EDAX analysis of corrosion products from corrosion coupons identified sulphur content in range 4.5 wt. %. There is good correlation between air pollution level and contamination of paint surface.

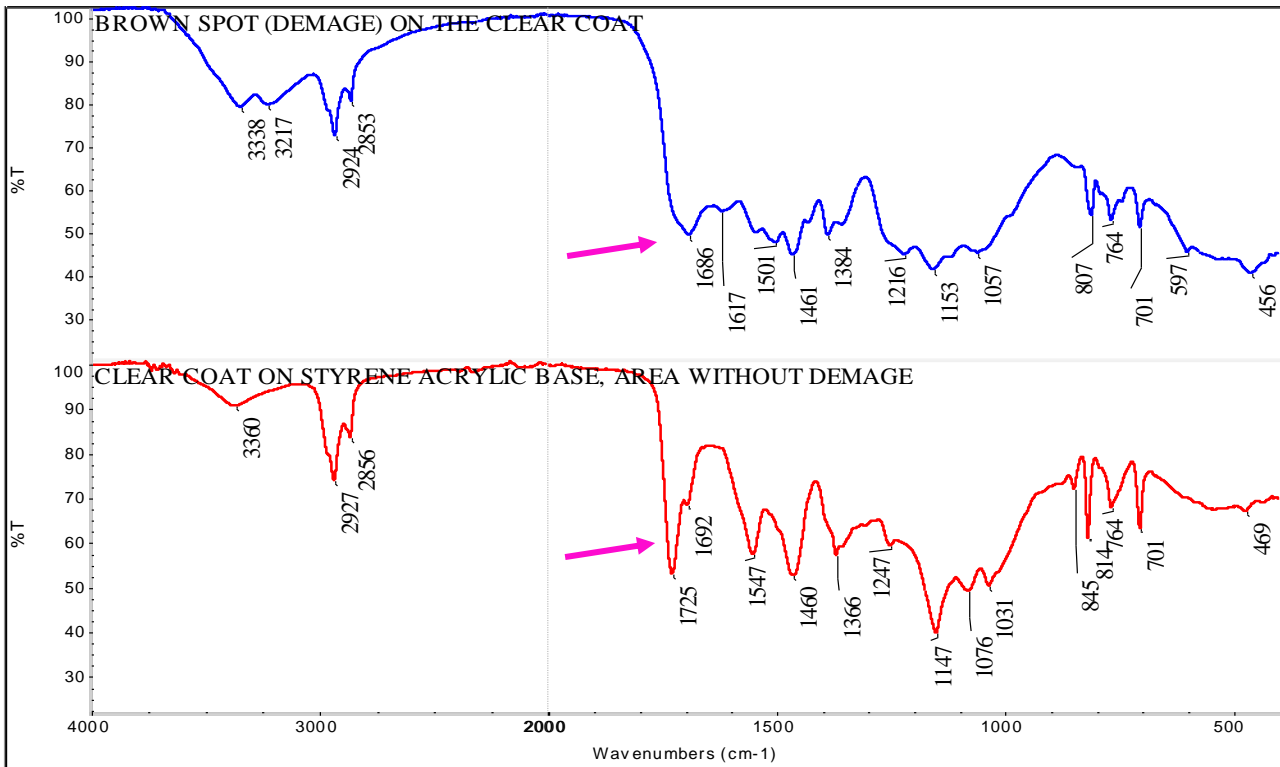
## 3. FTIR ANALYSIS

The special holder (**Figure 4**), has been developed for FTIR analysis of multilayer paint systems. This holder enables the analyses of individual, gradually removed layers using a very fine sandpaper in a given specific place of paint sample and improve quality of spectra. Holder ensure the measurement of the spectra of individual layers of coating systems exactly by removed 10  $\mu\text{m}$  due rotation of the micrometric screw.



**Figure 4** The special holder for FTIR analysis

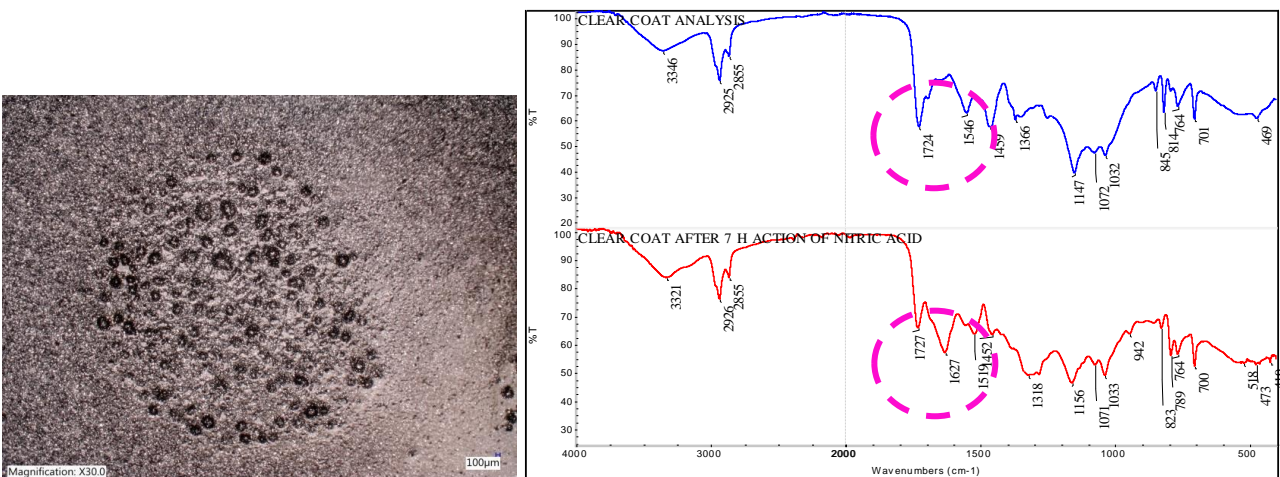
FTIR analysis of sample 1 top coat determinates styrene-acrylate copolymer like binder. In stain area the peak characteristic for C=O bond is missing. From the spectra it was found that hydrolysis of top layer had to take place (**Figure 5**).



Description: blue line – brown spot (damage) on the clear coat  
red line – clear coat on styrene acrylic base, area without damage

**Figure 5** FTIR spectra - difference of peaks in the area of hydrolysis (arrows)

The acidic contamination of paint surface by sulfuric and nitric acid into consideration as main air pollution sources. During laboratory simulation the mechanisms of degradation of the top paint layer caused by both acids was different. While sulfuric acid promoted by UV radiation caused hydrolyses, nitric acid caused swelling of the top coat without hydrolysis (**Figure 6**).



Description: blue line - clear coat analysis  
red line - clear coat after 7 h action of nitric acid and UV

**Figure 6** FTIR spectra of top layer after nitric acid effect and its swelling

#### 4. CONCLUSION

With a variety of paints and coatings come a variety of modes by which these products can experience failure. Paint analysis problems and paint failures present a unique challenge to the modern scientist, however the combined information obtained from both FTIR and EDS techniques allows for the interpretation and diagnosis of a great variety of paint analysis problems.

In this case study all the methods used to detect defects in the car top coat are indicative of degradation by sulfuric acid with the interaction with UV radiation. This pollutant takes specific defect mechanisms and kinetics. The laboratory simulation of dilute H<sub>2</sub>SO<sub>4</sub> drops application shows the same degradation like car body coating. The source of evaluated damage can be air pollution near the chemical areal. The other possible air pollution by HNO<sub>3</sub> does not show the same degradation mechanism.

Not only the different commercial paint formulations can be distinguished from each other when using FTIR, but degradation of each paint can be identified, too. For this evaluation the database of various paints is necessary to create.

#### ACKNOWLEDGEMENTS

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