Applied Comparison in Coating Analysis – Micro-XRF and Handheld XRF

Bruker Nano Analytics, Berlin, Germany
Webinar, July 5th 2017
• Presenters
• Principles of XRF Coating Analysis
• XRF Types and Comparison
• M1 MISTRAL Introduction
• S1 TITAN Introduction
• Live measurements
• Questions and Answers
Our Expertise

Presenters / Moderators

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Application Scientist
Bruker Nano Analytics, Berlin, Germany
X-Ray Fluorescence
In Coating Analysis

Excitation

- X-rays can penetrate and excite matter
- Signal from base material and covered layers can still be detected
- X-rays are attenuated in characteristic ways pathing through matter
- Intensity ratio of observed elemental lines, allows for calculation of respective layer thicknesses

XRF Detection

Silicon Drift Detector with XFlash® Technology
Comparison of stacks shows:

- Attenuation of signal from buried elements
- Increased signal strength from top layers for thicker layers
- Even though Ni reaches saturation thickness at about 25 µm, underneath 5 µm of gold the Ni intensity is reduced to about 2 %
- The overall thickness of stacked layers is limited to about 30 µm, depending on elements and matrix density
Approximate accessible thickness range for individual layers of one element

For analysis by XRF emission:

- The higher the photon energy the thicker the films that can be analyzed
- The lower the photon energy the more sensitive the method is for thinner layers
XRF Instrument Types
Bruker Overview

- S1 Titan, M1 Mistral, M2 Blizzard, Tracer 5i, M4 Tornado, M6 Jetstream
M1 MISTRAL
Key Features

- Benchtop “small spot” XRF spectrometer
- Full protection unit with German type approval (D)
- Element range from K to U
- **Collimator** changer (0.1 - 1.5 mm)
- Top view video camera (30x)
- **Programmable X-Y-Z-stage** (travel range / max. sample size in mm: 200x175x80)
- **Touch UI**
- **Method editor** for fast method setup and calibration
S1 TITAN
Key Features

• Ultimate portability, measure samples anywhere
• Multipurpose instrument for coatings, alloys, plastics and many others
• Rugged and weatherproof, designed for outdoors and harsh conditions.
• Very easy to use, minimal user training required

• 50kV X-ray tube and SDD detector for speed and low detection limits
• Weight only 1.5 kg
• Element range Mg - U
• Measurement spot size 3 to 8 mm

Portable  Fast an Easy  Large parts
## Instrument Comparison

### Handheld XRF & Microspot XRF

<table>
<thead>
<tr>
<th>Features</th>
<th>Microspot XRF</th>
<th>Handheld XRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single layer metal coatings (Zn/Fe, Cr/Fe etc.)</td>
<td>✔️ ✔️</td>
<td>✔️ ✔️</td>
</tr>
<tr>
<td>Multi layer analysis (Cr/Ni/Cu etc.)</td>
<td>✔️ ✔️</td>
<td>*</td>
</tr>
<tr>
<td>Very large and heavy parts</td>
<td>n/a</td>
<td>✔️ ✔️</td>
</tr>
<tr>
<td>Electronic components</td>
<td>✔️ ✔️</td>
<td>✔️</td>
</tr>
<tr>
<td>Small samples (&lt;1mm)</td>
<td>✔️ ✔️</td>
<td>✔️</td>
</tr>
<tr>
<td>Light element coatings (Mg, Al, Si, P)</td>
<td>*</td>
<td>✔️ ✔️</td>
</tr>
<tr>
<td>Plating bath liquid analysis</td>
<td>✔️ ✔️</td>
<td>✔️ ✔️</td>
</tr>
</tbody>
</table>

**Recommended ✔️ ✔️**  **Limited Performance ✔️**  **Upcoming ***
• Single points, lines, arrays within 200 mm x 175 mm (Z = 80 mm)
• Optional reference points for repeated sample load (2D)
• Easy load options (on, Z, XY)
M1 MISTRAL
Method Setup and Tolerances

- Calibration is automated to a great extent
- Optional user choice of polynomial degrees, calibration ranges or individual parameters to be added, weighted or excluded
- Methods available via hot keys
M1 MISTRAL
Automated Measurements

- Base correction for increased accuracy in case of repeated elements
- Multi-point measurements are evaluated with respect to preset tolerances
- Table of results or trend line can be displayed together with statistical values
M1 MISTRAL
Automated Reports

- Bruker ESPRIT report manager module
- Versatile report template creation
- Process relevant information easy to add to report
M1 MISTRAL
Applications

Coating analysis in General metal finishing

- Galvanic & Sanitary & Automotive
- Multilayer coatings for protection, hardening, decoration

Coating analysis in Electronics

- Industrial component production
- Multilayer coatings for functionality of contacts and components

Coating analysis in Jewelry

- Non- and precious metal contents according to governmental regulation
- Multilayer coatings for (fashion) decoration
### S1 TITAN Applications

<table>
<thead>
<tr>
<th></th>
<th>Cutting Tools</th>
<th>Construction/ Fasteners</th>
<th>Auto/Sports/ Architectural</th>
<th>Aerospace PowerGen</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>EXAMPLES</strong></td>
<td><img src="image1" alt="Cutting Tools" /></td>
<td><img src="image2" alt="Construction Fasteners" /></td>
<td><img src="image3" alt="Auto/Sports/Architectural" /></td>
<td><img src="image4" alt="Aerospace PowerGen" /></td>
</tr>
</tbody>
</table>
| **COATINGS**        | • Cobalt leaching  
• W-carbide/Tool Steel  
• Ti-carbide/Tool Steel | • Zn / Fe  
• Cr / Fe  
• Ni-P / Fe, Steel  
• Zn-Ni / Fe | • Cr / Steel  
• Ni only/substrate  
• SnCo/Fe  
• Ti / Steel  
• TiZr / Steel | • Y-TBC  
• Mo/Ti  
• Cu/Al |
| **PURPOSE**         | • Wear resistance  
• Corrosion resistance high temp | • Wear resistance  
• Corrosion resistance  
• Passivation coating | • Corrosion resistance  
• Decorative | • High temp resistance  
• Wear resistance  
• Lubricity |
S1 TITAN
Operation Modes

- Handheld
  - For point and shoot measurements

- Portable Desktop Stand
  - For field measurement

- Interlock secured Benchtop Stand
  - For stationary use
TiZr conversion coatings

• In the automotive industry TiZr coating is used widely for steel (CRS, EG, HDG) and aluminum

• TiZr offers multiple advantages over traditional tri-cation phosphate coating
  • Increased Corrosion Resistance
  • Salt Spray Performance
  • Reduced Environmental Impact
  • Reduced Wastewater Treatment

• Each sample was measured three times (different spots)
• Correlation coefficient $R^2$: 99.6%
• Calibration Error 0.7 mg/m$^2$
S1 TITAN
Coating Calibration Options

- Qualitative Coating Analysis
- Type Standardization
- Standard Coating Calibrations from factory
- EasyCal User Calibrations
Instrument Comparison
Summary of M1 and S1

• Microspot XRF and handheld XRF are complementary coating analysis techniques which both provide own unique benefits
• Micro-XRF is the best suited for quality control with repeating patterns, such as connector contacts on PCBs
• Micro-XRF instrument features programmable XYZ stage and spot sizes down to 100 µm for small sample analysis
• When a large sample size prevents analysis with tabletop instruments or portability is important, handheld XRF is the perfect alternative
• Bruker S1 TITAN can easily measure large parts or at difficult-to-reach locations in general metal finishing, automotive and aerospace industries
## Sample Overview

<table>
<thead>
<tr>
<th>Memory Chip</th>
<th>Faucet Handle</th>
<th>Automotive Part</th>
<th>Surprise</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Size</strong> (WxDxH)</td>
<td><strong>Layer</strong></td>
<td><strong>Question</strong></td>
<td><strong>Instrument</strong></td>
</tr>
<tr>
<td>70 x 30 x 5 mm</td>
<td>Au/Ni/Cu-PCB</td>
<td>Homogeneity of coating thickness</td>
<td>M1 and S1</td>
</tr>
<tr>
<td>120 x 60 x 60 mm</td>
<td>Cr/Ni/Cu/Zamac</td>
<td>Homogeneity of coating thickness</td>
<td>M1 MISTRAL</td>
</tr>
<tr>
<td>140 x 140 x 50 mm</td>
<td>Zn/Fe</td>
<td>Homogeneity of Zn coating thickness</td>
<td>S1 TITAN</td>
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<tr>
<td>?</td>
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Questions, Thoughts, or Comments?

To contact us during the Webinar please type your questions, thoughts, or comments in the Q&A box and press Submit.

Please accept our sincere apologies if we can’t discuss all comments and questions within the session!

We will of course answer and discuss those later on by e-mail or in another Webex meeting.
Innovation with Integrity