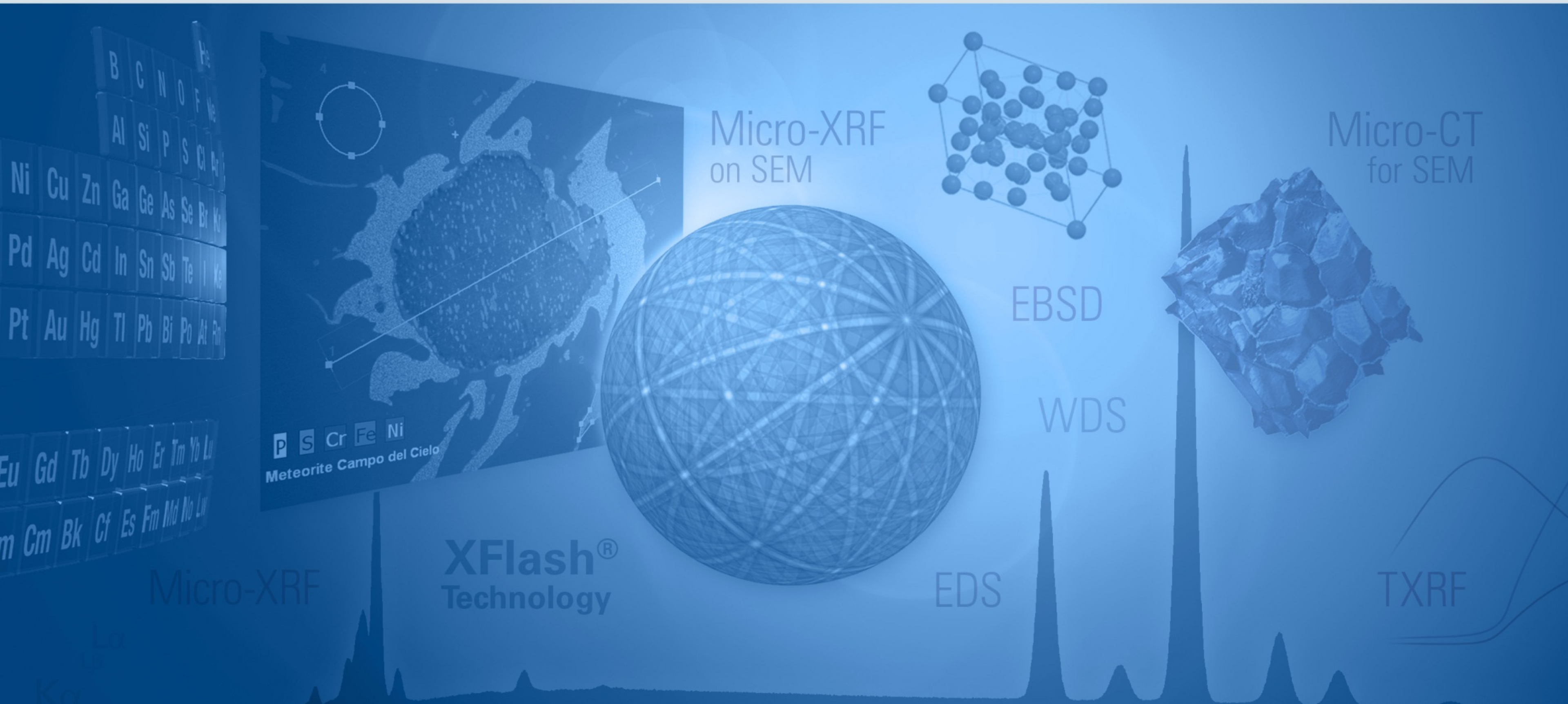
Spatially Resolved Layer Thickness Analysis of Thin Alloy Coatings with Micro-XRF

Bruker Nano Analytics, Berlin, Germany
Webinar, May 12, 2016



Presenters

Falk Reinhardt

Application Scientists micro-XRF,
Bruker Nano Analytics, Berlin, Germany

Samuel Scheller

Sr. Product Manager micro-XRF,
Bruker Nano Analytics, Berlin, Germany

Overview

- Introduction
- The M4 Tornado micro-XRF Instrument
- Example 1 – Ti / ZnAlMg coating on sheet metal
- Example 2 – Cu-Al coating on glass
- Summary
- Live demonstration
- Questions

Introduction

- Please also have a look at our Webinar on Layer Analysis:

  "Automated Analysis of Decorative and Functional Coatings with the M1 MISTRAL Micro-XRF Instrument" from the 4th May 2016

- The M4 Tornado is a high speed micro-XRF instrument for challenging applications and element distribution analysis
- We want to present you with a couple of analysis examples looking at metal and alloy layers,
- Also, we want to provide a live demonstration of the instrument
- Any questions you may have, we want to answer and discuss

M4 Tornado Micro-XRF Spectrometer
Standard configuration

30 W microfocus X-ray Tube

- Rh target
- Polycapillary lens with spot size < 20 µm
- Resolve micro structures
- Effective for all material types

Silicon drift detectors (SDD)

- 30 mm² with energy resolution < 145 eV (for Mn-Kα)
- Optional 60 mm² with energy resolution < 145 eV
- Second detector option for double speed

M4 Tornado Micro-XRF Spectrometer
Standard configuration

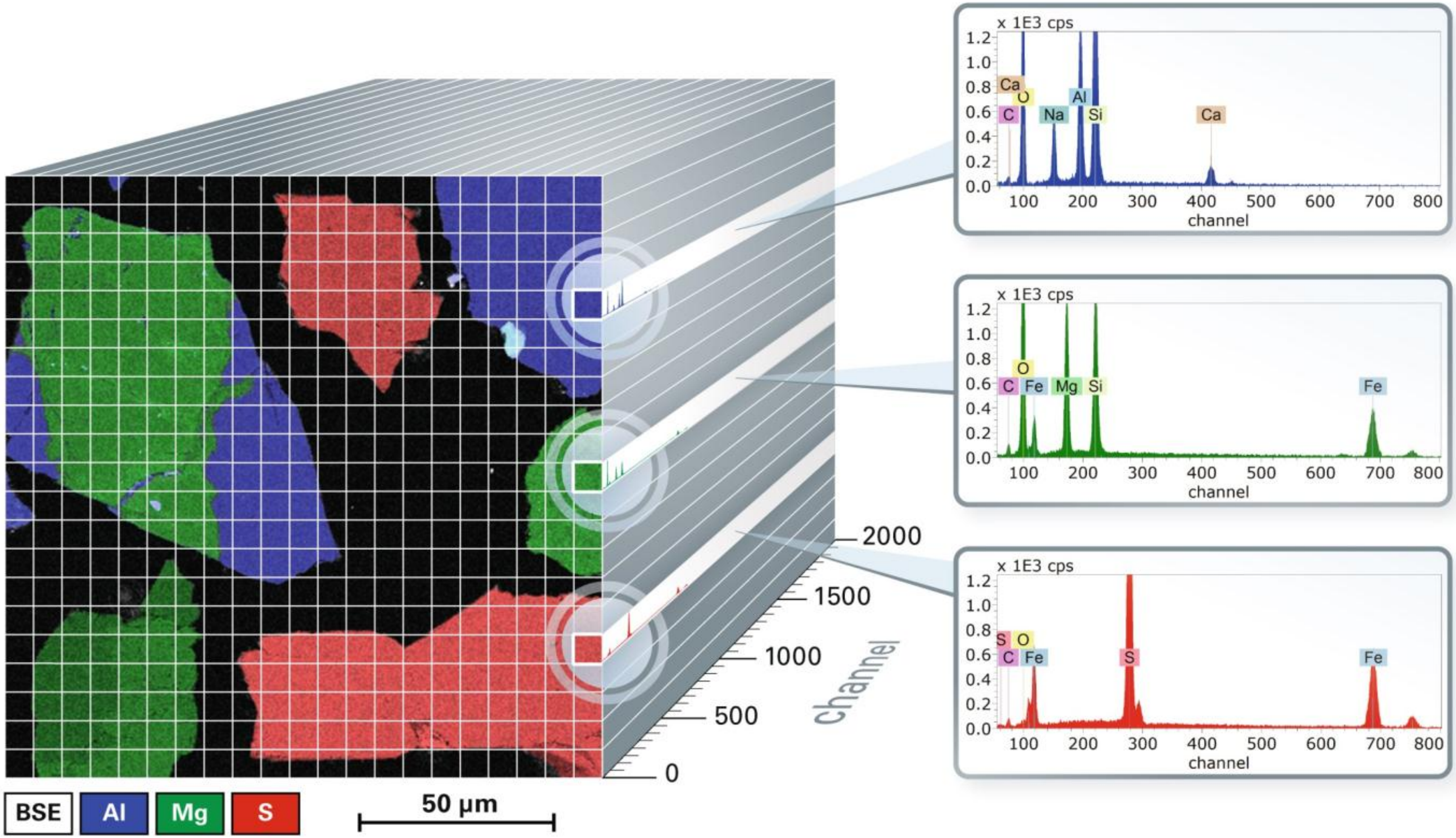
Vacuum sample chamber

- Pressure range from 1 mbar up to atmosphere
- Light element detection down to Sodium
- Stage 190 mm x 160 mm
- Maximum sample height of 120 mm
- 5 kg capacity
- Stage measurement speed up to 100 mm/s
- 4 µm minimum step size

Advantages

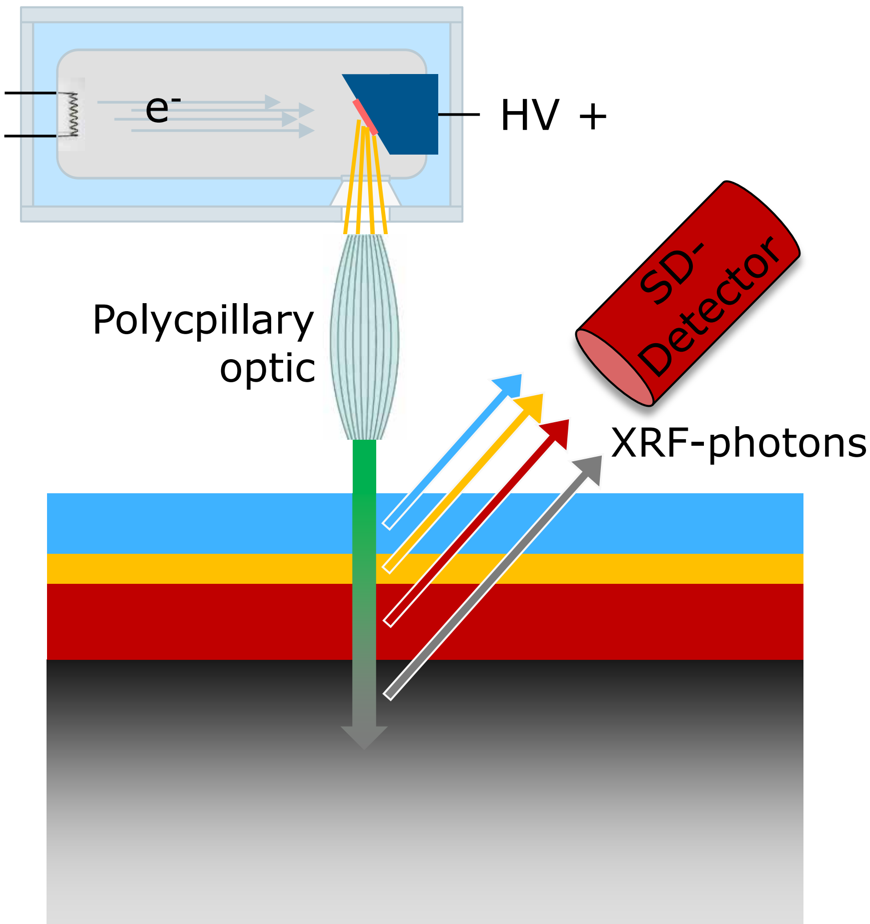
- Non-destructive, spatially resolved material analysis
- Requires no sample preparation
- Quality control screening measurement of structured samples
- X-ray „depth view" is ideal for analysis of thin alloy layers

Position Tagged Spectrometry Imaging
The HyperMap Database

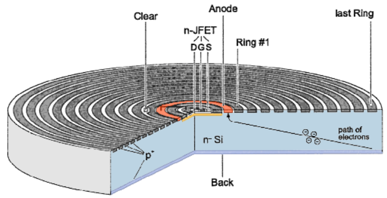


X-Ray Fluorescence
…in Coating Analysis

Excitation



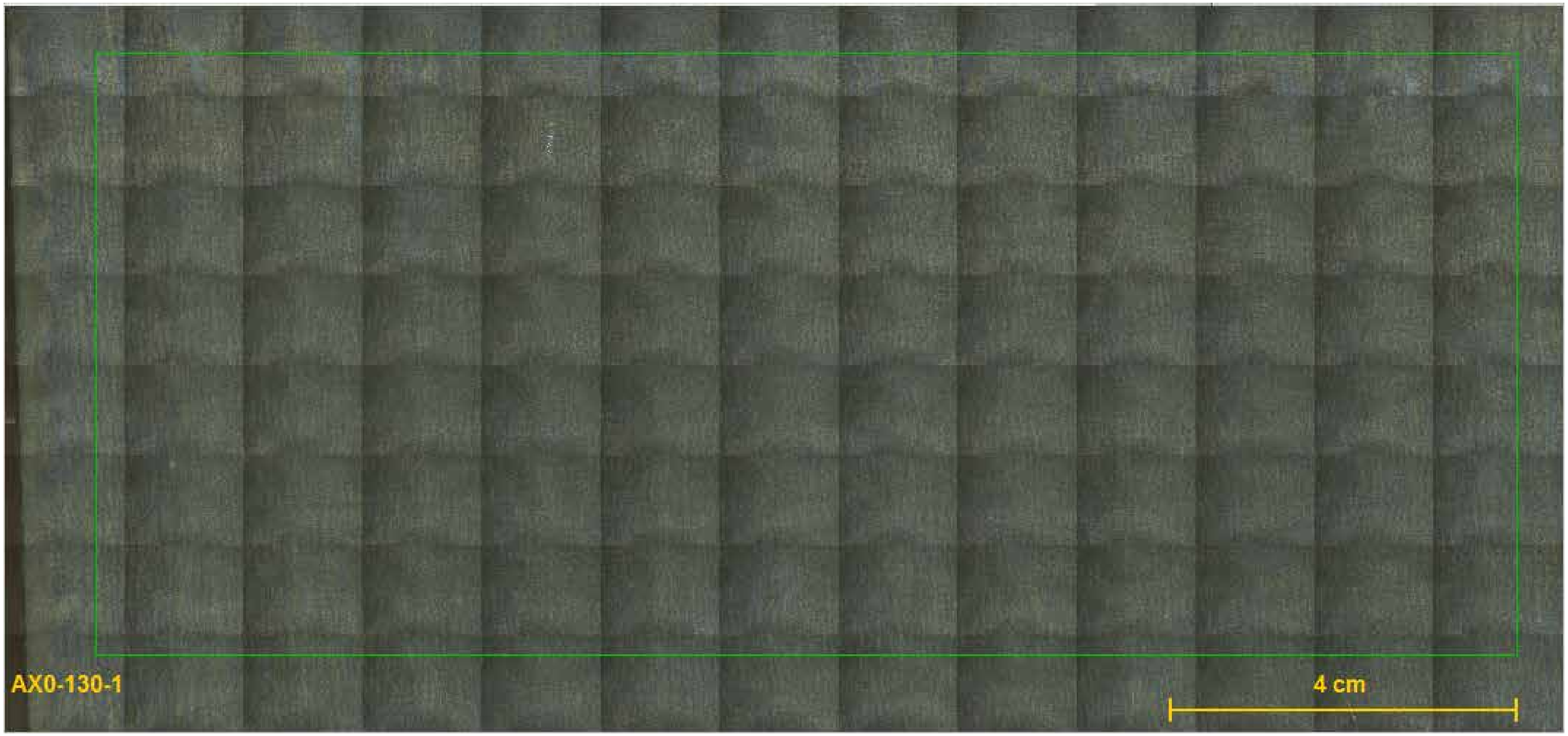
XRF Detection



Silicon **D**rift **D**etector
with XFlash® Technology

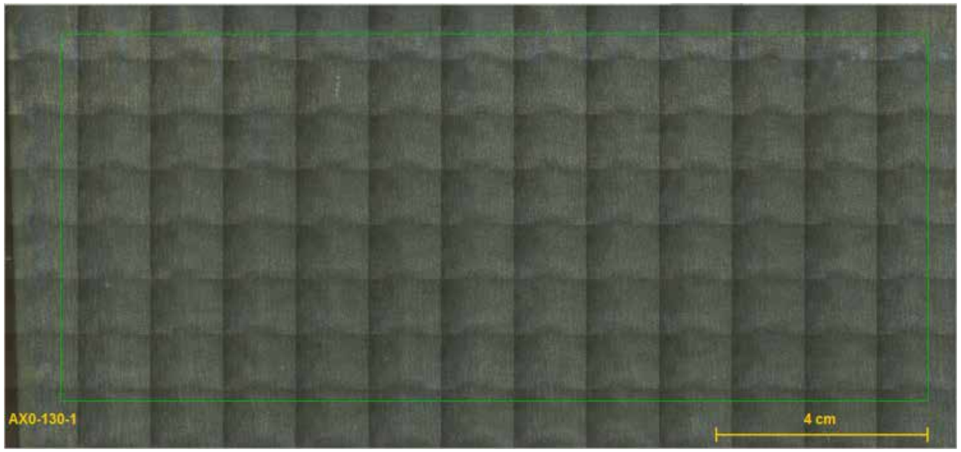
- X-rays can penetrate and excite matter
- Signal from base material and covered layers can still be detected
- X-rays are attenuated in characteristic ways on their path through matter
- Intensity ratios of observed elemental lines, allows for calculation of respective layer thicknesses

Example 1



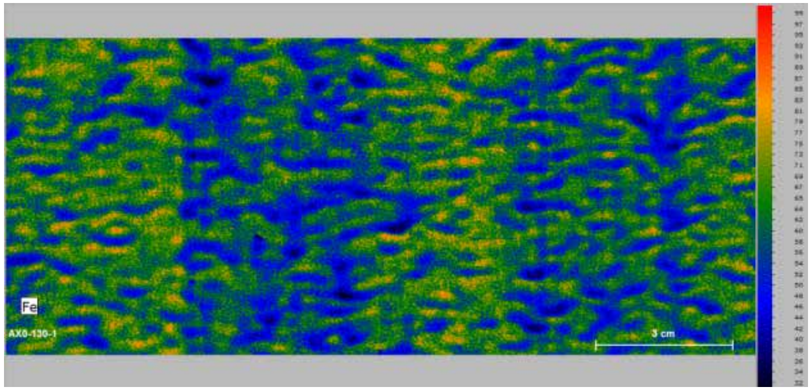
Ti /ZnAlMg coating on sheet metal

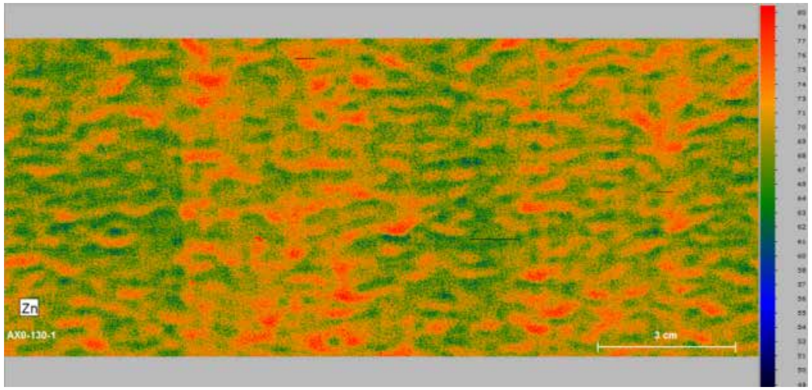
Ti /ZnAlMg: Coating on Sheet Metal
Overview and Measurement Data (Map)

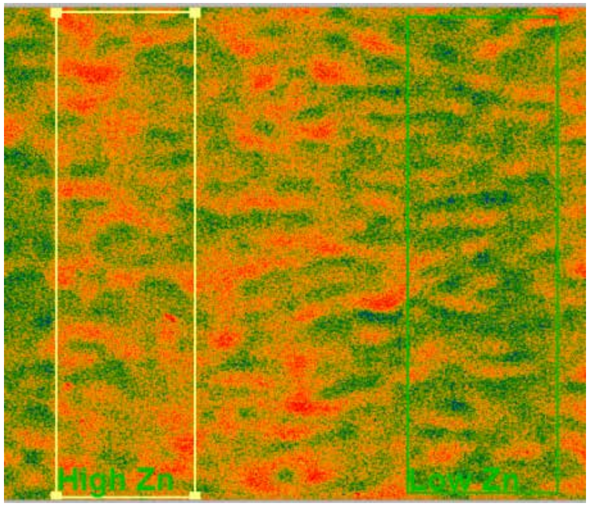


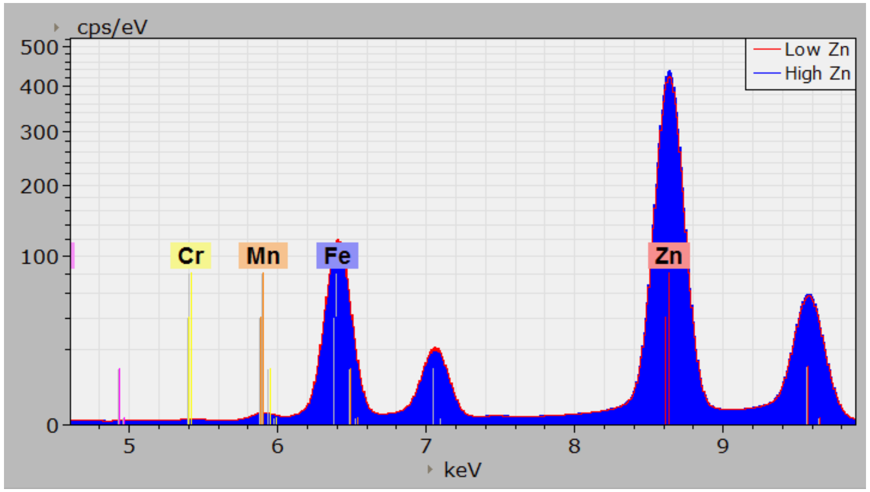
Mapping parameter		
Width:	1640	pixel
	164	mm
Height:	694	pixel
	69.4	mm
Pixel Size:	100	µm
Total number of pixel:	1138160	pixel
Acquisition parameter		
Frame count:	1	
Pixel time:	15	ms/pixel
Measure time:	17072	s
Overall time:	17705	s
Tube parameter		
High voltage:	50	kV
Anode current:	600	µA
Filter:	Al100	
Optic:	Lens	
Chamber at:	Air 1023.3	mbar

Ti /ZnAlMg: Coating on Sheet Metal
Element Distribution









Measurements show small variations of the Zn and Fe intensities. Ti peak is relatively small, <u>but looks to be distributed homogeneously</u> in the sample.

Example 2

Cu-Al coating on glass

Cu-Al Coating on Glass
Overview and Measurement Data (Map)

- Glass substrate is coated through Magnetron sputtering using a dual Cu-Al-target
- Layer thickness and gradient of Cu:Al ratio were to be measured
- Over night measurement (15 h) over the whole sample was performed
- 50 µm step size, 50 ms dwell per pixel

Cu:Al gradient layer, manufactured through Magnetron sputtering of a dual Cu/Al target by K. Harbauer, W. Kohrt, K. Ellmer, Helmholtz-Zentrum Berlin für Materialien und Energie

Cu-Al Coating on Glass
Overview and Measurement Data (Map)

Map information

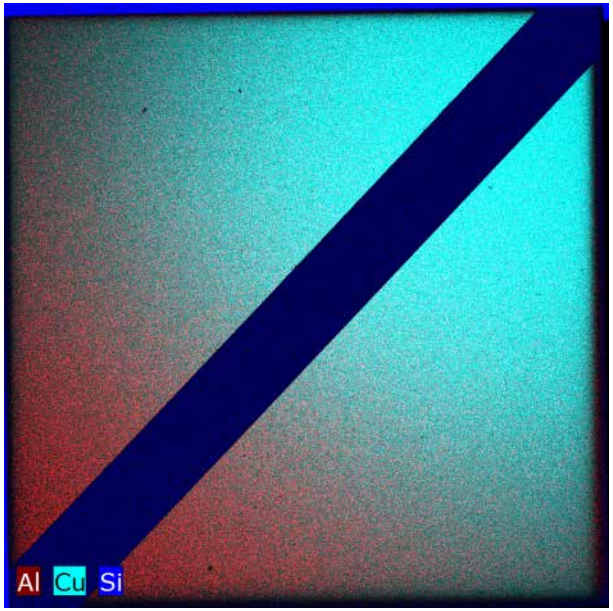
Mapping parameter		
Width:	1000	pixel
	50	mm
Height:	1000	pixel
	50	mm
Pixel Size:	50	µm
Total number of pixel:	1000000	pixel
Acquisition parameter		
Frame count:	1	
Pixel time:	50	ms/pixel
Measure time:	13:53 h	
Overall time:	14:58 h	
Tube parameter		
High voltage:	50	kV
Anode current:	200	µA
Filter:	Empty	
Optic:	Lens	
Chamber at:	Vacuum 20	mbar
Anode:	Rh	
Detector parameter		
Selected detectors:	1	

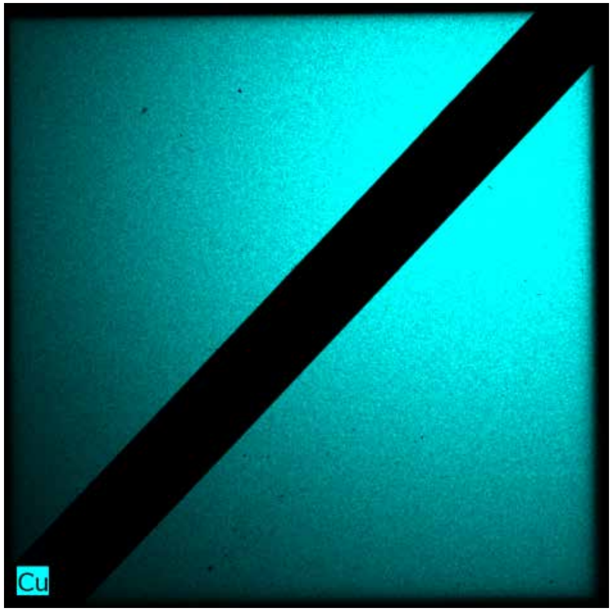
Close

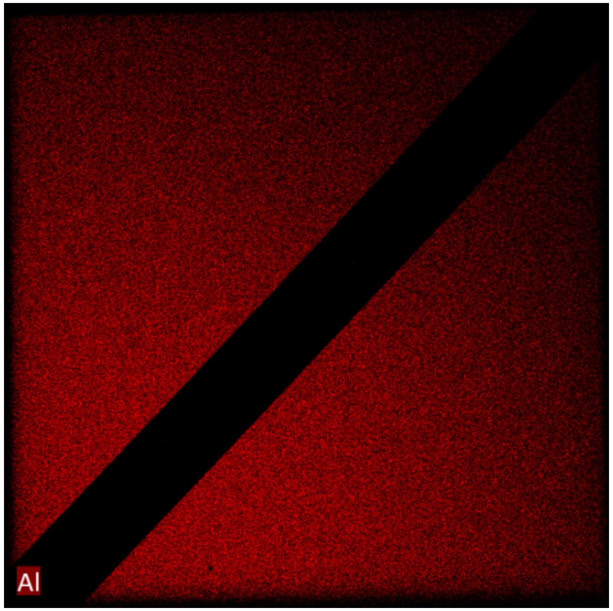
Cu:Al gradient layer, manufactured through Magnetron sputtering of a dual Cu/Al target by K. Harbauer, W. Kohrt, K. Ellmer, Helmholtz-Zentrum Berlin für Materialien und Energie

HZB Helmholtz Zentrum Berlin

Cu-Al Coating on Glass
Element Distribution



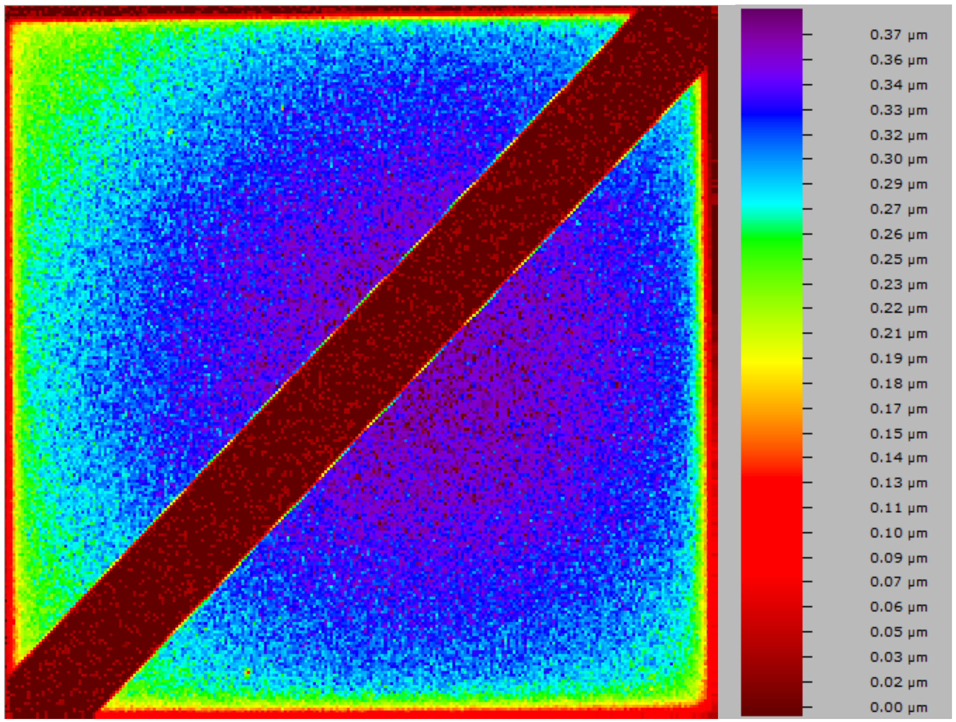




Qualitative distribution of the major elements comprised of base and layer

- Copper gradient distribution from top-right corner to bottom left
- The maximum of the Aluminum distribution seems offset to the left corner

Cu-Al Coating on Glass
Layer Thickness Analysis

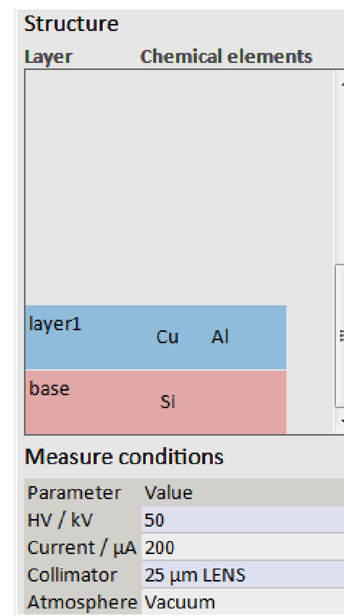


No standards available

Hence pure FP Quantification

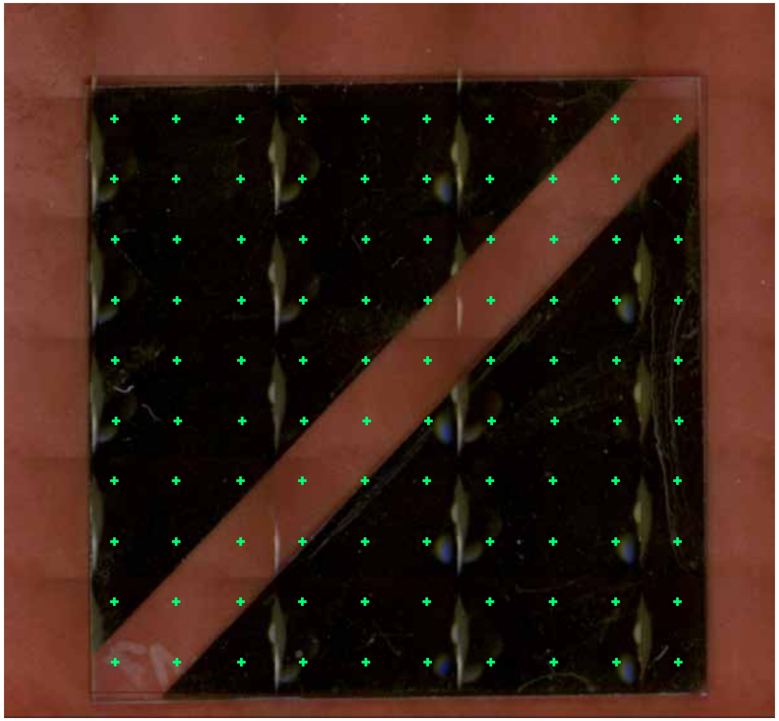
5x5-Binning:

- effectively 1.25 s per Pixel
- 250 µm spatial resolution



The layer is not homogeneous. The center of the radially symmetric thickness gradient is slightly eccentric. The thickness of the Cu:Al layer reduces from ~380 nm at the center down to ~250 nm at the edges.

Cu-Al Coating on Glass
Comparison (Point Measurements)



Alternatively, to reduce the measurement time, an array of point measurements can be performed.

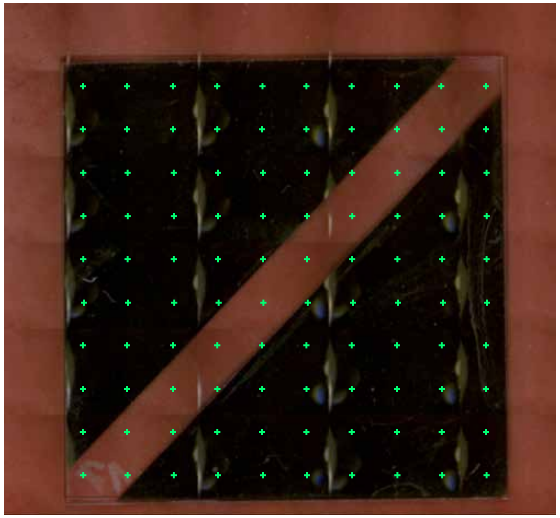
The inserting of a grid of measurement point is an automatic function in the software under Multi-Point.

Left shows a 10x10 grid.

Measurement time is 10 s per point.

Each spectrum can be quantified by the same layer analysis method. The results (layer thicknesses and composition) are shown in tabular from and can be exported to Excel in the .xls format. This allows, for example, to visualize the result of the element and layer thickness distribution.

Cu-Al Coating on Glass
Layer Thicknesses and Composition



Cu Concentration

77.2	79.0	79.3	81.3	82.4	83.2	84.5	85.6	0.0	0.0
76.6	77.4	78.6	80.4	80.1	82.3	82.6	0.0	0.0	86.3
75.4	76.9	77.7	78.6	80.0	80.8	0.0	0.0	85.2	85.1
74.4	74.4	77.5	78.1	78.3	80.0	0.0	82.2	83.6	84.0
72.9	73.0	74.7	75.8	76.4	0.0	79.6	80.4	81.6	83.0
71.3	72.6	72.7	74.5	0.0	76.4	77.0	79.5	80.5	82.2
69.7	70.4	71.2	0.0	74.7	74.5	76.9	77.3	79.3	81.8
69.3	69.7	0.0	0.0	72.9	74.3	74.2	77.1	78.2	79.8
67.5	0.0	0.0	70.4	71.9	72.8	74.6	76.3	77.2	78.4
0.0	0.0	68.2	69.4	71.4	72.7	74.3	75.8	76.9	79.0

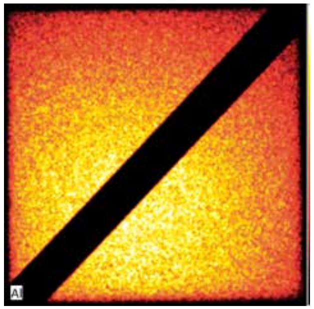
Layer thickness

0.239	0.259	0.291	0.301	0.311	0.321	0.325	0.314	0.000	0.000
0.258	0.287	0.307	0.318	0.346	0.340	0.350	0.000	0.000	0.300
0.269	0.298	0.322	0.342	0.355	0.361	0.000	0.000	0.332	0.316
0.277	0.314	0.324	0.347	0.369	0.366	0.000	0.367	0.347	0.327
0.284	0.325	0.342	0.359	0.378	0.000	0.372	0.370	0.355	0.331
0.283	0.312	0.346	0.360	0.000	0.379	0.386	0.367	0.357	0.324
0.281	0.314	0.338	0.000	0.301	0.382	0.369	0.370	0.355	0.309
0.264	0.294	0.000	0.000	0.350	0.356	0.370	0.347	0.337	0.305
0.249	0.000	0.030	0.319	0.326	0.342	0.336	0.327	0.319	0.291
0.000	0.000	0.274	0.289	0.295	0.303	0.302	0.295	0.286	0.256

Al Concentration

22.8	21.0	20.7	18.7	17.6	16.8	15.5	14.4	0.0	0.0
23.4	22.6	21.4	19.6	19.9	17.7	17.4	0.0	0.0	13.7
24.6	23.1	22.3	21.4	20.0	19.2	0.0	0.0	14.8	14.9
25.6	25.6	22.5	21.9	21.7	20.0	0.0	17.8	16.4	16.0
27.1	27.0	25.3	24.2	23.6	0.0	20.4	19.6	18.4	17.0
28.7	27.4	27.3	25.5	0.0	23.6	23.0	20.5	19.5	17.8
30.3	29.6	28.8	0.0	25.3	25.5	23.1	22.7	20.7	18.2
30.7	30.3	0.0	0.0	27.1	25.7	25.8	22.9	21.8	20.2
32.5	0.0	0.0	29.6	28.1	27.2	25.4	23.7	22.8	21.6
0.0	0.0	31.8	30.6	28.6	27.3	25.7	24.2	23.1	21.0

Cu-Al Coating on Glass
Results Comparison

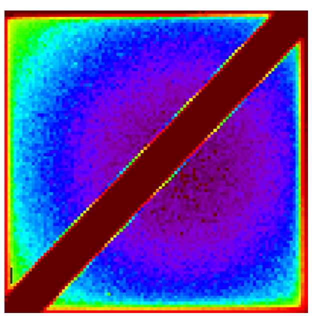


22.8	21.0	20.7	18.7	17.6	16.8	15.5	14.4	0.0	0.0
23.4	22.6	21.4	19.6	19.9	17.7	17.4	0.0	0.0	13.7
24.6	23.1	22.3	21.4	20.0	19.2	0.0	0.0	14.8	14.9
25.6	25.6	22.5	21.9	21.7	20.0	0.0	17.8	16.4	16.0
27.1	27.0	25.3	24.2	23.6	0.0	20.4	19.6	18.4	17.0
28.7	27.4	27.3	25.5	0.0	23.6	23.0	20.5	19.5	17.8
30.3	29.6	28.8	0.0	25.3	25.5	23.1	22.7	20.7	18.2
30.7	30.3	0.0	0.0	27.1	25.7	25.8	22.9	21.8	20.2
32.5	0.0	0.0	29.6	28.1	27.2	25.4	23.7	22.8	21.6
0.0	0.0	31.8	30.6	28.6	27.3	25.7	24.2	23.1	21.0

Element distribution of Al (qualitative, left) shows maximum is not in the corner, the layer composition (quantitative, right) shows that the highest Al:Cu ratio is in the corner.
The layer thicknesses is also thinner there.

77.2	79.0	79.3	81.3	82.4	83.2	84.5	85.6	0.0	0.0
76.6	77.4	78.6	80.4	80.1	82.3	82.6	0.0	0.0	86.3
75.4	76.9	77.7	78.6	80.0	80.8	0.0	0.0	85.2	85.1
74.4	74.4	77.5	78.1	78.3	80.0	0.0	82.2	83.6	84.0
72.9	73.0	74.7	75.8	76.4	0.0	79.6	80.4	81.6	83.0
71.3	72.6	72.7	74.5	0.0	76.4	77.0	79.5	80.5	82.2
69.7	70.4	71.2	0.0	74.7	74.5	76.9	77.3	79.3	81.8
69.3	69.7	0.0	0.0	72.9	74.3	74.2	77.1	78.2	79.8
67.5	0.0	0.0	70.4	71.9	72.8	74.6	76.3	77.2	78.4
0.0	0.0	68.2	69.4	71.4	72.7	74.3	75.8	76.9	79.0

Element distribution of Cu (qualitative, left) The layer composition (quantitative, right) shows the highest Cu:Al ratio in the corner.



0.239	0.259	0.291	0.301	0.311	0.321	0.325	0.314	0.000	0.000
0.258	0.287	0.307	0.318	0.346	0.340	0.350	0.000	0.000	0.300
0.269	0.298	0.322	0.342	0.355	0.361	0.000	0.000	0.332	0.316
0.277	0.314	0.324	0.347	0.369	0.366	0.000	0.367	0.347	0.327
0.284	0.325	0.342	0.359	0.378	0.000	0.372	0.370	0.355	0.331
0.283	0.312	0.346	0.360	0.000	0.379	0.386	0.367	0.357	0.324
0.281	0.314	0.338	0.000	0.301	0.382	0.369	0.370	0.355	0.309
0.264	0.294	0.000	0.000	0.350	0.356	0.370	0.347	0.337	0.305
0.249	0.000	0.030	0.319	0.326	0.342	0.336	0.327	0.319	0.291
0.000	0.000	0.274	0.289	0.295	0.303	0.302	0.295	0.286	0.256

Very similar results with respect to position and min/max-values of the **layer thickness distribution.**

Spatial resolution vs. measurement time (here 250 µm with 15 h vs. 5 mm with < 20 min)

Summary

- Micro-XRF allows measurement of thin (micrometer→nanometer) layer thicknesses with high (< 20 µm) lateral spatial resolution
- Through the HyperMap database, element distributions can be saved with spectra and processed afterwards
- Intensity variations are not necessarily proportional to the layer thickness changes
- To determine the thickness of a layer, it must be quantified or calculated by applying a layer thickness method
- This can then be visualized as distribution or layer thickness image
- Alternatively, using single point measurements, the layer can be analyzed and quantified, followed by additional processing in Excel to provide a visual representation of the element and layer thickness distribution

Q&A

Any Questions?

Please type in the questions you might have

in the Q&A box and press Submit.

More information

For more information, please contact us:

falk.reinhardt@bruker.com

samuel.scheller@bruker.com

info.bna@bruker.com

https://www.bruker.com/m4tornado

Innovation with Integrity