

Layer Thickness Analysis of Thin Metal Coatings with micro-XRF on SEM



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Presenters



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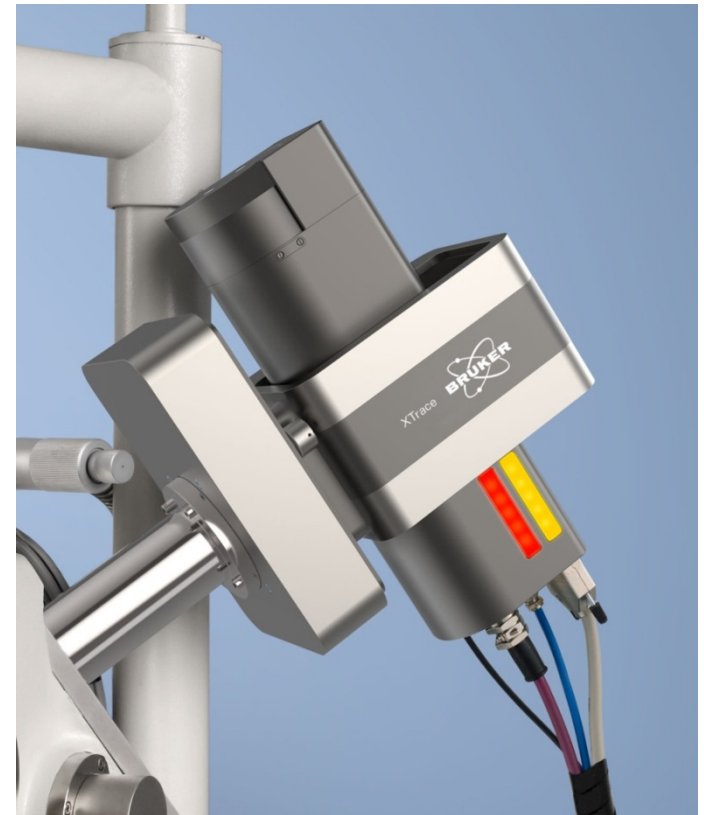
- XTrace setup and key features
- Basics: Layer thickness measurements with XRF
- Method editor
- Application examples + Live measurements
- Summary

Layer Thickness Analysis with micro-XRF/SEM

Key Features

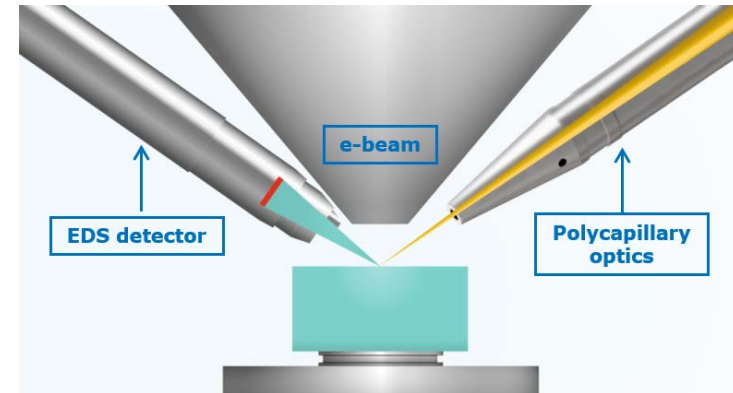


- Micro-XRF source attachment to an SEM works in conjunction with the (existing) Bruker EDS detector
- Element range from **Na** to **U**
- Focussing polycapillary X-ray optics enables a spot size of (standard) **35 μm** (smaller optics with a spot size of 15 μm available as well)
- Integrated user interface ESPRIT 2.x for Quantax EDS/micro-XRF ...
- **Method editor** for fast layer method setup is linked to ESPRIT
- Allows the quantification of layer systems measured either in point mode or extracted from line scans or X-ray maps



Layer Thickness Analysis with micro-XRF/SEM

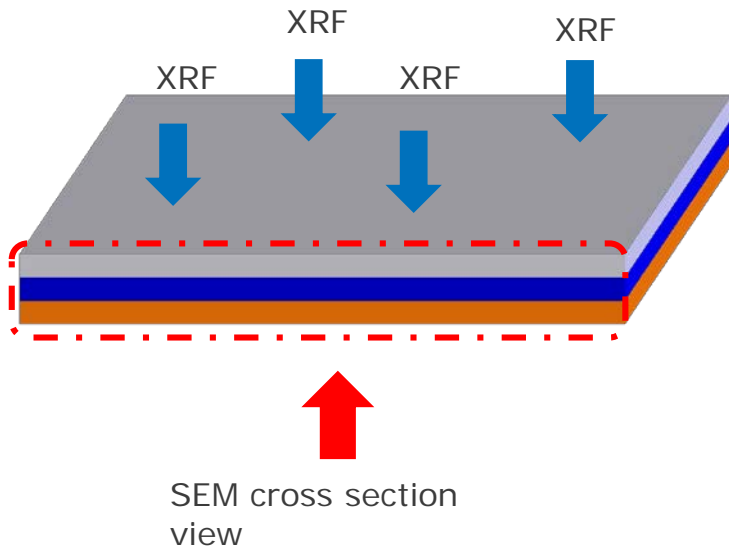
Example for instrument setup



Schematic of setup in a SEM for XRF with EDS detector

Layer Thickness Analysis

Differences in layer thickness analysis between SEM - XRF



SEM:

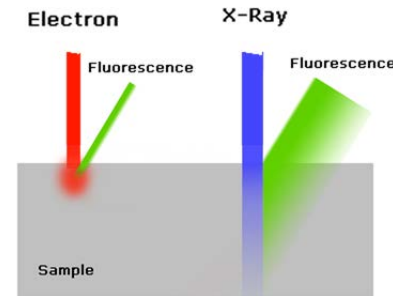
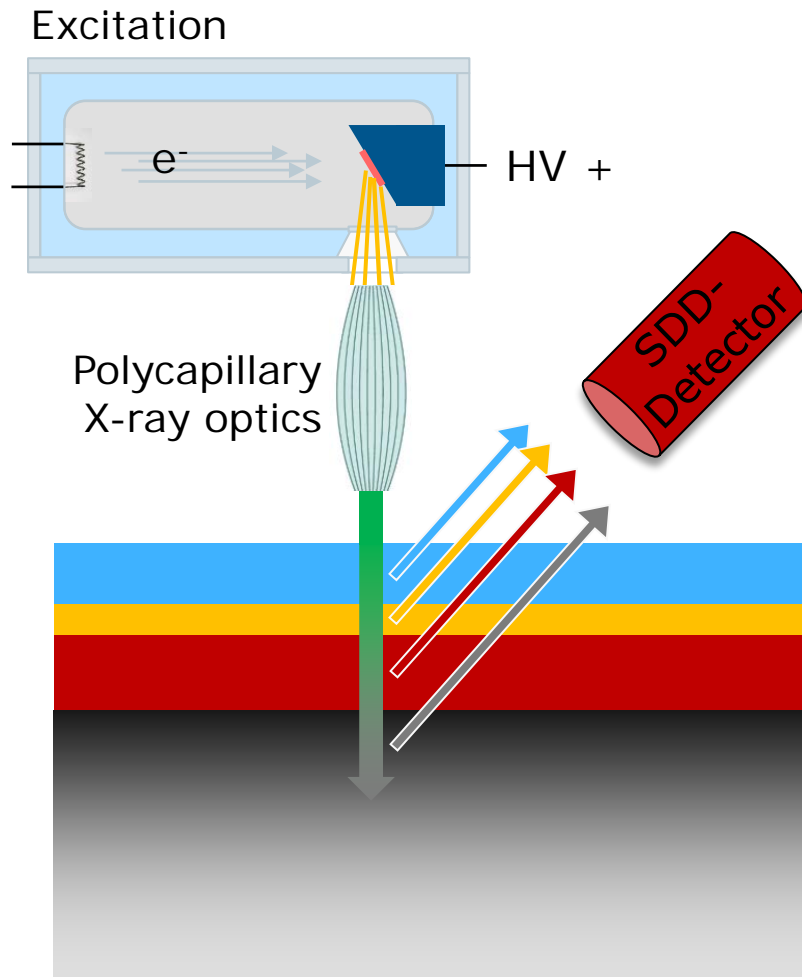
1. Sample require cross sectioned and sample preparation can be time consuming
2. Sample must be cut for cross sectioned observation and hence, it will be destroyed
3. Sufficient SEM resolution required to makes thin layer visible

XRF:

1. Fast and non- destructive method for measuring film thickness without any sample preparation
2. Additionally, the composition of the layer can be determined at the same time
3. Large numbers of samples or areas can be measured quickly

Layer Thickness Analysis with micro-XRF/SEM

Introduction



- Large information depth of X-ray excitation allows coating analysis
- Signal from base material and covered layers can be detected
- X-rays are attenuated in characteristic ways on their path through matter
- Intensity ratios of observed elemental lines were used for calculation of the respective layer thicknesses
- Generally, layers up to thickness of 40 μm can be analyzed (3- 4 layers)
- Several quantification models for layer systems available which works easily even standard-less

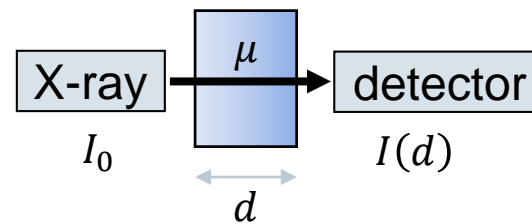
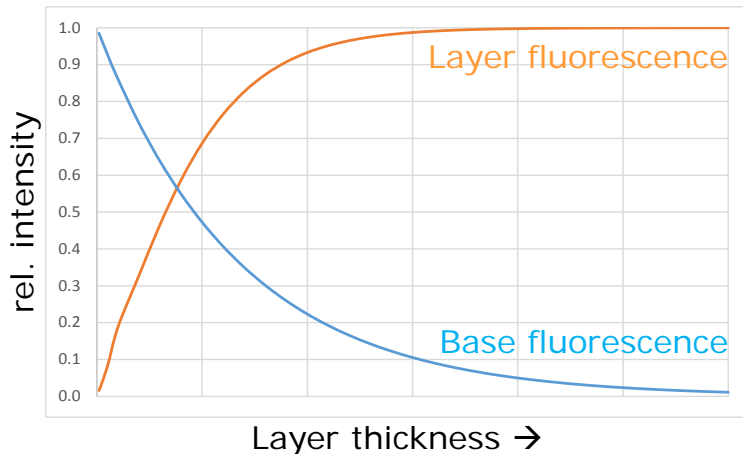
Physical Principles of Layer thickness analysis

Generation / Attenuation of XRF



For a single layer on top of a base material:

- Emission signal originating from coating increases with coating thickness up to the respective bulk intensity
- Emission from base material is attenuated stronger for thicker coatings, until it fades into the background
- Coating thickness can be calculated from absorption of base material, emission from coating, or a combination of both



Lambert-Beer-Law (absorption):

$$I(d) = I_0 \cdot e^{-\mu \cdot d}$$



X-ray transmission image of the hand of W.C. Roentgen's wife. 22nd December 1895

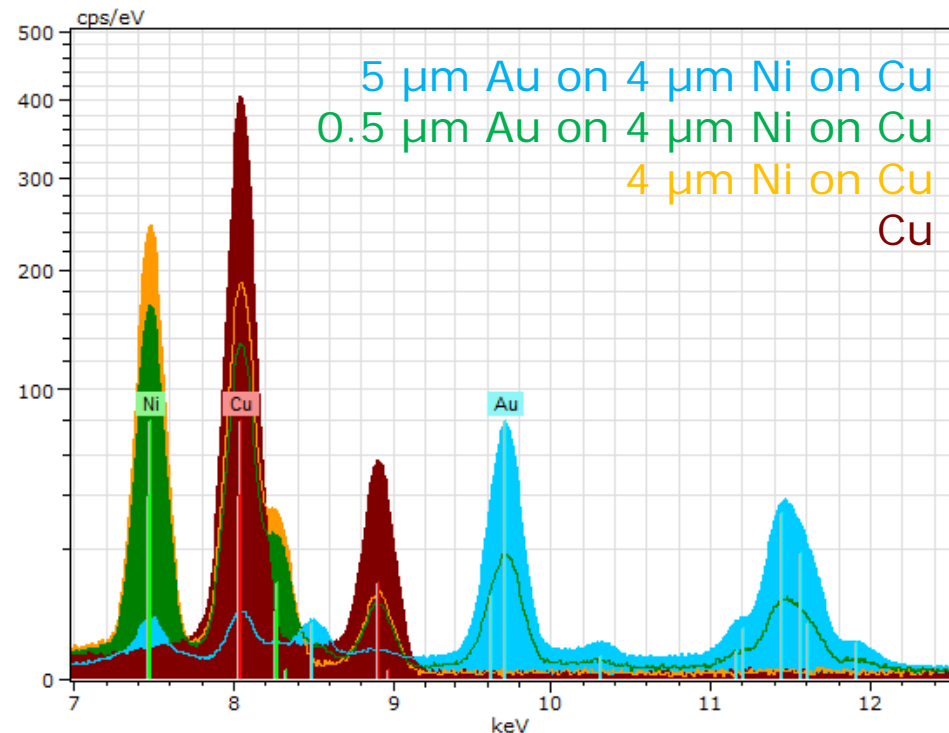
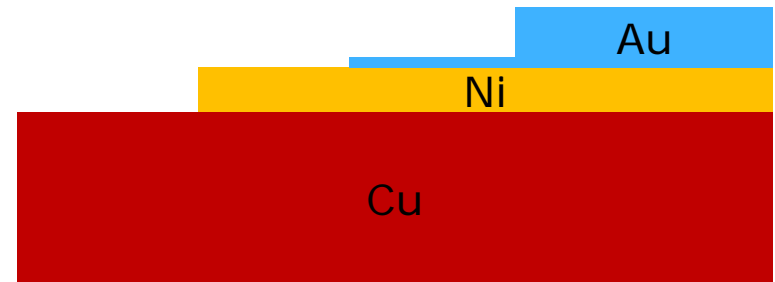
Physical Principles of Layer thickness analysis

Comparison of Layer Stacks



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 40 μm , depending on elements and matrix density

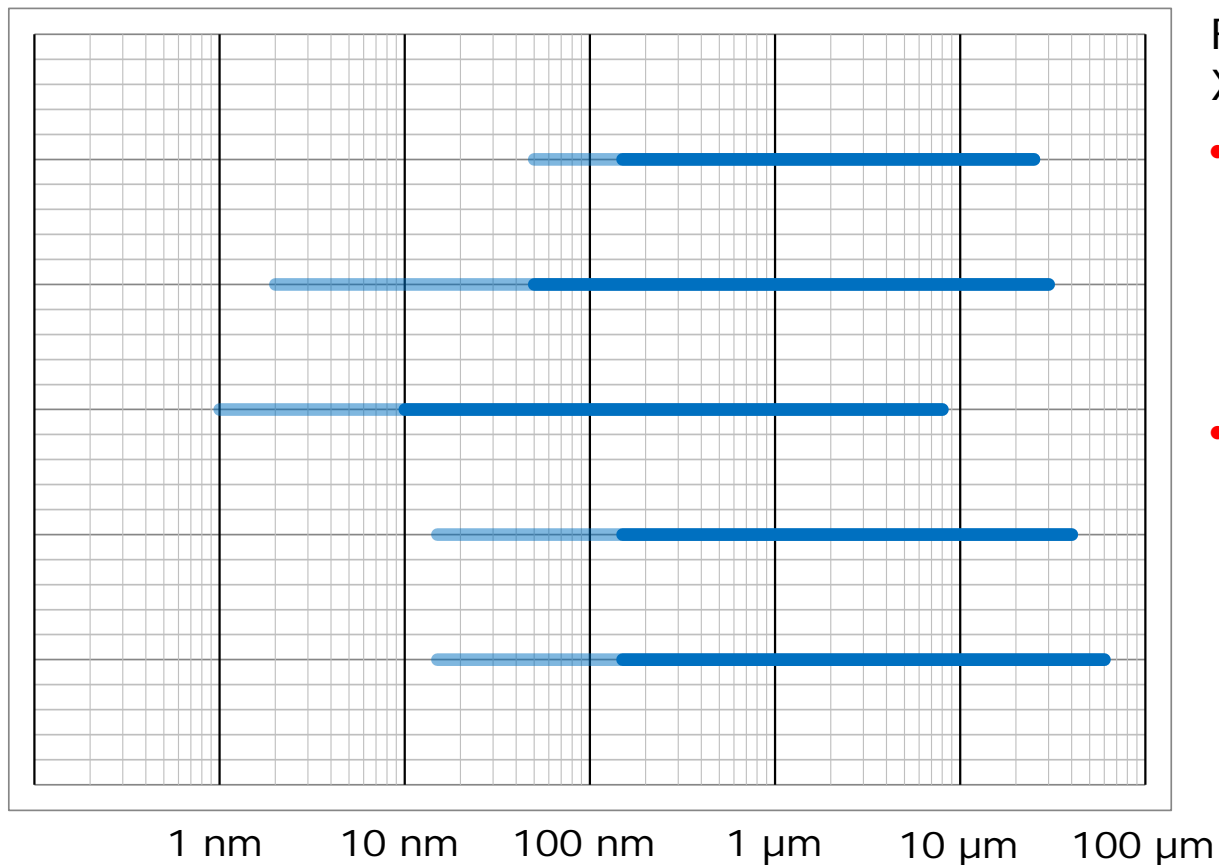


Layer Thickness Analysis with micro-XRF/SEM

Thickness Range for Element Emission



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

Layer Thickness Analysis with micro-XRF/SEM

Requirements



Requirements for layer analysis on the SEM:

1. Knowledge about the layer system is necessary (layer sequence)
2. Layer calculation is based on pure element spectra intensity → stable EDS + XRF conditions
3. For every layer a separate signal is required → the same element shouldn't be in different layers (or at least different X-ray series of this element should be available for analysis)
4. Tilt = 0, changing the Tilt angle will influence the TOA which isn't considered in XMethod

Coating Thickness Analysis on SEM with X-ray fluorescence XMethod



• Method creation

- For many physical different structured samples such as bulks, coatings, light matrices

• Method optimization

- By editing several details on the lines to investigate or the structure of the sample in the methods
- For bulk methods calibration ranges can be set

• Method calibration

- Methods can be applied based on fundamental parameter approach without any standard
- Standards can be utilized to enhance the precision or in order to build fully empirical calibrations

The image displays three screenshots of the Bruker XMethod software interface, illustrating the workflow from method creation to calibration.

Top Screenshot: Method Editor (ZnNi_Fe)
This window shows the configuration for a new method. Key parameters include:

- Method data:** Name (layer3), Description (ZnNi_Fe), Type (layer), Standard decomposition (Calc. mode), Standard spectrum deconvol (Empirical).
- Measurement parameters:** Meas time (s) set to 20.
- Layer parameters:** Name (layer3), Start thickness (0.00), Unit (µm (2)), Fixed thickness (0.00), Calc. mode (Empirical), Density (7.13), Low thickness (0.00), High thickness (0.00), Target value (0.00).
- Structure:** Layer (layer3), Chemical elements (Zn, Ni).
- Element overview:** Table showing elements Zn and Ni with their main lines and start concentrations.

Middle Screenshot: ELEMENT PROPERTIES EDITOR (Zn)
This window allows for detailed configuration of the Zn element line:

- Parameters:** Element type (Normal), Main line (KA), Sub line (KB).
- Start conc.:** 90.00, Unit (% (2)).
- Calculation mode:** Calculate.
- Use for thickness calculation:** Checked, Threshold (0.0005).
- Density:** Default (7.13) or User (1.00).
- Use tolerances?:** Checked, Low concentration (87), High concentration (93), Target value (90.00).
- Line data table:**

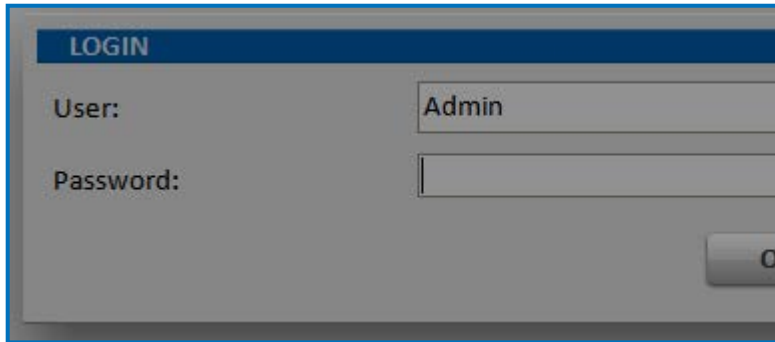
Line	Energy (keV)	Use	Used lines
KA	8.637	<input checked="" type="checkbox"/>	KA1,KA2
KB	9.570	<input checked="" type="checkbox"/>	KB1,KB2,KB3,KB5

Bottom Screenshot: Calibration Editor (ZnNi_Fe)
This window shows the calibration setup and results:

- Method data:** Same as the top screenshot.
- Calibration samples table:**

Layer	Parameter	conc	layer	conc	conc	conc	conc	
100	80041P010000_Fe	---	4.00	4.00	88.00	87.50	83.00	12.00
112	80240P010000_Fe	---	4.00	4.70	88.00	81.10	88.00	10.00
110	80240P010300_Fe	---	11.30	11.10	84.00	81.50	81.00	14.00
109	80240P010500_Fe	---	15.00	14.50	88.00	84.00	83.00	13.00
111	80240P010800_Fe	---	18.00	19.70	83.00	79.00	88.00	20.00
- Measurement parameters:** Meas time (s) set to 20.
- Calibration graph:** A plot of layer thickness (µm) vs. concentration (%) showing a linear relationship with data points and a fitted line.

XMethod Method Setup



- Easy to use method editor with all major settings on one screen
- Multiple users to allow only accredited people to change methods

METHOD EDITOR
ZnNi_Fe

Method data

Description: ZnNi_Fe
Type: layer
Spectrum deconvolution: Standard spectrum deconvol
Comment:

Measurement parameters

HV / kV: 50
Collimator / mm: 0.70
Atmosphere: Air
Current / μ A: 800
Measure time / s: 20
Smooth spectra:

Layer parameters

Name: layer1
Compound:
Start thickness: 0.00
Unit: μ m (2)
Fixed thickness:
Calc. mode: Emission
Normalize sample/standard:
Target value (%): 100.00
Density:
 Default: 7.43
 User: 0.00
 Use tolerances?
Low thickness: 4.50
High thickness: 4.70
Target value: 4.60

Structure

Layer: Chemical elements

layer1	Zn	Ni
base	Fe	

Element overview

Element	Z	Main line	Start conc.
Zn	30	KA	90.00
Ni	28	KA	10.00

XMethod Method Setup



LOGIN [X]

User:

Password:

Structure Normation Calibration Spectrum

Structure

Layer Chemical elements

layer1	Zn	Ni
base	Fe	

Element overview

Element	Z	Main line	Start conc.
Zn	30	KA	90.00
Ni	28	KA	10.00

Measurement parameters

Use tolerances?

layer1

0.00

μm (2)

Emission

Normalize sample/standard

Target value (%) 100.00

Density

Default 7.43

User 0.00

Use tolerances?

Low thickness 4.50

High thickness 4.70

Target value 4.60

Structure Normation Calibration Spectrum

Structure

Layer Chemical elements

layer1	Zn	Ni
base	Fe	

Layer parameters

Name

Compound

Start thickness

Unit

Fixed thickness

Calc. mode

Normalize sample/standard

Target value (%)

Density

Default

User

Use tolerances?

Low thickness

High thickness

Target value

- Starting values (to accelerate computation) as well as tolerances for results can be entered

XMethod Method Setup



LOGIN [X]

User:

Password:

Structure Normation Calibration Spectrum

Structure

Layer Chemical elements

layer1	Zn	Ni
base	Fe	

Structure Normation Calibration Spectrum

Structure

Layer Chemical elements

layer1	Zn	Ni
base	Fe	

Measurement parameters [X] Use tolerances?

Layer parameters

Name

Compound

Start thickness

Unit

Fixed thickness

Calc. mode

Normalize sample/standard

Target value (%)

Density

Default

User

Use tolerances?

Low thickness

High thickness

Target value

ELEMENT PROPERTIES EDITOR [X]

³⁰Zn

Line data

Main line

Sub line

Parameters

Element type

Show element in results

start conc.

Unit

Calculation mode

Use for thickness calculation

Threshold

density

Default

User

Use tolerances?

Low concentration

High concentration

Target value

Line	Energy(keV)	Use	Used lines
KA	8.637	<input checked="" type="checkbox"/>	KA1,KA2
KB	9.570	<input checked="" type="checkbox"/>	KB1,KB2,KB3,KB5

XMethod Method Setup



Element	Z	Main line	Start conc.
Au	79	LA	100,00

Description	M Type	Sub type	Supplier	Use	base	Ni layer	Au layer
Au500_Ni4380_Cu	layer		not specified	<input checked="" type="checkbox"/>	--	4,38	0,500
Au52_Ni960_Cu	layer		not specified	<input checked="" type="checkbox"/>	--	1,96	0,052

layer	base	Ni layer	Au layer			
Parameter	W. Std	calib.	Std	calib.	Std	calib.
7 Au500_Ni4380_Cu	1	--	4,38	4,38	0,500	0,500
8 Au52_Ni960_Cu	1	--	1,96	1,96	0,052	0,052

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



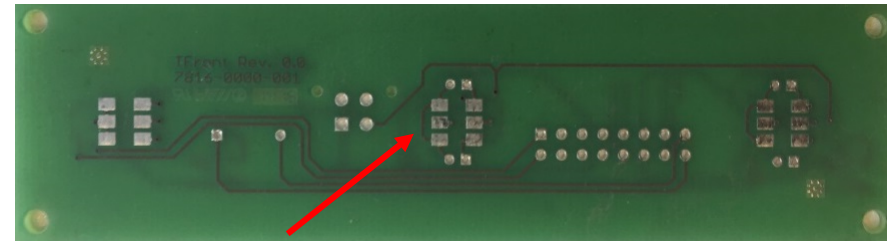
Application examples

Layer Thickness Analysis with micro-XRF/SEM



Example I: SnPb/Cu Solder bump

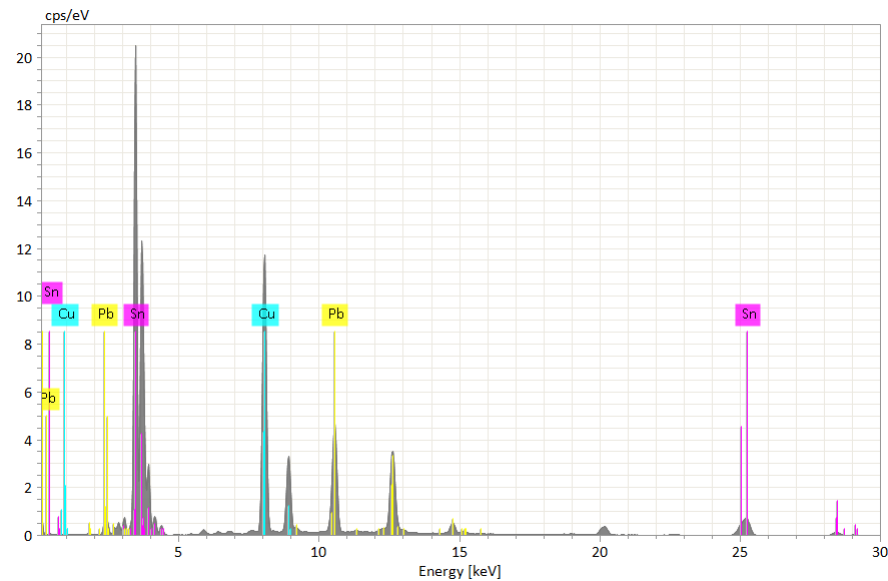
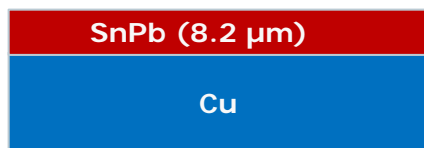
- Pb is a restricted material in consumer electronic devices
- But still used in some areas of medical (implants) or defense sector when reliability and durability are absolutely essential.



Example for solder bumps



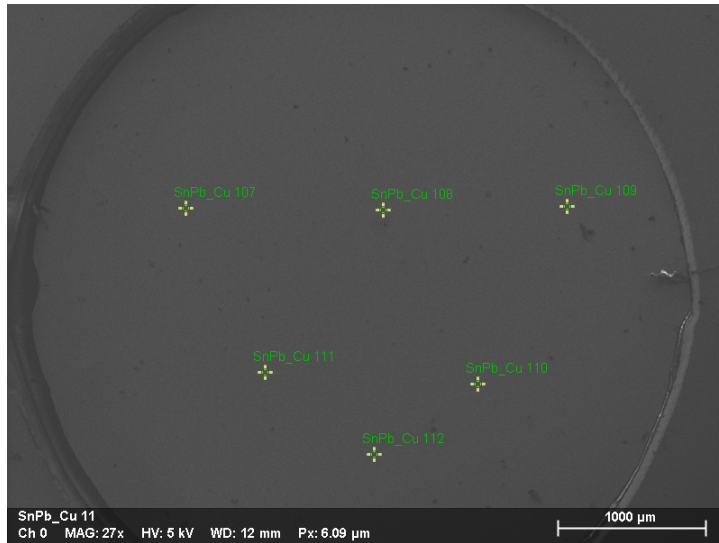
Certified SnPb standard



XRF spectra of the certified standard, 60 sec measurement time

Layer Thickness Analysis with micro-XRF/SEM

Example I: SnPb/Cu Solder bump



Normalized mass concentration [%]

Spectrum	Substrate	Layer 1		
	Cu [%]	thickn. [μm]	Sn [%]	Pb [%]
SnPb_Cu 107	100.00	8.78	91.48	8.52
SnPb_Cu 108	100.00	8.88	91.83	8.17
SnPb_Cu 109	100.00	8.91	91.38	8.62
SnPb_Cu 110	100.00	8.82	91.98	8.02
SnPb_Cu 111	100.00	8.71	91.88	8.12
SnPb_Cu 112	100.00	8.75	91.87	8.13
Mean value	100.00	8.81	91.74	8.26
Std dev.	0.00	0.07	0.24	0.24
Std dev. rel. [%]	0.00	0.85	0.26	2.94
Conf. interval	0.00	0.03	0.10	0.10



Measurement points on SnPb standard , 60 sec acquisition time (real time)

- Standardless quantification for the SnPb coating gives sufficient results but can be improved by using standards!

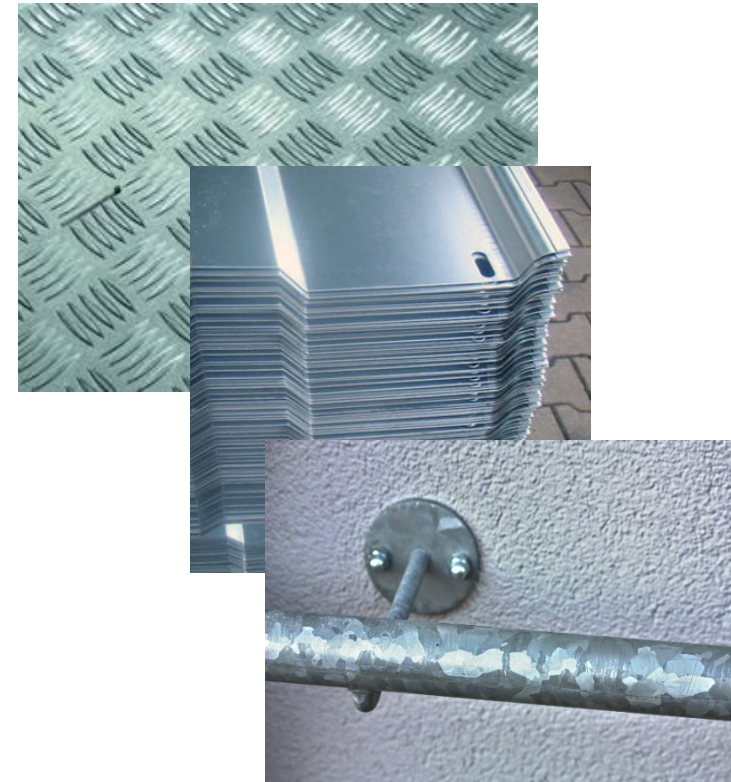


Layer Thickness Analysis with micro-XRF/SEM



Example II: Galvanized Metal Sheet

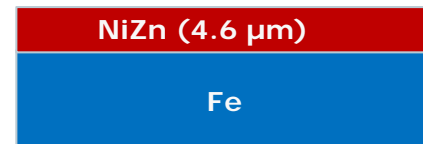
- Consists of a layer structure of NiZn/Fe (Fe = substrate)
- Zn is used for corrosion and fire protection
- Ni protects the material against mechanical wear
- Our (unknown) sample:



Examples for NiZn alloys

Fe Base	Zn Sat.	Ni Sat.	NiZn/Fe	NiZn/Fe	NiZn/Fe	NiZn/Fe	NiZn/Fe
			4,6 µm	15,0 µm	13,3 µm	20,6 µm	4,8 µm
			11,1 %	11,8 %	15,1 %	16,8 %	18,5 %
U6I10	U6I11	U6I12	U6I13	U6I14	U6I15	U6I16	U6I17

Certified NiZn/Fe standard



Layer Thickness Analysis with micro-XRF/SEM

Example II: Galvanized Metal Sheet



Step 1: Build a layer method in XMethod

1. Name the method

2. Define measurement conditions

3. Define layer structure

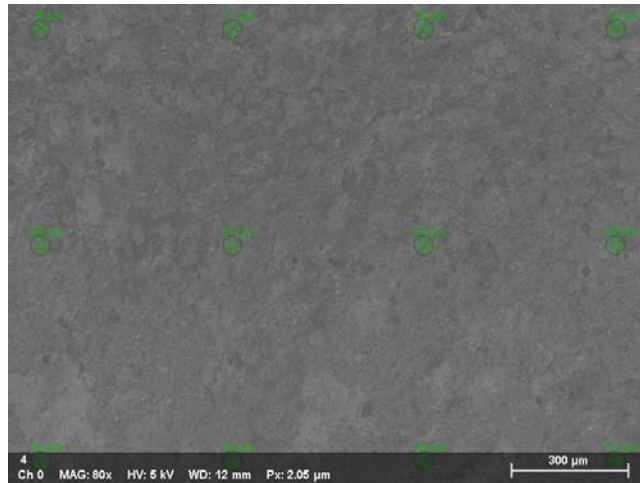
4. Save the Method (XADF) for further use

Coating Thickness Analysis

Example II: Galvanized Metal Sheet



Step 2: Acquire spectra in ESPRIT (Object mode)



Normalized mass concentration [%]

Spectrum	Substrate	Layer 1	Zn [%]	Ni [%]
	Fe [%]	Thickn. [µm]		
76.spx	100.00	3.37	85.91	14.09
77.spx	100.00	3.34	85.83	14.17
78.spx	100.00	3.23	85.79	14.21
79.spx	100.00	3.29	85.83	14.17
80.spx	100.00	3.20	85.60	14.40
81.spx	100.00	3.34	85.81	14.19
82.spx	100.00	3.36	85.92	14.08
83.spx	100.00	3.36	85.82	14.18
84.spx	100.00	3.50	85.99	14.01
85.spx	100.00	3.35	85.97	14.03
86.spx	100.00	3.31	85.88	14.12
87.spx	100.00	3.24	85.74	14.26
Mean value	100.00	3.32	85.84	14.16
Std dev.	0.00	0.08	0.11	0.11
Std dev. rel. [%]	0.00	2.37	0.12	0.76
Conf. interval	0.00	0.02	0.03	0.03

	Layer thickness	Ni %	Zn %
given	4.6 µm	11,10%	88,90%
Layer FP standard-free	3.32 µm	14,16%	85,84%

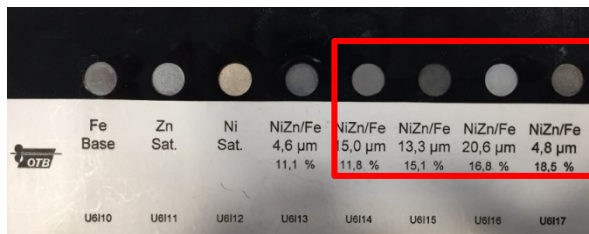
For this sample the standard-free FP is not accurate enough due to the secondary fluorescence effect (Zn excites Ni which overweight's the Ni concentration) → thickness calculation is based on the Zn intensity since Zn is the main element → **Method must be calibrated with standards!!**

Layer Thickness Analysis with micro-XRF/SEM



Example II: Galvanized Metal Sheet

Step 4: Measuring appropriated standards (with same layer structure)



Method data

Description: ZnNi_Fe_test_except_4_6

Type: layer

Spectrum deconvolution: Bayes deconvolution

Comment:

Measurement parameters

HV / kV: 50

Collimator / mm: 15 µm LENS

Atmosphere: Vacuum

Current / µA: 900

Measure time / s: 20

Calibration coefficients

0
1.25496268
2.723015074E-2

Calibration samples

No.	Sample	base W.	base Std	FP	layer1 d(µm)	FP	Zn[%] Std	FP	Ni[%] Std	FP
9	13300Zn84_9Ni15_1-Fe	1	---	---	13.30	8.80	84.90	81.85	15.10	18.15
5	15000Zn88_2Ni11_8-Fe	1	---	---	15.00	9.88	88.20	85.94	11.80	14.06
6	20600Zn83_2Ni16_8-Fe	1	---	---	20.60	12.89	83.20	79.34	16.80	20.66
4	4800Zn81_5Ni18_5-Fe	0	---	---	4.80	3.43	88.90	85.92	11.10	14.08
7	4800Zn81_5Ni18_5-Fe	1	---	---	4.80	3.57	81.50	77.11	18.50	22.89

Deviation (σ): 5.99, 4.02, 4.02

Start FP calculation

Normation type: Sample to 100.00 %

Norm target value: 100

Calibration curve offset: Use offset

Calibration parameter:

Order of polynom:

After clicking "start FP calculation" button, the chart will be updated with the FP results. The calculated FP values will be plotted against the given values of the selected standards (thickness or concentration).

Given values differs to the measured values → Calibration required!

Layer Thickness Analysis with micro-XRF/SEM



Example II: Galvanized Metal Sheet

Step 4: Calibrate the method

The screenshot displays the 'METHOD EDITOR' window for 'ZnNi_Fe_test_except_4_6'. The 'Calibration' tab is active, showing a table of calibration samples and a graph of layer thickness vs. Zn and Ni content.

No.	Layer Parameter Sample	base W.	base Std	calib.	layer1 d(μm)		Zn(%)		Ni(%)	
					Std	calib.	Std	calib.	Std	calib.
9	13300Zn84_9Ni15_1-Fe	1	---	---	13.30	13.17	84.90	85.36	15.10	14.64
5	15000Zn88_2Ni11_8-Fe	1	---	---	15.00	15.07	88.20	88.27	11.80	11.73
6	20600Zn83_2Ni16_8-Fe	1	---	---	20.60	20.72	83.20	83.28	16.80	16.72
4	4600Zn88_9Ni11_1-Fe	0	---	---	4.60	4.64	88.90	89.37	11.10	10.63
7	4800Zn81_5Ni18_5-Fe	1	---	---	4.80	4.85	81.50	82.90	18.50	17.10

Below the table, the 'Deviation (σ)' is shown as 0.11, 0.85, and 0.85. A graph titled 'layer1 (μm)' shows a blue line representing the calibrated results and a green dashed line representing the uncalibrated data. The x-axis ranges from 5.54 to 22.161, and the y-axis ranges from 5.54 to 22.161. A red arrow points to the 'Start calibration' button.

After clicking the Calibration button, the chart will be updated with the calibrated results. The results should be much closer to the blue line now. Method can be saved afterwards as standard based XADF method.

Layer Thickness Analysis with micro-XRF/SEM

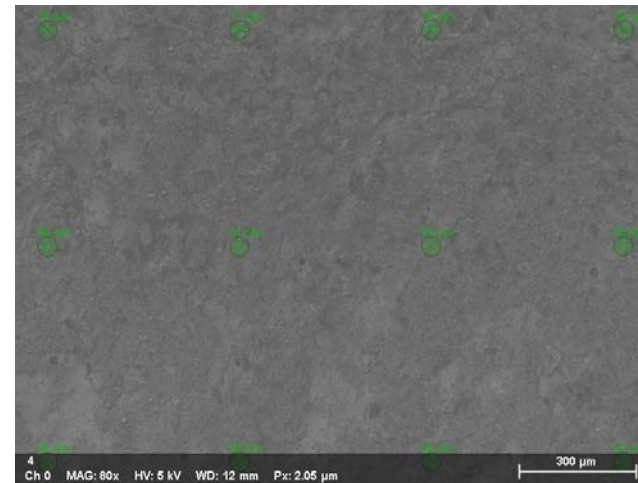


Example II: Galvanized Metal Sheet

Step 5: Quantify standard-based method

Normalized mass concentration [%]

Spectrum	Substrate	Layer 1	Zn [%]	Ni [%]
	Fe [%]	Thickn. [μm]		
76.spx	100.00	4.56	89.37	10.63
77.spx	100.00	4.51	89.27	10.73
78.spx	100.00	4.36	89.22	10.78
79.spx	100.00	4.44	89.28	10.72
80.spx	100.00	4.31	89.05	10.95
81.spx	100.00	4.52	89.26	10.74
82.spx	100.00	4.54	89.36	10.64
83.spx	100.00	4.54	89.27	10.73
84.spx	100.00	4.74	89.45	10.55
85.spx	100.00	4.53	89.41	10.59
86.spx	100.00	4.47	89.32	10.68
87.spx	100.00	4.36	89.16	10.84
Mean value	100.00	4.49	89.28	10.72
Std dev.	0.00	0.11	0.11	0.11
Std dev. rel. [%]	0.00	2.54	0.12	1.03
Conf. interval	0.00	0.03	0.03	0.03



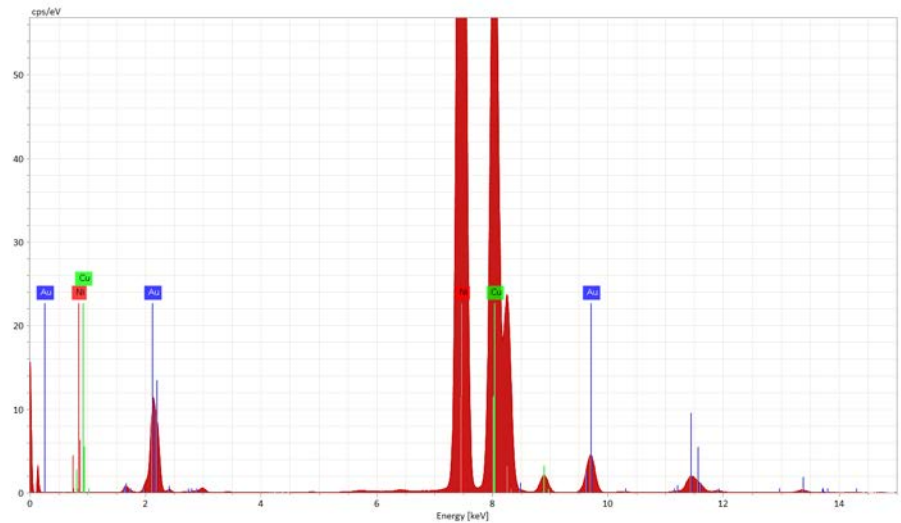
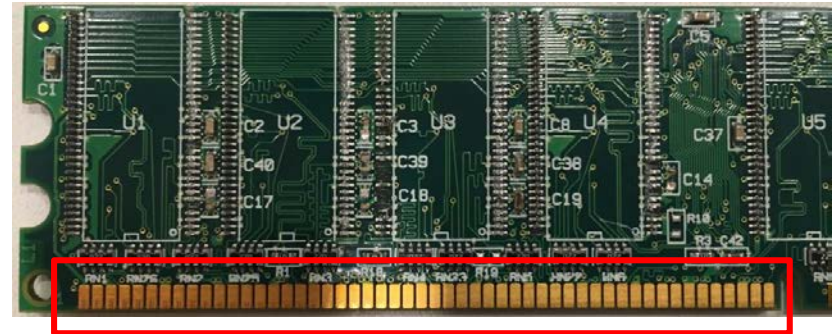
	Layer thickness	Ni %	Zn %
given	4.6 μm	11,10%	88,90%
Layer FP standard-free	3.32 μm	14,16%	85,84%
calibrated with standards	4.49 μm	10,72%	89,28%

Layer Thickness Analysis with micro-XRF/SEM



Example III: Au/Ni/Cu contact

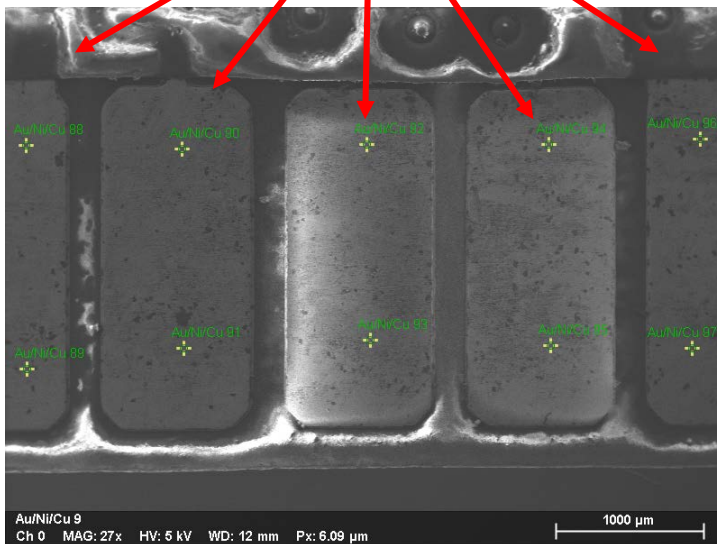
- Popular layer system which is used for contacts in micro-electronics
- For Higher- quality contacts also Pd is embedded as separate layer
- For cost reasons the precious metal coating are very thin and Ni is used as a diffusion barrier



Spectra of an Au-Ni/Cu layer system, acquisition time: 60 s

Coating Thickness Analysis

Example III: Au/Ni/Cu contact



Normalized mass concentration [%]

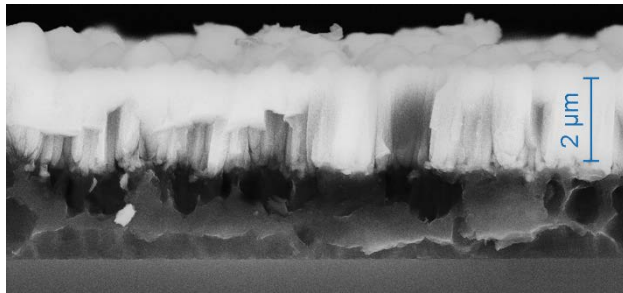
Spectrum	Substrate	Layer 1		Layer 2	
	Cu [%]	Thickn. [μm]	Ni [%]	Thickn. [nm]	Au [%]
Au/Ni/Cu 88	100.00	3.66	100.00	112.14	100.00
Au/Ni/Cu 89	100.00	3.71	100.00	111.43	100.00
Au/Ni/Cu 90	100.00	3.66	100.00	114.56	100.00
Au/Ni/Cu 91	100.00	3.78	100.00	112.43	100.00
Au/Ni/Cu 92	100.00	3.81	100.00	110.46	100.00
Au/Ni/Cu 93	100.00	3.69	100.00	104.50	100.00
Au/Ni/Cu 94	100.00	3.80	100.00	109.37	100.00
Au/Ni/Cu 95	100.00	3.73	100.00	101.50	100.00
Au/Ni/Cu 96	100.00	3.72	100.00	113.71	100.00
Au/Ni/Cu 97	100.00	3.78	100.00	102.39	100.00
Mean value	100.00	3.73	100.00	109.25	100.00
Std dev.	0.00	0.06	0.00	4.74	0.00
Std dev. rel. [%]	0.00	1.52	0.00	4.34	0.00
Conf. interval	0.00	0.02	0.00	1.50	0.00

Layer Thickness Analysis with micro-XRF/SEM

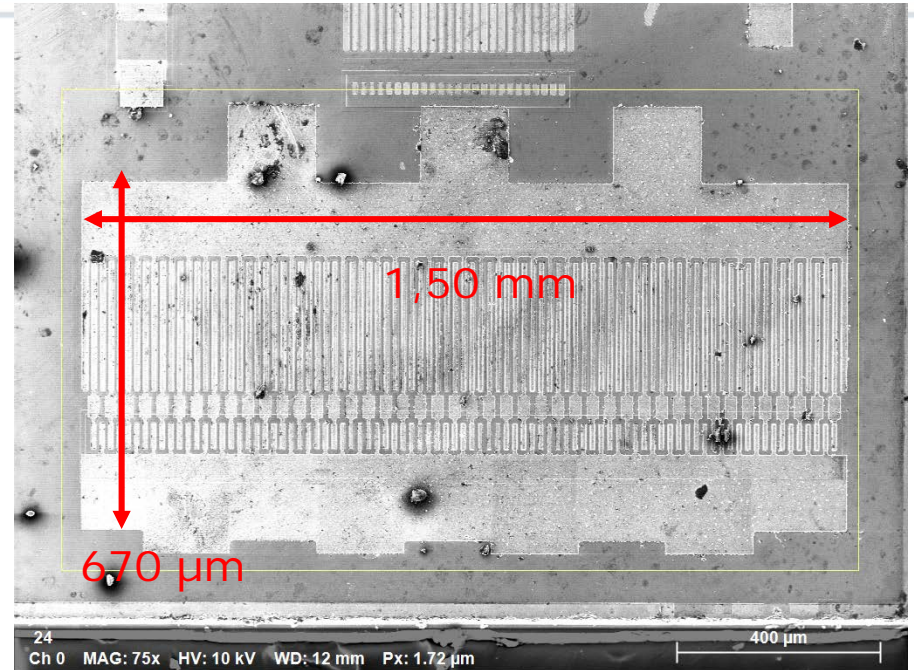
Example IV: Al/Si layer



Both samples mounted on graphite sample holder



Original sample 1: SEM image of a fracture edge of layer Al (bright) and substrate Si

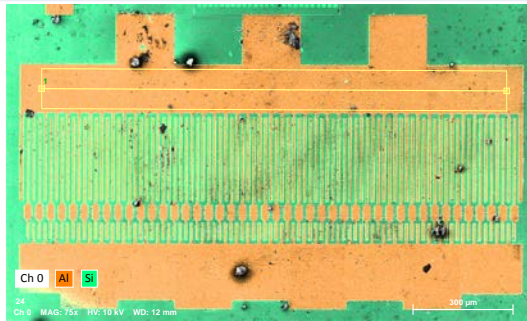


SE image of the sample 1

- Both samples were mounted on a graphite samples holder to avoid any influences coming from the sample holder due to the higher information depth of X-rays
- Both samples have the same dimensions 1.5 mm x 670 μm
- The thickness of the layer is ~ 2 μm for sample 1

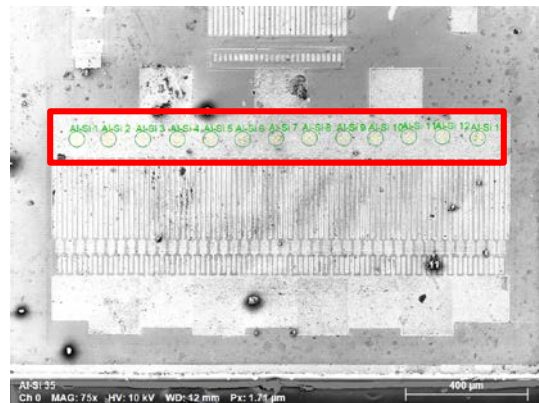
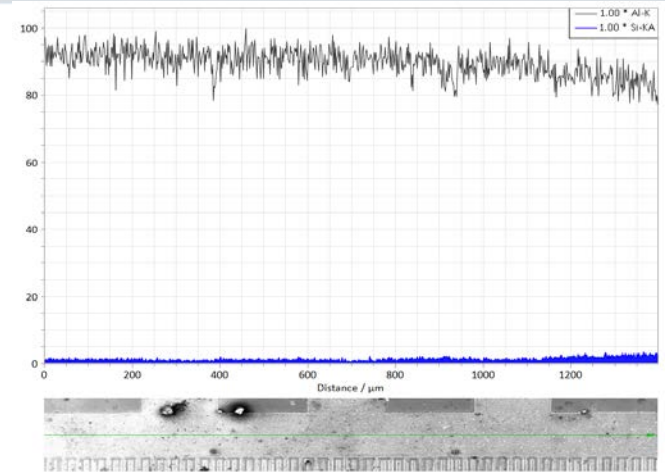
Layer Thickness Analysis with micro-XRF/SEM

Example IV: Al/Si layer → Sample I



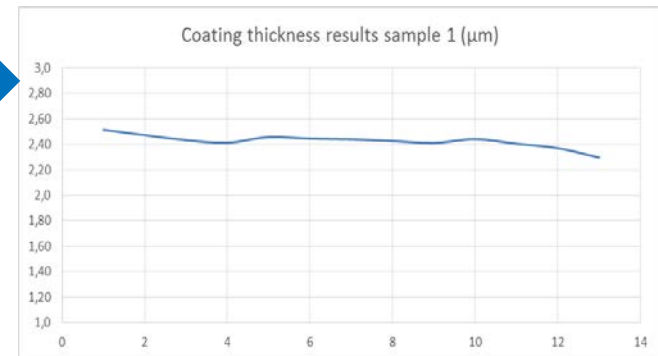
EDS Map of sample 1 with extracted line scan

Intensity profile of the extracted EDS line scan



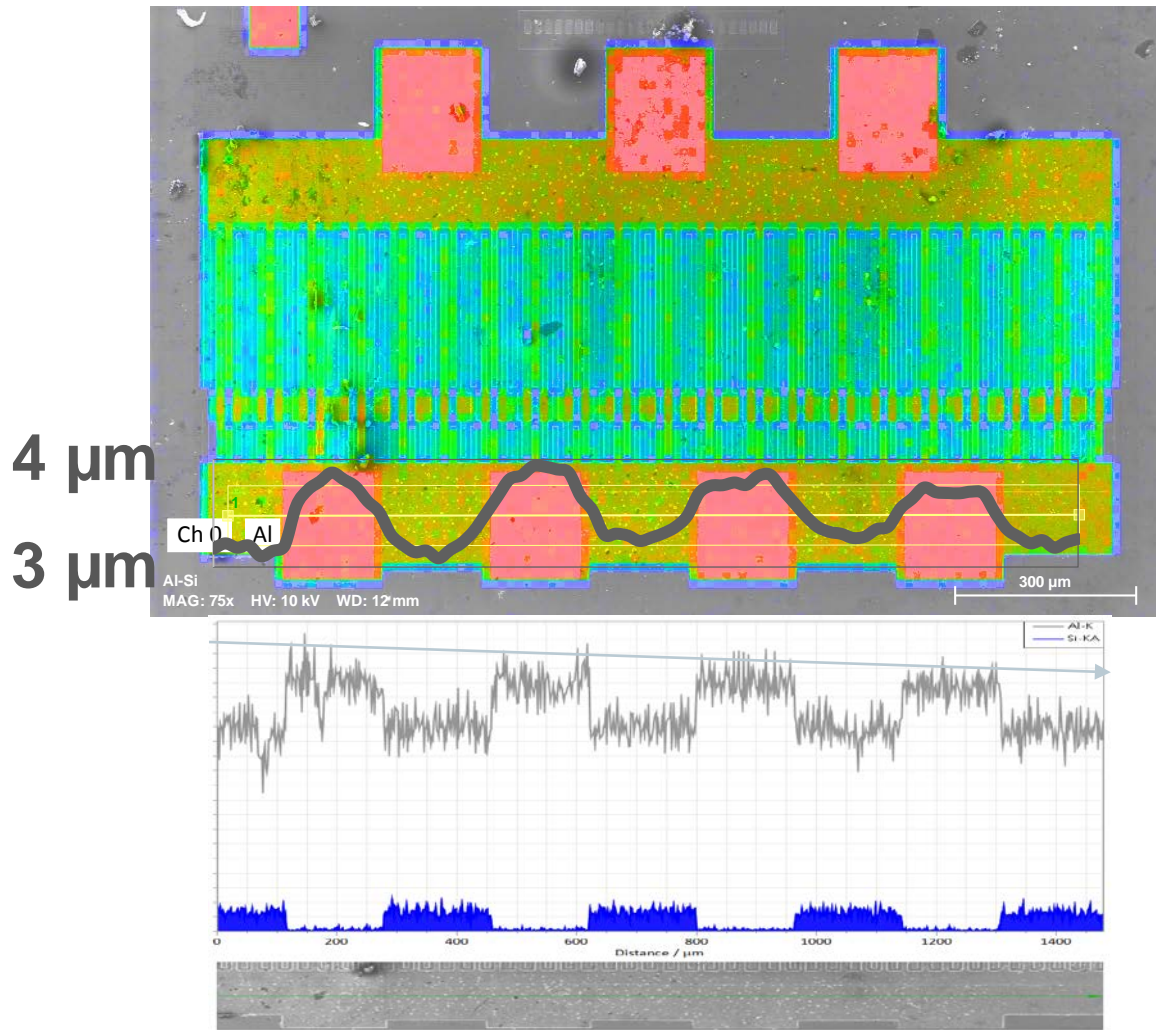
Normalized mass concentration [%]

Spectrum	Substrate		Layer 1	
	Si [%]	Thickn. [μm]	Al [%]	
Al-Si 1	100.00	2.51	100.00	
Al-Si 2	100.00	2.47	100.00	
Al-Si 3	100.00	2.43	100.00	
Al-Si 4	100.00	2.41	100.00	
Al-Si 5	100.00	2.46	100.00	
Al-Si 6	100.00	2.45	100.00	
Al-Si 7	100.00	2.44	100.00	
Al-Si 8	100.00	2.43	100.00	
Al-Si 9	100.00	2.41	100.00	
Al-Si 10	100.00	2.44	100.00	
Al-Si 11	100.00	2.41	100.00	
Al-Si 12	100.00	2.37	100.00	
Al-Si 13	100.00	2.30	100.00	
Mean value	100.00	2.43	100.00	
Std dev.	0.00	0.05	0.00	



Layer Thickness Analysis with micro-XRF/SEM

Example IV: Al/Si layer → sample 2



EDS Map of sample 2 in false color mode

XRF line-scan 75 points, Acquisition time: 30 sec / point

Example V: Analysis of solar cells by using Micro-XRF on SEM

Comparison of:

- SEM cross section – micro XRF to calculate the layer thickness
- EDS – micro XRF to calculate the layer composition

Layer Thickness Analysis with micro-XRF/SEM

CIGS- wafer



CIGS- wafer sample background:

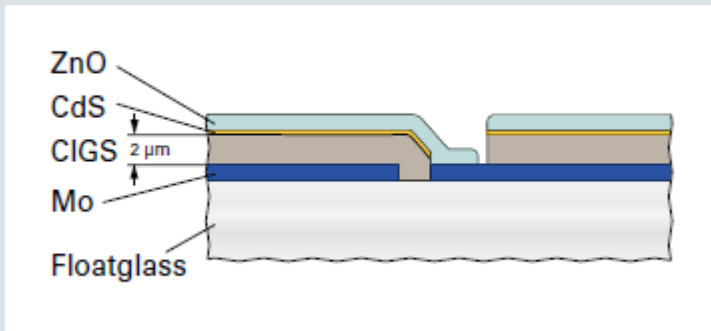
- The efficiency of solar cell is determined mainly by their chemical composition and physical structure
- Therefore the chemical composition is of particular interest for thin- film structures
- In most cases the absorber of the film cells is produced from CIGS- structures, which are Cu-In-Ga-Se or Cu-In-Ga-S compounds
- CIGS- solar cells are typically manufactured by deposition on glass substrates which are coated with a Mo- contact layer.
- For technology development and quality control during the manufacturing process it is important to check primarily composition but also thickness of these layers as well as their homogeneity
- A typical structure of a CIGS wafer is shown on the next slide

Coating Thickness Analysis

CIGS- wafer – structure and sample prep

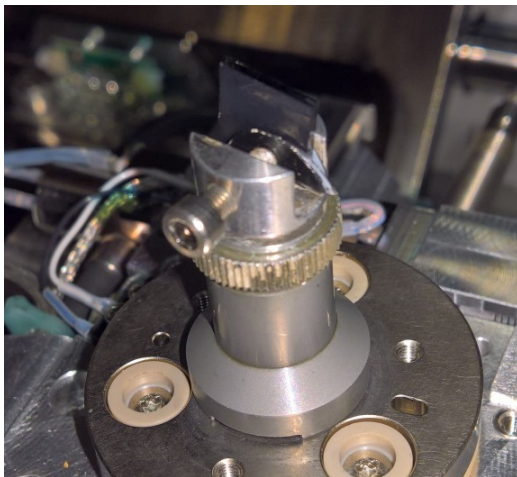


Sample structure



- Our sample contains of the float glass (substrate), Mo (1st layer), CIGS (2nd layer)
- No passivation layer
- Sample courtesy: PTB (Physical technical federation Berlin)

Typical structure of a CIGS solar cell



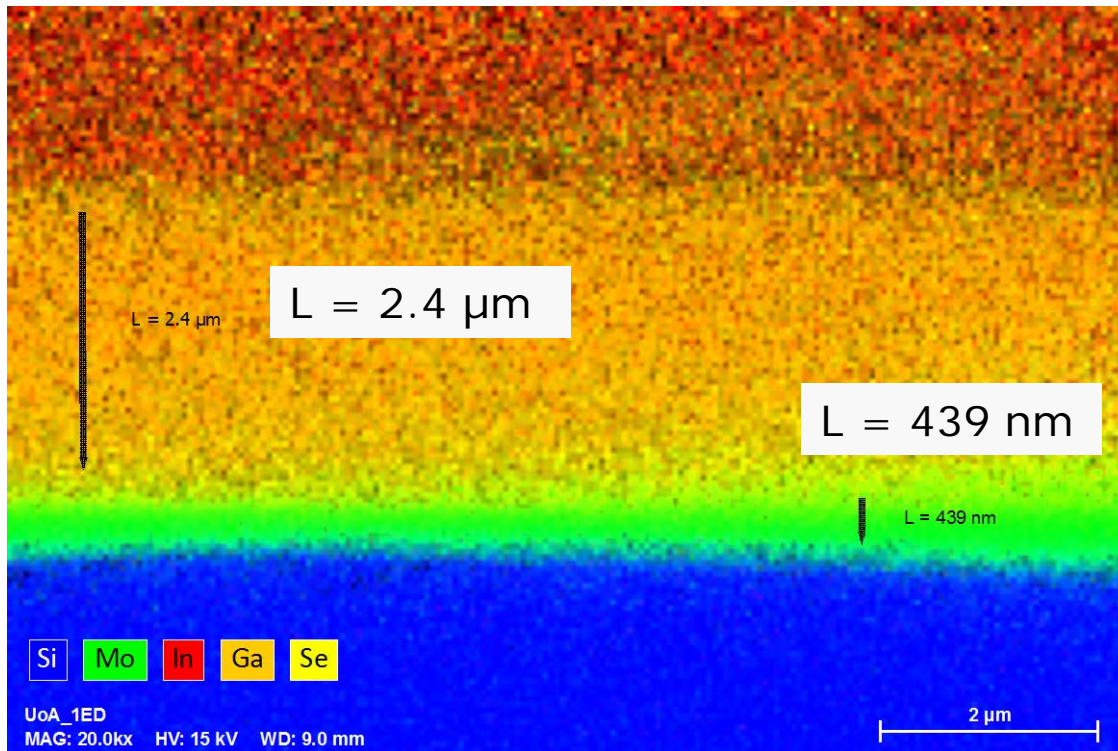
CIGS- wafer sample preparation for SEM cross section analysis in FEI NanoSEM 450



CIGS- wafer sample preparation for XRF analysis

Coating Thickness Analysis

CIGS- wafer – SEM layer observation



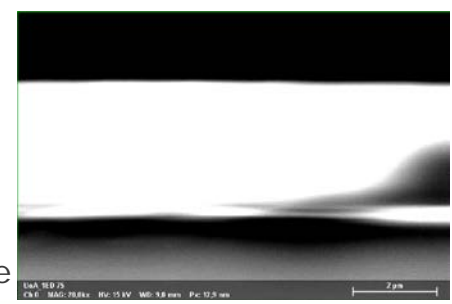
Noise (edge effect, due to sample prep) → Ignore it!

CIGS Layer structure

Mo – contact layer

Glass

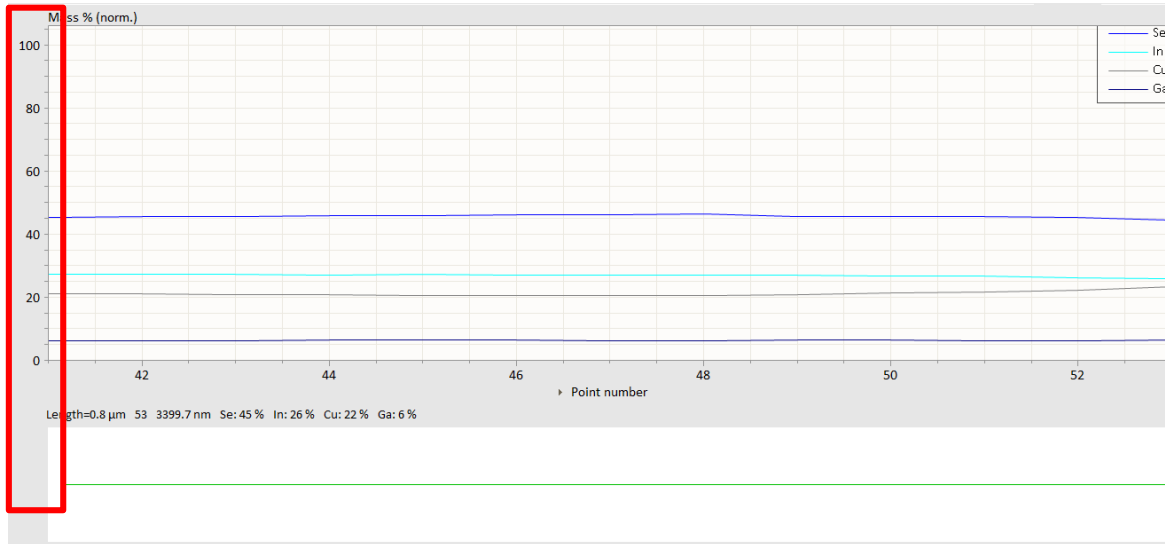
EDS map of a fracture edge of CIGS Wafer sample



SEM image of the map area, Backscattered image

Coating Thickness Analysis

CIGS- Wafer – EDS analysis



Extracted line scan area of the absorber (CIGS) for Cu, Ga, Se, In including concentration in wt%

Layer thickness results taken from the SEM cross section observation and quantification results of the CIGS layer taken from EDS analysis

Quantification of CIGS-layer

Method	Layer	Thickness /nm	Composition /wt.%			
			Cu	Ga	Se	In
SEM	Mo	440				
EDS	CIGS	2400	22	6	46	26

Coating Thickness Analysis CIGS- wafer – XRF analysis



Normalized mass concentration [%]

Spectrum	Substrate		Layer 1		Layer 2			
	Si [%]	Thickn. [nm]	Mo [%]	Thickn. [µm]	Cu [%]	In [%]	Ga [%]	Se [%]
CIGS 1	100.00	422.99	100.00	2.20	20.88	26.45	5.41	47.26
CIGS 2	100.00	420.67	100.00	2.21	20.94	26.31	5.41	47.35
CIGS 3	100.00	420.73	100.00	2.21	20.93	26.24	5.37	47.45
CIGS 4	100.00	418.65	100.00	2.20	20.92	26.42	5.38	47.27
CIGS 5	100.00	423.51	100.00	2.22	20.86	26.25	5.40	47.49
CIGS 6	100.00	421.52	100.00	2.21	20.96	26.37	5.37	47.30
CIGS 7	100.00	419.95	100.00	2.20	20.97	26.32	5.33	47.37
CIGS 8	100.00	424.57	100.00	2.22	21.04	26.39	5.40	47.17
CIGS 9	100.00	425.89	100.00	2.22	20.88	26.55	5.38	47.19
CIGS 10	100.00	428.78	100.00	2.23	20.88	26.45	5.37	47.31
CIGS 11	100.00	424.12	100.00	2.22	20.98	26.37	5.40	47.25
CIGS 12	100.00	420.95	100.00	2.21	20.95	26.31	5.41	47.33
CIGS 13	100.00	420.76	100.00	2.21	21.04	26.14	5.40	47.43
CIGS 14	100.00	425.46	100.00	2.23	20.88	26.51	5.42	47.19
CIGS 15	100.00	427.83	100.00	2.22	20.96	26.38	5.37	47.29
CIGS 16	100.00	420.92	100.00	2.23	21.22	26.15	5.36	47.27
CIGS 17	100.00	429.58	100.00	2.24	20.97	26.52	5.39	47.12
CIGS 18	100.00	426.31	100.00	2.22	21.03	26.19	5.37	47.41
CIGS 19	100.00	420.22	100.00	2.22	20.93	26.29	5.34	47.43
CIGS 20	100.00	428.18	100.00	2.23	20.93	26.29	5.43	47.34
Mean value	100.00	423.58	100.00	2.22	20.96	26.35	5.39	47.31
Std dev.	0.00	3.34	0.00	0.01	0.08	0.12	0.03	0.10
Std dev. rel. [%]	0.00	0.79	0.00	0.49	0.38	0.45	0.48	0.21
Conf. interval	0.00	0.75	0.00	0.00	0.02	0.03	0.01	0.02

Quantification of CIGS-layer

Method	Layer	Thickness /nm	Composition /wt. %			
			Cu	Ga	Se	In
Micro-XRF	Mo	424				
Micro-XRF	CIGS	2220	21	5.4	47.3	26.4

Coating Thickness Analysis

Comparing XRF – SEM thickness results



Quantification of CIGS-layer

Method	Layer	Thickness /nm	Composition /wt.%			
			Cu	Ga	Se	In
SEM	Mo	440				
Micro-XRF	Mo	424				
EDS	CIGS	2400	22	6	46	26
Micro-XRF	CIGS	2220	21	5.4	47.3	26.4

Conclusion:

- Measurements results agree very well between SEM layer thickness observation and
- for calculating the composition of the CIGS between EDS and XRF.

Summary

Layer Thickness Analysis with micro-XRF/SEM

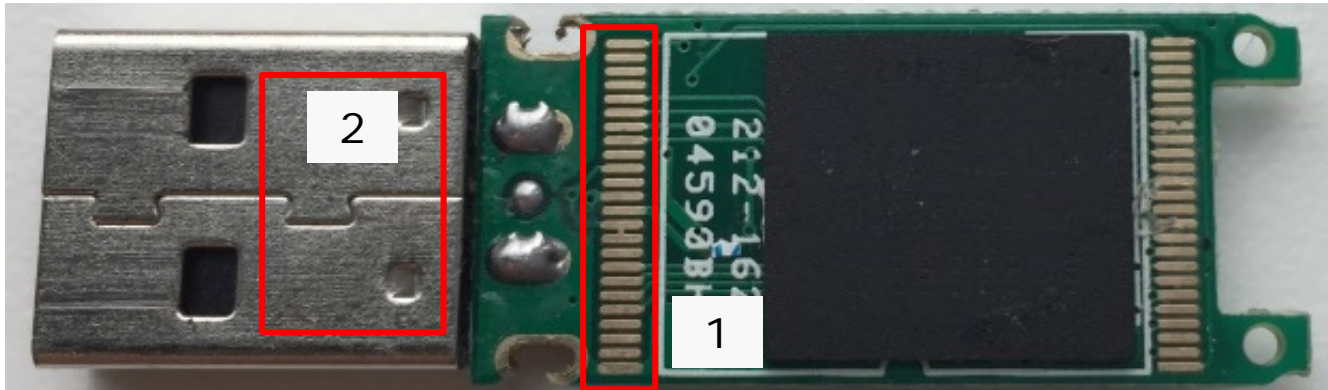


- XMethod software package for easy setup of analytical methods
- XRF can be utilized for coating thickness analysis of multilayer stacks
- Overall thickness of layer stack is limited (40 μm)
- Method editor for easy setup of wide range of methods, including option to choose lines to analyze
- Composition of multiple layers can be characterized
- Fundamental Parameter allows standard-less approach
- Standards can be applied in order to enhance precision



Live demonstration

Live demonstration USB stick



Areas of interest:

1. Au/Ni/Cu contact pins
2. USB housing → Ni/Cu/Fe

Are There Any Questions?

Please type in the questions you might have
in the Q&A box and press *Send*.

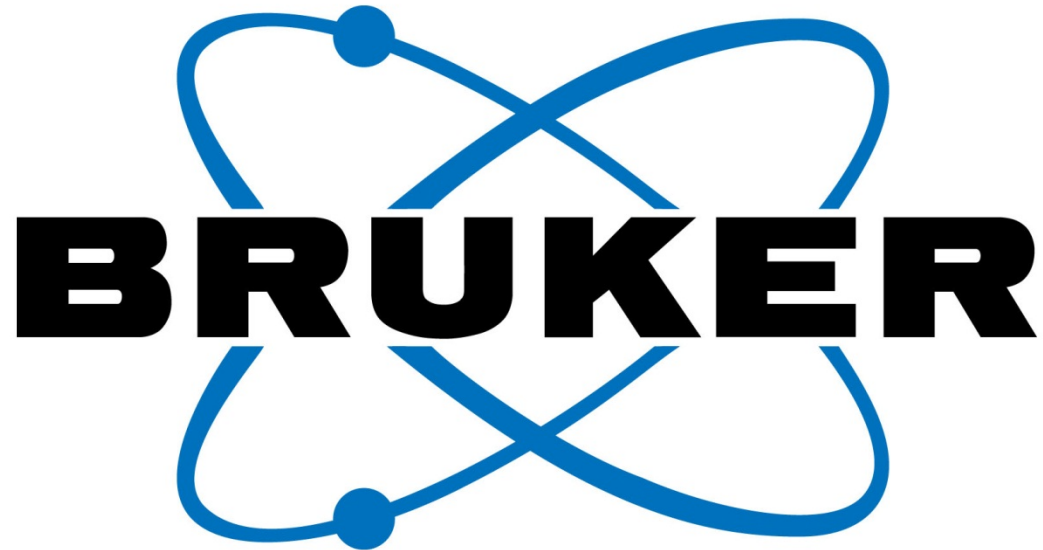


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