WEBINAR

Industrial applications of micro-XRF – Quality control at different stages

Bruker Nano Analytics - Micro-XRF applications

The speakers

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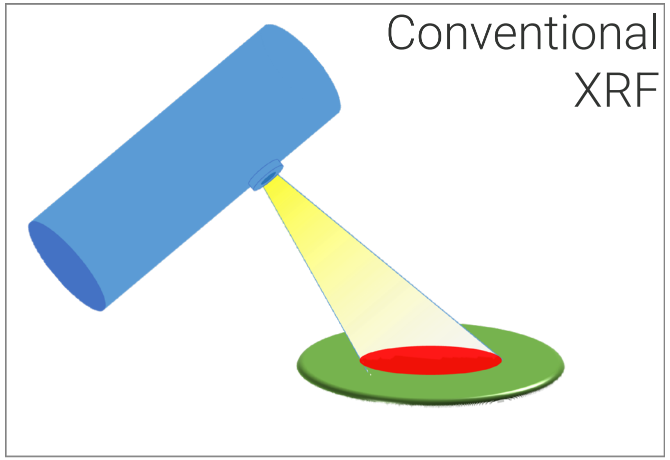
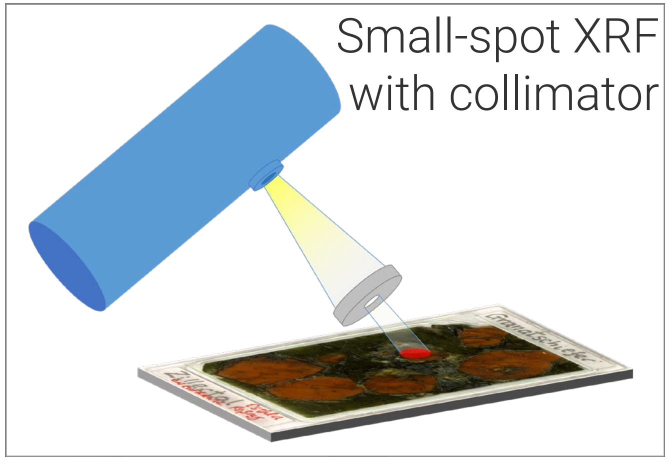
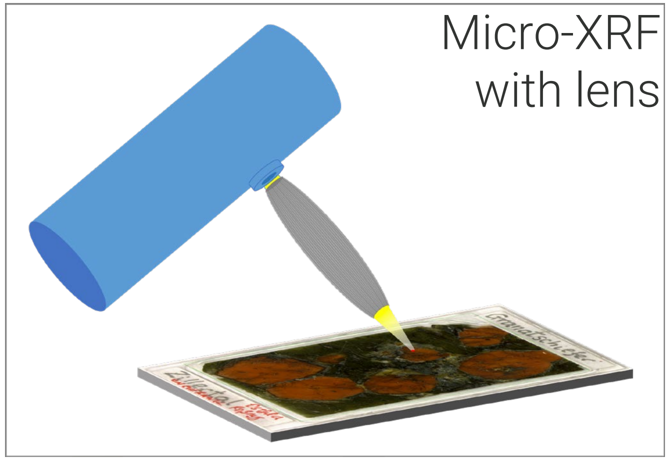
  Bruker Nano Analytics, Berlin, Germany

Agenda

From XRF to micro-XRF







Composition

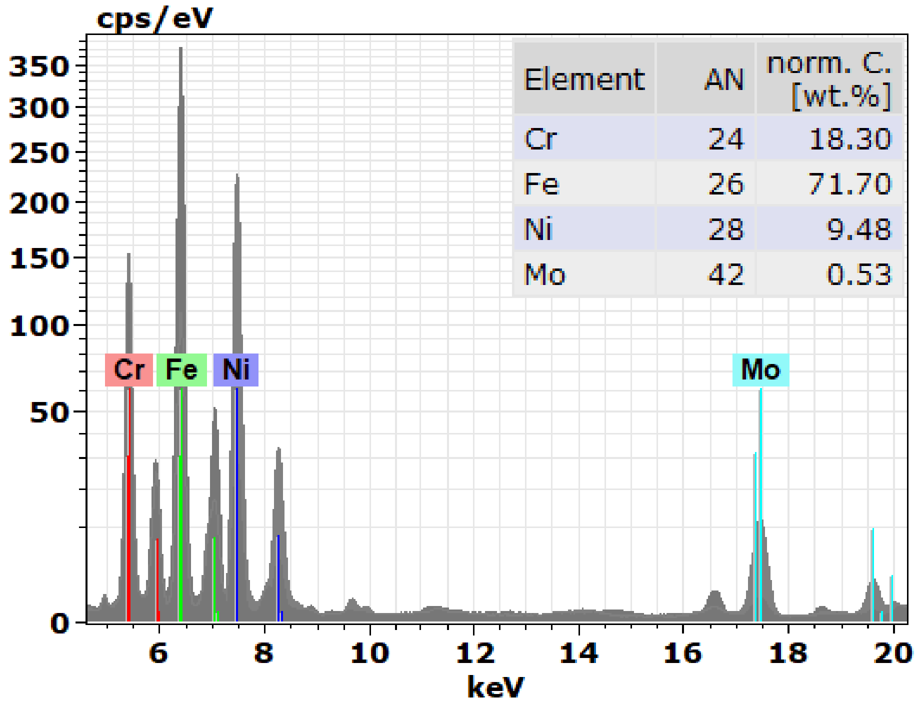
Compositional variations

From XRF to micro-XRF

- **Conventional X-ray fluorescence analysis** (XRF) is an analytical tool for qualitative and quantitative material analysis. It performs ideally in a **standardized workflow**.
- XRF tells you **which** elements are in the sample and **how much** of each one.
- Usually a sample needs “**preparation**”, including **homogenization** and/or **dilution** for matrix reduction.

Information is lost!

The compositional variations in a sample may be a crucial property of the material!



Element	AN	norm. C. [wt.%]
Cr	24	18.30
Fe	26	71.70
Ni	28	9.48
Mo	42	0.53

Micro-XRF

- Micro-XRF is XRF with a small spot (nowadays typically < 20 µm).

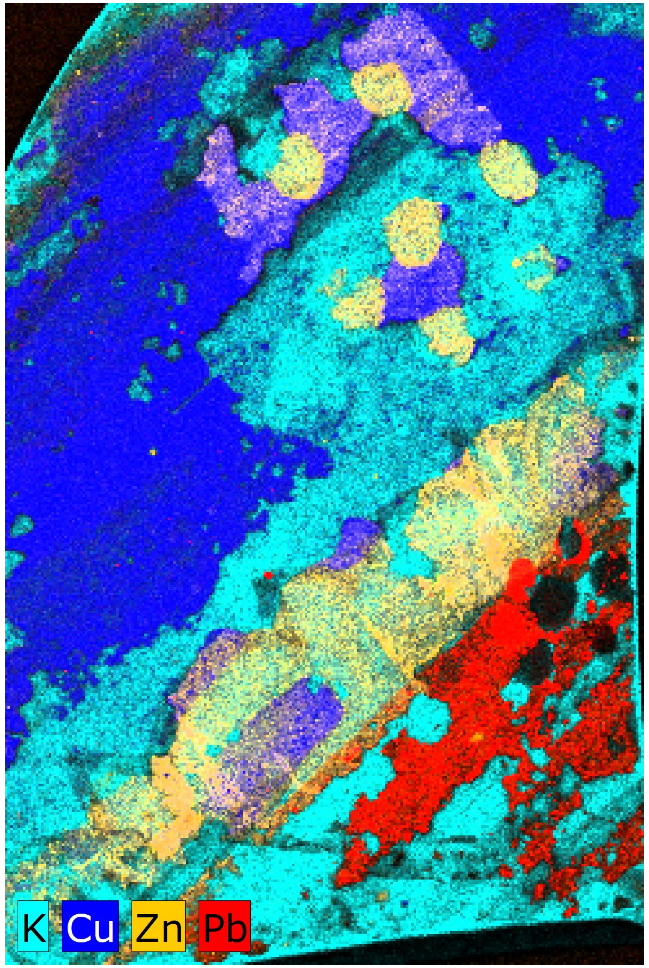
  → Micro-XRF reveals where elements are.

  → Micro-XRF is ideal for non-homogeneous samples.

- It usually requires minimal or no sample preparation.

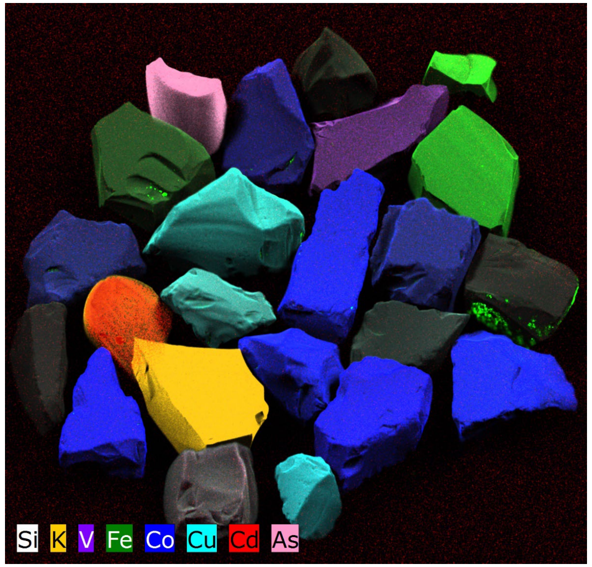
- **Quantitative micro-XRF** is feasible for sufficiently homogeneous areas of the sample, which can be even below 100 µm in diameter.

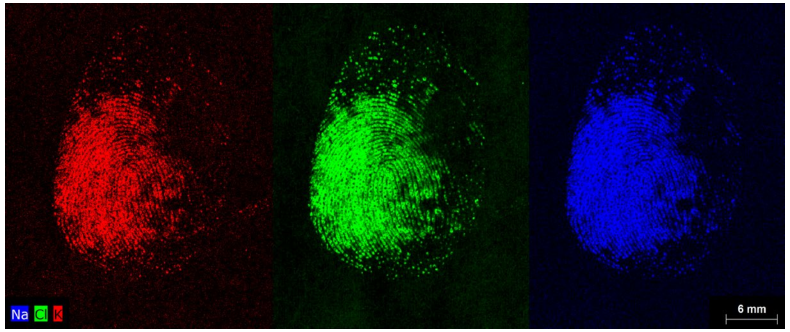
- The measurement conditions are very flexible in order to address different analytical tasks or requirements posed by the sample.



Micro-XRF
What is it used for?

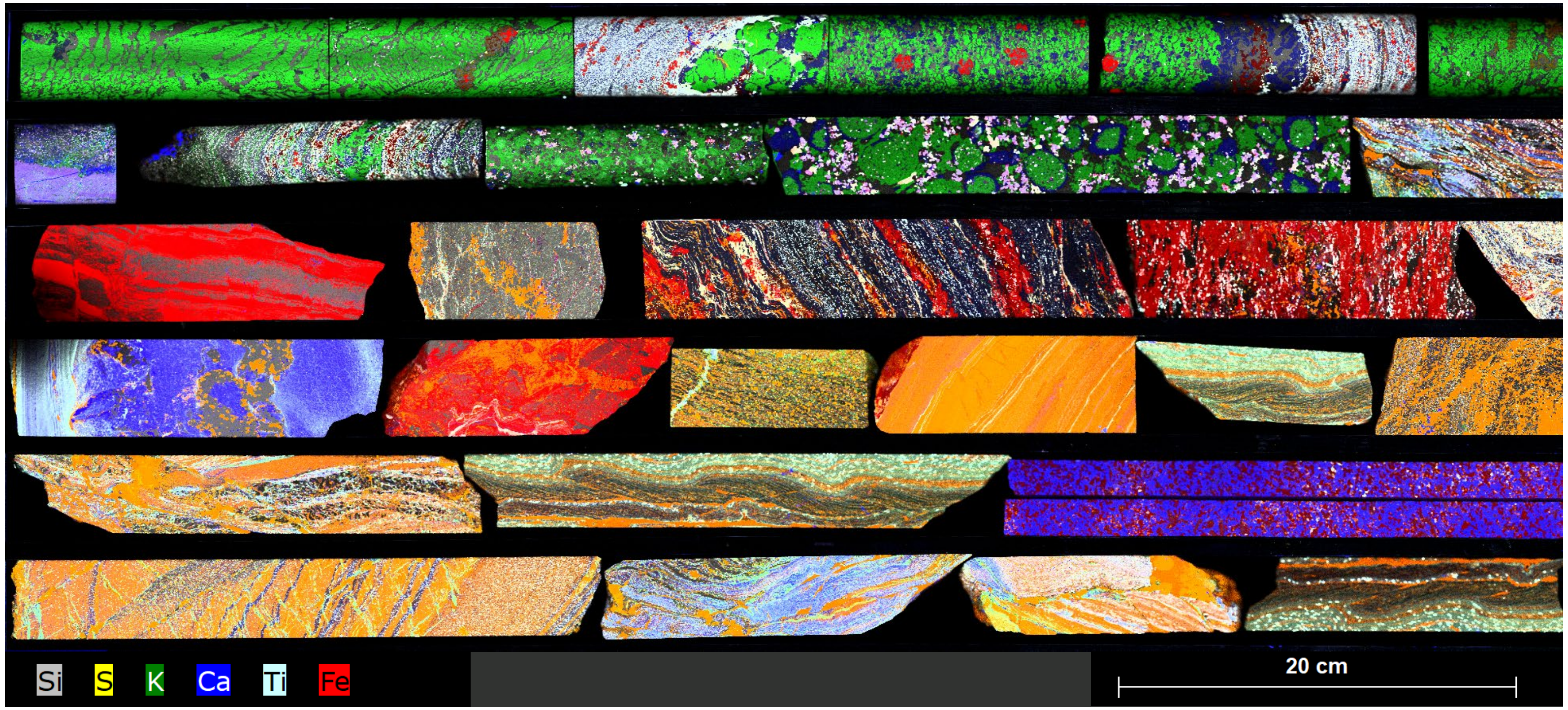
- Forensics and life sciences





Micro-XRF
What is it used for?

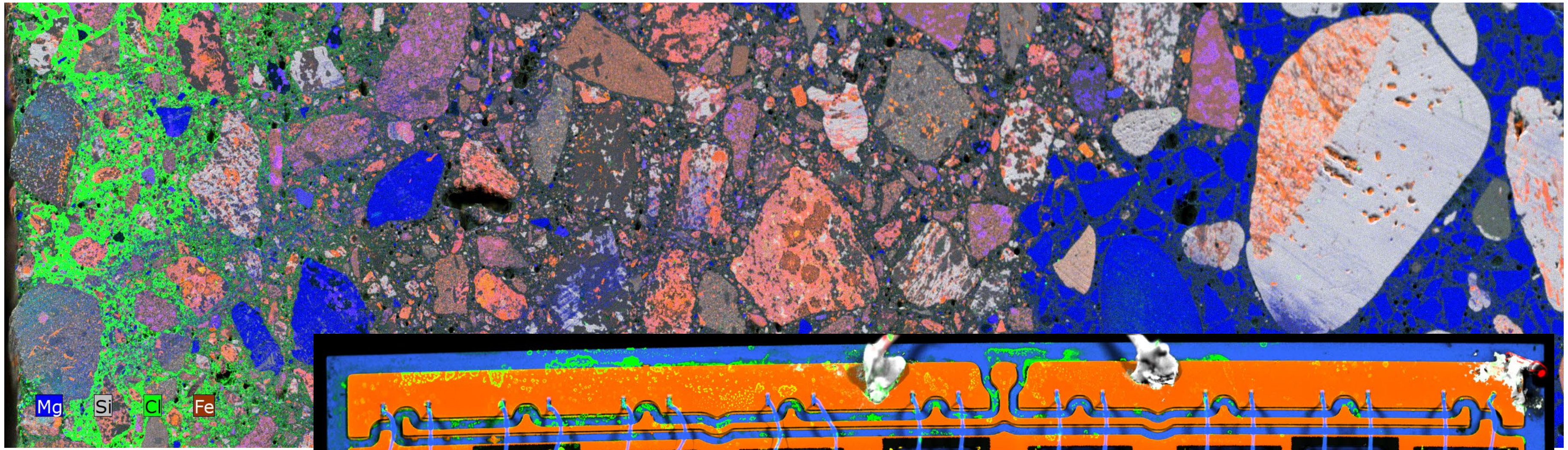
- Art, conservation, archaeology



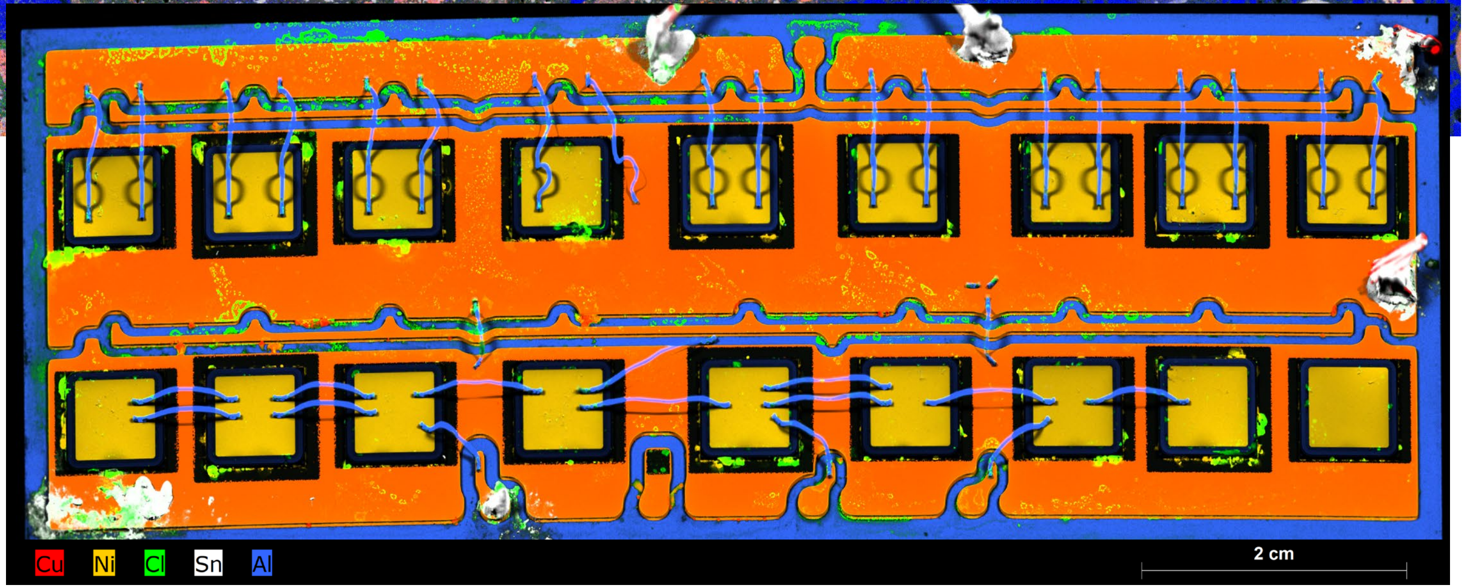
- Geology

Micro-XRF
What is it used for?

- Materials science



- R&D



Micro-XRF
Uses in industrial environment

- Micro-XRF is well established in academic environment and the R&D or central labs of large companies.

... but where does it fit in a more industrial environment?

- Even though it is possible to operate the M4 TORNADO in an automated environment, this will not be the topic of this presentation.
- We will discuss the standard operation of the instrument and where its analytical methods are beneficial in an industrial workflow.

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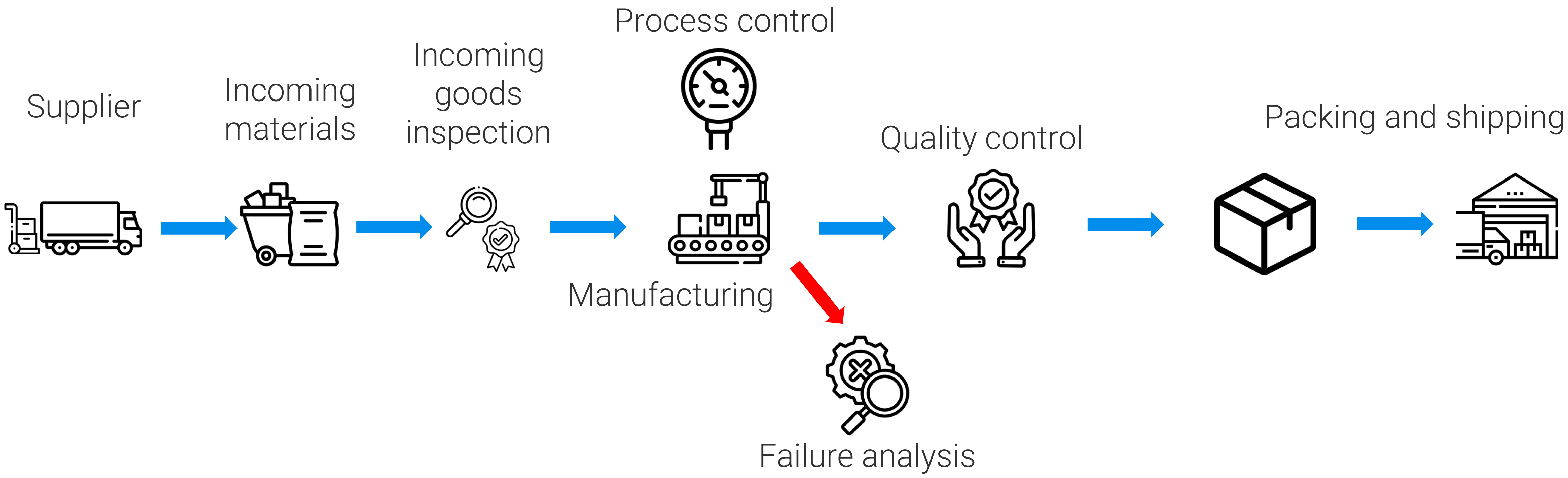
Micro-XRF – Quality control at different stages



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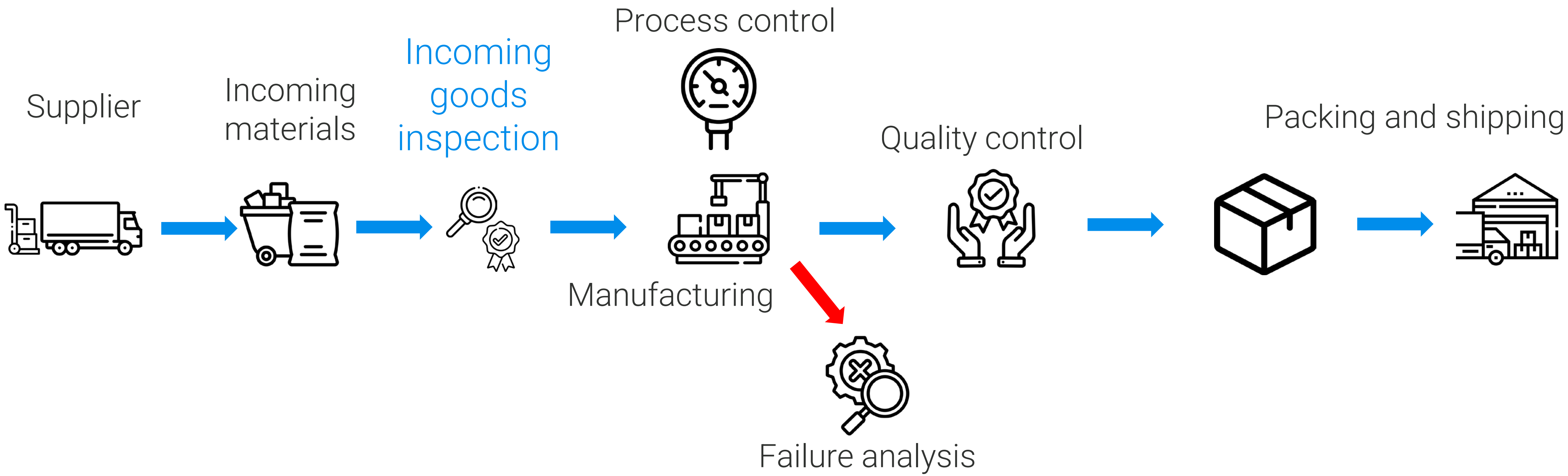
Micro-XRF – Quality control at different stages Incoming goods inspection



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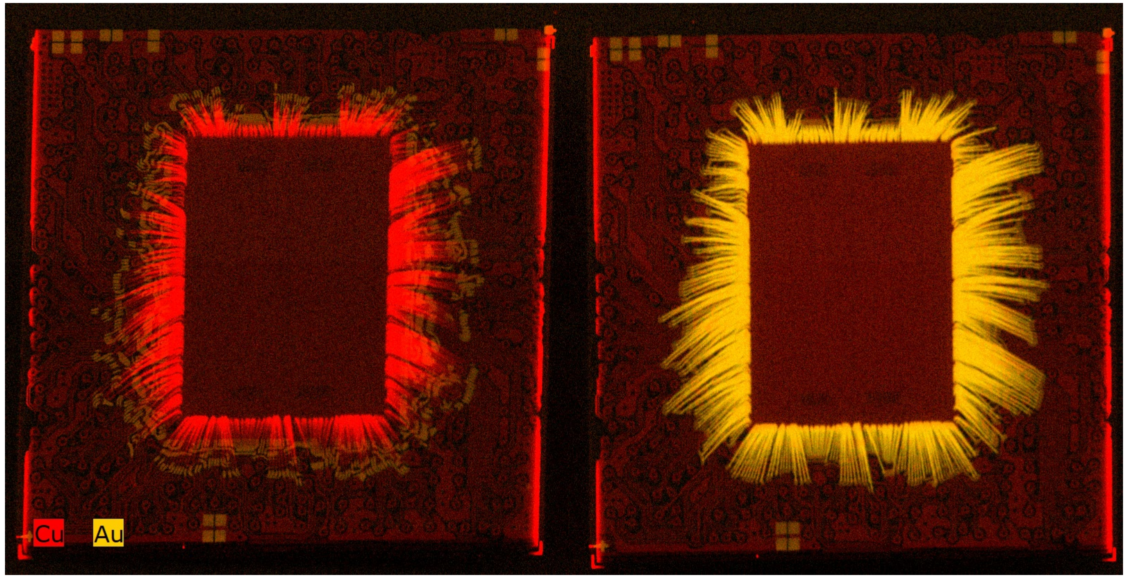
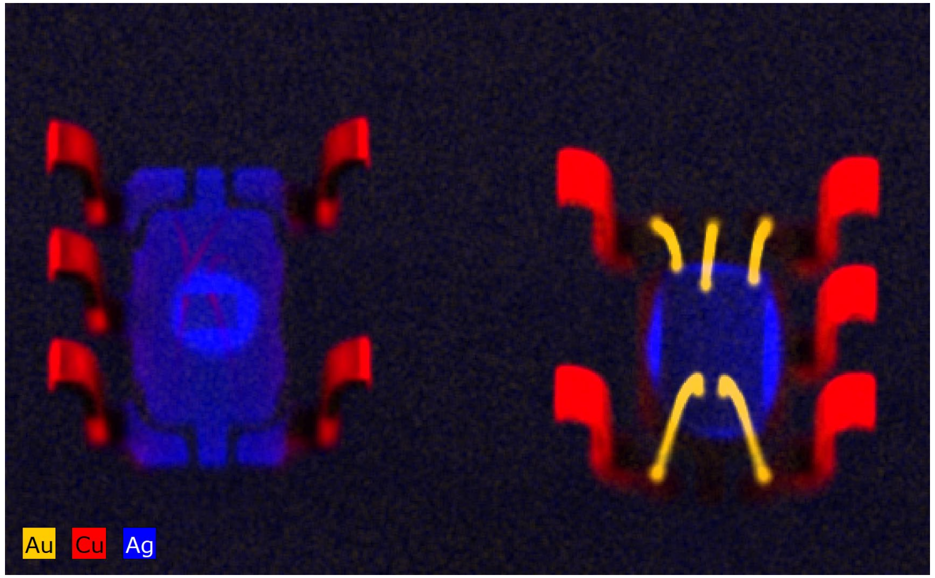
Incoming goods inspection Qualitative analysis

- Updated chip design, announced by the manufacturer:
  - Copper wires instead of gold to save costs.

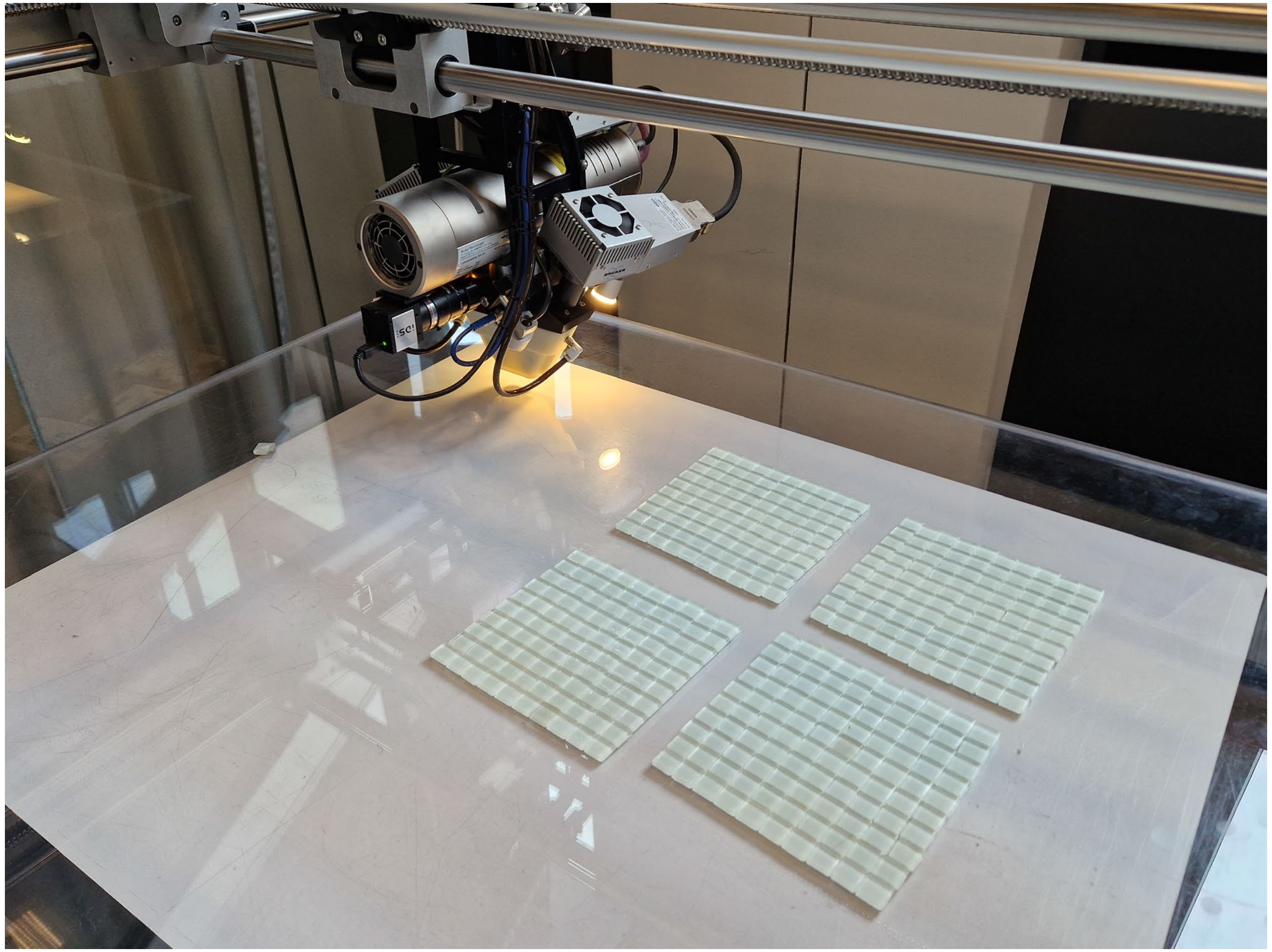
- Here a new supplier sent a batch of ICs with the same designation who behaved differently when tested.
- Micro-XRF showed a different inner design.



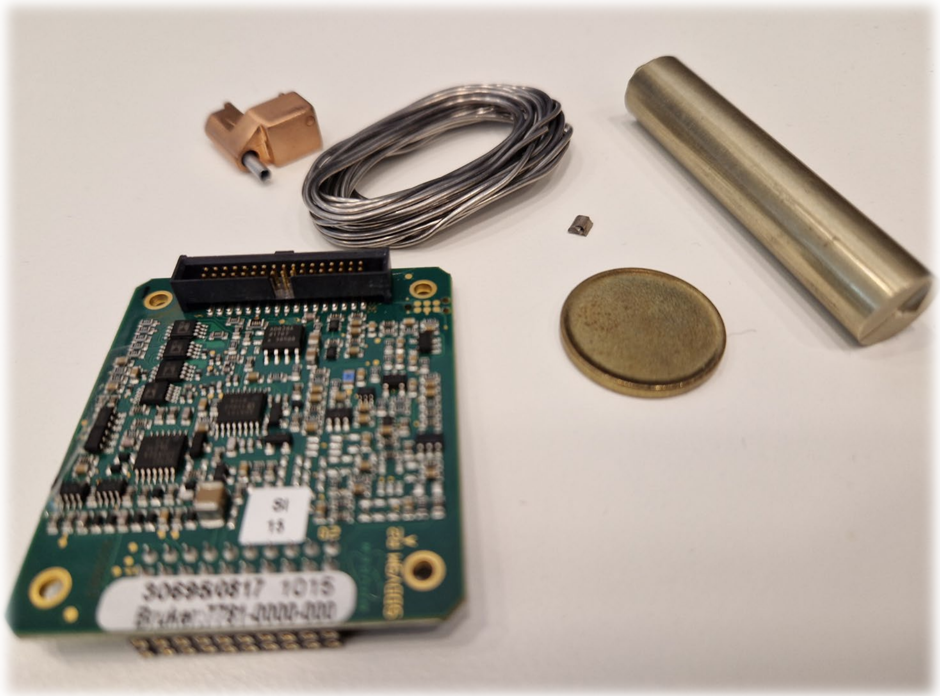


Incoming goods inspection
Qualitative analysis

- With sufficiently clear criteria (Au wires in ICs) even qualitative analysis can be done as point measurements, maybe with collimator.
- Example here: in batches of glass tiles, a specific subset of potassium-rich tiles needs to be identified.



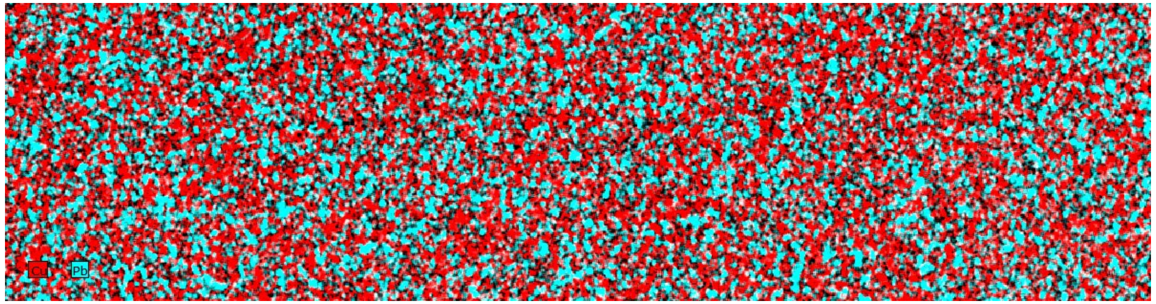
Incoming goods inspection
Quantitative analysis – Introduction



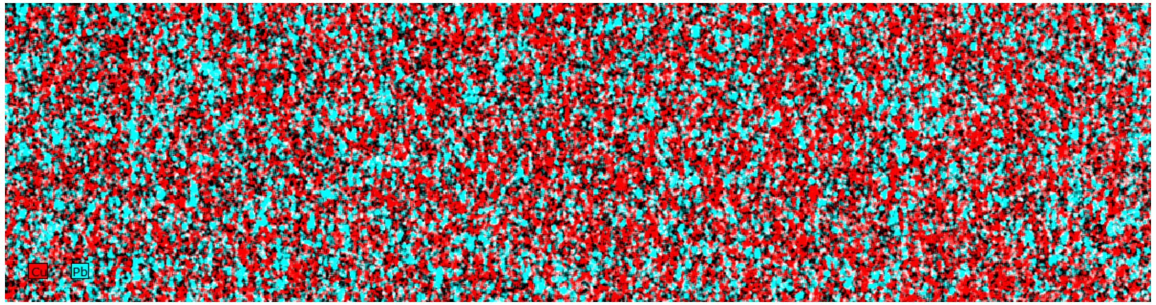
Some challenging samples

- When using metal alloys as raw materials a quantitative analysis of incoming goods is vital.
- Next to OES, XRF is the most common method for incoming goods inspection, be it handheld, benchtop or floor-standing.
- When the samples are too small for larger instruments, micro-XRF can be used with high results quality.
- Also, it has the benefit of being relatively forgiving when it comes to adverse sample shapes.
  - However, like for most techniques, a more sophisticated sample preparation (flat surfaces) and thorough sample placement (horizontal alignment) greatly improves the attainable quality of the results.

Incoming goods inspection
Quantitative analysis – Copper alloys



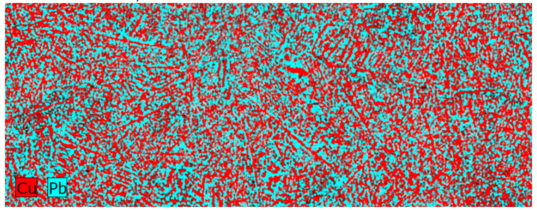
BAM
ERM-EB 375
Cu 58 %, Zn 39 % Pb 2.9 %



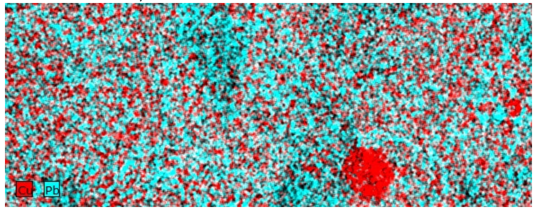
ARMI
CDA 360 73B
Cu 61.5 %, Zn 35.5 % Pb 1.9 %

- Element distribution in bronzes
- The alloy is clearly inhomogeneous, because the lead (added as cutting aid) forms inclusions.
- Quantification is possible with
  - **Collimator** measurements,
  - the **sum spectrum** of a map,
  - the **average values** of a sufficiently large set of point measurements.

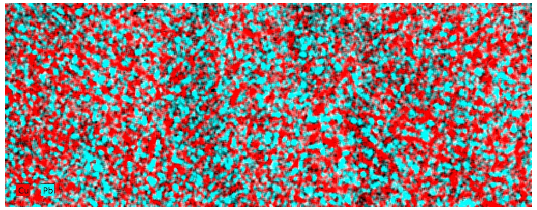
LGC Standards
SUS RC36
Cu 82.5 %, Zn 0.25 % Pb 9.6 %



ARMI
CDA 932
Cu 83.2 %, Zn 2.6 % Pb 6.8 %



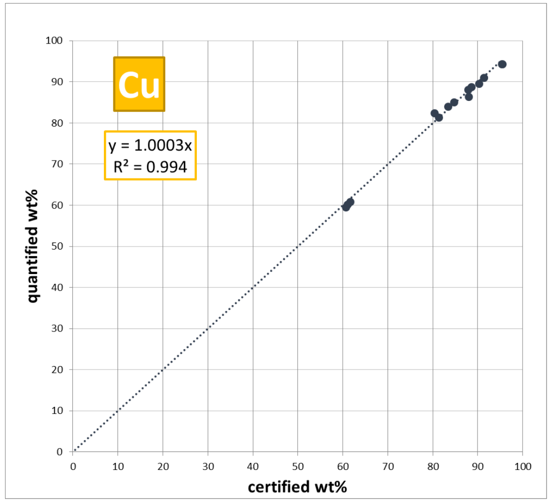
ARMI
CDA 397
Cu 80.2 %, Zn 0.04 % Pb 9.2 %

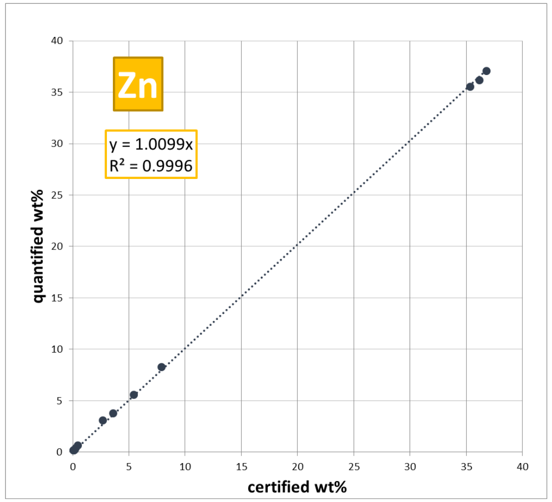


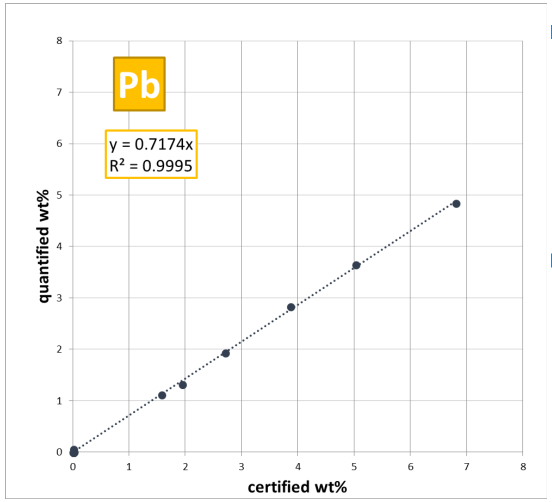
Incoming goods inspection
Quantitative analysis – Copper alloys

Grade-IARM	Al	Co	Cr	Cu	Fe	Mn	Mo	Nb	Ni	P	Pb	Si	Sn	Ti	V	W	Zn	Zr
CDA314-72B	T	T	T	90.1	0.0	T	T	T	0.0	0.0	2.0	0.0	0.0	T	T	T	7.8	T
CDA360-73B	0.0010	0.0	T	61.5	0.2	0.0	T	T	0.1	0.0	2.7	0.0	0.2	T	T	T	35.3	T
CDA485-76B	0.0050	0.0	T	60.5	0.1	0.0	T	T	0.0	0.0	1.9	T	0.7	T	T	T	36.7	T
CDA510-77B	0.0010	T	T	95.2	0.0	0.0	T	T	0.0	0.1	0.0	0.0	4.7	T	T	T	0.0	T
CDA544-78B	0.0020	T	T	87.7	0.0	0.0	T	T	0.1	0.2	3.9	T	4.7	T	T	T	3.6	T
CDA623-79B	9.1900	0.0	0.0	88.4	2.1	0.2	T	T	0.1	0.0	0.0	0.0	0.0	T	T	T	0.0	T
CDA630-80B	10.1900	0.0	0.0	81.2	3.3	0.5	T	T	4.7	0.0	0.0	0.0	0.0	T	T	T	0.1	T
CDA642-81B	6.7000	T	0.0	91.2	0.0	0.0	T	T	0.0	0.0	0.0	1.8	0.0	T	T	T	0.2	T
CDA655-82B	0.0020	T	0.0	95.3	0.1	1.0	T	T	0.0	0.0	0.0	3.2	0.0	T	T	T	0.4	T
CDA706-84B	0.0020	0.0	0.0	87.9	1.3	0.6	T	T	10.0	0.0	0.0	0.0	0.0	T	T	T	0.1	T
CDA836-86C	0.0020	T	T	84.6	0.2	0.0	T	T	0.3	0.0	5.0	0.0	4.4	T	T	T	5.4	T
CDA857-87B	0.2000	0.0	0.0	60.9	0.3	0.0	T	T	T	0.0	1.6	0.0	0.8	T	T	T	36.1	T
CDA932-91C	0.0020	T	T	83.2	0.0	0.0	T	T	0.5	0.1	6.8	0.0	6.8	T	T	T	2.6	T
CDA937-BS937B-1	T	T	T	80.2	0.0	T	T	T	0.4	0.0	9.2	T	9.7	T	T	T	0.0	T

- (Type-)calibration of the M4 TORNADO's FP quantification needs reference materials.
- Measure sets of samples of known composition and quantify them.



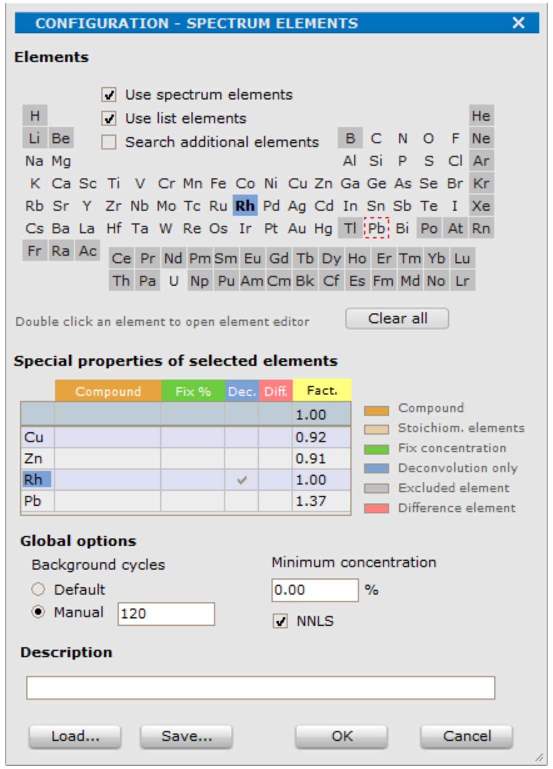


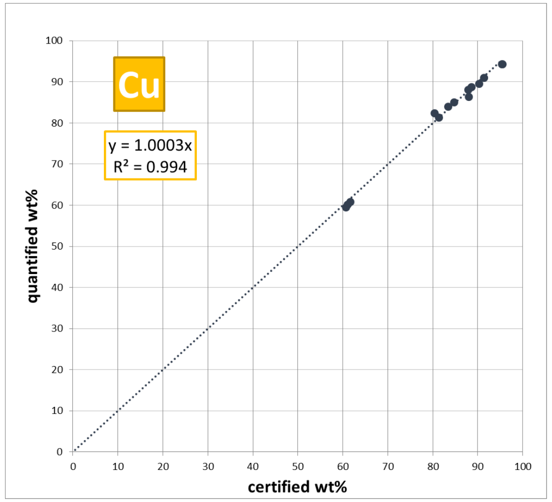


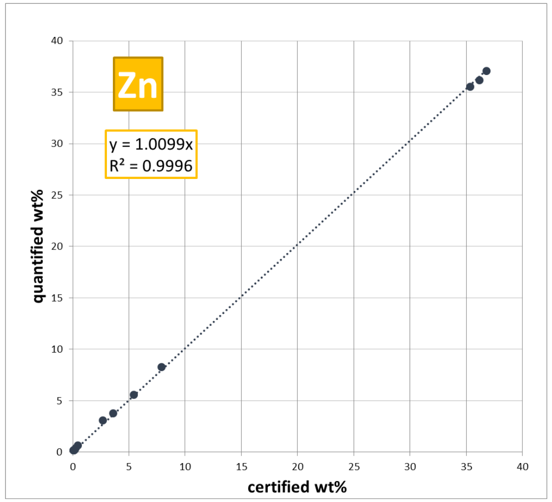
- The initial correlation already is usually very linear.
- The slopes of the correlation may need to be adapted (=calibrated).

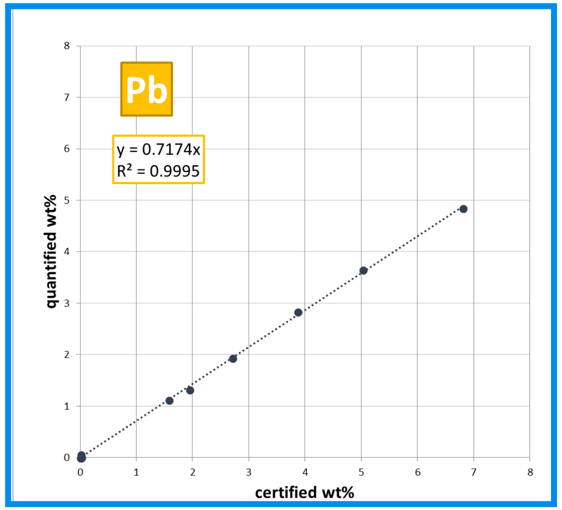
Incoming goods inspection
Quantitative analysis – Copper alloys

Grade-IARM	Al	Co	Cr	Cu	Fe	Mn	Mo	Nb	Ni	P	Pb	Si	Sn	Ti	V	W	Zn	Zr
CDA314-72B	T	T	T	90.1	0.0	T	T	T	0.0	0.0	2.0	0.0	0.0	T	T	T	7.8	T
CDA360-73B	0.0010	0.0	T	61.5	0.2	0.0	T	T	0.1	0.0	2.7	0.0	0.2	T	T	T	35.3	T
CDA485-76B	0.0050	0.0	T	60.5	0.1	0.0	T	T	0.0	0.0	1.9	T	0.7	T	T	T	36.7	T
CDA510-77B	0.0010	T	T	95.2	0.0	0.0	T	T	0.0	0.1	0.0	0.0	4.7	T	T	T	0.0	T
CDA544-78B	0.0020	T	T	87.7	0.0	0.0	T	T	0.1	0.2	3.9	T	4.7	T	T	T	3.6	T
CDA623-79B	9.1900	0.0	0.0	88.4	2.1	0.2	T	T	0.1	0.0	0.0	0.0	0.0	T	T	T	0.0	T
CDA630-80B	10.1900	0.0	0.0	81.2	3.3	0.5	T	T	4.7	0.0	0.0	0.0	0.0	T	T	T	0.1	T
CDA642-81B	6.7000	T	0.0	91.2	0.0	0.0	T	T	0.0	0.0	0.0	1.8	0.0	T	T	T	0.2	T
CDA655-82B	0.0020	T	0.0	95.3	0.1	1.0	T	T	0.0	0.0	0.0	3.2	0.0	T	T	T	0.4	T
CDA706-84B	0.0020	0.0	0.0	87.9	1.3	0.6	T	T	10.0	0.0	0.0	0.0	0.0	T	T	T	0.1	T
CDA836-86C	0.0020	T	T	84.6	0.2	0.0	T	T	0.3	0.0	5.0	0.0	4.4	T	T	T	5.4	T
CDA857-87B	0.2000	0.0	0.0	60.9	0.3	0.0	T	T	T	0.0	1.6	0.0	0.8	T	T	T	36.1	T
CDA932-91C	0.0020	T	T	83.2	0.0	0.0	T	T	0.5	0.1	6.8	0.0	6.8	T	T	T	2.6	T
CDA937-BS937B-1	T	T	T	80.2	0.0	T	T	T	0.4	0.0	9.2	T	9.7	T	T	T	0.0	T









Incoming goods inspection
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CDA544-78B	0.0020	T	T	87.7	0.0	0.0	T	T	0.1	0.2	3.9	T	4.7	T	T	T	3.6	T
CDA623-79B	9.1900	0.0	0.0	88.4	2.1	0.2	T	T	0.1	0.0	0.0	0.0	0.0	T	T	T	0.0	T
CDA630-80B	10.1900	0.0	0.0	81.2	3.3	0.5	T	T	4.7	0.0	0.0	0.0	0.0	T	T	T	0.1	T
CDA642-81B	6.7000	T	0.0	91.2	0.0	0.0	T	T	0.0	0.0	0.0	1.8	0.0	T	T	T	0.2	T
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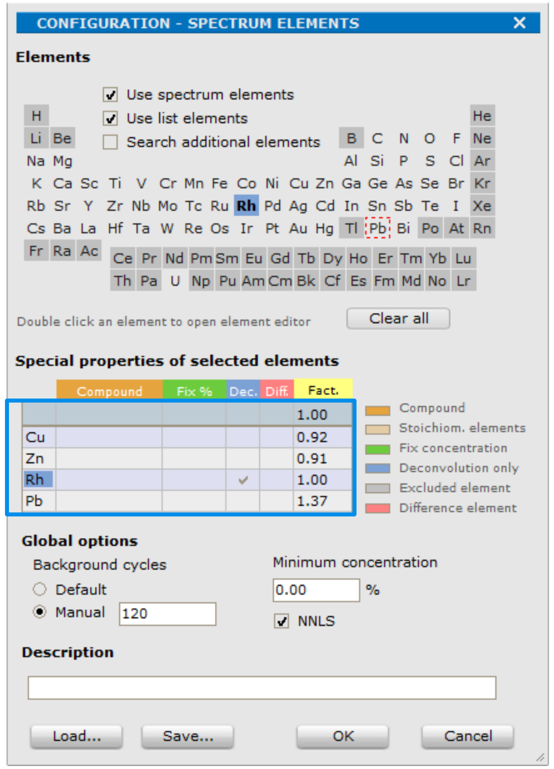
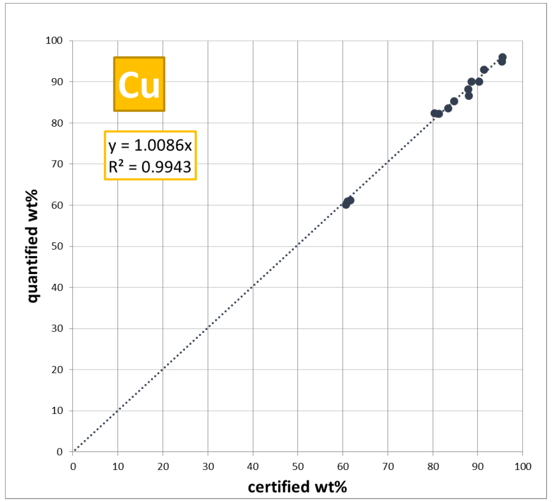
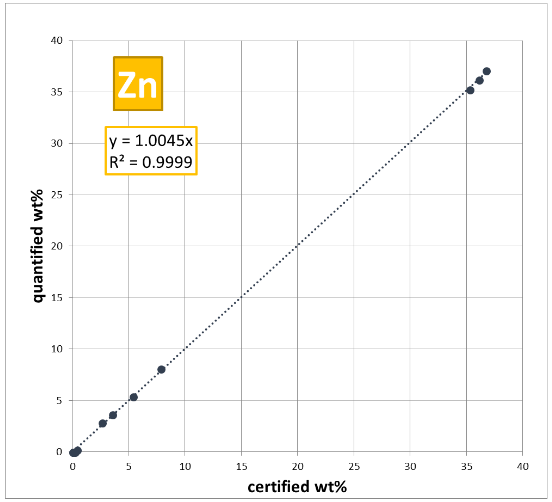
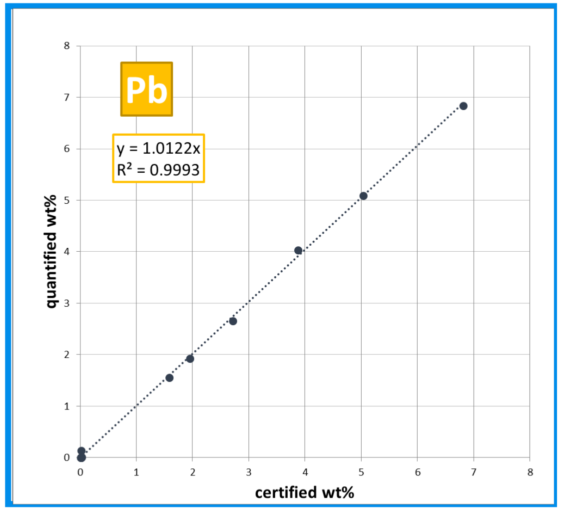
CDA937-BS937B-1	T	T	T	80.2	0.0	T	T	T	0.4	0.0	9.2	T	9.7	T	T	T	0.0	T

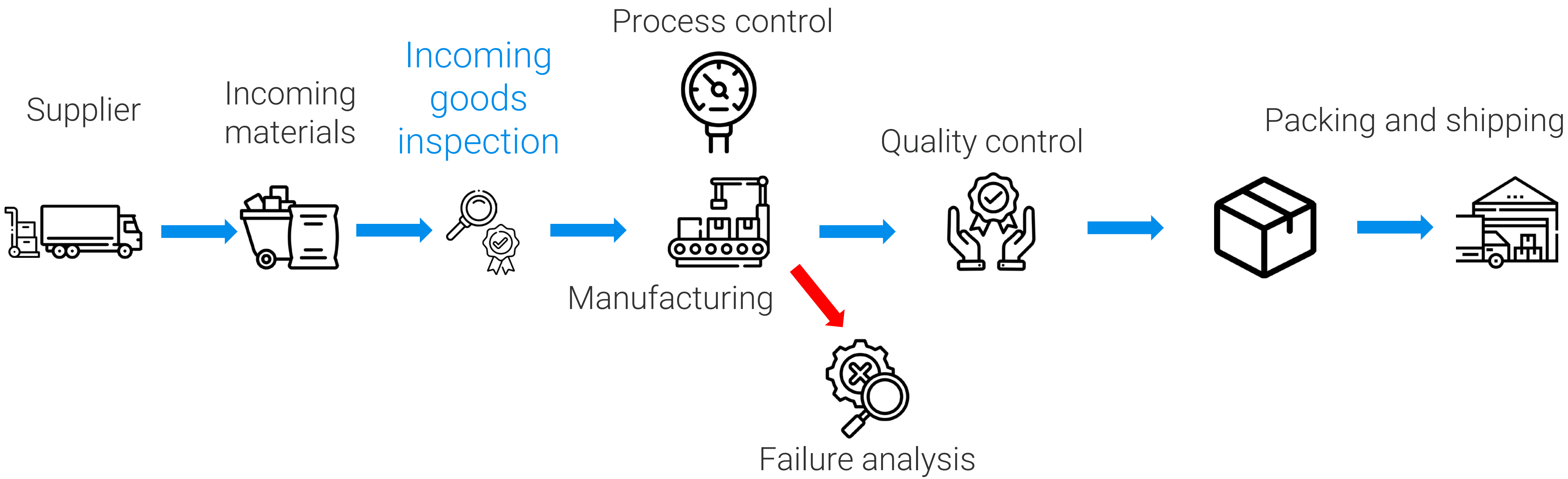








Micro-XRF – Quality control at different stages



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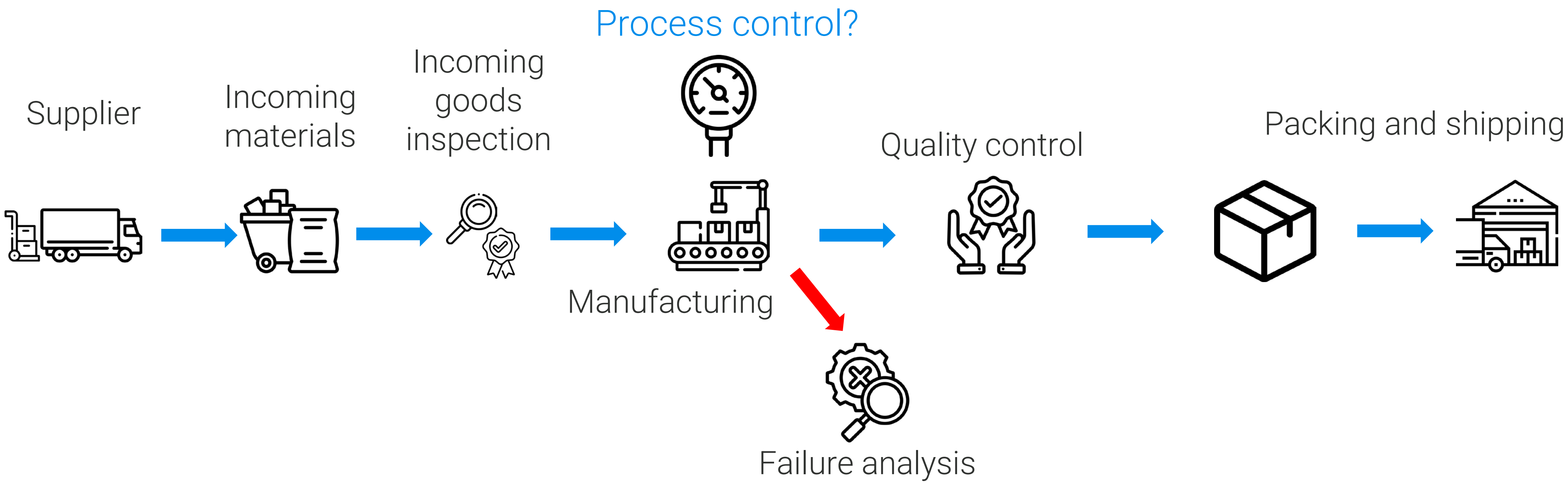
Micro-XRF – Quality control at different stages Process control



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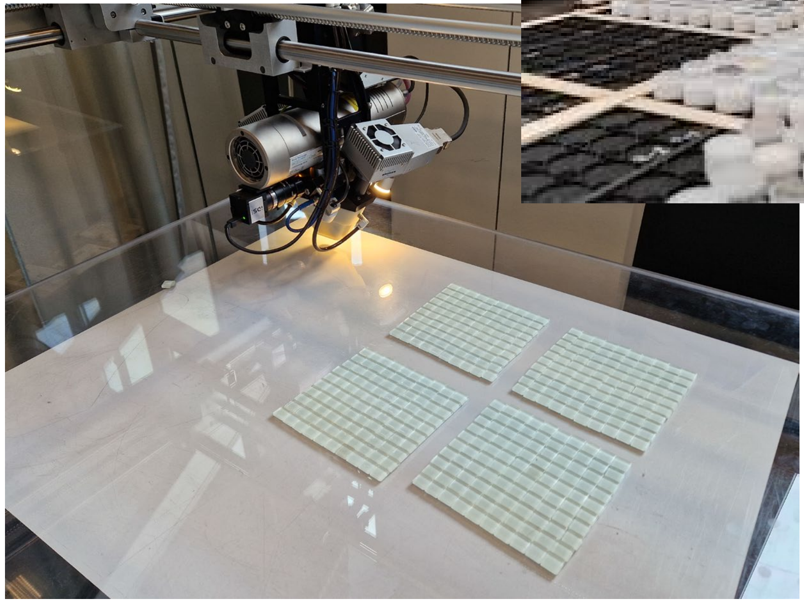
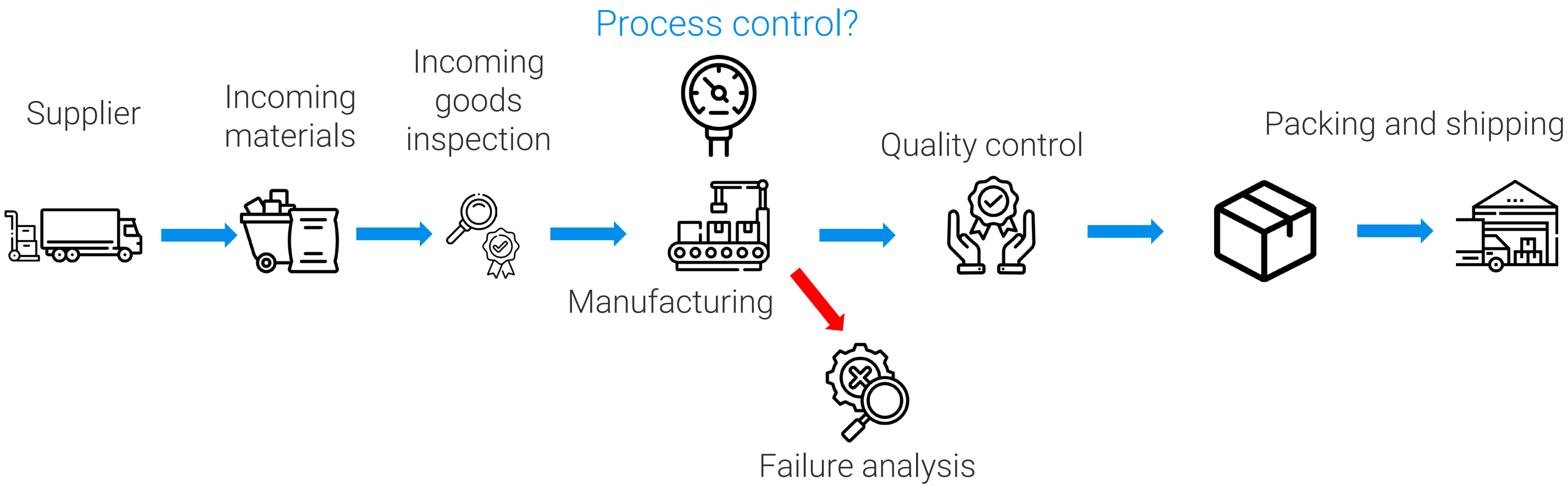
Process control
Is it feasible?

- True on-line analysis with XRF is possible – but with micro-XRF it is ... challenging.
- (semi-)automated point measurements with open-beam instruments are the immediate application.



Micro-XRF – Quality control at different stages



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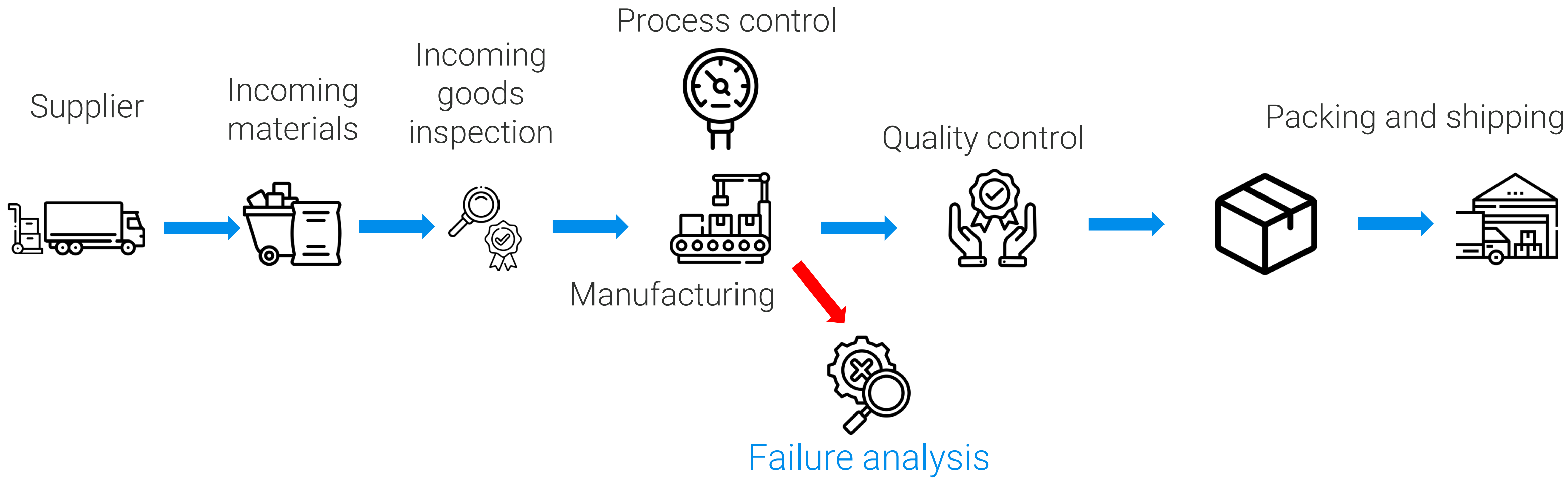
Micro-XRF – Quality control at different stages Failure analysis



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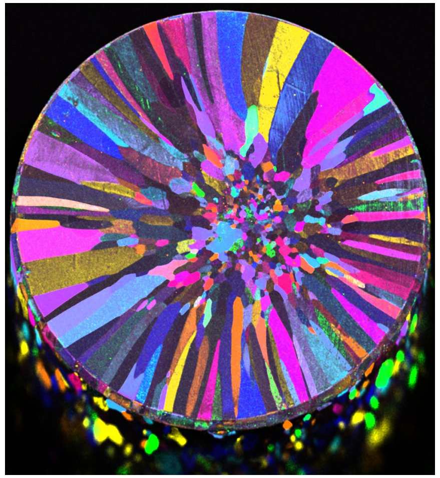
Failure analysis
Example – Stripped threads in aluminium

- Al 6063 is a commonly used aluminium alloy, well suited for aluminium extrusion with smooth surfaces that allow for anodizing.
- In one batch of products, it was not possible to insert the screws as usual because the threads could not withstand the mechanical stresses.
- In aluminium, even more than in other alloys, the mechanical properties are not only a matter of composition but above all of microstructure, which can be finely tuned by heat treatment (temper).

- Sometimes even the crystalline structure can be resolved by micro-XRF.



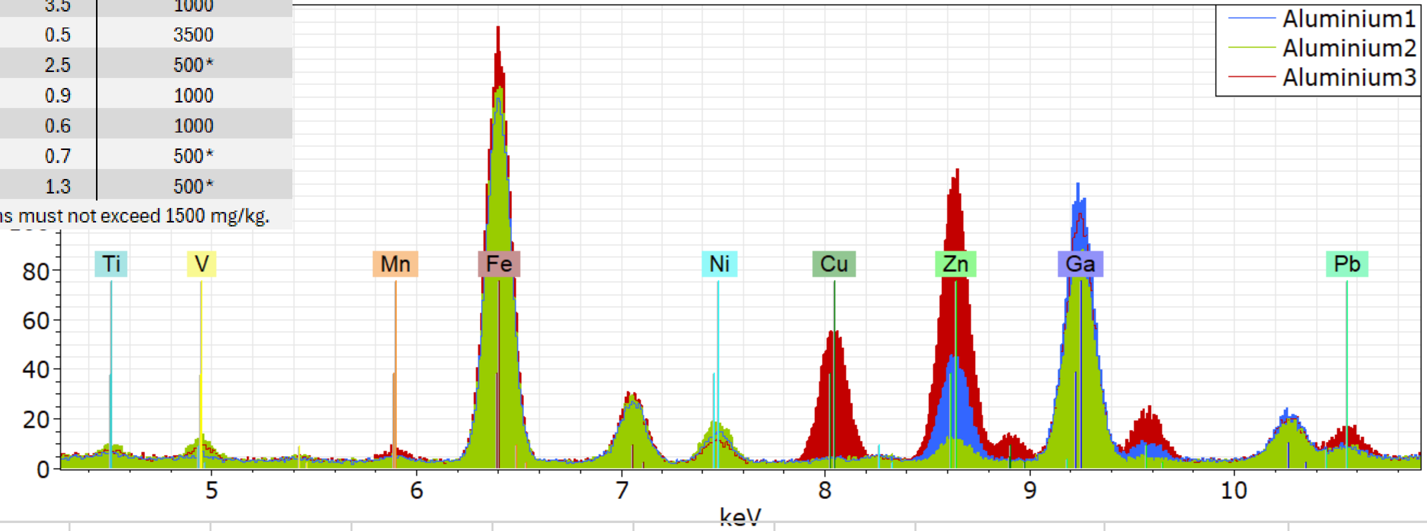
Failure analysis
Alloy element quantification

Element	Aluminium 1 concentration / mg/kg	Aluminium 1 uncertainty / mg/kg	Aluminium 1 uncertainty / %	Aluminium 2 concentration / mg/kg	Aluminium 2 uncertainty / mg/kg	Aluminium 2 uncertainty / %	Aluminium 3 concentration / mg/kg	Aluminium 3 uncertainty / mg/kg	Aluminium 3 uncertainty / %	Al 6063 grade compostion (upper limit) / mg/kg
Ti	**60**	3	5.0	**180**	5	2.9	**60**	3	5.5	1000
V	**20**	1	6.8	**160**	4	2.5	**80**	3	3.8	500*
Mn	**20**	1	4.1	**20**	1	4.2	**40**	1	3.5	1000
Fe	**860**	4	0.5	**890**	5	0.5	**980**	5	0.5	3500
Ni	**30**	1	1.8	**40**	1	1.5	**20**	0.9	2.5	500*
Cu	**1**	0.1	9.1	**2**	0.12	5.9	**100**	1	0.9	1000
Zn	**60**	1	0.9	**10**	0.2	2.0	**150**	1	0.6	1000
Ga	**110**	1	0.6	**90**	1	0.6	**90**	1	0.7	500*
Pb	**5**	0.1	1.9	**5**	0.1	1.9	**15**	0.2	1.3	500*

* - not graded. "Other" elements may be concentrated up to 500 mg/kg, but the sum of their concetrations must not exceed 1500 mg/kg.

- Quantified results

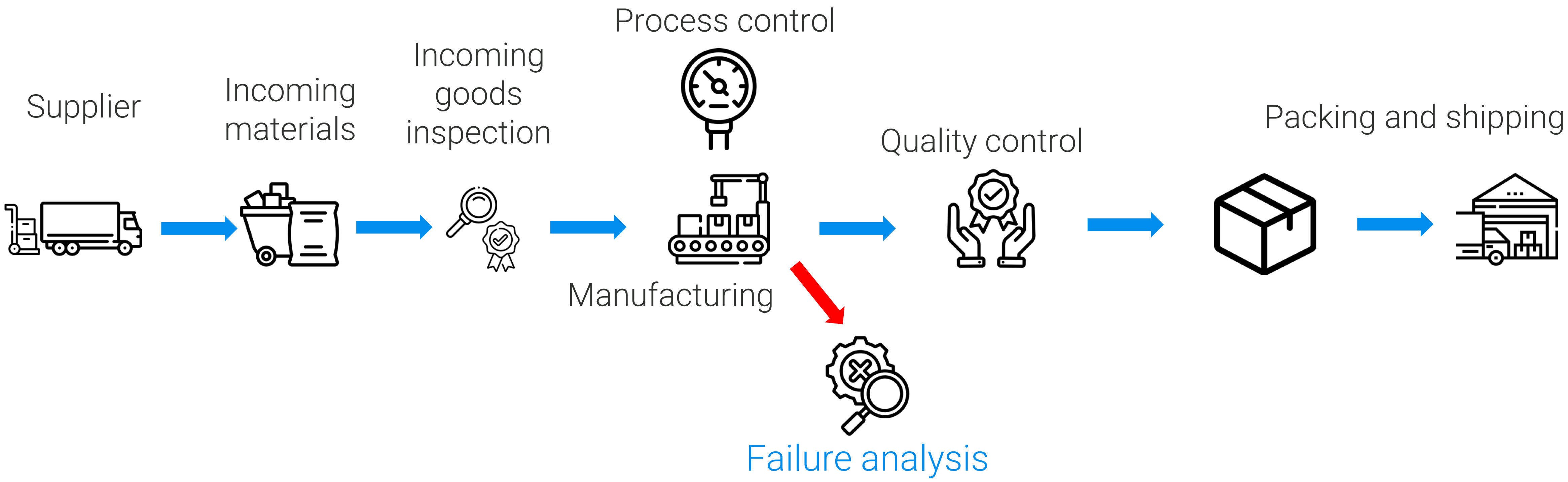
- Spectra of the samples



- Grade limits of selected Al alloys

Aluminium grades	Mg / wt%	Al / wt%	Si / wt%	Ti / wt%	Cr / wt%	Mn / wt%	Fe / wt%	Cu / wt%	Zn / wt%	residuals each	residuals total	Density / g/cm^3	Young's modulus / GPa	Ultimate tensile strength / MPa	Thermal expansion / 10^{-6} K^{-1}
6060	0.35 - 0.5	97.9 - 99.3	0.3 - 0.6	0 - 0.1	0 - 0.05	0 - 0.1	0.1 - 0.3	0 - 0.1	0 - 0.15	0 - 0.05	0 - 0.15	2.71	70	70 - 180	23.4
6063	0.45 - 0.9	97.5 - 99.35	0.2 - 0.6	0 - 0.1	0 - 0.1	0 - 0.1	0 - 0.35	0 - 0.1	0 - 0.1	0 - 0.05	0 - 0.15	2.7	69	120(190*) - 240	23.4
6105	0.45 - 0.8	97.2 - 99.0	0.6 - 1	0 - 0.1	0 - 0.1	0 - 0.1	0 - 0.35	0 - 0.1	0 - 0.1	0 - 0.05	0 - 0.15	2.69	70	120 - 260	21.8
6463	0.45 - 0.9	97.9 - 99.4	0.2 - 0.6			0 - 0.5	0 - 0.15	0 - 0.2	0 - 0.05	0 - 0.05	0 - 0.15	2.69	70	130 - 230	22.1
														*T6 temper	

Micro-XRF – Quality control at different stages



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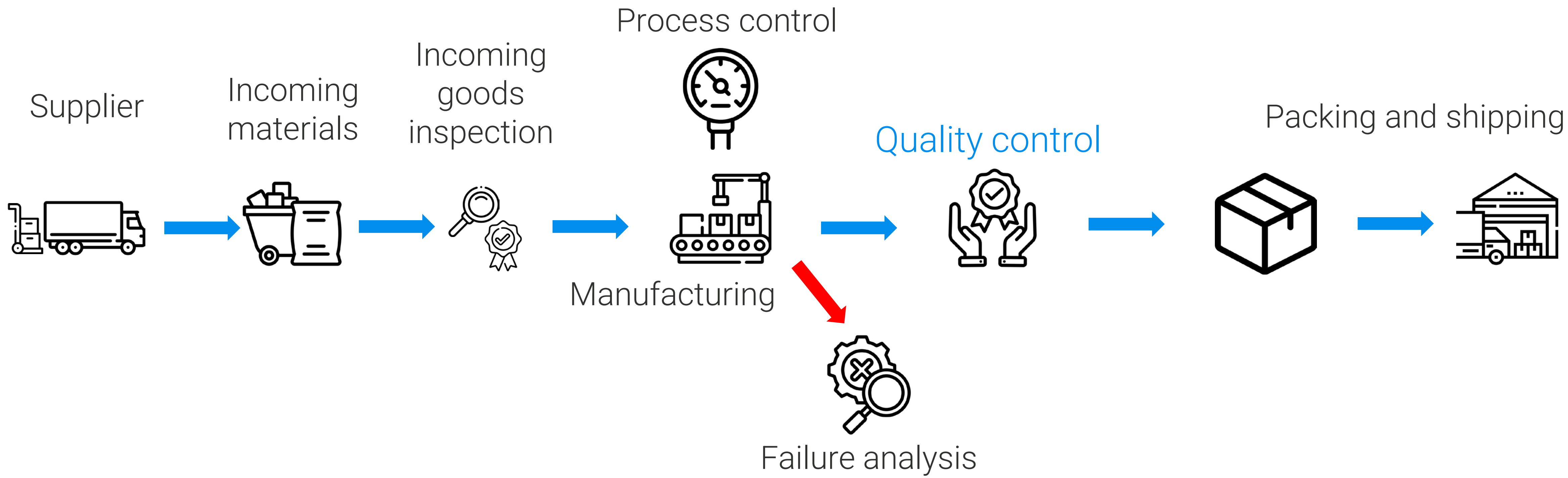
Micro-XRF – Quality control at different stages
Quality control



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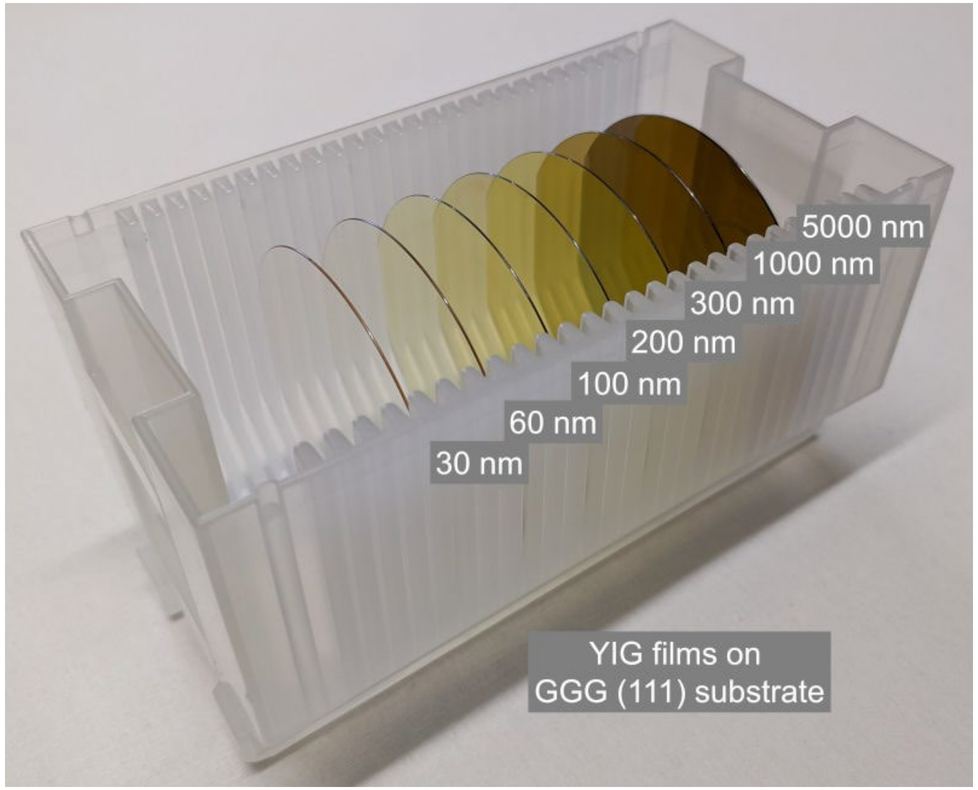
Quality control
YIG layer analysis

- Due to outstanding magnetic properties, Yttrium iron garnet (YIG) thin films grown on a lattice matched substrate (GGG, gadolinium gallium garnet) are increasingly being produced commercially.
- Important properties like magnetic damping factor scale with the thickness of the thin film.

→ Thickness needs to be quantified to gauge the material performance.

→ Micro-XRF may be used for this purpose.

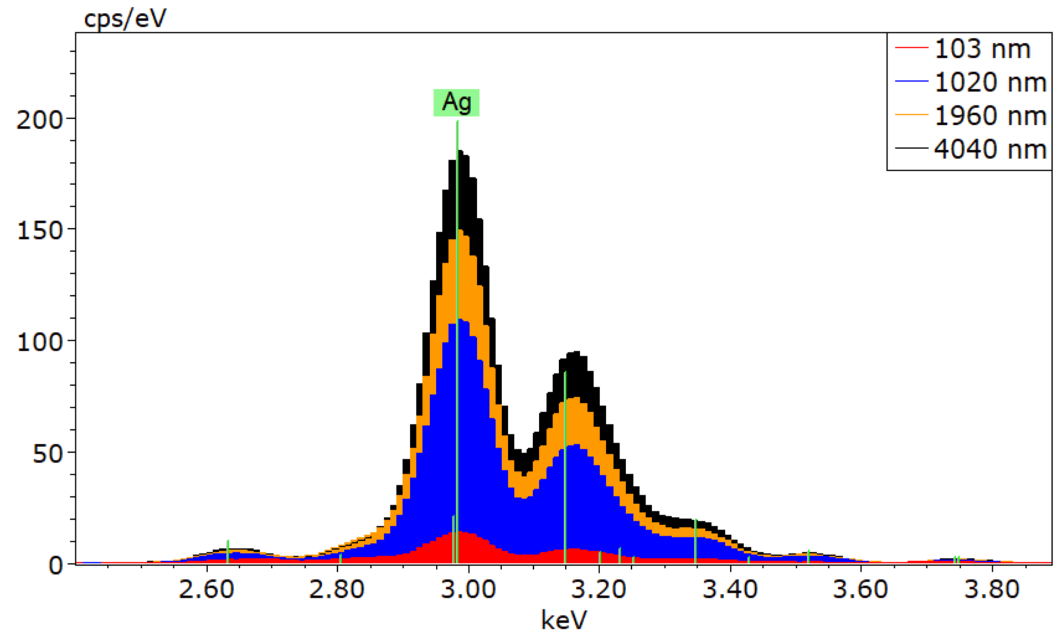


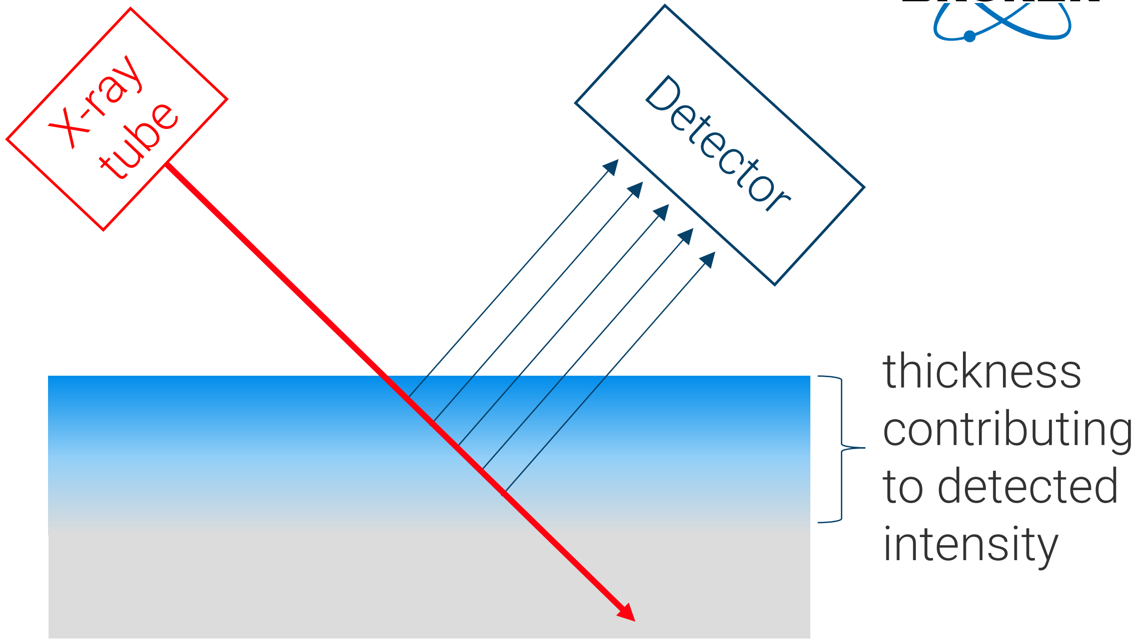
©Innovent e.V. – https://www.innovent-jena.de/mos/yig

Intro
Thickness analysis using micro-XRF

- X-rays can penetrate deeply into many materials.

→ Characteristic X-rays can be generated in varying sample depths.





- X-rays from high depths will be absorbed before reaching the surface.

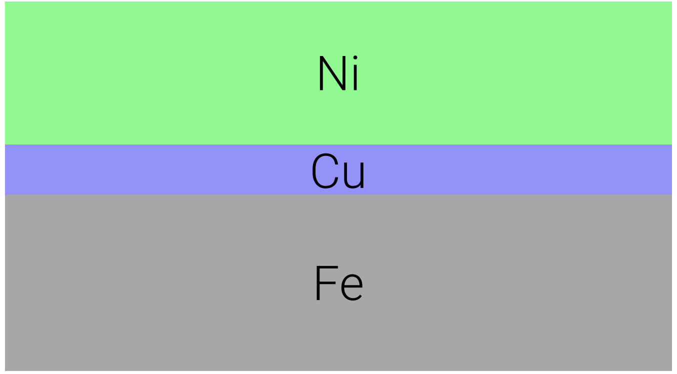
→ Detected intensities will increase with sample thickness up to a maximum.

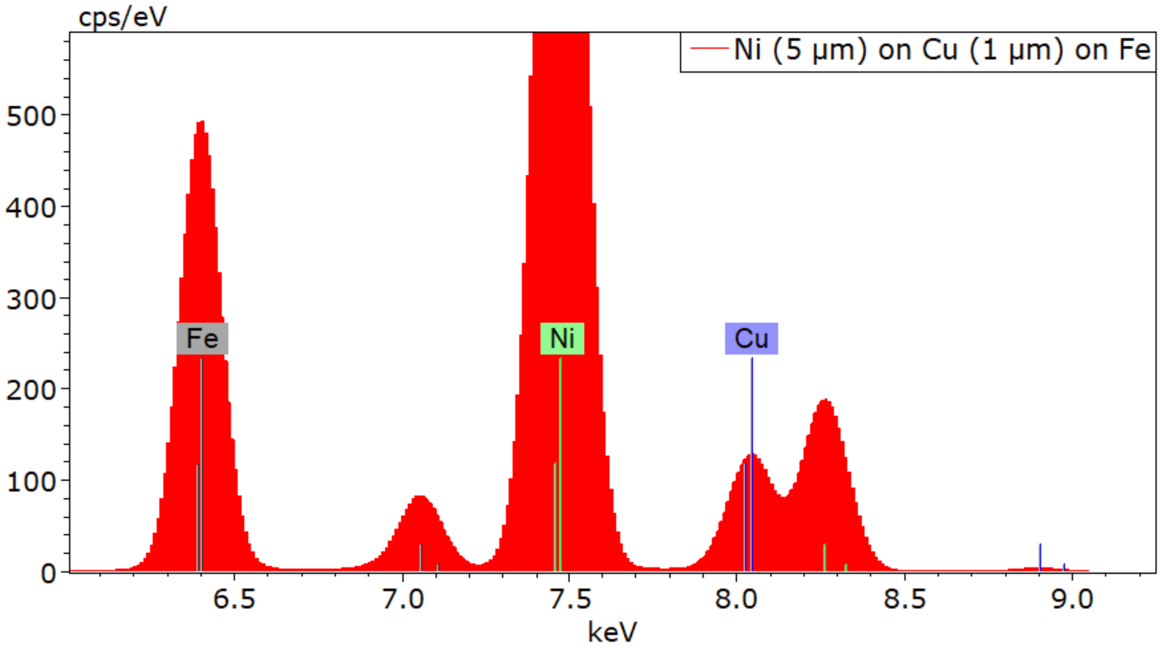
→ **Intensities of characteristic X-rays may be used to deduce layer thicknesses.**

Intro
Thickness analysis using micro-XRF

- Interlayer effects are often important.
- Layer stacking needs to be known.

- Quantification may be done purely based on reference-materials or in a fundamental parameter based approach that may, but must not necessarily be calibrated using reference materials.



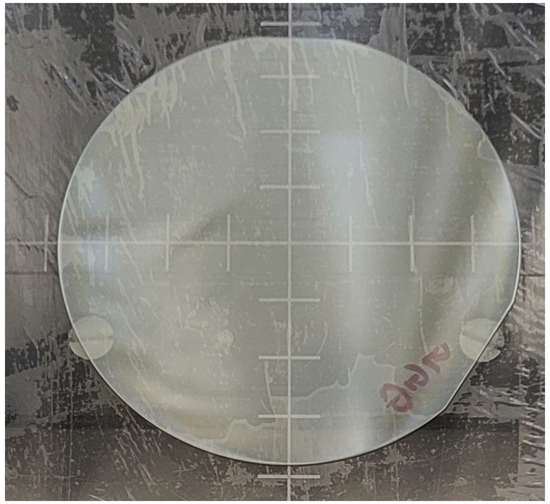


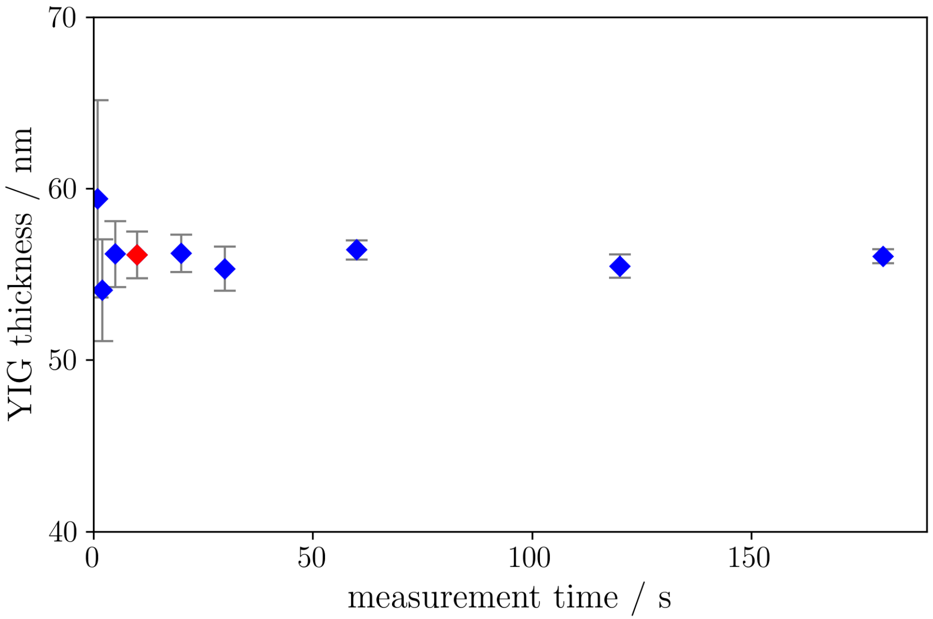
Quality control
YIG layer analysis – Measurement time I

- The objective of quality control might be to map variations in thickness of YIG on the sample surface.

→ Mappings or point analyses on a grid may be performed.

- Measurement time is a primary consideration. It is dictated by the required precision.
- For ~ 50 nm of YIG on GGG, measurements should likely not be less than 10 s (red marking) and anything above 60 s will not improve the results.



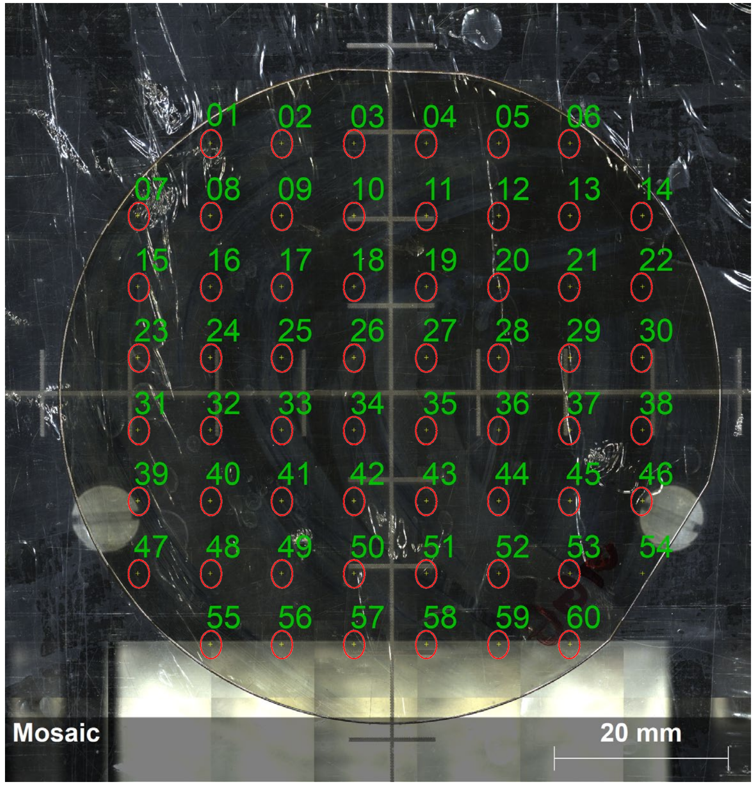


Quality control
YIG layer analysis – Measurement time II

- Given the required measurement times, doing point analyses rather than a mapping is more feasible.
- Decisions must be made regarding the number of points and time per point and will depend on what is required for the user.

Three options were explored:

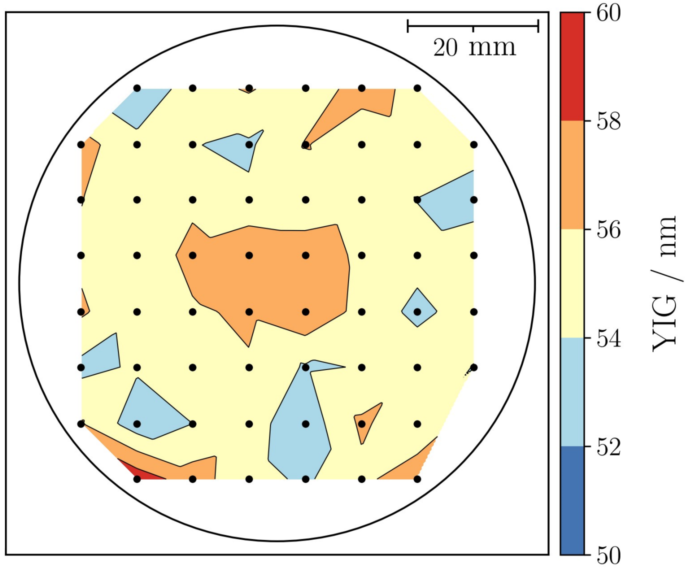
1. 60 measurements on a grid with 60 s per spot (shown on right).
2. 60 measurements on a grid with 30 s per spot if lower precision is acceptable.
3. 120 measurements on a grid with 60 s per spot if a higher measurement time is acceptable.



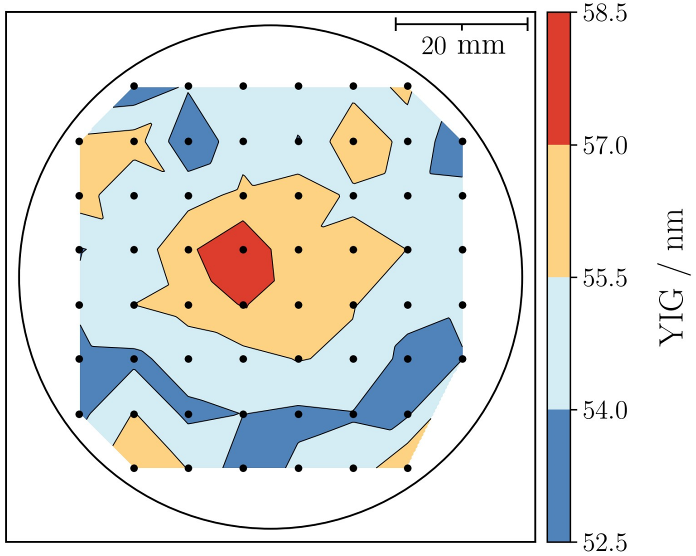
- To ensure that very localized variations are less reflected in the data, a 2000 µm collimator was used.

Quality control
YIG layer analysis – Layer thickness distribution

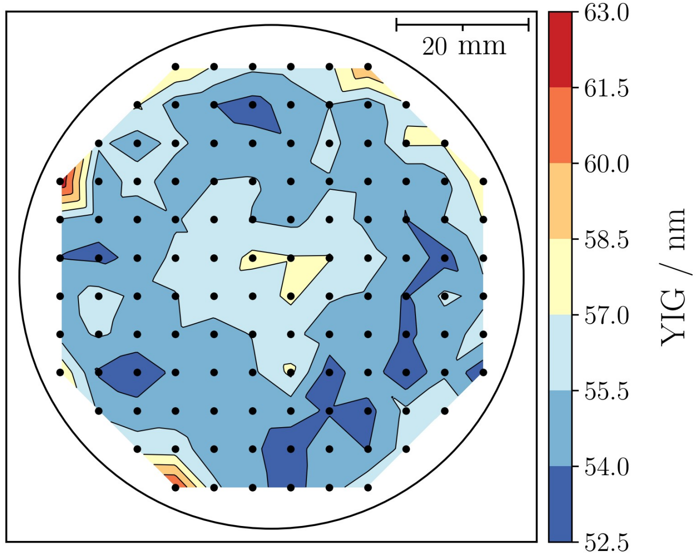
overall time: ~ 38 min



overall time: ~ 68 min



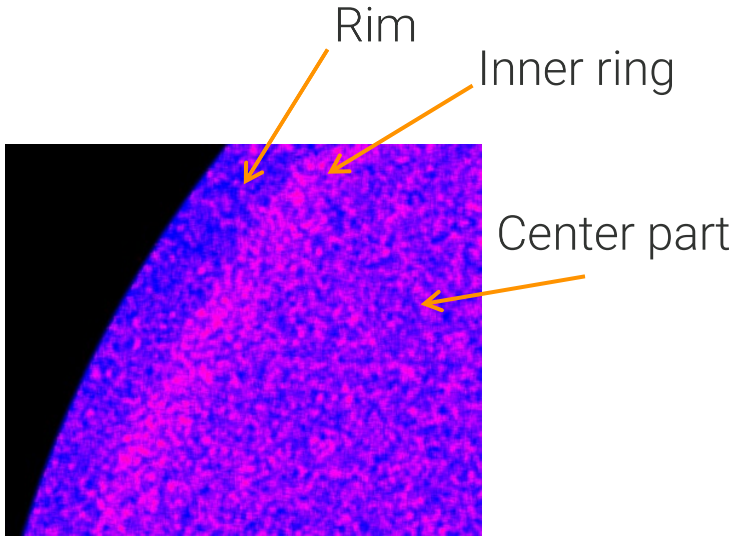
overall time: ~ 137 min



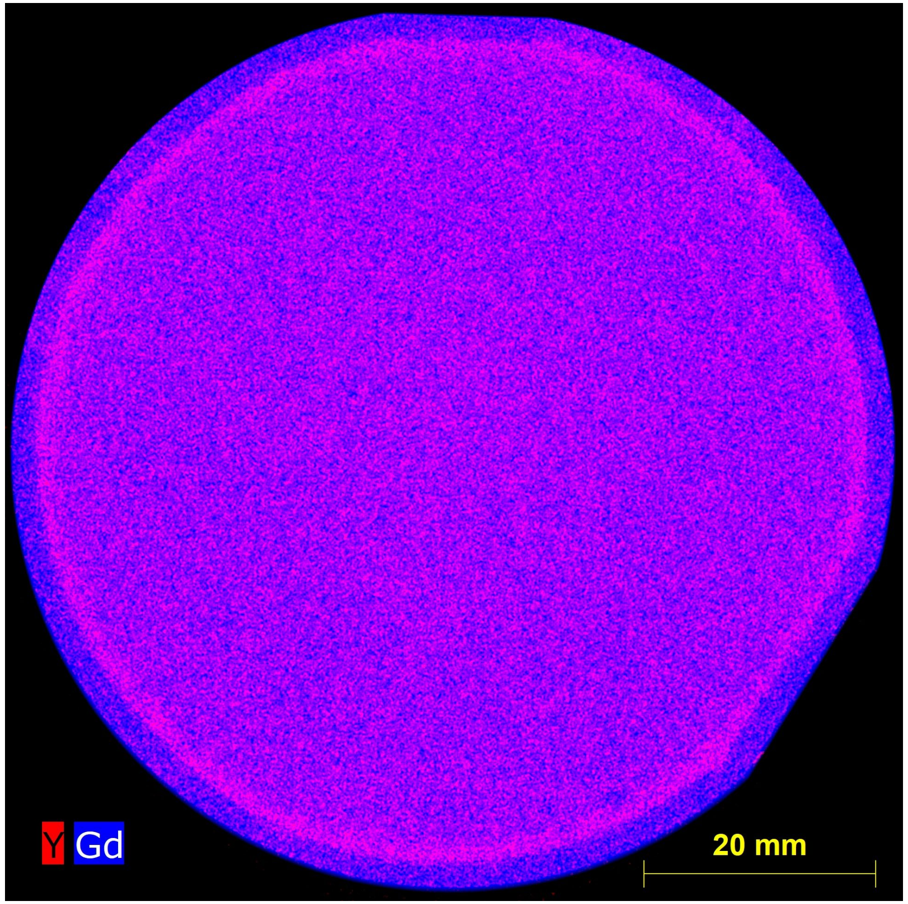
- All measurements show an increased thickness at the center.
- Denser map highlights often-times increasing thickness at the outside.
- Thicknesses were derived without introducing reference materials. YIG thickness according to XRR: 53 nm.

Quality control
YIG layer analysis – Rim structure

- A map with a polycapillary optic reveals the origin of the anomalies close to the edge.
- There is an outer ring with ~ 2.3 mm width, where very little YIG was deposited, followed by an ~1.7 mm inner ring with increased loading.
- Depending on which part of this ring structure at the edge was hit by the larger X-ray spots, either an increased or decreased thickness of YIG was quantified.

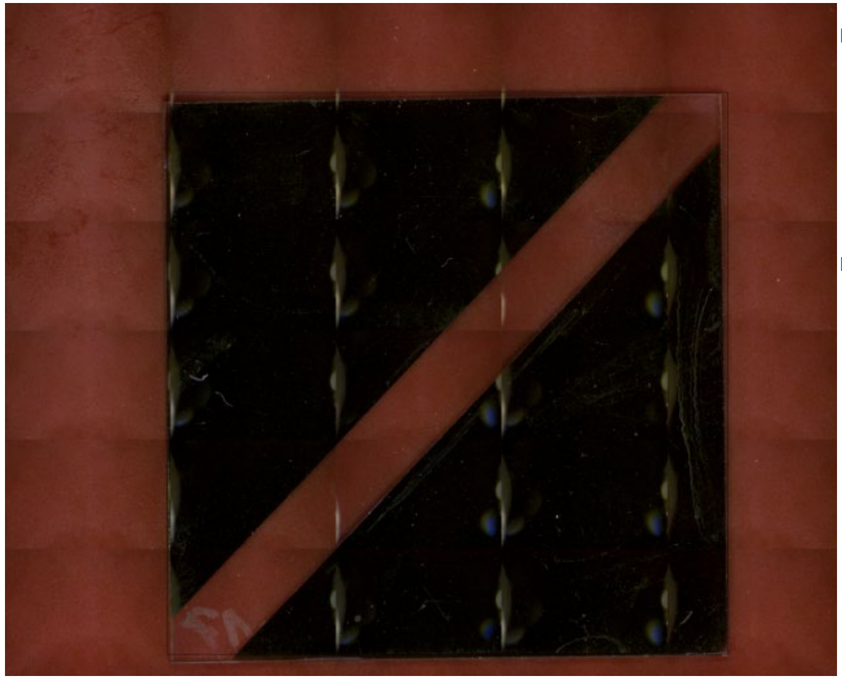


overall time: ~ 14 h 30 min



Quality control
Components for PEC cells

BRUKER



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

- Photoelectrochemical (PEC) cells can produce hydrogen from water when exposed to sunlight.
- The efficiency of the process, as well as the stress due to corrosion depend strongly on a finely tuned set of technical parameters – layer thickness and composition is one of them.

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

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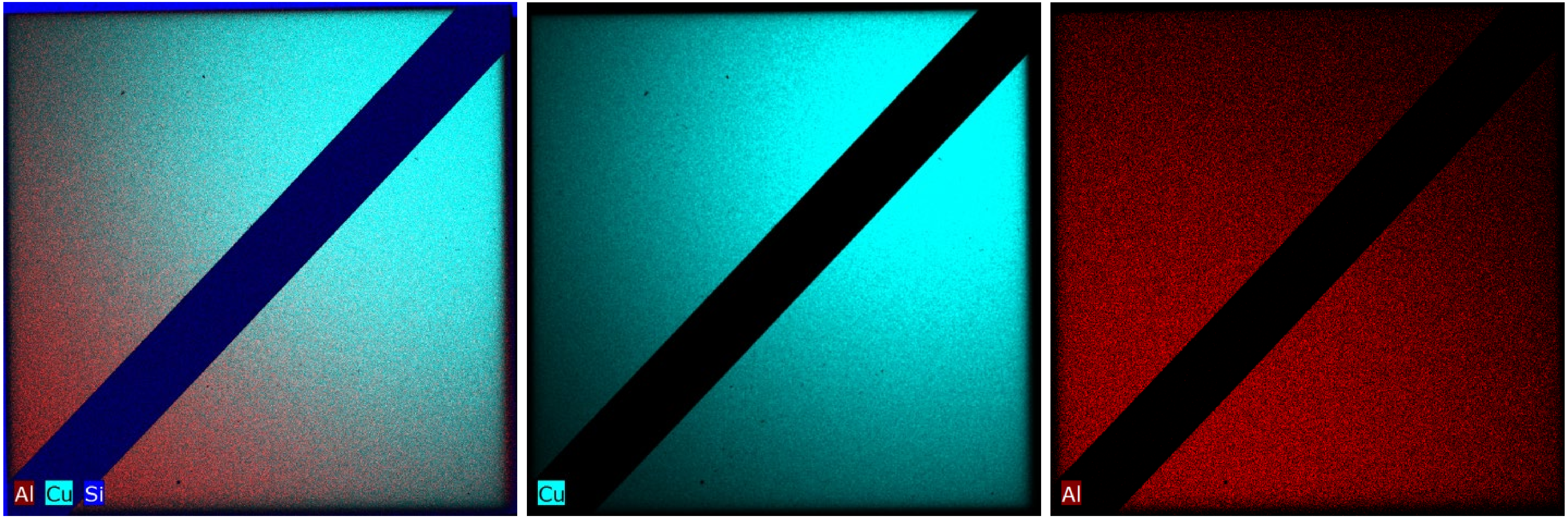
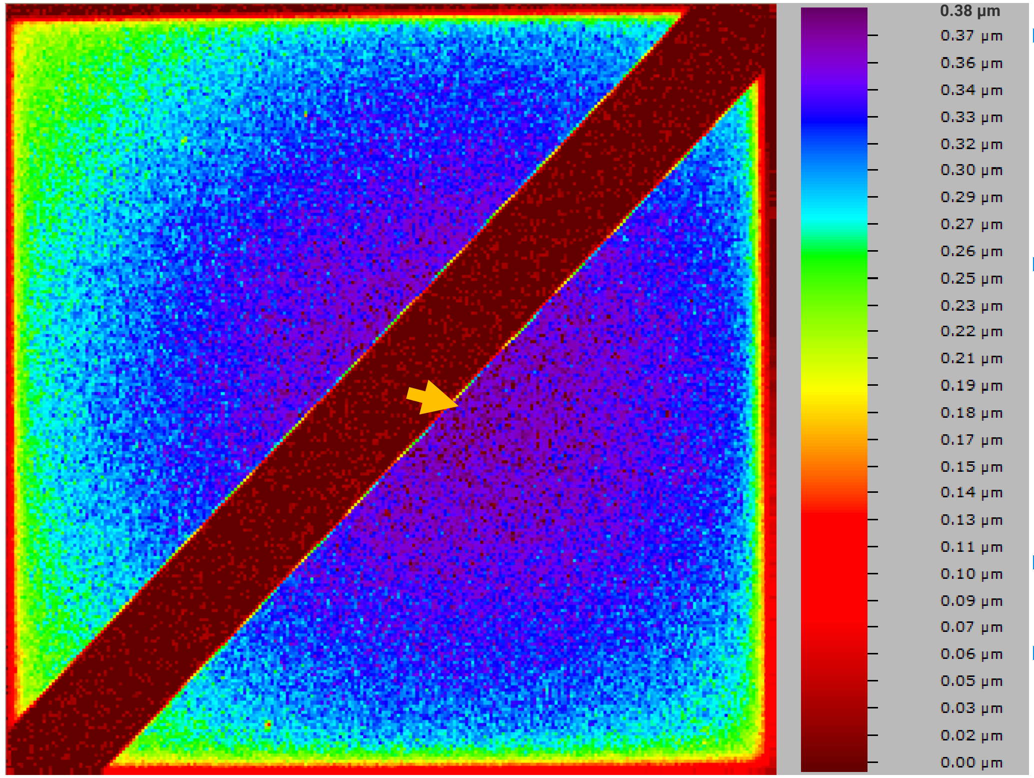
Quality control
Components for PEC cells – Qualitative analysis



- Cu/Al-layer on a glass substrate: the concentration gradient is clearly visible

Quality control
Components for PEC cells – Layer thickness quantification



- Knowing the structure of the sample, a method for layer thickness analysis can be set up quickly.
- A full quantification of the map data (here with 5x5 binning) shows the layer **thickness** distribution with high spatial resolution (250 µm per pixel).
- ... but it also takes time.
- The radially symmetric distribution pattern is visibly offset from the center.

Structure

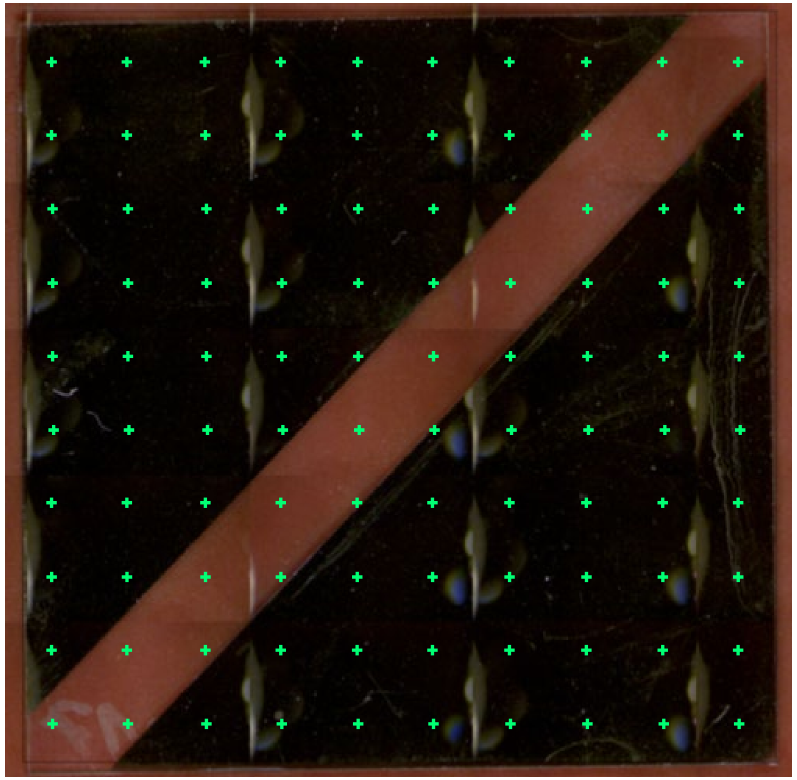
Layer	Chemical elements
layer1	Cu Al
base	Si

Measure conditions

Parameter	Value
HV / kV	50
Current / µA	200
Collimator	25 µm LENS
Atmosphere	Vacuum

- Note: for sample systems like this there are no certified reference materials.
  → the quantification is based on fundamental parameters and known physics (and can be supported by samples of trusted composition).

Quality control
Components for PEC cells – Quantitative point analysis



- 10x10 grid.
- Measurement time per point is 10 s.

- When the expected variations are on a millimeter-scale, there is no need to measure with micrometer resolution.
- To save measurement time, an array of points can be set and measured.
- Point spectra can be quantified using the same layer analysis method.
- The results (layer thicknesses and composition) are shown in tabular from and can be exported to text files.
- This allows, for example, to visualize the result of the element and layer thickness distribution.

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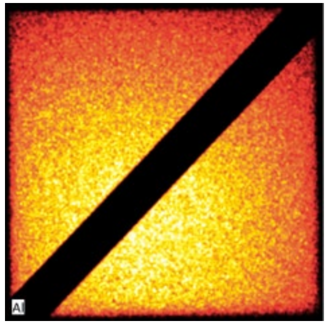
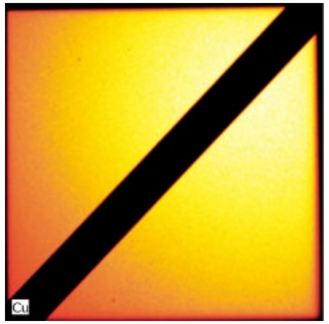
Quality control
Components for PEC cells – Comparison of results



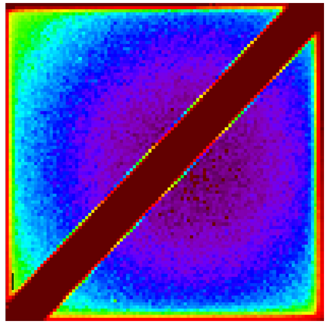
22.8	21.0	20.7	18.7	17.6	16.8	15.5	14.4	0.0	0.0
23.4	22.6	21.4	19.6	19.9	17.7	17.4	0.0	0.0	13.7
24.6	23.1	22.3	21.4	20.0	19.2	0.0	0.0	14.8	14.9
25.6	25.6	22.5	21.9	21.7	20.0	0.0	17.8	16.4	16.0
27.1	27.0	25.3	24.2	23.6	0.0	20.4	19.6	18.4	17.0
28.7	27.4	27.3	25.5	0.0	23.6	23.0	20.5	19.5	17.8
30.3	29.6	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 (33:67) is in the bottom-left corner.



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 (86:14) in the top-right 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)

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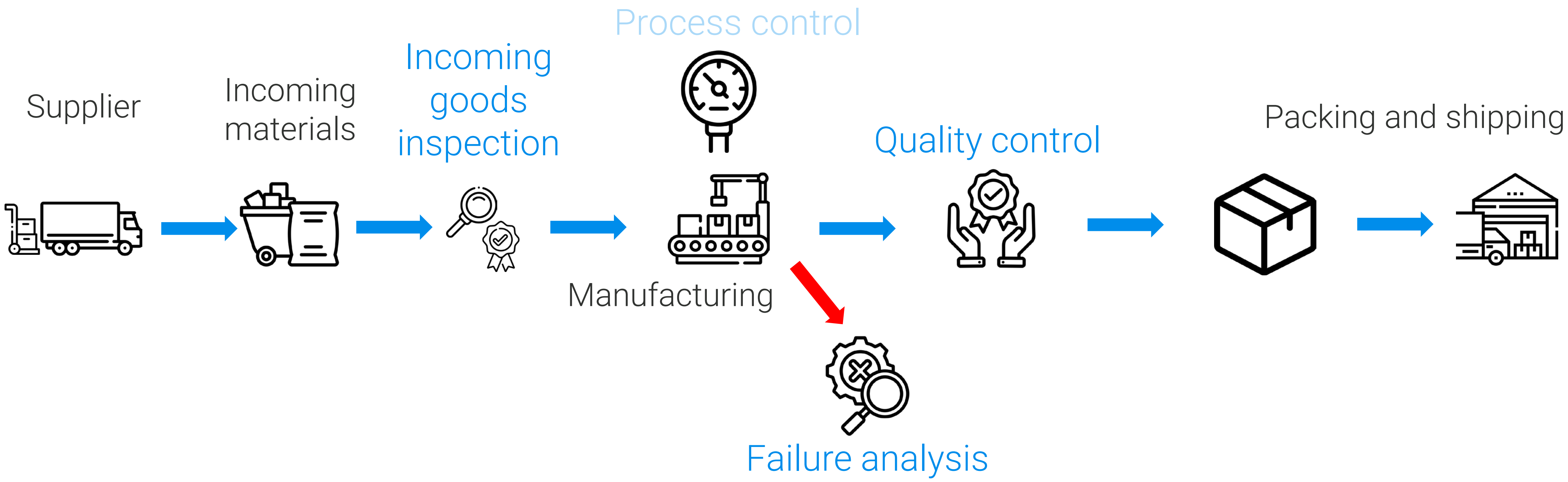
Summary



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Thank you!

Are there any questions?

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