

## Practical Spectrum Imaging: Rapid Collection for Routine Analysis

# Welcome

## Today's Topics

- Introduction
- X-ray Detectors for Microanalysis
- HyperMap Applications
- $\mu$ XRF Instrument Design - ARTAX
- ARTAX Element Mapping in Forensics and Industry
- Q&A



Don Becker  
Webinar Host

## Guest Speakers

- Ted Juzwak – Applications Lab Manager, Ewing, NJ
- Armin Gross – International Sales Manager, Berlin, Germany

# Bruker Companies

## Bruker BioSciences Corporation

(NASDAQ: BRKR)

### Bruker AXS

Advanced X-ray Solutions

XRF, Diffraction, Single Crystal, Microanalysis



S4 Pioneer



D8 Discover



Recombinase Fragment with  
3 DNA Strand – Dr. Yu Lau,  
University of Saskatchewan



XFlash 4010

### Bruker Daltonics

Mass Spectrometry



The innovative ultraflex™ III mass spectrometer

### Bruker Optics

FTIR, NIR, Raman



SENSOR 27 & HYPERION  
FT-IR Microscope

### Bruker BioSpin

(Affiliated privately held not part of Bruker BioSciences)

NMR, EPR, MRI



AVANCE III

# Bruker AXS Microanalysis



**Bruker AXS**

**Microanalysis**

**Est. 2006**



**Est. 1965**



**Est. 1991**

# Microanalysis **QUANTAX with XFlash®**



**Ted Juzwak**  
**Bruker AXS Microanalysis USA**

# X-ray Detectors for Microanalysis

## UHV Dewar Si(Li) Detector



- Conventional EDS Detector
- Cooling with LN<sub>2</sub> (ca. -190°C)
- 2 hours to cool down
- Technology from 1960's
- Weight approx. 8 kg

## XFlash<sup>®</sup> Detector



- SDD Technology
- no LN<sub>2</sub> (Peltier cooling, ca. -25°C)
- 30 seconds to cool down
- Up to 10 times faster
- High efficiency
- Stable resolution, reliable quantification
- Weight 2.5 kg

# History of the SDD

## 4<sup>th</sup> generation



XFlash<sup>®</sup>4010  
July 2006

## 3<sup>rd</sup> generation



XFlash<sup>®</sup>3001  
2002

## 2<sup>nd</sup> generation



XFlash<sup>®</sup>2000  
2000

## 1<sup>st</sup> generation



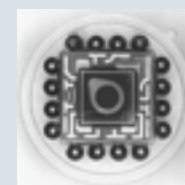
XFlash<sup>®</sup>1000  
1997

2007: 10th anniversary  
of the XFlash<sup>®</sup> Detector!

# XFlash<sup>®</sup> Detector 4010

## ■ 4<sup>th</sup> generation high resolution detector

- 10 mm<sup>2</sup> active area
- Energy resolution:  $\leq 125$  eV, specification at MnK $\alpha$ , 100 000 cps
- Detection of beryllium (4)
- Input count-rate:  $> 700,000$  cps
- 2-stage Peltier cooler
- Vibration-free
- Maintenance-free

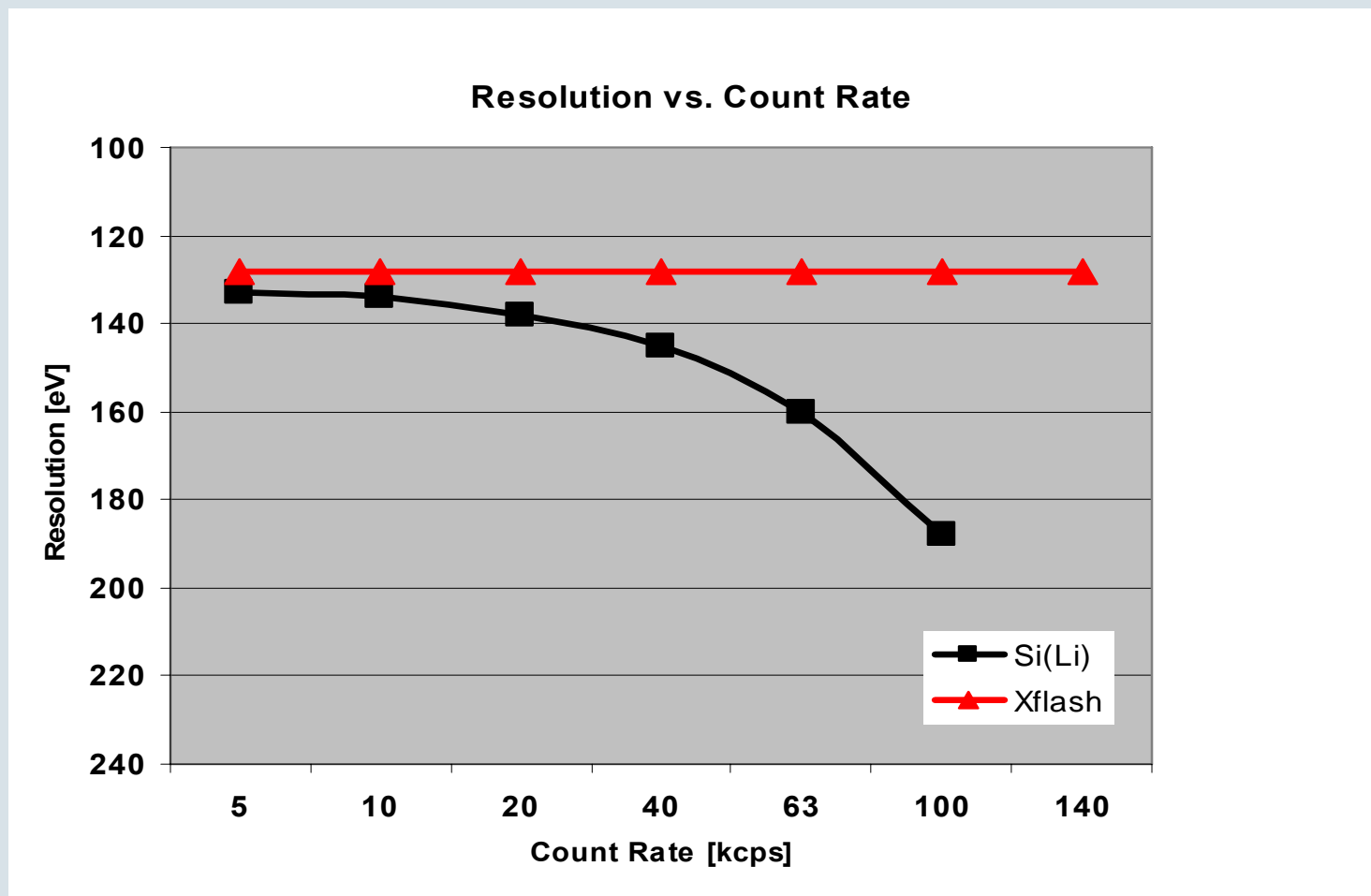


# 129eV Resolution at Mn: XFlash vs Si(Li)

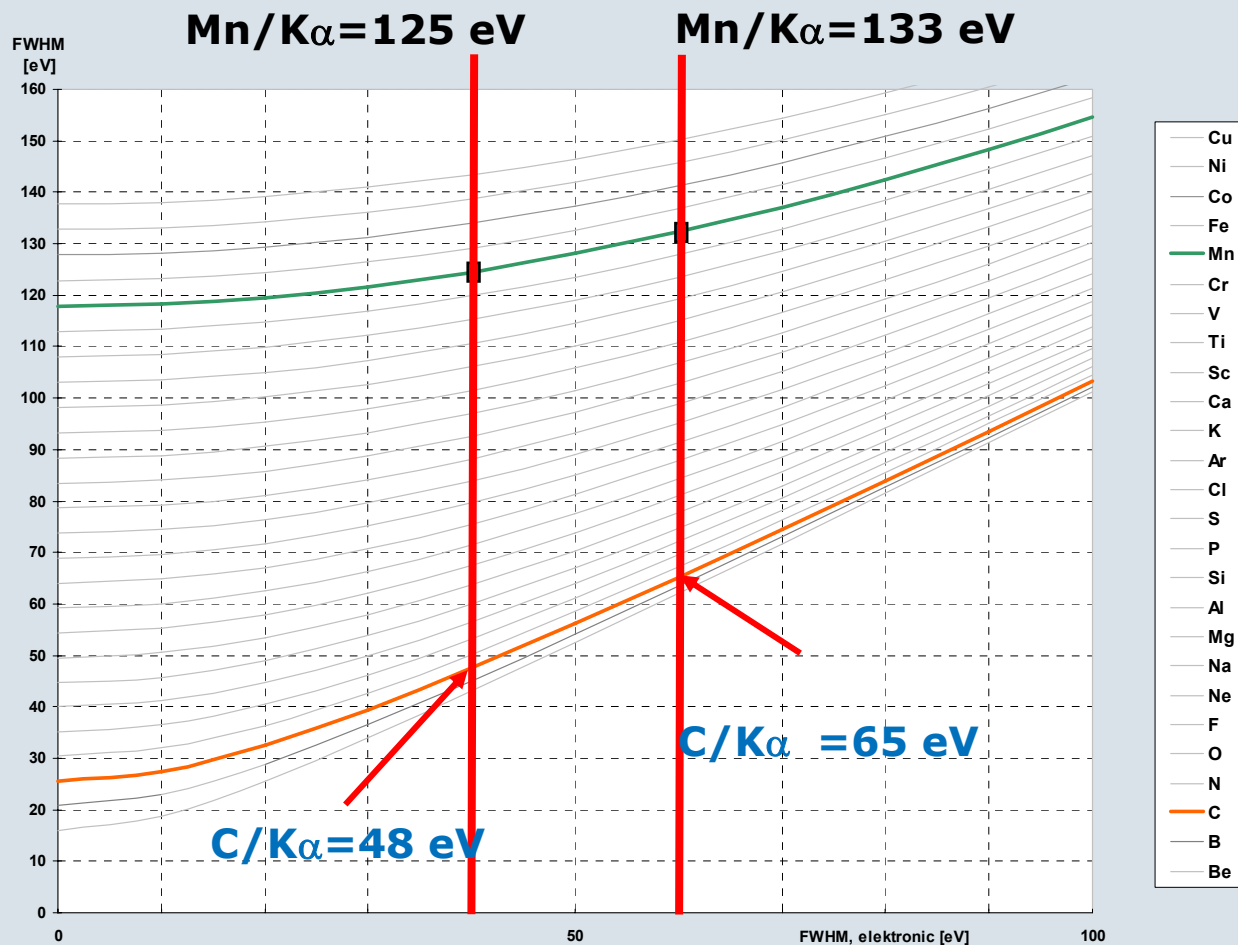
<u>Count Rate (Kcps)</u>	<u>Si(Li)</u>	<u>XFlash®</u>
■ 0-2	129	129
■ 2-8	131	129
■ 8-14	133	129
■ 14-20	138	129
■ 20-42	149	129
■ 42-62	161	129
■ 62-110	191	129
■ 110-150	NP	129
■ 150-200	NP	130
■ 200-300	NP	131

# XFlash 4010 SDD

## Resolution vs Count Rate



# Theoretical energy resolution of $K\alpha$ lines for Si-based detectors

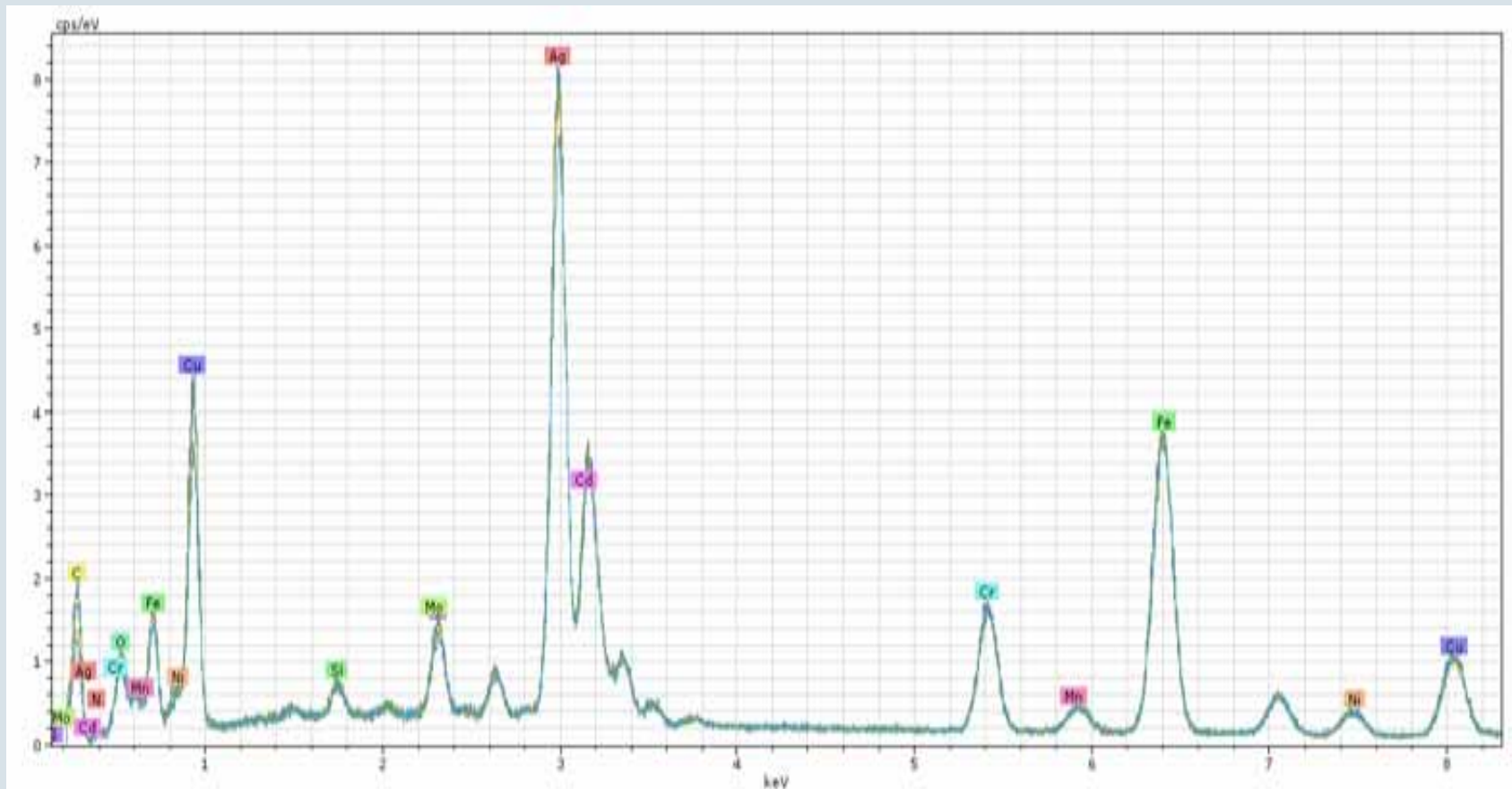


-8 eV at  
Mn/ $K\alpha$

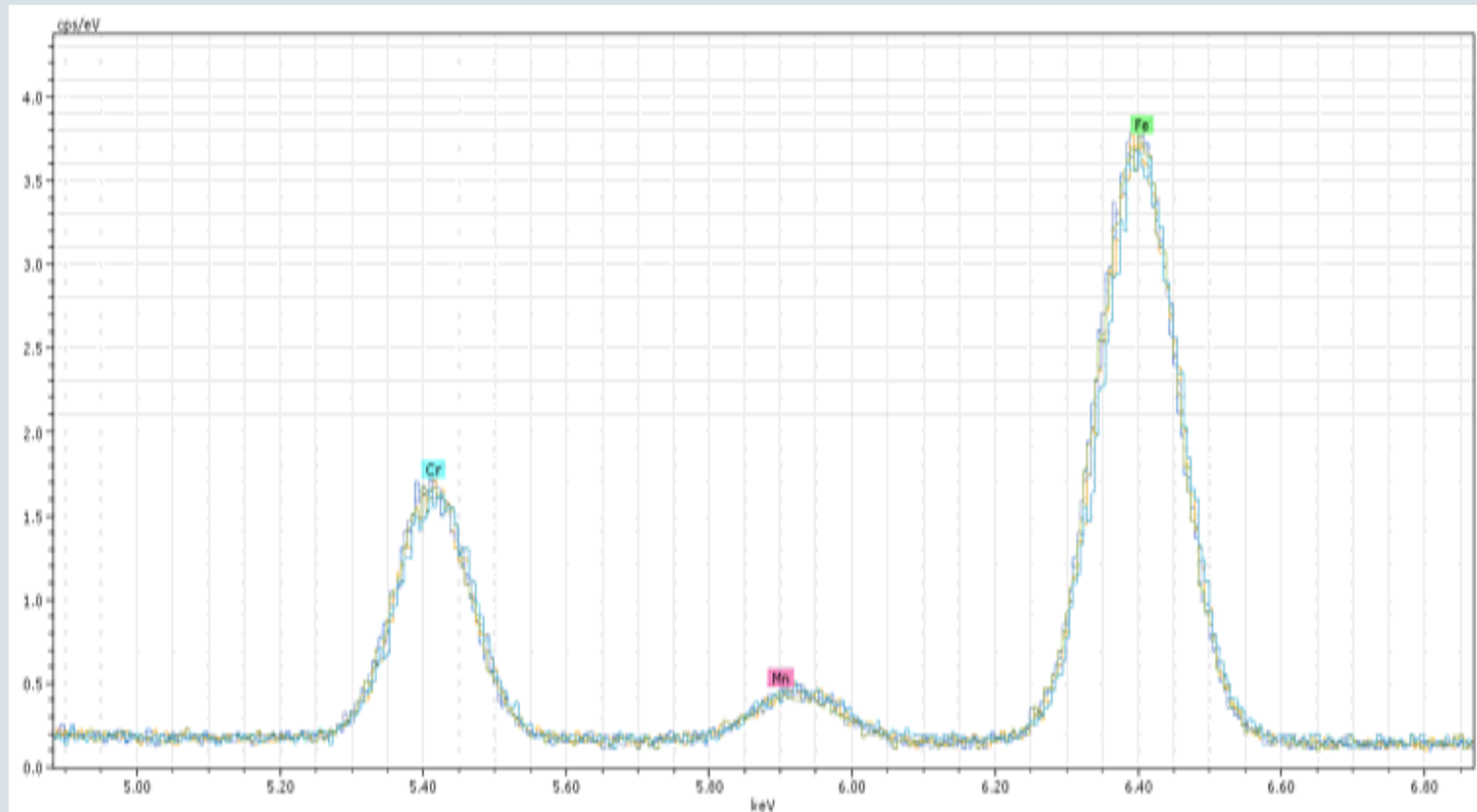


-17 eV at  
C/ $K\alpha$

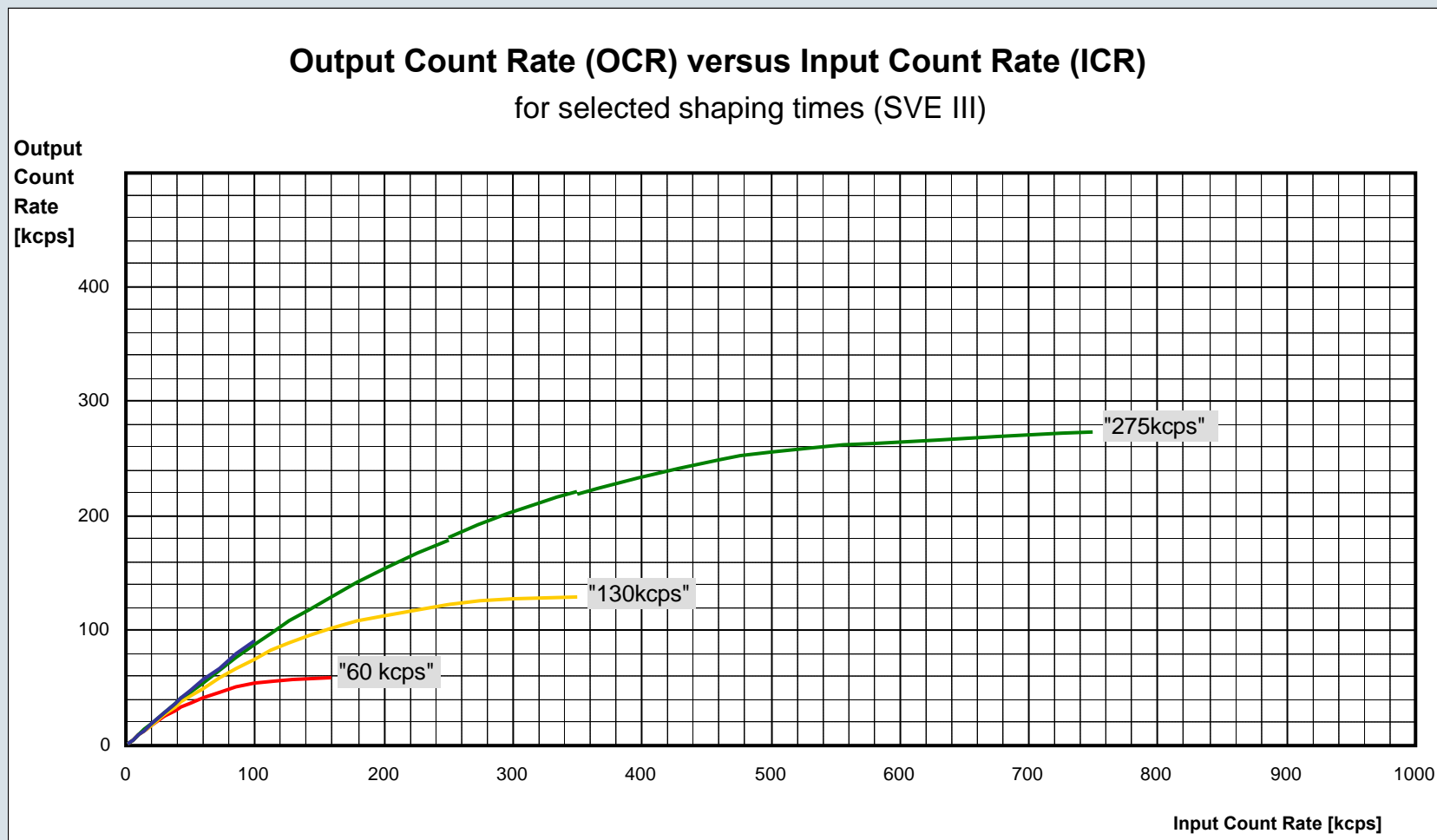
# XFlash SDD at 5K, 10K, 20K, 50K, 100K cps



5K, 10K, 20K, 50K, 100K cps –  
*No peak shift with count rate!*



# Output Count Rate vs Input Count Rate



# XFlash<sup>®</sup> Family Of Detectors



**XFlash 4010: 10mm<sup>2</sup>, 125eV, Be Detection**  
**XFlash 4030: 30mm<sup>2</sup>, 133eV, B Detection**



**XFlash Multi- Detectors**



**XFlash Quad 4040: 4 x 10mm<sup>2</sup>, 125eV, Be Detection**

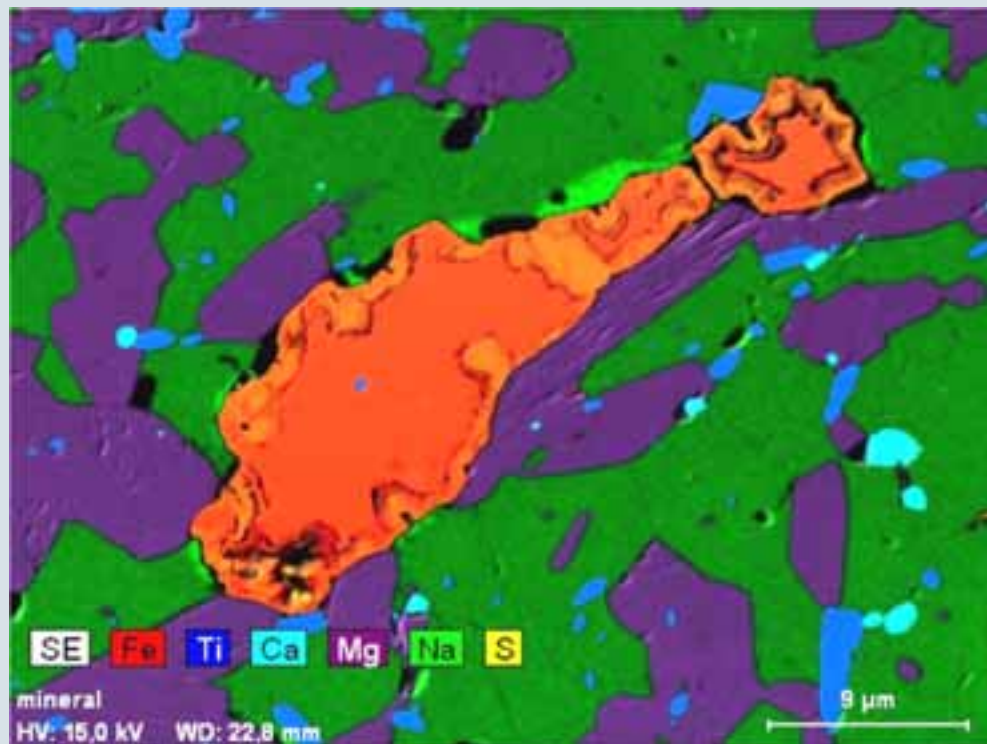
# Multiple Detector Systems



**Internal view chamber**

# Comparison: XFlash<sup>®</sup> with Si(Li)

- Si(Li) detector (30 mm<sup>2</sup>)
  - 138 eV resolution
  - 20,000 cps input
  - 60% dead time
  - **Duration: 6.25 hours**
  
- XFlash<sup>®</sup> QUAD 4040
  - 138 eV resolution
  - 720,000 cps input
  - 40% dead time
  - **Duration: 7 minutes**



Mineralogical sample, 15 keV, 600 x 450 Pixel

The 30 mm<sup>2</sup> Si(Li) needs 50x longer for the same result!

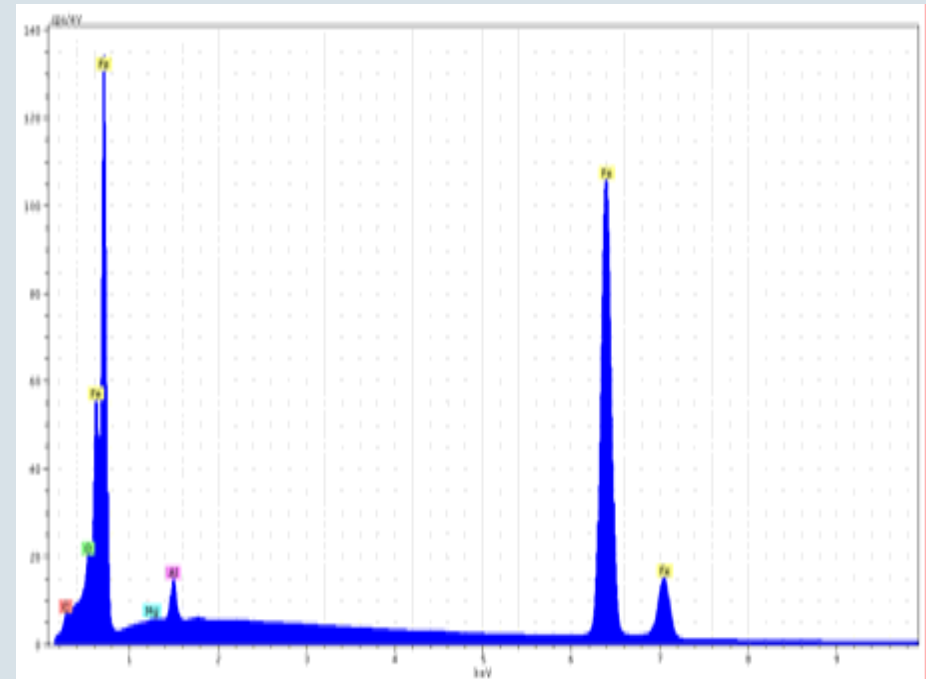
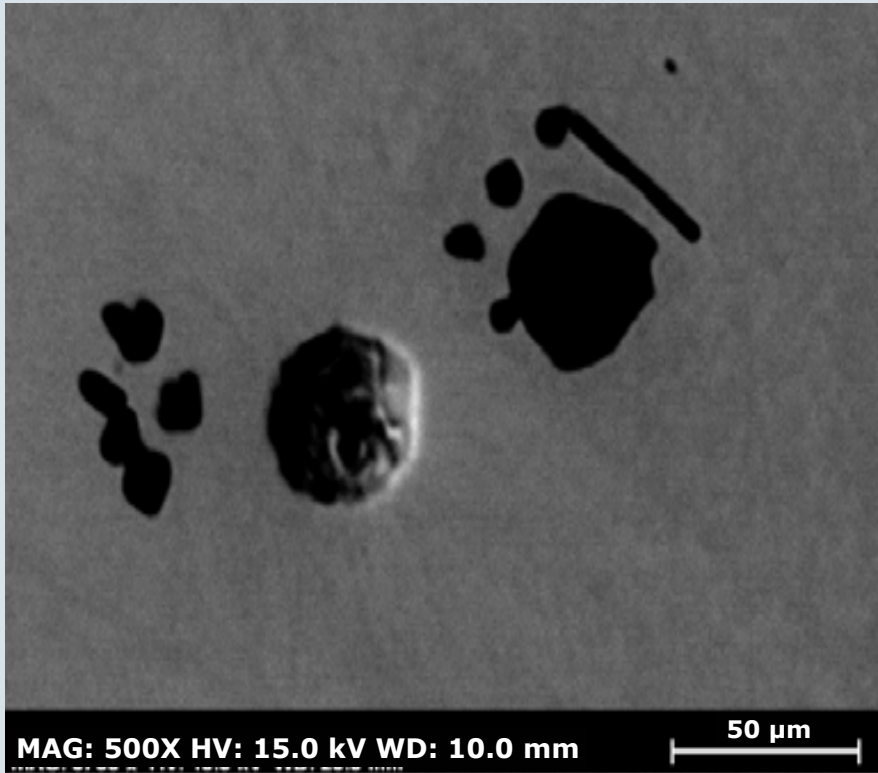
**Note: For an XFlash 4030 a 30mm<sup>2</sup> SD, Duration = ~ 8.75 minutes  
and for an XFlash 4010 a 10mm<sup>2</sup> SD, Duration = ~ 26.25 minutes**

# SD Detectors have become the EDS detectors of choice



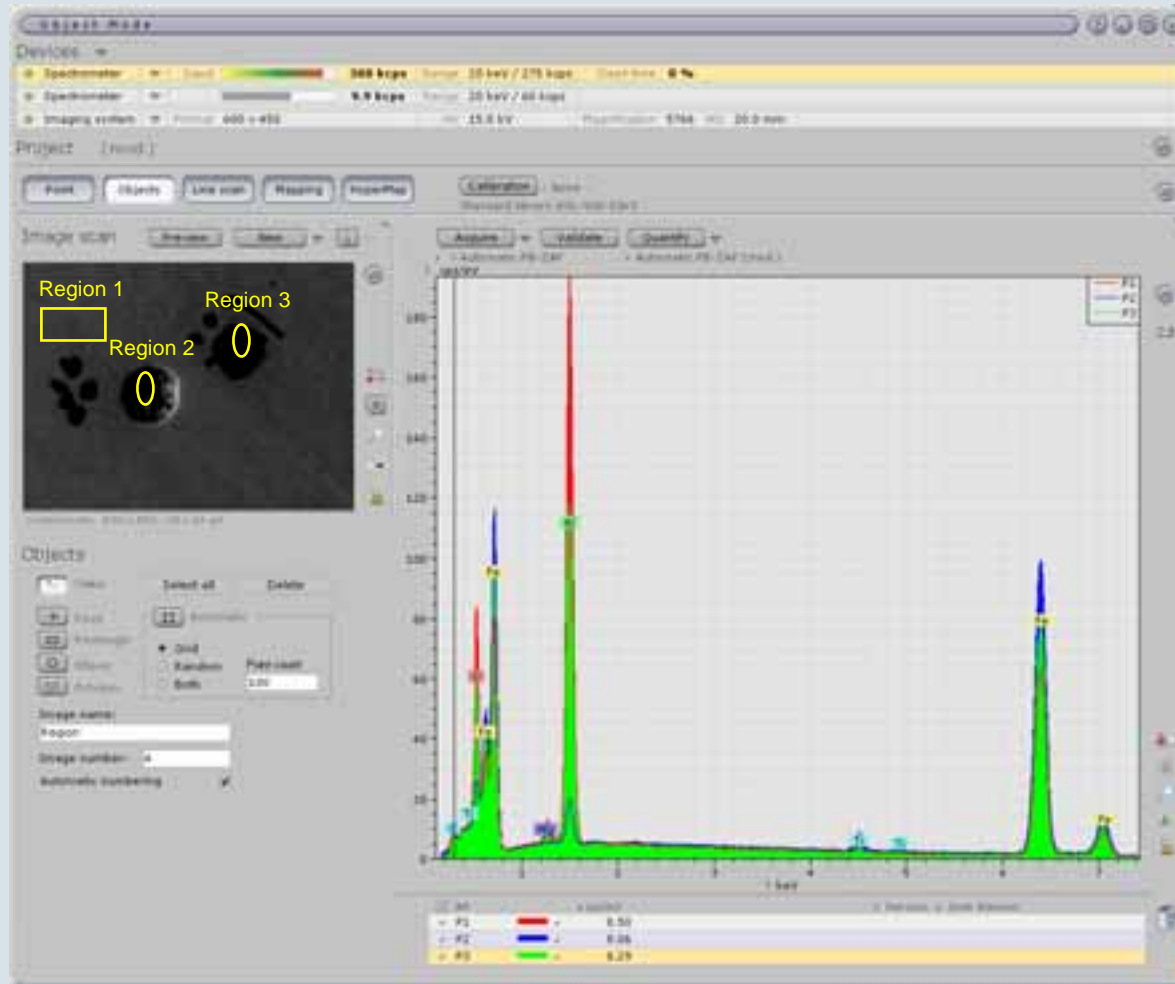
- No liquid nitrogen
- No vibration
- Ready to use in 30 seconds
- Does not have to be warm to vent chamber
- Better resolution than the traditional Si(Li)
- No change in resolution with count rate to 100,000 cps
- Count rates of up to 1,000,000 cps
- High resolution maps - Real time analysis of HyperMap
- Makes traditional mapping obsolete
- Faster survey of sample
- Easily moved to a new microscope – No Dewar!
- 10 mm sq., 30 mm sq. active areas
- QUAD: 4 x 10mm sq. array
- Multiple XFlash detectors: up to four SDDs together

# Sample: Steel with Inclusions

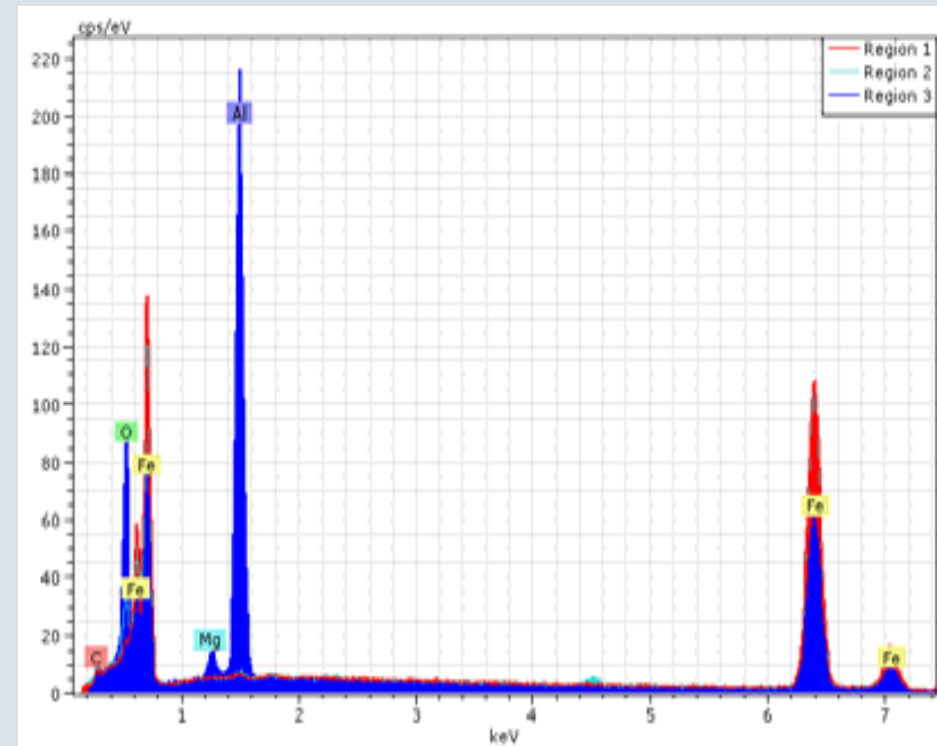
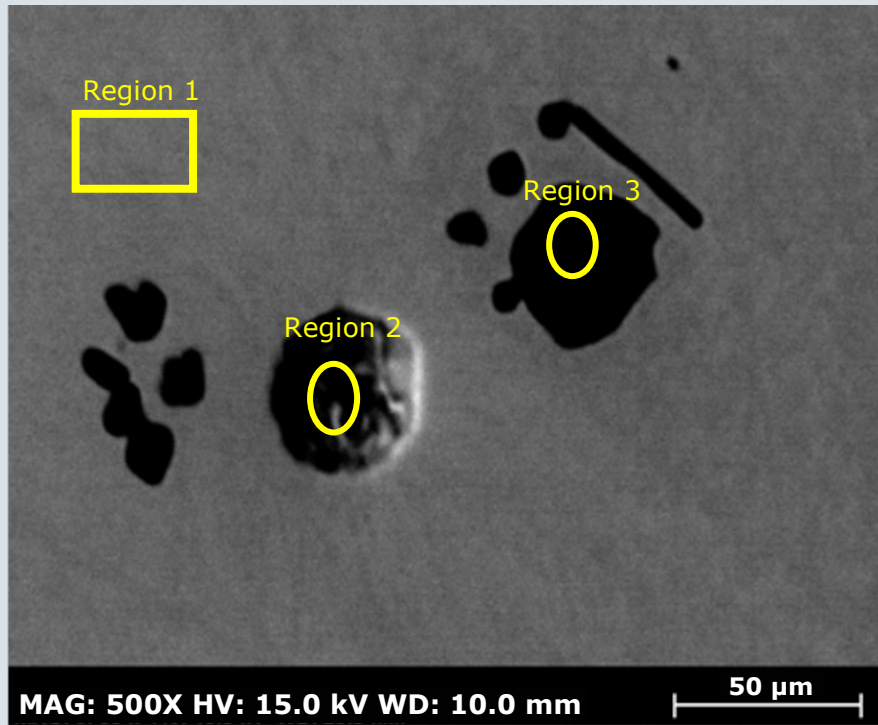


Overall Spectrum  
15kV, 35° $\Psi$ , 20nA  
80Kcps @ 40%DT

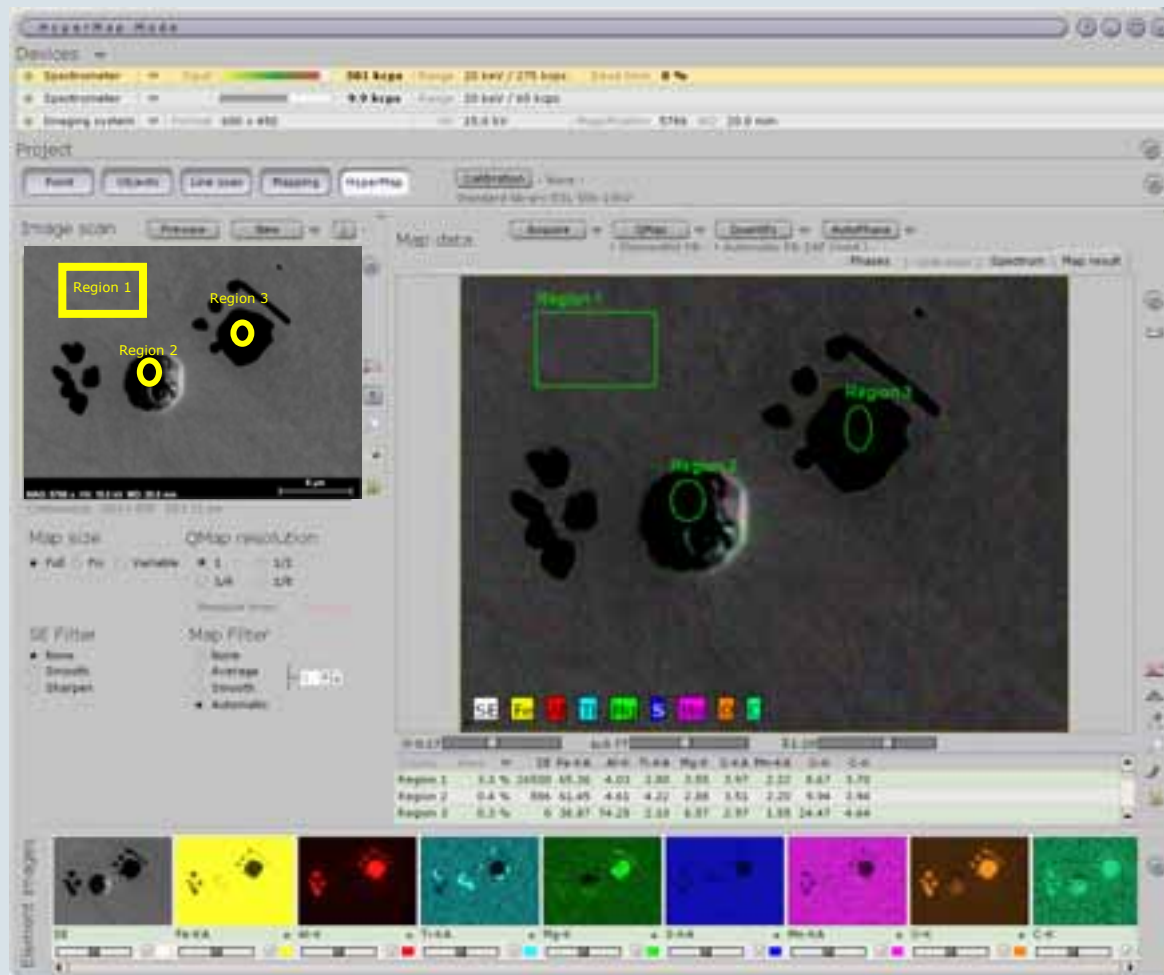
# ESPRIT: Multi-Point Analysis



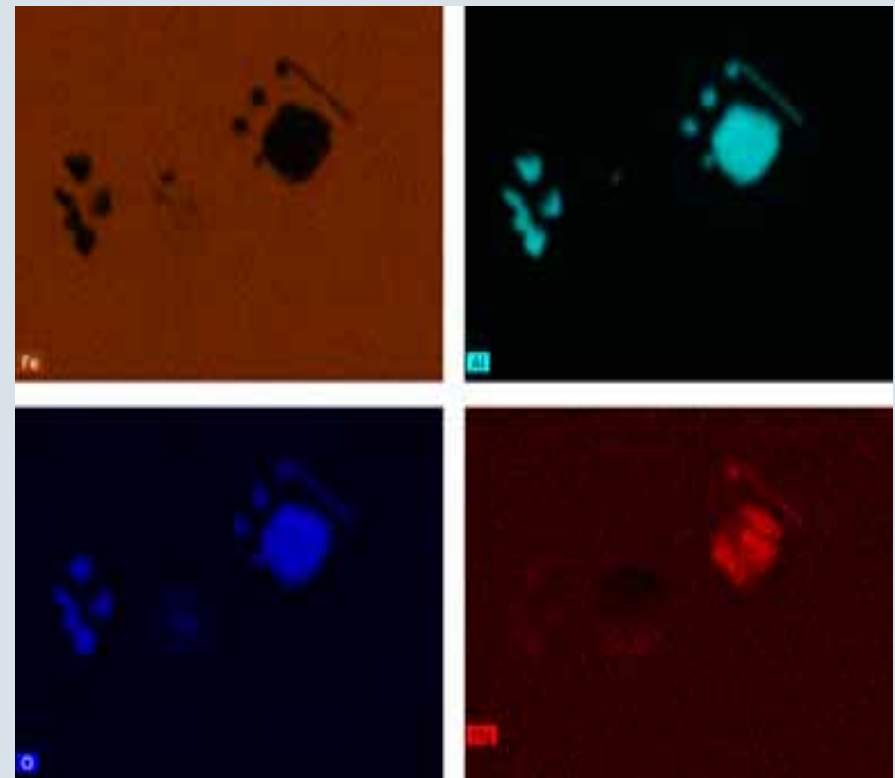
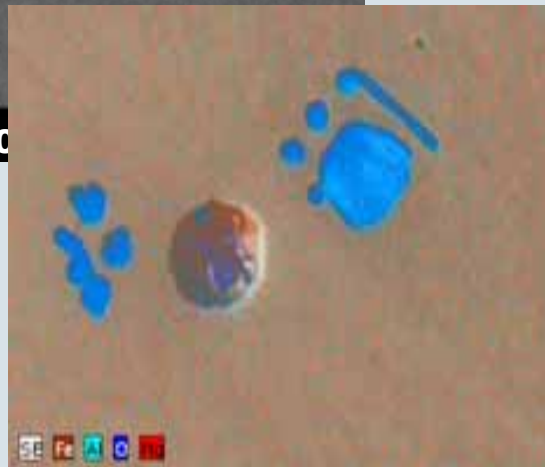
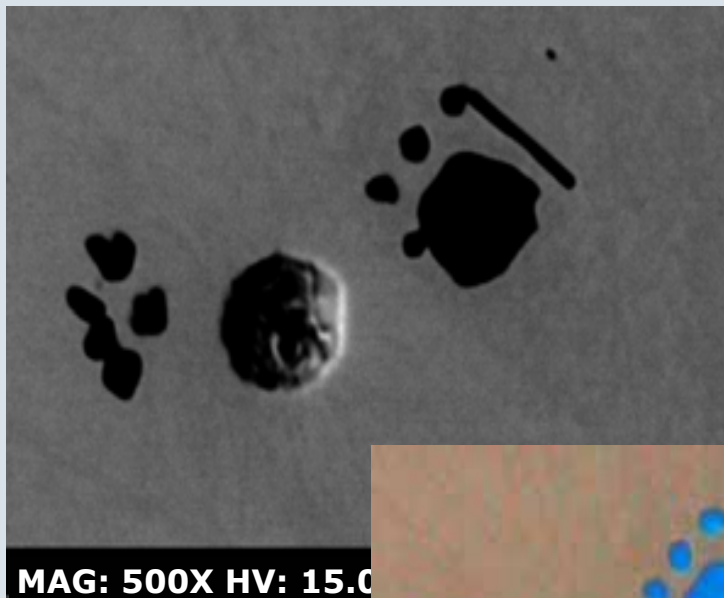
# Multi-Point Analysis



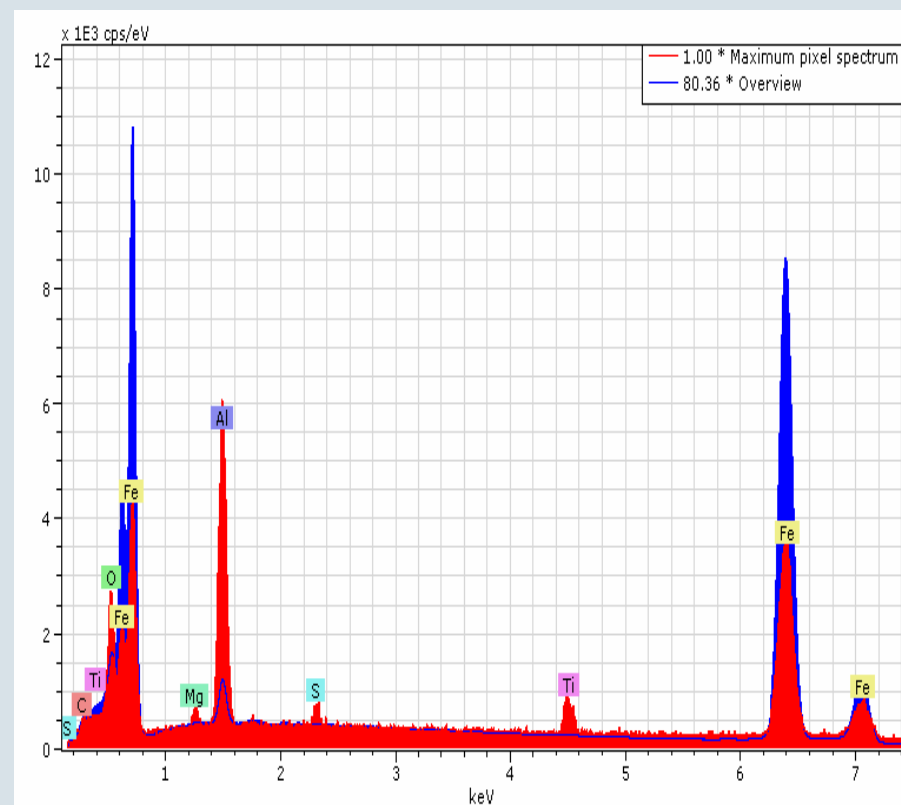
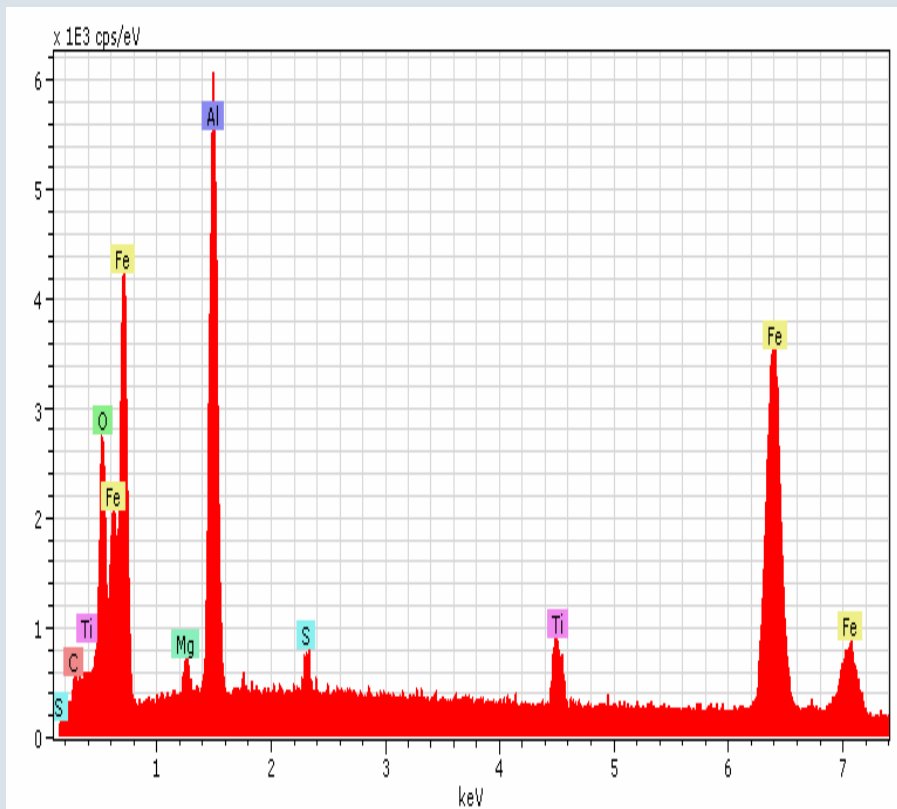
# HyperMap: Collection



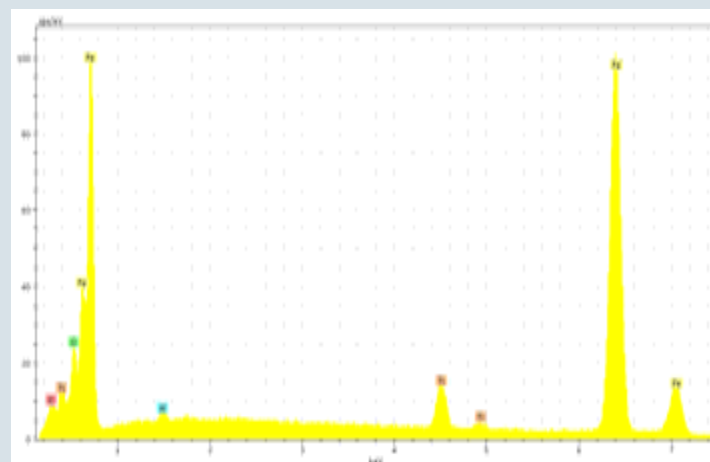
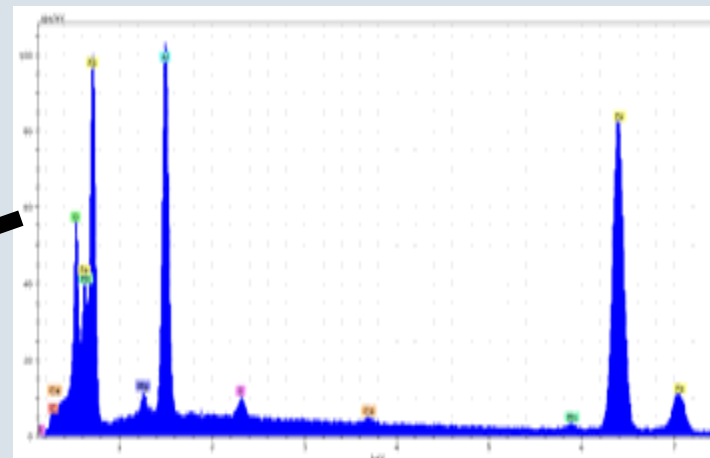
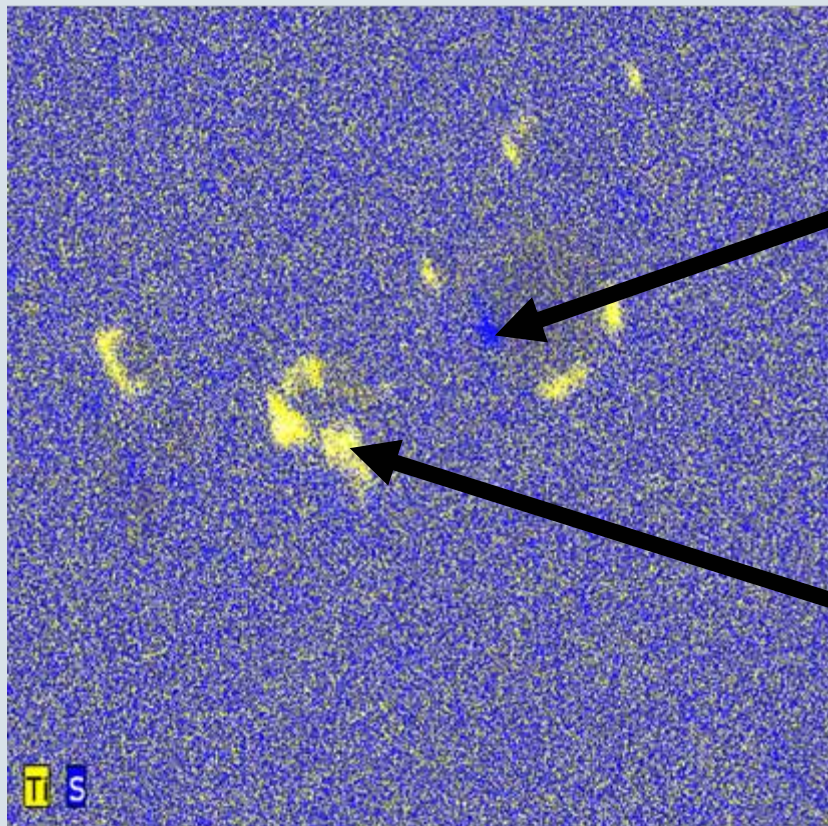
# HyperMap: SEI, Composite and Individual Maps



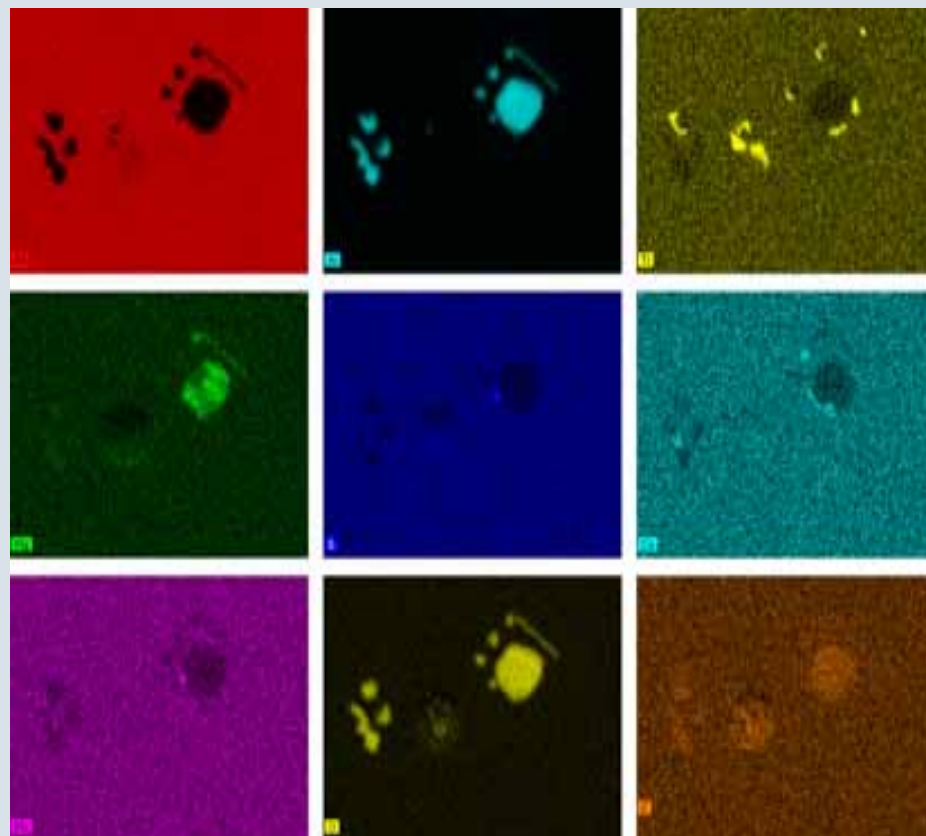
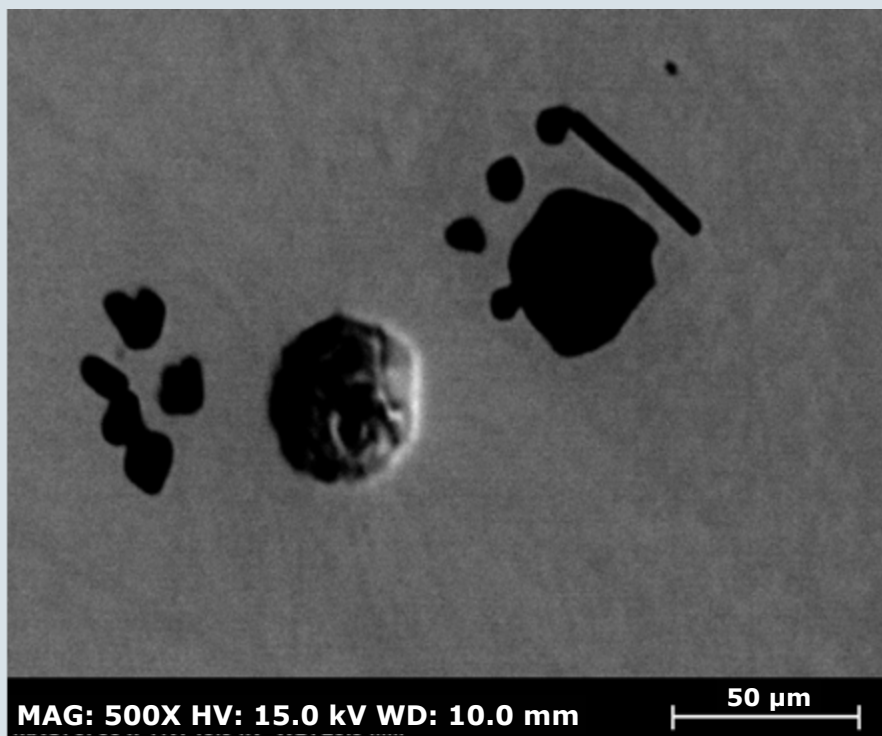
# Maximum Pixel Spectrum



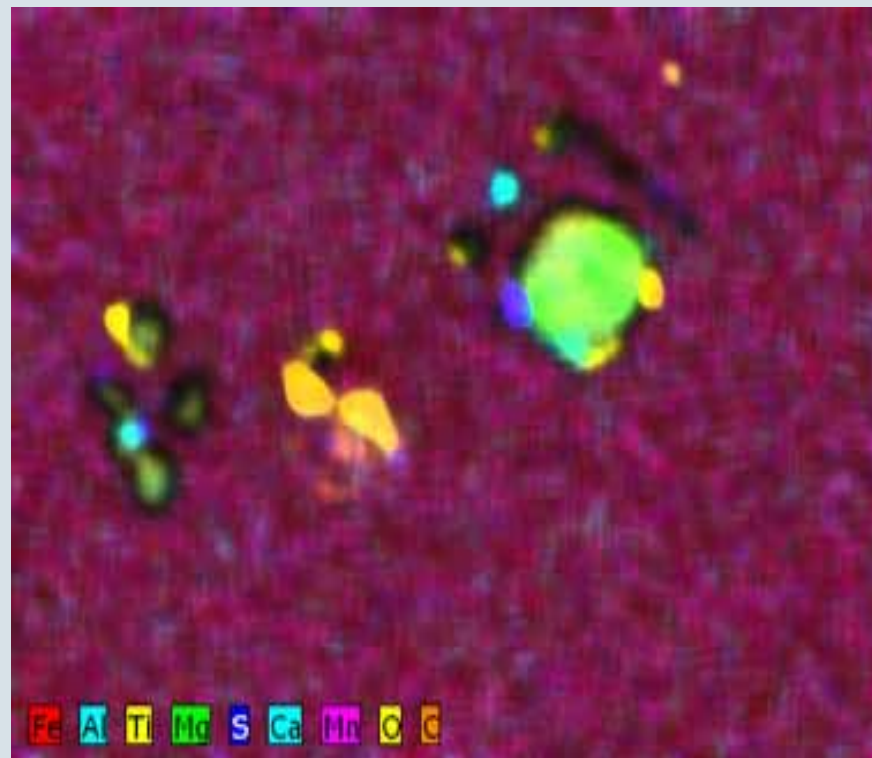
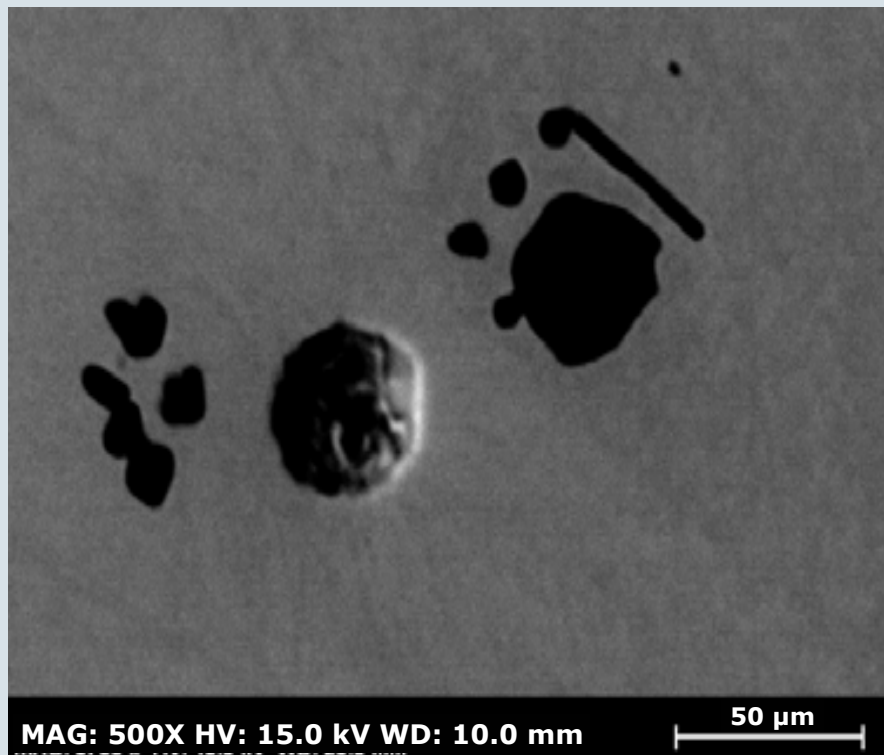
# Verification of Maximum Pixel Spectrum



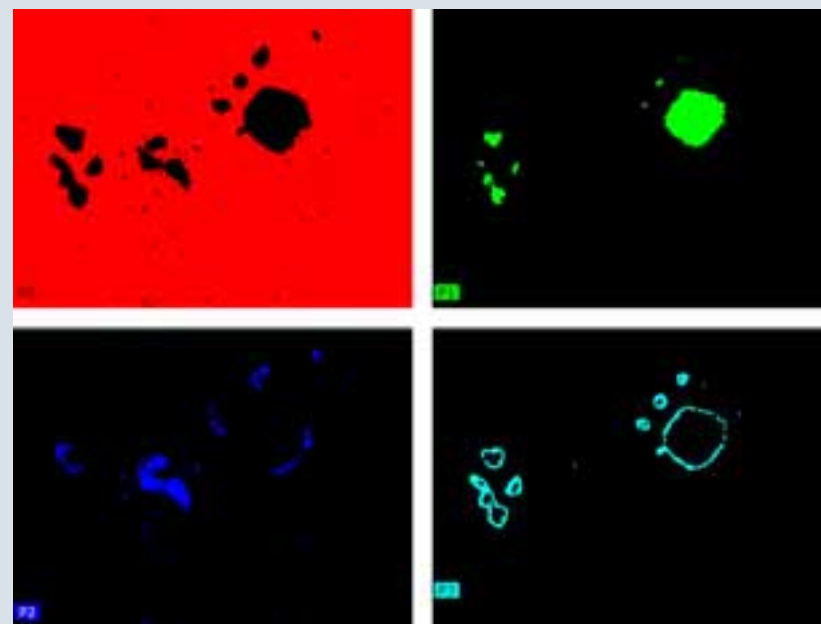
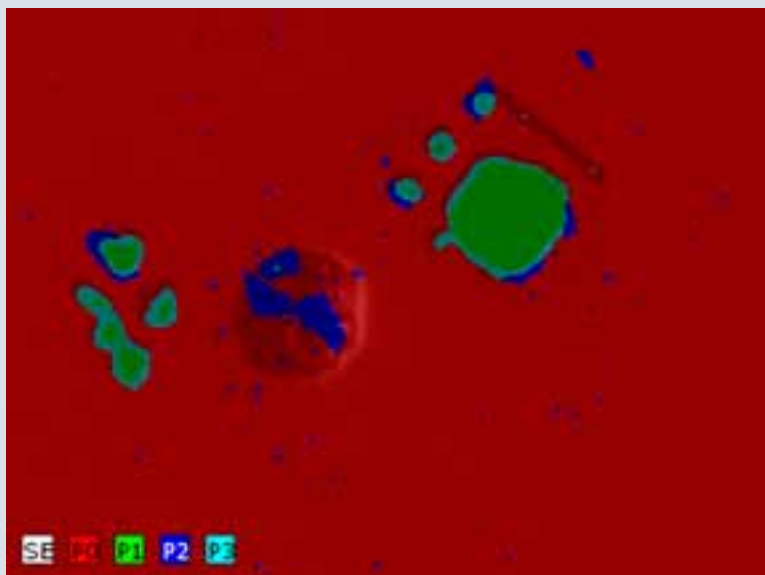
# HyperMaps: SEI and Individual Maps



# HyperMap: SEI and Composite

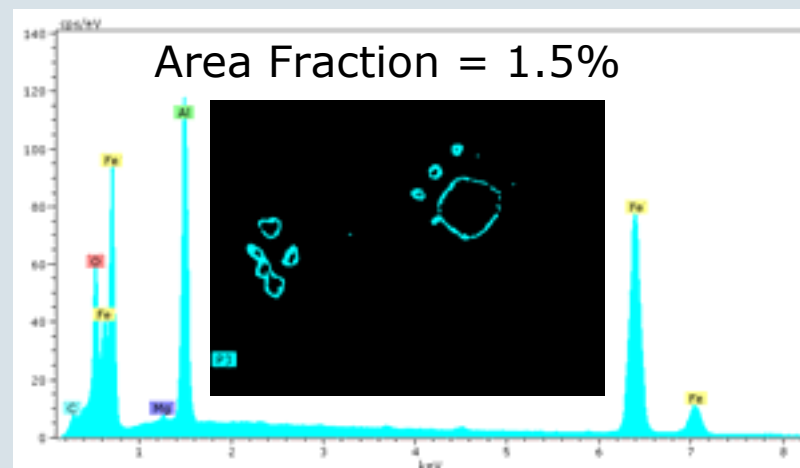
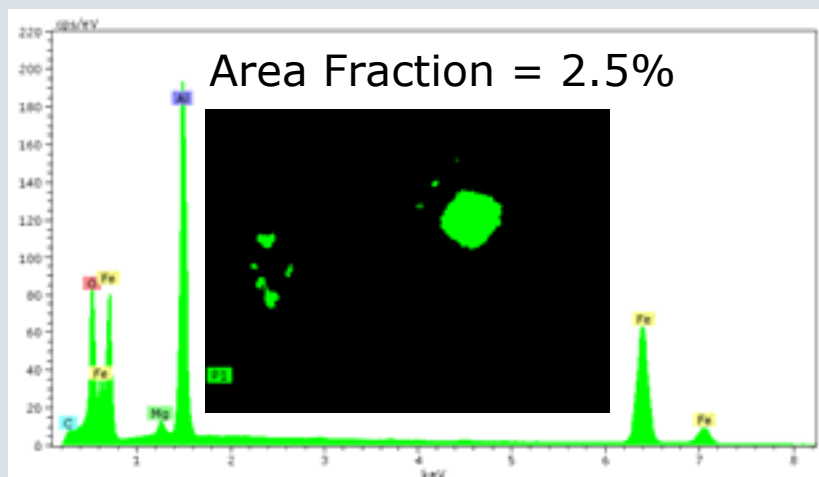
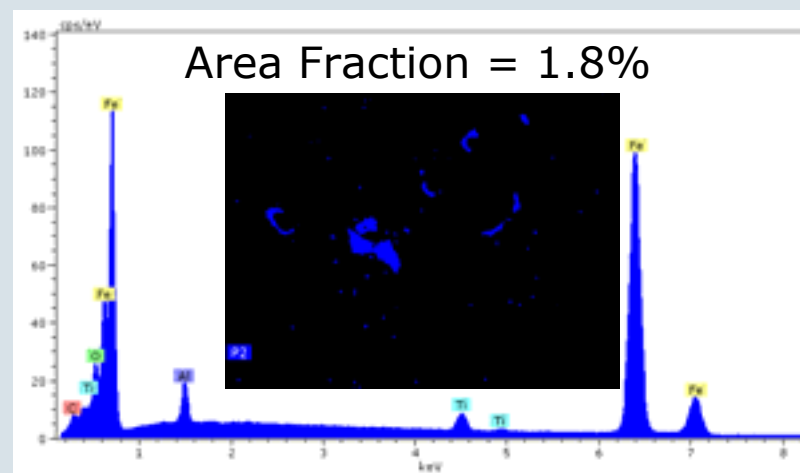
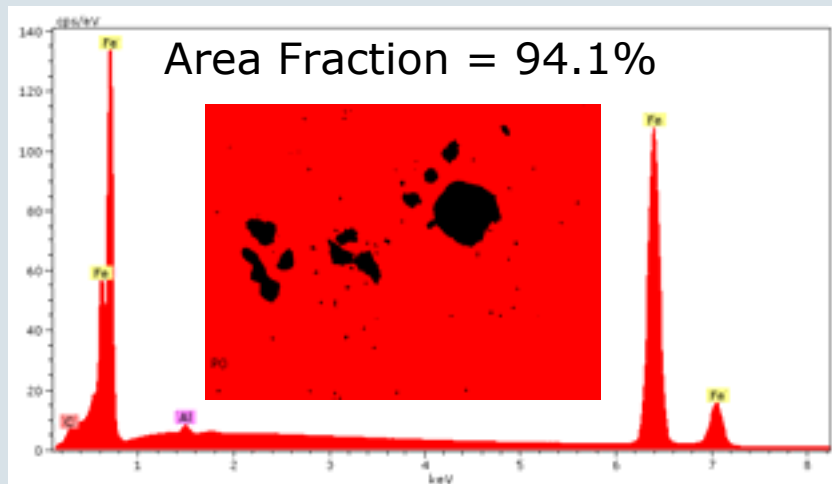


# AutoPhase



Counts	Area	SE	Fe-KA	Al-K	S-KA	Ti-KA	Mg-K
P0	94.1 %	--	64.11	4.01	3.45	2.50	2.81
P1	2.5 %	--	37.45	64.09	2.75	2.15	5.18
P2	1.8 %	--	58.89	7.72	3.23	5.41	2.73
P3	1.5 %	--	46.07	39.47	3.20	2.64	3.45

# AutoPhase Extracted Spectra



# XFlash<sup>®</sup>, QUANTAX & ESPRIT



- XFlash SDD
  - No maintenance
  - High speed data acquisition
  - Maintain spectral resolution
  - Maintain sensitivity for all elements
- Not limited to low resolution images
- Compositional image in seconds
- ID phases that backscatter cannot separate
- Immediate verification of data while collecting
  - Meaningful Maps available in seconds
  - Extract spectra to verify identification and phase
  - Maximum pixel calculation
  - Phase Analysis
- Full Quantification of any data



# Micro-XRF Spectrometer **ARTAX**

A portable spectrometer  
offering  $\mu\text{m}$  resolution

Armin Gross

Bruker AXS Microanalysis GmbH

# Contents

1. Introduction to  $\mu$ XRF
2. ARTAX instrument design
3. Line scan of solar cells
4. Element mapping in forensics
5. Element mapping in industry
6. Summary and outlook

# XRF Technologies

## $\mu$ XRF vs EDS and XRF



Technology	X-ray Microanalysis (EDS)	Micro X-ray Fluorescence Analysis ( $\mu$ XRF)	X-ray Fluorescence Analysis (XRF)
Capability	High resolution element mapping in the sub- $\mu$ m range	Non-destructive spatial investigation of element distribution	Elemental analysis of bulk samples
Limitation	Sample size limited by chamber	Analytical range of 10 $\mu$ m to 10 mm	No information about spatial element distribution

nm -  $\mu$ m

$\mu$ m - mm

mm - cm



# ARTAX

## Introduction to $\mu$ XRF

### ■ Micro X-ray fluorescence analysis ( $\mu$ XRF)

- Spatial resolved multi-element analysis
- Non-destructive analysis of the element composition
- Analysis at the location of the object
- Scan range 50 x 50 mm
- Target market art and conservation



# ARTAX Instrument Design

## Detail of the Measuring Head



Red LED

He purge

Sample illumination

XFlash detector

CCD camera

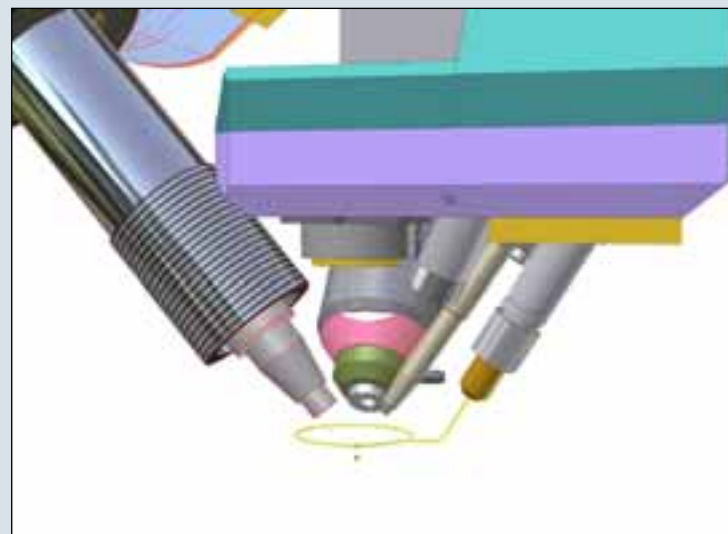
Collimator / Polycapillary lens

Tube housing

# Instrument Design

## Excitation Source

- Metal-ceramic X-ray tube
  - Mo, W or Rh target
- Beam focusing by polycapillary lens
  - Ensemble of several thousand glass capillaries
  - Each capillary  $\sim 1 \mu\text{m}$  in diameter
- Advantages for fast mapping
  - Minimal focal spot size about  $70 \mu\text{m}$
  - Outstanding intensity gain  $> 1000$  compared to collimator



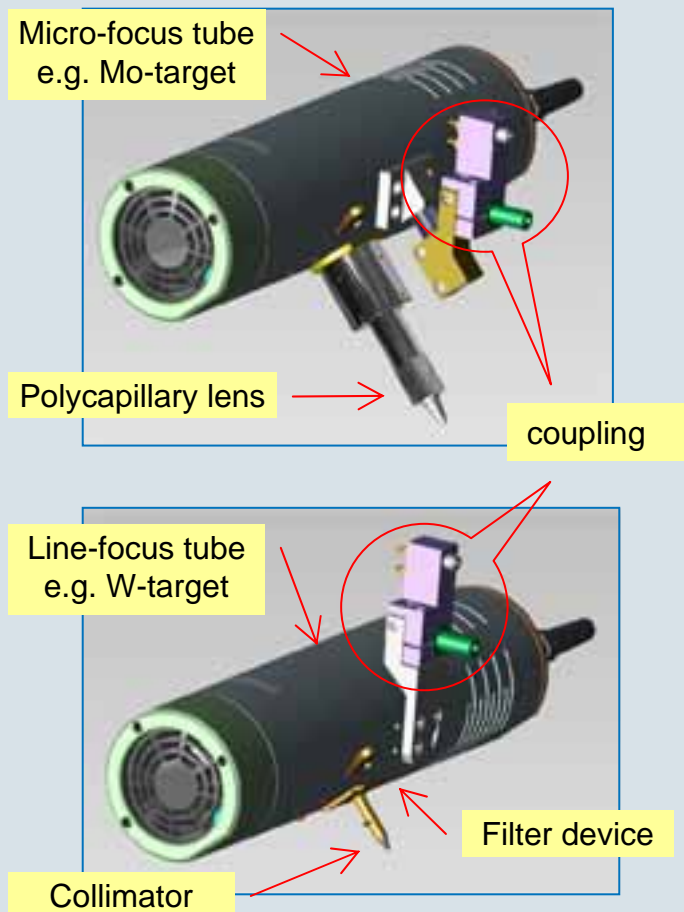
# Instrument Design

## Excitation Source

■ High precision coupling for fast exchange of excitation source

### A source contains:

- tube housing incl. shutter
- X-ray tube (Mo, Rh, W target etc.)
- X-ray optics (polycapillary lens, collimator)
- Filter device

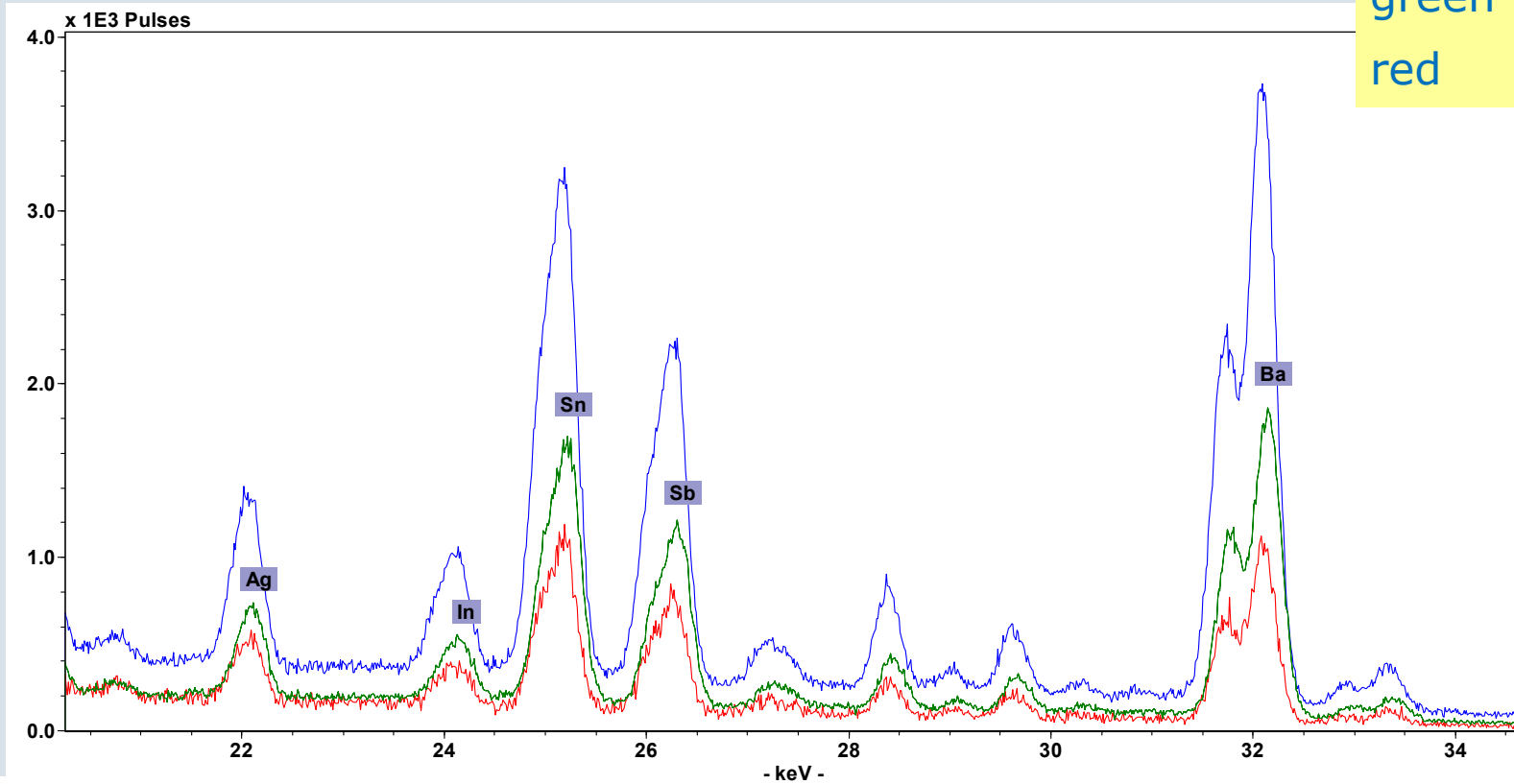


# Glass Standard BR E2

## Spectrum section above 20 keV

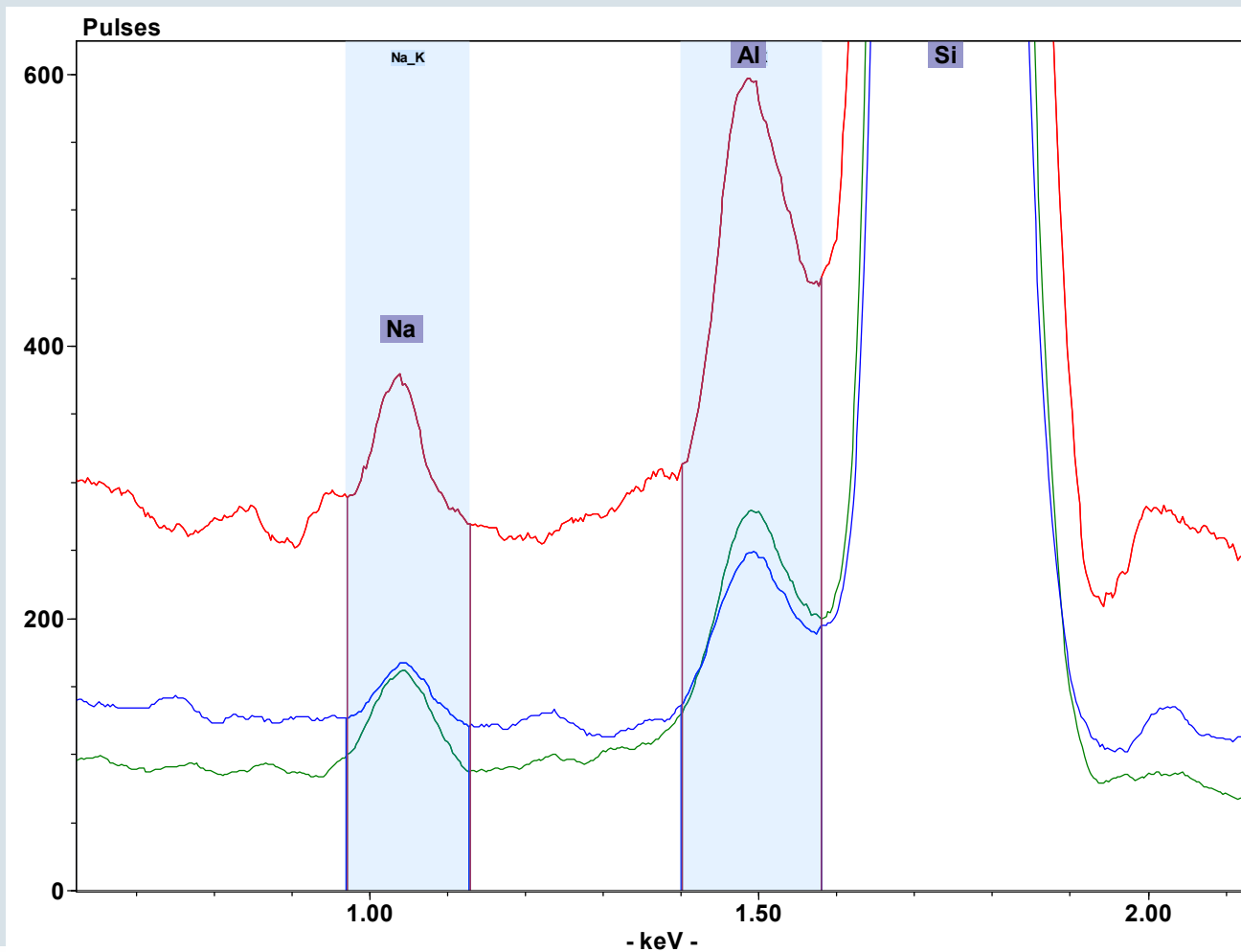


blue = W  
green = Mo  
red = Cr



# Glass Standard CRM 610

## Low energy range



blue = W  
green = Mo  
red = Cr

# Instrument Design

## XFlash<sup>®</sup> Silicon Drift Detector

### ■ Specifications

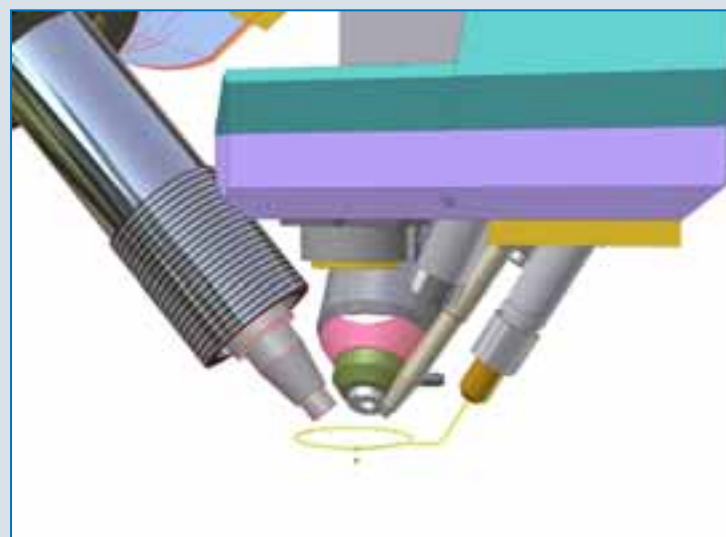
- Si drift chamber detector (SDD)
- 10 mm<sup>2</sup> active area, Be window
- Electro-thermal cooling

### ■ Advantages

- Energy resolution < 160 eV (Mn-K $\alpha$  line, 100 kcps)
- Highest throughput (> 100 kcps)
- No liquid nitrogen required

### ■ Upgrade option

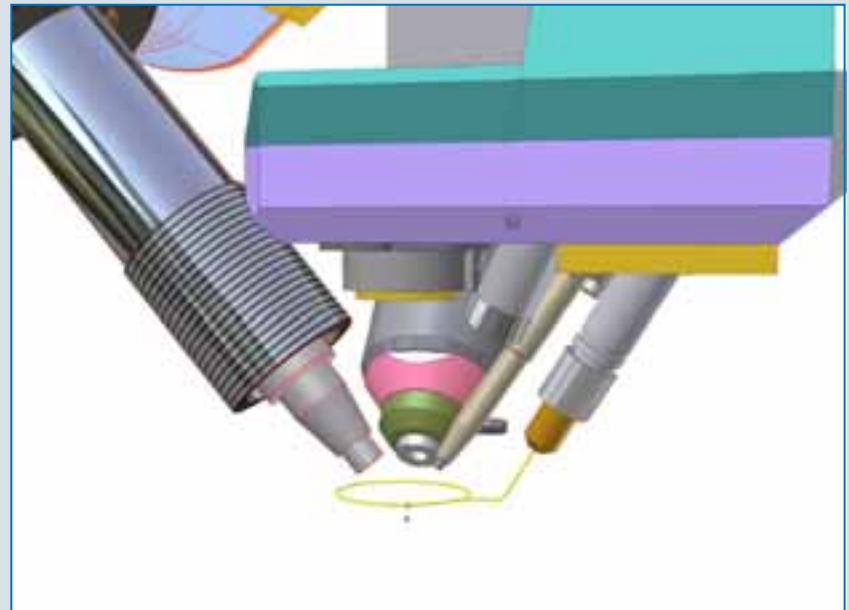
- Energy resolution < 145 eV



# Instrument Design

## CCD Camera and Sample Illumination

- Camera image of sample area under investigation  
Magnification about 20
- White LED for optimization of the image quality  
can be dimmed
- One red laser diode - can be dimmed
- Red spot in center of crosshair



# Instrument Design

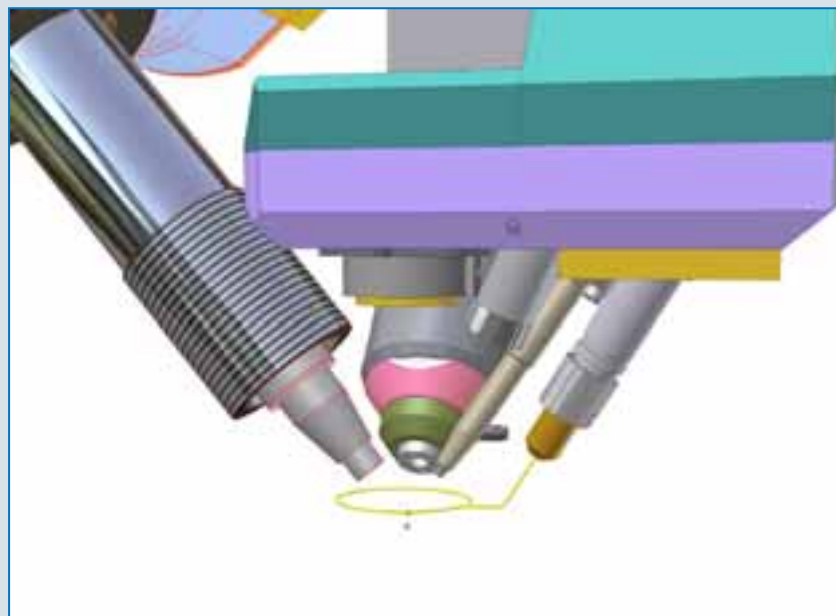
## Helium Purging

### Specifications

- Detection of light elements Na(11) to Ar(18)

### Advantages

- No vacuum required, avoids damage of fragile samples
- Hardly limitations in form and size of the sample
- Small time effort



# Art Applications

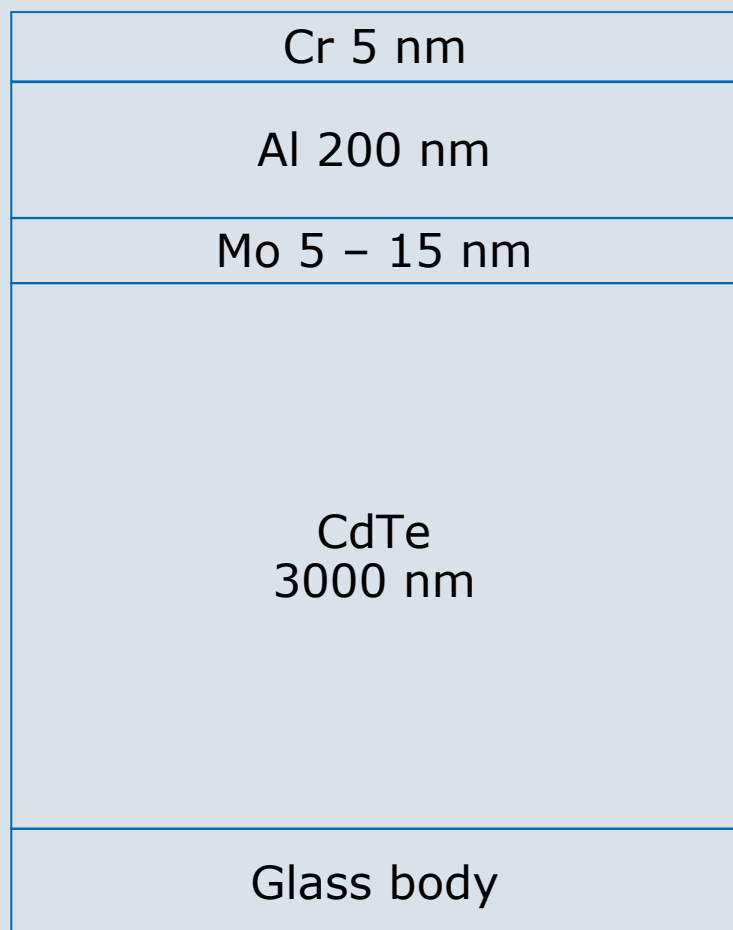
## Limoges Painted Enamels

- 15th to 17th century:  
peak in the painting of enamels in the french city Limoges
- End of 19th century:  
Limoges style replicas
- Line thickness of paintings about 150  $\mu\text{m}$
- Classification initially done by EDS



# Line Scan Application

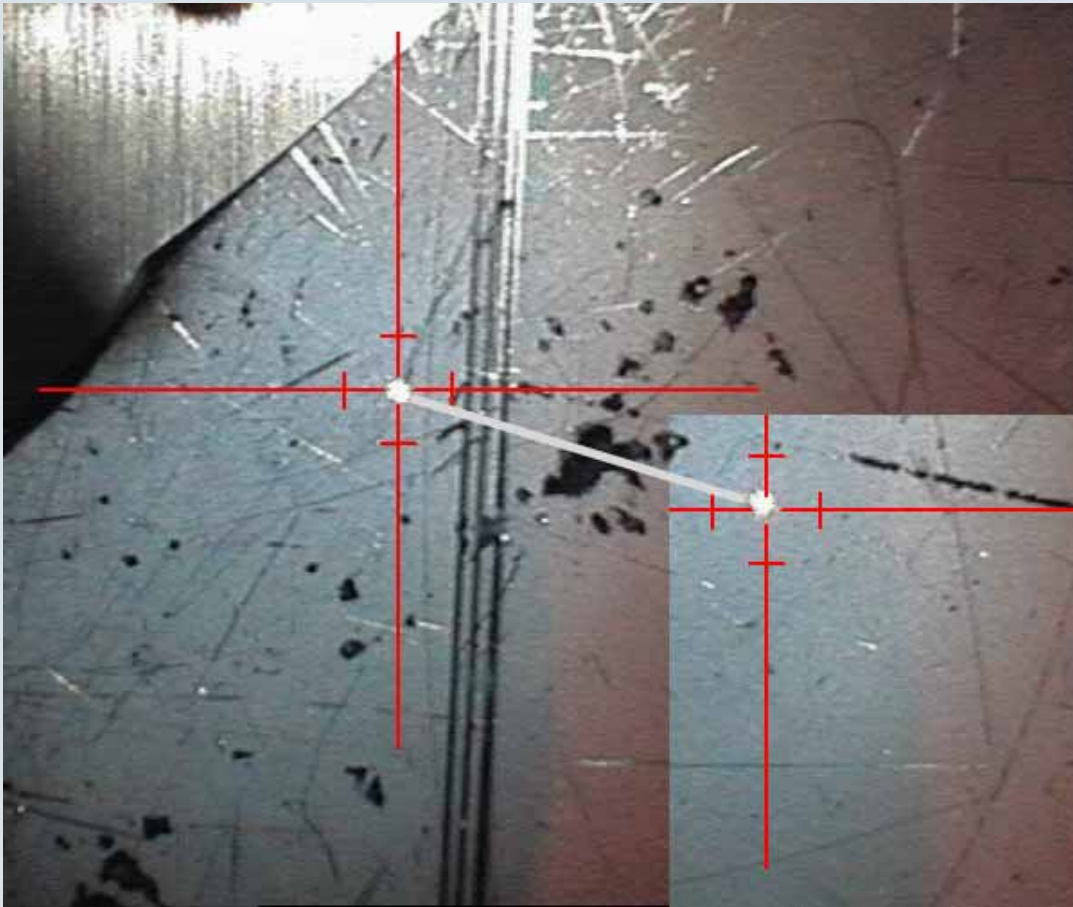
## Investigation of Solar Cells



- Production of solar cells
  - Typical layer structure of a CdTe solar cell
  - During production the homogeneity is tested routinely
- Measurement parameters
  - ARTAX line scan
  - 48 measurements
  - 50 kV, 600  $\mu$ A
  - 15 s

# Applications

## Investigation of Solar Cells



# Applications

## Investigation of Solar Cells



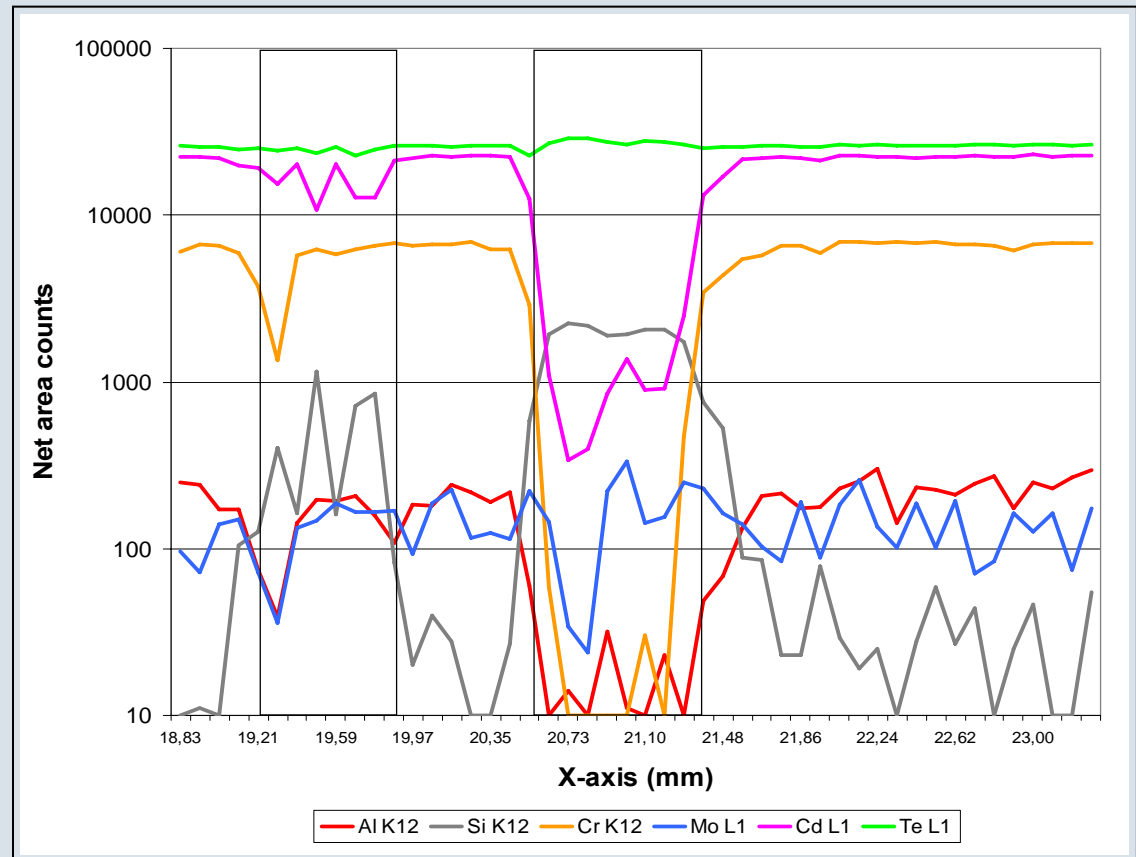
Cr: removed

Al: almost removed

Mo ?

Cd > 90% removed  
Te almost unchanged

Si: lower absorbance



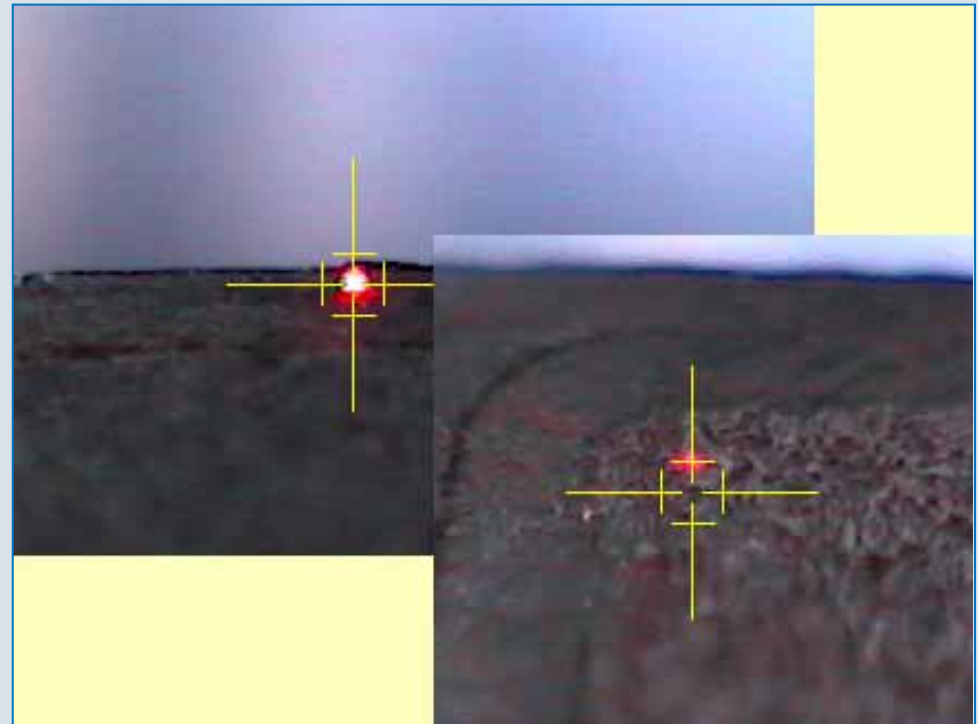
# Applications

## Element Mapping in Forensics



### ■ Section of a bullet

- Area 1.8 x 1.8 mm
- Meas. points 100
- Meas. time 5 s
- Total time 23 min



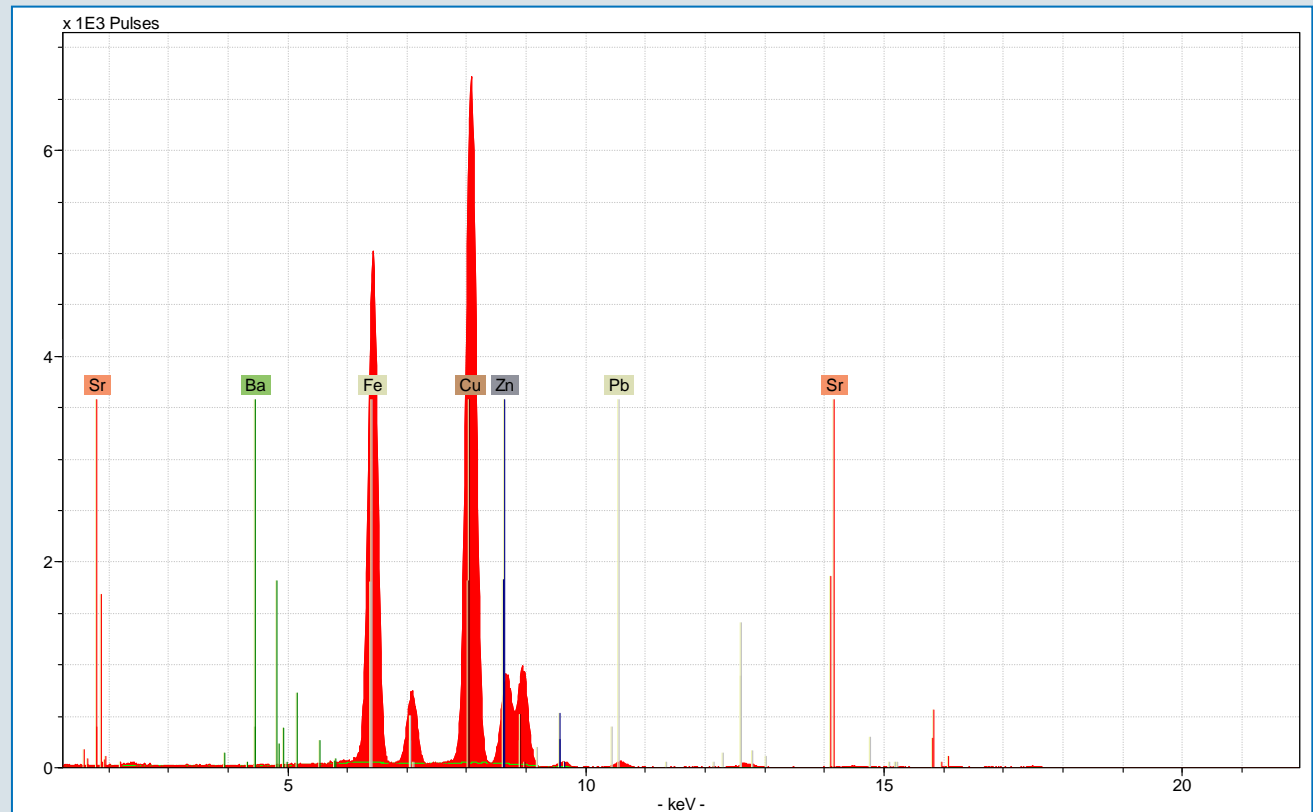
# Applications

## Element Mapping in Forensics



### ■ Section of a bullet

- Spectrum



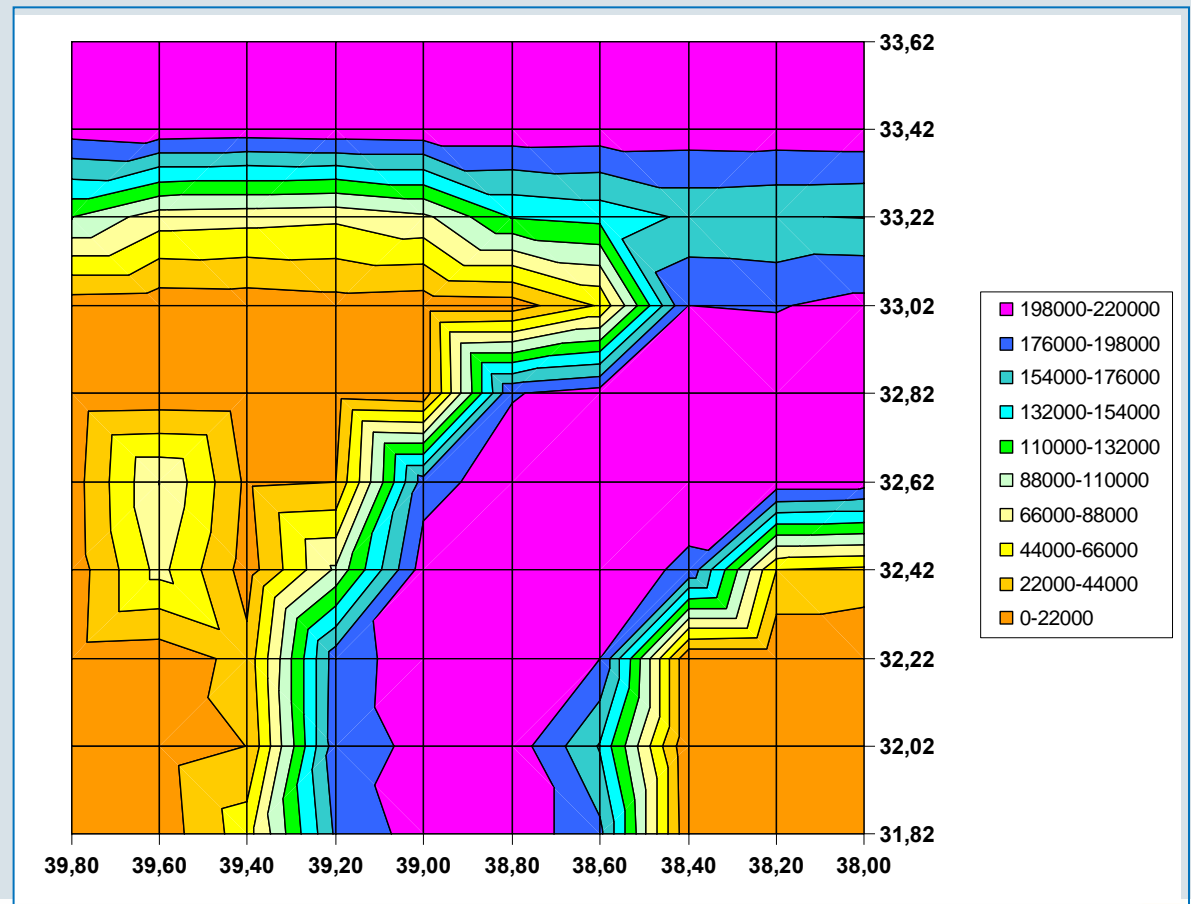
# Applications

## Element Mapping in Forensics



### ■ Section of a bullet

- Distribution of **Fe, Cu, Pb, Ba**



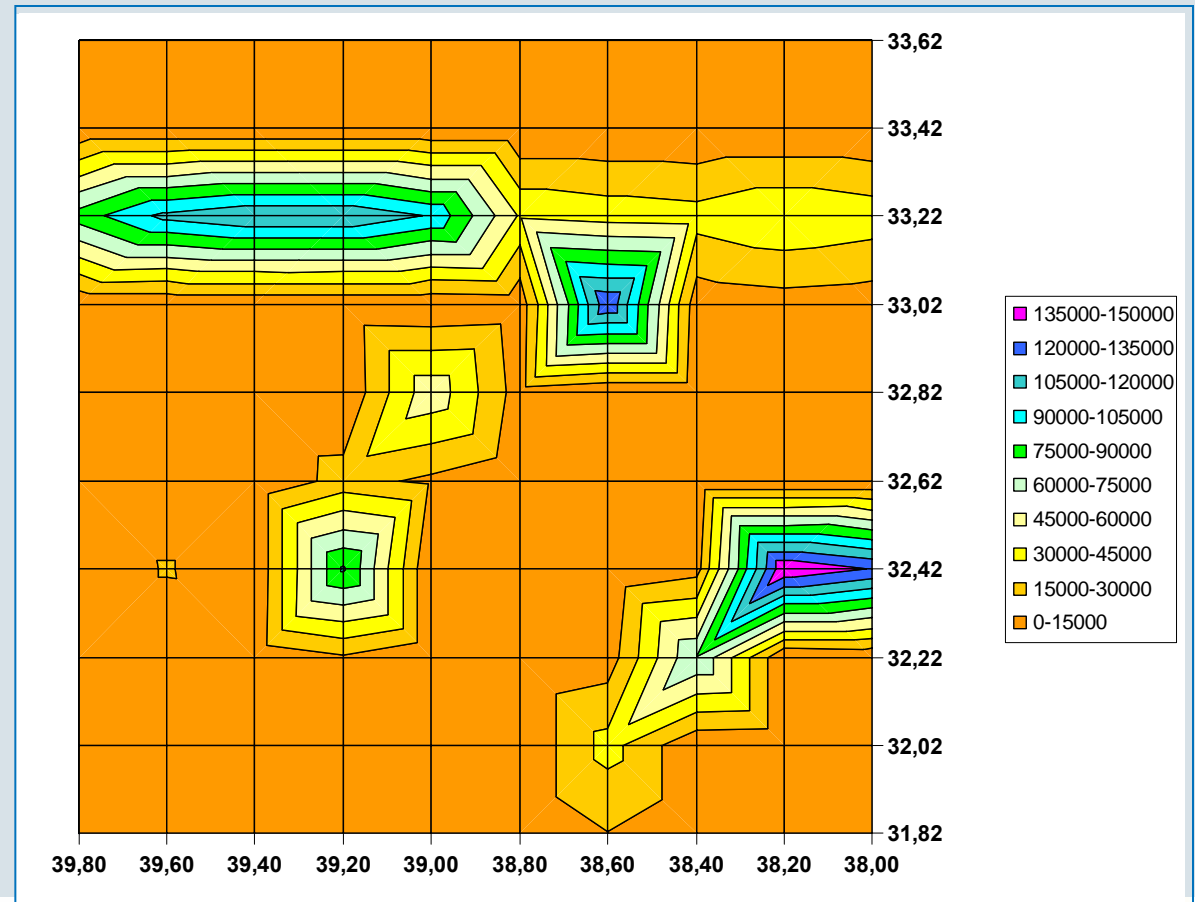
# Applications

## Element Mapping in Forensics



### ■ Section of a bullet

- Distribution of Fe, **Cu**, Pb, Ba



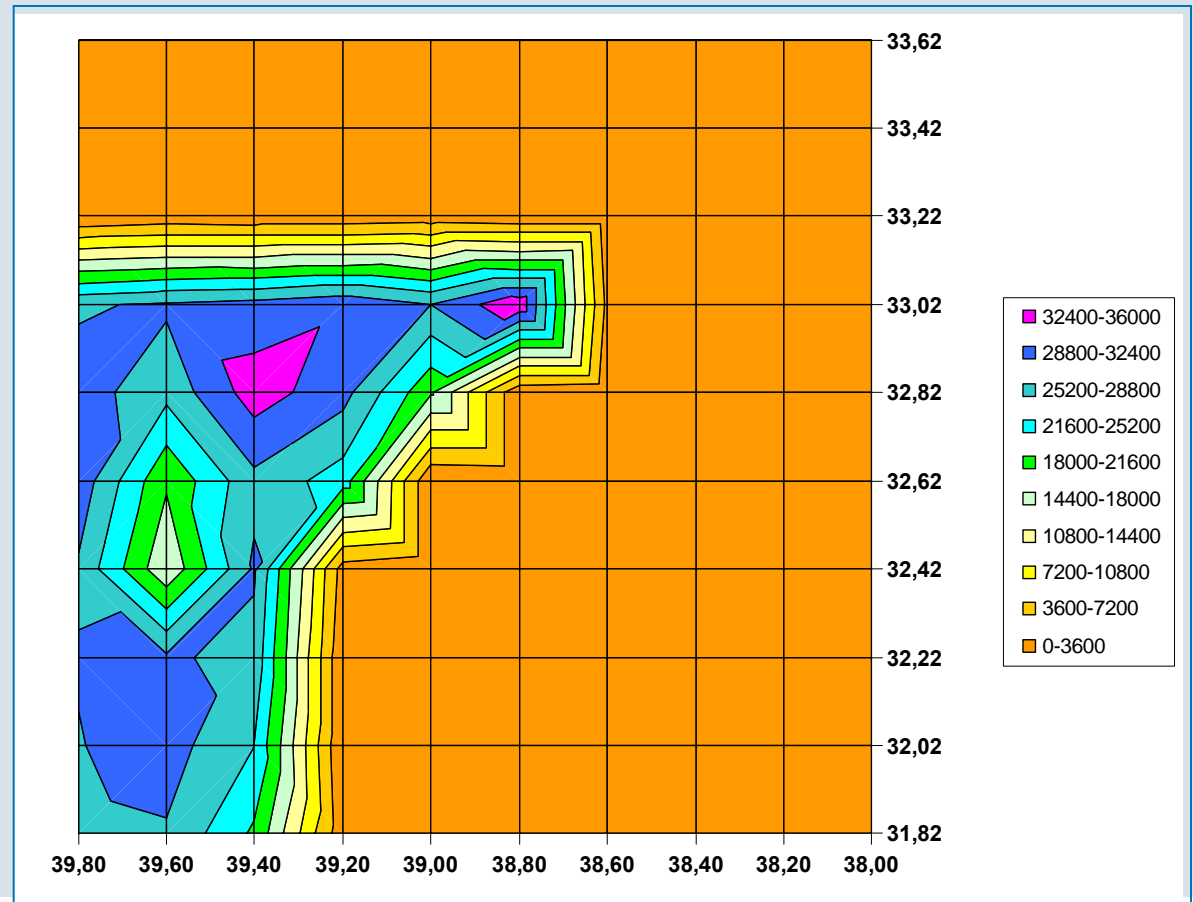
# Applications

## Element Mapping in Forensics



### ■ Section of a bullet

- Distribution of Fe, Cu, **Pb**, Ba



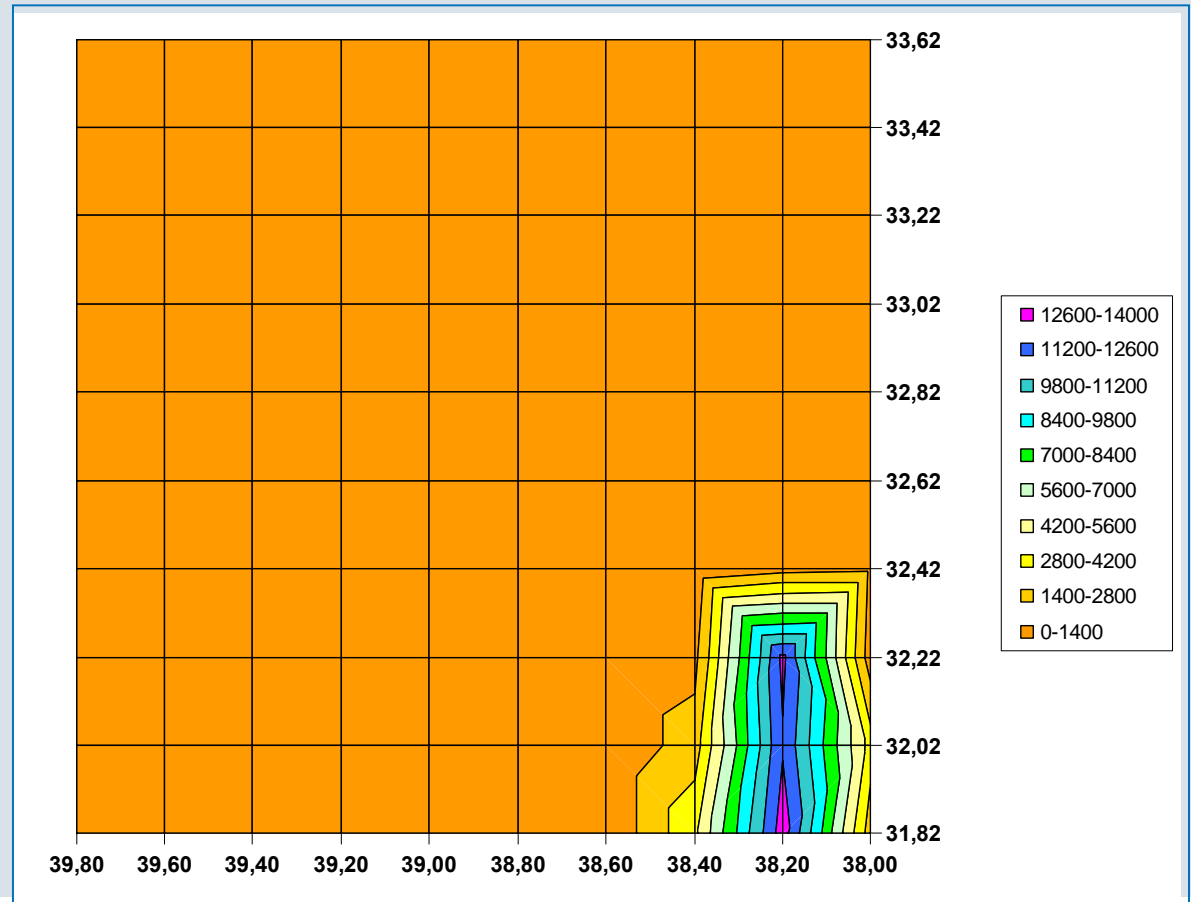
# Applications

## Element Mapping in Forensics



### ■ Section of a bullet

- Distribution of Fe, Cu, Pb, **Ba**



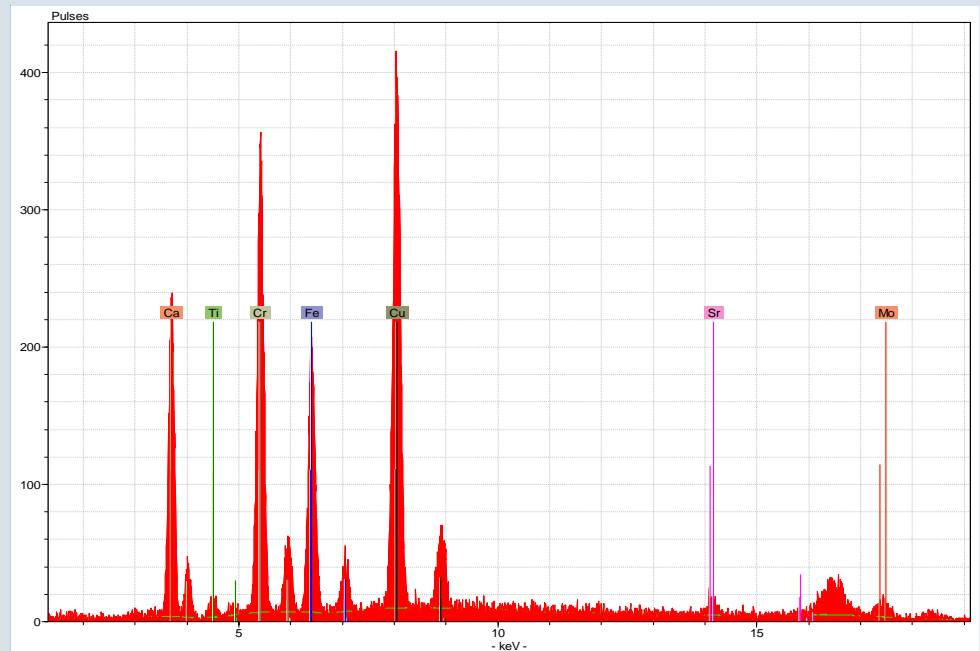
# Applications

## Element Mapping in Industry



### ■ Polymer mould with metal structures

- Area 2.9 x 2.9 mm
- Meas. points 900
- Meas. time 5 s
- Total time 5 h 10 min



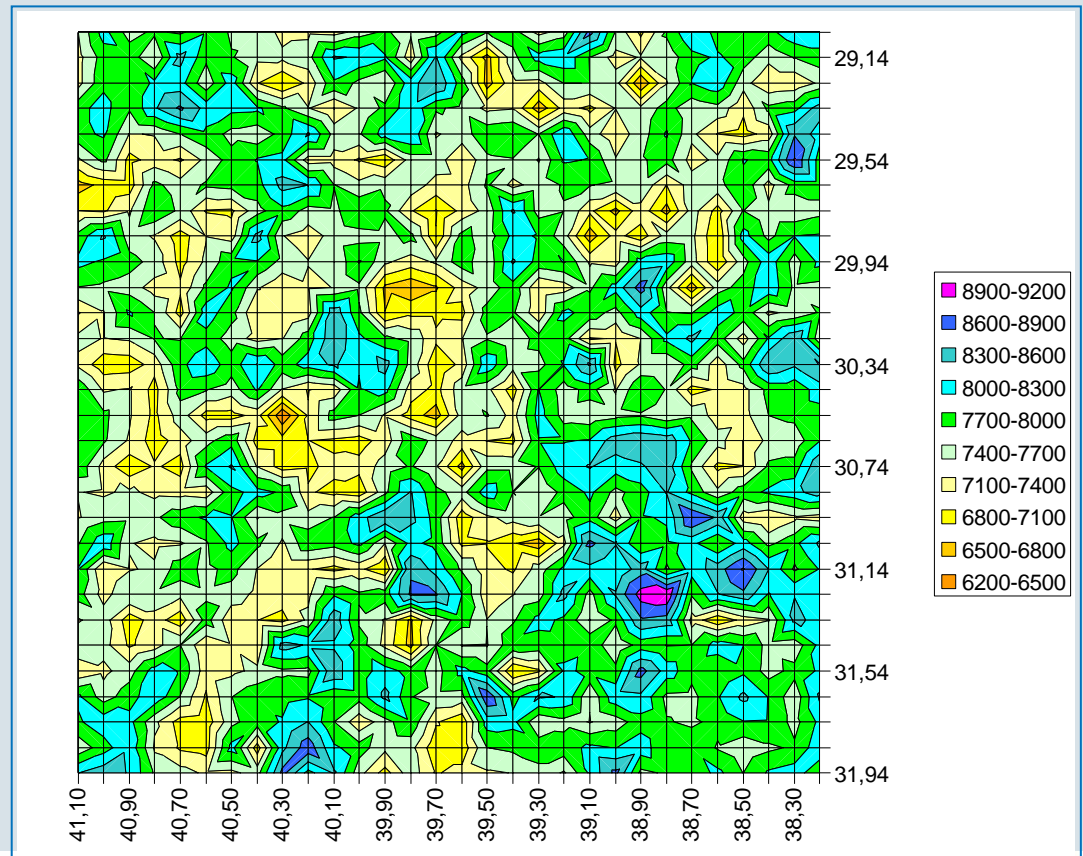
# Applications

## Element Mapping in Industry



### ■ Polymer mould with metal structures

- Distribution of **Cr** and **Ca**



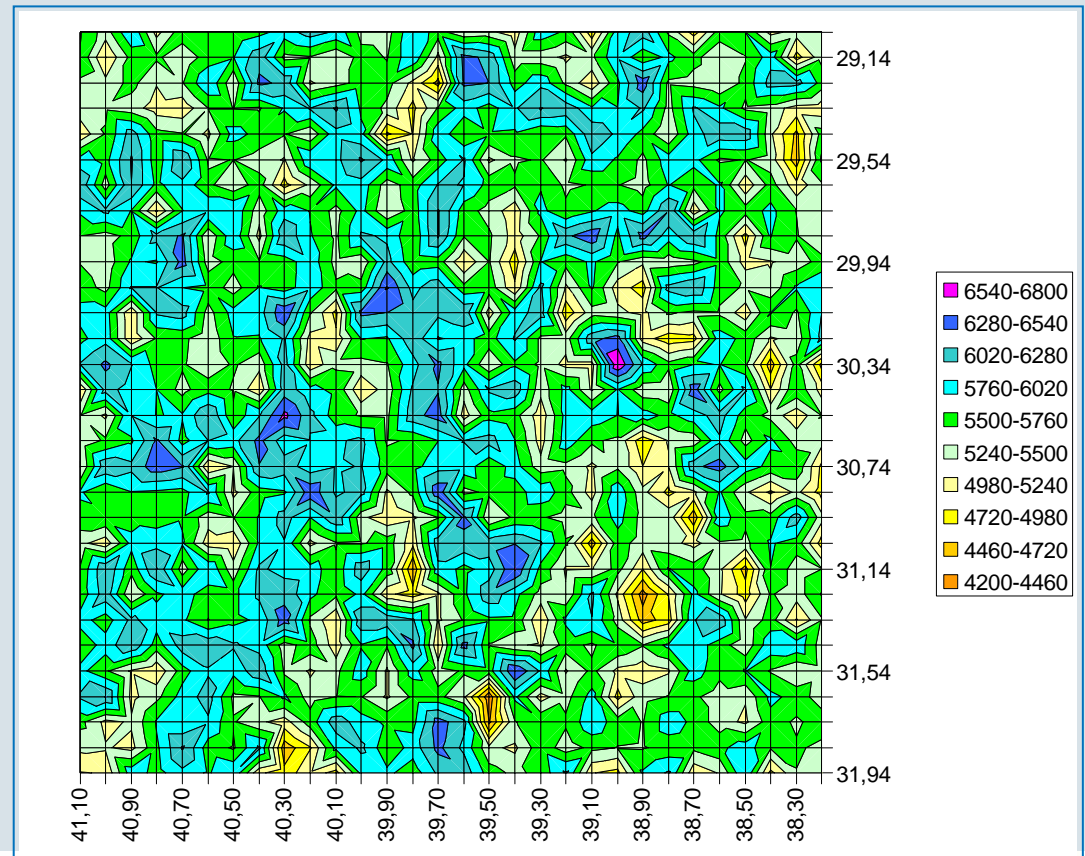
# Applications

## Element Mapping in Industry



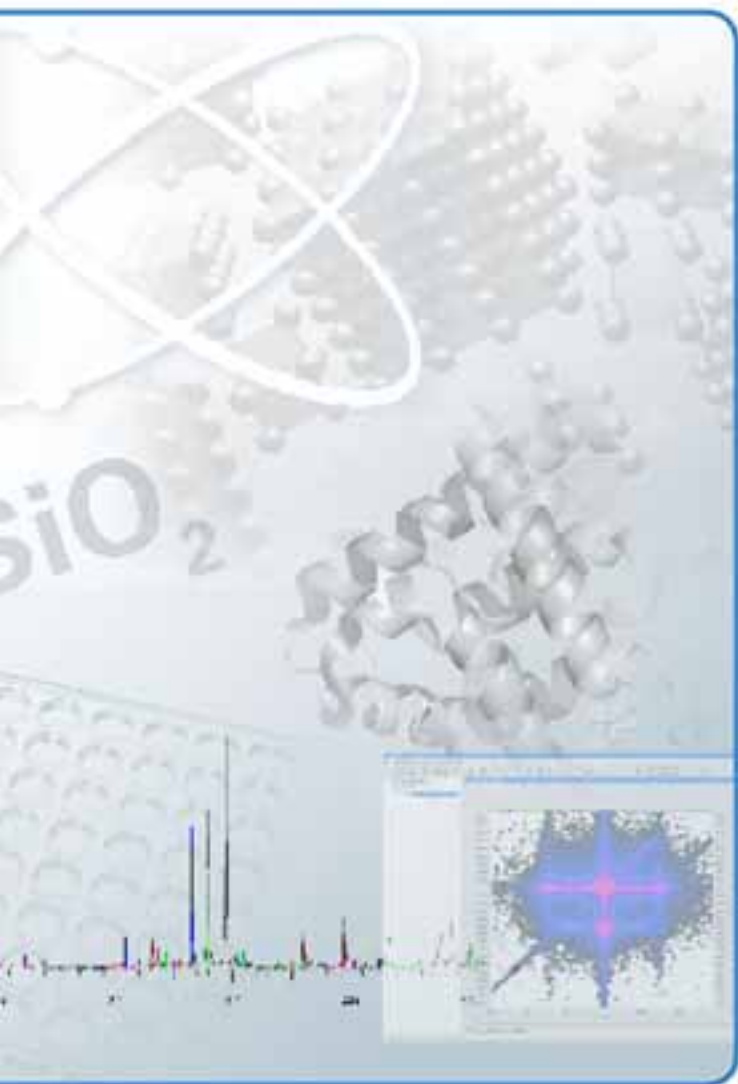
### ■ Polymer mould with metal structures

- Distribution of Cr and Ca



# Summary and Outlook

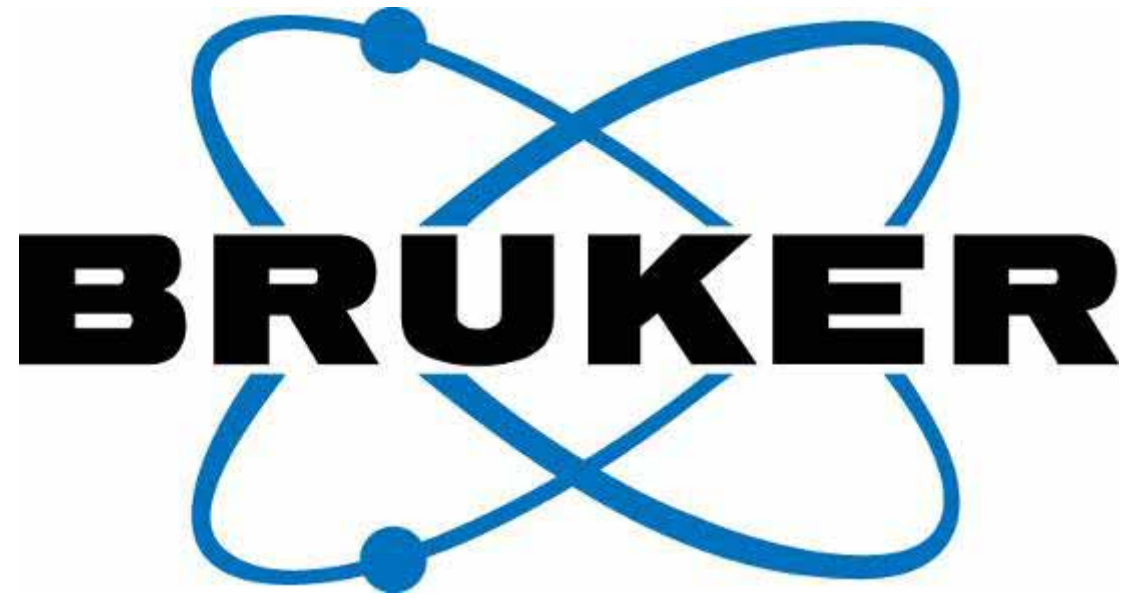
- The  $\mu$ XRF Spectrometer ARTAX features a fast multi-element mapping in the mm range for industrial and forensic samples
- The fast exchange of the excitation source (X-ray tube, collimator, polycap lens) allows optimization of spatial resolution and detection limits
- Bruker AXS continues its development in the field of  $\mu$ XRF spectrometry



# Thank you for attending!

Please type any questions you may have in the **Q&A** panel to the right of your screen and click **Send**.

Copies of this presentation and related microanalysis resource materials will be emailed to you.



[www.bruker-axs.com](http://www.bruker-axs.com)