Practical Spectrum Imaging: 
Rapid Collection for Routine Analysis
Welcome

Today’s Topics
- Introduction
- X-ray Detectors for Microanalysis
- HyperMap Applications
- μXRF Instrument Design - ARTAX
- ARTAX Element Mapping in Forensics and Industry
- Q&A

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

- **Bruker Daltonics**
  Mass Spectrometry

- **Bruker Optics**
  FTIR, NIR, Raman

**Bruker BioSpin**
(Affiliated privately held not part of Bruker BioSciences)
NMR, EPR, MRI

The innovative ultraflex™ III mass spectrometer

- S4 Pioneer
- D8 Discover
- XFlash 4010
- Recombinase Fragment with 3 DNA Strand – Dr. Yu Lau, University of Saskatchewan

- TENSOR 27 & HYPERION
  FT-IR Microscope

- AVANCE III
Bruker AXS Microanalysis

Microanalysis

Est. 2006

Est. 1991

Est. 1965
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$_2$ (ca. -190°C)
- 2 hours to cool down
- Technology from 1960’s
- Weight approx. 8 kg

XFlash® Detector
- SDD Technology
- no LN$_2$ (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

1st generation
XFlash®1000 1997

2nd generation
XFlash®2000 2000

3rd generation
XFlash®3001 2002

4th generation
XFlash®4010 July 2006

2007: 10th anniversary of the XFlash® Detector!
XFlash® Detector 4010

- 4th generation high resolution detector

- 10 mm² active area
- Energy resolution: ≤ 125 eV, specification at MnKα, 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)

<table>
<thead>
<tr>
<th>Count Rate (Kcps)</th>
<th>Si(Li)</th>
<th>XFlash®</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-2</td>
<td>129</td>
<td>129</td>
</tr>
<tr>
<td>2-8</td>
<td>131</td>
<td>129</td>
</tr>
<tr>
<td>8-14</td>
<td>133</td>
<td>129</td>
</tr>
<tr>
<td>14-20</td>
<td>138</td>
<td>129</td>
</tr>
<tr>
<td>20-42</td>
<td>149</td>
<td>129</td>
</tr>
<tr>
<td>42-62</td>
<td>161</td>
<td>129</td>
</tr>
<tr>
<td>62-110</td>
<td>191</td>
<td>129</td>
</tr>
<tr>
<td>110-150</td>
<td>NP</td>
<td>129</td>
</tr>
<tr>
<td>150-200</td>
<td>NP</td>
<td>130</td>
</tr>
<tr>
<td>200-300</td>
<td>NP</td>
<td>131</td>
</tr>
</tbody>
</table>
XFlash 4010 SDD
Resolution vs Count Rate

Resolution vs. Count Rate

Resolution [eV]

Count Rate [kcps]

Si(Li)

Xflash
Theoretical energy resolution of Kα lines for Si-based detectors

-8 eV at Mn/Kα
-17 eV at C/Kα
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

Output Count Rate (OCR) versus Input Count Rate (ICR)
for selected shaping times (SVE III)

Output Count Rate [kcps] versus Input Count Rate [kcps]

- "60 kcps"
- "130 kcps"
- "275 kcps"
XFlash® Family Of Detectors

XFlash 4010: 10mm², 125eV, Be Detection
XFlash 4030: 30mm², 133eV, B Detection

XFlash Quad 4040: 4 x 10mm², 125eV, Be Detection

XFlash Multi- Detectors
Multiple Detector Systems

Internal view chamber
Comparison: XFlash® with Si(Li)

- Si(Li) detector (30 mm²)
  - 138 eV resolution
  - 20,000 cps input
  - 60% dead time
  - **Duration:** 6.25 hours

- XFlash® 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 mm² Si(Li) needs 50x longer for the same result!

*Note:* For an XFlash 4030 a 30mm² SD, **Duration = ~ 8.75 minutes**
and for an XFlash 4010 a 10mm² 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°Ψ, 20nA
80Kcps @ 40%DT
ESPRIT: Multi-Point Analysis
Multi-Point Analysis

MAG: 500X HV: 15.0 kV WD: 10.0 mm

Region 1
Region 2
Region 3

50 µm
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

Area Fraction = 94.1%

Area Fraction = 2.5%

Area Fraction = 1.8%

Area Fraction = 1.5%
XFlash®, 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 µm resolution

Armin Gross
Bruker AXS Microanalysis GmbH
1. Introduction to μXRF
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

## µXRF vs EDS and XRF

<table>
<thead>
<tr>
<th>Technology</th>
<th>X-ray Microanalysis (EDS)</th>
<th>Micro X-ray Fluorescence Analysis (µXRF)</th>
<th>X-ray Fluorescence Analysis (XRF)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Capability</strong></td>
<td>High resolution element mapping in the sub-µm range</td>
<td>Non-destructive spatial investigation of element distribution</td>
<td>Elemental analysis of bulk samples</td>
</tr>
<tr>
<td><strong>Limitation</strong></td>
<td>Sample size limited by chamber</td>
<td>Analytical range of 10 µm to 10 mm</td>
<td>No information about spatial element distribution</td>
</tr>
</tbody>
</table>

- **nm - µm**
- **µm – mm**
- **mm - cm**
ARTAX

Introduction to µXRF

- **Micro X-ray fluorescence analysis (µXRF)**
  - 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 ~1 µm in diameter

- **Advantages for fast mapping**
  - Minimal focal spot size about 70 µm
  - 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

- keV -

x 1E3 Pulses

Ag  Sn  Sb  Ba
blue  green  red  = W  = Mo  = Cr
Glass Standard CRM 610
Low energy range

Pulses

blue = W  
green = Mo  
red = Cr
Specifications
- Si drift chamber detector (SDD)
- 10 mm² active area, Be window
- Electro-thermal cooling

Advantages
- Energy resolution < 160 eV (Mn-Kα 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 µm
- Classification initially done by EDS
Line Scan Application
Investigation of Solar Cells

<table>
<thead>
<tr>
<th>Layer</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr</td>
<td>5 nm</td>
</tr>
<tr>
<td>Al</td>
<td>200 nm</td>
</tr>
<tr>
<td>Mo</td>
<td>5 – 15 nm</td>
</tr>
<tr>
<td>CdTe</td>
<td>3000 nm</td>
</tr>
<tr>
<td>Glass body</td>
<td></td>
</tr>
</tbody>
</table>

- 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 µA
  - 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 $1.8 \times 1.8 \text{ mm}$
- Meas. points $100$
- Meas. time $5 \text{ s}$
- Total time $23 \text{ min}$
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 µXRF 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 µXRF 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.