EDXRD for Out-of-the-Box Crystal Orientation Measurement
Welcome

Host: Uwe Preckwinkel  
Armin Gross

Speakers

- **Uwe Preckwinkel**  
  Marketing & Sales Manager, XRD  
  Madison, WI, USA
- **Dr. Armin Gross**  
  Global Product Manager  
  Berlin, Germany

Topics

- Overview of Bruker Corporation
- Principles of energy-dispersive X-ray diffraction (EDXRD)
- Instrument design – hardware and software
- D2 CRYSO features, benefits and specifications
- Applications
- Summary
- Q & A
Overview of Bruker Corporation
Bruker Corporation

- The Bruker Corporation is one of the world’s leading analytical instruments companies, employing thousands of people worldwide
- Founded 1960
Bruker Corporation

- NMR
  - MR Imaging
  - EPR
- FT-IR
- FT-NIR
- Raman
- Terahertz Imaging
- MALDI-TOF MS
  - ESI Ion Trap MS
  - FT-MS
  - ESI-TOF MS
- SCD
- XRD
- XRF
- Micro Analysis
Operations & Centers of Excellence

**Madison, Wisconsin**
- Production/R&D
  - Life Science
  - Material Science

**Yokohama, Japan**
- Production/R&D
  - Material Science

**Delft, the Netherlands**
- Production/R&D
  - Life Science

**Karlsruhe/Germany**
- Production/R&D
  - Material Science
  - Lab & process automation
Our Mission

Growth in key markets

- Advanced materials research
- Industrial lab and process automation
- Life science / structural proteomics

through

- Technology leadership
- Excellence in customer support
Principles of EDXRD
Audience Poll

Please use your mouse to answer the question on your screen:

What method do you most frequently use to determine crystal orientation?

- In-house Laue camera
- In-house monochromatic XRD
- Outsourced Laue camera
- Outsourced monochromatic XRD
- None
Overview of Crystal Orientation Measurement

- **Area of applications**
  - Industrial production and processing of single crystals
  - Continuous testing of the lattice orientation related to the crystal surface

- **Requirements**
  - Crystal dimensions and masses vary significantly
  - Process related testing of the crystals
  - Tolerated deviation of the test varies from 0.01° to more than 0.1°
Laue Method

**Transmission Laue**
- The film is placed *behind* the crystal to record beams which are transmitted through the crystal.
- One side of the cone of Laue reflections is defined by the transmitted beam.
- The film intersects the cone, with the diffraction spots generally lying on an ellipse.

**Back-reflection Laue**
- The film is placed *between* the x-ray source and the crystal.
- The beams which are diffracted in a backward direction are recorded.
- One side of the cone of Laue reflections is defined by the transmitted beam.
- The film intersects the cone, with the diffraction spots generally lying on an hyperbola.

Laue Method

- Works well and is accurate

But...

- Complex sample mounting and alignment are required
- Slow when using film - expensive when using an area detector
Conventional Technology

- Conventional X-ray diffractometer
- Monochromatic X-ray beam towards single crystal
- Angle adjustment of one or several axis since reflection requirements of a lattice plane are fulfilled
- Crystal orientation = measured shift of angles
Energy-Dispersive X-ray Diffraction

- Single crystal
- Lattice plane
- Incident polychromatic X-ray beam
- Surface normal $n$
Energy-Dispersive X-ray Diffraction

- Single crystal
- Lattice plane
- Rotation angle
- Incident polychromatic X-ray beam
- Surface normal $n$
- $k$
Energy-Dispersive X-ray Diffraction

- Rotation angle
- Lattice plane
- Single crystal
- Incident polychromatic X-ray beam
- Surface normal n

- $E_1$
- $E_2$
- $k_1$
- $k_2$
Energy-Dispersive X-ray Diffraction

- Single crystal
- Lattice plane
- Incident polychromatic X-ray beam
- Rotation angle
- Energy dispersive detector
- Surface normal $n$
- $E_1$, $E_2$
- $k_1$, $k_2$
Energy-Dispersive X-ray Diffraction

- Rotation angle
- Lattice plane
- Single crystal
- Incident polychromatic X-ray beam
- Energy dispersive detector
- Surface normal $n$
- Energy dispersive detector
- Energy / keV
- $E_1$ and $E_2$

$\theta$ $k_1$ $k_2$
Energy-Dispersive X-ray Diffraction

- Rotation angle
- Lattice plane
- Single crystal
- Incident polychromatic X-ray beam
- Energy dispersive detector
- Surface normal n
Energy Dispersive X-Ray Diffraction – Orientation Angle

\[ E(\delta, \gamma, \varphi) = \frac{12.4}{2 \cdot d \cdot (\cos(\alpha) \cdot \sin(\delta) \cdot \cos(\gamma - \varphi) + \sin(\alpha) \cdot \cos(\delta))} \]

\( \delta, \gamma \) Orientation angles of the lattice plane normal (spherical coordinates)

\( \alpha \) Incident angle of the primary beam related to the crystal surface (constant)

\( \varphi \) Turning angle of the surface normal of the crystal

\( d \) distance of the lattice planes
Principles of Energy-Dispersive Detectors

- Energy-dispersive detectors show significantly different energy resolution
- Two types of nitrogen-free detection systems are available
  - PIN diode (25 mm²): 260 eV
  - Silicon Drift Detector XFlash® (30 mm²): 150 eV
- Benefits of XFlash®: enhanced peak fit for calculation of crystal orientation angles
The XFlash® offers stable energy resolution even at the highest count rates above 100 kcps.

Benefit: D2 CRYSO measurements according to the Laue method take only a few seconds.

- Count rate: 100.0 kcps
- FWHM \(M_{\text{nk1,2}}\): 147.6 eV
- Time: 2 s
- DT: 24 %
D2 CRYSO - Instrument Design

- Low power X-ray tube with high voltage generator
- X-ray optics, mono-capillary
- Energy dispersive detector with multi-channel analyzer
- Rotation sample table
- PC for control of the measurement, data recording and analysis
- Large samples as well as small samples
- Goniometer fixture as optional accessory
D2 CRYSO - Instrument Design

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D2 CRYSO - Instrument Design

Mono-capillary X-ray optics

Energy-dispersive XFlash® detector

Low power X-ray tube
Flat - or azimuthal - orientation: orientation of a scattering plane perpendicular to the main orientation

Measurement of a “help” plane with the same flat

δ (meas) <37°
Principle of Flat Determination – Measurement of Main Orientation

- Single crystal
- Polychromatic X-ray beam
- Main orientation e.g. (111)
- 2 \theta_1
- Detector at Pos. 1
- Chi circle drive
Principle of Flat Determination – Measurement of Flat Orientation

Single crystal

Main orientation e.g. (111)

Flat orientation e.g. (331)

Chi circle drive
Principle of Flat Determination – Measurement of Flat Orientation

Single crystal

Polychromatic X-ray beam

Flat orientation e.g. (331)

$2 \Theta_2$

Detector at pos. 2

Chi circle drive

Bruker AXS
D2 CRYSO - Software

**CRYSOCONTROL**
- Control of all hardware functions
- Energy and angle calibration
- Measurement, display and analysis of the energy spectra
D2 CRYSO - Software

- CRYSOCONTROL
  - Preset of all parameters
  - Definition of methods
D2 CRYSO - Software

CRYSOMEASURE
  • for routine operation
CRYSOMEASURE - for routine operation

- Selection of the method
- Start - automatic measurement and analysis
- Record and output of the results
# D2 CRYSO Specifications

<table>
<thead>
<tr>
<th>Specifications</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Instrument size (HxWxD, mm)</td>
<td>500 x 560 x 540</td>
</tr>
<tr>
<td>Weight</td>
<td>approx. 60 kg</td>
</tr>
<tr>
<td>Power consumption</td>
<td>150 W</td>
</tr>
<tr>
<td>Side window X-ray tube, Rh anode</td>
<td>max. 50 kV, 1mA</td>
</tr>
<tr>
<td>Mono capillary for beam collimation, focal spot size at sample</td>
<td>0.7 mm</td>
</tr>
<tr>
<td>XFlash Silicon Drift Detector, energy resolution thermo-electrically cooled, active area</td>
<td>&lt; 150 eV, 30 mm²</td>
</tr>
</tbody>
</table>
# D2 CRYSO Specifications

## Measuring parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max. specimen diameter</td>
<td>385 mm</td>
</tr>
<tr>
<td>Max. specimen thickness / height</td>
<td>140 (210) mm</td>
</tr>
<tr>
<td>Max. specimen weight</td>
<td>35 kg</td>
</tr>
<tr>
<td>Incident angle of the primary beam (adjusted ex works)</td>
<td>ca. 30°</td>
</tr>
<tr>
<td>Max. “off angle”</td>
<td>≤ 5°</td>
</tr>
<tr>
<td>Measurement error for orientation angles (2σ)</td>
<td>&lt; 0.02°</td>
</tr>
<tr>
<td>Max. deviation of the flat lattice plane from the crystal surface</td>
<td>≤ 37°</td>
</tr>
<tr>
<td>Measurement error for flat orientation angles</td>
<td>approx. 0.2°</td>
</tr>
</tbody>
</table>
Reference Sites - Europe

Research

- Institute for Crystal Growth, Berlin
  all kinds of single crystals
- Max-Planck- Institute for chemical physics of solid materials, Dresden
  exotic materials: \( \text{YbRh}_2\text{Si}_2 \), \( \text{Sr}_2\text{V}_3\text{O}_9 \) etc.
- High Magnetic Field Laboratory Dresden-Rossendorf
  exotic materials
- European Synchrotron Radiation Facility, Grenoble
  Si monochromator production

Industry

- leading suppliers of Ge, CaF\(_2\), SiC
- Production of industrial diamonds
D2 CRYSO
Applications
Please use your mouse to answer the question on your screen:

What types of single-crystal materials do you manufacture and/or analyze for crystal orientation? (Check all that apply.)

- Semiconductor (Si, Ge, GaAs, SiC, etc.)
- Optical (SiO₂, Al₂O₃, MgO, etc.)
- Metals (Au, Ni, Pt, etc.)
- Fluorides, Chlorides or Nitrides
- Other
D2 CRYSO Applications – Crystalline Materials

- Semiconductor substrates
  - Si, Ge, GaAs, GaP, InAs, ZnS, CdTe etc.
- Components for optical applications
  - SiO$_2$, LiF, CaF$_2$ etc.
- Monochromator and analyzer crystals
  - SiO$_2$, Ge, Si, Graphite, etc.

The D2 CRYSO is not suited for oscillator Quartz crystals (e.g. watches)
# D2 CRYSO Applications - Semiconductor Single Crystals

<table>
<thead>
<tr>
<th>Crystal</th>
<th>Chem. Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon</td>
<td>Si</td>
</tr>
<tr>
<td>Gallium Arsenide</td>
<td>GaAs</td>
</tr>
<tr>
<td>Germanium</td>
<td>Ge</td>
</tr>
<tr>
<td>Silicon Carbide</td>
<td>SiC</td>
</tr>
<tr>
<td>Indium Phosphate</td>
<td>InP</td>
</tr>
</tbody>
</table>
# D2 CRYSO Applications – Metallic Single Crystals

<table>
<thead>
<tr>
<th>Crystal</th>
<th>Chem. Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gold</td>
<td>Au</td>
</tr>
<tr>
<td>Platinum</td>
<td>Pt</td>
</tr>
<tr>
<td>Nickel</td>
<td>Ni</td>
</tr>
</tbody>
</table>
D2 CRYSO Applications – Fluorides, Nitrides and Others

<table>
<thead>
<tr>
<th>Crystal</th>
<th>Chem. Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calcium Fluoride</td>
<td>CaF&lt;sub&gt;2&lt;/sub&gt;</td>
</tr>
<tr>
<td>Graphite</td>
<td>C</td>
</tr>
<tr>
<td>Magnesium Fluoride</td>
<td>MgF&lt;sub&gt;2&lt;/sub&gt;</td>
</tr>
<tr>
<td>Silver Chloride</td>
<td>AgCl</td>
</tr>
<tr>
<td>Zinc Telluride</td>
<td>ZnTe</td>
</tr>
</tbody>
</table>
D2 CRYSO Applications – Optical Crystals: Oxides

<table>
<thead>
<tr>
<th>Crystal</th>
<th>Chem. Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corundum - Aluminum Oxide</td>
<td>( \text{Al}_2\text{O}_3 )</td>
</tr>
<tr>
<td>Gadolinium Titanate</td>
<td>( \text{Gd}_2\text{Ti}_2\text{O}_7 )</td>
</tr>
<tr>
<td>Lanthanum Aluminate</td>
<td>( \text{LaAlO}_3 )</td>
</tr>
<tr>
<td>Magnesium Aluminate</td>
<td>( \text{MgAl}_2\text{O}_4 )</td>
</tr>
<tr>
<td>Magnesium Oxide</td>
<td>( \text{MgO} )</td>
</tr>
<tr>
<td>Magnesium Silicate</td>
<td>( \text{Mg}_2\text{SiO}_4 )</td>
</tr>
<tr>
<td>Neodymium Gallium Oxide</td>
<td>( \text{NdGaO}_3 )</td>
</tr>
<tr>
<td>Silicon Dioxide</td>
<td>( \text{SiO}_2 )</td>
</tr>
<tr>
<td>Sanidine</td>
<td>( (\text{K,Na})(\text{Si,Al})_4\text{O}_8 )</td>
</tr>
<tr>
<td>Strontium Lanthanum Aluminate</td>
<td>( \text{SrLaAlO}_4 )</td>
</tr>
<tr>
<td>Strontium Titanate</td>
<td>( \text{SrTiO}_3 )</td>
</tr>
<tr>
<td>Titanium Dioxide</td>
<td>( \text{TiO}_2 )</td>
</tr>
<tr>
<td>Zirconium Dioxide</td>
<td>( \text{ZrO}_2 )</td>
</tr>
</tbody>
</table>
Application – Si and CaF$_2$ Crystals

Task:
- Surface orientation measurement of Si and CaF$_2$ crystals

Spectra:
- PIN diode: broad blue peaks
- XFlash: narrow red peaks

Challenge:
- Materials like Ge show strong fluorescence peaks, which discriminate the formation of Laue peaks
Application – Si and CaF$_2$ Crystals

Result - PIN diode measurements:
Limitation in max. count rate requires reduction of excitation current
⇒ long measurement times

<table>
<thead>
<tr>
<th>PIN diode detector</th>
<th>XFlash detector</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PIN diode detector</td>
</tr>
<tr>
<td>Si (400)</td>
<td>I (µA)</td>
</tr>
<tr>
<td>600</td>
<td>2500</td>
</tr>
<tr>
<td>CaF$_2$ (400)</td>
<td>170</td>
</tr>
<tr>
<td>CaF$_2$ (333)</td>
<td>170</td>
</tr>
</tbody>
</table>

Result - XFlash measurements:
Maximum excitation current applied
⇒ measurement times reduced by a factor of about 20
Summary

- Conventional instrument

- D2 CRYSO
  - Energy-dispersive X-ray diffraction suitable for process related measurements of single crystal orientations
  - Small size
  - Easy to use
  - Short measuring times
# Benefits of EDXRD Technique

| Fixed arrangement of tube - detector | ➥ compact design  
|                                   | ➥ portable bench top device  
|                                   | ➥ reduced purchasing costs |
| No goniometer                     | ➥ no fixation of the crystal required  
|                                   | ➥ samples are positioned and aligned  
|                                   | ➥ analysis of large samples possible |
| Fully protected X-ray device      | ➥ Radiation protection guaranteed  
|                                   | ➥ No installation in controlled environment |
| Automatic measurement             | ➥ Short measuring times  
|                                   | ➥ automatic measurement and analysis  
|                                   | ➥ Easy-to-use during routine work |
Any Questions?

Please type any questions you may have in the Q&A box and then click Send.
Thank you for attending!

Please take a moment to complete the brief survey on your screen. Your feedback is very important to us.

Copies of this presentation and related EDXRD resource materials will be emailed to you.
See us at:

ICMCTF – San Diego, CA – Apr 28 - May 2
SEMICON West – San Francisco, CA – July 15-17
Denver X-ray Conference – Denver, CO – Aug 4-8
FXMA West – Portland, OR – Sep 16-18