Outline

- X-rays interactions with the matter
- Fundamentals of diffraction
- Powder diffraction methods
  - Size / strain analysis
  - Search / Match, structure determination
  - Quantitative analysis, whole pattern fitting
- X-ray parallel beam methods
  - Thin film crystallographic orientation
  - Glancing / Grazing angle XRD methods
- Texture - preferred orientation methods
- Residual stress analysis methods
- High resolution XRD methods
  - Rocking curve analysis
  - Reciprocal lattice mapping
- X-ray reflectivity methods
- X-ray fluorescence methods
- X-ray analysis summary
- Comparison with other techniques
- Quick guide to the FS-MRL x-ray analysis facilities
- Recommended literature.
**X-ray interactions with matter**

- **Coherent scattering** (Thompson scattering / diffraction):
  - Incident photon $h\nu_0$ interacts with e- with no energy loss and no phase change.

- **Incoherent scattering** (Compton scattering):
  - e- absorbs incident energy $h\nu_0$ (excited photoelectron);
  - Part of the energy is emitted at different energy $h\nu_1$ and different phase.

- **Fluorescence**:
  - K/L shell e- absorb incident energy $h\nu_0$;
  - Outer shell e- "cascade" down filling the "holes" causing secondary photons emission ($h\nu_2$).

- **Photoelectron emission**:
  - Energy is used to eject electron e- with kinetic energy $h\nu_0 - E_B$ (binding energy).

- **Auger electron emission**:
  - Incident $h\nu_0$ used to eject e- from atom;
  - 2nd e- "drops" to lower levels to fill the "hole" and a photon is emitted;
  - The emitted photon is absorbed by valance e-, which ionizes and leaves the atom.

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**X-ray interactions with matter**

- **Coherent scattering** (Diffraction, Thompson or Rayleigh scattering):
  - Incoming photon
  - Oscillating electron
  - Scattered photon
  - No loss of energy.

- **Incoherent scattering** (Compton scattering):
  - Incoming photon
  - Energy is partially transferred to electron
  - Scattered photon (energy loss).

- **Fluorescence**:
  - E$_0$ = E$_L$ + E$_K$
  - E$_L$ = Energy of inner shell electron
  - E$_K$ = Energy of outer shell electron

- **Auger electron**:
  - Incoming photon excites inner shell electron
  - Excitation energy is transferred to outer electron
  - Electron ejected from atom (Auger electron)
Fundamentals of diffraction

"Real" space
Set of planes

Reciprocal space
Point

(h k l)

M. von Laue 1879-1960
X-rays from crystals, 1912.
Fundamentals of diffraction

“Real” space \( \rightarrow \) Reciprocal space

- **Elastic (Thompson’s) scattering**
  - \( 2d \sin \theta = n \lambda \)

- **Bragg’s law**
  - \( 2d \sin \theta = n \lambda \)
  - \( q = k_1 - k_0 \)
  - \( q \): scattering vector
  - \( q = (4\pi\lambda)\sin\theta \)

- **Ewald’s sphere**
  - \( q \)
  - \( k_0 (= \text{radius}) \)

- **X-ray source**

- **Detector**

- **Incident beam**

- **Diffused beam**

**References:**
- 1862–1942
- 1890–1971

**Authors:**
- Paul P. Ewald
  - 1888–1955
Fundamentals of diffraction

"Real" space

Detector scan

$\Delta(2\theta)$

$\Delta(2\theta/\omega)$

20/\omega scan

Rocking curve (sample) scan

$\Delta(\omega)$

$h k l$

Scattering vector

Incident beam

Diffracted beam

Detector Scan: around Ewald's sphere

$\Delta(2\theta)$

$\Delta(2\theta/\omega)$

$\Delta(\omega)$

Rocking curve scan: \perp to scattering vector

20/\omega scan: radial, // to scattering vector

Reciprocal space

$\omega$

$\omega$

Detector scan

$\Delta(2\theta)$

$\Delta(2\theta/\omega)$

20/\omega scan

Rocking curve scan: \perp to scattering vector

$\Delta(\omega)$

$h k l$

Scattering vector

Incident beam

Diffracted beam

Detector Scan: around Ewald's sphere

$\Delta(2\theta)$

$\Delta(2\theta/\omega)$

$\Delta(\omega)$

Rocking curve scan: \perp to scattering vector

20/\omega scan: radial, // to scattering vector

Fundamentals of diffraction

"Real" space

Detector scan

$\Delta(2\theta)$

$\Delta(2\theta/\omega)$

20/\omega scan

Rocking curve (sample) scan

$\Delta(\omega)$

$h k l$

Scattering vector

Incident beam

Diffracted beam

Detector Scan: around Ewald's sphere

$\Delta(2\theta)$

$\Delta(2\theta/\omega)$

$\Delta(\omega)$

Rocking curve scan: \perp to scattering vector

20/\omega scan: radial, // to scattering vector

Reciprocal space

$\omega$

$\omega$

Detector scan

$\Delta(2\theta)$

$\Delta(2\theta/\omega)$

20/\omega scan

Rocking curve scan: \perp to scattering vector

$\Delta(\omega)$

$h k l$

Scattering vector

Incident beam

Diffracted beam

Detector Scan: around Ewald's sphere

$\Delta(2\theta)$

$\Delta(2\theta/\omega)$

$\Delta(\omega)$

Rocking curve scan: \perp to scattering vector

20/\omega scan: radial, // to scattering vector

• Defects, material quality
• Mosaicity
• Texture
• Texture strength

• Glancing angle
• Constant sampled volume
• Phase analysis
• Grain size analysis
• Unit cell determination
• Stress analysis
• Lattice distortion, strain
• Composition, alloying
• Defects, material quality
• Mosaicity
• Texture
• Texture strength
Typical contents from XRD pattern (diffractogram)

- **Peak position:** identification, structure, lattice parameter
- **Peak width:** crystallite size, strain, defects
- **Peak area or height ratio:** preferred orientation
- **Peak tails:** Diffuse scattering, point defects
- **Background:** amorphous contents

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**Powder diffraction methods**

- Crystalline? Amorphous?
- What elements, compounds, phases are present?
- Structure? Lattice constants?
- Strain?
- Grain sizes? Grain orientations?
- Is there a mixture? What %?
- Powders, bulk materials, thin films, nanoparticles, soft materials.
Bragg-Brentano focusing configuration

X-ray source

Secondary Optics: Scatter and soller slits

Detector

Single crystal monochromator (\(\lambda\))

Detector rotation (2\(\theta\))

Angle of incidence (\(\omega\))

specimen

Divergence slit

Receiving slit

Focusing circle (variable during measurement)

Diffractometer circle (fixed during measurement)

Focusing circle

Sample height positioning is critical

Divergent beam not good for grazing incidence analysis
Instrumentation: powder diffraction

- **Cu x-ray source**
- **Soller slits**
- **Divergence slit**
- **Sample**
- **2θ**
- **θ**
- **Scatter slit**
- **Detector**
- **Δ2θ ~ 0.2°**
- **Curved graphite monochromator**

**Rigaku D/Max-b XRD system**

**CMM XRD facilities**

Instrumentation: stress, phase analysis

- **Bragg-Brentano configuration** with programmable slits
- **X-ray source:** Cu, line focus
- **Optional filter or attenuator**
- **Vertical mask**
- **Programmable divergence slit** [≥ (1/32) deg]
- **Programmable receiving slits**
- **Detectors**
- **Curved graphite monochromator**

**Panalytical X'pert #2 XRD system**

Programmable slits can be used for applications requiring limited or constant illuminated area in the sample.
Crystallite size analysis

**Scherrer’s equation:**

\[
\text{Size} = \frac{k \cdot \lambda}{\cos(\theta) \cdot (\text{FWHM})}
\]

- \( k \): shape factor (0.8-1.2)
- \( \lambda \): x-ray wavelength
- FWHM: full width at half maximum (in radians)

Not accounting for peak broadening from strain and defects

Measured along the specific direction normal to the (hkl) lattice plane given by the 2\( \theta \) peak position

Peak width (FWHM)

Peak position 20

FWHM vs. integral breadth

It does not take into account the lower part of the profile

\[ \text{Int. breadth} = \frac{(\text{total area})}{(\text{peak height})} \]

The square-topped profile has the same area and peak height as the measured curve.
Peak shape analysis

Peak fit:
- Gaussian
- Lorentzian
- Pearson-VII (sharp peaks)
- Pseudo-Voigt (round peaks)

Information from fit:
- Position
- Width (FWHM)
- Area
- Deconvolution
- Skewness

Correction for instrument resolution

Use FWHM curve as a function of 2θ from standard sample (NIST LaB₆)
- specific for each diffractometer

FWHM: β

D: deconvolution parameter

D : 1 (~ Lorentzian)

D : 2 (~ Gaussian)

β₁.₅ = (β_{meas})₁.₅ – (β_{instr})₁.₅

Software: MDI’s Jade + 8.0
### Potential artifacts in size determination

#### FWHM: $\beta$

$\beta^D = (\beta_{\text{meas}})^D - (\beta_{\text{instr}})^D$

<table>
<thead>
<tr>
<th>Measured peak width ($^\circ$)</th>
<th>Size, nm $D = 1$ (Lorentzian)</th>
<th>Size, nm $D = 1.5$</th>
<th>Size, nm $D = 2$ (Gaussian)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.3</td>
<td>56.4</td>
<td>38.0</td>
<td>32.6</td>
</tr>
<tr>
<td>0.5</td>
<td>24.2</td>
<td>19.2</td>
<td>17.7</td>
</tr>
<tr>
<td>0.75</td>
<td>14.1</td>
<td>12.0</td>
<td>11.5</td>
</tr>
<tr>
<td>1</td>
<td>10.1</td>
<td>8.8</td>
<td>8.6</td>
</tr>
<tr>
<td>1.5</td>
<td>6.3</td>
<td>5.8</td>
<td>5.7</td>
</tr>
<tr>
<td>2</td>
<td>4.6</td>
<td>4.3</td>
<td>4.2</td>
</tr>
</tbody>
</table>

Assume:
- Instrument resolution ~ 0.15$^\circ$
- $2\theta = 40^\circ$
- Cu radiation

- Large difference (up to 48%) for narrow peaks (large sizes)
- Smaller difference (~ 10%) for broad peaks (small sizes)

### Strain effects in diffraction lines

- **Macrostrain**
  - Uniform strain (lattice distortion)
  - Peak position shift (lattice constant change)
  - No strain

- **Microstrain**
  - Nonuniform strain (lattice distortion)
  - Peak width change
  - FWHM

- $\Delta(2\theta)$
- $d$
**Size and strain in peak shape analysis**

\[(FWHM)\cos(\theta) = \frac{k\lambda}{(size)} + \text{(strain)}\sin(\theta)\]

Intercept \(\sim \frac{1}{(size)}\)

Slope \(\sim\) micro strain

(Rieke, 1932-)

**Other methods of grain size analysis**

**Rietveld refinement method:**
- Refines the whole diffraction pattern (including background)
- Needs detailed data over a wide angular range
- Gives one average size value
- Works better for powder samples (not well with films with strong preferred orientation)
- Data processing (refinement and “play” with parameters) is time consuming.

**Warren-Averback method:**
- Standard sample -> Instrumental Broadening -> Correct measured peaks assuming “error”-type of function (main assumption) \(I \sim \exp(-p\phi^2)\) -> Use Scherrer’s equation

**Jones’ method:**
- Measured profile = convolution (“pure”, instrument)
- Use Fourier integrals for all the profiles (measured, “pure”, instrument)
- Use ratio of the integrals to determine sizes.
Whole pattern fitting and structure refinement

- Bragg-Brentano or parallel beam x-ray analysis.
- Powders, polycrystalline films or nanostructures, mineralogy, cements, ceramics, pharmaceutics, polymers, biomaterials.
- Identification, quantification (w%) and structure determination of mixtures, impurities, multiple phases and amorphous fractions.
- Quantification of crystallinity, texture, twinning, grain size and strain.
- Pattern indexing, lattice parameters determination, unit cell refinement, atomic positions, bonds distances and angles.
- Pattern simulation and structure refinement (Rietveld analysis).

Results obtained from the measured pattern:

- Search / Match: LiNi0.7Co0.3O2, major (minor: C graphite)
- Structure: Hexagonal R-3m (166) a=2.86285 Å, c=14.17281 Å
- Unit cell volume: 100.6 Å³
- Density: 4.8583 g cm⁻³
- Linear Absorption Coefficient: 385.5 cm⁻¹

Average bond distances (Rietveld):
- Ni-O: 1.9570 Å, Ni-Li: 2.8729 Å
- Co-O: 1.9570 Å, Co-Ni: 2.8629 Å
- Li-O: 2.1117 Å
- Li-Li: 2.8729 Å
- Ni-Ni: 2.8629 Å

Quantitative analysis:
- LiNi0.7Co0.3O2: 82.9 w%, C: 17.1 w%

Crystallite size:
- > 1000 Å

Averaging:
- 0.05%
New: S/M assisted by whole pattern fitting

Multi-phase mixture powder data

Whole pattern fitting using possible pdf hits in search for best phase id’s

“R” is being minimized (whole pattern fitting)
New: S/M assisted by whole pattern fitting

Unmatched peaks are now fitted

New pdf hits are now automatically added

Whole pattern fitted

3 phases matched: Rutile, Hematite, Anatase

R: 45.5 w%, XS: 463 nm
H: 31.0 w%, XS: 273 nm
A: 23.5 w%, XS: 28 nm
X-ray parallel beam methods

Rough, irregular surfaces

Film / Substrate systems

Glancing / grazing angle applications. Phase, stress gradients (depth profiles)

Parallel beam configuration

Negligible sample displacement issues (rough and curved samples OK)

Specific optics to maximize intensities

X-ray source

Primary Optics: mirror, slits, lens

Parallel beam

Parallel plates collimation

Specimen

Detector

Single crystal monochromator (λ)

Detector rotation (2θ)

Excellent for glancing angle (fixed ω) applications
Instrumentation: thin film, texture, stress analysis

Parallel plate collimator configurations

Top view

Primary optics option 1: Crossed-slit collimator

Optional filter or attenuator

X-ray source: Cu, line or point focus

Primary optics option 2: x-ray lenses

Primary optics option 3: programmable divergence slit + programmable attenuator

Flat graphite monochromator

Detector

0.27-parallel plates collimator

\( \phi \), \( \theta \)

\( \Delta \theta \sim 0.03-0.2^\circ \)

CMM instrument: Panalytical X’pert #2 XRD system

Thin film orientation analysis

Example:

\( \phi \) scan (in-plane surface direction)

\( \theta/\phi \) scan (surface normal direction)

Data: Shin, Petrov et al, UIUC

Cube on cube epitaxy:

(001)$_{\text{TaN}}$/[001]$_{\text{MgO}}$

(100)$_{\text{TaN}}$/[100]$_{\text{MgO}}$
Glancing angle x-ray analysis

- Conventional Bragg-Brentano configuration: $2\theta - \omega$ scans probe only grains aligned parallel to the surface.
- Parallel-beam grazing incidence configuration: $2\theta$ scans probe grains in all directions.

X-ray penetration depth vs. angle of incidence

- Type of radiation
- Angle of incidence
- Material ($Z$, $A$, $\rho$, $\mu$)

Low angle region
X-ray penetration and information depths

(1) Penetration depth \( \tau_{1/e} \):
   depth for intensity \( I/e \approx 37\% I_0 \)
   \[ \tau_{1/e} = (\sin \alpha) / \mu \]

(2) Penetration depth \( \tau_63 \):
   depth for \( A_{GIXRD} = 1-1/e \) (~ 63%, \( \mu_k c = 1 \))
   \[ \tau_{63} = 1 / \mu_k c = \frac{\sin \alpha \sin(2\theta - \alpha)}{\mu \sin \alpha + \sin(2\theta - \alpha)} \]
   Bragg peak \( 2\theta \) dependent penetration depth!

(3) Information depth \( \tau \):
   weighted average of sampling depth
   \[ \tau = \frac{1}{\mu_k c} + \frac{1}{1 - \exp(\mu_k c)} \]
   It will not exceed the thickness value!

(4) Penetration depth \( \tau_{1/e} \) for \( \alpha \sim \alpha_c \):
   Includes correction with refractive index
   \[ \tau_{1/e} = \sqrt{\frac{2\lambda}{4\pi}} \left[ (\alpha^2 - \alpha_c^2) + \beta^2 \right]^{1/2} - (\alpha^2 - \alpha_c^2) \]

Excel spreadsheet calculator available at:
http://www-ssrl.slac.stanford.edu/materialscatter/gixs-calculator.xls

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### Table: X-ray penetration and information depths

<table>
<thead>
<tr>
<th>Angle of incidence (°)</th>
<th>Penetration depth (( \tau_{1/e} )) (nm)</th>
<th>Information depth (( \tau )) (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.9</td>
<td>298</td>
<td>180 - 182</td>
</tr>
<tr>
<td>1.8</td>
<td>595 (mtl)</td>
<td>212 - 214</td>
</tr>
<tr>
<td>2.7</td>
<td>892 (mtl)</td>
<td>224 - 226</td>
</tr>
</tbody>
</table>

From: M. Birkholz, "Thin Film Analysis by X-ray Scattering" Wiley-VCH 2006
Grazing angle vs. Bragg-Brentano configurations

Grazing incidence analysis with parallel beam optics
- Detect grains in various orientations (including tilted to the sample surface).
- Fixed incident angle: constant probed depth in the sample during analysis.
- Low angle of incidence:
  - high sensitivity to (ultra) thin films;
  - avoid artifacts from substrates;
  - can be used for “depth profiling” at different incidences.
- Slightly lower 2θ resolution: ok for broad peaks.
- Thin film collimation optics: enhanced sensitivity to thin films.
- Parallel beam: insensitive to sample displacement errors.

Conventional analysis with focusing configuration
- Detect only grains with orientation parallel to the sample surface.
- Sample probed depth may vary during the analysis.
- Lower sensitivity to (ultra) thin films; substrate artifacts are problem.
- Very good 2θ resolution and well-known mathematical formalism.
- Typical optics are ideal for powder samples and thick films with no preferred orientation.
- Focusing configuration: very sensitive to sample displacement errors.

Example:
Poly-Si (~ 100 nm)
Si(001) substrate

Glancing angle x-ray analysis

Conventional 2θ/0 XRD

Substrate
Grazing incidence x-ray analysis

- Grazing incidence x-ray scattering (GIXS) analysis uses low angle of x-ray beam incidence relative to the surface in order to enhance diffraction from very thin layers.
- Analysis of ultra-thin films (<10 nm), nanostructures or topmost surface regions of the material.
- Allows structure determination and quantification of lattice parameters, rocking curve widths and crystallite size along the surface direction (different from conventional XRD, which probes surface normal directions).

GIXS from a ScN thin film on MgO (001):
- The results allow the determination of in-plane lattice parameter and crystallite size.
- Notice the differences in the relative intensities of the film and substrate peaks at different in-plane angles of incidence $\alpha$.

Texture and preferred orientation methods

Texture / Preferred orientation: anisotropy of grain orientation distribution.

Are the grain orientations distributed randomly? Or is there a preferred orientation?

What is the preferred orientation?

What is the % of random grains?

How strong and sharp is the texture?

For film / substrate systems: what is the crystallographic orientation between the substrate and the layers?
Determination of preferred orientation

Crystalline grains in a material may be preferentially distributed along one orientation (preferred orientation). This may complicate the analysis using conventional XRD methods derived from powder techniques.

Methods:

1. Compare relative peak height or area obtained from a $2\theta/\theta$ scan with the expected relative intensity from a standard (same material) with no preferred orientation (~ powder): Lotgering factors.

2. Use the relative intensity method above combined with March-Dollase preferred orientation corrections to obtain % grains that are more oriented in a specific direction.

3. Use the rocking curve analysis of a strong film diffraction. The width of the rocking curve peak is used as texture parameter.

4. Perform pole figures to determine the presence of grains of a certain orientation in all sample directions.

5. Use multiple pole figures from multiple orientations to obtain Orientation Distribution Functions: % of grain orientation distributions in all wafer directions.

X-ray pole figure analysis of textured materials

Texture results from a rolled Cu foil

- Texture orientation and quantification.
- Volume fraction of textured grains, twinning and random distributions.
- Texture strength and sharpness.
- Crystallographic orientation.
- Crystallographic relationship between layers and substrate.

Orientation distribution function (ODF)

Data: Sardela, UIUC
**Pole plot projections**

- **Wulff projection** (stereographic or equal-angle projection)
  - Side view (normal to equatorial plane)
  - Top view (parallel to equatorial plane)
  - Equatorial plane
  - \( OW = r \tan(\psi/2) \)

- **Schmidt projection** (equal-area projection)
  - Side view (normal to equatorial plane)
  - Top view (parallel to equatorial plane)
  - Equatorial plane
  - \( OS = 2r \tan(\psi/2) \)

**Basics of pole figure analysis**

- **Surface normal**
  - \( \phi \)
  - \( hkl \)

- **Example:** (100) cubic crystal

- **Pole figure plot**
  - \( \phi \) (azimuthal rotation)
  - \( \psi \) (radial / tilt)

- **(100) Pole figure**
  - Azimuth \( \phi = 0, 90^\circ, 180^\circ, 270^\circ \)
  - Tilt \( \psi = 0, 90^\circ \)
  - \( \psi: [100],[110] \)

- **(111) Pole figure**
  - Azimuth \( \phi = 45^\circ, 135^\circ, 225^\circ, 315^\circ \)
  - Tilt \( \psi = 54.7^\circ \)
  - \( \psi: [100],[111] \)

- **(110) Pole figure**
  - Azimuth \( \phi = 0, 90^\circ, 180^\circ, 270^\circ, 45^\circ, 135^\circ, 225^\circ, 315^\circ \)
  - Tilt \( \psi = 45^\circ, 90^\circ \)
  - \( \psi: [100],[110] \)
Basics of pole figure analysis

Example: (111) cubic crystal

Pole figure plot

ψ (radial / tilt)
φ (azimuthal rotation)

(100) Pole figure
ψ
Azimuth
φ = 30°, 150°, 270°
Tilt ψ = 54.7°
ψ: [111],[100]

(111) Pole figure
ψ
Azimuth
φ = 90°, 210°, 330°
Tilt ψ = 0, 70.5°
ψ: [111],[111]

(110) Pole figure
ψ
Azimuth
φ = 60°, 120°, 240°, 300°, 90°, 210°, 330°
Tilt ψ = 35.3°, 90°
ψ: [111],[10]

Fiber texture analysis

Example: Cu thin film on Si (001) substrate

Conventional 2θ/θ scan:

Cu(111)
Grains:
56 nm

Cu(222)

Fiber texture analysis

Example: Cu thin film on Si (001) substrate

Conventional 2θ/θ scan:

Cu(111)
Grains: 56 nm
(200)
(220)
(311)
Cu(222)

All grains are <111> oriented along the film growth direction.

Grains are randomly oriented along the surface

(111) Pole figure:

ψ = 70.53°

ψ = 54.74°

(200) Pole figure:

ψ = 0

(200) Pole figure:

ψ = 70.53°

ψ = 54.74°

Example: Cu thin film on Si (001) substrate

(200)
(220) (311)

All grains are <111> oriented along the film growth direction.

Grains are randomly oriented along the surface

Fiber texture analysis

(111) Pole plot
Cu film on Si (001) substrate

Detector
X-ray source
Sample

 Texture direction
Random

Fiber texture:

Random

<111>
HWHM: 1.09° (texture sharpness)

3.5% <5713>+<112>+<225> twinning
4.6% <511> twinning
9.0% random grains

Data after background subtraction
Residual stress analysis methods

Residual stress?
How much? (MPa – GPa)
Type?
Direction(s)?
Stress gradients?

X-ray analysis of residual stress

- Quantification of residual stress.
- Compressive (-) and tensile (+) stress.
- Crystallographic orientation of stress.
- Sin²ψ and RIM methods.
- ψ and ω scan methods.
- New glancing angle method (texture).
- Determination of stress tensor.
- Requires crystallinity (no amorphous).

Stress results from a steel sample

Data: Sangid et al, UJIC
**Residual stress analysis: the \( \sin^2 \psi \) method**

\[
\varepsilon_\psi = \frac{a_\psi - a_\sigma}{a_\sigma} = \frac{1 + \nu}{E} \sigma_\psi \sin^2 \psi - \frac{2\nu}{E} \sigma_\beta
\]

\[a_\psi = \frac{a_\sigma \sigma_\psi}{E} \left( (1 + \nu) \sin^2 \psi - 2\nu \right) + a_\sigma = \delta \sin^2 \psi + \xi\]

\[a_\sigma = \xi + \frac{2\delta\nu}{1 + \nu} = 4.533 \text{ Å}\]

\[\sigma_\phi = \frac{E\delta}{2\delta\nu + (1 + \nu)\xi} = -1.47 \text{ GPa (Compressive)}\]

**High resolution XRD methods**

**Single crystals:**

Accurate measurements of \( a, b, c, \alpha, \beta, \gamma \)

Detailed peak shapes: defects, mosaicity.

**Film / substrate epitaxial systems:**

Measure small variations \( \Delta a, \Delta c, \ldots (~ 10^{-5}) \).

Measure layer tilts \( \Delta \phi, \ldots \)

Detailed peak shapes: defects, strain, mosaicity.
Instrument resolution in reciprocal space

Beam angular divergence, detector acceptance and diffractometer sampling volume

Angular acceptance of the secondary optics

Diffractometer sampling volume

Ewald sphere

Primary beam divergence (from primary optics)

Incident beam

Diffracted beam

Sample

Instrumentation: high resolution configuration

High resolution configuration with mirror and 4-reflections monochromator

4-reflection Ge(220) monochromator

x-ray mirror

x-ray source

3-bounce analyzer crystal

Programmable attenuator

“Lower” detector (triple axis: 12 arc-sec acceptance)

“Upper” detector (open: < 1º acceptance)

Crossed slits (variable slit+mask)

Variable slit

Variable mask

Sample

Delta theta = 12 arc-sec

Delta lambda/lambda = 5x10^-5

Line focus;
Parabolic x-ray mirror;
4-reflection monochromator (12 arc-sec resolution);
Open detector or analyzer crystal.

CMM instrument: Panalytical X’pert #1 XRD system
High resolution x-ray analysis

- High resolution methods using multi-reflection monochromator and possibly analyzer crystal ($\Delta\theta = 0.003^\circ$).
- Sensitive to lattice distortions within $10^{-5}$.
- Rocking curve analysis.
- Film thickness measurements.
- Strain relaxation and lattice parameter measurements.
- Determination of alloy composition and superlattice periods.
- Dynamical scattering simulation to determine composition variations and interface smearing in heterostructures.

Single crystals
Epitaxial films
Heterostructures
Superlattices
Quantum dots

Instrumentation: high-resolution configuration

Hybrid Mirror
(2-bounce monochromator + X-ray mirror)

3-bounce analyzer crystal

“Upper” detector
(open: $< 1^\circ$ acceptance)

“Lower” detector
(triple axis: 12 arc-sec acceptance)

Programmable attenuator

High resolution configuration with hybrid mirror

Line focus:
- Hybrid mirror (30 arc-sec resolution).
- Open detector or analyzer crystal.

CMM instrument: Panalytical X'pert #2 XRD system

InAs quantum dots on GaAs
Data: Wu et al, UCB

SiGe / Ge superlattice
Data: Sardela, UCB

InAs / GaAs multilayer
Data: Zhang et al
High resolution x-ray analysis

Example: strained \( \text{In}_x\text{Ga}_{1-x}\text{As} \) on GaAs (001) substrate

Lattice structure

Data: Sardela
Sample: Highland, Cahill, Coleman et al., UIUC

Takagi-Taupin dynamical scattering simulation

Data: Sardela
Sample: Highland, Cahill, Coleman et al., UIUC
High resolution reciprocal space mapping

No strain relaxation:
\[ a_u = a_s \]

Strain relaxation:
\[ a_u \neq a_s \]

- High resolution reciprocal lattice mapping requires multi-reflection monochromator and analyzer crystal in order to separate strain from mosaicity.
- Sensitive to lattice distortions within \( 10^{-5} \).
- Accurate lattice parameter determination (in and out of plane).
- Determination of strain and composition variations, strain relaxation, mosaic size and rotation, misfit dislocation density, nanostructure dimensions, lattice disorder and diffuse scattering.

**Layer structure**

- Strained Si (top layer, very thin)
- Relaxed Si\(_{1-x}\)Ge\(_x\) (thick, many microns)
- Si(001) substrate

**Lattice structure**

- Strained Si
- Relaxed Si\(_{1-x}\)Ge\(_x\) (virtual substrate)
- Si(001) substrate

**Example: strained Si layer on Si\(_{1-x}\)Ge\(_x\) / Si substrate**

**Reciprocal lattice**

- (004) Strained Si
- (224) Si substrate
- Si\(_{1-x}\)Ge\(_x\)

\[ \Delta q_{001} \]
\[ \Delta q_{110} \]
High resolution reciprocal space mapping

Layer structure
- Strained Si (top layer, very thin)
- Relaxed Si$_{1-x}$Ge$_x$ (thick, many microns)
- Si(001) substrate

Lattice structure
- Strained Si
- Relaxed Si$_{1-x}$Ge$_x$ (virtual substrate)
- Si(001) substrate

Strain?
Lattice distortion?
% of relaxation?
at % of Ge?
Defects?

Example: strained Si layer on Si$_{1-x}$Ge$_x$ / Si substrate

Reciprocal lattice
- (004)
- (224)

Δq┴
Δq∥

Si(001) substrate
Relaxed Si$_{1-x}$Ge$_x$
Strained Si

High resolution 2θ/θ scan near Si(004)

High resolution reciprocal lattice map

Strained Si (top layer)
Relaxed Si$_{1-x}$Ge$_x$
Si(001) substrate

Map near Si(224)

Data: Sardela
Sample: Zuo, UIUC
High resolution reciprocal lattice map

Strained Si
$\varepsilon_{\perp} = -0.77\%$
$\varepsilon_{//} = 0.64\%$

4.60 at% Ge
7.52 at% Ge
11.45 at% Ge

Relaxed Si$_{1-x}$Ge$_x$
18.70 at% Ge
100% strain relaxation

Si substrate

Map near Si(224)

Data: Sardela
Sample: Zuo, UIUC

Finite size
Composition and strain gradients
Mosaicity and dislocations
Analyzer streaks
Coherent length in any direction:
$2\pi / \Delta q_i$
$i = x, y, z, //, \perp$

High resolution reciprocal lattice map

Strained Si (top layer)
Relaxed Si$_{1-x}$Ge$_x$
Si(001) substrate

Map near Si(224)

Data: Sardela
Sample: Zuo, UIUC
High resolution reciprocal lattice map

- Finite size
- Vertical coherent length: 14 nm
- Composition and strain gradients
- Mosaicity and dislocations
- Misfit dislocations: average separation: 21 nm density: $5 \times 10^5 \text{ cm}^{-1}$

Strained Si (top layer)
Relaxed Si$_x$Ge$_{1-x}$
Si(001) substrate

Map near Si(224)
Analyzer streaks

Coherent length in any direction:
$$\frac{2\pi}{\Delta q_i}$$

Misfit dislocations
- average separation: 21 nm
- density: $5 \times 10^5 \text{ cm}^{-1}$

Vertical coherent length: 14 nm

Comparison with TEM

EDS line scan

Data: Zuo's group, UIUC
The “shape” of the reciprocal lattice point

- Changes in lattice parameter (radial direction)
- Lateral sub-grain boundaries (along $q_\parallel$)
- Mosaicity, curvature, orientation (circumferential direction)
- CTR, finite layer thickness, superlattice (along $q_\perp$)

X-ray reflectivity methods

**Bulk materials:**
- Near surface region

**Liquids:**
- Near surface and interface information on:
  - Density
  - Porosity
  - Roughness
  - Thickness in films (ultra thin to thick)
  - Amorphous or crystalline materials

**Multilayered systems:**
- Near surface and interface information on:
  - Density
  - Porosity
  - Roughness
  - Thickness in films (ultra thin to thick)
  - Amorphous or crystalline materials
X-ray reflectivity

- Film thickness measurements: 2 – 300 nm.
- Applicable to ultra-thin films, amorphous or crystalline materials, multilayers and liquids.
- Simulation and fitting: determination of interface roughness (rms) at each interface, roughness correlation and film porosity.
- Very sensitive to density variations.
- Determination of critical angle, refractive index and density.

X-ray reflectivity analysis of thin films

- Ultra-thin
  - 2.3 nm thick polymer on Si
  - Data: Heitzman et al, UIUC
  - Sample: Aasadi et al, SIU

- Complex multilayers
  - Data: Sardela, UIUC

- Metallic multilayer
  - Data: Mikalsen et al, UIUC

- Non crystalline
  - Amorphous PZT film
  - Data: Mikalsen et al, UIUC

\[
\sin \theta = \left( \frac{\lambda}{2d} \right)^2 \eta^2 - (\eta^2 - 1)
\]
(modified Bragg's law to include refractive index)
X-ray reflectivity information content

- Critical angle: density, porosity
- Thickness (Kiessig) fringes: angular separation between oscillations gives thickness
- Two sets of oscillations: gives thickness of two layer structure
- Amplitude of fringes: interface roughness, density variations
- Slope / shape of the decay of the curve: used to quantify interface roughness in all interfaces

\[ \theta_{\text{critical}} = \frac{(2\delta)}{1} \]

Refractive index = 1 - \( \delta + i\beta \)

\[ t = \frac{(\lambda/2)}{(\sin\theta_2 - \sin\theta_1)} = \frac{(\lambda/2)}{\Delta\theta} \]

X-ray reflectivity from superlattices

- Critical angle: gives density of each layer in the superlattice (model dependent)
- Thickness fringes: angular separation gives total superlattice thickness
- Number of fringes gives the total number of layers in the superlattice
- Superlattice main peaks: angular separation gives period thickness
- Slope: gives interface roughness for each layer in the superlattice (model dependent)

Example: 20 periods [1 nm SiO\(_2\) / 1 nm Si] superlattice on Si wafer
X-ray reflectivity data fitting in ultra-thin films

Polymer (few nm)

SiO\(_2\) (~100 nm)

Si substrate

Data: Sardela
Sample: Zhang, Rogers et al, UIUC

Best fit

Polymer thickness

SiO\(_2\) thickness

1,3,5-tribromo-2-nanyloxybenze: C\(_{15}\)H\(_{21}\)Br\(_3\)SiO\(_3\)

Polymer thickness

Polymer thickness

rms: 0.26 nm

rms: 0.45 nm

rms: 0.24 nm

Polymer

2.0 nm, 1.30 g/cm\(^3\)

SiO\(_2\)

98.9 nm, 2.19 g/cm\(^3\)

Si substrate
Fitting sensitivity to the polymer thickness (best fit: 2.00 nm)

- 1.8 nm (- 10 %) simulation
- 2.2 nm (+ 10 %) simulation

Fitting sensitivity to the polymer top roughness (best fit: 0.26 nm)

- 0.2 nm (- 0.06 nm) simulation
- 0.4 nm (+ 0.14 nm) simulation
**X-ray reflectivity data fitting**

Fitting sensitivity to the polymer density (best fit: 1.3 g/cm³)

**X-ray reflectivity: summary**

* Non destructive method
* Applicable to whole wafers (wafer mapping option)
* Fast method (in most cases)
* Do not depend on crystalline quality of the films (can also be used in amorphous layers).

Quantification of:

* Layer thickness in thin films and superlattices: 1 nm ~ 1 μm (± 0.5-1%).
* Layer density and porosity (± 1-2%).
* Interface roughness: 0.1 – 10 nm (model dependent; reproducibility ~ 3%).
* Layer density gradients (variations > 2%).
* Interface roughness correlation in superlattices and multilayers.

Alternative techniques:

* Thickness: optical methods (TEM, SEM) poor contrast issues.
* Density: RBS (issues for ultra thin layers).
* Interface roughness: AFM (surface only – not buried interfaces).
X-ray fluorescence methods

**Bulk materials:**

What elements are present down to ppm levels?

What is the elemental composition (%)?

Fast, accurate.

Liquids, solids, amorphous, crystalline materials.

**Liquids:**

**Multilayered systems:**

Fast, accurate.

Liquids, solids, amorphous, crystalline materials.

X-ray fluorescence (XRF)

Data: Panalytical (www.panalytical.com)

Transition notation:

IUPAC: <element><"hole" shell><"originating" shell>
Ex.: Cr-KLii

Siegbehn: <element><"hole" shell><α,β,γ, etc.>
Ex.: Cr-Kα1

(for Lα to K transition in Cr)

- Typical plot: Intensity vs. Energy.
- Line position associated to element and specific transition
- Line positions: fingerprint of the element.
- Element identification Na-U or Be-U.
- Peak assignment uses database and search/match.
- Peak area/height: composition (sub ppm to 100%).
- Peak overlap requires deconvolution.
- Composition determination requires standards, calibration.
- Fast data acquisition (seconds to ½ hr).
- Solids, liquids, powders, thin films, etc.
- Minimum or no sample preparation required.
### EDXRF and WDXRF

#### Energy Dispersive XRF (EDXRF)

- **X-ray tube**
- **Sample**
- **Secondary target**
- **Detector**
- **Analyzer crystal**

#### Wavelength Dispersive XRF (WDXRF)

- **X-ray tube**
- **Sample**
- **Collimator**
- **Detector**
- **Analyzer crystal**

### Peak assignment:

- Search/match software

### Concentration:

- Down to sub-ppm

### Energy resolution:

- **EDXRF**: 150-300 eV
- **WDXRF**: 5-20 eV

### Data:


---

### Comparison Table

<table>
<thead>
<tr>
<th></th>
<th>EDXRF</th>
<th>WDXRF</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Dispersive system</strong></td>
<td>Energy</td>
<td>Wavelength</td>
</tr>
<tr>
<td><strong>Raw data</strong></td>
<td>Intensity vs. Energy (keV)</td>
<td>Intensity vs. Detector angle (2θ)</td>
</tr>
</tbody>
</table>
| **Basic set up** | • X-ray tube  
• Secondary Target  
• Sample  
• Detector | • X-ray tube  
• Sample  
• Collimator (for // beam)  
• Analyzer Crystal  
• Detector (+ goniometer) |
| **Elemental Range** | Na – U                       | Be – U                         |
| **Detection limit** | Good for heavier elements (less optimum for light elements) | Good for all range |
| **Sensitivity**   | Good for heavier elements (less optimum for light elements) | Moderate for light elements. Good for heavy elements. |
| **Resolution**    | Good for heavy elements (less optimum for light elements) | Good for light elements (less optimum for heavy elements) |
| **Cost**          | Moderate                      | Relatively expensive           |
| **Measurement**   | Simultaneous                  | • Sequential (moving detector on goniometer)  
• Simultaneous (fixed detector) |
| **Moving parts**  | No                             | Crystal, goniometer            |
| **Detector**      | Solid state detector          | • Gas-filled (for Be – Cu)  
• Scintillation (for Cu – U) |
| **Qualitative analysis** | Peak area                    | Peak height                   |

---

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Basic EDXRF system configuration

Two configurations:
--- direct collimated
--- secondary target

Automatic 16 samples changer

Si (Li) detector

Automatic filter changer

Automatic secondary target changer

CMM instrument: Kevex Analyst 770 XRF system

XRF application

The Virgin and the Child, Unknown author
Museum of the Serbian Orthodox Church, Belgrade

Surface sample:
• Original made of Au.
• Restoration with Au imitation Schlagmetal (Cu and Zn).
• Fe: natural Fe-Al-silicate red boile used for gilding the icon.

Frame sample: Cu and Zn (no Au) -> Schlagmetal

XRF and IR results show pigments and binders widely used in the XIX century confirming that the work is from late XIX to XX centuries

"Spectroscopy investigation of icons painted on canvas"
Lj. Damjanovic et al. Belgrade
http://www.srs.ac.uk/research/heritage/presentations/Olgica_Marjanovic.pdf
### X-ray analysis summary

| Information contents | • % of crystallinity and amorphous contents  
| | • Identification and quantification of phases and mixtures  
| | • Chemical information (if crystalline)  
| | • Texture (type and strength) and fraction of random grains  
| | • Grain / crystallite size (> 2 nm)  
| | • Strain (> 10⁻⁷)  
| | • Stress (type, direction and value)  
| | • Relative crystallographic orientation  
| | • Lattice constants and unit cell type  
| | • Structure determination  
| | • Thickness (1.5 nm – 3 nm)  
| | • Roughness (including buried interfaces)  
| | • Density and porosity  
| | • Relative fraction of domains  
| | • Defects and dislocation densities  
| | • Mosaic tilts and sizes  
| | • ...  

| Detection limits | > 0.1 – 0.5 w%  

| Depth information | Up to 20 – 50 mm (typical)  
| | > 10 nm and variable with glancing angle XRD  

| Lateral resolution | ~ a few cm (typical)  
| | 10 mm (microdiffraction) – 5 cm  

| Angular resolution | 0.1°²q (typical powder diffraction)  
| | 0.003²q (high resolution methods)  

| Sample requirement | Mostly non destructive  
| | Sample sizes from ~ 50 mm to many cm  
| | No vacuum compatibility required  
| | Solids, liquids, gels  

### Comparison with other techniques

| X-ray analysis methods | Other techniques  
| | Surface analysis and electron microscopy techniques will require vacuum compatibility and in many cases sample preparation.  
| | Optical techniques will do analysis on air.  

| Sample preparation and vacuum compatibility | ☐ No vacuum compatibility required (except XRF on vacuum).  
| | ☐ "Any" sample size (depends on the goniometer size/weight capability).  
| | ☐ Rough surfaces acceptable (parallel beam configuration).  
| | ☐ No sample preparation required (prep recommended for the detection of unknown phases or elements in XRD/XRF).  

| Composition and impurity determination and quantification | ☐ ~ 0.1 w % (XRF > ppm); may require standards.  
| | ☐ XRD: also phase information and % of crystallinity.  
| | ☐ Data averaged over large lateral area.  
| | ☐ XPS: > 0.01 – 0.1 at % (may require depth profiling).  
| | ☐ SIMS: > 1 ppm (requires sputtering depth profiling).  
| | ☐ EDS: > 0.1 – 1 w % over small volume 1µm².  
| | ☐ Little with phase information; averages over small lateral areas (< 100 µm).  

| Lattice constants | ☐ Better than within 10⁻⁵  

| Thickness in thin films | ☐ HR-XRD or XRR: direct measurement (no modeling for single or bi-layers).  
| | ☐ Requires flat interfaces.  
| | ☐ RBS: > 10 nm (requires modeling).  
| | ☐ Ellipsometry: requires modeling.  
| | ☐ TEM: requires visual contrast between layers.  

| Grain size | ☐ Measures Crystallite Size.  
| | ☐ Typically ~ 1-2 nm – microns, requires size/strain assumptions/ modeling.  
| | ☐ "Volume average" size.  
| | ☐ SEM: grain size distribution averaged over small area.  
| | ☐ TEM/SEM: "number average" size.  

* Semiconductors  
* Coatings  
* Pharmaceutical Cements  
* Metals  
* Ceramics  
* Geology  
* Archeology  
* Biosciences  
* Forensics  
* Medical applications  
* Nanotechnology  
* Polymers  
* Food science  
* Combustion  
* Energy
## Comparison with other techniques

<table>
<thead>
<tr>
<th></th>
<th>X-ray analysis methods</th>
<th>Other techniques</th>
</tr>
</thead>
<tbody>
<tr>
<td>Texture</td>
<td>Type and distribution averaged over large sample volume.</td>
<td>EBSD: within grain sizes dimensions, better sensitivity at the surface.</td>
</tr>
<tr>
<td>Residual Stress</td>
<td>10 MPa, averaged over large sample volume (large number of grains). Needs crystallinity. Measures strain and obtains stress from Hooke's law. Averages macro and micro stresses over large area of a layer.</td>
<td>Wafer curvature: No need for crystallinity. Direct measurement of stress, but only interlayer stress between film and substrate (macrostress).</td>
</tr>
<tr>
<td>Depth dependent information</td>
<td>Phase, grain sizes, texture and stress “depth profiling” – requires x-ray information depth modeling</td>
<td>Surface analysis depth profiling: compositional depth profiles.</td>
</tr>
<tr>
<td>Surface or interface roughness</td>
<td>XRR: interface roughness 0.01 ~ 5 nm, including buried interfaces</td>
<td>SPM: top surface only; rsm~ 0.01-100 nm.</td>
</tr>
<tr>
<td>Defects</td>
<td>Misfit dislocations (HR-XRD). Point defects (diffuse scattering with model). Extended defects (powder XRD with model). Average over larger sample area (&gt; mm).</td>
<td>TEM: accurate identification of defects and their densities; average over small sample area. Sample preparation may introduce artifacts.</td>
</tr>
<tr>
<td>Instrument cost</td>
<td>Portable instruments ~ $ 60 K. Average well-equipped: ~ $ 200 ~ 300 K. Top of the line ~ $ 500 K (including microdiffraction and 2D detectors).</td>
<td>Surface analysis instruments &gt; $ 500 K. Electron microscopes ~ $ 300 K ~ 1 M. RBS ~ $ 2 M. Raman, ellipsometry &gt; $ 100 K.</td>
</tr>
</tbody>
</table>

## Amazing x-ray samples

- Pieces from Egyptian mummy
- Powder from Inca ruins
- Corrosion in pipes from IL water supply
- Rocks from Mississippi river
- Mud from a cadaver’s shoes
- Dove chocolate
- Corn starch
- Train tracks
- Heavy machinery valves
- 100 µm micro chips
- 10 µm superconductor single crystal
- Next generation CPU processors
- Micro extraction from Dutch paintings
- Virus, bacteria, DNA, proteins
- Powder from Mars terrain
- Gasoline, electrical car battery
- Bone implants
- Pork tissue
### Amazing x-ray samples

**W. C. Roetgen wife’s hand (1895)**  
© Science and Society Picture Library | Science Museum

It “frightened Bertha terribly” as a “premonition of death”  
After Boston Globe 11/8/95

**W.C. Roetgen**  
(1845-1923)  
1st Physics Nobel

**A. Bertha Roetgen**  
(1833-1919)

---

### Quick guide to our x-ray analysis instruments (1)

**X-ray analysis instrumentation available as user facility in the FS-MRL**

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Set up</th>
<th>Applications</th>
</tr>
</thead>
</table>
| Panalytical X’pert (#1) | • Source: Cu Kα1, line or point focus.  
• High resolution configuration.  
• 4 or 2 reflection Ge monochromator (12 or 30 arc-sec parallel beam).  
• 3 reflection Ge analyzer crystal (12 arc-sec parallel beam).  
• Eulerian cradle.  
• Proportional detectors.  | • High resolution XRD capabilities.  
• Rocking curve.  
• Single crystals, epitaxial systems.  
• Reciprocal space mapping.  
• Reflectivity.  
• Curvature, wafer mapping, miscut, diffuse scattering.  
• Topography.  
• Glancing angle.  
• Parallel beam applications.  
• Max sample size: 10 cm diameter x 2 cm thick. |
| Panalytical X’pert (#2) | • Source: Cu Kα1+Kα2, line or point focus.  
• Crossed slit collimator (variable aperture).  
• X-ray lens.  
• Programmable divergence slit.  
• Eulerian Cradle.  
• Parallel plate collimator and flat graphite monochromator.  
• Programmable receive and scatter slits and graphite monochromator.  
• Proportional detectors.  | • Phase, size, strain, stress, texture, crystallinity  
• Parallel beam applications  
• Bragg-Brentano applications  
• General thin film analysis  
• Glancing/grazing angle  
• Max sample size: 10 cm diameter x 2 cm thick |
Quick guide to our x-ray analysis instruments (2)

X-ray analysis instrumentation available as user facility in the FS-MRL

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rigaku D/Max b</td>
<td>• Source: Cu Kα1+Kα2, line focus.</td>
</tr>
<tr>
<td></td>
<td>• Bragg-Brentano focusing configuration.</td>
</tr>
<tr>
<td></td>
<td>• Theta/2theta goniometer.</td>
</tr>
<tr>
<td></td>
<td>• Divergence,oller, scatter and receiving slits.</td>
</tr>
<tr>
<td></td>
<td>• Curved graphite monochromator.</td>
</tr>
<tr>
<td></td>
<td>• Scintillation detector.</td>
</tr>
<tr>
<td></td>
<td>• Phase, size, strain, crystallinity.</td>
</tr>
<tr>
<td></td>
<td>• Bragg-Brentano applications.</td>
</tr>
<tr>
<td></td>
<td>• Rietveld analysis.</td>
</tr>
<tr>
<td></td>
<td>• Mostly for powder, bulks and thin film with small preferred orientation.</td>
</tr>
<tr>
<td>Rigaku Laue</td>
<td>• Source: Mo point focus.</td>
</tr>
<tr>
<td></td>
<td>• Four circle sample stage (manual).</td>
</tr>
<tr>
<td></td>
<td>• Polaroid film camera detection system.</td>
</tr>
<tr>
<td></td>
<td>• Single crystal orientation.</td>
</tr>
<tr>
<td></td>
<td>• Miscut information.</td>
</tr>
<tr>
<td></td>
<td>• Crystallographic alignment prior to crystal cutting.</td>
</tr>
<tr>
<td>Bruker / Siemens D5000 (Fall 2008)</td>
<td>• Source: Cu Kα1+Kα2, line focus.</td>
</tr>
<tr>
<td></td>
<td>• Bragg-Brentano focusing configuration.</td>
</tr>
<tr>
<td></td>
<td>• Theta / theta goniometer.</td>
</tr>
<tr>
<td></td>
<td>• Horizontal sample load.</td>
</tr>
<tr>
<td></td>
<td>• No sample movement required during analysis.</td>
</tr>
<tr>
<td></td>
<td>• Divergence, scatter and receiving slits.</td>
</tr>
<tr>
<td></td>
<td>• Scintillation detector.</td>
</tr>
<tr>
<td></td>
<td>• Ideal for powder and soft samples (horizontal load).</td>
</tr>
<tr>
<td></td>
<td>• Phase, size, strain, crystallinity.</td>
</tr>
<tr>
<td></td>
<td>• Bragg-Brentano applications.</td>
</tr>
<tr>
<td></td>
<td>• Rietveld analysis.</td>
</tr>
<tr>
<td></td>
<td>• Mostly for powder, bulks and thin film with small preferred orientation.</td>
</tr>
<tr>
<td>Kevex Analyst 700 XRF</td>
<td>• Source: Rh (side window) 3.3mA, 80kV</td>
</tr>
<tr>
<td></td>
<td>• 6 secondary targets, 2 detector collimators, 3 filters.</td>
</tr>
<tr>
<td></td>
<td>• Si (Li) solid state detector.</td>
</tr>
<tr>
<td></td>
<td>• Elemental identification: Na – U.</td>
</tr>
<tr>
<td></td>
<td>• Liquids, solids, powder samples.</td>
</tr>
<tr>
<td></td>
<td>• Composition: &gt; ppm (some standards available).</td>
</tr>
</tbody>
</table>

Recommended literature

Basic applications of x-ray diffraction:
• Sample Preparation Methods:
• Rietveld Analysis:
• Thin Analysis by X-ray:
  "Thin Film Analysis by X-ray Scattering", M.Bieldsole, Wiley-VCH, 2006
• High-resolution X-ray analysis:
  "X-ray scattering from semiconductors", P. Forster, Imperial College, 2001.
• Industrial applications of x-ray analysis:
  "Industrial Applications of X-Ray Diffraction" by F. Smith (Editor), CRC, 1999.
• Glancing/grazing incidence methods and reflectometry:
Acknowledgements

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