Goals and results of the SNL crystal calibration work

Goals
1) Measure accurate rocking curve of bent crystals to help the understanding of measurements taken on Z (opacity, photoionized plasmas, non-thermal emission, liner spectra…) e.g. The iron opacity measurements on Z require integrated reflectivity, crystal resolution, and 2\textsuperscript{nd} to 1\textsuperscript{st} order reflectivity ratio (2\textsuperscript{nd} order correction below 950eV)

2) Evaluating absolute source intensities, instrumental broadening, plasma line-widths.

3) Develop intuition and a path to model crystal performance that could be used in a larger parameter space (various crystal curvatures, geometries, orders, photon energy…)

Results
1) We accurately measured integrated reflectivities for a set of KAP crystal curvatures (flat, 2, 4, 6 and 9 in) and diffraction orders (1\textsuperscript{st}, 2\textsuperscript{nd} and 3\textsuperscript{rd})

2) Integrated reflectivities show good agreement with XOP multilamellar model with a set of Debye-Waller factors (temperature factors) depending on crystal curvature and order of diffraction. It is possible that they could also depend on photon energy but the present data is too limited to be conclusive.

4) Width for 2\textsuperscript{nd} and 3\textsuperscript{rd} order agree relatively well with XOP multilamellar model

5) Widths in 1\textsuperscript{st} order are systematically measured higher than any calculation due to extra instrumental broadenings, this might be solved through deconvolution.

6) Width and spectral shape in 1\textsuperscript{st} order are measured with a conventional x-ray source

7) Crystal efficiencies were measured to high accuracy using NIST calibrated KERMA source
Crystal spectrometric properties are required to unfold instrumental effects in HEDP experiments

Crystal spectrometric properties are:

1) Crystal integrated reflectivity ($R_{\text{int}}$)
2) Crystal resolution and spectral shape

These are readily obtained through the measurement of crystal *rocking curve*, nevertheless either one quantity is more readily measurable accurately.

Outline of this presentation

1) Synchrotron beamline measurements $\rightarrow R_{\text{int}}$ and some resolution
2) Uncalibrated conventional x-ray source $\rightarrow$ resolution and spectral shape
3) Calibrated conventional x-ray source $\rightarrow R_{\text{int}}$
1) Using the ALS LBNL beamline to measure crystal rocking curve and probe the effect of bending in KAP
Rocking curve calibrations principle and requirements

Principle of measurement

1) Select photon energy (double-monochromator)
2) measure $I_0$ with the crystal out of the way
3) rock the crystal ($\theta$) and detector ($2\theta$) and measure reflected counts

Requirements

*Integrated reflectivity*
1) No significant higher order contamination in the counts
2) Rock the crystal about a fixed point (alignments crystal, beam, stages)
3) High x-ray flux for reasonable S/N and fast data collection

*Shape and width* \(\rightarrow\) limit systematic broadenings introduced by other effects
1) beam intrinsic spectral width small (high spectral purity)
2) beam size small
3) bending effect small or understood
4) keep misalignments small
Rocking curve calibrations principle and requirements

Principle of measurement

1) Select photon energy (double-monochromator)
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3) rock the crystal (\( \theta \)) and detector (\( 2\theta \)) and measure reflected counts

Requirements

Integrated reflectivity
1) No significant higher order contamination in the counts ✔✔ (ALS+ detector)
2) Rock the crystal about a fixed point (alignments crystal, beam, stages) ✔ ✔ (precision stage)
3) High x-ray flux for reasonable S/N and fast data collection ✔✔✔ (ALS)

Shape and width \( \rightarrow \) limit systematic broadenings introduced by other effects
1) beam intrinsic spectral width small (high spectral purity) ✔✔✔ (ALS beamline)
2) beam size small ✔ (pinhole and collimated beamline)
3) bending effect small or understood
4) keep misalignments small

“rocking” angle

crystal surface

\( 2\theta \)
\( \theta \)
Predicted effect of bending on integrated reflectivities

KAP (001)

Predicted effect of bending on FWHM

KAP (001)

Photon energy (eV)

FWHM (mrad)

Potassium K-edge

Henke tabulated

XOP* 2in
4in
6in
9in
flat

The ALS beamline 9.3.1 has high photon flux in 2.3-5.2 keV

Source characteristics
Bend magnet
Energy range 2.3-5.2 keV
Monochromator Double Si(111) crystal
Measured flux (1.9 GeV, 300 mA) $10^{11}$ photons/s in 30 bunches mode.
Resolving power (E/ΔE) 3000-7200
Beam size Adjustable with 2nd mirror
Focused: 1.0 mm x 0.7 mm (~0.5 mm square at 2800 eV)
Unfocused: 10 mm x 10 mm or larger
ALS beamline 9.3.1 experimental setup

Ring electron current 35.2mA ± 0.3mA, high stability, ~24/7

**Monochromator**
- 2x Si(111) crystals, energy range 2300eV-5200eV, resolving power 3500-7000

**Imager**
- x-ray beam imaged (4mrad width, ½ mrad height) at the crystal location
- imager = 2 Ni toroidal mirrors (1m long each), Mag=1
- grazing 11mrad incidence (rejection at > ~5.4keV)
Calibration end-station

Crystal stages: $\vartheta$ rotation (Bragg angle), resolution=0.001°, z translation
Detector stage: $2\vartheta$ rotation, res=0.005°
Pinhole mount: vertical translation
Pinhole sizes: 5.0, 3.0 or 1.2µm

Crystals: Saint Gobain crystal flat, 2, 4, 6 and 9 in radius of curvature, all new
Detection and statistics

The Amptek SDD (Silicon Drifted Detector) is an energy **dispersive** detector. Can count up to 1e6 cts/s, linearly in 1-50keV.

*Simultaneous* events appear as a ‘second order’ signal, such that 2 counts add up together and appear as a 2x energy single count.

Rocking curves RC are obtained by dividing the reflected counts to the direct beam counts number at a given Bragg angle.

Rocking curve 1-σ uncertainty is inferred through Poisson statistics of counts $C$:

$$ RC = \frac{C}{C_0} \quad \sigma = \frac{C}{C_0} \sqrt{\frac{1}{C} + \frac{1}{C_0}} $$

with $C_0$ the non-reflected (direct beam) counts, assuming detector dominated statistics.

Example of detected signal at a given Bragg angle

- **counts**
- **energy**
- **region of summation**
- **<1%**
- **integration time 3-30s / angle**

Channel (photon energy)
Data can be collected 24/7
We collected ~200 rocking curves, each energy/crystal set measurement is doubled - 184 processed here

<table>
<thead>
<tr>
<th>Date</th>
<th>Crystal set</th>
<th>Phenan crystal (°)</th>
<th>Rocking curve set</th>
<th>Order of diffraction</th>
<th>time stamps1</th>
<th>time stamps2</th>
<th>Ren2 (μrad)</th>
<th>Ren3 (μrad)</th>
<th>Ren4 (μrad)</th>
<th>FWHM2 (μrad)</th>
<th>FWHM3 (μrad)</th>
<th>FWHM4 (μrad)</th>
<th>X (μrad)</th>
<th>Y (μrad)</th>
<th>Z (μrad)</th>
</tr>
</thead>
</table>
| Sunday April 12th
| 9          | 7854-2      | 1.71               | -                  | -                    | -            | -            | -           | -           | -           | -            | -            | -         | -        | -        |
|            | 1184-1      | 1.71               | -                  | -                    | -            | -            | -           | -           | -           | -            | -            | -         | -        | -        |
|            | 7854-2      | 1.71               | -                  | -                    | -            | -            | -           | -           | -           | -            | -            | -         | -        | -        |
|            | 1184-1      | 1.71               | -                  | -                    | -            | -            | -           | -           | -           | -            | -            | -         | -        | -        |

*Table data continued...*
Example of rocking curve (4)

KAP Flat
2400 eV
1st order

Voigt peak = 0.45642801
Voigt integral = 0.079761257
Voigt FWHM = 0.13351575
Example of rocking curve (2)

KAP 2in
2293.16eV
1st order

Reflectivity

Bragg angle (mrad)

Voigt peak = 0.087180386
Voigt integral = 0.11721647
Voigt FWHM = 1.038857

Exp. data

Voigt, $\chi^2 = 47.5$, bkg = 0.00065 + 0.0x + 0.0x^2

Lorentz, $\chi^2 = 113$, bkg = -0.0020

Gauss, $\chi^2 = 112$, bkg = 0.0037
Example of rocking curve (2)

KAP 2in
2293.16eV
2nd order

<table>
<thead>
<tr>
<th>Function</th>
<th>Chi-squared</th>
<th>Background</th>
</tr>
</thead>
<tbody>
<tr>
<td>Voigt</td>
<td>3.06</td>
<td>2.5e-05 + 0.0x + 0.0x^2</td>
</tr>
<tr>
<td>Lorentz</td>
<td>10.1</td>
<td>-0.00021</td>
</tr>
<tr>
<td>Gauss</td>
<td>8.89</td>
<td>0.00028</td>
</tr>
</tbody>
</table>

Voigt peak = 0.009484988
Voigt integral = 0.0080358051
Voigt FWHM = 0.66323464
Example of rocking curve (3)

KAP 2in
2293.16eV
3rd order

Reflectivity

Bragg angle (mrad)

Voigt peak = 0.00021695432
Voigt integral = 9.996444E-05
Voigt FWHM = 0.34115678
The Voigt fit parameters are compared with XOP model for $R_{\text{int}}$ and FWHM

XOP 2.4 Multi-Lamellar model, dynamical theory of diffraction, Zachariasen equations, imperfect mosaic crystal.

- 5 curvatures: flat, 9in, 6in, 4in, 2in crystals
- orders: 1, 2, 3
- polarization: $\sigma$, $\pi$ & none
- temperature factors: 0.8, 0.9, 1.0
- energy grid 500pts $[2270 – 4533]$ eV

The KAP $c$ lattice parameter was updated in XOP to correct lattice spacing and 013 plane tilt.

Integrated Reflectivity

1st order
FLAT – 1\textsuperscript{st} order

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{FLAT_1st_order.png}
\caption{Graph showing the Rint (mrad) vs. Photon energy (eV) for different DW values.}
\end{figure}

- DW = 1.0
- DW = 0.9
- DW = 0.8
9in – 1\textsuperscript{st} order

\begin{figure}
\centering
\includegraphics[width=\textwidth]{figure.png}
\caption{Graph showing \textit{Rint} (mrad) vs. Photon energy (eV) for different DW values.}
\end{figure}

- DW = 1.0
- DW = 0.9
- DW = 0.8
6in – 1\textsuperscript{st} order

![Graph showing Rint (mrad) vs. Photon energy (eV) for DW=1.0, DW=0.9, and DW=0.8.](image)
4in – 1\textsuperscript{st} order
2in – 1\textsuperscript{st} order

\begin{align*}
\text{DW} &= 1.0 \\
\text{DW} &= 0.9 \\
\text{DW} &= 0.8
\end{align*}
Integrated Reflectivity

2nd order
FLAT – 2nd order

![Graph showing Rint (mrad) vs. Photon energy (eV) for DW=1.0, DW=0.9, and DW=0.8]
9in – 2\textsuperscript{nd} order

\begin{figure}
\centering
\includegraphics[width=\textwidth]{9in_2nd_order.png}
\caption{Graph showing Rint (mrad) vs. Photon energy (eV) for different DW values.}
\end{figure}

- DW = 1.0
- DW = 0.9
- DW = 0.8
6in – 2\textsuperscript{nd} order

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{graph.png}
\caption{Graph showing the relationship between photon energy (eV) and Rint (mrad) for different DW values.}
\end{figure}

\begin{itemize}
\item DW=1.0
\item DW=0.9
\item DW=0.8
\end{itemize}
4in – 2\textsuperscript{nd} order

\begin{itemize}
\item DW=1.0
\item DW=0.9
\item DW=0.8
\end{itemize}
2in – 2\textsuperscript{nd} order

![Graph showing photon energy versus Rint (mrad) for different DW values.]
Rocking curve FWHM
1\textsuperscript{st} order
FLAT – 1\textsuperscript{st} order

\begin{align*}
\text{FWHM}_v (\text{mrad}) & \quad \text{Photon energy (eV)} \\
\text{DW}=1.0 & \quad \text{DW}=0.9 \\
\text{DW}=0.8 & \quad \sim 1.6 \times \text{larger}
\end{align*}
9in – 1\textsuperscript{st} order

![Graph showing Fwhmv vs. Photon energy for different DW values (1.0, 0.9, 0.8)]
6in – 1\textsuperscript{st} order

\begin{figure}
\centering
\includegraphics[width=\textwidth]{figure}
\caption{Graph showing the relationship between Fwhmv (mrad) and Photon energy (eV) for different DW values (1.0, 0.9, 0.8).}
\end{figure}
4in – 1st order

![Graph showing a relationship between Fwhmv (mrad) and Photon energy (eV) for different DW values: DW=1.0, DW=0.9, DW=0.8.](image-url)
2in – 1\textsuperscript{st} order
Rocking curve FWHM

2nd order
FLAT – 2\textsuperscript{nd} order

![Graph showing energy resolution for different DW values]
9in – 2\textsuperscript{nd} order

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure.png}
\end{figure}

- DW = 1.0
- DW = 0.9
- DW = 0.8
6in – 2\textsuperscript{nd} order

\[ \text{FwhmV (mrad)} \]

\[ \text{Photon energy (eV)} \]

\begin{align*}
\text{DW}=1.0 \\
\text{DW}=0.9 \\
\text{DW}=0.8
\end{align*}
4in – 2\textsuperscript{nd} order
2in – 2\textsuperscript{nd} order

![Graph showing photon energy vs. FWHM (mrad) with different DW values: DW=1.0, DW=0.9, DW=0.8.](image-url)
Contributions to rocking curve width

1) crystal rocking curve
2) beam size at crystal (depends on angle of incidence)
3) crystal bending
4) intrinsic energy width
5) misalignments

**Width Voigt components:**

\[
\begin{align*}
Lorentzian & : & w_L &= w_{L,xtl} + w_{L,intrinsic} \\
Gaussian & : & w_g^2 &= w_{g,xtl}^2 + \left[ \left( \alpha d_p + 1.22 \frac{r \lambda}{d_p} \right) \frac{1}{R_c \sin \theta} \right]^2 + w_{g,intrinsic}^2
\end{align*}
\]
Contributions to rocking curve width

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The unknowns can be searched using under this large dataset to perform a forward deconvolution.

43
2) Using the un-calibrated Manson source to measure crystal spectral resolution and shape in KAP
The rocking curve shape and width in 1\textsuperscript{st} order can be measured with a conventional x-ray source* 

Methodology

- The detected spectral line (characteristic line) is made of the intrinsic line (at the source) convolved with crystal rocking curve.
- The intrinsic line shape and width is measured with a high resolution setup (here 2\textsuperscript{nd} order measurement)
- The 1\textsuperscript{st} order shape and width are obtained by deconvolution of the 2\textsuperscript{nd} order measurement

*G. Loisel et al, RSI, 2012
KAP 6in - Al Kα in 2\textsuperscript{nd} order

- Exp. data 2nd order
- Gaussian fit $Q^2=0.128$
- Lorentzian fit $Q^2=0.099$
- Voigt fit $Q^2=0.054$

FWHM $\approx 5.98$ mÅ
KAP 6in - Al Kα in 1\textsuperscript{st} order

Exp. data 1st order
- Gaussian fit $Q^2=8.14$
- Lorentzian fit $Q^2=2.15$
- Voigt fit $Q^2=0.25$

FWHM\nobreak\textsuperscript{a} $\approx 10.38$ mÅ
## KAP 6in results for Al, Mg and Si Kα lines

<table>
<thead>
<tr>
<th>Element / Kα wavelength</th>
<th>2nd order width (mÅ)</th>
<th>1st order width (mÅ)</th>
<th>Crystal broadening (mÅ)</th>
<th>Crystal 1st order resolving power</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si / 7.126 Å</td>
<td>5.87 ($\alpha=0.67$)</td>
<td>9.72 ($\alpha=0.83$)</td>
<td>6.24 ($\alpha=0.67$)</td>
<td>1142±73</td>
</tr>
<tr>
<td>Al / 8.34 Å</td>
<td>5.98 ($\alpha=0.88$)</td>
<td>10.38 ($\alpha=0.99$)</td>
<td>6.64 ($\alpha=0.77$)</td>
<td>1256±73</td>
</tr>
<tr>
<td>Mg / 9.89 Å</td>
<td>7.03 [13] ($\alpha=0.94$)</td>
<td>11.81 ($\alpha=1.04$)</td>
<td>7.47 ($\alpha=0.13$)</td>
<td>1415±348 [13]</td>
</tr>
</tbody>
</table>

The found crystal profiles are Voigt profiles with RP=1100

**NB:** the convolution of a Voigt (RP=1100) is similar to a Gaussian (RP=700)
3) Using the NIST absolutely calibrated KERMA facility to measure crystal efficiency in Quartz transmission
Absolute calibration setup @ NIST KERMA facility

100kV tube, W anode

Beam qualities (NS) used for the calibration are international standards with 4% relative uncertainty
End-to-end instrument calibration

101 data

The NS resolution should be used to convolve the crystal dispersed spectrum

XOP calc rescaled with a flat 4.7 ratio

2nd/1st ratio ~10 is in reasonable agreement
End-to-end instrument calibration

203 data
crystal 203-1-1

XOP calc rescaled with a flat 5.9 ratio

crystal mount clipping

1st order (203)

2nd order (406)

2nd/1st ratio ~20 is correct
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