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Measuring the X-ray Resolving Power of Bent Potassium Acid Phthalate Diffraction Crystals[a)]

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This report presents the results from measuring the X-ray resolving power of a curved potassium acid phthalate (KAP(001)) spectrometer crystal using two independent methods. It is part of a continuing effort to measure the fundamental diffraction properties of bent crystals that are used to study various characteristics of high temperature plasmas. Bent crystals like KAP(001) do not usually have the same diffraction properties as corresponding flat crystals. Models that do exist to calculate the effect of bending the crystal on the diffraction properties have simplifying assumptions and their accuracy limits have not been adequately determined. The type of crystals that we measured is being used in a spectrometer on the Z machine at Sandia National Laboratories (SNL) in Albuquerque, NM. The first technique for measuring the crystal resolving power measures the X-ray spectral line width of the characteristic lines from several metal anodes. The second method uses a diode X-ray source and a dual goniometer arrangement to measure the reflectivity curve of the KAP(001) crystal. The width of that curve is inversely proportional to the crystal resolving power. The measurement results are analyzed and discussed.

I. JUSTIFICATION AND GOALS

The throughput and the resolving power of a spectrometer are limited by the diffraction properties of the crystal that is used as the spectrometer monochromator. These properties and their energy dependence can be altered by bending the crystal. We have measured these properties for various crystals that were in spectrometers used on National Ignition Facility (NIF) at the Lawrence Livermore National Laboratory and the Z machine at Sandia National Laboratories (SNL). Measuring the reflectivity curve width is very challenging when the beam diverges. There are several corrections needed to correct for this divergence. An independent method was needed to corroborate the curve width measurement. The independent method chosen was to measure the X-ray spectral line width.

II. INTRODUCTION

A reflectivity curve for a flat crystal is given in FIG. 1 illustrating the diffraction properties being presented. This curve was measured using National Security Technologies, LLC (NSTec) dual goniometer and illustrates that when the system is used to measure a flat, perfect crystal, the measured full width at half maximum (FWHM) agrees with the dynamical diffraction theory for a double crystal as calculated using the X0h subset of the X-ray Server[1] from the Argonne National Laboratory. A spectrometer's throughput is proportional to area under the curve, which is referred to as the integrated reflectivity (R_I). The spectrometer's resolving power is inversely proportional to the reflectivity curve width taken as the full width at half maximum (FWHM). This term will be referred to as ΔΘ. The crystal resolving power (for Bragg angles far from 90º) is then given as

$$E/_{\Delta E} = \tan\theta_B/_{\Delta\theta} \qquad (1)$$

where E is X-ray spectral energy and Θ_B is the Bragg angle. Both of these diffraction properties are functions of spectral energy.

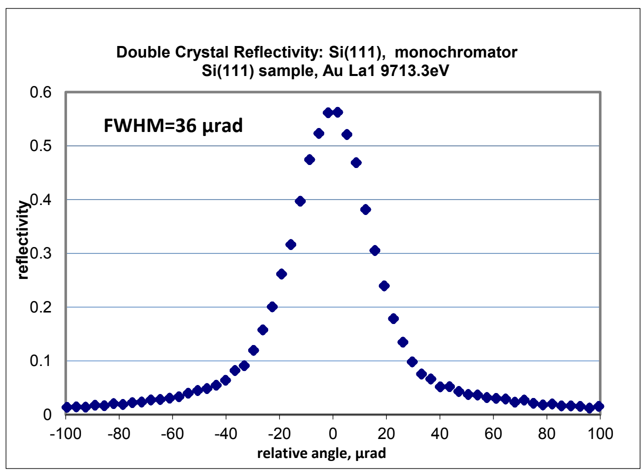

FIG. 1. Measured reflectivity curve for Si(111) sample crystal using Si(111) monochromator

Bending the crystal usually changes the reflectivity curve; R_I increases and ΔΘ increases, similar to the way the properties change for a mosaic crystal. We have published measurements of the increased integrated reflectivity for elliptically bent pentaerythritol (PET(001)) crystals and find the R_I increases significantly with decreasing radius of curvature.[2] There are several models for calculating the change but they all have tight

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constraints and limited applicability. None have been well tested. For cylindrical crystals in reflection, such as the KAP(001) of this study, there are the multi-lamellar model and the quasi-mosaic model. The multi-lamellar model decomposes the crystal in the beam direction into several layers of equal thickness. The output of a layer is the input to the next layer. The curvature alters the direction of each lamella. The term quasi-mosaic was first used by Russian scientists in the 1960's[3] and was selected because the crystal takes on mosaic like qualities when bent but will return to the perfect crystal qualities when unbent. According to this concept, the bent diffraction properties are determined by the crystal stress tensor. Recent publications show the importance of the quasi-mosaic effect for applications on beam lines and astrophysics.[4, 5]

III. MEASURING ΔΘ USING FILM

The sample crystal was a KAP(001) cylindrically bent with a 6 in radius of curvature. A sheet of X-ray film, curved into a cylinder with the same center as the KAP crystal, was mounted at an angle with respect to the beam axis so that it would receive the Bragg diffracted reflection. The crystal and film were mounted in a light tight enclosure that was attached to a vacuum arm of the X-ray source. The X-ray source was a multi-anode Manson type and it was 2 m from the crystal. A more complete description of this arrangement is found in Reference 6. We measured the X-ray source spot size as 0.7 mm using a pinhole camera.

Spectra were taken using five different anodes: Mg, Al, Si, Mo, and Ti. The first four spectra were taken on the same film. The KAP crystal and the film had to be reoriented at the smaller Bragg angle of Ti spectral energy, so the Ti spectrum was on a second film. The films were developed and converted to a digitized intensity spectrum. The spectrum wavelength was calibrated using known wavelengths of the spectral lines. The FWHM for the characteristic K_α and L_α and identified as Δλ. The crystal resolving power was calculated as λ/Δλ=E/ΔE. The results are shown in Table I.

TABLE I. KAP Resolving Power, 6 in. curved KAP crystal, measured resolving power, film spectrum

Anode	Spectral Energy (eV)	Resolution, Flat KAP Crystal E/ΔE	Resolution, 6 in curved KAP Crystal λ/Δλ	Ratio, Flat to Curved
Mg	1254	2000	821	2.4
Al	1487	2100	802	2.6
Si	1740	2150	728	3.0
Mo La	2293	2180	555	3.9
Mo Lb	2395	2180	518	4.2
Ti	4511	2250	380	5.9

The anodes used and the spectral energy of the characteristic X-ray lines for each anode are given in columns 1 and 2. The resolving power of a perfect flat KAP(001) crystal is given in column 3. The resolving power of the sample bent KAP(001) crystal having a 6 in radius of curvature is given in column 4. The resolving power of the bent KAP crystal is significantly lower than that of the flat KAP crystal. The ratio of the resolving power for the flat KAP to that of the curved KAP is given in column 5. Note that, whereas the resolving power for the flat KAP increases slowly with increasing spectral energy, that of the curved KAP decreases rapidly with increasing spectral energy.

IV. MEASURING ΔΘ USING A DUAL GONIOMETER SYSTEM

The device used for the second method to measure the bent KAP resolving power is the NSTec dual goniometer high resolution X-ray source as shown schematically in FIG. 2. A monochromator crystal located adjacent to the exit slit and on the rotation axis of the first goniometer is used to isolate a characteristic spectral line from the broad band X-ray source. The second rotation axis of the first goniometer is a large plate that has the sample stage attached to it, and the plate is rotated to the $2\Theta_B$ of the monochromator crystal. The sample stage comprises the second goniometer and the target crystal is mounted on its first rotation axis. An energy dispersive detector is mounted on the second rotation axis. The sample stage has three translation axes that are used to locate the target crystal so that the X-ray beam strikes the target at the desired point. The system has a laser that is aligned to the X-ray beam. The sample is aligned to the X-ray beam, and the measurement point on the sample is oriented over the rotation axis using the laser. The source and measurement chambers are then evacuated and the X-ray source is energized. Once the desired Bragg reflection from the sample is found, the sample is rotated around the measurement point to obtain a reflection curve.

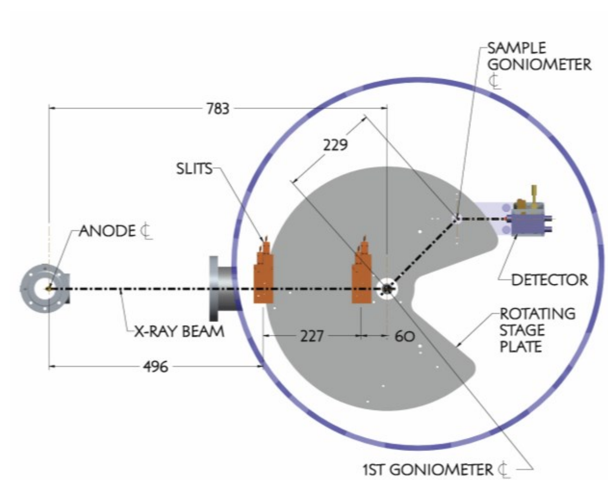

FIG. 2. Schematic image of the NSTec dual goniometer high resolution X-ray source showing the major elements of the measurement system. All measurements are in mm.

The reflectivity curve width is strongly affected by the X-ray beam divergence which is determined by the slit aperture and the separation between the slits. The reflectivity curve is measured at several slit apertures ranging from 100 µm to 10 µm. It has not been possible to get reasonable measurements below 10 µm. The graph in FIG. 3 shows the reflectivity curve width measurements for bent KAP crystal using the Al Ka spectral line. Similar results were obtained using the Mg Ka and the Mo La spectral lines. As shown by the trend line, this extrapolates to a ΔΘ=538 µrad with slits closed. The corresponding film measurement is ΔΘ=411 µrad when the resolving power from Table 1 is converted to ΔΘ using Equation (1). The reflectivity width using the dual goniometer system is 30% higher compared to the film, but further improvements in the measurement can be done as discussed in the following section.

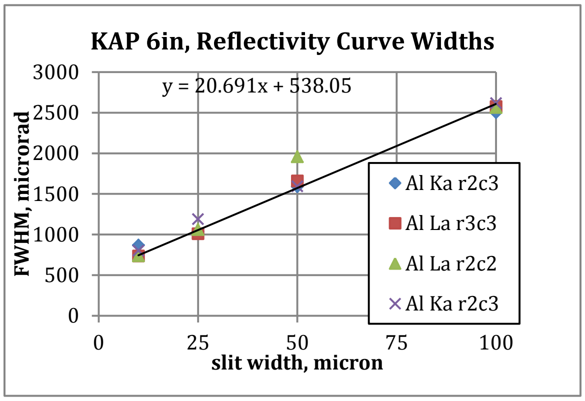

FIG. 3. Graph showing FWHM as function of slit width at three locations on crystal, with one location repeated at a later date.

V. ANALYSIS AND DISCUSSION

This section discusses factors affecting the reflectivity curve width measurement arising from the X-ray beam divergence. The width obtained from the film measurement will be considered as the "true width." There are three terms that arise from the beam divergence:

- $\Delta\Theta_D$: Beam divergence because the slits are not infinitely separated and the source has a finite size and does not produce a plane wave.
- $\Delta\Theta_E$: apparent angle arising from X-ray beam energy spread (spectral beam width).
- $\Delta\Theta_S$: range of angles at the curved sample surface due to the finite beam size hitting the sample surface.

Our estimates for these terms for the reflectivity curve measurement using the Al Ka spectral line are shown in Table II. The monochromator for these measurements and estimates was a flat KAP crystal. Our estimate of the beam divergence factors do correlate with the measured width but not sufficiently well (yet) to allow a useful estimate of the true reflectivity curve width. The ray trace done for estimating $\Delta\Theta_S$ was crude and an accurate ray trace is needed. This term is usually dominating for the KAP measurements because of the small Bragg angle; the projection of the slit width on the curved crystal is large. The next effort will be to learn how to improve the width measurement by correcting for these effects. We will measure several more KAP crystals having radius of curvatures from 2 in to 9 in. This and the accurate ray trace should give us a better understanding, especially for the impact of $\Delta\Theta_S$.

TABLE II. Reflectivity Curve Measurements on 6 inch KAP Crystals at 1487 eV

slit (μm)	Beam Divergence $\Delta\Theta_d$ (μrad)	Beam Spectral Energy Content ΔΘE (μrad)	Beam Width at Sample $\Delta\Theta_s$ (μrad)	Film Measured ΔΘ true (μrad)	Reflection Curve Measured ΔΘ meas (μrad)
100	930	944	2400	411 ±40	2617
50	465	465	1188	411 ±40	1593
25	233	282	580	411 ±40	1188
10	93	185	250	411 ±40	800
5	47	171	108	411 ±40	810

TABLE III. Reflectivity Curve Width Measured, Compared to Flat and Lamellar Model, 6 inch Curved Crystal

Anode	Spectral Energy (eV)	Film Measured, ΔΘ (μrad)	Film Measured, ΔΘ (μrad)	Film Measured, ΔΘ (μrad)
Mg	1254	487	232	232
Al	1487	411	177	177
Si	1740	381	147	148
Mo	2293	373	106	137
Ti	4511	273	51	228

Film measurements of the spectral resolving power for a cylindrically curved KAP(001) crystal having a 6 in radius of curvature is much lower the perfect flat KAP crystal. The NSTec dual goniometer X-ray was compared to the film measurements. System improvements have moved the reflectivity curve width measurements close to those of film (width measure approximately 30% high) so further work is needed. This will include an accurate beam ray trace and comparative measurements of several other cylindrically curved KAP crystals having different radii of curvature. Our long term goal is to achieve sufficient accuracy so that we can test the limits of various models on a variety of bent crystals.

VII. ACKNOWLEDGMENTS

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