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DEVELOPMENT OF THE ROCKING CURVE IMAGING SETUP AT BL17-2 AT SSRL

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Abstract

We report the implementation of a silicon (111) channel cut crystal as an x-ray beam expander for the rocking curve imaging (RCI) at Stanford Synchrotron Radiation Light Source (SSRL). Recently, we utilized an undulator source located at beamline 17-2 with the new beam expander to the perform rocking curve imaging of diamond (400) and (220) reflections. The beam expander is installed as part of an existing RCI optics setup, previously commissioned at beamline 10-2. We achieved horizontal beam magnifications of up to $M = 1.38$ at 6.951 keV and $M = 2.25$ at 9.831 keV. This work presents the updated RCI setup, and the experimental results validating its performance. Future improvements to the setup are also mentioned.

INTRODUCTION

Rocking curve imaging (RCI) is a common technique for characterizing diffraction Bragg crystals. The essence of the technique is to prepare a very well collimated beam and rock the crystal in the diffraction plane along the Darwin window. This measurement is typically done in the non-dispersive geometry. Synchrotron light sources, such as bend magnet or wigglers are perfect for RCI as they provide high average photon flux and a spatially large beam size. However, recently more and more synchrotrons employ undulator insertion devices, and with the 4-th generation upgrades, the beam sizes are now much smaller. This fact requires beam expansion, in addition to angular collimation, to perform RCI measurements. In this paper, we report on recent RCI experiments at the SSRL beamline 17-2, using an undulator insertion device.

We previously reported on the RCI program developed at the SSRL beamline 10-2. In brief, the RCI setup supported Bragg and Laue geometry measurements for 5 different photon energies. We refer the reader to the [1] for more information.

At beamline 17-2, asymmetric Bragg reflections are employed for double beam expansion. The experimental setup is schematically shown in Fig. 1(a). The horizontal expander consists of an symmetric-asymmetric channel-cut Si(111) crystal fabricated according to relation described in [2, 3], with dimensions designed for a theoretical magnification of $M = 2.5$ at 9.831 keV (Fig. 1(b)). This value represents a practical compromise between beam size expansion and combined reflectivity in a channel-cut. Figure 2 presents the calculated reflectivity curves (red) for different magnifications, where the observed angular shifts between reflections originate from x-ray refraction at the asymmetric surface (blue). As M increases, the angular overlap between the two reflections reduces, leading to a noticeable reduction in peak reflectivity and integrated diffracted intensity. These findings guide the practical design limits of Si(111) channel-cut beam expander.

The calculated overall diffraction efficiency is summarized in Fig. 3, showing that as M increases from 1 to 7, and a higher-resolution calculation for the practical range $M = 2$ to 4, where a nearly linear reduction in total output intensity and a gradual decrease in peak reflectivity is observed. We also note that for practical reasons, the Bragg angle in the channel-cut expander must be away from 45°, since the x-rays are $\sigma-$polarized. In our setup, the Bragg angle is 11.6° for 9.831 keV, and 16.5° for 6.951 keV.

Also note that, the Si(111) expander is mounted on a precision expander mount designed so that the center of the rotation axis coincides exactly with the center of the symmetric reflection surface. This geometry ensures that the Bragg angle can be adjusted easily when switching between different photon energies, without introducing additional alignment errors other than a horizontal shift. This horizontal shift of the expanded beam when switch between photon energies is less than the width of the analyzer crystal and well within the horizontal travel range of subsequent analyzer crystal and sample mount. The theoretical center-to-center beam offset between incoming beam and expanded beam is 1.7 cm and 2.14 cm for 6.951 keV and 9.831 keV, respectively. As a result, during experiments, the photon energy can be switched without moving either the analyzer or the sample stage.

After the beam has been horizontally expanded, it is propagated into the previously built and commissioned RCI setup with the asymmetric Si (531) crystal [4]. The vertical expansion ratio in the setup is about 30 [1]. The final rectangular, highly collimated beam then illuminates the sample, which is rocked around its Bragg reflection.

EXPERIMENT

Setup

The proof-of-principle experiment was carried out on beamline 17-2 at SSRL. The incoming 9.831 keV x-ray beam of 4 × 0.2 mm was expanded horizontally first to 9 × 0.2 mm with the channel cut expander (Fig. 1(a)), resulting in the magnification value $M = 2.25$. A final beam size of 9 × 8 mm was attained at the sample position (Fig. 1(b)). We also tested the magnification of the channel cut expander at 6.951 keV, with the measured magnification value is $M = 1.38$. We measured the total x-ray transmission (beamline output to the sample) of about 3% at 9.831 keV

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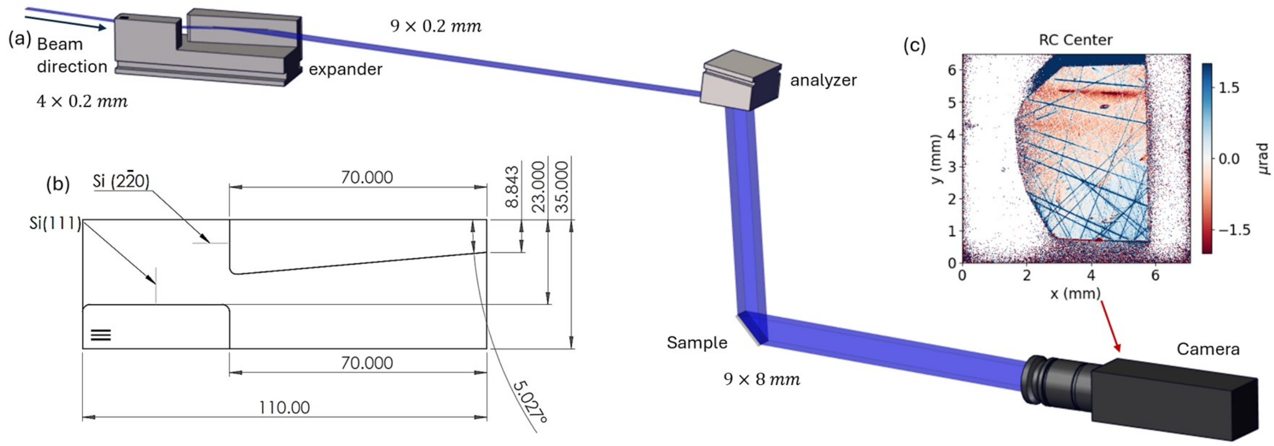

Figure 1: (a) Layout of the RCI setup (not to scale) for 9.831 keV. The incoming beam is first horizontally expanded in the symmetric-asymmetric Si(111) channel-cut, then vertically expanded by the Si(531) analyzer and illuminates the sample; (b) dimensions of the Si(111) beam expander; (c) an example rocking curve map of the Diamond (220) reflection.

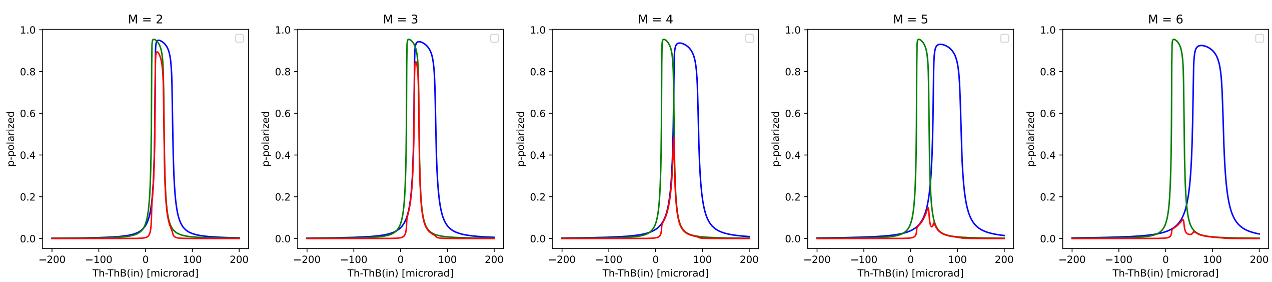

Figure 2: Overlap of the Darwin curves for Si(111) reflection at 9.831 keV in the channel-cut expander at different magnification M from 2 to 6 due to refraction in the Si.

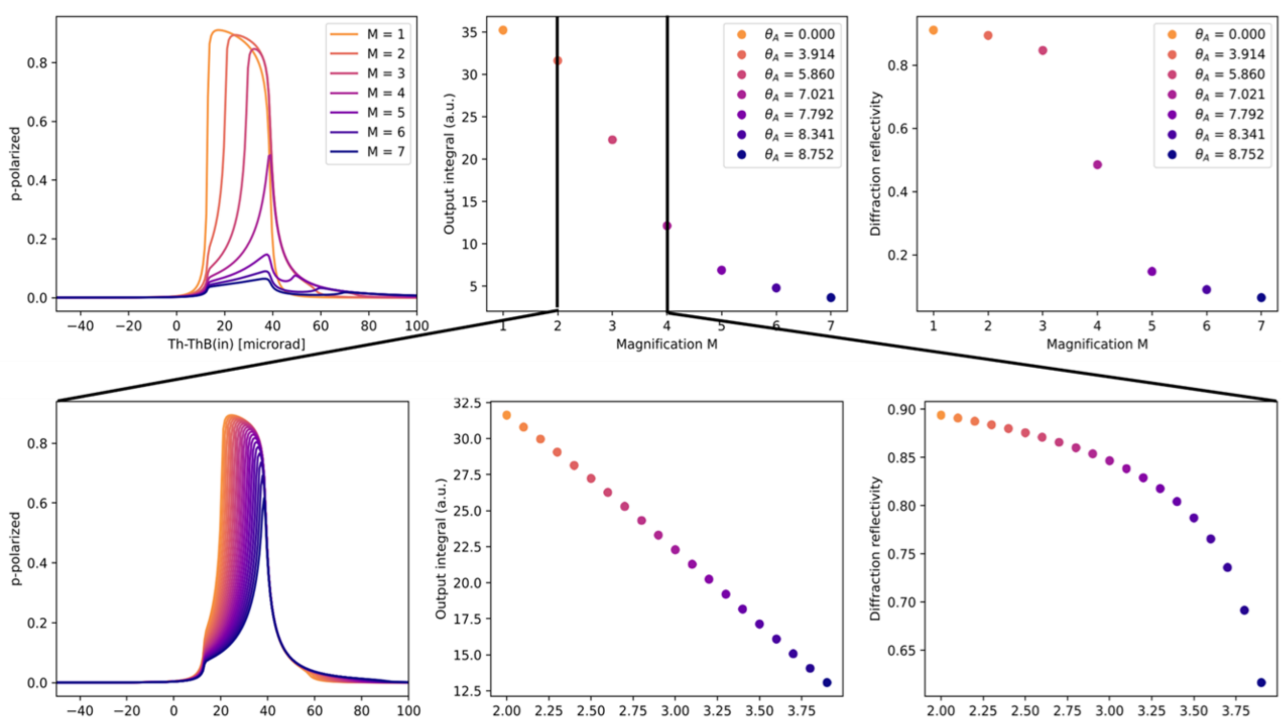

Figure 3: (left column) Calculated Darwin curves for different asymmetry angles for 9.831 keV; (middle column) calculated total reflected intensity (arb. units); (right column) calculated reflectivity peak amplitude, at M from 1 to 7 (top row) and from 2 to 4 (bottom row).

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photon energy. The samples are rocked around its Bragg reflection with a step of 0.00005°. The images recorded in the camera are combined and the rocking curve center is determined at each pixel for the scanned angle range.

Results

In the experiment, we measured the rocking curve maps of two diamond crystals with (400) and (220) reflections (Fig. 4). The (400) diamond has been previously characterized at beamline 10-2 and the RCI setup at the 1-BM beamline at APS. The results of our mapping corroborate previous measurements [1].

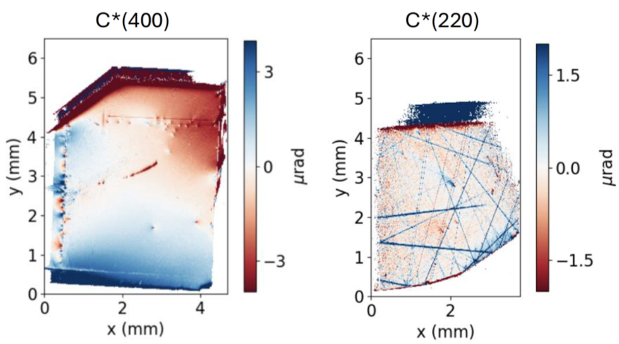

Figure 4: Recorded RCI maps of two diamond samples with (400) and (220) Bragg reflections.

DISCUSSION

The expanded x-ray beam from beamline 17-2 has been successfully used for crystal characterization. Further improvements of the setup can be made. First, the asymmetry ratio can be increased in the Si (111) expander, to further increase the horizontal beam size, at the expense of reducing the flux. We also note that the channel-cut geometry becomes more challenging with larger M due to larger geometrical offsets. Yet, the channel-cut geometry is much simpler for alignment. In a more permanent setup, one can consider separating reflecting surface of the expander and align them individually.

Second, one can also consider replacing the current Si(111) expander with Ge(111), which has a Darwin curve more than twice as wide as that of Si(111) at 9.831 keV. This broader angular acceptance of Ge(111) allows maintaining high diffraction efficiency at large angular offset, which improves the total photon throughput at same magnification compared to present Si(111) expande, or making larger magnification M such as $M = 4$ or $M = 5$ practical choices for beam expansion without the steep efficiency losses with Si(111).

The Ge(111) channel-cut expander uses the same symmetric-asymmetric Bragg reflection configuration as the current Si(111) expander. Importantly, at the photon energies of interest, (e.g., 6.951 keV, 9.831 keV, etc.), the Bragg angle for Ge(111) are very close to Si(111); hence the asymmetric angles for different magnifications are also similar. This ensures that the working geometry and the beam trajectory remain similar to those of the Si(111) expander. This close match means that the Ge(111) expander could be fabricated to fit the existing expander mount with minimum modification to the rest of mechanical components. The alignment procedure for Ge(111) would also remain essentially the same, avoiding the need for any major changes to the existing procedure.

CONCLUSION

We have successfully commissioned an RCI setup at the SSRL beamline 17-2, demonstrating the use of an undulator source with with a precision-mounted asymmetric–symmetric channel-cut Si(111) beam expander. This configuration allows rapid and reproducible Bragg angle adjustments when switching photon energies. The expander design achieves a horizontal magnification of $M = 2.5$ at 9.831 keV and $M = 1.38$ at 6.951 keV. Using this setup, we performed rocking-curve mapping of the Diamond (400) and (220) crystal reflections. These crystals will be employed in the future diffractive optics at LCLS. Several future upgrade plans are presented in the discussion section.

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