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Improvement of the Self-Referenced Lattice Comparator: from Using a Pencil Beam to a Brush Beam

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Abstract. We report improvement of the Self-referenced lattice comparator using a wide brush beam with size of $16 \text{ mm} \times 0.3 \text{ mm}$ and a pair of one-dimensional position sensitive X-ray counters instead of using a pencil beam with size of $1 \text{ mm} \times 0.3 \text{ mm}$ and a pair of PIN photodiode detector in the former system. The experimental time is accelerated by a factor of more than ten times. Since we use a brush beam for the measurement, the wavelength along the horizontal beam width positions are different, it is necessary to establish a data correction procedure for the lattice comparator. We reported our first data after the system improved, the spatial resolution of mapping measurement reached to $0.5 \text{ mm} \times 0.3 \text{ mm}$, that is higher than the previous system.

INTRODUCTION

In 2003, Zhang *et al.* proposed the self-referenced lattice comparator (SRLC) method¹ by using the wavelength selectivity of synchrotron radiation (SR). The use of high-intensity SR brings the gospel to overcome the problems relating the ultra-precision d -spacing mapping measurement of large-scale silicon crystals, that means this method is available for characterizing perfection of the ²⁸Si crystal used in the International Avogadro Coordination (IAC) project.

In 2017, Waseda *et al.* used a self-referenced lattice comparator to evaluate the uniformity of the lattice spacing of large silicon crystals samples with a resolution of 3×10^{-9} .² Although the high-intense synchrotron radiation makes to measure d -spacing of a large-scale sample possible, because of the photodiodes with photocurrent mode and a pencil beam were used in the experiment, the signal to noise ratio (SNR) was restricted, the pencil beam can only be narrowed to the size of $1 \times 0.3 \text{ mm}^2$, mapping a sample with size of $20 \times 40 \text{ mm}^2$ takes more than 20 hours.

Because the SR of bending magnet is a fan beam, if we use a pair of one-dimensional position sensitive detectors in our comparator system, the mapping measurement can be improved from a pencil beam scanning to a brush beam scanning, the mapping speed can be greatly increased. In this manuscript, we report preliminary results of using a brush beam and a pair of Mythen 1K detectors in the experiment of d -spacing measurement.

Method and Experimental Setup

The measurement method of SRLC is the same as previously reported, except for the detectors behind the monolithic double channel-cut monochromator (MDCM)³ (Fig. 1). The MDCM is composed of two channel-cut of high indices diffraction plane of the same crystal plane group. For example, the diffraction plane indices of two channel cut were (10 0 2) and (10 2 0) or (7 7 5) and (7 5 7), respectively. Different combinations of diffraction planes allow different X-ray energy to pass through. Theoretically, the MDCM with a certain diffraction plane

indices allows only a fixed energy beam to pass through. In this paper, the MDCM diffraction plane indices were (10 0 2) and (10 2 0), the allowable energy of the beam is 12.7536keV and the resolution ($\Delta E/E$) of 7×10^{-6} .

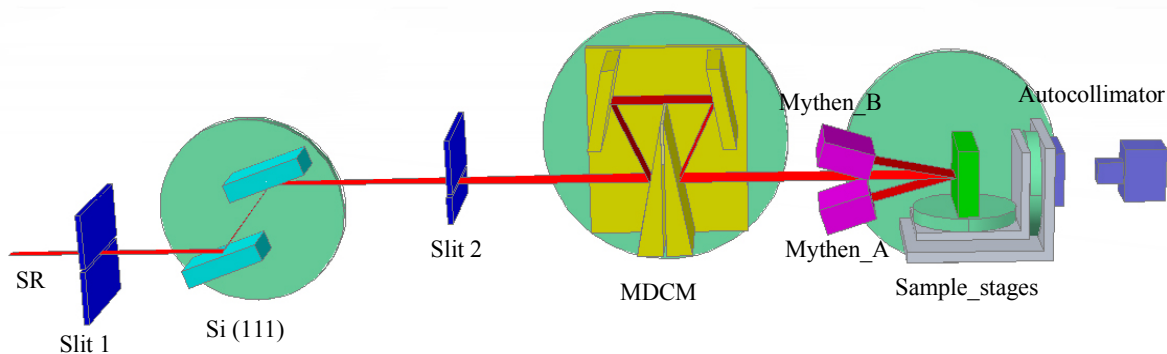


FIGURE 1. Sketch of the SRLC equipped with Mythen 1K detectors and using the brush X-ray beam for mapping measurement.

A wide beam of synchrotron radiation emitted from bending magnet is incident to MDCM after passing through the double crystal Monochromator Si (111). For pencil beam, the MDCM only allows a specific wavelength to go through. For brush beam, there is a horizontal divergence. The wavelength of monochromatic brush beam after MDCM changes slightly with the position, which is equivalent to turning the MDCM's tilt when using pencil beam. This tiny difference brings a significant effect on the ultra-high resolution of lattice. The wavelength distribution of brush beam satisfies the following equation:

$$\frac{\Delta\lambda}{\lambda} = \frac{1}{2} \cos(2\theta_B) \cdot \phi^2 \quad \text{a)}$$

where θ_B is the Bragg angle which can make MDCM diffracted, and ϕ is the angle between a certain direction and the central incident direction in brush beam. For the pencil beam, the horizontal energy resolution is 3×10^{-10} , and for the brush beam, the energy resolution is 8×10^{-8} . While in the experiment with a brush beam, the measurement results will be different at different sample positions even if the d -spacing is the same. Therefore, it is necessary to compensate the measurement results at different positions, when using brush beam for efficient and ultra- accurate lattice length measurements.

Figure 2 shows the experimental setup of the core part for system correction1. A pair of Mythen detectors replace the original photodiode detector. An analytical crystal together with a small slit (0.5 mm wide) placed facing the brush beam emitted by the MDCM. After adjusting the two angles of the analyzed crystal, the slit and the crystal are moved across the brush beam. The same area of analyzer is measured using the different pencil beam intercepted from the brush beam. Based on the theory mentioned above, it is possible to use the brush beam for the mapping lattice parameter measurement.

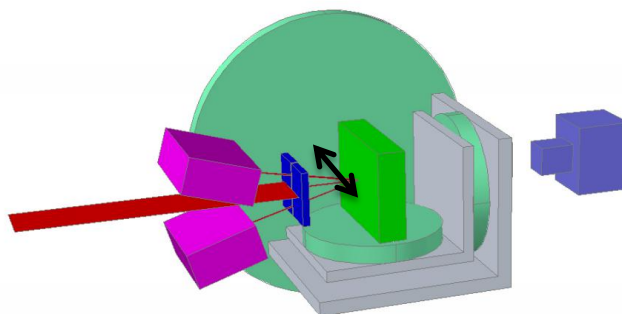


Figure 2. The experimental setup of the core part for system correction1. The direction of the black arrow is the direction of the analytical crystal and the slit scanning.

Figure 1 shows a scheme of the SRLC at the BL-3C of the Photon Factory, Japan. The brush beam emitted from the double crystal Monochromator Si111 is further monochromatic by the MDCM at location of 18 m downstream the SR source, and then the brush beam irradiated on the sample under measurement. A pair of Mythen 1K detectors are used to record intensities of the rocking curves. The posterior experimental processes are the same as those of using the pencil beam. Because of making full use of SR resources, the efficiency of brush beam scanning is more than ten times higher than that of the previous one. As a result, the experiment with more than 20 hours can be completed in less than 2 hours.

On the other hand, the Mythen detector is a photon counting type detector, which has a better SNR than the previous PIN detector. The reflection area is further reduced on the sample at the same intensity of incident beam. Mythen detector is composed of $50 \mu\text{m} \times 1280$ channels. The horizontal spatial resolution of 0.5mm can also be obtained by making 10 channels into a group.

Data analysis and discussion

Systematic correction of the SRLC with the brush beam was carried out using the analyzer crystal of ^{28}Si 4R.1 and a narrow slit, its results is shown in Fig. 3. Even a same area on the crystal was measured, but it is in sight that $\Delta d/d$ along the beam width is not a constant, because of that the horizontal divergence of the brush beam causes the wavelength distribution after the MDCM, and tilt angle difference at positions along the beam width. The relative d -spacing deviation is 1.2×10^{-7} .

This deviation can be calculated as follows:

$$\frac{\Delta d}{d} = \frac{\Delta \theta}{\tan \theta_B}$$

here $\Delta \theta$ contains two parts, one comes from the deviation from the central wavelength that combined with the Eq.(a),

$$\Delta \theta = \frac{1}{2} \cos(2\theta_B) \tan \theta_B \cdot \phi^2$$

The other part comes from the different tilt angle deviation to central position along the brush beam, given by

$$\Delta \theta = \frac{1}{2} \tan \theta_B \cdot \phi^2$$

The sum of two parts is

$$\frac{\Delta d}{d} = \frac{1}{2} (1 + \cos 2\theta_B) \cdot \phi^2 \quad \text{b)}$$

The beam width of 16mm at 18m from the SR source corresponds to 0.89 mrad beam divergence. For the MDCM used in the experiment, θ_B is 82.03° , and the deviation of relative d -spacing is about 4×10^{-7} , which is almost the same as the observed value.

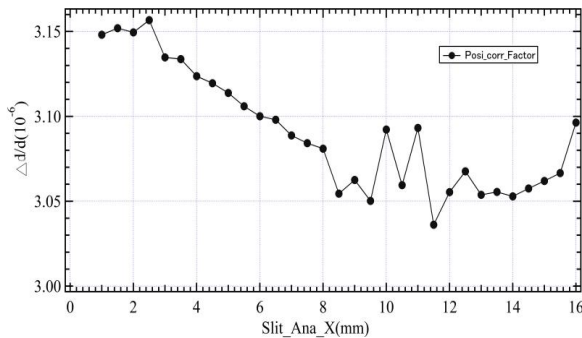


Figure 3. Correction factors as function of position X across of the brush beam

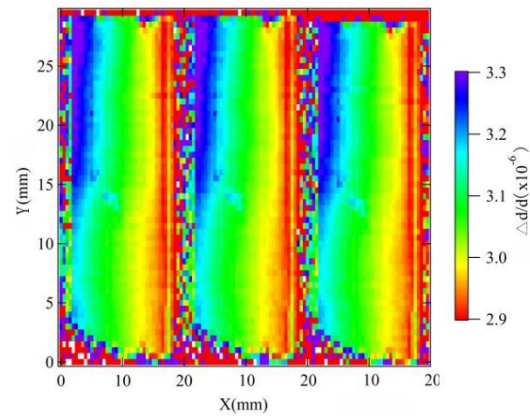


Figure 4. The results of three times mapping measurements at the same position on the sample using brush beam

We have obtained a preliminary d -spacing measurement using the brush beam scan. After considering the temperature correction, the final results are shown in Fig. 4, which is almostly consistent with results of the previous pencil beam mapping measurement. Furthermore, due to the use one pair of one-dimensional photon counting detectors with spatial resolution of 50 μm , the SRLC not only be improved in a point of view of the mapping measurement speed, but also be improved in a point of view of the spatial resolution, where the improvement is from the previous 1mm to less than 0.5mm.

Conclusion

We improved the original SRLC by equipped a pair of Mythen 1K detectors and effectively utilizing the brush beam of the bending magnet SR. After the improvement, the measurement was expedited by a factor of ten times; the one set of measurement time can be shortened to 2 hours from more than 20 hours before. At the same time, because of we use the photon counting detector of Mythen 1K, SNR of the rocking curve measurement is also improved. Under the same incident beam intensity, the diffraction area on the crystal can be reduced, the better spatial resolution of mapping measurement can be realized. The correction problem caused by different positions of the brush beam was overcome by establishment of the calibration method. With the system calibration and the data correction method, the new SRLC equipped with Mythen detectors and using the brush beam, it will be more powerful for characterization of silicon crystals.

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