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# Energy-resolved X-ray imaging method with a counting-type pixel detector

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ABSTRACT

We have developed an energy-resolved X-ray imaging method using the counting-type pixel detector PILATUS-100K. X-ray intensities were recorded as a scan of threshold energies, and the X-ray energy was determined by an s-curve fitting analysis. As a capability study of ultra precise energy-resolved imaging, X-ray beam intensities at 15.75, 15.76, 15.77, 15.78, 15.79, and 15.80 keV were measured and their threshold scan distributions could be clearly separated from each other. Laue diffraction patterns of a silicon steel sample were recorded with white X-ray beams. A grain image of silicon steel was obtained with a sample position scan. The reflected X-ray energy was also measured at three sample positions to analyze the lattice constant of the sample crystal grain.



## 1. Introduction

Synchrotron radiation science is part of the application of particle accelerators. Many common technologies are investigated for accelerators and detectors in high energy physics and synchrotron radiation science. Pixel detector technologies have progressed complementarily over the past 10 years. In particular, at PSI [1], two pixel detector projects, a compact muon solenoid (CMS) inner tracking detector for the Large Hadron Collider (LHC) at CERN and a pixel apparatus for the Swiss Light Source (PILATUS) have been developed. For these two projects, an indium bump-bonding technology was developed [2]. SPring-8 [3] has closely collaborated in the PILATUS project and contributed to module fabrication and then the development of calibration methods. Thus, a PILATUS-I single-module detector $217\,\mu m \times 217\,\mu m$ in pixel size and $157 \times 366$ in format was developed [4], and the PILATUS-1M with $3 \times 6$ modules was assembled [5]. In the next steps, a PILATUS-II single-module detector (PILATUS-100K) $172\,\mu m \times 172\,\mu m$ in pixel size and $195 \times 487$ in format was developed [6–8], and further developments of PILATUS-2M with $3 \times 8$ modules and PILATUS-6M with $5 \times 12$ modules were successfully completed [9,10].

In this study, we have developed a precise energy-resolved X-ray imaging method using the counting-type pixel detector PILATUS-100K, which is a hybrid silicon pixel detector containing a charge-sensitive amplifier, a shaper, a lower level discriminator and a 20-bit counter in one cell. X-ray intensities were measured as a scan of threshold energies and its distribution could be well reproduced by an s-curve fitting [7,8]. However, this threshold scan method is mostly only used for test purposes and calibrations in pixel detector developments. In this study, we have actively applied this technique to X-ray energy measurements and developed a color Laue method for a challenging application to create a next-generation synchrotron radiation analytic method.

## 2. Threshold energy calibration for PILATUS-100K

The PILATUS detector is based on the hybrid-pixel detector technology composed of a monolithic silicon sensor with pixelated two-dimensional pn-diodes and CMOS readout chips designed in $0.25\,\mu m$ UMC technology. Each pixel contains a charge-sensitive amplifier, a shaper, a single level discriminator and a 20-bit counter in one cell of the readout chip. The comparator thresholds can be controlled with a global threshold $V_{comp}$ commonly applied to all pixels and the trim voltage $V_{trim}$, individually adjusted with a 6-bit DAC for each pixel. In this study a custom-designed PILATUS-100K detector with a silicon sensor, $450\,\mu m$ in thickness, was applied. Threshold voltage calibration has been performed with fluorescent X-ray irradiation from standard samples such as Cu, Ge, Br, Zr, Mo, Pd, Cd, and Sn, which emits $K_\alpha$ fluorescent X-rays of 8.05, 9.89, 11.92, 15.77, 17.48, 21.23, 23.17, and 25.27 keV, respectively [11,12].

In the threshold calibration, the global voltage $V_{comp}$ is adjusted with that of all pixels count X-rays and the trim voltage $V_{trim}$ is adjusted with that of no pixel counts with all DAC bits on; then each DAC value is scanned and set at the actual threshold point. The global threshold voltage $V_{comp}$ is approximately represented by a linear function of the X-ray energy. The threshold trimming settings of $V_{trim}$ and the DAC values, on the other hand, depend only weakly on the X-ray energy. Therefore, the required threshold energy can be approximated using the closest trimming

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setting and by scaling the global threshold voltage $V_{comp}$ along the calibration curve.

## 3. X-ray beam energy measurements

In our previous study, the capability of the threshold scan method has been reported, where X-ray beam energies were measured to be 11.971 ± 0.007, 12.086 ± 0.006, and 12.203 ± 0.005 keV for the 12.0, 12.1, and 12.2 keV X-ray beams, respectively [12]. Such excellent accuracies did result from the precise energy calibration performed with fluorescent X-ray irradiation as described in Section 2.

In this study, we took the challenge of further obtaining fine energy steps of 10 eV. The experiment was carried out at BL14B2 of SPring-8 [13]. X-ray beams were attenuated with metal plates and irradiated onto the detector directly. The beam intensities were recorded as a scan of the threshold voltage from 3.75 to 850 mV with a step size of one DAC unit (about 2.42 mV). The trimming parameters were fixed at the result of Zr $K_\alpha$ fluorescent (15.77 keV) calibration.

The threshold scan distribution could be well reproduced by the s-curve fitting:

$$I = \frac{I_0}{2}\left(1+erf\left(\frac{V_{comp}-V_0}{\sigma\sqrt{2}}\right)\right)+A(V_{comp}-V_0)+B$$

where $I_0$, $V_0$, and $\sigma$ indicate the flux, the threshold voltage at the inflection point, and the square root of the threshold dispersion, respectively. The linear function for A and B is the correction term of charge sharing around the pixel boundary. Fig. 1(a) shows the measured X-ray beam intensities for 15.75, 15.76, 15.77, 15.78, 15.79, and 15.80 keV X-ray beams as a scan of the global threshold voltage $V_{comp}$ from 250 to 450 mV. The vertical axis values were normalized with $I_0$.

Fig. 1(b) shows the threshold scan distributions around the inflection points in the s-curve analysis and these patterns could be clearly separated from each other. The threshold voltages determined at the inflection points are plotted in Fig. 2. The errors were estimated using the s-curve fitting analysis. The solid line is the result of a linear function fitting, where the slope parameter is 71.7 mV/keV. In assuming 15.77 keV as the reference energy because of the use of Zr $K_\alpha$ fluorescent calibration, the X-ray energies were measured to be 15.745 ± 0.004, 15.759 ± 0.004, 15.775 ± 0.004, 15.786 ± 0.005, and 15.798 ± 0.005 keV for the 15.75, 15.76, 15.78, 15.79, and 15.80 keV X-ray beams, respectively.

## 4. Color Laue method

The Laue method is a traditional technique for X-ray crystallography. A sample crystal is irradiated with white X-ray beams and X-ray reflection spots are obtained symmetrically under certain conditions that satisfy the Bragg equation $n\lambda = 2d\sin\theta$, where $n$ is an integer (1, 2, 3, …, $n$), $\lambda$ the wavelength of the reflected X-ray, $d$ the distance between atomic planes, and $\theta$ the angle of incidence of the X-ray beam and the atomic planes. Under the standard method, a Laue diffraction pattern is usually measured with an integration-type area detector such as an imaging plate, a CCD-based detector or a flat-panel detector, and the indexing of the diffraction plane is performed only with the diffraction pattern information. If $\lambda$ for each diffraction spot is measured additionally, even an indexing deformed crystal is feasible and the lattice constant $d$ is exactly determined using the Bragg equation. Especially, the determination of $d$ is important for evaluating stress and strain information. One solution is a position scan with an energy-dispersive point detector of SDD,

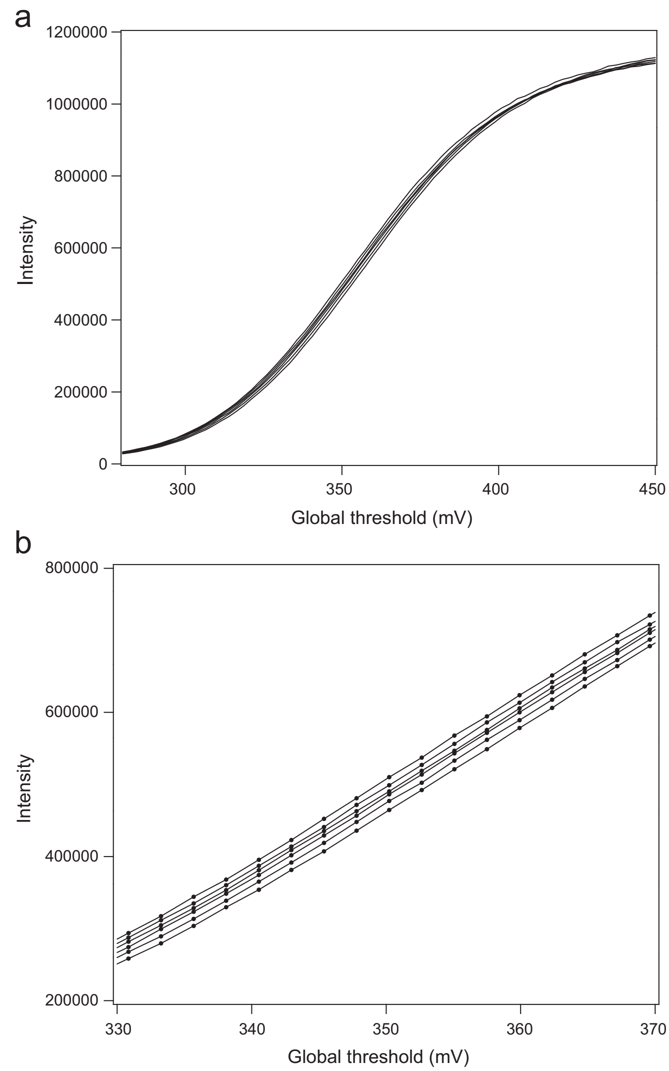


**Fig. 1.** (a) Global threshold scans for 15.75, 15.76, 15.77, 15.78, 15.79, and 15.80 keV X-ray beams. (b) Global threshold scans around the inflection points for 15.75, 15.76, 15.77, 15.78, 15.79, and 15.80 keV X-ray beams.

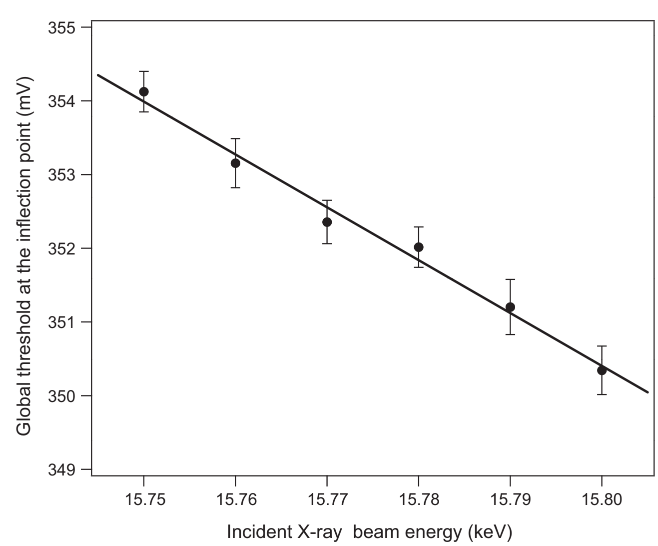


**Fig. 2.** Estimated threshold voltages at the inflection points for 15.75, 15.76, 15,77, 15,78, 15.79, and 15.80 keV X-ray beams.