Performance of *K*-edge subtraction tomography as an application of parametric x-ray radiation

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We developed a novel method for element detection utilizing an x-ray source based on parametric x-ray radiation (PXR), an accelerator-based light source at the Laboratory for Electron Beam Research and Application (LEBRA), Nihon University. The method is a type of *K*-edge subtraction (KES) imaging that uses the drastic change of x-ray absorption around the *K*-shell absorption edge of the target element. Using the properties of PXR, simultaneous KES imaging is possible, and can easily be applied to 3-dimensional (3D) computed tomography (CT). We demonstrated the feasibility of simultaneous KES-CT in our previous work. In this study, we investigate the quantitative performance of simultaneous KES imaging for a sample containing the element strontium (Sr) for which the *K*-edge energy is 16.1 keV. Results of a simultaneous KES-CT experiment employing the LEBRA-PXR source confirm that the imaging method can provide a 3D distribution of the element with a value proportional to the Sr concentration. Concerning sensitivity to element concentration, at least in the region of 0.5%-concentration, the sample was successfully distinguished from the region without Sr in the 3D tomographic image obtained using the element-imaging technique.

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I. INTRODUCTION

The Laboratory for Electron Beam Research and Application (LEBRA) is an accelerator-based light source facility for user applications at Nihon University [1]. The accelerator of LEBRA is a conventional electron linac with a typical energy of 100 MeV. Three kinds of light sources from the THz range to the x-ray region are actualized utilizing the LEBRA linac [2,3]. In particular, the x-ray source with a dedicated beamline is unique; it is based on parametric x-ray radiation (PXR), which is electromagnetic radiation resulting from the interaction between a relativistic charged particle and a crystal medium with periodic structures [4–6]. The x-ray energy from the PXR source is almost monochromatic, and can be adjusted by the angle between the crystal plane and the electron beam axis, regardless of the electron beam energy. In the PXR source a double-crystal system is used to extract the x-ray beam to the next experimental hall through the fixed port [7]. Since the electron energy of 100 MeV is much lower than that of GeV-scale synchrotrons, the angular spread of the radiation cone is relatively large compared with third-generation synchrotron radiation (SR) sources. The x-ray beam at the exit port has a substantially uniform profile of 100 mm in diameter. The main application of the PXR source, therefore, has been x-ray imaging, including advanced methods developed at SR facilities [8,9].

The LEBRA-PXR source is a pulsed x-ray source depending on the linac pulse structure. Consequently, the x-ray average intensity is restricted, due to the low duty cycle of the linac, and a considerably long exposure time is required for an x-ray image with a practical quality. The LEBRA linac, however, has sufficient long-term stability to perform experiments for computed tomography (CT) involving several hundred projection images. Actually, 3-dimensional (3D) tomographic images have been obtained using the LEBRA-PXR source [10]. The typical measurement time is in the range of 1-4 h, and it is acceptable as a user application in a machine time at the LEBRA facility. In addition to tomographic imaging, the PXR source has been frequently applied to *diffraction-enhanced imaging* (DEI), a kind of phase-contrast imaging that is capable of detecting the x-ray refraction and scattering caused by passing through a sample [11–14]. Since the x-ray refraction angle is limited to the μ rad order, the method requires strict spatial coherence for the x-ray source. In the case of the PXR source, DEI is possible despite the cone-like x-ray beam with an angular spread on the order of mrad, which can be compensated with the energy dispersion of x-rays in the PXR beam profile in the Bragg condition at the analyzer crystal [15,16]. PXR properties also allow us to form x-ray crossing

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beams with slightly different energies. When the energies of the two x-ray beams are on opposite sides of the *K*-shell absorption edge of a specific element, dual-color images across the *K*-edge energy can be obtained simultaneously. Since the x-ray absorption of a specific element drastically varies at the *K*-edge, subtraction of one image from the other provides information on the spatial distribution of the element. The method is a kind of element imaging called the *K*-edge subtraction (KES) method [17].

Using the LEBRA-PXR source, simultaneous KES imaging can be performed and a 2-dimensional (2D) image of a specific element can be obtained directly from one exposure. Therefore, 3D tomography of the element can be carried out by one-axis scanning of the sample. In our previous study, we demonstrated simultaneous KES experimentally using the LEBRA-PXR source [18]. This unique imaging technique could be a distinctive application of PXR. For practical user applications, however, the performance of this method needs to be investigated quantitatively. Here, therefore, we evaluate the sensitivity of the method to the concentration of strontium (Sr).

II. SPECIFICATION OF THE LEBRA-PXR SOURCE

The LEBRA linac has two beamlines for light sources. The first is an infrared free electron laser (FEL) beamline, and the second is used for PXR generation and coherent THz-wave radiation. In the PXR mode, the electron energy is set at 100 MeV. The target crystal–as PXR radiator–is placed in the PXR beamline after the 90° bending section following the linac. The intensity and duration of the electron macropulse are usually adjusted to be around 100 mA and 5 μ s, respectively, at the target crystal plate. The beam parameter must be empirically determined to avoid the destruction of the target crystal, generally a silicon monocrystal, due to the electron beam bombardment [19]. The repetition rate of the macropulse is 5 pps in ordinary operation, and the average current of the electron beam is restricted to approximately 3.3 μ A.

The target crystal is mounted on a goniometer stage in a vacuum chamber to control the PXR photon energy. Let us define the Bragg angle θ as the angle between the incident electron velocity v and the crystal plane with a reciprocal lattice vector g. When a PXR photon is emitted in the same plane as v and g, with an emission angle ϕ to v, the PXR energy is expressed as

$$\hbar\omega = \frac{\hbar c^* |\mathbf{g}| \sin\theta}{1 - \beta \cos\phi},\tag{1}$$

where c^* is the light speed in the crystal medium and $\beta = |\mathbf{v}|/c^*$ [20]. The PXR center energy at $\phi = 2\theta$ is equal to the ordinary Bragg energy $\hbar\omega_B = \hbar c^* |\mathbf{g}|/2 \sin \theta$. When the angle ϕ is slightly different from 2θ , by $\Delta\theta$, the PXR energy is approximated by

$$\begin{split} \hbar\omega &\approx \hbar\omega_B + \frac{\mathrm{d}(\hbar\omega)}{\mathrm{d}\phi} \Delta\theta \approx \hbar\omega_B \left(1 - \frac{\sin 2\theta}{1 - \cos 2\theta} \Delta\theta\right) \\ &= \hbar\omega_B \left(1 - \frac{\Delta\theta}{\tan \theta}\right), \end{split} \tag{2}$$

where $\beta \approx 1$ is used in these approximations [21,22]. Equation (2) indicates that the PXR beam has an energy dispersion in its radiation cone, the aperture of which depends on the electron energy. The center energy and energy shift due to the dispersion property depend only on the geometrical condition, and are independent from the electron energy. The direction of the PXR beam axis changes according to the variation of the PXR energy. Fortunately, we can diffract the whole PXR beam by employing a flat crystal plate with the same crystal plane as the target. When the second crystal is placed in the (+, -) arrangement against the target, the energy variation according to Eq. (2) and the angular shift in the cone-like shape compensate each other. As a result, all x-rays in the PXR beam simultaneously satisfy the Bragg condition at the second crystal. Strictly speaking, the spatial-spectraldistribution of PXR does not satisfy the condition of selfdiffraction in the target crystal [23]. The second crystal, however, can effectively diffract the whole of the PXR beam when the Bragg angle of the second crystal is slightly smaller ($\sim 0.01^{\circ}$) than the target crystal angle [7]. Using the double-crystal system, a PXR beam with wide tunability can be transported through the fixed port. Since the angular width of the rocking-curve at the second crystal depends on the electron energy, the reflectivity at the peak of the rocking-curve also depends on that energy [7,24]. In the case of a silicon monocrystal with high perfectness for the double-crystal system, however, reflectivity of at least 10% can be obtained for the 100-MeV electron beam. Actually, we have observed the rocking-curves at the second crystal with the widths almost equal to the result

TABLE I. Parameters of the LEBRA linac and the PXR source.

Electron energy	100 MeV (typical)
Electron energy spread	$\leq 1\%$
Accelerating frequency	2856 MHz (S-band)
Macropulse beam current	100-130 mA (typical)
Macropulse duration	$4-5 \ \mu s$ (typical)
Macropulse repetition rate	2–5 pps
Average beam current	1–3.3 µA
Beam size on the target	~0.5 mm in diameter (rms)
PXR energy range	
Si(111) target:	4.0–20.5 keV
Si(220) target:	6.5–33.6 keV
X-ray window	125-µm-thick PET film
Irradiation field	100 mm in diameter
Total x-ray photon rate	$\sim 10^7$ /s @17.5 keV
at x-ray window	
X-ray flux at 3rd crystal	$10^2 - 10^3 / \text{mm}^2 \text{ s}$



FIG. 1. Schematic top view of the PXR beamline and setup of the simultaneous KES imaging.

of the ray-tracing calculation based on the PXR property and the Darwin curve of a silicon perfect crystal [3].

The typical parameters of the LEBRA linac and PXR source are listed in Table I. The double-crystal system can cover the Bragg angle from 5.5° -30° corresponding to the x-ray energy range from 4.0–20.5 keV or from 6.5–33.6 keV for Si(111) or Si(220), respectively. Here, the irradiation field and the total x-ray photon rate are the values at the x-ray exit window of the polyethylene terephthalate (PET) that is behind a 2-m shield wall and located at a distance of 7.3 m from the target crystal [25]. The photon rate also includes the reflectivity of the second crystal and the cutoff effect due to the duct size in the x-ray transport line.

III. SIMULTANEOUS KES IMAGING

Energy dispersion in the PXR beam profile obeying Eq. (2) is conserved after the second crystal diffraction. Thus, the PXR beam extracted from the double-crystal system can also be efficiently diffracted by a third crystal in the (+, -, +) arrangement. In the case of the third crystal, the width of the rocking-curve is almost equal to the intrinsic width of a Si perfect crystal, and the reflectivity is improved to approximately 80%. The third crystal can be used as an analyzer crystal in the DEI experiments. Actually, we have obtained many phase-contrast images by the DEI method.

In contrast to usual DEI experiments performed at SR facilities, the x-ray beam used in the measurement has significantly large energy dispersion estimated to be several percentage points. When the third crystal is placed on the energy side lower than the center of the PXR beam, dualenergy crossing beams can be formed by reflecting only half of the PXR beam using the third crystal, and the crossing angle is equal to just twice the Bragg angle. Figure 1 contains a schematic of the setup with the double-crystal system of the PXR source. When the center energy of the PXR beam is adjusted at the *K*-edge energy of the element of interest, the two x-ray beams have energies on opposite sides of the *K*-edge. Then, the crossing beams allow simultaneous KES imaging for a sample located at

the intersection using an x-ray image detector with a large active area [18]. Although the sample size is restricted by the third crystal area, simultaneous KES-CT is possible by rotating the sample on one vertical axis. This is an advantage of the PXR-based KES compared to the simultaneous KES conducted using a bent-crystal system, referred to as spectral-KES at some SR facilities [26–28].

IV. INVESTIGATION INTO THE SENSITIVITY OF SIMULTANEOUS KES-CT

A. The sample and measurement conditions

To investigate the performance of simultaneous KES-CT using PXR, we carried out an experiment for Sr detection. Schematic top view of the experimental setup is shown in Fig. 2. Si(220) planes were used for the PXR source and the third crystal, and the PXR center energy was adjusted to the Sr K-edge energy of 16.1 keV. Since the Bragg angle was 11.54°, the parallax angle between the two images observed simultaneously was estimated to be 23.08°. Parameters other than the sample were the same as in our previous study, including the x-ray image detector, which was a flatpanel detector (FPD) with a Gd₂O₂S:Tb-based x-ray converter. In this study, the sample shown in Fig. 3 was prepared to investigate the sensitivity of this method to the concentration of Sr. The sample was five epoxy resin pellets colored with white pigment SrTiO₃ (STO), with Sr concentrations of 5%, 1%, 0.5%, 0.1% and 0% in weight ratios from the top.

In the simultaneous KES-CT experiment for this sample using a 16.1-keV PXR beam, 360 projection images were taken by 0.5°-step CT scanning. The exposure time of each image was 20 s, and total measurement time (gross) including margins for motor moving and data saving was estimated to be 7632 s. Figure 4 is a typical projection image taken using the FPD of 100 μ m × 100 μ m pixel size after the average dark image taken beforehand was subtracted and with some digital noise reduction. The dualcolor images of the sample are obtained from one projection image. The energy of the direct beam was higher than that of the reflected beam across the Sr *K*-edge, and the



FIG. 2. Schematic explanation of the experimental setup for simultaneous KES-CT.



FIG. 4. A typical projection image of the simultaneous KES-CT experiment taken by using the FPD with an active area of $128 \text{ mm} \times 128 \text{ mm}$.

energy difference between the beams was estimated to be approximately 200 eV at the position of the sample center. We found that the two x-ray transmittances were obviously different. In addition, the actual reflectivity of x-rays at the third crystal was estimated to be approximately 73%. The image by the reflected beam, therefore, had a statistical quality comparable to the direct beam image.



FIG. 3. Photograph of the sample for the simultaneous KES-CT experiment with the scale in mm. The sample was composed five epoxy pellets colored with STO at Sr concentrations of 5%, 1%, 0.5%, 0.1%, and 0% in weight ratios from the top.



FIG. 5. The fluctuations of image pixel values during the CT measurement as functions of the projection image number or time. The mean values of the pixel values in the dark field corresponding to the rectangle of the dashed yellow line in Fig. 4 are plotted with the black line. The blue line indicates the fluctuation of the mean value in the bright field (the dashed blue rectangle in Fig. 4) relative to the average overall projection images.



FIG. 6. The relative fluctuation of mean value of the pixel values in the rectangle area of the dashed red line in Fig. 4 after brightness correction.

B. Stability during the KES-CT measurement

Since the measurement time of the simultaneous KES-CT was longer than 2 h, it was possible that the instability of the PXR source, including the linac and measurement system, affected the image quality of reconstructed tomographic images. For instance, the dark noise of the FPD depends strongly on the room temperature because of the characteristic of the CMOS sensor. Actually, the mean values of the pixel values in the dark field defined as the region of interest (ROI; indicated by the dashed yellow line in Fig. 4) fluctuated during the CT measurement, as shown by the black line in Fig. 5. The blue line in Fig. 5 indicates the relative fluctuation of the mean pixel values in the bright field surrounded by the dashed blue line, which contains both the fluctuations of the dark noise and the x-ray intensity. As the brightness fluctuation was not overly large, we simply performed a brightness correction by dividing each projection image by the mean value in the bright field after subtracting the dark noise variation. As a result, the fluctuation of the mean value of the pixel values in the reflected beam area surrounded by the dashed red line was suppressed, as shown in Fig. 6. The remaining fluctuation was less than $\pm 5\%$ at most. Considering the diffraction width of the third crystal was less than 0.001°, the stability of the system was sufficient during the simultaneous KES-CT scanning.

C. 3D distribution of the Sr element

From the projection images of the simultaneous KES-CT, two 3D tomographic images could be reconstructed using the filtered back projection (FBP) method, which is commonly used for CT reconstruction [29,30]. Figures 7(a) and 7(b) show the 3D volume rendering for these tomographic images and its cut-surface, respectively. The images on the left are those of the energy lower than the Sr-K-edge, while those on the right are the energy higher than the K-edge. In these figures, the parallax between the images due to the beam crossing angle of 23.08° is corrected, and the image contrast is normalized at the epoxy resin region without STO (0%). The image pixel value of the tomographic images expressed using a pseudocolor set corresponds to the absorption power for x-rays in an arbitrary unit. The absorption power clearly depends on the concentration of Sr, especially in the case of higher energy than the K-edge.

We considered that only the absorption power contributed by Sr was obtained by subtracting the lower energy image from the higher energy image. The results of the subtraction are shown in Fig. 7(c) and 7(d). Since the two tomographic images have been already normalized with respect to the intensity and the profile of the PXR beam, the subtraction was simply performed without additional normalization. The tomographic images correspond to the 3D distribution of the Sr element in the sample. Here, relatively strong artifact noise is conspicuous around the 5%-concentration pellet in the higher-energy tomographic



FIG. 7. (a) 3D volume rendering for tomographic images reconstructed from the KES-CT data and (b) its cut-surface. The volume rendering (c) and cut-surface (d) are for the subtraction between the two tomographic images. The contrast of these images is expressed using a pseudo-color set.



FIG. 8. The image value of the subtraction tomographic image as a function of Sr concentration (weight ratio) in the sample. Red and blue lines represent linear regression for the data set with and without 5%-concentration point, respectively.

image. The x-ray absorption in this pellet is too strong, and it is difficult to accurately measure x-ray transmittance in each projection image due to the limitation of the photon flux of the PXR source. This leads to increased error in the CT reconstruction process.

D. Sensitivity to Sr concentration

To quantitatively confirm the sensitivity of simultaneous KES-CT, we plotted the image value of the subtraction tomographic image as a function of Sr concentration in the sample, as shown in Fig. 8. The image value was defined as the mean pixel value in a 3D ROI set within each pellet volume, and the error was calculated as the standard deviation of the pixel values. The red line represents the linear regression for all five data points, and the estimated correlation coefficient was 0.9592. The image value was almost linear to the Sr concentration, and at least the 0.5%-concentration point would be significantly distinguished from the absence of Sr.

In addition, the spatial uniformity of the 5%-pellet was lower than others. Thus, the reliability of the 5%-concentration data was somewhat inferior, and the error bar of the point is much larger than the others. If the 5%-concentration data were masked, the linear regression line obtained would be the blue line in Fig. 8, and the correlation coefficient would be improved to 0.9976, even though the number of the data points is small.

V. CONCLUSIONS

Based on the results of the simultaneous KES-CT experiment for the sample containing Sr, we conclude that the proposed method is capable of providing 3D distribution of the concentration of a target element. The retrieval

value of each pixel in the tomographic image had sufficiently good linearity to the concentration of the element. This means that the element-imaging technique can be applied to quantitative analysis in a nondestructive manner. In the case of Sr, the method was sensitive to at least 0.5% concentration in weight ratio. On the other hand, a too high concentration of the target element makes it difficult to accurately evaluate the concentration due to the lack of photons passing through the sample. An exposure time longer than 20 s for one projection image is necessary to measure the 5% sample using the LEBRA-PXR source and the existent x-ray image detector. Taking the practical machine time of the linac into account, the upper limit of the concentration that the method can treat at present seems to be several percentage points. With respect to the lower concentration limit, a concentration of a couple of 0.1% might be detected if we could suppress the system instability, especially the detector dark-noise.

Although the absolute sensitivity to element concentration is far inferior to that of x-ray fluorescence analysis, the method can treat samples with cm-scale thickness. We consider this novel approach to element imaging to be one of the promising applications of a PXR-based x-ray source.

When PXR sources based on new accelerators are developed that can provide substantially more photon flux than the present LEBRA-PXR source, it is expected that the dynamic range of element detection will be expanded to become a practical analytic tool [31].

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