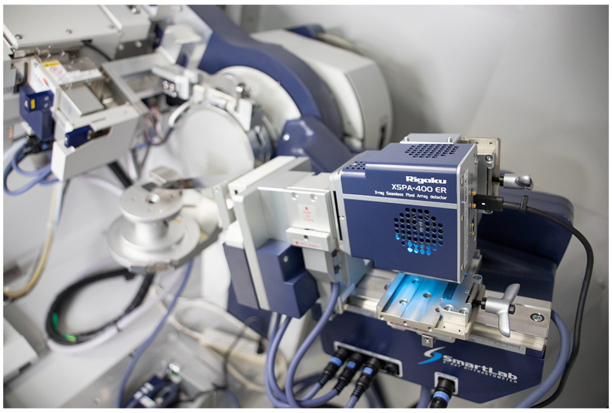
X-ray seamless pixel array detector

XSPA-400 ER



1. Introduction

The XSPA-400 ER (XSPA: X-ray Seamless* Pixel Array, ER: Energy Resolution) is a next-generation 2D semiconductor detector with a higher energy resolution than conventional models. With this higher energy resolution, the XSPA-400 ER reduces X-ray fluorescence, which can be a significant source of background intensity for powder diffraction patterns on samples containing transition metal elements. In addition to 0D and 1D measurements, 2D measurements are also available. The 2D mode allows the user to observe Debye-Scherrer rings, which provide information about sample orientation and the existence of coarse particles. Furthermore, the $75\,\mu\mathrm{m} \times 75\,\mu\mathrm{m}$ pixel size provides high spatial resolution. These features contribute to improved accuracy in quantitative analysis of trace crystalline phases, precise analysis of lattice constants, and 2D stress analysis of samples such as steel and battery materials that contain transition metal elements.

*Seamless Pixel Detectors: Typical hybrid pixel detectors use a tiled array of readout ASICs (Application Specific Integrated Circuits). With these tiled array detectors the pixel shapes at the IC boundary differ from those in other areas on the IC, requiring correction of the IC boundary. In addition, the correction sometimes remains incomplete depending on the measurement conditions, leaving an intensity difference between the boundary and other non-boundary areas on the IC. As a better solution, the seamless pixel detector has the same pixel shape over the whole IC, eliminating the need for IC boundary correction and thus producing a uniform image.

2. Multidimensional Measurements with the Same Detector

The XSPA-400 ER can be used not only as a 2D detector but also as a 0D and 1D detector. This flexibility allows this detector to be used for optical system alignment using 0D mode, as well as 1D and 2D powder pattern measurements.

3. Low Background Measurement

The XSPA-400 ER has very high energy resolution, which can reduce the influence of X-ray fluorescence from the sample, and thus gives a lower background (BG) in the measured data. Figure 1 shows the sensitivity of different types of detectors where the energy distribution is calculated assuming a normal distribution centered at Cu Kα. Compared to conventional detectors, the XSPA-400 ER has a much narrower energy distribution and can discriminate between Cu Kα and the X-ray fluorescence from transition metal elements such as Mn, Fe, Co, and Ni.

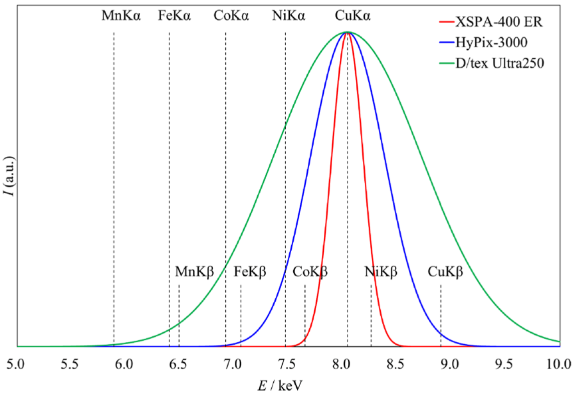


Fig. 1

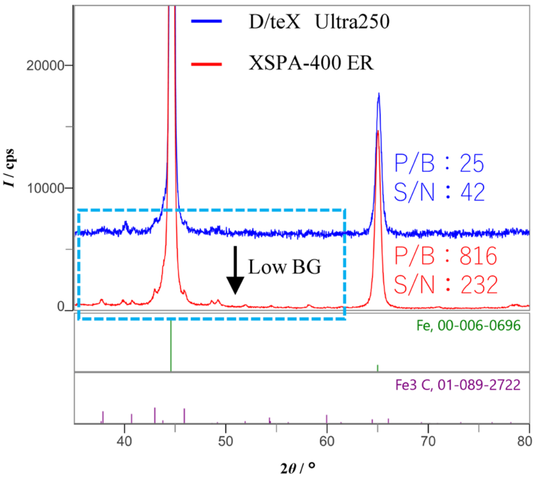


Fig. 2

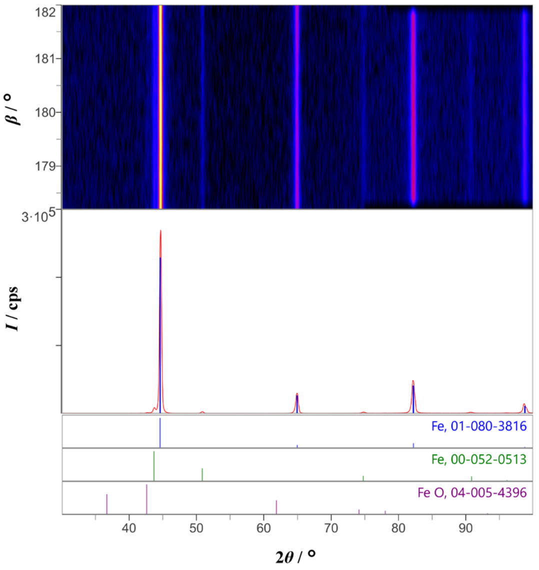


Fig. 3

This enables low-background X-ray diffraction patterns to be obtained even if the sample contains substantial levels of transition metal elements.

Figure 2 shows the measurement results of a chrome-molybdenum-steel sample using the XSPA-400 ER. The peak-to-background (*P*/*B*) and signal-to-noise (*S*/*N*) ratios are higher than those of conventional detectors because the background is suppressed, making it easier to find small peaks.

Furthermore, as shown in Figure 3, the Debye-Scherrer rings can be clearly observed in 2D detector mode with a low background. With the enhanced P/B and S/N performance of the XSPA detector in 2D mode, it is ideal for high-speed in-situ measurements, stress analysis and texture determination using a small spot X-ray source.

4. Combination with Flat Multilayer Mirror CBO-α

Although the function of the XSPA-400 ER alone is sufficient to suppress fluorescence background, there can be specific measurements that require further reduction of additional background contributions. As an example, the Cu-Kα distribution in Figure 1 shows that the Co Kβ and Ni Kβ lines overlap with the Cu-Kα energy distribution of XSPA-400 ER. The intensities of these Kβ components are no more than about 20% of the corresponding Kα components. Still, a slight energy overlap remains even after being reduced by the detector's high energy resolution, contributing to the increase in background level. This is especially noticeable when measuring the cathode material $\mathrm{Li(Ni, Co, Mn)O_2}$. The background contributions of these additional fluorescence contributions can be further suppressed by employing CBO-α(1), a flat multilayer mirror unit. The CBO-α is an optical element located on the incident X-ray side that can monochromatize the continuous and characteristic X-rays emitted from the X-ray tube into an almost pure Kα component and can be used with Bragg-Brentano para-focusing geometry. With this unit, the continuous X-rays, which are the primary source of excitation for X-ray fluorescence, do not irradiate the sample, and the generation of X-ray fluorescence from the sample can be suppressed.

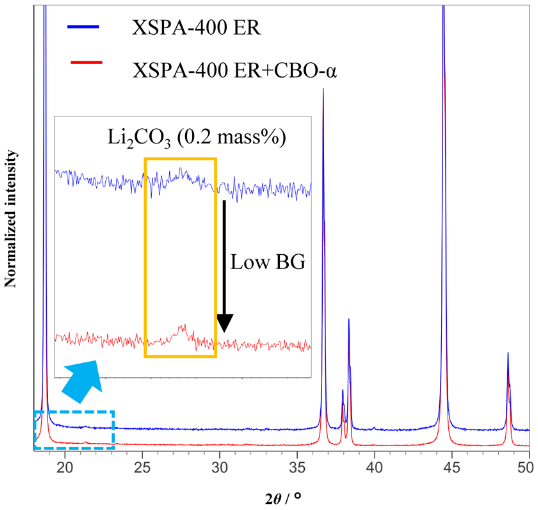


Fig. 4

Figure 4 shows the measurement results for $\mathrm{Li(Ni, Co, Mn)O_2}$, a common cathode material for lithium-ion batteries, with and without the CBO-α. It demonstrates that the measurement with the CBO-α gives significantly lower BG than the one without. This makes the peak of Li_2CO_3, which accounts for 0.2 mass% of the sample, easier to observe. The combination of the XSPA-400 ER and CBO-α can contribute to the detection of trace constituents and the improvement of their quantification accuracy.

5. Excellent Kβ/Kα Ratio

Generally, characteristic X-rays emitted from an X-ray tube include Kα and Kβ lines, while most X-ray diffraction measurements use only the Kα line for analysis. Ideally, the Kβ component should be removed and the beam should be monochromatized into a pure

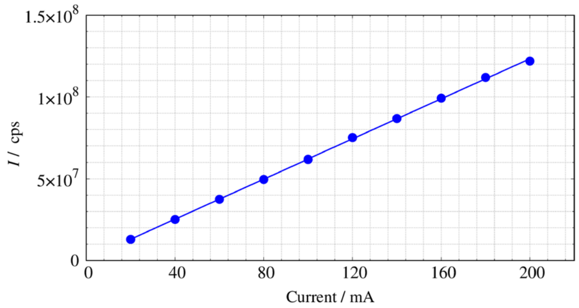


Fig. 5

Kα line. For Bragg–Brentano para-focusing geometry, for example, a metal filter or crystal monochromator is commonly used. Although these tools have been in use for a long time, they unavoidably weaken the Kα component when they remove the Kβ line. With its high energy resolution, the XSPA-400 ER can reduce the effect of diffracted Kβ lines by itself. This eliminates the need for conventional monochromatization methods and avoids unwanted attenuation of Kα lines.

6. High Counting Rate

In general, the count rate becomes lower when energy resolution is prioritized due to the principle of detector functionality. As count rate is usually limited per detection element, by having more pixels involved in measuring strong peaks using a 2D pixel detector rather than a 1D strip detector, the overall count rates and linearity for the XSPA detector greatly exceed those of traditional strip detectors. Because of this, the XSPA-400 ER pixel detector is suitable for both high-intensity measurements using a high-power source, and for measurements that require highly sensitive detection, such as epitaxial thin films. Figure 5 shows the results of 004 diffraction peak intensity measurements of a Si substrate with various tube current levels. With the high counting rate of the detector, the diffraction intensity does not saturate and shows a good linearity. Therefore, the XSPA-400 ER can be used for a wide range of sample types, from powder samples to thin films.

7. Switching between the Vertical and Horizontal Mounts of the Detector

The XSPA-400 ER has a rectangular detector surface measuring 9.6 mm (H) × 38.4 mm (W), and can be mounted in either vertical or horizontal orientation. For example, as shown in Figure 6, the vertical configuration can be used for 2D exposure measurement when a wide range in the 2θ direction is desired, and the horizontal configuration can be used when a wide range in the β direction is desired. For 1D measurements, on the other hand, the horizontal configuration provides a narrower detection area than the vertical configuration, but enables high-resolution measurement of diffraction peak widths by reducing the influence* of aberrations in the optical system.

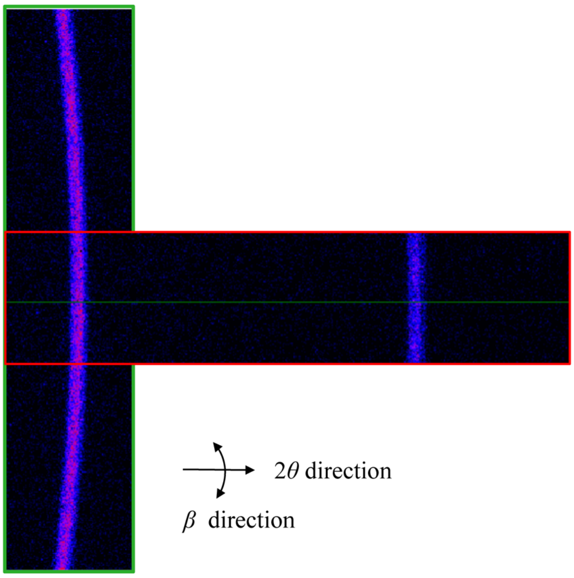


Fig. 6

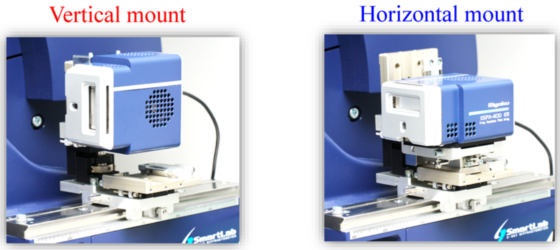

Vertical mount
Horizontal mount

Fig. 7

*The focal plane of the para-focusing method is cylindrical while the detection surface is flat. The X-ray focal point coincides with the center of the detection surface, but as one moves toward the edge of the detection surface (2θ direction), it no longer coincides with the focal point. This phenomenon is called defocusing, and the diffraction peak width becomes wider.

The newly developed detector holder (Fig. 7) allows anyone to easily and reproducibly switch between vertical and horizontal mounting.

Technical specifications	
Detection type	Direct detection, single photon counting
Detection element	Silicon
Pixel size	$75\,\mu m \times 75\,\mu m$
Number of pixels	65,536 pixels
Detection area	$9.6\,mm \times 38.4\,mm = 368.64\,mm^2$
Max count rate	$> 1 \times 10^5$ cps/pixel
Energy range	Cr, Co, Cu, Mo, Ag
Energy efficiency (Cu Kα)	99%
Energy resolution (Cu Kα)	340 eV (XRF reduction mode)

References

T. Osakabe: Rigaku Journal, **33**(1) (2017), 15–19.