X-ray Seamless Pixel Array Detector

XSPA-200 ER

—High energy resolution detector for a benchtop X-ray diffractometer—

Abstract

In X-ray diffraction measurements using a Cu source, transition metals in the sample—for example, batteries and steel materials—generate fluorescent X-rays. These fluorescent X-rays raise background intensities in the measured data, making it difficult to detect peaks derived from trace crystalline phases. The new "XSPA-200 ER" detector, which can be mounted on a benchtop X-ray diffractometer, has high energy resolution, enabling measurements with low background intensities.

1. Introduction

The XSPA-200 ER X-ray Seamless Pixel Array detector is a multidimensional pixel detector for the MiniFlex benchtop X-ray diffractometer. The most significant difference between the XSPA-200 ER and the conventional detector for MiniFlex is its energy resolution. The XSPA-200 ER is a compact detector with the same high energy resolution as the "XSPA-400 ER"[(1)], which can be installed in the SmartLab automated multipurpose X-ray diffractometer. X-ray diffraction (XRD) patterns measured by XSPA-200 ER have low background intensities because the detector discriminates fluorescent X-rays generated from the sample. The XSPA-200 ER enables the MiniFlex to obtain higher quality measurement data. This paper introduces the features of the XSPA-200 ER with actual examples.

2. Features of XSPA-200 ER

2.1. Low background measurement

CuKα radiation is generally used for XRD. When transition metals such as Cr, Mn, Fe, and Co are contained in a sample, fluorescent X-rays derived from these transition metals are generated by CuKα radiation. The energy of these X-rays is known to be close to that of the CuKα X-rays. Figure 1 shows the energy distribution of three different detectors calculated as a normal distribution using the energy resolution for CuKα X-rays, and the fluorescent X-ray energy of each element. A narrower energy distribution indicates higher energy resolution. If the energy resolution of the detector is low, the background intensity of the diffraction pattern increases because the CuKα line cannot be discriminated from the fluorescent X-rays of transition metals. The XSPA-200 ER has a narrow energy distribution, and its energy resolution is high enough to discriminate between the CuKα line and transition metal fluorescent X-rays. Therefore, the XRD patterns obtained by the XSPA-200 ER have low background intensity.

Figure 2 shows the XRD patterns of lithium nickel cobalt manganese oxide ($Li(Ni_{1/3}Co_{1/3}Mn_{1/3})O_2$: NCM111), a positive electrode material for lithium ion batteries, as an example of measurement. The XRD patterns were obtained by the XSPA-200 ER and a conventional detector, D/teX Ultra2. The XRD pattern by the XSPA-200 ER showed low background intensities compared to the results from the D/teX Ultra2. The PB (peak to background) ratio calculated from the strongest peak of NCM111 was 19 with the D/teX Ultra2 and 54 with the XSPA-200 ER, resulting in an improvement of about 2.8 times. As a result, the peak of trace lithium carbonate was clearly observed by the XSPA-200 ER.

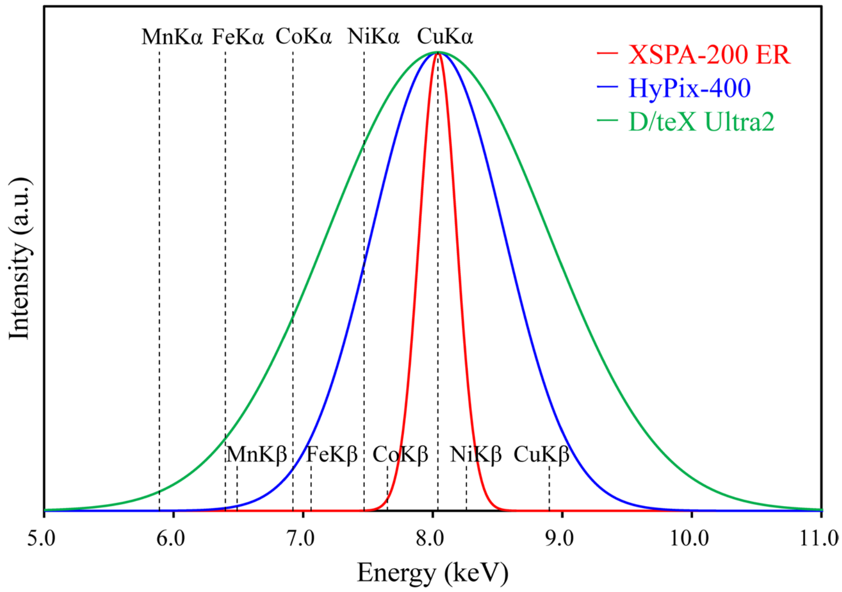

Fig. 1. The energy distribution of three detectors calculated using the energy resolution for the CuKα line, and the fluorescent X-rays energy of each element.

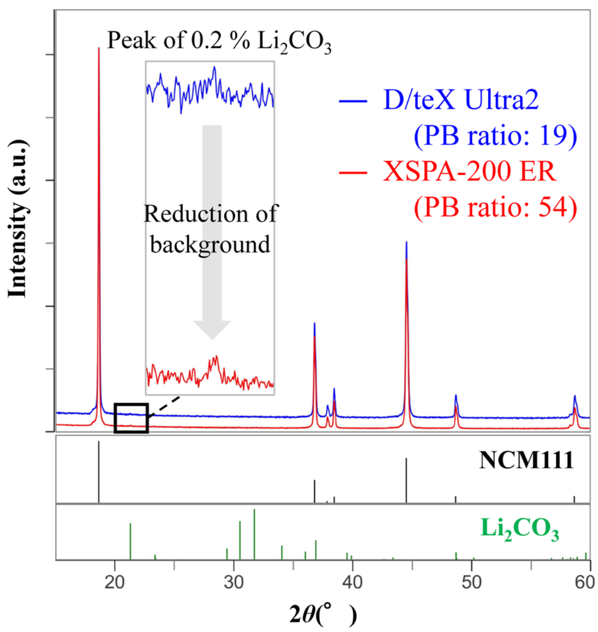

Fig. 2. Comparison of XRD patterns of NCM111 measured by two different detectors.

2.2. High reduction of Kβ line

Characteristic X-rays emitted from an X-ray tube include both Kα and Kβ lines. Generally, XRD measurements use only the Kα line for analysis. Therefore, the Kβ line should be removed, leaving only the Kα line. For example, a metal filter or a crystal monochromator is commonly used in the Bragg-Brentano parafocusing geometry(2). Although these methods are effective, it is inevitable that the Kα line intensity decreases when removing the Kβ line. XSPA-200 ER with its high energy resolution can reduce the intensity of Kβ without attenuating the Kα line. By selecting filterless measurement, the XSPA-200 ER enables measurement with high intensity.

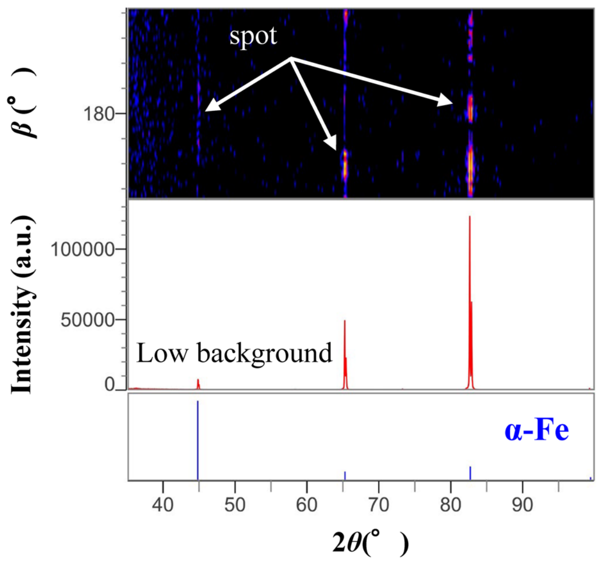

Fig. 3. 2D measurement data of steel material.

2.3. Multidimensional measurements of XSPA-200 ER

The XSPA-200 ER can be used as a 0D, 1D, and 2D detector. Alignment of the instrument using 0D measurement, data collection of high-quality XRD patterns by 1D measurement, and observation of Debye rings by 2D measurement can all be performed without replacing the detector. Figure 3 shows the results of a 2D measurement of a steel sample using the XSPA-200 ER. Low background data were obtained even for this steel material, which generates fluorescent X-rays derived from iron. Because the Debye rings are spotty, the sample contains coarse particles. The 2D measurement by the XSPA-200 ER enables low-background measurement and provides information on the sample condition, such as the presence of coarse particles and preferred orientation.

Table 1. XSPA-200 ER specifications.

Counting method	Direct detecting photon counting
Sensor	Silicon
Pixel size	75 μm × 75 μm
Number of pixels	32,768 pixels
Detect area	9.6 mm × 19.2 mm = 184.32 mm^2
Count rate	> 1 × 10^5 cps/pixel
Correspond wavelength	CoKα, CuKα
Detect efficiency (at CuKα)	99%
Energy resolution (at CuKα)	340 eV (in fluorescent X-ray reduction mode)

2.4. Specifications

Table 1 shows the specifications of the XSPA-200 ER.

3. Conclusion

The MiniFlex benchtop X-ray diffractometer equipped with XSPA-200 ER enables you to obtain higher-quality data. As mentioned above, the most important feature of the XSPA-200 ER is its high energy resolution. The XSPA-200 ER is useful especially for analyzing samples containing transition metals such as Cr, Mn, Fe, and Co; for example, steel and battery materials.

References

(1) *Rigaku Journal*, 39 (2023), No. 1, 23–26.
(2) M. Omori: *Rigaku Journal*, 37 (2021), No. 1, 12–19.

