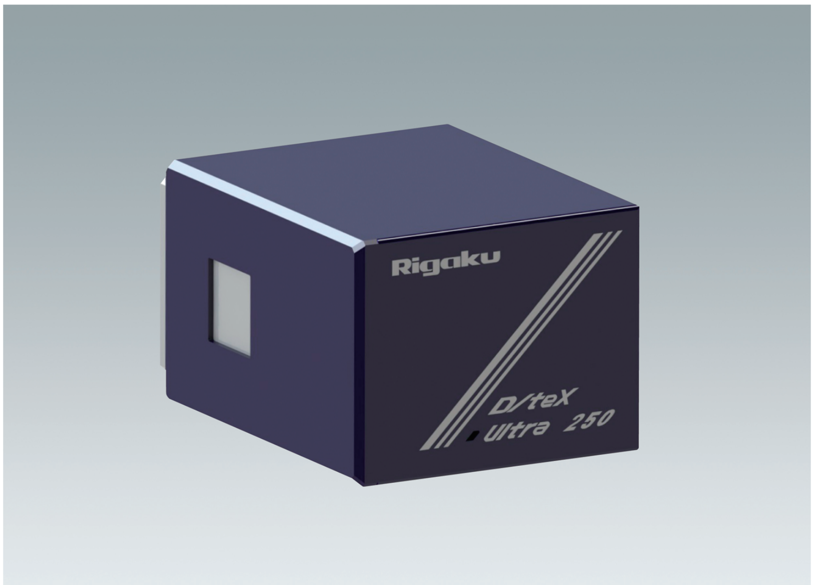
High-speed 1D silicon strip X-ray detector

D/teX Ultra 250



1. Introduction

In X-ray diffractometry, a variety of detectors are used depending on the purpose of the measurement. In recent years, in addition to the previously used 0D detectors (scintillation counters etc.), there has been increasing use of 1D detectors in which multiple detector elements are packaged into a single unit using the latest semiconductor technology. These semiconductor 1D detectors enable measurement with high-intensity using multiple elements, high-resolution with a strip width of 100 microns or less, and a high P/B (peak/background) ratio.

The D/teX Ultra 250 1D silicon strip X-ray detector introduced here is a new type of detector that makes major improvements on previous 1D detectors, and achieves dramatically improved performance. The D/teX Ultra 250 can measure extremely high-intensity data, approximately 150 times that of a scintillation counter (SC) (Fig. 1). In addition to detection of even smaller trace components and micro-area measurements on

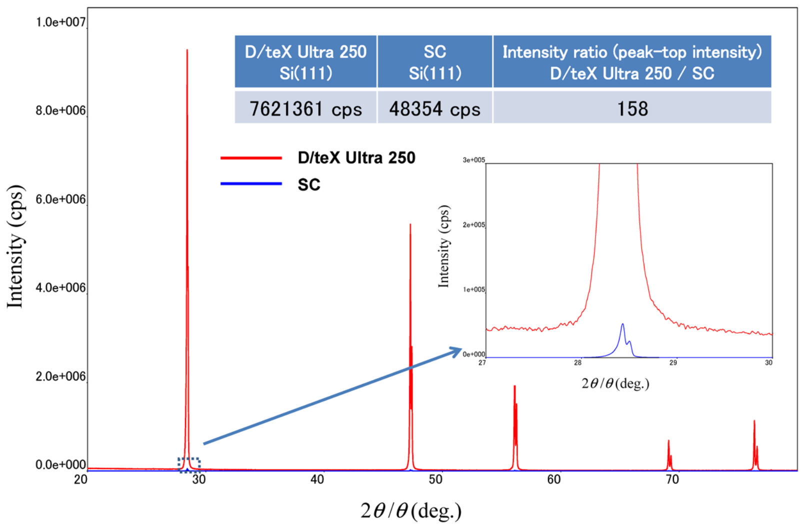


Fig. 1. Comparison of diffraction intensity of D/teX Ultra 250 and scintillation counter (SC) (sample: Si powder).

small samples, it also enables increased speed in tasks such as multi-point measurement, in-situ measurement and reciprocal space mapping measurement. These detectors work well in a variety of fields, including R&D and quality control.

2. Product features

2.1. High-intensity, high-resolution measurement

In focusing and the Bragg-Brentano parafocusing geometry measurement using a 1D silicon strip detector, X-ray diffraction intensity increases in proportion to the active area of the detector. The D/teX Ultra 250 detector has the largest active area in the industry among 1D silicon strip detectors, 384 mm^2 (width 19.2 mm × height 20.0 mm), and this enables measurement at a high-intensity 1.5 times the previous D/teX Ultra (Fig. 2(a), (b)). However, expansion of the detection width with respect to the scanning direction causes a drop in angle resolution. Thus, with the D/teX Ultra 250, the width of a strip was decreased to 75 microns for higher angular resolution (the previous D/teX Ultra had a width of 100 microns), and also a larger active area was achieved while suppressing the drop in resolution by increasing the number of channels to 256 (the D/teX Ultra had 128 channels).

2.2. XRF reduction mode

The D/teX Ultra 250 has an energy discrimination function, and can selectively capture only a specific energy range. By using this function, it is possible to efficiently reduce X-ray fluorescence generated from the sample. For example, when measuring iron-based samples with a high absorption coefficient using CuKα radiation, the Fe atoms in the sample are strongly excited by the CuKα X-rays which are the incident X-rays, and thus extremely high FeK X-ray fluorescence is observed as the background. With the D/teX Ultra 250, however, it is possible to selectively count only the CuK rays, and thus it is possible to obtain measurements with a high P/B ratio in which the background has been suppressed (Fig. 3(a), (b)). In addition, a special-purpose monochromator is available as an option, and this enables even further improvement in the P/B ratio.

2.3. High count rate

The D/teX Ultra 250 is equipped with an ASIC

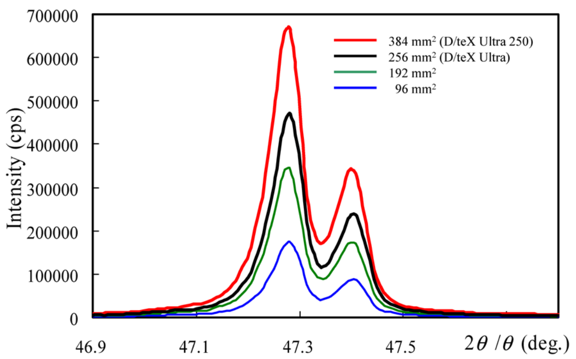


Fig. 2(a). Measurement results for Si powder (220) reflection using high-speed 1D silicon strip detectors with different active areas.

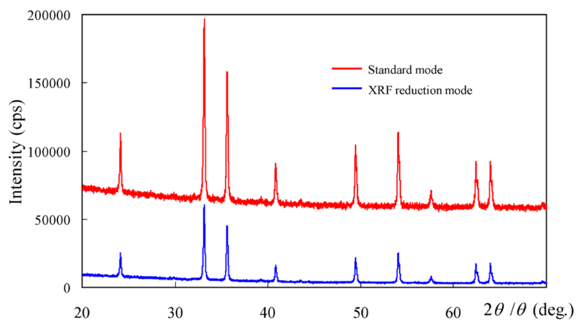


Fig. 3(a). Effect of XRF reduction mode of the D/teX Ultra 250 (sample: iron oxide power, measured with the CuKα rays.)

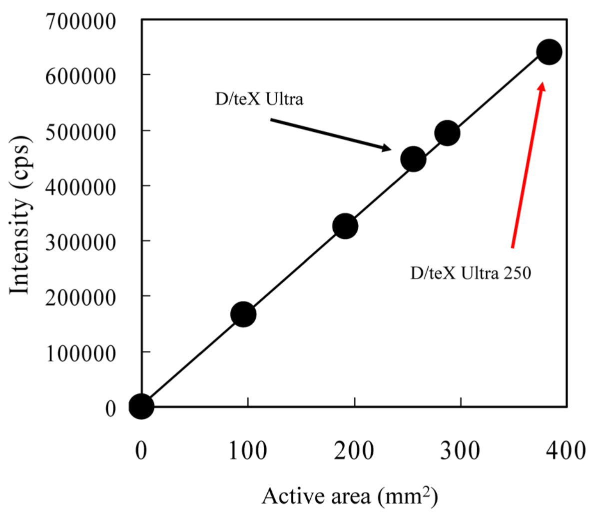


Fig. 2(b). Relationship of active area and diffraction intensity (peak-top intensity).

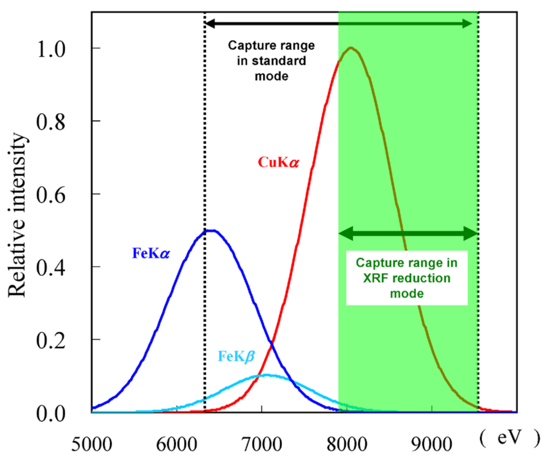


Fig. 3(b). X-ray energy spectra obtained from D/teX Ultra 250.

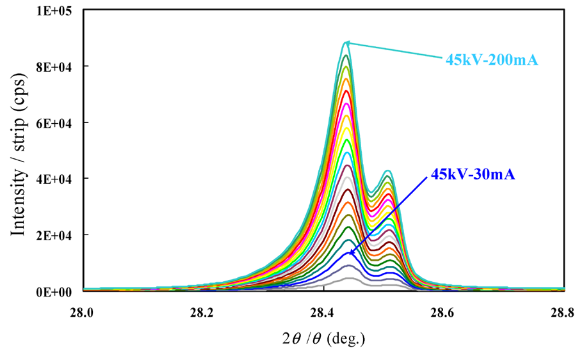


Fig. 4(a). Results of high-speed parafocusing geometry measurement of Si powder (111) reflection. The intensity axis is converted to the value per strip. Measurement was done with a tube voltage of 45 kV and tube current of 10–200 mA.

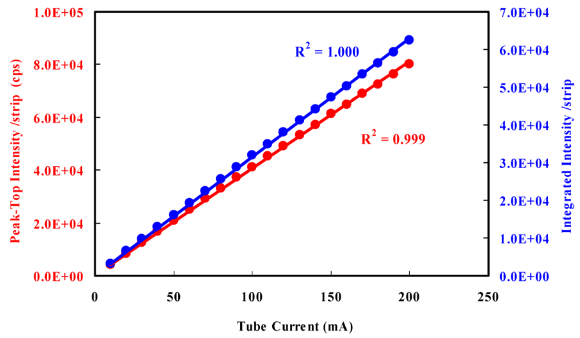


Fig. 4(b). Results of measuring counting linearity of D/teX Ultra 250. The intensity axis is converted to the value per strip. Si powder (111) reflection was measured with a tube voltage of 45 kV and tube current of 10–200 mA.

(Application Specific Integrated Circuit), produced by making Rigaku's unique counting circuit into a custom chip. This ASIC has unprecedented high speed, and thus each of the detector strips configured in 256 channels has a high counting linearity of 10^6 cps (value for the detector as a whole is 2.5×10^8 cps or more). Therefore, it is possible to conduct measurements using a high brightness rotating anode X-ray generator, and guaranteed counting linearity can be achieved (Fig. 4(a), (b)).

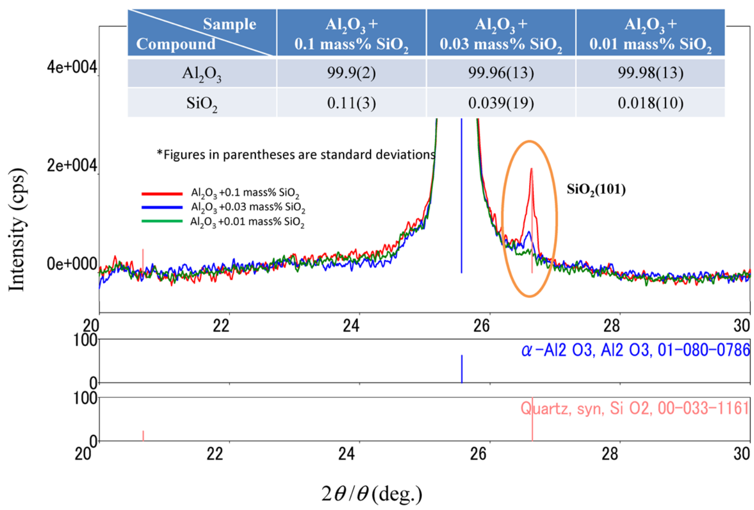
2.4. Applications

2.4.1. High-speed measurement of trace samples

It is extremely difficult to detect trace components of a few % or less whose diffraction intensity is faint. However, the detection limit can be greatly improved with a high-speed parafocusing geometry measurement using the D/teX Ultra 250. As an example of this type of analysis, Fig. 5 shows the results of measuring trace SiO_2 in Al_2O_3. The measurement time is short, approximately 6 minutes per sample, and the SiO_2 (101) reflections of samples adjusted to extremely trace SiO_2 concentrations of 0.1, 0.03 and 0.01 mass% are clearly observed. In addition, when quantitative analysis was performed using the Rietveld method, the values obtained through analysis exhibited an extremely good match with adjusted values. Even if the system is like that in this example, where there is a mixture of a main component (Al_2O_3) observed with a high diffraction intensity, and a trace component (SiO_2) which exhibits a minute diffraction intensity, accurate quantitative analysis can be done with the D/teX Ultra 250 due to its outstanding counting linearity.

2.4.2. High-speed measurement of multiple samples (multi-point high-speed measurement)

With a high-speed parafocusing geometry measurement using the D/teX Ultra 250, a measurement can be



Sample / Compound	Al_2O_3 + 0.1 mass% SiO_2	Al_2O_3 + 0.03 mass% SiO_2	Al_2O_3 + 0.01 mass% SiO_2
Al_2O_3	99.9(2)	99.96(13)	99.98(13)
SiO_2	0.11(3)	0.039(19)	0.018(10)

Fig. 5. Example of high-speed parafocusing geometry measurement by the D/teX Ultra 250 of samples containing a trace component.

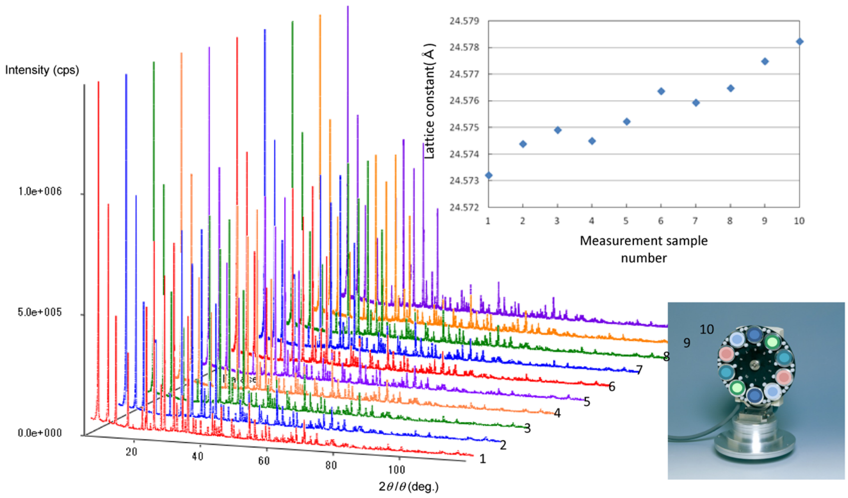


Fig. 6. Example of high-speed parafocusing geometry measurement of multiple samples (sample: Zeolite-LTA powder, measurement time 1 minute/sample).

done in an extremely short time, and this is ideal for automated multi-point measurements using an automatic sample changer and XY stage. Furthermore, the steps from measurement to analysis can also be automated by combining with Rigaku's PDXL integrated powder X-ray analysis software. As an example of analysis, Fig. 6 shows the results of measuring Zeolite-LTA samples with different composition ratios due to solid solutions, over a time of approximately 1 minute per sample. The figure also shows the results of calculating the lattice constant of each sample through automatic analysis using PDXL. Thus the process from multi-point measurement to analysis can be automated, and workflow efficiency can be dramatically improved in areas such as screening during R&D and quality control.

2.4.3. High-speed reciprocal space mapping measurement for thin film samples

Previously, reciprocal space maps that were used for tasks such as evaluation of epitaxial films, the reciprocal space was measured in a mesh pattern using a scintillation counter (i.e., a 0D detector), and thus an extremely long measurement time was required. In reciprocal space mapping measurement using the D/teX Ultra 250, it is possible to measure the diffraction angle 2θ for a number of positions at once, and as a result, the measurement time can be greatly reduced. Fig. 7 shows reciprocal space maps measured with a scintillation counter and a D/teX Ultra 250. It is evident that measurement can be done in approximately 1/10 the time, even though the measurement range is roughly the same.

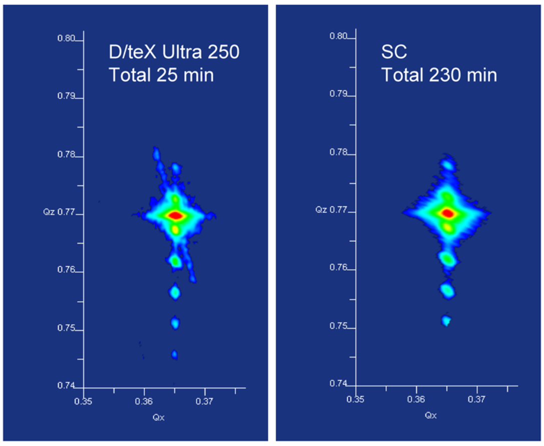


Fig. 7. High-speed reciprocal space mapping measurement of a thin-film sample (sample: InGaN MQW).