Microarea X-ray Diffractometer DicifferX™ Microarea Edition

Ultrahigh-Flux Laboratory X-Ray Diffraction System Optimized for Microarea XRD Measurements

Abstract

In recent years, as electronic devices have become smaller, electronic components have become increasingly miniaturized and highly integrated. In electronic components such as multilayer ceramic capacitors (MLCCs), the evaluation area for X-ray diffraction has been reduced to several tens of micrometers. To meet these needs, the microarea X-ray diffractometer DicifferX Microarea Edition was developed. This apparatus can form a point beam of approximately $\phi 20\,\mu$m (full width at half maximum of the direct beam) at the sample position by using a collimator with a minimum diameter of $\phi 10\,\mu$m. In addition, this diffractometer is equipped with a high-intensity X-ray source, a high-precision goniometer, and a two-dimensional detector. These features enable high-precision, high-speed and high-intensity measurements of microareas as small as several tens of micrometers in the laboratory. In this article, we introduce an example in which the cross-section of a zinc-plated nut was measured at $20\,\mu$m intervals and qualitatively analyzed.

1. Introduction

In recent years, the miniaturization of electronic components, such as multilayer ceramic capacitors (MLCCs), has increased the demand for material characterization in areas as small as several tens of micrometers. The automated multipurpose X-ray diffractometer SmartLab can only focus the beam to a point of approximately $\phi 100\,\mu$m (FWHM of the direct beam) at the sample position. Therefore, the microarea X-ray diffractometer DicifferX Microarea Edition has been newly developed to enable high-precision measurement of microareas as small as several tens of micrometers in the laboratory. This apparatus can form a point beam of approximately $\phi 20\,\mu$m (FWHM of the direct beam) at the sample position using a collimator with a pinhole diameter of $\phi 10\,\mu$m. Figure 1 shows the results of scanning the direct beam along the Y-axis (horizontal) and Z-axis (vertical) using a collimator with a diameter of $\phi 10\,\mu$m.

For microarea X-ray diffractometers, it is important that high intensity is obtained and the X-ray beam does not shift from the target position during the measurement, even with a small irradiation area on the sample surface. To meet these requirements, the DicifferX Microarea Edition is equipped with the following three key features: (1) ultrahigh-flux X-ray

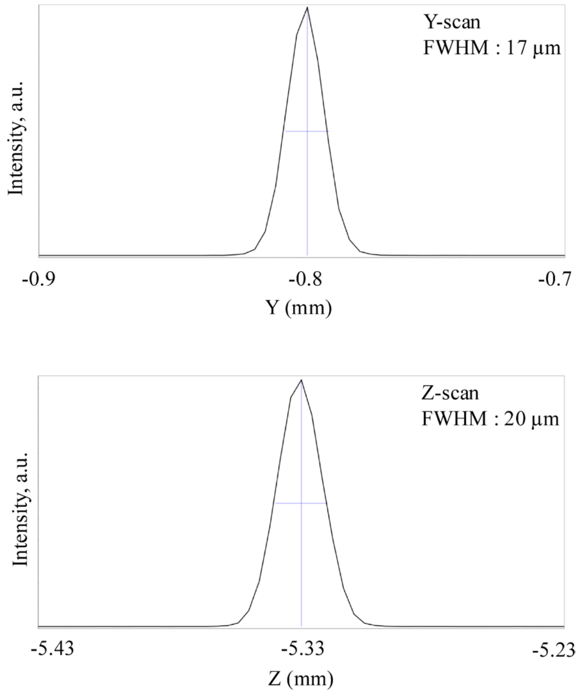

Fig. 1. Direct beam profile and FWHM with a $\phi 10\,\mu m$ collimator.

source, (2) high-precision goniometer for position control with no shift, and (3) 2D photon counting X-ray detector for efficient measurement. The following sections describe these features in detail.

2. Features

2.1. Ultrahigh-flux X-ray source

In general, X-ray diffractometers often use line-beam optics, and to measure microareas, the beam size is restricted to a point using slits or collimators. However, this causes a decrease in X-ray intensity, and sufficient diffraction intensity cannot be obtained. On the other hand, the DicifferX Microarea Edition is equipped with an ultrahigh-flux X-ray source (MicroMax-007HF) and adopts a point-beam optical system utilizing a beam with an effective focus size of $\phi 70\,\mu m$. The generated X-rays are efficiently focused and monochromated by two reflections using a confocal mirror (VariMax). This enables the formation of a point beam of approximately $\phi 200\,\mu m$ (FWHM of the direct beam) at the sample position without using a collimator. The path between the focusing mirror and the collimator is kept under vacuum to minimize intensity attenuation caused by air. In addition, it is possible to switch between high-intensity and high-resolution settings by adjusting the aperture width of the mirror slit. In high-resolution setting, the mirror slit aperture is narrowed to extract part of the focused X-rays, which reduces convergence and divergence angles and results in higher resolution. In high-intensity setting, the mirror slit is fully opened, so resolution is lower, but higher X-ray intensity can be obtained.

Figure 2 shows that NIST Si (Standard Reference Material 640f) was measured using SmartLab and DicifferX Microarea Edition, each equipped with a $\phi 100\,\mu m$ collimator. If the intensity measured with the SmartLab is normalized to 1, the intensity measured with the DicifferX Microarea Edition is approximately 100 to 330 times higher. This means that a measurement that previously took 10 minutes per point can be shortened to approximately 2 to 6 seconds. In high-resolution setting, the FWHM obtained with SmartLab and DicifferX Microarea Edition is approximately the same. Therefore, with microarea optics, DicifferX

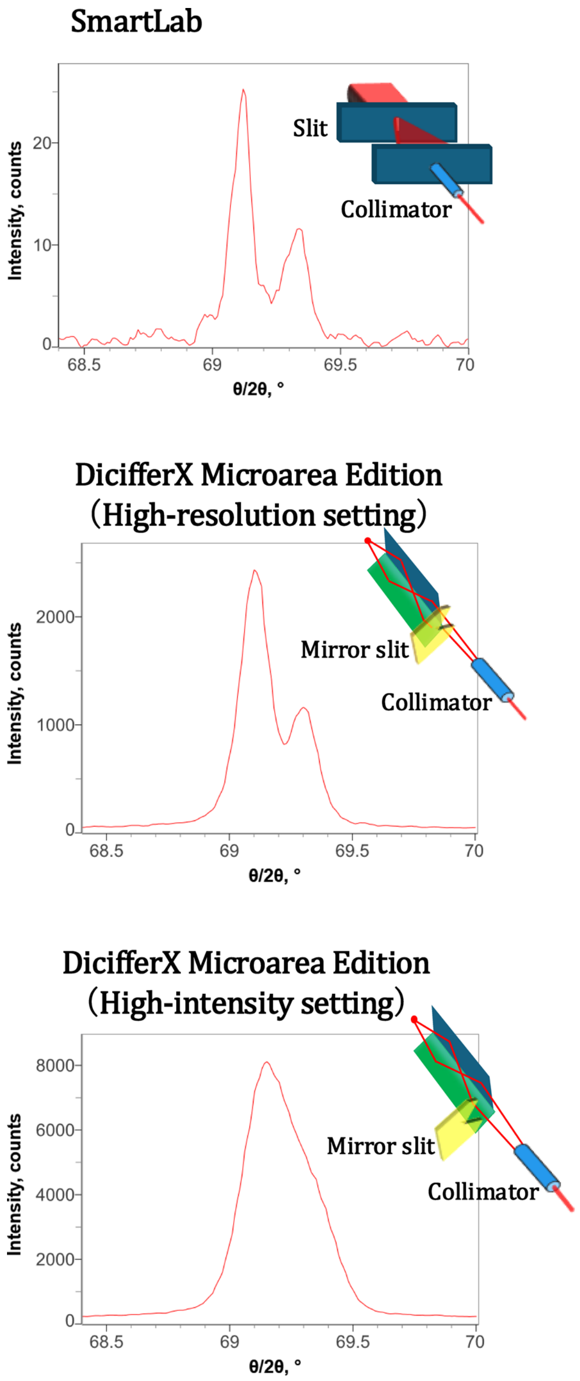

Fig. 2. Results of NIST Si measurements using SmartLab and DicifferX Microarea Edition with a $\phi 100\,\mu m$ collimator.

Microarea Edition can acquire data with the same resolution as the SmartLab in a much shorter time, resulting in significantly improved measurement efficiency.

2.2. High-precision goniometer and offset function

For microarea X-ray diffractometers, it is important to maintain precise goniometer positioning so that the X-ray beam continuously irradiates the target area during the measurement. The DicifferX Microarea Edition is equipped with a high-precision goniometer and a sample stage with an XY offset function. This function corrects the XY position for each χ-axis (tilt axis) movement to compensate for any shift of the measurement point away from the rotation center when the χ-axis is moved (Fig. 3). With this high-precision goniometer and the XY offset function, a positioning accuracy of within $\pm 10\,\mu$m can be achieved. Therefore, measurements can be performed without any deviation from the intended position even when the axes move.

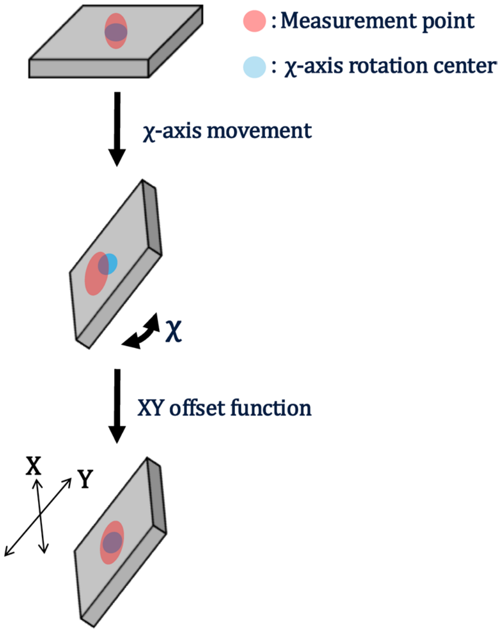

Fig. 3. Schematic diagram of the XY offset function.

2.3. 2D photon counting X-ray detector

The DicifferX Microarea Edition is equipped with the 2D photon counting X-ray detector, HyPix-3000 (active area: 77.5 mm × 38.5 mm)(1).

The HyPix-3000 supports 0D, 1D, and 2D measurement modes. Measurements in 2D mode can be performed in two ways: 2D scanning using TDI (Time Delay and Integration) mode, and 2D exposure with the detector fixed. The 2D scanning enables measurements over a wide angle range, making it applicable to qualitative and quantitative analysis of minute discolored areas or foreign inclusions. The 2D exposure enables simultaneous measurement over a fixed 2θ range. The focus is on one diffraction peak, and tens or even hundreds of images are obtained. This is suitable for residual stress measurement, pole figure analysis, and mapping analysis in microareas.

Figure 4 shows that NIST LaB_6 (SRM 660c) was measured using the DicifferX Microarea Edition equipped with a $\phi 100\,\mu$m collimator in the 2D scan.

In 2D mode, the intensity along the circumferential direction of the Debye ring (β direction) is integrated and converted into a 1D profile. This enables the

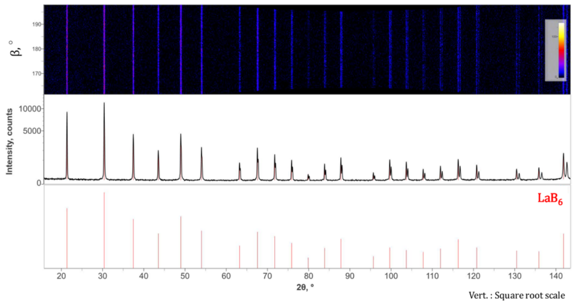

Fig. 4. Result of the 2D scan of NIST LaB_6 using DicifferX Microarea Edition.

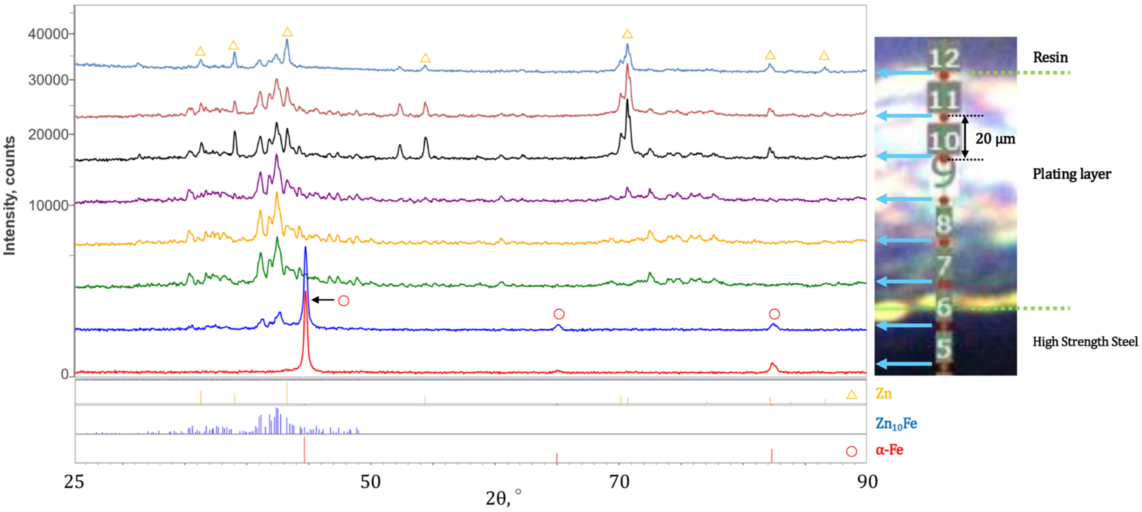

Fig. 5. Qualitative analysis result of a zinc-coated nut using DicifferX Microarea Edition.

acquisition of higher-intensity data in a shorter time compared to 0D and 1D modes. When performing measurements in microareas, high-speed and high-intensity measurements in 2D mode, which fully utilize the large detection area of the HyPix-3000, are highly effective.

3. Measurement Example

The example of a mapping measurement on the cross section of a zinc-coated nut using the DicifferX Microarea Edition is presented. Using a $\phi 10\,\mu$m collimator, measurements were performed at $20\,\mu$m intervals across the plating layer and its surrounding area. Figure 5 shows that the acquired 2D images were converted into 1D profiles and qualitatively analyzed

In the area approximately $40\,\mu$m away from the resin–plating interface, Zn is mainly present. In areas further away, Zn–Fe alloy ($Zn_{10}Fe$) becomes predominant. The DicifferX Microarea Edition can measure microareas as small as several tens of micrometers with high precision. These features enable detailed characterization of the distribution of crystalline phases within the plating layer.

4. Specifications*

Max. output (Cu X-ray source)	1.2 kW
Supported wavelength	Mo, Cu, Co, Cr,
Collimator	Min. $\phi 10\,\mu$m Max. $\phi 500\,\mu$m
Camera length	65～150 mm
X, Y axis (XY-52 mm attachment)	Stroke: ±26 mm
Z axis	Stroke: −9～+1 mm
Max. sample thickness that can be set	50 mm
Max. sample weight that can be set	1 kg (300 g when intersection accuracy of $10\,\mu$m is required)

*Specifications as of July 2025.

References

(1) *Rigaku Journal*, **30**, (2014), No. 2, 38–40.