High-sensitivity EDX analysis using QUANTAX FlatQUAD (EDX) with SU8200 Series Instruments

Toshiya Watanabe (Hitachi High Technologies, Applications Development Dept., Tokyo Solutions Group)

The SU8200 Series of ultra-high-resolution scanning electron microscopes, which are equipped with new cold field emission guns, offer great potential for observation and analysis at low accelerating voltages. The reduced speed of the optical system allows measurements of polarized surfaces with irradiating voltages starting at 10 V, while the optional filtering feature further enables high-contrast structural observations and capture of STEM images. For EDX analysis, these instruments may be combined with the QUANTAX FlatQUAD from Bruker AXS to enable elemental analysis with greater sensitivity.

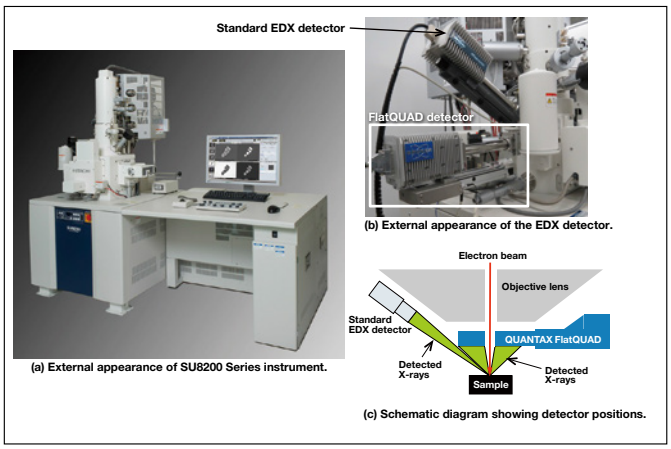

Fig. 1 External appearance and schematic diagram showing EDX positions.

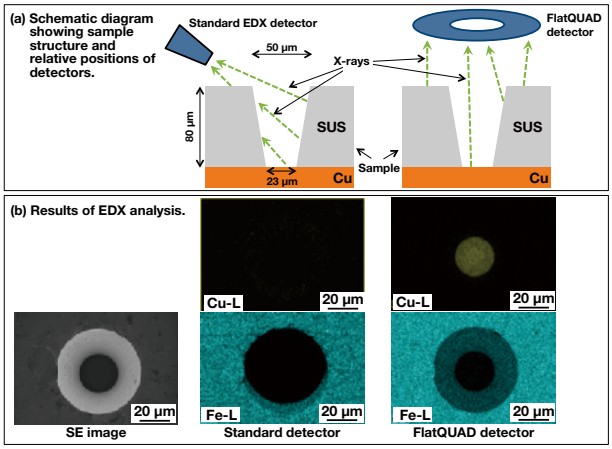

Fig. 2 Comparison of analytical results obtained with a standard detector and with the FlatQUAD detector.
•Accelerating voltage: 5 kV
•Magnification: 1,300×
•Analysis time: 5 minutes

Figure 1 shows the external appearance of the SU8200 and FlatQUAD instruments and indicates the relative positions of the detectors. As shown in Figure 1(b), the FlatQUAD detector is designed to be inserted horizontally. The standard EDX detector is inserted diagonally, as shown on the left of Figure 1(c), and X-rays are detected with the regions to be analyzed read off in the diagonal direction. In contrast, the FlatQUAD is designed with the detector inserted between the objective lens and the sample. The detector is positioned immediately above the sample—as shown on the right of Figure 1(c)—allowing the detection of X-rays emitted over a wide range of angles, including directly upward. FlatQUAD allows analysis over a solid angle of approximately 1.1 sr, enabling more detailed analyses to be performed in a short period of time. This facilitates analysis of fragile samples and allows a reduction in the total time required for analysis. It is also possible to achieve improved spatial resolution at low accelerating voltages, and the standard EDX detector may also be installed simultaneously. Moreover, by exploiting the possibility of placing detector elements directly above the sample, the system offers powerful capabilities for analyzing the lowest regions of deep trenches.

Figure 2 shows example images of a sample of stainless steel on a Cu substrate after etching to yield tapered sidewalls, obtained using the standard detector and the FlatQUAD detector. These analyses were conducted at an accelerating voltage of 5 kV and a magnification of 1,300× with an analysis time of 5 minutes. The standard detector fails to detect both the Cu at the bottom of the trench and the Fe in the tapered region. The reason for this is explained by the schematic diagram in the inset of the figure: the side walls form an obstruction to the X-rays emitted from the bottom of the trench and the tapered regions, preventing their detection by the standard detector. In contrast, the FlatQUAD is able to detect Fe from the stainless steel all the way down to the tapered region, and also clearly reveals the Cu at the bottom of the trench. This is because the FlatQUAD detector is positioned directly above the sample, ensuring that X-rays emitted from the tapered regions and the bottom of the trench are properly detected. Thus, we see that this system is capable of analyzing both materials with a large surface roughness and trench bottoms that would be difficult to analyze using conventional approaches.

The combination of the SU8200 Series with the QUANTAX FlatQUAD effectively exploits the performance of a cold field emission gun to enable high-resolution observations and EDX analysis with high sensitivity over a large solid angle. This allows good throughput for elemental mapping and for qualitative analysis of features that do not depend on sample shape.