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Crystalline analysis by W-SEM using a newly developed EBSD detector

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Background incl. aims

Understanding crystalline properties of material is essential for controlling its physical characteristics. EBSD method in SEM (SEM-EBSD) is a well-established technique for investigating the phase orientation and identification of single or polycrystalline materials. It is commonly used to determine crystalline properties in micro and nanotextures [1]. The development of SEM-EBSD significantly contributes to the analysis of microstructure, crystallography and physical properties in various fields, including materials science and mineralogy, such as metals, ceramics, and minerals. Thus, the SEM-EBSD method is widely recognized as a requisite tool in these fields now.

To obtain high-quality EBSD measurement results, large probe currents ranging from several 10 to 100 nA are generally required. Therefore, the SEM equipped with a Schottky-type field emission gun (FE-SEM) has been basically employed for EBSD measurements due to its ability to maintain high resolution even when large probe currents are applied. On the other hand, the crystal grains to be measured by EBSD are usually a few to several 10 of micrometers in size, which falls within the measurable range of the SEM with Tungsten thermal filament (W-SEM). However, there are few cases where W-SEM is installed together with the EBSD detector in laboratory, because the spatial resolution of Tungsten filament gun when using a large probe current is significantly inferior to that of field-emission guns [2].

Recently, CMOS has increasingly replaced CCD as the image sensor in EBSD detectors. This shift has made it possible to obtain fast and clear EBSD patterns with less probe current, resulting in more practical EBSD measurement by W-SEM. Here, we present a new EBSD detector from Bruker (e-Flash XS) in combination with JEOL's entry-level W-SEM. Although the body of e-Flash XS is significantly smaller than the conventional EBSD detectors, its CMOS image sensor provides high-definition EBSD patterns even at a small beam current, enabling fast analysis. The EDS detector is also included in this system, called ED-XS, which allows an elemental analysis during the EBSD measurement simultaneously. This report presents an overview of the e-Flash XS with ED-XS system and one of its applications.

Methods

The ED-XS system including an EDS (XFlash 630; Bruker Nano GmbH) and an EBSD detector (e-Flash XS; Bruker Nano GmbH) is installed on the entry model W-SEM, JSM-IT200 (JEOL, Ltd.). Details are described in the following “Results” section. Cross section of zirconia ceramic made with Cross Section Polisher (IP-19520CCP; JEOL, Ltd.) was used as a specimen. The crystal structure of cubic zirconia (ZrO2, Fm-3m, No. 225) was used for EBSD pattern analysis. The acquisition parameters were an acceleration voltage of 20 kV, a probe current of 15 nA, a working distance (WD) of 7 mm, and a chamber vacuum of 10 Pa.

Results

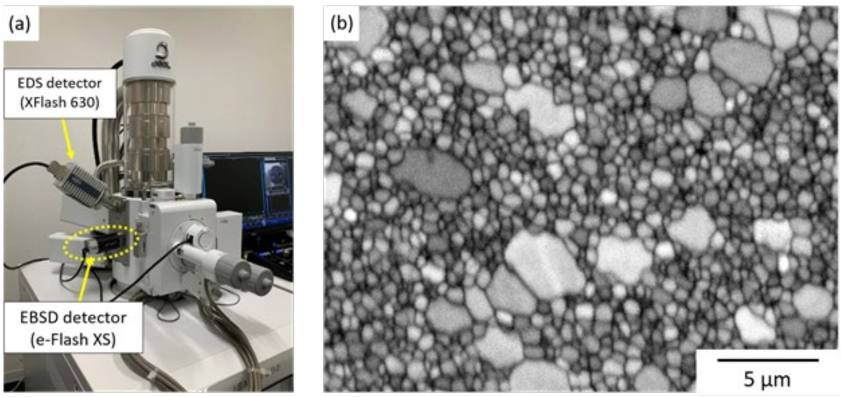
Graphic (a) displays an external view of the complete ED-XS system with JSM-IT200. Simultaneous acquisition of EDS and EBSD is possible because the detectors are located in the same direction. The phosphor screen inside the sample chamber can be manually attached and detached. A specimen to be measured is mounted on a pin-type stub and placed in a designated holder with the specimen surface tilted at 70 degrees. This holder allows high-resolution EBSD measurements with a short WD of less than 15 mm (minimum 6 mm) without tilting the stage.

The pattern quality map of the zirconia ceramic obtained by e-Flash XS is shown in graphic (b) at a magnification of 5,000. Although this ceramic is the non-conductive material, EBSD measurements can still be performed without a conductive coating using the low-vacuum feature. The pattern quality map reflects the shape of the grains and their boundaries based on the clarity value of the Kikuchi pattern image. Then, the crystal grain size of the zirconia ceramic measured in this study was found to be approximately 2-3 µm for the largest ones and several 100 nms for the smallest ones. Additionally, inverse pole figure (IPF) map indicates that the orientation of the individual grains are random, while kernel average misorientation (KAM) map demonstrates that there is little local plastic deformation in the ceramic. Moreover, simultaneous EDS elemental map revealed that Zr and O were mainly present thoughout the measurement area, with small amounts of Y and Hf also detected. No segregation of other elements was observed, indicating the absence of foreign materials.

Conclusion

In this report, we present a new EBSD detector e-Flash XS and ED-XS integrated system designed for the compact model of W-SEM. As described, this small detector can measure the properties of crystal grains as small as several 100 nm, even under low-vacuum conditions using W-SEM. Electron channeling contrast images are generally used for measuring crystal grains. However, it is important to note that the contrast of these images can be affected by changes in the acceleration voltage or angle of incidence of the electron beam. On the other hand, crystal grain measurement via EBSD can be performed independently for acceleration voltage and angle of incidence. In addition, it is possible to analyze distortion and deformation resulting from differences in crystallographic orientation. This system is optimal for all SEM users, especially W-SEM users, to enhance their crystallographic analysis.

Graphic:



Keywords:

EBSD
W-SEM
Crystal analysis

Reference:

[1] J. I. Goldstein, D. E. Newbury, J. R. Michael, N. W. M. Ritchie, J. H. J. Scott, D. C. Joy, New York: Springer Nature, (2018).
[2] P. McSwiggen, IOP Conf. Ser.: Mater. Sci. Eng., 55 (2014) 012009.