Orthogonally arranged FIB-SEM for serial sectioning observation; concept and applications

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1. Introduction

To understand the microstructure of materials accurately, 3D observation is necessary because it is 3D in nature. Serial-sectioning using a FIB-SEM is now widely applied for such purpose. We introduced the first orthogonally arranged FIB-SEM in 2011[1], which is specially designed to observe the serial-sectioning images precisely; with higher contrast and higher spatial resolution. From the installation of this instrument, we have opened it to all researchers and have developed a methodology with them. In my presentation, I focus on the features and some applications of the orthogonal FIB-SEM, then discuss the role of a FIB-SEM for 3D microstructure analysis.

2. Features of the Orthogonal FIB-SEM

The basic concept of the orthogonal FIB-SEM is shown in Fig. 1. A FIB and a SEM column are set perpendicularly while in the standard arrangement, both columns are tilted around 60deg. each other. The orthogonal arrangement realizes high-contrast 3D reconstruction image of several tens of micron cube with the spatial resolution of below 10nm. Figure. 2 is an outlook of the instrument. An EDS and EBSD detectors are equipped to obtain various information simultaneously with serial-sectioning. Especially, for the EBSD, this instrument is a static setup; a sample stage does not move during 3D-EBSD measurement, that is realized by placing the EBSD detector perpendicular to both the SEM and FIB.

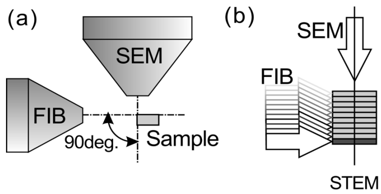

Figure 1. (a) Concept of orthogonal FIB-SEM. (b)Configuration of serial-sectioning.

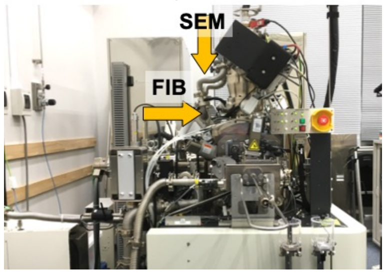

Figure 2. Outlook of the orthogonal FIB-SEM.

3. An example of applications

Figure. 3 is an example of 3D reconstructed image. The sample is heat-resistant steel, and the purpose of this observation is performed to evaluate the distribution of two types of precipitates, chromium-based carbide ($M_{23}C_6$) and vanadium-based nitride (MX), especially on a phase boundary. By conventional SEM and TEM observations, we cannot observe it directly. We obtained 240 images with a slice pitch of 10nm. Observation conditions are; acceleration voltage of 1kV using an on-axis annular SE detector. With its high contrast, we can distinguish these two types only by contrast. From this observation, we can analyze the distribution of precipitates on a phase boundary, which cannot observe by conventional 2D methods.

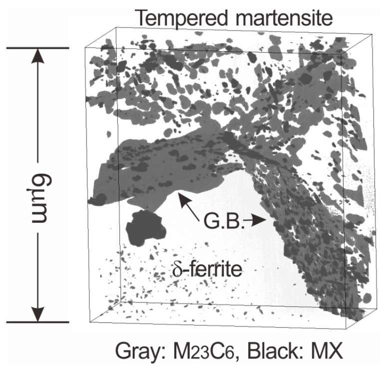

Figure 3. An example of 3D reconstruction image showing precipitates distribution in a heat resistant steel.

4. Requests from users and future perspectives

For around 8 years, we have collaborated with many researchers who covers broad research fields to develop a methodology of a 3D serial-sectioning by this FIB-SEM. We have received many requests from users, they can be classified into following three; i) Large volume observation, ii) Obtaining other information (concerning to detector), iii) Analysis methods, such as image processing. I will introduce some of them and discuss future perspectives of FIB-SEM serial sectioning method.

[1] T.Hara, K.Tsuchiya, K.Tsuzaki, X.Man, T.Asahata, A.Uemoto, J. Alloys and Compounds, 577, (2013), 717-721