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Preparation of TEM Samples from Specific Orientations Using FIB-SEM

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Introduction

High resolution observation of crystal samples using transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) is performed from a specific crystal orientation of a sample. In order to get the specific crystal orientation that we want, we need to adjust a tilting angle of the sample with a goniometer stage in TEM. However, if the tilting angle is larger, the sample effectively becomes thicker to reduce image resolution. And in that case, the shadow effect of the sample itself and/or the sample holder becomes larger to reduce the effective X-ray intensity of the energy dispersive X-ray spectrometer (EDS) detector. Powder and polly-crystal samples such as ceramics and minerals typically include random oriented grains. Then, it is difficult to obtain a TEM thin film in a specific orientation of the sample.

If we can know the crystal orientation of the sample surface before preparing a TEM thin film, it helps us to obtain a TEM thin film with a desired specific crystal orientation. Recently we developed a new method for preparing TEM thin films with specific crystal orientation from the interest area using a focused ion beam (FIB) method combined with an electron backscatter diffraction (EBSD) method. In this paper, we will report the procedure of the method and results.

Sample and Methods

The sample that we examined in this study is Allende meteorite. This meteorite mainly contains coarse-grained (up to 1 mm in size) objects (chondrules and inclusions) embedded in a fine-grained matrix, composed mainly of Fe-rich olivine grains (< 50μm in size). The previous study suggests that the olivine grains contain very thin (< 1nm in thickness) lamellas of Fe-rich oxide perpendicular to the [100] of host olivine [1]. In order to obtain TEM thin films of olivine [001], [010], and [100] in the Allende meteorite, we performed the following method. First, crystal orientation distribution of matrix olivine grains from the surface of a thick Allende section was examined with an EBSD detector (EDAX EBSD system) attached to FIB-SEM (JEOL, JIB-4700F). Figure 1 is an example showing a crystal orientation map. In Fig.1, blue-colored grains correspond to grains having a [001] crystal orientation that is roughly parallel (< 20 degrees) to the Z direction of the section (perpendicular to the paper). From these grains, we extracted and prepared a TEM thin film of olivine in the [100] orientation using a conventional FIB milling method (Fig. 2). In the same manner, TEM thin films of olivine [010] and [001] orientations were prepared from the crystal grains oriented in the [100] and [010] directions, respectively, which are roughly parallel to the Z direction of the section. After that, we observed these TEM thin films by using our aberration corrected microscope (JEOL, JEM-ARM300F) at 300 kV.

Results and Discussion

We firstly adjusted the crystal orientation to each zone axis of olivine with a goniometer stage in TEM using selected area diffraction patterns (Fig.3), and then we checked the amount of the tilting angle of the goniometer stage for each TEM thin film. Even after adjusting the crystal orientation, all the thin films are thin enough to be able to take atomic resolution STEM images (Fig.3). Table 1 shows the summary of the tilting angle of the goniometer stage for all TEM thin films after the adjustment. The amount of the tilting angle was within 5 degrees or less for all the thin films (Table 1). It should be noted that those angles include the sum of the angular errors resulting from all steps of the sample preparation and mounting process for TEM thin films such as selection of sample position/orientation, block preparation, loading of thin films to TEM sample holder. Despite even such many factors, we need tilting adjustment of only less than 5 degrees with a goniometer. These results suggest that our new method is very effective for obtaining TEM thin films with specific crystal orientation from fine-grained aggregates such as the Allende matrix. We believe that this method can apply to many samples such as ceramics and metals.

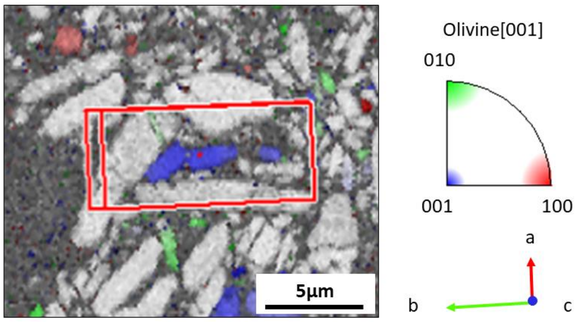

Figure 1: Crystal orientation map

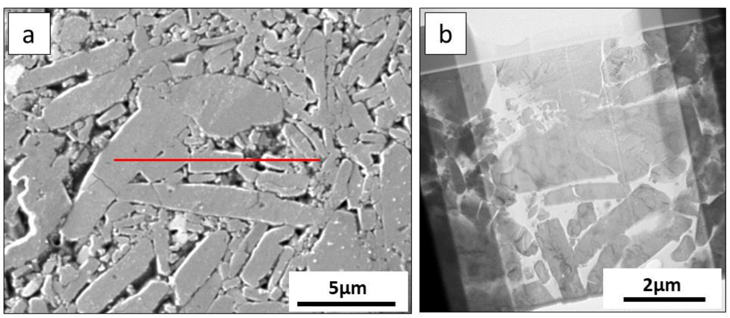

Figure 2: (a) SEM image of the area near the location of TEM sample preparation (red line represents the location of TEM sample preparation); (b) TEM image of the resulting TEM sample

Figure 1. Fig. 1 & 2

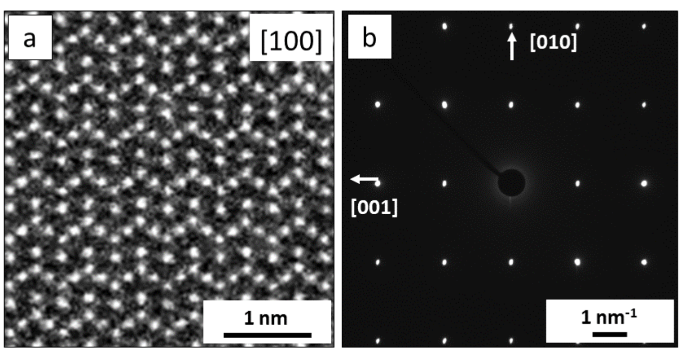

Figure 3: (a) HAADF STEM image; (b) Diffraction pattern

Table 1: Tilt angles of prepared samples in TEM imaging

	[100]	[010]	[001]
Tilt X	-1.7	-3.5	+3.4
Tilt Y	+2.6	+2.7	-0.5

Figure 2. Fig 3 & Table 1

References

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