PREPARATION OF TEM SPECIMENS FOR OLIVINE WITH SPECIFIC CRYSTAL ORIENTATIONS FROM THE MATRIX IN ALLENDE CV3 CHONDRITE USING FIB-SEM AND EBSD.

I. Ohnishi[1], M. Kadoi[1], and M. Shibata[1],
[1]JEOL Ltd., 3-1-2 Musashino, Akishima, Tokyo 196-8558, JAPAN (ionishi@jeol.co.jp)

Introduction: Iron-rich olivine is a major mineral in the matrices of CV3 carbonaceous chondrites. Although it has been widely studied so far, the origin of the matrix olivine has been still poorly understood. Recently, the matrix olivine in the Allende chondrite was studied using an aberration corrected scanning transmission electron microscope (STEM) [1], which reveals that very thin planar precipitates (~1 nm in thickness) occur in the olivine and their long axis is parallel to (100) plane of its host olivine. Based on these results and the previous studies, the precipitates were inferred to be Fe, Cr-rich oxides and formed by the oxidation process of olivine [1]. However, as the number of olivine grains previously examined is limited, it is still controversial whether the precipitates commonly occur in the matrix olivine. In order to observe the precipitates parallel to the (100) plane, we need to observe them in the specific orientations (e.g., [010] or [001]). Thus, we prepared the TEM specimens from the randomly oriented olivine grains in the Allende matrix by a method combined with FIB (Focused Ion Beam) and EBSD (Electron Back Scattered Diffraction). Here, we report details of the procedure and its results.

Methods: TEM specimens were made using multi-beam system (JEOL JIB-4700F) equipped with an EDAX DigiView EBSD detector. Prior to the FIB milling, the crystal orientation maps of matrix olivine were obtained from the surface of Allende thick section using EBSD method. And using the maps, we selected the grains oriented to [001], [100] and [010] having mis-orientation angles less than 20 degrees. Those three orientations are orthogonal to each other since the olivine belongs to the orthorhombic crystal system. Therefore, if we perform milling for the samples parallel to surface with FIB, they should be TEM specimens of olivine with [001], [100] and [010] orientations. According to this idea, TEM specimens of [100] orientation were prepared within mis-orientation of 20 degrees. In the same way, TEM specimens for observing in the [010] and [001] directions were prepared. The preparation of the TEM specimens by FIB was done with the bulk pickup method, which is divided to three processes. In the first process, small bulk block having the specific orientation is hewn with an FIB. And, next, the block is laid down so that the plane of specific orientation is vertical. Finally, the thin TEM specimen is sliced from the block by an FIB. The TEM specimens prepared by the thus-mentioned way were examined using an aberration corrected 300 kV STEM (JEOL JEM-ARM300F) equipped with a cold field emission electron gun.

Results & Discussion: Our atomic resolution STEM observations revealed that the planar precipitates (<1 nm in thickness) parallel to the (100) plane occur along the [010] and [001] directions and that no distinct defects were found in the [100] direction. These results are consistent with the previous study [1]. It indicates that the planer precipitates might commonly occur in the matrix olivine of Allende meteorite.

The mis-orientation of these specimens were found to be less than 5 degrees judging from amounts of specimen stage tilt angle, after crystal axis adjustment for selected area diffraction patterns and STEM imaging. The mis-orientation angles are supposed to be resulted from many reasons, including the sample tilt when we load to the grid on TEM specimen holder, and so on. Finally, we successfully achieved the TEM specimens having small mis-orientation angles (<5 degrees). This suggests that such sample preparation method is very effective to make TEM specimens with the specific orientations from bulk sample of variously orientated crystalline grains. It greatly helps to reduce the TEM observation time. Moreover, it can apply the presented method not only to the matrix olivine, but also to the poly-crystal object composed of randomly oriented grains such as chondrules and CAIs in chondrite.

References: [1] Ohnishi I. et al. (2018) *Meteoritics & Planetary Science* **53**:6105. [2] Ohnishi I. et al. (2016) *Micros. Microanal.* **22**: 313-314.