

# PRELIMINARY RESULTS OF SAMPLE PREPARATION OF ULTRA-THIN FILM BY USING FOCUSED ION BEAM TECHNIQUE FOR EXTRATERRESTRIAL MATERIALS IN JAXA

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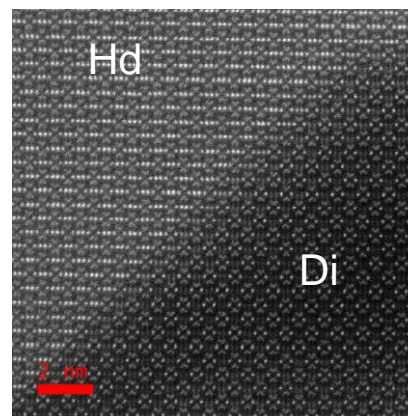
**Introduction:** Sample return mission is an useful project for planetary material sciences, because it gives us opportunities for direct handling of extraterrestrial materials known of their astronomical/geological occurrence such as in comets and asteroids. However, the returned samples are often limited in mass and volume. For instance, the nominal mass returned are expected to be 0.1 g for JAXA Hayabusa2 and 1 g for NASA OSIRIS-Rex. Transmission electron microscopy (TEM) does not need large volume of a sample and it is very effective to analyze small sample at micro- to nano-meter level. However, conventional ion milling methods using Ar<sup>+</sup> ion could lose large amount of sample to make ultra-thin film for TEM. Then, ultramicrotomy was widely used in sample preparation of ultra-thin film for micrometeorites [1]. Recently, a focused ion beam (FIB) method using Ga<sup>+</sup> ion has been utilized for sample preparation of ultra-thin film from extraterrestrial materials such as meteorites and cosmic dusts [e.g., 2]. The method can precisely make ultra-thin film from any interested area in a sample with spatial resolution of micrometer to sub-micrometer. In 2018, we installed a FIB instrument at Extraterrestrial Sample Curation Center (ESCuC) of JAXA and started to construct the procedure of ultra-thin film sample preparation of extraterrestrial materials for TEM. We present here the detail of procedure and preliminary results of evaluation of ultra-thin film made with the FIB.

**Material and Methods:** In this study, ultra-thin films were made from a Ca, Al-rich inclusion (CAI) in thin section of the Allende CV3 chondrite using triple beam FIB instrument (HITACHI NX2000) at ESCuC, equipped with a variety of detectors such as secondary and backscattered electron detectors and silicon drift X-ray detector (Oxford X-Max 80). The petrological and mineralogical studies were conducted using the function of scanning electron microscope in the FIB before ion thinning. The CAI studied here was classified to fluffy type A inclusion and its primary minerals such as anorthite and diopside have been significantly altered by the secondary minerals including nepheline, hedenbergite and so on. Ultra-thin films were extracted from the altered area in the CAI. After rough thinning with Ga<sup>+</sup> ion at 30 kV and 5kV, fine thinning with Ar<sup>+</sup> ion at 1 kV was performed to reduce amorphous layer at the surface of films. The films were studied using conventional 120 kV TEM (JEOL JEM-1400) at ESCuC and an aberration corrected 300 kV STEM (JEOL JEM-ARM300F) at JEOL R&D factory.

**Results & Discussion:** Our conventional TEM observations indicate that all the ultra-thin films made in this study are very thin enough for electron beam to transmit even at 120 kV of accelerating voltage. Although a variety of minerals, which have different etc

hing rates of Ga<sup>+</sup> ion each other, coexisted in the films, the surface of films seems to be homogenously flat. In addition, re-deposition of sample seems to be negligible. The results suggest that the fine thinning with Ar<sup>+</sup> ion after rough thinning with Ga<sup>+</sup> ion is very effective to remove damage layers and contamination from the surface of films. Because all the ultra-thin films made in this study are enough thin, they are also use to observe atomic resolution images as shown in Figure 1.

**References:** [1] Bradley J.P. and Brownlee D.E. (1986) *Science* **231**:1542–1544. [2] Lee M.R. et al. (2003) *Mineralogical Magazine* **67**:581-592.



**Figure 1:** Atomic resolution STEM high angle annular dark field image showing the boundary between diopside (Di) and hedenbergite (Hd) in an ultra-thin section from altered CAI. The image was obtained with JEM-ARM300F at 300 kV.