

## Sample Results Summary Sheet

Please return this form to the Curator for each allocated Sample

**Sample ID:** RA-QD02-0032

**PI:** Takaaki Noguchi

**Type and date of analysis performed:**

Potted butt: Scanning electron microscopy (SEM) at Ibaraki University on Feb. 7, 2011.

Ultrathin sections: Scanning transmission electron microscopy (STEM) at Hitachi

High-technologies Co., on Feb. 9, 2011.

FIB samples: TEM at Ibaraki Univ. on Feb. 21 and 22, 2011.

TEM tomography: STEM at Hitachi High-technologies Co., on Jul. 25, 2011

**Elements or phases identified:** (Mg, Si, olivine, pyroxene, aromatic carbon, etc.)

Olivine, high-Ca pyroxene, plagioclase, and troilite.

Nano particles were observed on the surface of olivine in ultrathin sections and a FIB section.

Nano particles on the surface contain sulfur.

**Contaminant phases identified:** (Al, SUS, carbon particles, etc.)

Aluminum, Tin oxide nanoparticles were observed at the boundary of the cross section of the particle and the embedding epoxy. Please see the summary seat of this particle.

**Sample handling:** (e.g. exposed in atmosphere, embedded in resin, polished, sliced by FIB or UMT)

Embedding in epoxy resin in atmosphere at Ibaraki University on Feb.4, 2011 (Because this sample has been already enclosed in a thin layer of epoxy resin at the curation facility during preparation for the mainstream analyses, this sample was not exposed directory to the earth's atmosphere.)

Ultramicrotomy at Ibaraki University in atmosphere on Feb. 6, 2011. The ultrathin sections on TEM grids were preserved in a vacuum desiccator just after ultramicrotomy.

Carbon coating of the potted butt at Ibaraki University on Feb. 6, 2011. The potted butt was preserved in a vacuum desiccator just after carbon coating.

Focused ion beam (FIB) of potted butt at Hitachi High-technologies Co., on Feb. 10, 2011. 2 samples were prepared for TEM observation. Another FIB sample (a needle-like sample) was prepared for STEM tomography on Jul. 25, 2011. They were preserved in a vacuum desiccator just after FIB. During transportation from Hitachi High-technologies Co. to Ibaraki University, it

was kept in a reduced pressure. At Ibaraki University, the sections were preserved in a vacuum desiccator.

However, when one of the FIB sample for TEM and the sample for STEM-tomography were observed by STEM on Nov. 17, we identified morphological changes. There are many sulfide particles up to  $\sim 1 \mu\text{m}$  across on the surface of the sample, where coincides the boundary between the sample and the embedding epoxy resin. They seem to have been formed from the amorphous S-bearing material that were originated from the "zone I" of Noguchi et al. (2011). To observe the boundary, we again performed FIB to remove the sulfide particles on Nov. 18. The sample for STEM-tomography was swelled. Perhaps the sample itself was reacted with the residual atmosphere ( $\sim 1/100$  atmospheric pressure) in the vacuum desiccator in which it was kept. Although we reshaped the sample by FIB, nanoparticles were not identified in the sample on Nov. 18. Probably, due to the high surface/volume ratio, the nanoparticles had reacted with the residual atmosphere. Unfortunately, no image was saved during FIB processing on Nov. 18.

**State of sample pre-analysis:** (e.g. N<sub>2</sub> hold, atmosphere, resin embedded, polished section, UTS) (please describe treatments and/or modifications for the sample you have done before your analysis)

STEM observation: ultramicrotomed sections embedded in epoxy resin and FIB sections that were lifted out and mounted on a TEM grid for FIB lift-out sections.

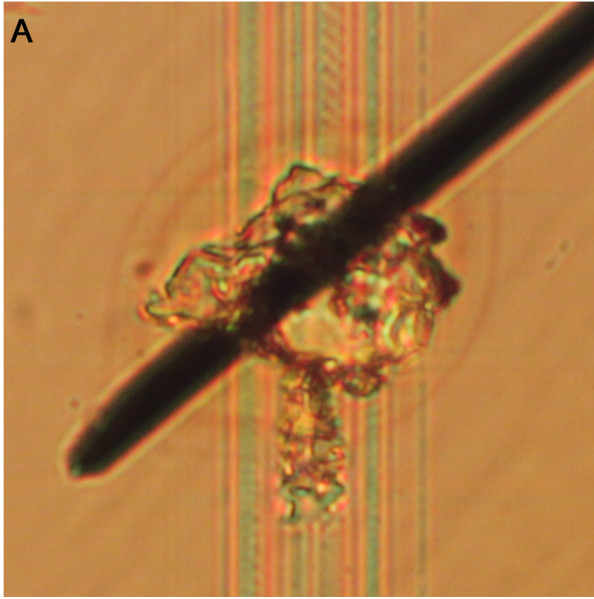
SEM observation: Carbon coated potted butt

**State of sample post-analysis:**

All the ultrathin sections and FIB sections were preserved in a vacuum desiccator.

**Analysis data Notes:** (summary of the attached analysis data and/or images)

Please see the summary seat of this particle.

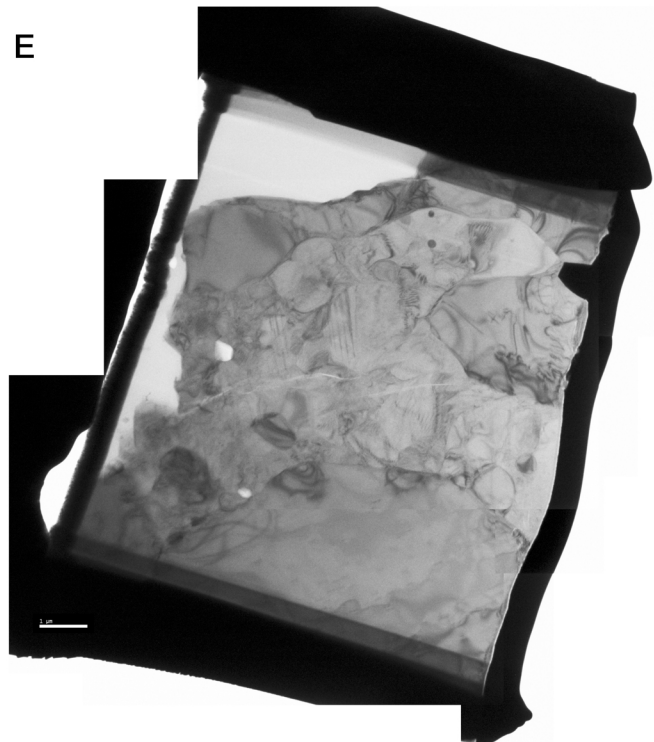
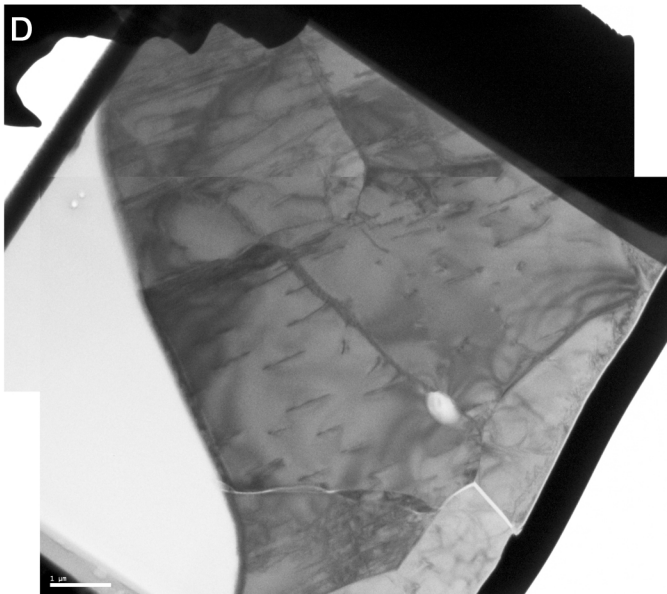
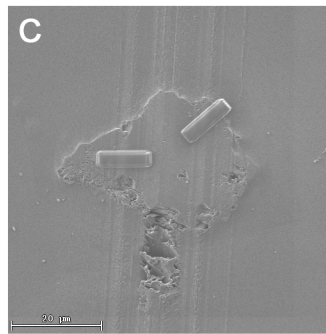
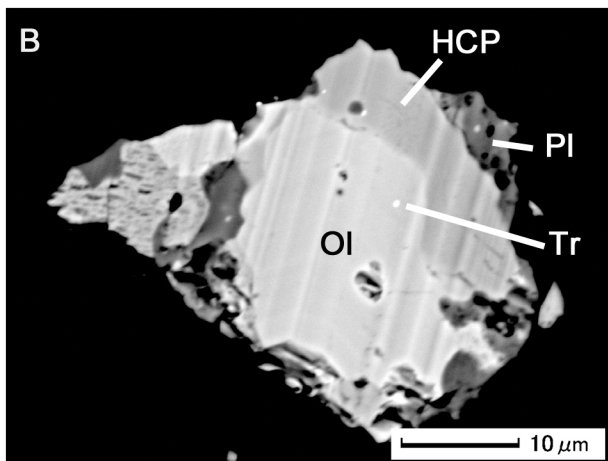


## Sample handling history

1. Embedding in epoxy resin at Ibaraki Univ. on Feb.4, 2011
2. Ultramicrotomy at Ibaraki Univ. on Feb. 6, 2011
3. Potted butt: Carbon coating at Ibaraki Univ. on Feb. 6, 2011
4. Potted butt: SEM at Ibaraki Univ. on Feb. 7, 2011
5. Ultrathin sections: STEM at Hitachi High-tech., on Feb. 9, 2011
6. Potted butt: FIB at Hitachi High-tech., on Feb. 10, 2011 (2 samples were prepared)
7. FIB samples: TEM at Ibaraki Univ. on Feb. 21 and 22, 2011
8. Sample for TEM-tomography: FIB at Hitachi High-tech., on Jul. 25, 2011

Processes No. 1 and 2 were performed in the earth's atmosphere.

Samples (1) Potted butt: RA-QD02-0032, (2) Ultrathin sections: RA-QD02-0032-1 to 4  
(3) FIB samples: RA-QD02-0032-5 and 6 (for TEM), and 7 (TEM tomography)



**Figure A)** Potted butt: An optical image after UM (2). **B)** BSE image of PB (4). Abbriiations; Ol: olivine, Pl: plagioclase, Tr: troilite. Striations are artifacts formed during ultramicrotmy. **C)** SIM of RA-QD02-0032, on which W depositions protecting FIB sampling areas are observed (6). **D and E)** HAADF-STEM images of RA-QD02-0032-5 and 6, respectively (6).