**TECHNIQUES FOR ION MICROPROBE ANALYSIS OF TINY PARTICLES: COMBINATION OF FIB MARKING AND $^{16}O^-$ ION IMAGING AND SAMPLE MOUNTS USING INDIUM.** D. Nakashima[1], D. E. Brownlee[2], D. J. Joswiak[2], N. T. Kita[1], and T. Ushikubo[1]. [1]WiscSIMS, Dept. Geoscience, University of Wisconsin-Madison, Madison, WI 53706 (naka@geology.wisc.edu), [2]Dept. Astronomy, University of Washington, Seattle, WA 98195.

**Introduction:** High precision oxygen isotope analyses (±1-2‰) of tiny extraterrestrial particles, such as interplanetary dust particles (IDPs) and comet Wild 2 particles, have been successfully made using an IMS-1280 secondary ion mass spectrometer (SIMS) at WiscSIMS laboratory [1]. In the previous studies, particles were individually embedded in epoxy resin with a standard grain, which were grinded and polished to flat disks with 8 mm radius. These small epoxy disks were suitable for accurate oxygen isotope analyses in terms of minimal sample topography and hydride production [e.g., 2]. However, the mounting technique would be difficult for microtomed samples and at risk of consuming significant portion of remaining samples by the regrinding processes [3]. Another technical difficulty in the previous studies was the accuracy of aiming of the analysis locations, which was limited by the optical resolution of the reflected light microscope (originally ~3.5μm, which is recently improved to 1.8μm). Inaccurate aiming caused significant beam overlap with surrounding resin and adjacent mineral phases (and adhering aerogel in the case of Wild 2 particles), which results in significant bias on the measured oxygen isotope ratios.

Resolution to the above difficulties has an advantage of analyzing smaller particles (<10μm) that were previously studied by TEM sections. Here we report two modifications to the analytical procedures: (1) use of indium to mount microtomed samples and (2) use of focused ion beam (FIB) marking on the sample surface that is identified by the $^{16}O^-$ secondary ion imaging.

**Sample mounting procedure using indium:** Indium has been widely used for mounting polished grains for SIMS, though mainly for volatile element analyses. A particle with microtomed surface was mounted in a ~100×100μm square top of mesa of an acrylic potted butt with an 8 mm diameter. An acrylic cube (~100×100×100μm) that contains the particle was cut out from the acrylic potted butt. The acrylic cube and a San Carlos olivine standard grain (~100-200μm in size) were embedded in indium, which was mounted in the hole of 1.4mm diameter on the center of an aluminum disk (8mm diameter and 2.5mm thickness; Fig. 1). The acrylic cube and San Carlos olivine grain were pushed into indium using a flat and clean glass slide so that the acrylic cube and San Carlos olivine grain are on the same plane with the surface of indium as well as aluminum disk. The aluminum disk had an open blind hole where extra indium was pushed (Fig. 1a).

The flatness of the sample mounts was examined by a ZYGO NewView white light profilometer at the Material Science Center, University of Wisconsin. The flatness of the entire aluminum disk including indium, acrylic cube, sample particle, and San Carlos olivine is typically better than 5μm. When we first apply this mounting method to the Wild 2 particle Bidi [1], the San Carlos olivine standard grain was depressed about several dozen μm relative to acrylic and indium thus not used as a running standard of the ion microprobe analyses. Instead, San Carlos olivine grains mounted in other holes were used for standardization. In the later analyses, San Carlos olivine standard grains were always within 1μm of the acrylic cubes [4].

The particle and San Carlos olivine standard grain were located within a 0.7 mm-radius from the center of the aluminum disk (Fig. 1), where instrumental mass fractionation is insignificant (≤±0.5‰ in $\delta^{18}O$; [2]). Oxygen isotope ratios of San Carlos olivine grains embedded with particles in indium did not show detectable difference from those of a San Carlos olivine grain in a single flat 25mm mount.

**FIB marking:** Zeiss 1500XB CrossBeam workstation equipped with gallium ion source at the University of Wisconsin was used to remove carbon coating on the surface of the particles. A 30keV focused $Ga^+$ ion beam was set to 2pA of intensity and was rastered within 1×1μm square on the sample surface ~100s so that only carbon coating was removed without milling silicate from the sample surface (Fig. 2a). The sputtering time depends on the thickness and coating material (carbon or palladium). The depth of milled area relative to the surface of palladium or carbon coating was only ~50nm, which is comparative to the thickness of coating, so that the amount of sample sputtered by the $Ga^+$ ion beam is minimal.

**$^{16}O^-$ ion imaging:** Prior to the oxygen isotope analysis, the secondary $^{16}O^-$ ion image of the particles with a FIB square was obtained. $Cs^+$ ion beam was focused to ≤1μm of diameter (~1pA) that was rastered over an area of 10×10μm. The secondary $^{16}O^-$ ions was detected by an multi-collector EM by applying an X-deflector in the detection chamber without moving other multi-collection detectors nor changing magnetic field that were set up for the oxygen isotope analysis. A similar configuration was originally used for detect-

ing surface contaminated oxygen deposited by the electron probe analyses in U. Hawaii IMS-1280 [K. Nagashima, personal communication]. Since the sample surface is coated by carbon except for the 1×1μm FIB square mark, secondary $^{16}O^-$ signals were generated only from the FIB square within 5min of sputtering (Fig. 2b). We then readjusted the stage position in order to locate the FIB square at the center of the 10×10μm rastered area (center of cross-wire in Fig. 2b), where subsequently the oxygen three-isotope analysis was made in the spot mode. The 10×10μm $^{16}O^-$ ion images were taken after each analysis to confirm the positions of analyzed spots (Fig. 2c). The SIMS pits were also checked using a SEM after the SIMS analysis session (Fig. 2d).

**Test analysis and evaluation:** We analyzed seven FIB squares on a San Carlos olivine standard grain under the same conditions with Wild 2 particles. We evaluate the performance of the new analytical protocols in terms of aiming accuracy and accuracy of oxygen isotope data. Centers of SIMS pits were not significantly deviated from those of the FIB squares, with average and maximum deviations of 0.3μm and 0.7μm, respectively. The minimum resolution of the primary beam deflection and stage motion of the IMS-1280 is 1μm, so that the average deviation of 0.3μm is consistent with best possible accuracy with the current system. We did not find any resolvable bias between standard analyses on FIB squares and regular carbon coated areas within the analytical precision of ~1‰ (2SD).

**Evaluation of Wild 2 sample aiming:** The deviations of SIMS-pit centers from FIB-square centers on the Wild 2 particles are ~0-1.0μm (0.4μm on average), excluding SIMS pits that were purposely deviated from the FIB squares [4]. In the previous oxygen isotope analyses without FIB marking [1], the deviations of SIMS-pit centers from aimed spots on the Wild 2 particles and IDPs are ~0-2.3μm (1.3μm on average; n=10). Thus, we significantly improved our aiming skills to the level that the deviation is much smaller than the SIMS spot sizes.

In summary, the aiming technique that combined FIB and $^{16}O^-$ ion imaging enabled isotope analysis with 0.4μm aiming accuracy. New protocols has an advantage of analyses of smaller particles as small as ~4μm [4] without degrading the accuracy of analyses.

**References**: [1] Nakashima D. et al. (2011a) *LPSC, XLII,* #1240. [2] Nakashima D. et al. (2011b) *MAPS, 46,* 857-874. [3] Nakashima D. et al. (2011c) *MAPS,* In Press. [4] Nakashima D. et al. (2012) This volume.

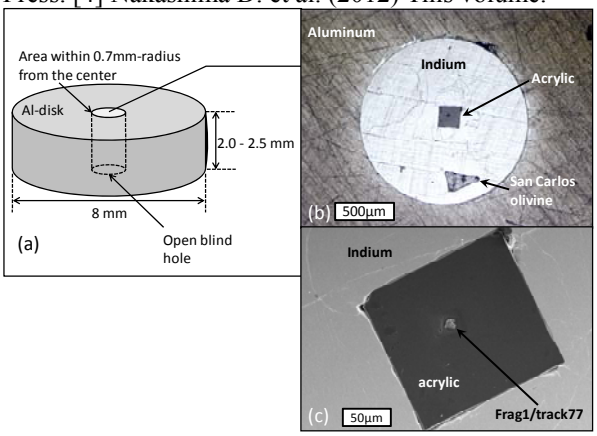


**Fig. 1:** A schematic drawing of an Al-disk (a), an optical microscope image of a Wild 2 particle (fragment 1 from track 77; [4]) (b), and a secondary electron (SE) image of the fragment 1 from track 77 (c).

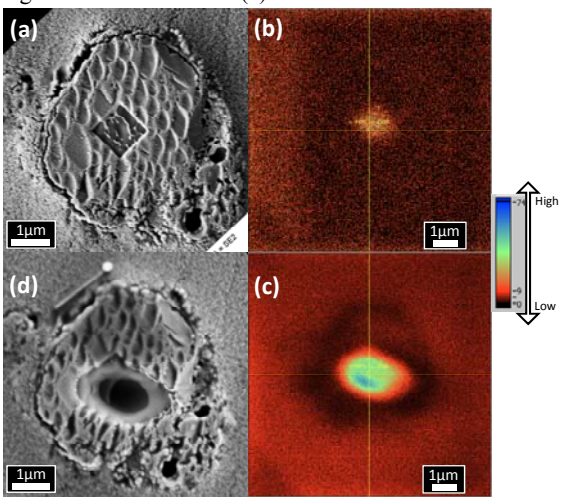


**Fig. 2:** SE image of a Wild 2 particle (fragment 4 from track 77; [4]) before the oxygen isotope analysis (a), $^{16}O^-$ ion image before the oxygen isotope analysis (b), $^{16}O^-$ ion image after the oxygen isotope analysis (c), and SE image of fragment 4 from track 77 after the oxygen isotope analysis (d).