ANALYTICAL DEVELOPMENTS FOR ION MICROPROBE ANALYSIS OF TINY PARTICLES.

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Introduction: Oxygen isotope analyses of tiny extraterrestrial particles (≤100µm), such as particles returned from comet Wild2 and asteroid Itokawa and interplanetary dust particles, have been successfully made using an IMS-1280 secondary ion mass spectrometer (SIMS) at WiscSIMS laboratory [1-4], in which we applied two techniques for accurate analyses: (1) indium mounting and (2) combination of FIB marking and $^{16}O^{-}$ ion imaging. Here we report these two techniques, which are beneficial to high-spatial resolution analysis of terrestrial/extraterrestrial samples which have a few-µm scale fine structure.

Indium mounting [4]: Multiple tiny samples with standards in a flat 25mm disk are desirable to minimize the surface topography effects on high precision isotope analyses [5]. However, preparation (casting and polishing) of a 25mm disk with multiple tiny samples is at risk of consuming a significant portion of samples. To avoid the risk, we prepared a 6mm epoxy disk containing a tiny particle with polished surface, and then epoxy disk was pressed into indium inside of a 25mm diameter Al-disk with standard grains. With this method, we achieved a single flat surface for sample and standard grains with the flatness of the entire disk of better than 40µm. Test analyses with 10µm spots showed that instrumental bias is within 0.3‰ in $\delta^{18}O$ among three San Carlos olivine grains pressed in the Al-disk as long as the tilt of the grains is within 1µm across the 1-2mm diameter.

FIB marking and $^{16}O^{-}$ ion imaging [3]: Accuracy of aiming of the analysis locations in tiny particles (≤10µm) was limited by the optical resolution of the reflected light microscope of SIMS (originally ~3.5µm, which is recently improved to 1.3µm). To improve aiming accuracy, we applied FIB marking and ion imaging. Carbon (or gold) coating of 1×1µm square within a tiny particle is removed by focused ion beam (FIB; Ga^{+}). The FIB marking on the particle surface is identified by the 10×10µm $^{16}O^{-}$ secondary ion imaging using ~1µm Cs^{+} beam of SIMS. We did not find any resolvable bias between standard analyses on FIB squares and regular carbon coated areas within the analytical precision of ~1‰; the average $\delta^{18}O$ value of FIB squares is 0.0±1.3‰ (n=7; 2SD; not corrected for instrumental bias of San Carlos olivine), while that of regular carbon coated areas is +0.1±0.7‰ (n=8; 2SD). The new aiming technique enabled isotope analysis with 0.4µm aiming accuracy and has an advantage of analyses of particles as small as ~4µm.

References: [1] Nakamura T. et al. 2008. *Science* 321:1664-1667. [2] Nakashima D. et al. 2012. *M&PS* 47:197-208. [3] Nakashima D. et al. 2012. *EPSL* 357-358:355-365. [4] Nakashima D. et al. 2013 *EPSL* In Press. [5] Kita N.T. et al. 2009. *Chem. Geol.* 264:43-57.