DEVELOPMENT OF MULTIPLE HOLE DISK FOR ISOTOPE ANALYSIS OF TINY SAMPLES USING ION MICROPROBE. D. Nakashima[1,2], T. Ushikubo[1], N. T. Kita[1], and J. W. Valley[1]. [1]Department of Geoscience, University of Wisconsin-Madison, Madison, WI 53706, USA (naka@geology.wisc.edu), [2]Laboratory for Earthquake Chemistry, University of Tokyo, Tokyo 113-0033, Japan.

Introduction: One of the keys in successfully analyzing stable isotope ratios using ion microprobes in tiny particles such as Antarctic micrometeorites, interplanetary dust particles, and Stardust particles is to obtain maximum polished surface area of the particles or of target minerals within the particles. To do so, it is necessary to polish the tiny samples embedded within resins individually. In addition, the multiple samples should be loaded in an ion microprobe at once for proper standardization and efficient operation.

Nakamura et al. [1] performed oxygen isotope analyses of Stardust particles with WiscSIMS Cameca IMS-1280 ion microprobe at UW-Madison, and they used a special disk with seven holes, which satisfies the requirements shown above. For those analyses, a 1-2µm Cs^+ primary beam was used, and the typical analytical uncertainties of bracketing standard analyses were 1-4‰ (2SD) for $\delta^{18}O$ and $\delta^{17}O$ [1]. Aiming at higher precision analyses, we are developing the new sample holding disk (Fig. 1). The use of multiple-hole sample mounts creates a trade-off of precision and accuracy vs. analysis of a single flat 25mm mount. Here we report the evaluation of the 7-hole performance.

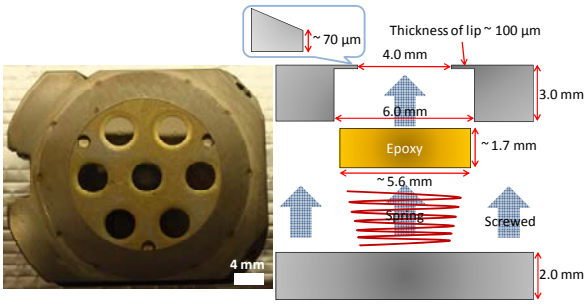

Fig. 1: A photograph of the 7-hole disk mounted in the CAMECA holder (left) and a schematic illustration showing mounting an epoxy disk in the 7-hole disk (right).

7-hole disk: The previous 7-hole disk (25mm in diameter) which was used by [1] consists of three parts: tungsten plate with a thickness of 100µm, the upper disk (3.0mm in thickness; stainless steel), and the bottom disk (2.0mm in thickness; stainless steel). The tungsten plate has 7 windows with a diameter of 4.0mm, and the upper disk has 7 holes with a diameter of 6.0mm. The tungsten plate is spot-welded on the upper disk and works as a lip to keep the polished sample surface to be parallel to the disk surface. Small epoxy disks (~5.6mm in diameter) that contain samples and running standards are put in the upper disk. The bottom disk presses sample surface against the tungsten plate through a spring between sample and the disk and the bottom disk is screwed to the upper disk. The tungsten plate surface has dents due to spot-welding. This may produce a deformed electrostatic field on a sample surface, which leads to low count rates of secondary ions and inaccurate isotopic ratios.

To avoid this possible problem, we developed the new 7-hole disk (Fig. 1). The dimension is the same as that of the tungsten-lipped disk, but the upper disk is fabricated so that it has a 4mm diameter window with a lip machined from a single stainless steel block without a tungsten plate. In addition, lips slope down to holes (~30µm/1.0mm; Fig. 1), which is expected to diminish deformation of electrostatic field around the lip. If epoxy disks are tilted or show surface topography, the measured isotope ratio would be biased [2]. The flatness of the upper disk surface and epoxy disks was checked by a ZYGO™ white light profilometer at the Material Science Center, UW-Madison. The flatness of the disk is better than 5µm and the tilt of epoxy disks is less than ~10µm across 4.0mm window.

Performance test of the new 7-hole disk: We measured oxygen two isotope ratios ($^{18}O/^{16}O$) in San Carlos olivine (SC-Ol) grains mounted in epoxy disks in order to see if there is an analytical bias among different holes. A primary Cs^+ beam was set to a size of 7×12µm with a current of ~2.0nA. Secondary O^- ions were detected with two Faraday cups simultaneously. Typical count rate of $^{16}O^-$ was 3×10^9 cps. Other analytical conditions and measurement procedures were similar to those in [2].

$\delta^{18}O$ variation of SC-Ol grains in outer holes relative to the SC-Ol grain in the center hole was within the 1‰ range (Fig. 2), which is comparable to the result of test analyses using the tungsten-lipped disk [1]. Secondary ion yields (^{16}O count rate/primary beam current) were 94-102% relative to those at the center. In individual SC-Ol grains (~0.5×1mm), $\delta^{18}O$ variations at different locations (north, south, east, and west) relative to the center of the grains were within

the 0.5‰ range. Secondary ion yields were 97-103% relative to those at center of the grains. From these results, oxygen isotope analyses performed within a ~500µm-radius of the center of each hole may not show significant analytical artifacts. Using a flat quartz glass disks (5.6mm in diameter), we found the secondary ion yields decreased by ~5% outside of 500µm radius. This indicates that the electrostatic field may be more deformed towards the lip of the window, which may result in the deviation of secondary ion trajectory even though automatic centering of the secondary ions to the field aperture is applied (DTFA; [2]).

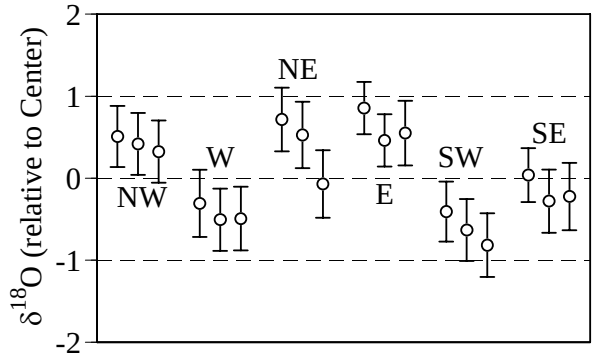

Fig. 2: Relative $\delta^{18}O$ values at outer holes from the bracketed analyses at the center hole. Locations at the center of each hole were analyzed. Bearings correspond to the positions of holes shown in Fig. 1. The three sets of analyses were made at outer holes, bracketed by eight sets of analyses (±0.3‰, 2SD) at the center hole before and after. The Error bars are internal error from counting statistics (2SE).

Chondrule analysis with the new 7-hole disk: Using the new 7-hole disk, we analyzed O three isotopes in CH chondrite chondrules (100-200µm in diameter) with large primary beam (10×15µm; ~2.5nA) and small primary beam (3×4µm; ~28pA) [3]. In every analysis session, an epoxy disk with a SC-Ol grain was mounted in the center hole, and used as a running standard for chondrules, which have no SC-Ol grain exposed on the epoxy surface. Isotopic ratios and ^{16}O count rates of the center SC-Ol grain were comparable to those of the polished thin section of the large San Carlos olivine grain (~20mm), analyzed in advance.

In the first session (large beam), we observed $\delta^{18}O$ values of the outer SC-Ol grains consistently lower by 1‰ than those of the center SC-Ol grain (Fig. 3), which ceased after the sample change ("session 1 continued" in Fig. 3). Secondary ion yields of SC-Ol grains in outer holes were almost comparable (90-98%) to those of the center SC-Ol grain. The lower delta values may be related to sample preparation such as thickness of carbon coating, position of epoxy disks within the holes, and position of the 7-hole disk within the sample holder. It is important to mount running standards with samples in the same epoxy disks so as to correct (unexpected) instrumental fractionation. Nonetheless, potential analytical biases are at the level of 1‰ or less and the raw $\delta^{18}O$ and $\delta^{17}O$ values shift along the slope 0.5 mass fractionation line. Therefore, results obtained without standard grain in the same epoxy disk may not show significantly large bias on $\delta^{18}O$ and $\delta^{17}O$ values and have no measureable effect on the $\Delta^{17}O$ ($=\delta^{17}O-0.52\times\delta^{18}O$) values.

The new 7-hole disk is applicable to tiny particle analysis, although it may induce a small isotopic fractionation. A single well-prepared 25 mm sample mount can yield more precise data. It would be needed to do further improvements including enlargement of surface area of the disk windows, considering the narrow surface area of the disk windows increases deformation of electrostatic field. Currently we are developing a new disk with three holes. The diameter of lip window is 6.0mm and larger than that of the 7-hole disk, which is expected to diminish deformation of electrostatic field. We will report the test analysis of this new 3-hole disk.

References: [1] Nakamura T. et al. (2008) *Science, 321*, 1664-1667. [2] Kita N. T. et al. (2009) *Chem. Geol., 246*, 43-57. [3] Nakashima D. et al. (2010) This volume.

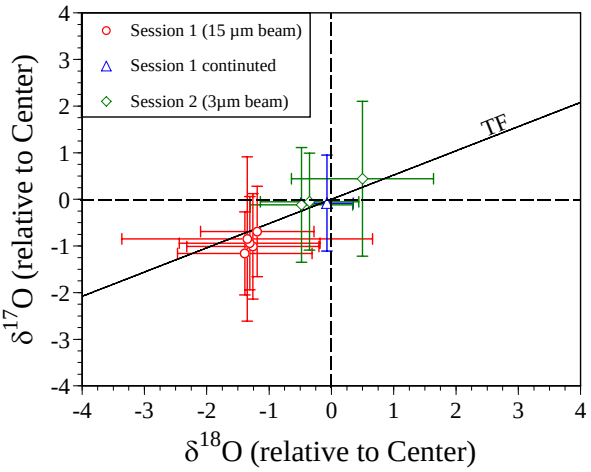

Fig. 3: Relative $\delta^{18}O$ and $\delta^{17}O$ values of outer SC-Ol grains from the analyses at the center SC-Ol grain. In individual holes, the SC-Ol grains were used as running standards bracketing chondrule analyses (6-8 sets of analyses on the SC-Ol grains). Error bars are external reproducibility (2SD). TF represents the terrestrial fractionation line.