REPRODUCIBILITY OF ION PROBE OXYGEN ISOTOPE MEASUREMENTS IN STARDUST COMETARY SAMPLES R. C. Ogliore[1], Z. Gainsforth[2], G. R. Huss[1], K. Nagashima[1], A. J. Westphal[2], [1]Hawai'i Institute of Geophysics and Planetology, University of Hawai'i at Mānoa, Honolulu, HI 96822, USA, [2]Space Sciences Laboratory, University of California Berkeley, Berkeley, CA 94720, USA.

Introduction

The accuracy of ion probe measurements can be reduced by various systematic errors caused by, for example, sample topography or conductivity, changes in the primary beam or secondary ion optics, or variable electron-multiplier efficiency. The magnitude of these systematic errors in ion probe measurements of oxygen isotopes can be large compared to the per-mil level precision needed to address cosmochemical questions in the returned samples from comet Wild 2. For example, FeO-poor ferromagnesian Wild 2 particles cluster at $\Delta^{17}O$ = -2‰ which is lower than the -1.5‰ to +2.5‰ range of FeO-rich particles [1], and a type II Stardust chondrule fragment showed slightly heavy O composition along the terrestrial fractionation line [2]. It is therefore necessary to prove that systematic errors in ion probe measurements of small Stardust grains are small compared to the statistical error of the measurement and can safely be ignored.

Many types of systematic errors in ion probe analyses can be quantified by measurements of known and homogenous isotope standards which are prepared and mounted analogous to the unknown. Repeated measurements of standards before and after the unknown can be used to estimate systematic errors caused by, for example, drifts in electron-multiplier efficiencies.

Sample topography and location as well as differences in charging between sample and standard, on the other hand, produce unpredictable electric-field variations which can be significant [3] and cannot be quantified with measurements of the standard during the measurement session. To quantify these systematic errors, it is necessary to measure the same sample in more than one measurement session. However, the small size of Stardust samples and difficult sample preparation makes this challenging.

With the UH Cameca ims 1280 ion probe, we remeasured the O isotopic composition of grains measured previously [4, 2] to investigate the contribution of systematic uncertainties between measurement sessions separated by years.

Methods

We have developed a reversible sample-preparation technique for small Stardust cometary particles in potted butts for SIMS analysis [5]. This technique allows us to measure the same particle by SIMS more than once at different depths. Our technique to measure fine-grained cometary material in the bulb of Stardust tracks [4] only consumes a small portion of the grains, so the remaining sample can be removed by FIB for reanalysis.

The type II chondrule fragment Iris ($23\times10\times15\mu$m) from Stardust Track C2052,2,74 was measured for oxygen isotopes [2], microtomed, and remounted for SIMS analysis. We measured the same olivine grains for O isotopes using similar analytical conditions as the first measurement: 25–30 pA Cs^+ primary beam focused to a $\sim$2 μm spot, multicollection of O isotopes with ^{16}O on a Faraday cup and ^{17}O, ^{18}O on electron multipliers, $\sim$5500 mass-resolving power for $^{17}O^-$ to minimize the contribution of the $^{16}OH^-$ interference on $^{17}O^-$. We corrected our measurement for any shift seen between the center and ring in an analogously prepared mount of San Carlos olivine. The microtomed faces of Iris before the first and second isotope measurement is shown in Figure 1. It is unlikely that the olivine grains in Iris are isotopically zoned—the O isotopic composition of Iris olivine should be the same at all depths.

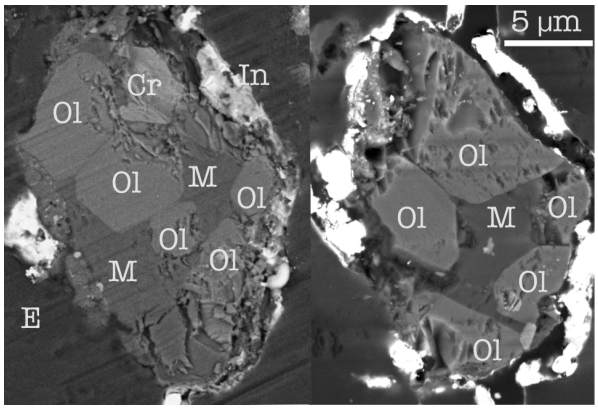

Figure 1: Left: The exposed face of Iris in a potted butt before the first SIMS measurement of O isotopes. Ol=olivine, Cr=chromite, M=mesostasis, In=indium (embedding), E=epoxy (embedding). Right: Iris after further microtoming, before the second SIMS measurement of O isotopes.

Two Fe-rich $\sim$1-μm grains in the wall of Stardust Track C2052,2,74 were measured to have high $\Delta^{17}O$ compositions [4]. We removed a slice of the compressed aerogel containing these particles and others by FIB and placed the slice on gold foil, exposing the particles for additional ion probe analysis (Figure 2). We obtained isotope maps of three oxygen isotopes, ^{16}OH, and $^{56}Fe^{16}O$ of cometary material in this FIB slice, us-

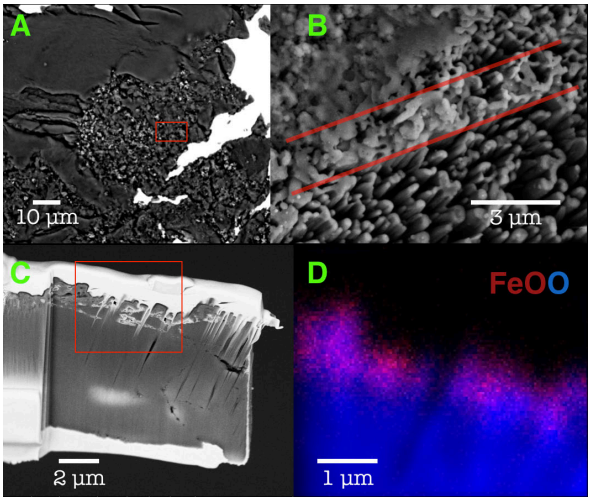

Figure 2: A) Secondary electron image of compressed aerogel from the bulb wall of track C2052,2,74. B) Fe-rich area after the first SIMS measurements, red lines indicate region of FIB cut. C) Backscatter electron image of FIB slice containing Fe-rich grains. D) Secondary ion image of Fe-rich grains.

ing a < 3 pA Cs^+ primary beam focused to $\sim$250 nm, slightly lower beam current and spot size than used previously [4]. TEM analysis of the FIB slice showed these grains to be mostly amorphous.

Results

Our previous O isotope measurement of an Iris olivine was δ^{17}O=4.6$\pm$2.3, δ^{18}O=7.2$\pm$1.6 (2σ uncertainties). Analyses of other olivines in Iris showed that they all have similar O composition. Our measurement of an olivine grain in the re-microtomed potted butt of Iris is δ^{17}O=4.4$\pm$3.1, δ^{18}O=6.3$\pm$2.4, consistent with the first measurement made 2.5 years prior (Figure 3). Uncertainties were slightly larger in our second measurement due to slightly worse reproducibility of the standard measurements.

Our previous measurements of small grains from the bulb wall of track C2052,2,74 showed a wide range of O isotopic compositions, with two grains showing high values of Δ^{17}O. Measurements of four particles in a removed FIB slice generally confirmed the results of our first measurement, as shown in Figure 4.

Conclusions

We remeasured different faces of previously measured Stardust cometary grains in order to understand the contribution of systematic uncertainties that may arise in different measurement sessions. No significant systematic uncertainties were seen in the potted butt measurement of a large terminal fragment. Remeasurements of

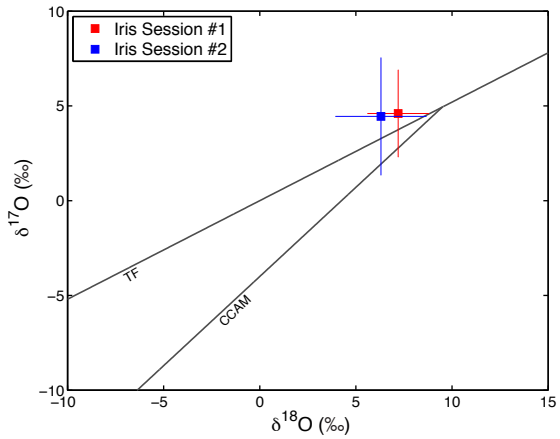

Figure 3: O isotope measurements of olivine in Iris from Session #1 (red squares, left image in Figure 1) and Session # 2 (blue squares, right image in Figure 1). Error bars are 2σ.

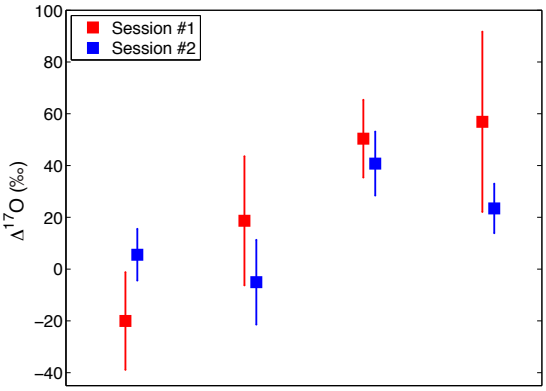

Figure 4: Δ^{17}O of four μm-sized grains from the wall of track C2052,2,74 made in compressed aerogel (red squares) and in the same four grains in a removed FIB section (blue squares). Error bars are 2σ.

four grains in the fine-grained bulb material overlapped Δ^{17}O 2σ uncertainties with the original measurement. Small differences between the two measurements of the bulb grains could be due to analyzing a different phase in the same aggregate grain (the measured face in the FIB slice is rotated 90° from the face in the first measurement). These results indicate that future measurements of Stardust samples prepared and measured in a similar fashion are not likely to be significantly compromised by systematic uncertainties.

References: [1] D. Nakashima, et al. (2012) *Earth Planet Sc Lett* 357:355. [2] R. C. Ogliore, et al. (2012) *Astrophys J Lett* 745(2):L19. [3] N. T. Kita, et al. (2009) *Chem Geol* 264:43. [4] R. C. Ogliore, et al. (2013) *44th LPSC* 2950. [5] A. J. Westphal, et al. (2011) *Meteorit Planet Sci* 74:5274.