CONTINUED STUDIES OF STARDUST IN IDPS. S. Messenger¹, L. P. Keller², and R. M. Walker¹,
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Introduction: We recently reported the first identification of stardust in IDPs [1]. Here we present the results of a detailed analysis of the original and subsequent O isotopic studies of anhydrous cluster IDPs. Anhydrous cluster IDPs were selected because they have preserved molecular cloud material, as evidenced by large enrichments in deuterium [2]. Further, they have escaped aqueous and thermal processing, which could destroy or modify their mineralogy.

Experimental: We performed oxygen isotopic imaging of fragments from 9 anhydrous cluster IDPs with the Washington University NanoSIMS ion microprobe. The NanoSIMS enables O isotopic measurements with a spatial resolution of 100 nm at unprecedented sensitivity. The O imaging techniques generally followed those described in [1]. We have found that the instrumental mass discrimination can be sensitive to the sample stage position for given tuning conditions. Consequently, the matrix material in each IDP is used to correct for variation in detector sensitivity and mass fractionation. The standard deviations of the O isotopic ratios for the subgrains in each IDP were used to estimate the degree of additional random error in the isotopic measurements for the final calculation of the measurement uncertainties.

Three different sample preparation techniques were employed: 5-10 µm fragments pressed into Au substrates, 70 nm sections mounted on transmission electron microscopy (TEM) grids, and 70 nm sections deposited directly onto Au. Five of the sections were analyzed in detail by TEM prior to measurement by ion probe for coordinated mineralogical and isotopic analysis. All grains in these sections > 100 nm in diameter were characterized by electron diffraction, dark-field imaging, and quantitative EDS. In one case, we sliced an IDP which had previously been analyzed by NanoSIMS. Here, a 200 x 200 µm area of the Au substrate containing the IDP was extracted by hand with a scalpel blade, affixed to a flattened epoxy bullet with a cyanoacrylate adhesive, trimmed with a glass knife, embedded in resin and finally sliced by ultramicrotome. With the exception of the first few slices, the material is generally unaffected by the prior ion probe measurement.

The data were analyzed by defining the outlines of subgrains within the images by hand using custom written IDL software. The outline of each grain and its persistence with depth was checked manually in subsequent image layers.

Results: Of all the subgrains defined in 25 images from 9 cluster IDPs, roughly 1031 were measured with sufficient precision to distinguish solar material from circumstellar dust ($1\sigma \delta^{15}O < 75‰, 1\sigma \delta^{18}O < 50 ‰$) as shown in Figure 1. Only grains > 200 nm were measured with this level of precision. Six of these grains have O isotopic compositions which fall well outside the range of solar system materials, marking them as stellar condensates. In all six cases, the Si/O secondary ion ratios fall within the range observed for silicates. We estimate a preliminary abundance of presolar silicates in IDPs of ~5,500 ppm by taking the ratio of the total area imaged to that of the presolar grains.

![Figure 1a: O isotopic ratios of 1031 subgrains measured in 25 images from 9 anhydrous cluster IDPs. Six of these grains are identified as presolar silicates.](image1a)

We compare the O isotopic ratios of the presolar silicates with presolar oxide grains from meteorites in Figure 2. Three of the silicates have large $^{17}O$ excesses and solar $^{18}O/^{16}O$ ratios. Their probable stellar sources are O-rich red giant (RG) or asymptotic red giant branch (AGB) stars of different masses. One grain is $^{16}O$-rich, and is likely from a low metallicity source.

![Figure 1b: O isotopic ratios the 750 subgrains with the smallest errors ($\delta^{17}O < 50‰$) shown at an expanded scale.](image1b)
AGB star. Two grains are enriched in $^{17}$O and $^{18}$O. These grains do not have a known stellar source, but possibilities are high metallicity stars and type II supernovae [4].

Figure 2: Comparison of the O isotopic compositions of presolar silicates with those of circumstellar corundum [3-5] and spinel grains [6].

Approximately 112 of the subgrains in the 5 mapped microtome thin sections were measured with the minimum precision given above. Two of the grains in these sections were determined to be presolar: a 300 nm forsterite grain and a 400 nm GEMS (glass with embedded metal and sulfides, [7]) grain. In one case, we determined the mineralogy of a presolar grain (also a GEMS grain) by slicing the grain for TEM following NanoSIMS analysis (Fig. 2). This grain was subsequently re-measured by NanoSIMS to confirm its unusual $^{16}$O-poor isotopic composition.

These results confirm the mounting circumstantial evidence linking GEMS to the common interstellar silicates [7]. The identification of GEMS as interstellar grains is consistent with the abundant spectroscopic evidence that most (> 95 %) interstellar silicates are amorphous [8].

The remaining 110 isotopically solar characterized grains include 36 enstatites, 40 GEMS, 23 olivines (Fo 100-60), 5 anorthites, 3 Ca,Al,Mg-rich glassy grains, 2 low-Ca pyroxene grains and 1 grain each of diopside and chromite. Many of these grains, including the GEMS are good candidates for stardust. Although it is possible that the GEMS formed in the ISM by reaccretion of sputtered material, the abundant crystalline material in these particles cannot have formed in a similar way. This implies that, despite being rich in stardust, these IDPs are not pristine aggregates of interstellar materials. It is possible that most of the crystalline silicates and GEMS are of solar system origin, variably mixed with a minor population of circumstellar grains.

The imminent return of cometary samples by the Stardust spacecraft will shed light on many unresolved problems. A priority measurement will be to investigate the isotopic compositions of the abundant crystalline silicates in comets, as this could determine whether there was large-scale radial transport of material in the solar nebula.

An important focus for future isotopic studies of IDPs will be the smallest (100-200 nm) grains, which are more representative of the interstellar grain population. Among the three sets of samples, the 70 nm sections deposited directly onto Au yielded the highest precision measurements for a given grain size. Taking the additional step of transferring TEM-mounted sections to Au will be essential to push these measurements to the finest scales.

Figure 2: TEM images of IDP L2036 CI4 showing the location of a presolar GEMS grain (G). The lacy gray material surrounding this grain is abundant indigenous carbonaceous matrix. The black material at the bottom is Au from the sample mount. A fold in the slice is also apparent.