ENABLING Al-Mg ISOTOPIC MEASUREMENTS ON COMET WILD 2's MICRO-CAIs. H. A. Ishii[1] , D. Joswiak[2], J. P. Bradley[1], N. Teslich[1], J. Matzel[1], I. D. Hutcheon[1], D. Brownlee[2], G. Matrajt[2], G. MacPherson[3] and K. D. McKeegan[4], [1]Institute of Geophysics & Planetary Physics, LLNL, 7000 East Avenue, Livermore, CA 94550; hope.ishii@llnl.gov, [2]Dept. of Astronomy, Box 351580, University of Washington, Seattle, WA 98195, [3]US National Museum of Natural History, Smithsonian Institution, Washington DC 20560, [4]Dept. Earth and Space Sci., University of California, Los Angeles, CA 90095.

Introduction: A few small CAIs (calcium-aluminum inclusions) , ~15 microns and smaller, have been identified among comet 81P/Wild 2 dust returned by Stardust. We have proposed to measure ^{26}Mg excesses in these objects, resulting from decay of initial radiogenic ^{26}Al, in order to compare their crystallization ages to those of meteoritic CAIs, many with 'canonical' initial $^{26}Al/^{27}Al \sim 5\times10^{-5}$. Meteoritic CAIs characterized in this manner are typically an order of magnitude or larger in mass than the Stardust CAIs [1-2, for example]. To successfully measure these micro-CAIs, the sub-micron probe size of a NanoSIMS is necessary. Assuming a canonical initial $^{26}Al/^{27}Al$ ratio, Al/Mg ~100 and a few % precision, an estimated minimum volume of ~10^7 nm^3 is required. Given that the majority of relevant samples are available as TEM sections <100 nm in thickness, an area of ~500x500 nm^2 is required for analysis.

In such small and complex CAIs, technical challenges have included 1) finding suffiently large volume of minerals, such as plagioclase or gehlenitic melilite, that are candidates for these measurements and 2) ensuring any Mg-bearing minerals remaining nearby are sufficiently small as to not over-dilute the measurements. We demonstrate the search for appropriate samples and the selective removal of interfering minerals in preparation for isotope measurements on a sub-micron scale for Stardust CAIs. Nano-scale "surgery" on small samples is an important new approach enabling isotope measurements otherwise not possible.

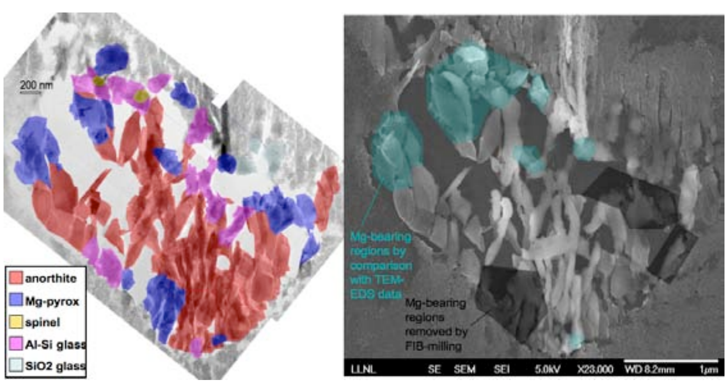

Figure 1. Mineral map overlaid on a TEM brightfield image (left) of Coki-B-5, fragment 2, grid 3C (anorthite: red, pyroxenes: blue, Mg-Al spinel: yellow, Al-Si glass: magenta, silica glass: light blue. The corresponding FE-SEM image is shown (right) prior to FIB preparations with Mg-bearing regions indicated.

Results: We have searched for plagioclase regions of suffient size to allow measurement of ^{26}Mg excesses in two Stardust CAIs named "Inti" and "Coki". Inti has been the subject of intense study, and its mineralogy and O isotopes are characteristic of those of CAIs in primitive meteorites [3-5]. Coki displays similar mineralogy to Inti, and a relatively large region of plagioclase (anorthite, $CaAl_2Si_2O_8$) was identified by TEM analysis in a thin section prepared by ultramicrotomy. Careful mineral mapping (Fig. 1) identified fragments of anorthite surrounded by Mg-rich minerals including pyroxenes and Mg-Al spinel and Al-Si glass. To reduce the dilution of excess ^{26}Mg by neighboring Mg-rich areas, a FIB (focused ion beam instrument) was employed to remove areas of interfering Mg-bearing material (Fig. 2). The anorthite thus isolated is >1.3 micron on a side, sufficient for Al-Mg isotope measurements in progress.

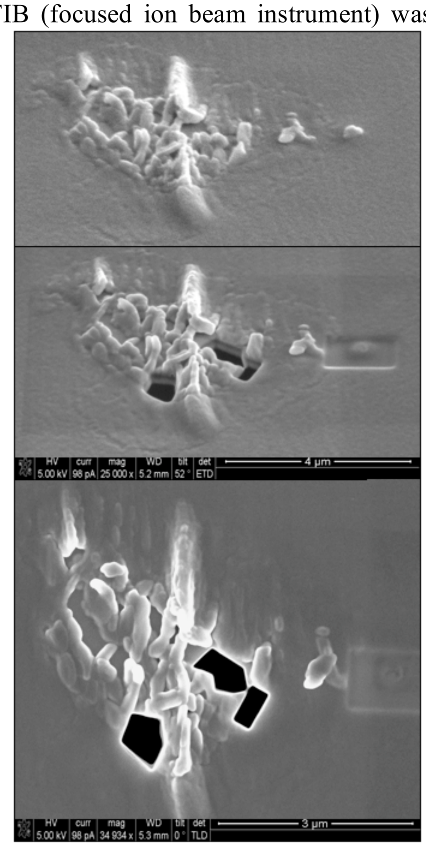

Figure 2. FIB-SEM images, before (above at 52°) and after FIB milling of Mg-bearing regions (middle at 52° and bottom at 0°). The surrounding regions of the sample experienced minimal ion damage. The grain to the far right was sacrificed in order to focus and stigmate the ion beam prior to imaging the region of interest.

Preparation Methods: The Coki CAI fragment was embedded in acrylic, ultramicrotomed, and placed on Au TEM grids with C substrates at University of Washington. Initial characterization indicated a large region of anorthite in the Coki-B-5 section, and it was further mapped at LLNL by TEM-EDS to locate all Mg-bearing regions in preparation for FIB milling (Fig. 1, left). FE-SEM imaging (Fig 1., right) was carried out to assist in identifying target regions once the sample was transferred to the FIB. To provide additional mechanical support to the CAI material for the subsequent FIB milling and for the final NanoSIMS sputtering, a Pt strap was first deposited behind the thin section of Coki from one grid bar to the other on the backside of the C film over the TEM grid opening. A second Pt strap was deposited perpendicular to the first (Fig. 3). The highly focused Ga+ ion beam of the FIB was used to precisely and accurately mill away three regions of Mg-bearing material nearest a region dense with anorthite shards. This created a region free of Mg-bearing materials for less dilution of NanoSIMS measurements of excess ^{26}Mg.

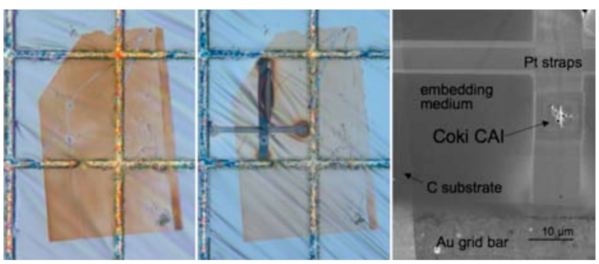

Figure 3. (left) Optical image of the ultramicrotomed slice of Coki, embedded in acrylic, on a Au TEM grid with a C substrate. (middle) Optical image after the deposition of Pt straps on the reverse side and FIB milling of several Mg-rich areas. (right) FIB-SEM image of the region containing the Coki CAI after FIB milling. Coki is located just below the intersection of two Pt straps (under the C substrate and the ultramicrotomed section).

In addition to the CAI Coki, thin sections of Inti were searched through by TEM to locate regions of sufficient area for ^{26}Mg excess measurements. Inti displays very fine-grained mineralogy, and in the most plagioclase-rich section analyzed, anorthite was found interspersed with Mg-bearing minerals (diopside, pyroxene, spinel) on size scales of a few 100 nm (Fig. 4) making successful isotope measurements impossible. We will continue to search for a good candidate for Al-Mg isotope measurements in Inti for comparison with Coki and with meteoritic CAIs.

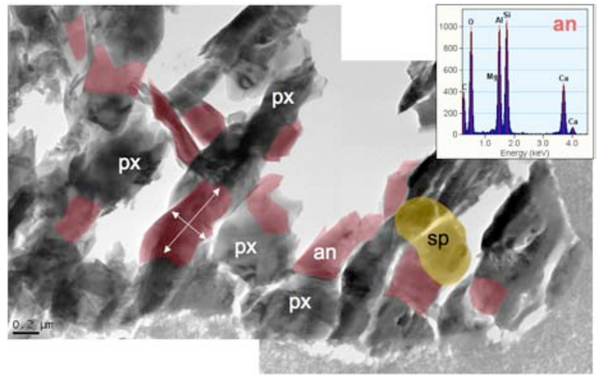

Figure 4. Anorthite-rich region of a thin section of Inti (Inti-2-A3). Anorthite is interspersed with Mg-bearing pyroxene and spinel on a size scale of a few 100 nms. The largest shard of anorthite is indicated by arrows and is ~300x700 nm. An energy-dispersive spectrum from an anorthite shard is inset.

Conclusions: The ability to precisely and selectively remove – or alternatively mask – interfering minerals in meteoritic samples by FIB milling or deposition makes possible isotope measurements that would otherwise be impossible. In the case of the micro-CAIs from comet 81P/Wild 2, the small grain sizes of the constituent minerals make Al-Mg isotopic measurements a severe challenge. With TEM mineral mapping and FIB nano-scale "surgical methods", we have freed an area of anorthite of Mg-bearing minerals in an ultramicrotomed section of the Coki CAI to provide a large enough volume for measuring the ^{26}Mg excess in this unique cometary CAI.

References: [1] MacPherson et al. (2007) *LPS XXXVIII*, Abstract #1378. [2] Makide et al. (2008) *LPS XXXIX*, Abstract #2407. [3] Zolensky et al. (2006) *Science, 314,* 1735. [4] McKeegan et al. (2006) *Science, 314,* 1724. [5] Simon et al. (2008) *Meteoritics & Planet. Sci., in press*.

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