LACK OF EVIDENCE FOR IN SITU DECAY OF ALUMINUM-26 IN COMET 81P/WILD 2 CAI-LIKE REFRACTORY PARTICLES 'INTI' AND 'COKI'. H. A. Ishii[1], F. J. Stadermann[2], C. Floss[2], D. Joswiak[3], J.P. Bradley[1], N. Teslich[1], D. E. Brownlee[3], G. Matrajt[3], G. MacPherson[4] and K. D. McKeegan[5], [1]Institute of Geophysics and Planetary Physics, Lawrence Livermore National Laboratory, 7000 East Avenue, Livermore, CA 94550 (hope.ishii@llnl.gov); [2]Laboratory for Space Sciences and Physics Dept., CB 1105, One Brookings Drive, Washington University, St. Louis, MO 63130; [3]Dept. of Astronomy, Box 351580, University of Washington, Seattle, WA 98195; [4]U.S. National Museum of Natural History, Smithsonian Institution, Washington DC 20560. [5]Dept. of Earth and Space Sciences, University of California, Los Angeles, CA 90095.

Introduction: To date, a few small calcium-aluminum-rich-inclusion-like (CAI-like) refractory particles have been identified in comet 81P/Wild 2 samples returned by the Stardust mission. 'Inti' is a fine-grained polymineralic refractory particle, originally perhaps ~30 µm across, that produced several mineralogically-related fragments on capture into silica aerogel [1-2]. Inti is comprised of sub-micron anorthite; fassaite; aluminous diopside, spinel and gehlenitic melilite that contain minor < 100 nm grains of osbornite [3-4]; and minor perovskite. The ~15 µm Inti terminal particle is ^{16}O-rich like refractory inclusions in chondrites [5]. 'Coki-B' is another ~5 µm polymineralic refractory object dominated by anorthitic plagioclase with < 200 nm spinel inclusions, minor diopside and fassaite [6].

For comparison with (typically much larger) meteoritic CAIs, Al-Mg isotopic measurements of these refractory particles are of great interest: The decay of radiogenic ^{26}Al incorporated in Al-rich minerals leaves excess ^{26}Mg that can provide a measure of the time at which these objects crystallized relative to those with 'canonical' initial ($^{26}Al/^{27}Al$) of ~5×10^{-5} [7-8]. Such comparisons assume a uniform starting distribution of ^{26}Al in the early solar system and minimal disturbance of host minerals by later reprocessing. The first successful Al-Mg isotopic measurement of Wild 2 cometary material was recently reported on a sample of Coki [9-10], but our prior attempts to look for evidence of decayed ^{26}Al in Inti have been hampered by the fine-grained nature of this CAI [6].

Here we present TEM and modified focused ion beam (FIB) preparation methods for NanoSIMS measurements and the resulting $(^{26}Al/^{27}Al)_o$ values from an ultramicrotomed section of Inti as well as possible implications for this CAI's history prior to incorporation in comet Wild 2. We also report independent confirmation of an upper limit on $(^{26}Al/^{27}Al)_o$ in Coki [9-10].

Samples and Methods: Ultramicrotomed thin sections of particles Inti-G and Coki-B embedded in acrylic [11] were prepared at UW and placed on thin C substrates on Cu TEM grids. Analytical TEM analyses were carried out at UW and LLNL on 200kV FEI Tecnai and 300kV FEI Titan field emission analytical scanning transmission electron microscopes ((S)TEM) to produce mineral maps. An FEI Nova NanoLab600 dual-beam FIB was used to deposit a Pt support strap behind each particle section and to "surgically" mill out interfering Mg-rich (spinel, pyroxene) fragments [6] leaving isolated anorthite. In this work, an extra-thick Pt pad (~2.5-4.0 µm) was grown behind each particle region (Fig. 1). The Pt provided excellent stability and durability for the samples which otherwise tend to be short-lived during NanoSIMS analyses due to failure of the C substrate of the TEM grid.

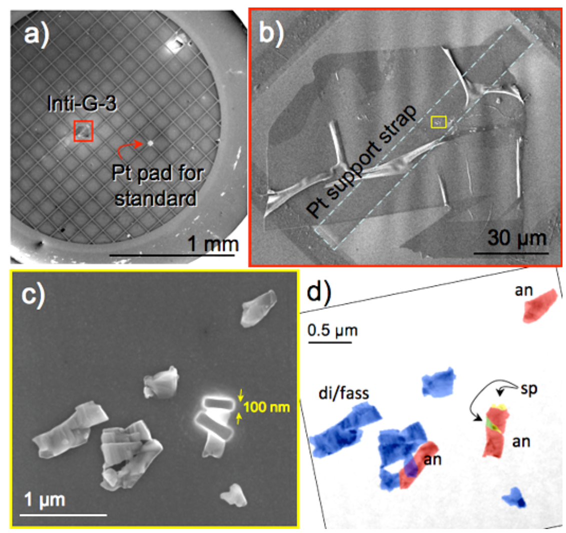

Figure 1. TEM and FIB preparation for NanoSIMS measurements on Inti-G. SEM image showing (a) TEM grid with ultramicrotomed section (in red box). (b) Region with section. Dashed outline indicates the extent of the FIB-deposited Pt support strap on the underside of the sample. Yellow box indicates region magnified in (c) showing removal of Mg-rich regions from an anorthite grain by FIB nano-scale surgery. (d) Corresponding false-color TEM brightfield image. (an=anorthite, sp=spinel, di=diopside, fass=fassaite).

Al-Mg isotope measurements were carried out with the Washington University NanoSIMS 50, using an O^- primary ion beam of ~25 pA. The measurements were made in imaging mode, with simultaneous detection of $^{24}Mg^+$, $^{25}Mg^+$, $^{26}Mg^+$, and $^{27}Al^+$, by rastering the ion beam over 6 x 6 to 10 x 10 μm^2 areas of the Coki-B and Inti-G ultramicrotomed sections. Mg isotopic ratios were extracted from carefully defined regions of interest in the images and were normalized to the Mg isotopic compositions of a Lake County labradorite standard measured before and after the sample measurements. Measured $^{27}Al/^{24}Mg$ ratios were corrected for the relative sensitivity factor determined from a plagioclase standard (USNM anorthite).

Results: The $(^{26}Al/^{27}Al)_o$ value obtained from Inti plagioclase is (-1.4 ± 3.8) x 10^{-5}. Thus, Inti shows no evidence for *in situ* decay of ^{26}Al. Indeed, Inti plagioclase and fassaite in the same isotope image produce essentially identical $\delta^{26}Mg^*$ (Fig. 2) providing internal calibration and confirmation of the lack of ^{26}Mg excess in Inti. Assuming Gaussian statistics, the results indicate an 84% probability that the Inti $(^{26}Al/^{27}Al)_o$ is $<2.4x10^{-5}$, less than half the canonical value. The large uncertainty is due to the miniscule sample volume (estimated to be ~$6.4x10^{-3}$ μm^3) that was sufficiently separated from nearby Mg-bearing phases given the lateral extent of the NanoSIMS O^- beam.

Coki plagioclase yields $(^{26}Al/^{27}Al)_o$ = (8.6 ± 8.4) x 10^{-6}. Again, assuming Gaussian statistics, there is an 84% probability that the Coki $(^{26}Al/^{27}Al)_o$ is $<1.7x10^{-5}$, consistent with previous results [9-10].

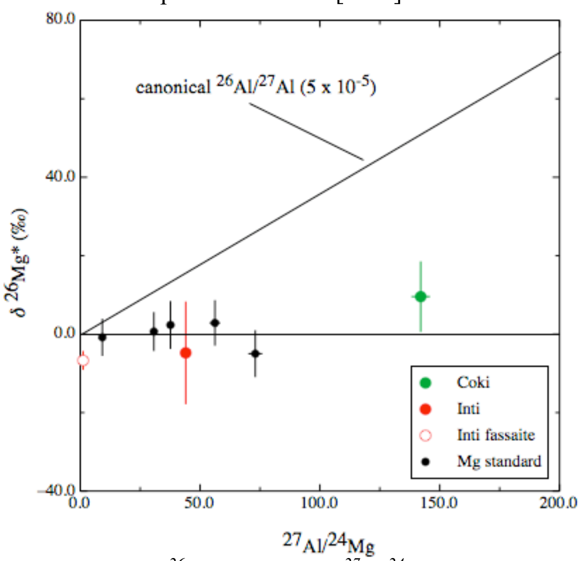

Figure 2. Excess ^{26}Mg (‰) versus $^{27}Al/^{24}Mg$ for Inti plagioclase, Coki plagioclase, Inti fassaite and a Mg isotope standard (Lake County labradorite). Error bars are 1σ.

Discussion and Conclusions: Both of the Wild 2 CAI-like particles analyzed to date, Inti and Coki, show no evidence for *in situ* decay of ^{26}Al. Neither particle demonstrates unusual Mg fractionation, and Inti has O isotopes typical for CAIs [5], so neither particle is an F- or FUN-CAI with unusual isotopes [7]. Most "normal" CAIs have near-canonical $(^{26}Al/^{27}Al)_o$, and although many exhibit complex histories of reprocessing leading to lower-than-canonical ratios, these are typically still resolvable from zero excess.

We consider several possible scenarios for Inti: (A) Inti formed or recrystallized long after canonical CAIs (few Myr), i.e. after the decay of "live" ^{26}Al, a scenario that has been proposed for Coki [9-10]. Late-stage melting of Inti, however, is unlikely given its irregular shape, complex and non-igneous texture, and its ^{16}O-rich composition typical of canonical CAIs. (B) Inti formed contemporaneously with canonical CAIs but from a reservoir devoid of ^{26}Al. Most CAIs formed from what appears to be a fairly uniform reservoir of ^{26}Al, but there are suggestions of heterogeneity from isotopically anomalous CAIs (FUN, F, UN) that lack ^{26}Al. Since Inti shows no detectable mass-dependent fractionation, it might be UN [cf. 12]. (C) Inti formed contemporaneously with canonical CAIs and incorporated canonical ^{26}Al, but in the intervening 4.6 Gyr, Mg isotope redistribution within the particle diluted the original excess ^{26}Mg beyond our ability to resolve it. Inti shows little evidence for significant reprocessing: It has a diopside rim but lacks secondary alteration minerals such as Fe-rich spinel and feldspathoids indicative of parent body alteration [2]. However, it is very small (perhaps ~30 μm originally and irregularly shaped), so interdiffusion of isotopes might readily have occurred in the solid state without effect on petrography. Closure temperatures for the Al-Mg system are typically calculated for grain sizes orders of magnitude larger than those in the Wild 2 CAI-like particles [e.g. 13]. For the small grain sizes in Inti and Coki, significant isotope exchange may have occurred during initial cooling of the CAIs or later at very modest temperatures, perhaps not requiring residence on a parent body. If interdiffusion played a significant role, we may be stymied in our attempts to date comet Wild 2's micro-CAIs due to their diminutive size. We plan Al-Mg isotope analyses of additional, hopefully larger, Wild 2 refractory inclusions as they are discovered.

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Acknowledgements: J. Matzel and I. D. Hutcheon are thanked for useful discussions on the first Al-Mg measurements on Coki. Portions of this work were performed under the auspices of the U.S. DOE by LLNL under contract DE-AC52-07NA27344. This work was supported by NASA Grants NNH07AG46I to HAI, NNX07AM63G to FJS and LDRD Grant 09-ERI-004 to JPB.