

NEW INTEGRATED ANALYTICAL CAPABILITY AT THE UNIVERSITY OF HAWAI‘I. H. A. Ishii¹, J. P. Bradley¹, K. Nagishima¹ and G. R. Huss¹, ¹Hawai‘i Institute of Geophysics and Planetology, University of Hawai‘i at Mānoa, 1680 East West Road, POST 602, Honolulu, HI 96822, USA (hope.ishii@hawaii.edu).

Introduction: The benefits of integrating instruments to enhance science yield were demonstrated during the Stardust preliminary examination [1-3]. We are implementing this approach at the University of Hawai‘i (UH). Existing electron microprobe capabilities in the Dept. of Geology and Geophysics and ion microprobe, SEM and Raman capabilities in the W. M. Keck Cosmochemistry Lab are now augmented with the new Advanced Electron Microscopy Center (AMEC), which hosts a 60-300 keV monochromated and dual spherical (C_s) aberration-corrected Titan TEM/STEM and our newest addition, a Helios NanoLab 660 dual-beam focused ion beam (FIB) instrument. The Titan has a high-angle annular dark field (HAADF) detector, Tridium Gatan imaging filter (GIF) for imaging and spectroscopy, and an EDAX Genesis 4000 Si(Li) energy dispersive x-ray spectrometer. The FIB is equipped with an Oxford Instruments Xmax N80 SD detector for x-ray spectroscopy and mapping, retractable back-scatter and STEM detectors, EasyLift in-situ manipulator and C, Pt and W gas chemistries. These capabilities permit us to integrate TEM data with isotope data from the UH Cameca IMS 1280 ion microprobe, which provides $<1 \mu\text{m}$ resolution scanning or, with SCAPS, direct ion imaging, as well as with Raman spectroscopy from the Witec Confocal Raman Scanning System. Titan analyses can be performed at UH or from Lunar and Planetary Institute (LPI) in Tucson via commercial fiber optic network and a remote Titan control platform (PI T. Zega).

Examples: One of the key capabilities of the FIB is to reconfigure a tiny TEM specimen like a thin section of a $\sim 10 \mu\text{m}$ IDP into a SIMS compatible specimen. (Fig. 1a). Using the Helios FIB, a Pt support strap and platform are deposited on the underside of the carbon film (Fig. 1b). Without this platform the lifetime of the specimen in the ion beam is too short to reliably measure its isotopic compositions. Conversely, an isotopic “hot spot” in a thick-flat SIMS specimen can be harvested and reconfigured into an electron transparent TEM specimen. Figure 2 is an example of extraction of an extreme ^{15}N anomaly for TEM analysis [4].

Figure 3 illustrates a key Titan capability, a sub-Å electron probe as demonstrated by single atom imaging. Sub-Å probes and $0.7+$ steradian x-ray detectors provide access to the largely unexplored realm of nano-petrography where low-electron-dose mapping with spatial resolution of 1-2 nm is now routine in suitably thin specimens. Figure 4 shows an example applied to FeNi sulfides, the most abundant crystalline

phase in IDPs. Hexagonal 2C pyrrhotite is the predominant polytype. The relative distributions of S, Fe and Ni in the maps provide evidence of low temperature thermal alteration consistent with previous work [5]. For example, S and Fe distributions correlate in the

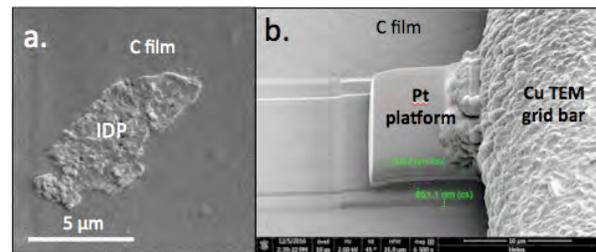


Figure 1: Secondary electron images of (a) an ultramicrotomed thin section of CP U217B19 on C support film and (b) the underside of support film after FIB deposition of a Pt strap and platform beneath the thin section shown in (a).

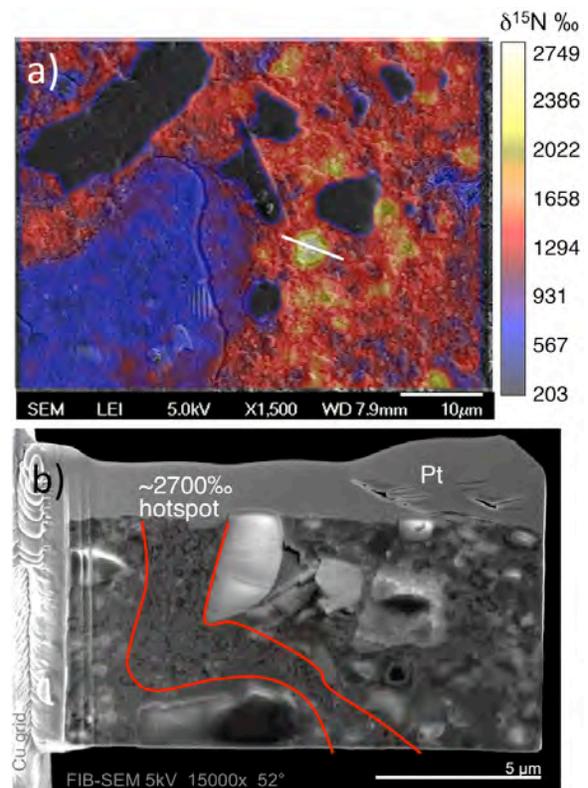


Figure 2: Lithic clasts in the Isheyevo meteorite show the highest ^{15}N anomalies ever measured. (a) ims 1280 $d^{15}\text{N}$ isotope map overlaid on SEM image of lithic clast from Isheyevo (CH/CB). White line indicates location from which FIB section was extracted. (b) FIB-SEM image of FIB section through ^{15}N -rich hotspot corresponds to vein of altered material rich in organics prior to final thinning [4].

FeNi-sulfide grain “1” but Ni and Fe are decoupled. Other grains are Ni-enriched and S-depleted to varying degrees (“2” and “3”). Grain “4” shows a S-enriched

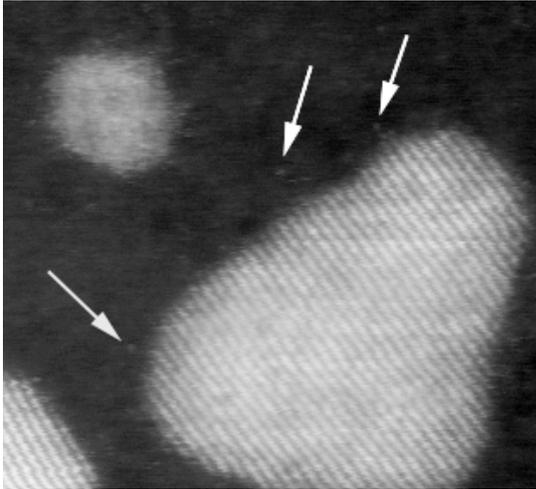
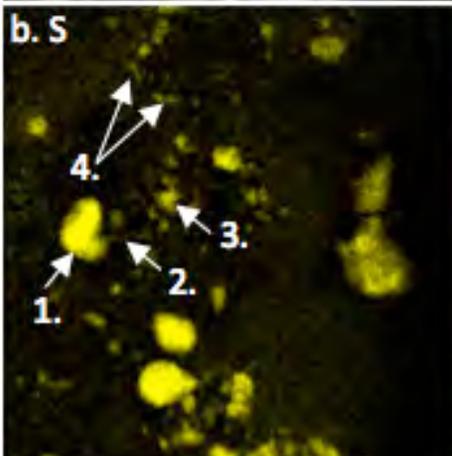
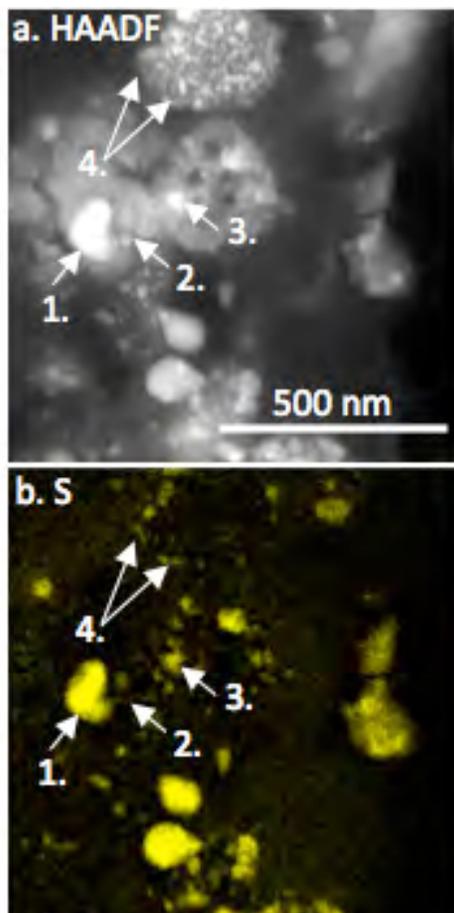


Figure 3: HAADF image shows several individual Au atoms (arrowed) teased off the edge of a gold nanoparticle using the sub-Å electron probe (300 keV Titan STEM image).



rim with a corresponding S-depleted interior of a GEMS grain. These S-enriched rims have been interpreted as a primordial property that show GEMS formed in the solar nebula [6], but *in-situ* experiments using a TEM heating stage implicate atmospheric entry heating as the origin of S-enriched rims [7].

Conclusions: Integration of new sample preparation and analytical capabilities at UH, the Titan TEM/STEM and Helios dual beam FIB, with existing capabilities, Cameca ims1280 ion microprobe, JEOL LV SEM and Witec Confocal Raman Scanning System, will enable assessment of fundamental properties of extraterrestrial materials with improved resolution and fidelity. Of particular interest are those fine-grained components, minimally explored thus far, and features that can now provide distinctions between primordial and secondary alteration effects.

References: [1] Graham G. et al. (2008) *MAPS* 43, 561-569. [2] Ishii H.A. et al. (2010) *LPSC* 41, Abs. 2317. [3] Matzel J. et al. (2010) *Science* 328, 483-486. [4] Bonal L. et al (2010) *GCA* 74, 6590-6609. [5] Dai Z.R. and Bradley J.P. (2001) *GCA* 61, 3601-3612. [6] Keller L.P. and Messenger S. (2011) *GCA* 75, 5336-65. [7] Bradley J.P. et al. (2014) *LPSC* 45, Abs. 1777.

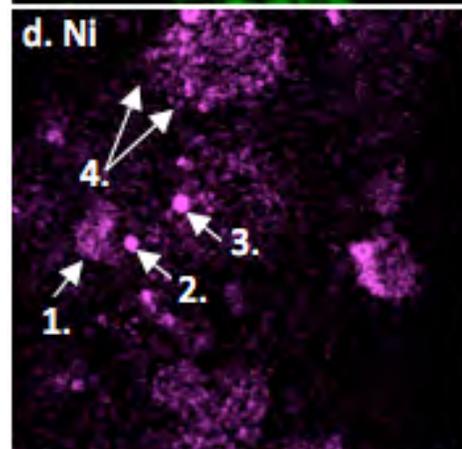
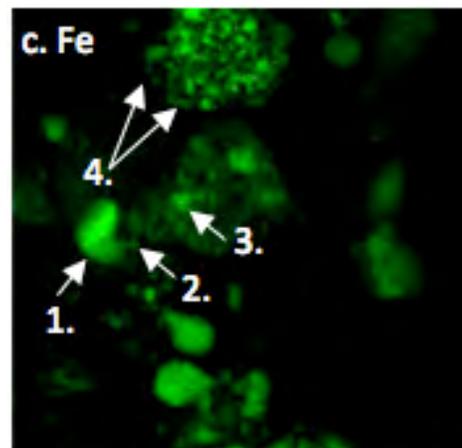


Figure 4: HAADF image and S (↑), Fe and Ni maps (→) of a thin section of a CP IDP (1-2 nm spatial resolution).