NEW INTEGRATED ANALYTICAL CAPABILITY AT THE UNIVERSITY OF HAWAI‘I.

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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 (C₃₄) 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 µm 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 ~10 µm 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 ¹⁵N 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...
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 inter-
preted 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 prepara-
tion 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 Sys-
tem, 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.