Analytical Laboratories for Astromaterials Research

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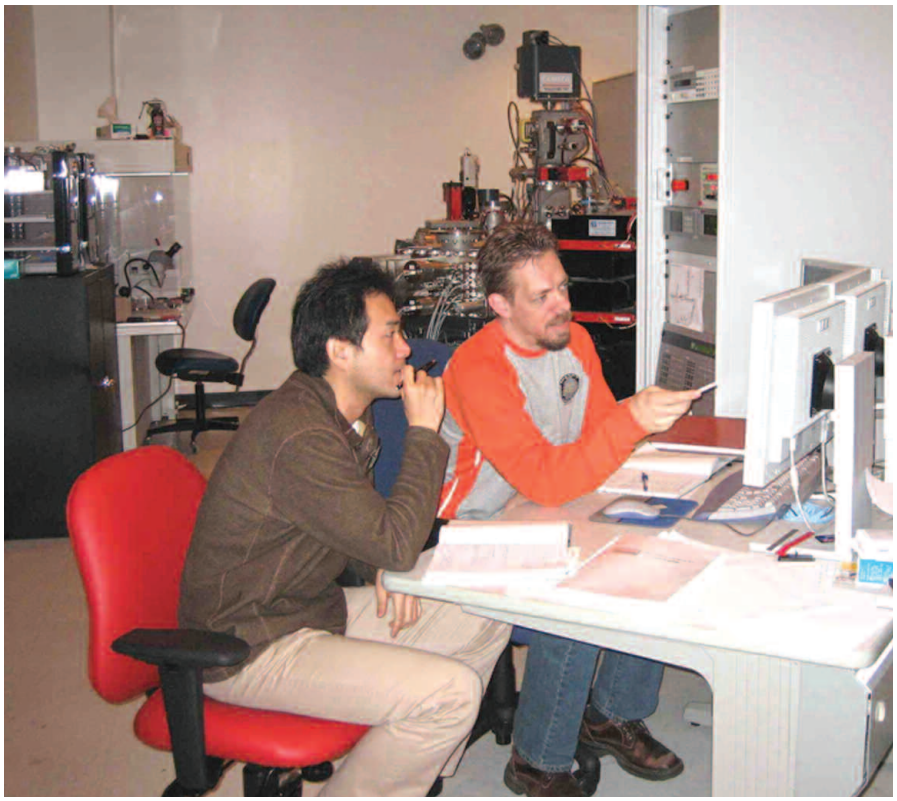
In the past few years, we have expended significant effort in upgrading our facilities for Astromaterials research. The building that houses most of these facilities is over 30 years old and has undergone minimal preventive maintenance during the previous decade. Recently, basic infrastructure obsolescence actions have included: installing a new liquid nitrogen tank and associated gas plumbing; connecting all air handling systems to the center-wide operations control; renovating the basic wet chemistry laboratory; and replacing the antiquated carbon monoxide monitoring system in the experimental petrology laboratory.

A major effort to expand the Image Science and Analysis Laboratory began after the *Columbia* accident in 2003, resulting in the tools necessary to perform our expanded role in shuttle imagery analysis and damage inspections.



Scott Messenger (right) and Motoo Ito in ARES NanoSIMS Laboratory, Building 31.

In preparation for the return of the Stardust mission in January 2006, we completed a processing and curation laboratory. In addition, new laboratories to support operation of the new NanoSIMS and scanning transmission electron microscope (STEM) in time for Stardust sample analysis was a highlight for the staff (see figure).

The laboratories and associated suite of instruments installed to support the Stardust sample return mission are particularly unique. These various instruments have pushed Astromaterials Research and Exploration (ARES) capabilities to new levels.

- The JEOL 2500 STEM has been optimized for quantitative chemical imaging and is capable of directly visualizing major and minor element distributions at the nm-scale. The new STEM is equipped with a field-emission source, a 50-mm^2 active area energy-dispersive X-ray (EDX) spectrometer, a Gatan Tridiem Imaging Filter for energy-filtered imaging and electron energy-loss spectroscopy, and a high-angle annular darkfield detector for z-contrast imaging of samples. We have demonstrated the remarkable analytical capabilities of the new STEM with the first *quantitative* X-ray maps (spectrum images) of interplanetary dust and lunar soil grains showing the nm-scale distribution of major and minor elements. The spectrum images contain a high count-rate EDX spectrum *at each pixel* in the image, enabling us to determine quantitative mineralogy along with major and minor element chemistry in the submicrometer constituent grains in interplanetary dust

particles and in Stardust samples. The new STEM combines the power of a conventional transmission electron microscope for high-resolution imaging, crystallographic measurements, and microstructure analysis with the ability to rapidly obtain quantitative compositional maps for elements heavier than helium.

- The NanoSIMS 50L builds on the strengths of the first-generation NanoSIMS and was designed with the goals of improving the reproducibility of isotopic measurements and increasing the information that can be obtained from the smallest samples. It was designed for our specific needs, installed in a new laboratory, and passed acceptance in February 2006. Two new mobile detectors were added, bringing the total to six mobile and one fixed detectors (either electron multipliers or faraday cups). The turning radius of the magnet is larger than that of the original NanoSIMS (650 mm vs. 550 mm), and each detector has been augmented with a 60-deg cylindrical electrostatic deflector. Together, these modifications enable simultaneous detection of adjacent mass lines up to 60 amu over a mass range of 21. The instrument is also equipped with a dedicated nuclear magnetic resonance probe, for use with low magnetic field analyses, that has benefited H-isotopic imaging. These features now enable simultaneous measurements of multiple isotopic systems, thus maximizing the scientific information that can be obtained from the smallest samples. Owing to the fine grain size and heterogeneous nature of Stardust samples, we encountered many situations where a valuable sample could only be subjected to a single measurement. The expansion of the multicollection system to seven detectors has, therefore, already benefited our analyses of Stardust samples during preliminary examination.
- The *ultra*-L^2 mass spectrometer instrument is a one-of-a-kind, two-step laser desorption laser ionization mass spectrometer designed for the sub-femtomole detection of organic matter at high spatial resolution. A spatially focused infrared laser pulse (λ 10.6 or 1.064 μm) is used to non-thermally desorb material from a sample surface, and the laser fluence is carefully controlled so that desorbed material is liberated from the sample substrate exclusively as neutral species. A second ultraviolet (UV) laser pulse is directed through the desorbed neutrals photoionizing specific molecules. Ionized species generated by the photoionization process are injected into a reflectron time-of-flight mass spectrometer. Under appropriate laser wavelength (λ 212, 266 or 355 nm), fluence (10^9-10^{15} $W \cdot cm^{-2}$) and pulse duration (10^{-9}-10^{-12} s) of the UV laser pulse, ionization occurs with negligible fragmentation. The types of organic species that can now be analyzed have been expanded dramatically by the development of an ultrafast, picosecond laser ionization system that acts as an efficient, universal, soft ionization source for many types of molecular species.

In summation, ARES' state-of-the-art facilities are critical to support our research activities. For this reason, we intend to continue the current obsolescence planning and facility renovations on a regular schedule to maintain the strong analytical capability that keeps our research efforts successful.