THE KUIPER MATERIALS IMAGING AND CHARACTERIZATION FACILITY AT THE UNIVERSITY OF ARIZONA: A NEW LABORATORY FOR THE COORDINATED ANALYSIS OF PLANETARY MATERIALS AND SAMPLES TO BE RETURNED BY HAYABUSA 2 AND OSIRIS-REx.

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Introduction: The primary objective of the Hayabusa 2 and OSIRIS-REx Missions is the return and analysis of pristine carbonaceous regolith to study the nature and history of asteroids Ryugu and Bennu [1,2]. Successful completion of that objective requires laboratory techniques capable of providing information on composition and structure of the sample over a range of spatial scales. Such information includes chemical composition, isotopic composition, spatial relationships, texture, microstructure, crystal chemistry, crystal structure, and local atomic order. Here we describe a new laboratory facility for electron and ion microscopy at the Lunar and Planetary Laboratory (LPL), University of Arizona (UA), newly constructed and motivated by the need for state-of-the-art instrumentation in support of planetary-materials research programs and sample-return missions.

The Kuiper Materials Imaging and Characterization Facility (KMICF) is located in the sub-basement of the Gerard P. Kuiper building for Space Sciences, which was constructed in 1966 with funds provided by NASA. Located approximately 30 ft. below ground and slab-on-grade, the basement was renovated and repurposed to house electron and ion microscopes. The KMICF serves the research needs of planetary-science investigations and is open to the UA community, including regional private- and public-sector users, as well as other NASA planetary-science investigators. For planetary science, materials investigated at KMICF include but are not limited to: meteorites, lunar samples, interplanetary dust particles, micrometeorites, samples returned from the first Hayabusa Mission, circumstellar dust grains from ancient stars, organic compounds in meteorites, chondritic sulfides, and refractory inclusions in meteorites, *e.g,* [3-7].

The costs of facility operation are shared among LPL, the UA College of Science, and the UA Office of the Vice President for Research. The KMICF is staffed by professional scientists who manage daily operations and train users. The Scientific and Technical Oversight Committee, composed of faculty users, provides oversight and scientific direction. Additional information can be found at https://kmicf.lpl.arizona.edu.

Layout and Instrumentation: The facility consists of parallel instrument bays and includes space for sample preparation, meeting and workshops, and offices for laboratory scientists and visitors (Fig. 1). A utility corridor was constructed behind several labs to house service equipment, *e.g.,* roughing pumps, chillers, and uninterruptible power supplies for the instruments. We describe the various laboratories below.

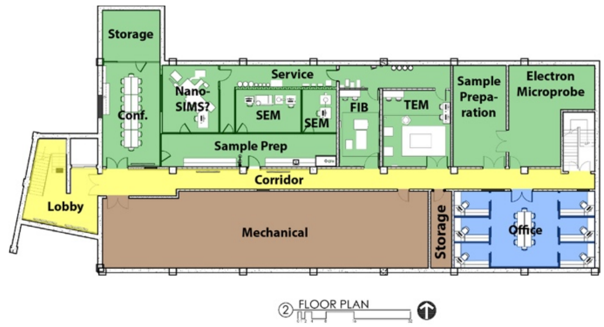

Figure 1. Floor plan for KMICF.

The *Scanning Electron Microscope* (SEM) suite currently houses two microscopes as well as a room designed for a next-generation nanoscale secondary ion mass spectrometer (NanoSIMS). The Hitachi S-4800 cold-field emission gun (cold FEG) can operate between 0.5 keV and 30 keV. It is equipped with a Thermo-Noran Si(Li) energy dispersive X-ray spectrometer (EDS) running Noran SystemSix (NSS) software. The Hitachi S-3400 is a W thermal emitter with a variable-pressure chamber. It is equipped with a Renishaw InVia confocal Raman system (514 nm and 785 nm lasers) and automated stage for mapping and spectral scans as well as a Structural and Chemical Analyser (SCA) interface and Reflex microscope that can be run stand alone. It is further equipped with secondary-electron and backscattered-electron detectors and a Thermo-Noran SDD EDS system operating NSS software. The SEM suite also has laboratory benchtop space for sample-preparation, including a broad-beam Ar-ion mill and plasma cleaner for TEM samples, and includes a chemical fume hood. See Fig. 2a,b.

The *focused-ion-beam scanning electron microscope (FIB-SEM)* lab houses a ThermoScientific Helios NanoLab 660 G^3. The Helios is equipped with an Elstar electron gun and monochromator, and is capable of electron beam resolution down to 0.6 nm from 15 kV to 2 kV. Its Tomahawk Ga^+ ion column can be operated between 65 nA and 500 V for, respectively, removal of large volumes of material and final sample polishing. Under standard operating conditions, an ion beam resolution of 2.5 nm at 30 kV is achievable. The Helios is equipped with *in situ* micromanipulation for creation and transfer of lamellae for transmission electron

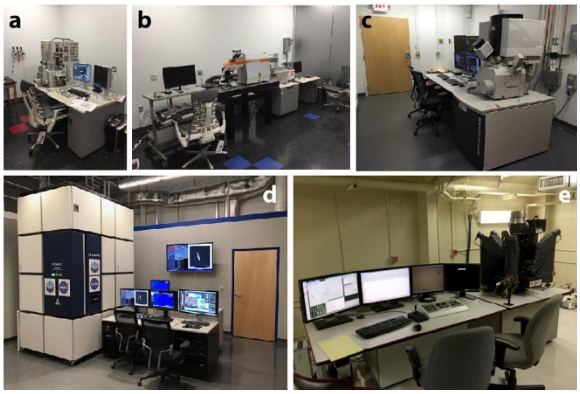

Figure 2. Photographs of KMICF laboratories. (a) Hitachi 4800 SEM. (b) Hitachi 3400 SEM. (c) ThermoScientific Helios FIB-SEM. (d) Hitachi HF5000 S/TEM. (e) Cameca SX-100 microprobe.

microscope (TEM) analysis. It is also equipped with an EDAX EDS system and electron backscatter diffraction (EBSD) analysis system for compositional and crystallographic analysis in two and three dimensions. Furthermore, FIB tomography via slice-and-view software is available and can be coupled with EDS and EBSD for 3-D volume reconstructions. Multiple polygons are supported for device patterning as well as the ability to directly import customized shapes for patterning or deposition. The Helios is equipped with C and Pt gas-injection systems. See Fig. 2c.

The *Transmission Electron Microscope (TEM)* lab houses a newly installed Hitachi HF5000. The HF5000 is equipped with a cold FEG and optical alignments at 200 keV and 60 keV. It is equipped with a Hitachi 3rd-order spherical aberration corrector for the scanning TEM (STEM) probe. Spectroscopic capabilities include: (1) Oxford Instruments twin silicon-drift detectors (SDD) for EDS providing a total solid angle approaching 2.0 sr; and (2) a Gatan Quantum Imaging Filter (GIF) for electron energy-loss spectroscopy (EELS). The HF5000 is capable of $\leq$0.4 eV energy resolution for EELS under full emission and 78 pm point-to-point resolution in STEM mode for atomic-resolution imaging. The HF5000 is also equipped with a full array of sample holders including single- and double-tilt analytical (low background) for EDS analysis as well has a heating holder (Hitachi Blaze) for in situ thermal studies inside the TEM [3]. See Fig. 2d and Fig. 3a-d.

The *Electron Microprobe (EMPA)* lab houses Cameca SX-50 and Cameca SX-100 Ultra instruments. The SX-50 was installed in 1991 and has been in continuous service for 26 years. It is equipped with a 30 keV W thermal emission gun, four wavelength dispersive X-ray spectrometers (WDS), 12 diffracting crystals, and a Princeton-Gamma Tech Si(Li) EDS system, allowing analysis of elements with Z$\geq$4. The SX-100 Ultra was installed in 2011 and has been in service for nearly eight years. It is equipped with a 30 keV LaB_6 filament, five WDS, 14 diffracting crystals, and an SDD-EDS system, allowing analysis of elements with Z$\geq$5. See Fig. 2e.

References: [1] Lauretta D.S. et al. (2017) *Space Science Reviews* 212:925-984. [2] Watanabe et al. (2017) *Space Science Reviews* 208:3-16. [3] Thompson M. et al. (2017) *Meteoritics & Planet. Sci.* 52, 413-427. [4] Haenecour P. et al. (2019) *Nature Astronomy* 3, 626-630. [5] Zega T.J. et al. (2019) *LPS L* Abstract #2127. [6] Seifert L. B. et al. (2019) *LPS L* Abstract #2585. [7] Ramprasad T. et al. (2019) *LPS L* Abstract #2129.

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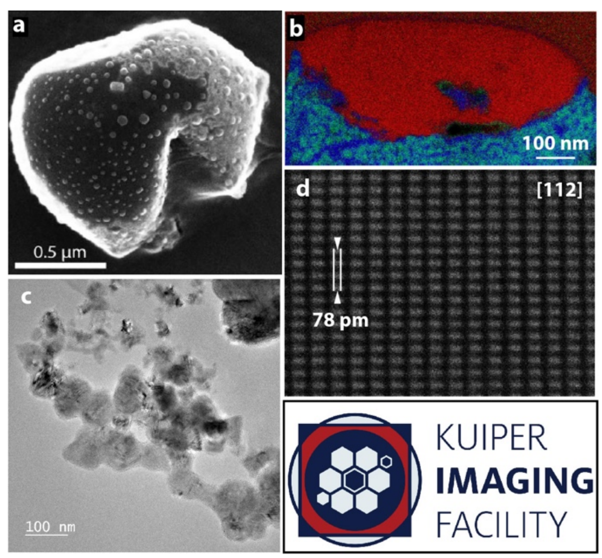

Figure 3. Example data acquired using KMICF instrumentation. (a) Secondary electron image of lunar soil grain heated in situ in the TEM using the Hitachi Blaze heating holder from [3]. (b) False-color EDS map of a presolar graphite spherule from a CO Nova from [4]. (c) Bright-field TEM image of synthetic 3C SiC subjected to in situ heating and irradiation from [5]. (d) Atomic-resolution HAADF image of Si showing 78 pm spacing between columns of Si atoms in the [112] orientation.