Planetary Science Capabilities at National Synchtrotron Light Source-II, Brookhaven National Laboratory

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Introduction: Synchrotron-based structural and chemical (micro)-analysis is a well-established, non-destructive tool used to study a wide range of materials, including planetary materials, meteorites and interplanetary dust particles [1]. X-ray absorbance techniques provide information on the chemical speciation of the sample, while diffraction can be used to resolve the mineralogical composition of a sample. Focusing of the x-ray beam allows for spatially resolved chemical and structural information. The National Synchrotron Light Source-II (NSLS-II) at Brookhaven National Laboratory (BNL), a Department of Energy user facility, is one of the newest and most advanced synchrotron facilities in the world. NSLS-II, providing world-leading capabilities for X-ray imaging and high-resolution energy analysis, enables chemical speciation and diffraction studies of planetary materials at nanoscale resolution.

Specific capabilities: NSLS-II is a state-of-the-art, medium-energy electron storage ring (3 billion electron-volts) designed to deliver world-leading intensity and brightness. The new facility currently operates seven beam lines, twenty-one are under various stages of construction, and there is a capacity for a 60 to 70 beamlines in total (https://www.bnl.gov/ps/). One of the discriminating features of NSLS-II is the ability to take up to seven beamtimes out of the ring building. Out of the seven operational beamlines, two are well-suited for the study of planetary materials. These are: the Hard X-ray Nanoprobe (HXN) beamline and the Sub-micron Resolution X-ray (SRX) beamline .

HXN provides x-ray imaging capabilities with a world-leading spatial resolution. It offers a suite of x-ray analytical tools for structural, elemental and chemical imaging, with an initial resolution of 15 nm and an ultimate goal of sub 5 nm resolution.

SRX provides world-leading, high-throughput spectroscopic imaging capability (2D and 3D) with sub-100 nm spatial resolution and millisecond dwell times/pixel with fly-scan capability. Simultaneous x-ray fluorescence and transmission measurements can be collected at sub-µm to sub-100 nm spatial resolution with an incident x-ray beam energy of 4.65-25 keV. Sample stages enable fast 2D scanning and tomography capabilities. X-ray fluorescence (XRF) imaging and tomography provides elemental mapping in 2D and 3D, respectively. Spatially resolved X-ray Absorption Near Edge Structure (XANES) spectroscopy can be performed in fluorescence or transmission mode. Full-field imaging and tomography are also possible to provide morphology information. Both, SRX and HXN, are working on implementing coherent diffraction imaging and ptychography capabilities into their beamline portfolio. These techniques enable even higher spatial resolutions than achievable right now.

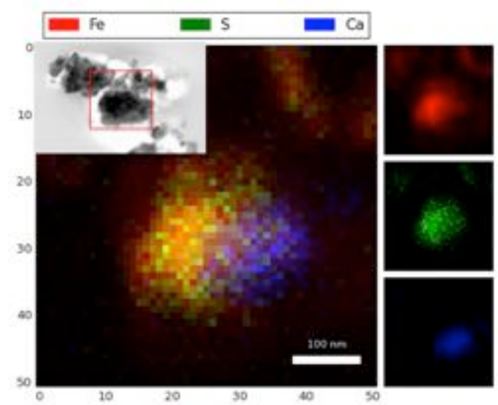

Figure 1. Elemental map of GEMS particle collected at HXN beamline, NSLS-II. Stepsize of 10 nm (Flynn, irick, Keller unpublished work) .

Examples of Planetary Science applications:

Trace elements in Glassy Embedded Metals and Sulfides (GEMS). GEMS are small, < 1 micron, amorphous, SiO_2-containing objects found in both Interplanetary Dust Particles (IDPs) and comet Wild 2 particles. There is uncertainty over the origin of GEMS, since their infrared spectra match interstellar silicate, but their S content is consistent with that of Solar System grains. A way to determine the origin of GEMS is to compare the trace metals concentrations in GEMS to solar concentrations reported by Lodders [2]. Until the advent of 3rd generation synchrotron sources trace metal concentrations could not be obtained from GEMS due to the lack of sufficient spatial resolution and insufficient flux to enable the collection of trace metals from very small objects. GEMS vary in size but are mostly smaller than 1 micron. Using the HXN beam line with a spatial resolution on the order of 20-30 nanometers we were able to obtain maps and some trace metal concentrations on GEMS. This data is preliminary and more work needs to be done to obtain a larger suite of trace metals before any conclusive statement can be made on the origin of GEMS; however, this study illustrates the capabilities of HXN in support of planetary sciences.

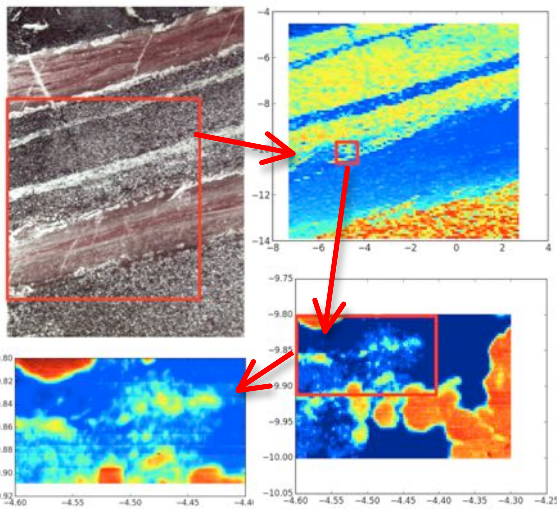

Figure 2. XRF imaging of banded iron formation. Light microscopic image 16 x 12 mm^2 (top left), XRF images 1 x 1cm^2 step size 100 µm (top right), 300 x 200 µm^2, step size 3 µm (bottom right), and 200 x 100 µm^2, step size 1 µm. The red squares indicate the respective scan areas (Schoonen and Thieme, unpublished).

Oxidation state of Iron in Banded Iron Formation (BIF). BIF's are sedimentary rocks characterized by thick sequences of thin alternating iron oxide-rich and silica-rich layers. BIF deposits are prevalent around 2.4 billion years ago when free molecular oxygen became available on Earth. The conditions and reaction mechanism that led to BIF formation are, however, not fully resolved. One of the key questions is what the oxidation state of the iron was at the time of deposition. The dominant iron oxide minerals in BIF's (hematite, Fe_2O_3; and magnetite, Fe_3O_4) are typically micron to submicron in size. The SRX beamline was used to study a BIF sample and map the distribution of Fe(II) and Fe(III) at submicron spatial resolution. The capability to resolving iron mineralogy at the submicron-level would also be of great importance in future studies of samples returned from Mars.

Future capabilities: Under development at NSLS-II are several other beamlines that could be of interest to planetary scientists. For example, the X-ray Fluorescence Microprobe (XFM) can be used to map P to Tc in heterogenous samples with a resolution of 1 to 10 micron. The Tender Energy Spectroscopy (TES) beamline is capable of mapping biogenic elements, such as Ca, Si, and P at micron-level spatial resolution. One of the unique future capabilities at NSLS is a beamline designed to study batteries in operando. This requires passing the x-ray beam through metal containments and studying processes within the battery while being charged or discharged. With this capability it might be possible to study samples returned from Mars without removal from their sample container.

With about half of the beamlines at NSLS-II still to be developed, there is also the possibility of designing new beamlines optimized for planetary studies, including the capability to build a satellite building associated with NSLS-II suitable to meet planetary protection requirements, security requirements, and combine synchrotron-based techniques with other analytical techniques.

References:

[1].Sutton, S.R., et al., *Microfluorescence and microtomography analyses of heterogeneous Earth and environmental materials*, in *Applications of synchrotron radiation in low-temperature geochemistry and environmental science*, P.A. Fenter, et al., Editors. 2002, The Mineralogical Society of America: Washington, D.C. p. 429-483. [2]. Lodders, K., *Solar system abundances and condensation temperatures of the elements.* Astrophysical Journal, 2003. **591**: p. 1220-1247.