Objectives:

This funding is to develop a new Synchrotron user facility for microbeam X-ray absorption spectroscopy (XAS) and quantitative X-ray fluorescence (XRF) imaging, at the National Synchrotron Light Source (NSLS) and NSLS-II. It includes design, purchase of components, and construction of the microprobe endstation and controls. Initial development, commissioning, and application is ongoing at NSLS Beamline X15B, with planned transition in 2014-15 to the NSLS-II TES (Tender-Energy Spatially Resolved X-ray Absorption Spectroscopy) beamline. It is optimized for the “tender” energy range of 1-5 keV, reaching up to 8 keV. Thus it uniquely covers the K absorption edges of critical elements Mg, Al, Si, P, S, Cl, and Ca, and can reach up to Co.

A stable, high-flux microbeam focus, user-tunable from ~50 to ~5 microns, has been achieved using two-stage achromatic focusing. Existing beamline optics collimate, monochromate, and macro-focus the X-ray beam to ~1 mm at a secondary source aperture (SSA). Beam from the SSA is then re-focused by a pair of mirrors in KB geometry to the microbeam scale. Size of the microbeam is tunable, at the expense of flux, by adjusting the size of the SSA as a virtual source.

The new experimental endstation consists of 1) a sample chamber operable as a radiation enclosure with helium atmosphere to facilitate measurements in this energy range, 2) the KB microfocusing optics, 3) a sample-positioning stage for raster-scanning and positioning the sample, 4) X-ray fluorescence detectors, an existing Ge detector for low-signal sensitivity and a new Si detector for high count rates, 5) an optical camera for viewing samples and locating target locations, 6) beam intensity monitors and diagnostics, and 7) controls and data acquisition system. An important aspect of this project is the added capability for fast, on-the-fly scanning of the monochromator (energy), required for fast XAS and advanced XAS imaging. This instrument will be available for initial PI and Co-I measurements, and for General Users to apply for beamtime to use, at NSLS X15B for the remaining operation of NSLS, to September 30, 2014. Phase two of this project will transition this facility, both the primary optics and the endstation, to NSLS-II after NSLS ceases operation. While this transition itself is beyond the scope of the current grant, preparation and planning for it is included.

Summary of project progress.

This project is ongoing, but the core funded activity is essentially complete, and is continuing via shared funding by NSF and NASA coupled with development by NSLS-II of the TES beamline. Core components are all in place and are being commissioned; additional components and enhancements continue to be incorporated. The current status is summarized here.

1) The new sample chamber has been designed, fabricated by a local vendor, installed, connected to NSLS safety interlocks, inspected and approved by the NSLS review committee, and aligned to the X-ray beam. This meets the shielding requirements for both NSLS and NSLS-II, being a composite aluminum-lead-copper construction. It is ~1 x 0.5 x 0.5 m in size and is outfitted to accommodate glove-box function to maintain helium atmosphere.

2) The primary microfocusing optics, the KB mirror system, has been designed in collaboration with the vendor (IDT) and Peter Eng (U. Chicago, GSECARS), fabricated, delivered, connected to the new control system, bench-tested, installed in the experimental chamber, aligned with beam, live-tested, and applied to a variety of test and user experiments.

3) Sample-positioning stages have been purchased, connected to controls, bench-tested, assembled to an XYZ geometry at 45 degrees to the beam, and installed. This provides a scanning area of 100 x 100 mm with resolution of 0.3 microns. A sample holding frame has been fabricated and installed. The system has been tested for step- and on-the-fly scanning modes.
4) The existing Ge X-ray fluorescence detector, for low-signal sensitivity, has been installed in the new experimental chamber and tested with the new data acquisition hardware. A new Si detector, for high count rates to work in tandem with the Ge detector, has been custom-designed with the vendor (Hitachi), installed, and commissioned.
5) The optical microscope camera system has been designed, the parts purchased, and the system constructed and implemented in the new chamber and aligned to the sample stage.
6) Beam intensity monitors and diagnostics have been installed.
7) A new control and data acquisition system has been developed in collaboration with NSLS (Ryan Tappero), NSLS-II (Jun Ma), and GSECARS (Steve Sutton). This included system design, the purchase, modification and integration of new components, and extensive testing. This new system is a prototype NSLS-II system utilizing NSLS-II selected standard hardware components, so that it will be fully supported by NSLS-II after transition there. However, as a prototype implementation this required substantial time and effort, resulting in some delay of the overall project. Successful testing of this system is reported below. The beta-version data acquisition software has been written and implemented: we now have a functional system to collect XRF image data in X26A/X27A (GSECARS) format, that includes both step- and on-the-fly scan modes. It also collects step and fly scans of energy for quick XANES (~15 seconds per scan) and EXAFS (~1 minute per scan), and can do XANES imaging (energy fly-scan at each pixel of a line or 2D map).

Fast on-the-fly energy scanning has been tested on the existing X15B monochromator with excellent results. Such fast scanning, within certain speed ranges, has been demonstrated to have vibrational stability comparable to step-scanning while saving as much as 2 seconds per data point of step-scanning overhead. This test was critical to prove the mechanical suitability of the X15B monochomator for transition to NSLS-II. Initial testing of the integrated data-acquisition hardware and beta-version software has included use by General Users.

Initial commissioning activities have identified several enhancements and adjustments that will improve operation. These continue to be implemented.

This facility will transition to the TES Beamline of the new NSLS-II facility following shutdown of NSLS September 30, 2014. Therefore, extensive planning effort has been devoted to development of the TES beamline within the NSLS-II NxtGen Project. PI Northrup is now the Lead Scientist for the TES Beamline in addition to his position as Research Scientist at Stony Brook University. The transition plan to NSLS-II, including detailed cost and schedule for the TES beamline, will be further defined.