MICROCAL & I0_UP

IRR Functional Description

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1 INTRODUCTION

Primary Research Capabilities

The National Institute of Standards and Technology (NIST) and the Department of Energy (DOE) have more than 30 years of ongoing partnership at the NSLS and NSLS-II developing advanced synchrotron measurement methods and delivering excellence in material science impacting important societal challenges in energy, health, environment, national security, and improvement of quality of life. This partnership continues to promote innovation and enhance US industrial competiveness for inorganic and organic semiconductors, photovoltaics, self-assembled-monolayers, biological and environmental materials, batteries, catalysts, fuel cells, polymers, superconductors, thermoelectrics, ferroelectrics, and ferromagnets.

Currently, the NIST Synchrotron Science Group operates a suite of three beamlines at NSLS-II: the beamline for materials measurement (BMM) at 6-BM, and the spectroscopy soft and tender beamlines (SST-1 and SST-2) at 7-ID; studying materials that span the entire periodic table. The microcalorimeter (MICROCAL) is an X-ray fluorescence spectroscopy tool located on SST-1. It was previously installed and operated at the NIST U7A beamline at NSLS.

MICROCAL performs chemical spectroscopy of low concentration and radiation-sensitive samples and devises. It uses a transition-edge sensor microcalorimeter to perform high-throughput, highly efficient and high-resolution UHV X-ray Fluorescence Spectroscopy for low concentration samples. It also performs X-ray absorption fine structure spectroscopy utilizing the sample drain current especially for radiation-sensitive samples and devices.

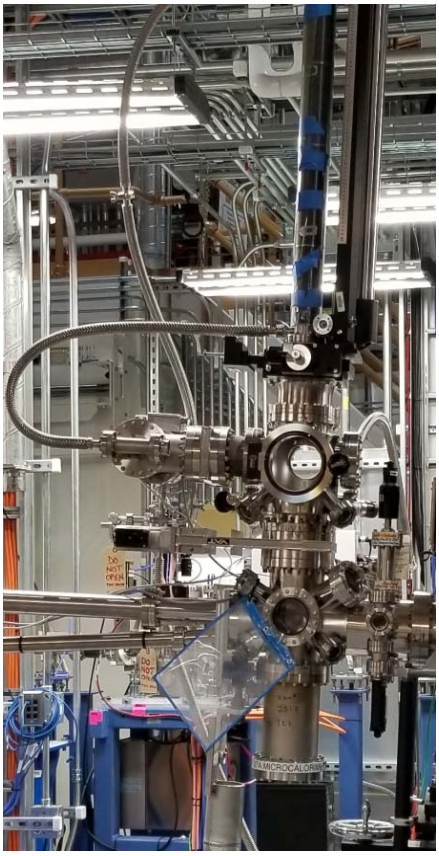

Figure 1: Picture of the MICROCAL station as installed at SST-1.

Beamline Staff

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2 EXPERIMENTAL STATION DESIGN AND COMPONENTS

2.1 Location on Beamline

The MICROCAL experimental station is a through-station located upstream of the NEXAFS experimental station on SST-1. A layout diagram of the NIST beamline suite at NSLS-II is shown in Figure 2. An upstream photon shutter, which serves to isolate MICROCAL from the rest of the beamline, is located just downstream of RSoXS.

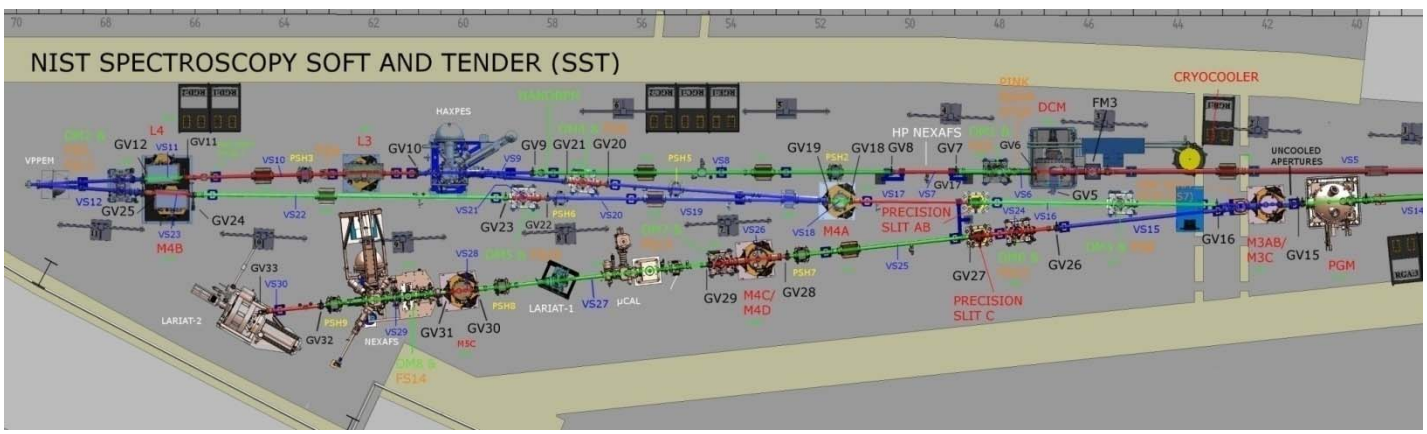

Figure 2: Layout of the NIST SST BL. MICROCAL is located downstream of PSH7/RSoXS.

2.2 Experimental Station Components

The MICROCAL through-station consists of a stainless steel vacuum chamber contiguous with the LARIAT1 chamber. Figure 3 shows a picture of the MICROCAL experimental station, I0_UP including the plain dithering mirror, M5W. The main vacuum chamber consists of a stainless steel vessel with number of ports. An 8-inch port is located at the top of the main vessel unto which an isolation gate valve and a sample loadlock chamber are attached. The loadlock is equipped with a motorized sample manipulator which has X, Y and Z translation stages as well as θ stage (for rotation about the vertical axis.) An 8-inch conflat flange is located upstream of the chamber to allow entry of beam for experiment in this chamber or downstream including the LARIAT1, which uses a downward deflected and dithered beam. The chamber has two exit ports for the through-beam - one downward 2.75-inch conflat for the LARIAT1 and another, 4⅝- inch conflat, for the rest of the downstream stations. Perpendicular to the axis of the beam is an 8-inch conflat port for a gate valve and the microcalorimeter detector. Other ports of various sizes with blanks or viewports can be found on the chamber.
The manipulator is equipped with electrical feedthru, which is used to collect sample drain current. The sample drain current provides the means to perform X-ray absorption spectroscopy measurements on samples.

Samples are mounted in a loadlock chamber and positioned into the main vacuum chamber for experiments using the motorized sample manipulator. The loadlock is equipped with another gate valve and a turbo pump (Loadlock Turbo). The two turbo pumps are used to maintain the vacuum in the chamber below 10^{-7} Torr.

Associated with the MICROCAL, is a diagnostic section called the I0_UP section. This section is equipped with an array of meshes of elements whose K- or L-edges fall in the range 280 ~ 1100 eV. The mesh drain current is set up to provide X-ray absorption structure spectroscopy and are used for calibrating relevant edges during an experiment downstream. This section is also equipped with a phosphor screen to visualize the beam, a pair of photodiodes to measure the intensity of the beam, two pairs of horizontal and vertical slits for trimming the beam to a desired size. In addition, a gold-coated plain mirror, M5W, for dithering and deflecting the beam down to the LARIAT1 sample position; a rotatable stainless steel mirror mask and a gold mesh for beam normalization are located just downstream of this section.

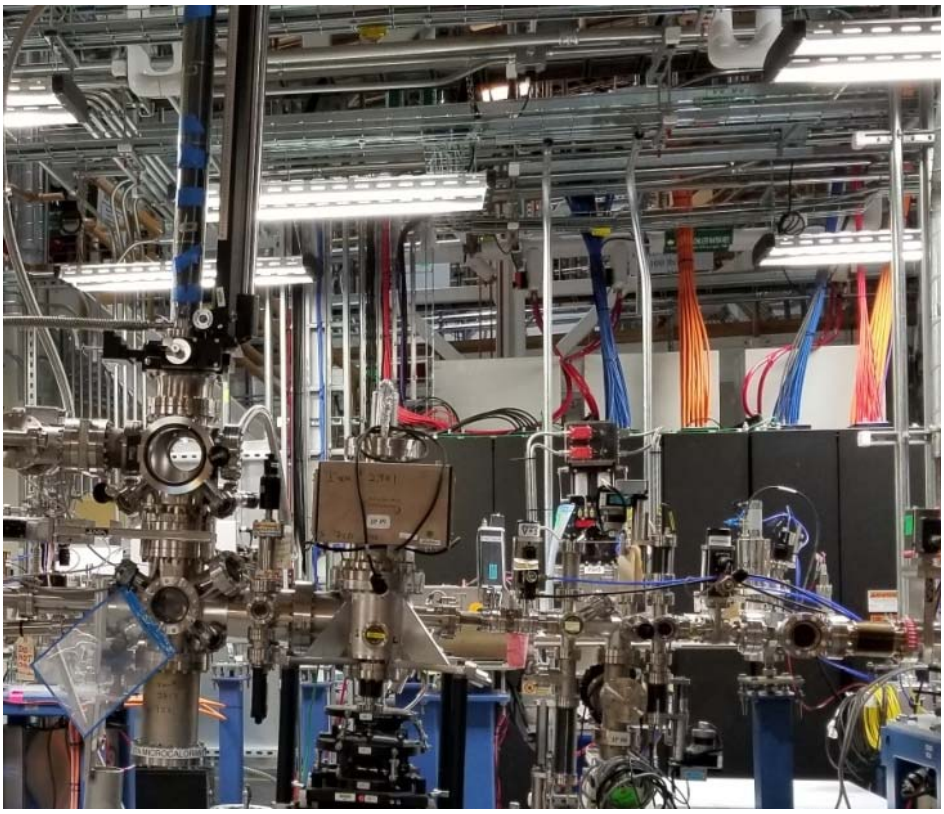

Figure 3: A picture of MICROCAL with I0_UP and M5W as installed at SST1

3 EXPERIMENTAL STATION SAFETY

3.1 Radiation Shielding

The experimental station radiation shielding design is based on the calculations presented in NSLS-II Tech Note #275. These calculations show that with incident soft monochromatic X-ray beam, 1 mm of stainless steel equivalent is sufficient to reduce radiation external to chamber to below 0.05 mrem/hr and as low as reasonably achievable. The minimum chamber thickness is 3 mm stainless steel. If the sample is moved away from the beam, the beam propagates downstream to the beam termination point/beamstop located on LARIAT-2 chamber or to either of the photon shutters PSH8 and PSH9.

3.2 Radiation Safety Components

The MICROCAL through-station has vacuum switches mounted on the main chamber and an associated photon shutter (PSH7) located upstream of the chamber. These act as radiation safety components and were previously reviewed as part of the 07-ID SST Beamline Instrument Readiness Review.

3.3 Configuration Control

All radiation safety components are under configuration control, in accordance with the NSLS-II Radiation Safety Component Configuration Management procedure (NSLSII-ESH-PRC-030). These are located in Sections C and D of the radiation safety component checklist and are listed on the checklist as items 7-ID1-PSH-07, 7-ID1-VVS-03 and 7-ID1-SW-11.

3.3 Personnel Protection System (PPS)

The PPS system monitors the vacuum switches on the chamber, which are interlocked with the upstream photon shutter, PSH7. In the event of a vacuum loss, PSH7 will close immediately.

3.4 Hazard Identification and Mitigation

The MICROCAL poses no unique hazards from the other experimental stations, which are already in operation at SST and NSLS-II. A USI evaluation has been conducted and it was determined that the anticipated activities at the beamline do not violate the existing SAD and ASE. All relevant NSLS-II procedures and safety practices were followed during the design and construction of the beamline to mitigate or control the hazards identified in these documents.

4 INSTRUMENT READINESS

4.1 Survey and Alignment

MICROCAL is installed according to the specifications and final designs. Installations of the components are verified and documented by the NSLS-II Survey group, working closely with the beamline staff.

4.2 Utilities

The following services/capabilities have been deployed for MICROCAL:

- *Electrical power distribution system,* which is unchanged from the beamline IRR.
- *Network connectivity,* which is part of the existing beamline network for motor control and data acquisition.
- *Cabling support structures,* which are in place for all the utilities.

4.3 Vacuum System and Pressure Safety

The vacuum pressure for MICROCAL is to be maintained at less than 10^{-7} Torr. MICROCAL is contiguous with LARIAT-1 and shares vacuum with the rest of the SST-1 beamline.

Venting of vacuum chambers will be performed following the NSLS-II vacuum venting procedure.

4.4 Controls

All motorized components have been tested by beamline staff and documented. The individual motors are accessible using the MICROCAL instrument software and will be integrated into standard EPICS commands.