Final Technical Report

Canted Undulator Upgrade for GeoSoilEnviroCARS Sector 13 at the Advanced Photon Source

Key Personnel: Stephen Sutton (PI), Peter Eng, Mark Rivers, Matthew Newville

DOE-BES-SISGR: Grant DE-SC0002580; ER16092 (Sept. 15, 2009 - Sept. 14, 2011)
NSF-EAR-IF-ARRA: Grant EAR-0841622 (Aug. 1, 2009 - July 31, 2012)
NASA-SRLIDAP (LARS): Grant NNX09AP65G (July 21, 2009 - July 20, 2012)

University of Chicago, 5801 S. Ellis Avenue, Chicago, IL 60637

Submitted February 2013

Contents

1. Executive Summary

Support for the beamline component of the canted undulator upgrade of Sector 13 (GeoSoilEnviroCARS; managed and operated by the University of Chicago) at the Advanced Photon Source (APS; Argonne National Laboratory) was received from three agencies (equally divided): NASA-SRLIDAP (now LARS), NSF-EAR-IF (ARRA) and DOE-Single Investigator Small Group (SISGR). The associated accelerator components (undulators, canted front end) were provided by the APS using DOE-ARRA funding.

The intellectual merit of the research enabled by the upgrade lies in advancing our knowledge of the composition, structure and properties of earth materials; the processes they control; and the processes that produce them. The upgrade will facilitate scientific advances in the following areas: high pressure mineral physics and chemistry, non-crystalline and nano-crystalline materials at high pressure, chemistry of hydrothermal fluids, reactions at mineral-water interfaces, biogeochemistry, oxidation states of magmas, flow dynamics of fluids and solids, and cosmochemistry. The upgrade, allowing the microprobe to operate 100% of the time and the high pressure and surface scattering and spectroscopy instruments to receive beam time increases, will facilitate much more efficient use of the substantial investment in these instruments. The broad scientific community will benefit by the increase in the number of scientists who conduct cutting-edge research at GSECARS.

The user program in stations 13ID-C (interface scattering) and 13ID-D (laser heated diamond anvil cell and large volume press) recommenced in June 2012. The operation of the 13ID-E microprobe station began in the Fall 2012 cycle (Oct.-Dec 2012).

The upgraded canted beamlines double the amount of undulator beam time at Sector 13 and provide new capabilities including extended operations of the X-ray microprobe down to the sulfur K edge and enhanced brightness at high energy. The availability of the upgraded beamlines will advance the research being conducted at Sector 13.

2. Scientific and Programmatic Impact

GSECARS beam lines have been designed for maximum flexibility to allow virtually all X-ray-based analytical techniques of utility to earth, environmental and planetary scientists to be implemented. As a result, a wide range of research is being undertaken covering topics including the chemistry, nature, and dynamics of Earth's core and mantle; processes at work in the Earth's crust that control the transport and bio-availability of toxic species; and the evolution of our solar system revealed through the study of extraterrestrial materials. The majority of this research can be divided into the following categories: (A) High Pressure Mineral Physics and Chemistry, (B) Non-crystalline and Nano-crystalline Materials at High Pressure, (C) Chemistry of Hydrothermal Fluids, (D) Reactions at Mineral-Water Interfaces, (E) Biogeochemistry, (F) Flow Dynamics of Fluids and Solids, (G) Oxidation States of Magmas, and (H) Cosmochemical Studies of Extraterrestrial Materials. The three agencies that have supported this upgrade are aligned with these topics. The National Science Foundation – Earth Sciences Division has deep earth, high pressure studies (A and B) as a major emphasis. The Department of Energy – Geosciences program is focused on near surface processes (C, D, E, and F). And the National Aeronautics and Space Administration – SRLIDAP and Cosmochemistry programs are concerned with unraveling the histories of celestial bodies through studies of extraterrestrial materials (A, G, and H).

The upgrade addresses priorities documented in the DOE-BES report "Basic Research Needs for Geosciences: Facilitating 21st Century Energy Systems", specifically, the priority research directions of Mineral-Water Interface Complexity and Dynamics, Nanoparticulate and Colloid Chemistry and Physics, and Biogeochemistry in Extreme Subsurface Environments. This project also addresses the DOE-BES mission to serve the research needs of the scientific community by facilitating access to cutting edge research instrumentation at their major research facilities.

The upgrade addresses specific priorities in the National Science Foundation's "Investing in America's Future: Strategic Plan FY2006-2011". The project fosters research that improves our ability to live sustainably on Earth. It provides enabling research infrastructure and supports the next generation of large research facilities. It contributes to the development of new capabilities, technologies, and instrumentation that could lead to the establishment of next-generation facilities. And, the project leverages other community, industry, federal agency and international investments in research, education and infrastructure, broadening participation in science and engineering.

The upgrade addresses NASA's Strategic Goals and Research Objectives to understand the Sun and its effects on Earth and the solar system and advance scientific knowledge of the origin and history of the solar system. Specific NASA Research Objectives that will be addressed include understand the fundamental physical processes of the space environment from the Sun to Earth, to other planets, and beyond to the interstellar medium, and learn how the Sun's family of planets and minor bodies originated and evolved. This facility will advance the analysis of samples returned by Stardust and Genesis, and will have future applicability for the analysis of other extraterrestrial samples, including particles collected by future Earth orbit collectors, stratospheric IDPs, and future NASA sample return missions to Mars, asteroids and comets.

Science in these areas will be advanced in three ways. First, a unique sub-micron microprobe in a dedicated enclosure allows detailed speciation research on light and heavy elements in systems of geochemical, environmental and cosmochemical significance. Second, an optimized high energy insertion device advances the quality of surface and high pressure diffraction data. Third, the doubling of undulator beam time opens up the capabilities of GSECARS to many more investigators than previously possible. In the context of the APS Strategic Plan, the upgrade addresses four of the scientific opportunities identified: environmental science, inelastic scattering, high energy and X-ray imaging. In addition, we provide new capabilities, particular in terms of the intermediate energy, sub-micron microprobe, for the General User Program.

This upgrade project supported a variety of scientific and technical positions involving the design, procurement, installation and commissioning of the various instruments.

3. Upgrade Accomplishments

The pre-upgrade sector consisted of an insertion device (undulator) beamline (13ID), a bending magnet beamline (13BM), and two support laboratories. Each beamline had four enclosures: A first optics enclosure (13ID-A and 13BM-A), a second optics enclosure (13ID-B and 13BM-B), and two experimental stations (13ID-C, 13ID-D, 13BM-C, and 13BM-D). The single 13ID X-ray source was an undulator A used to supply 4 to 40 keV monochromatic X-rays, or 4 to ~120 keV white beam to our experiments.

The upgrade plan (Figure 1) involved addition of a second undulator, replacement of the magnetic array in the existing undulator, dividing an existing experimental enclosure into two independent enclosures, and addition and modification of beamline components. The two undulators have been installed in a canted geometry to provide two independent undulator sources. The existing 13ID-C enclosure was partitioned into two enclosures by adding a dividing wall, door, and pass-through shielded beam pipe. X-rays are provided to this new enclosure (13ID-E) by installing the second undulator, two horizontal deflecting mirrors, a monochromator and two shutters. The existing 13ID-C/D monochromator was modified to allow the new canted beam to pass outboard of the first crystal, and the large KB mirror pair was relocated. The new configuration provides X-radiation from the new medium-energy undulator (2.3 to 28 keV) to the X-ray microprobe, which will also be able to perform non-laser heated diamond anvil cell (DAC) spectroscopy and diffraction experiments. The downstream undulator supplies both doubly-focused monochromatic X-radiation from 5.6-65 keV and white beam to the surface diffractometer, the laser-heated DAC, and the multi-anvil press. These three instruments each receive an increase in beam time of ~33% and the microprobe operates continuously, doubling the total beam time available on the undulator beamline.

The upgrade tasks included the following:

- Undulator 1 (design, fabricate, install): 2.2 m, 2.3-28 keV
- Undulator 2: (design, fabricate, install): 2.2 m, 5.5-65 keV
- Front end (design, fabricate, install): canted ID components
- Bendable deflecting mirrors (2) (design, fabricate, install): silicon, 590 mm
- Monochromator for 13ID-E line (design, fabricate, install): Si(111) and Si(311) double crystal.
- Monochromator for 13ID-C/D line: modify for improved performance, add Si(311) crystal, and to pass 13ID-E beam
- Shutters for 13ID-E and 13ID-C/D (design, fabricate, install)
- Station 13ID-C modifications: install interior wall, install new door for 13ID-E
- Beamline KB mirrors (13ID-C/D): move to new locations in 13ID-B and 13ID-C
- Shielded pipe in 13ID-E (design, fabricate, install)
- Personnel Safety System (PSS) and Equipment Protection System (EPS) modifications

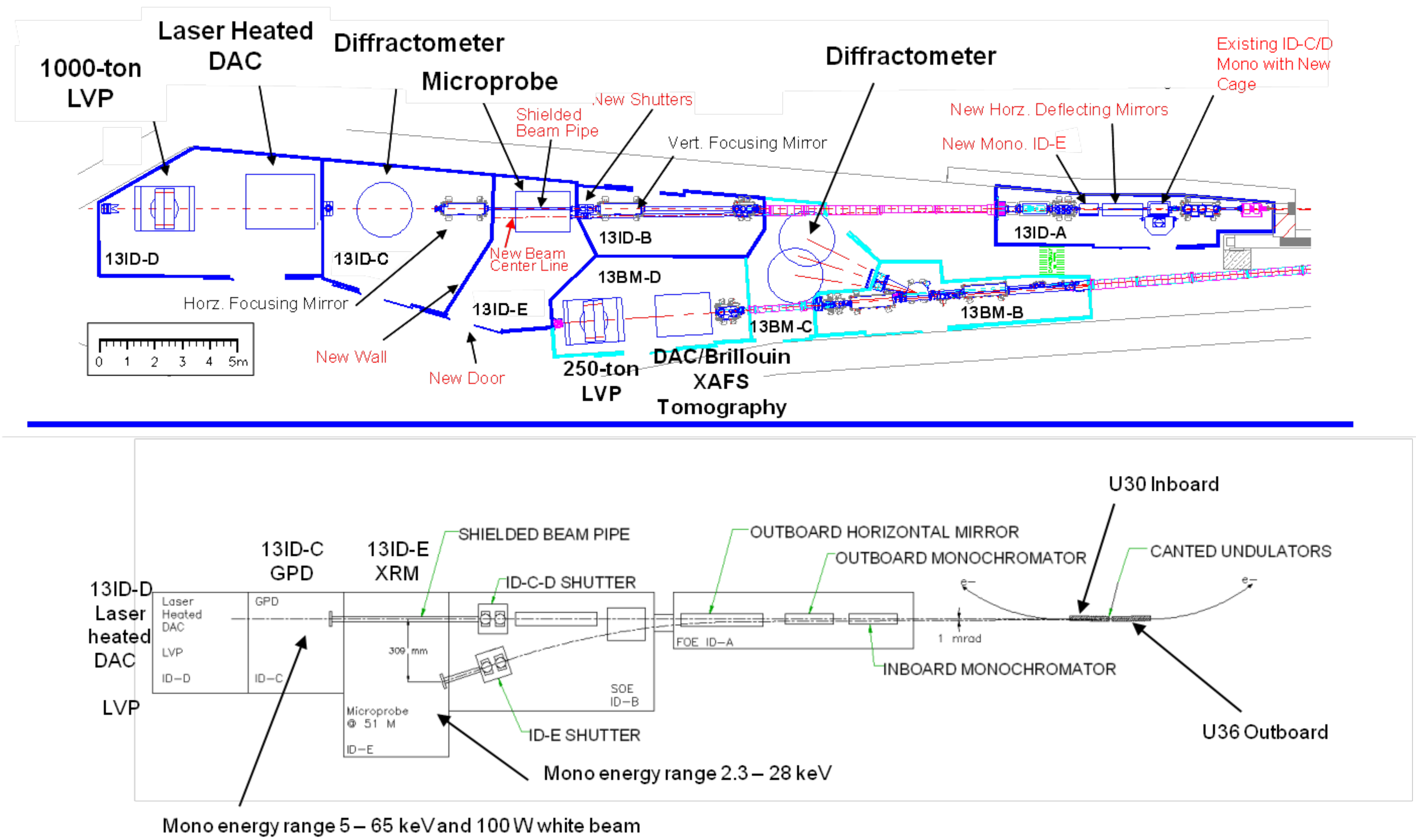

Figure 1: Canted undulator upgrade schematics including sector layout (top) and X-ray optics configuration (bottom, not to scale).

4. Upgrade Components

The three federal awards and ARRA contributions from the APS have been used to procure/install the following major components (relevant stations and funding breakdown shown in parentheses). The major components are described in the sections below.

Component	Experimental Station(s)	Funding Source (%)			
		NSF	DOE	NASA	APS
Undulator (upstream)	E				100
Undulator (downstream)	C/D				100
Canted Front End	C/D and E				100
Experimental Station Subdivision	C/D and E	100			
White Beam Slits/Tank Upgrade	C/D		50	50	
Monochromator Upgrade	C/D			100	
Monochromator	E		39	61	
Horizontal Mirrors	E	20	80		
Secondary Source Aperture/WBS	E		86	14	
Beam Position Monitors	C/D and E		86	14	
Oxford Cryo-cooler	E			100	
Power Limiting Aperture	C/D			100	
Shutters	C/D and E	100			
Shielded Vacuuum Pipes	C/D and E	100			
KB Microfocusing Mirror System	E	80*		20	

* GSECARS operations grant

4.1 Undulators and Front End

The APS allocated DOE-ARRA funds to provide two new undulators and the canted undulator front end (masks and shutters for the two beams). The two undulators reside in the 5 m straight section in the accelerator with their generated photon beams canted by 1 mrad (Figure 2). Figure 3 shows the calculated brilliance of the previous GSECARS 33mm period Undulator A, the new 3.6 cm period undulator for the dedicated microprobe (ID-E) beamline, and the 3.0 cm period undulator for the surface scattering (ID-C) and high-pressure (ID-D) beamline. Note that the 3.6 cm period undulator extends to lower energy than Undulator A (2.3 keV) and the 3.0 cm period undulator has twice the brilliance of Undulator A at the high energies used in the large-volume press. The canted undulator front end is designed to handle 20 kW of power (at eventual 200 mA operations) and deliver the two independent photon beams to the experimental stations.

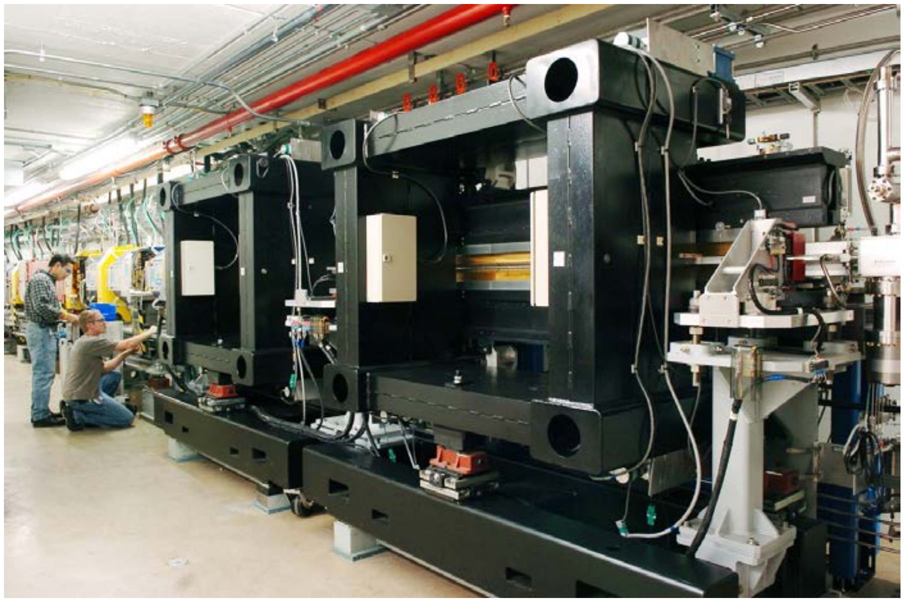

Figure 2: A canted, two undulator arrangement in the APS storage ring similar to that in the upgraded Sector 13. The downstream (right) device together with beamline optics provides X-rays in the 5-5-65 keV range to stations 13ID-C (interface scattering) or 13ID-D (laser-heated diamond anvil cell and large volume press. The photon beam from the upstream (left) device is canted outboard by 1 mrad and together with beamline optics provides X-rays in the 2.3-28 keV range for the X-ray microprobe in 13ID-E.

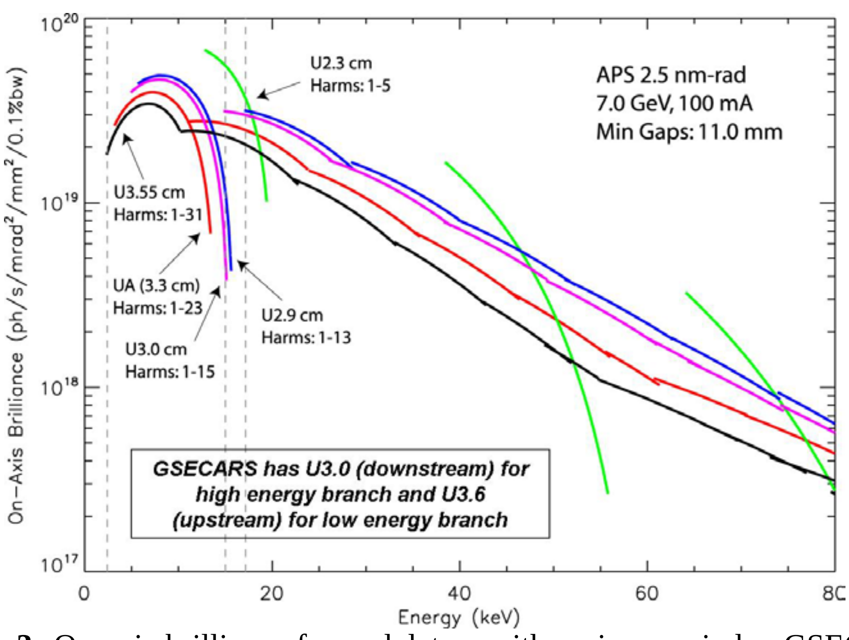

Figure 3: On-axis brilliance for undulators with various periods. GSECARS has U3.0 (purple) installed in the downstream position to supply the high energy branch (13ID-C/D) and U3.6 (similar to U3.55, black) in the upstream position to supply the lower energy branch (13ID-E).

4.2 Experimental Station Subdivision

Modifications were made to the 13ID-C station to divide it into two separate stations (Figure 4). 13ID-E is the new dedicated X-ray microprobe station and 13ID-C continues to house the large Newport diffractometer. A shielded wall now divides 13ID-C and 13ID-E and a new door for 13ID-E was installed. The upstream shielded wall in 13ID-E was rebuilt to allow two beam pipes to enter this station and to provide the required shielding so that personnel can be in this station when there is beam in the 13ID-B optics enclosure. A shielded transport pipe was installed in 13ID-E to allow the beam from the downstream undulator to be transported down to 13ID-C or D when personnel are in 13ID-E. The Newport diffractometer was moved to the downstream end of 13ID-C to allow the horizontal focusing mirror to be installed upstream of it. The Personnel Safety System was modified to allow the new door in 13ID-E to be used to access this station.

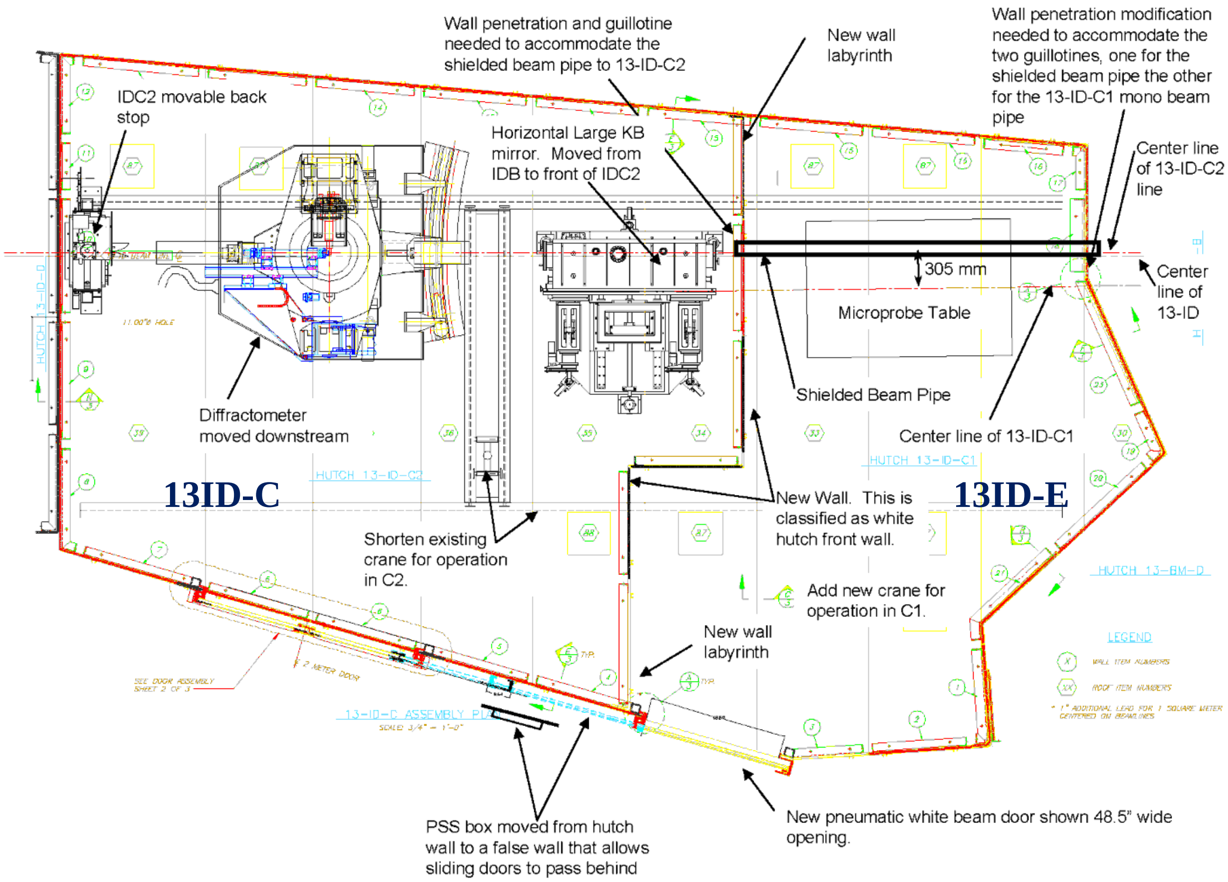

Figure 4: Final configuration of the 13ID-C and 13ID-E stations.

4.3 White Beam Slits and Vacuum Tank Upgrade (13ID-C/D and E)

The existing primary white beam slits were upgraded to allow canted beam operation. This involved retrofitting of slit blade geometry to prevent beam interference, fabrication of a second slit assembly for the second undulator beam and fabrication of a custom vacuum tank to accommodate both slit assemblies in as little space along beam as possible (Figure 5). The existing tank stand was reused. This slit tank was outfitted with both slit assemblies (Figure 3) and installed in the 13ID-A first optics enclosure in May 2012.

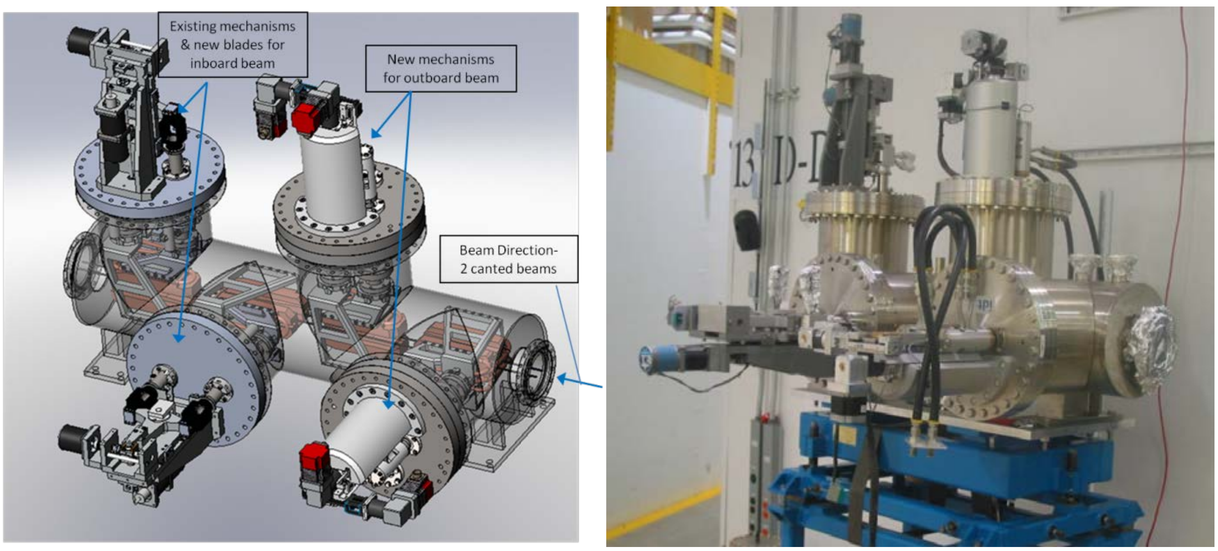

Figure 5: GSECARS Canted Primary White Beam Slit Assembly drawing (left) and photograph of the fully-assembled instrument (right) with horizontal and vertical slits for both canted undulator beamlines.

4.4 Double Crystal Monochromator Upgrade (13ID-C/D)

The undulator beamline monochromator, which we have used for nearly two decades, is a cryogenically cooled, double-crystal Si(111) operating with a fixed height exit beam. This monochromator is continuing to serve 13ID-C/D and has been modified to eliminate previous issues with the second crystal performance (poor cooling and carriage non-linearity) which have caused complicating beam motions (Figure 6). The second crystal was made much longer, so that for a single EXAFS scan there is no required along-beam motion of the second crystal (Figure 7). The second crystal is now being better cooled for improved temperature stability. A Si (311) crystal was added to increase the high-energy limit and to improve resolution at low-energy. The entire monochromator is translated about 1 cm to change between the two Si crystals. The vertical translation has been replaced with one that has the required straightness and is encoded to allow quantitative motions. The crystal cage assembly has also been modified to allow the 13ID-E beam to pass through it. The upgraded monochromator is now in operation (Figure 8).

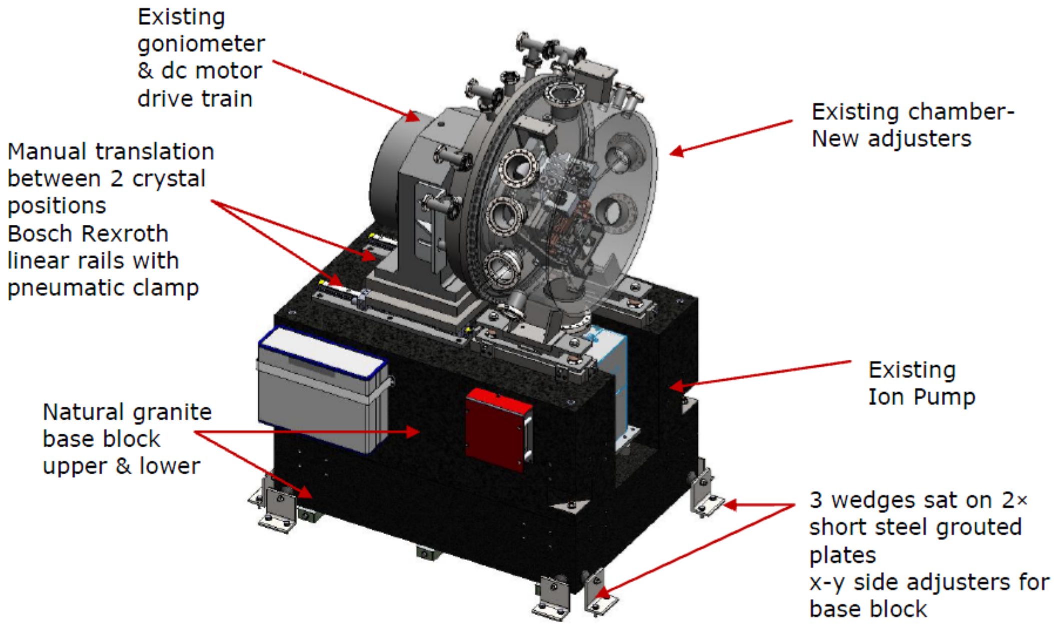

Figure 6: Overall 13ID-C/D Monochromator Assembly drawing.

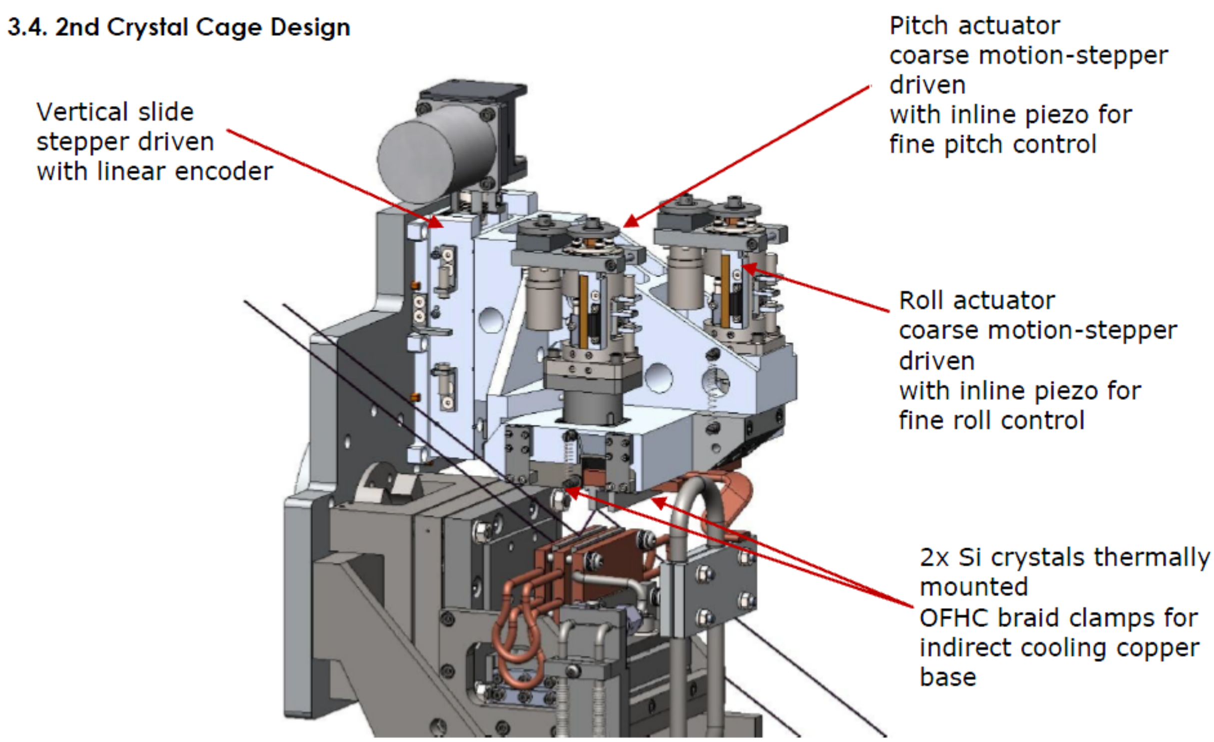

Figure 7: Double crystal monochromator 2nd crystal cage with pitch & roll linear coarse stepper actuators, in line pitch & roll linear fine piezo actuators and strain gauge.

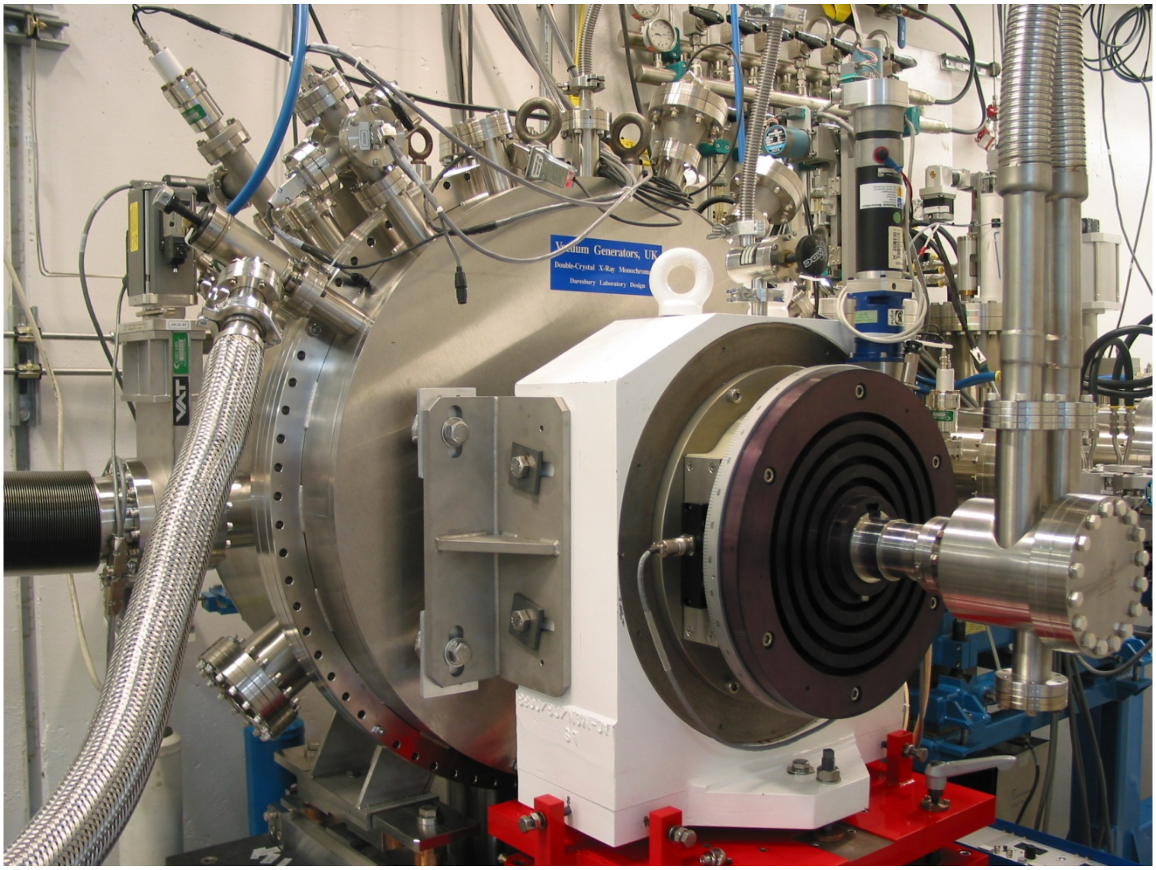

Figure 8: Fully assembled and installed 13ID-C/D upgraded double crystal monochromator

4.5 Double Crystal Monochromator (13ID-E)

Located just upstream of the deflecting mirror assembly is a new monochromator (Figure 9 through Figure 12) for the 13ID-E microprobe station, designed to allow the 13ID-C/D monochromatic and/or white beam to pass inboard (Figure 11). This new monochromator houses Si(111) and Si(311) crystal pairs with a translation capability between the two. It has fixed offset, bounces the beam up by 25 mm and is optimized to operate between 2.3 to 28 keV. A state-of-the-art air-bearing turntable is employed for highly stable rotations along with encoder systems using latest products and technologies.

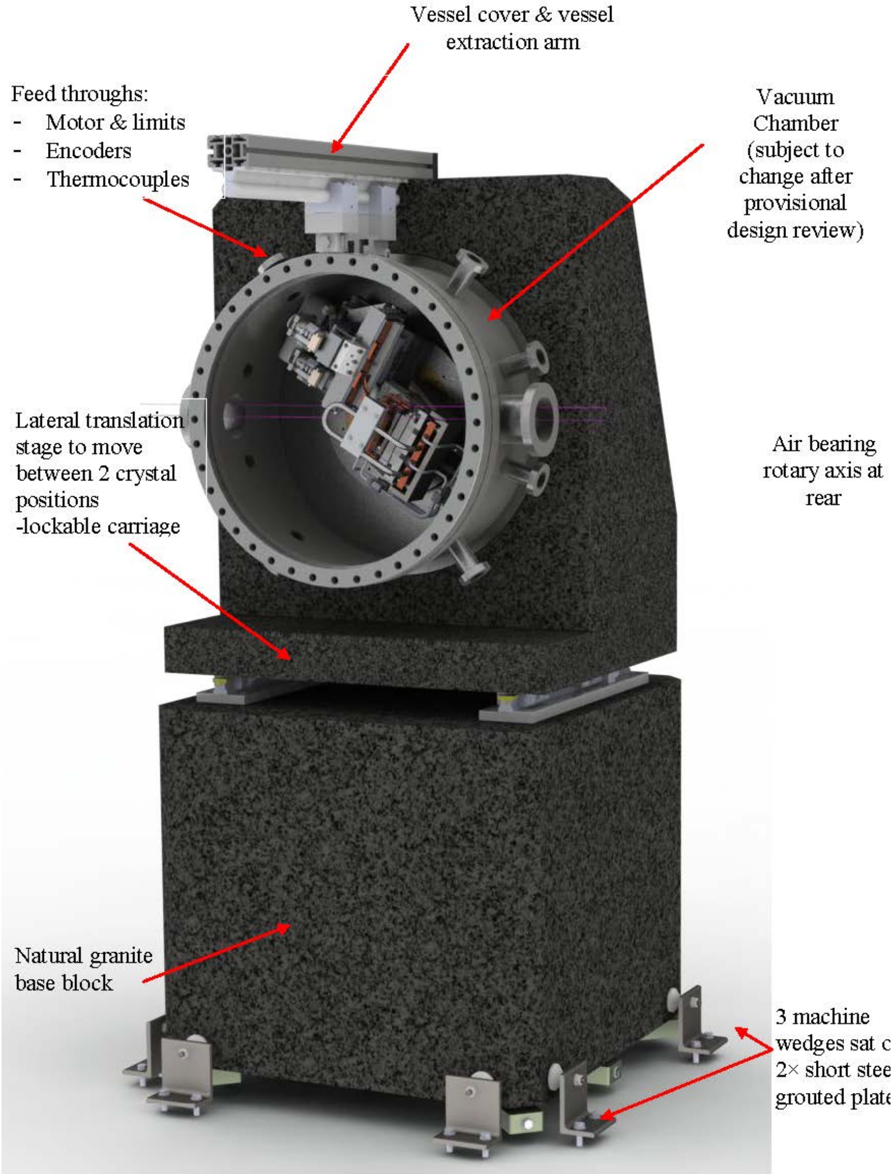

Figure 9: Schematic of the 13ID-E monochromator employing an air-bearing rotary assembly for angular adjustment.

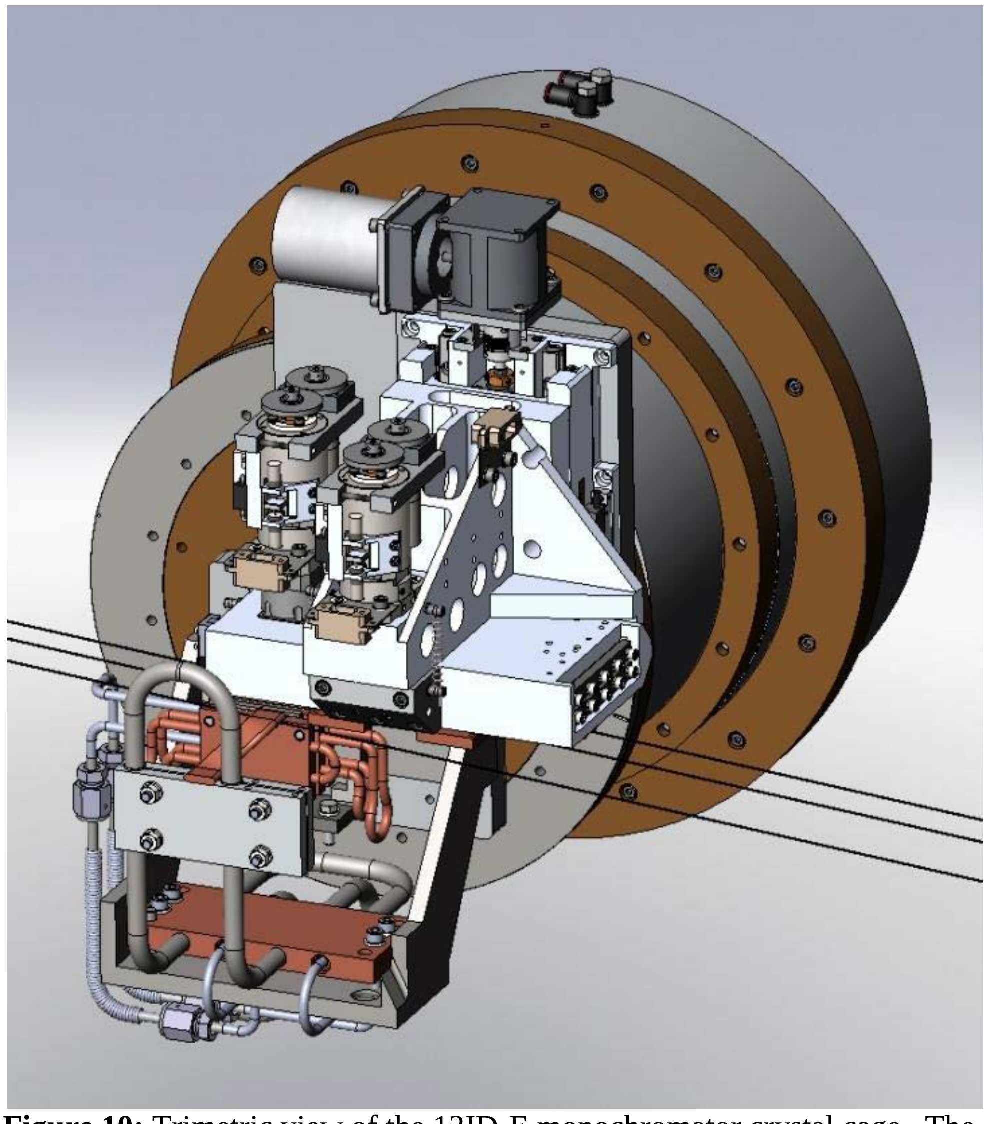

Figure 10: Trimetric view of the 13ID-E monochromator crystal cage. The rotation stage is set in this drawing for 28 keV monochromatic beam operations.

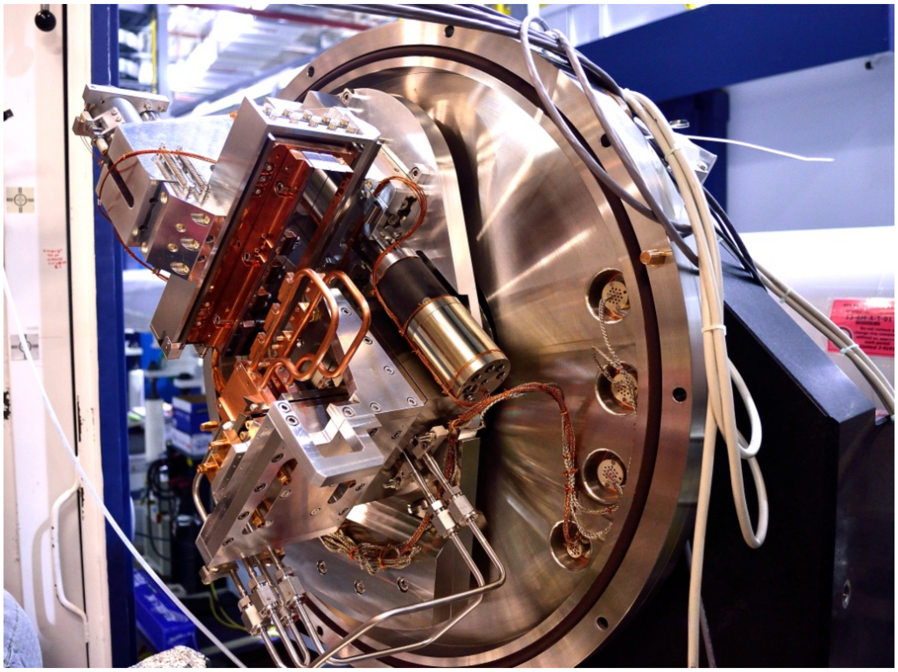

Figure 11: Photograph of the assembled 13ID-E crystal cage mounted to the lid of the monochromator vessel.

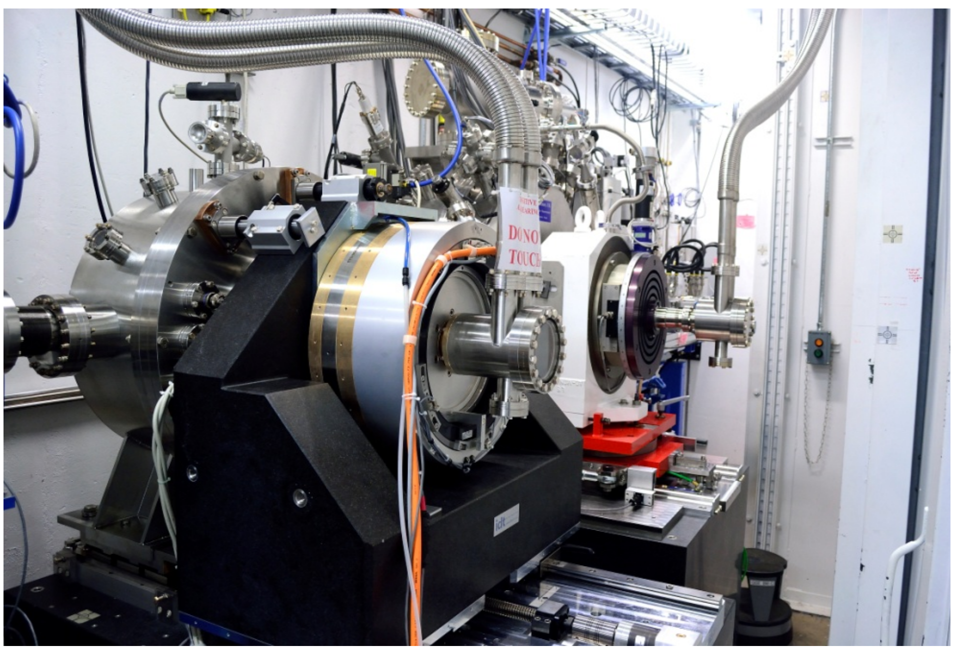

Figure 12: Upstream view in optics enclosure 13ID-A showing the installed 13ID-E (foreground) and 13ID-C/D (background) monochromators.

4.6 Double Horizontal Focusing Mirror System (13ID-E)

This system (Figure 13) is required to provide sufficient separation between the two undulator beams to allow them to be used independently. The beam from the 13ID-E monochromator strikes a 590 mm long flat horizontally deflecting mirror located at 30.5 m and operated at a fixed angle of 3 mrad (coated with Pt, Rh, and bare Si stripes and having a maximum cutoff energy of approximately 28 keV) bouncing the beam outboard by an additional 6 mrad. A second bendable horizontally deflecting mirror is located immediately after this

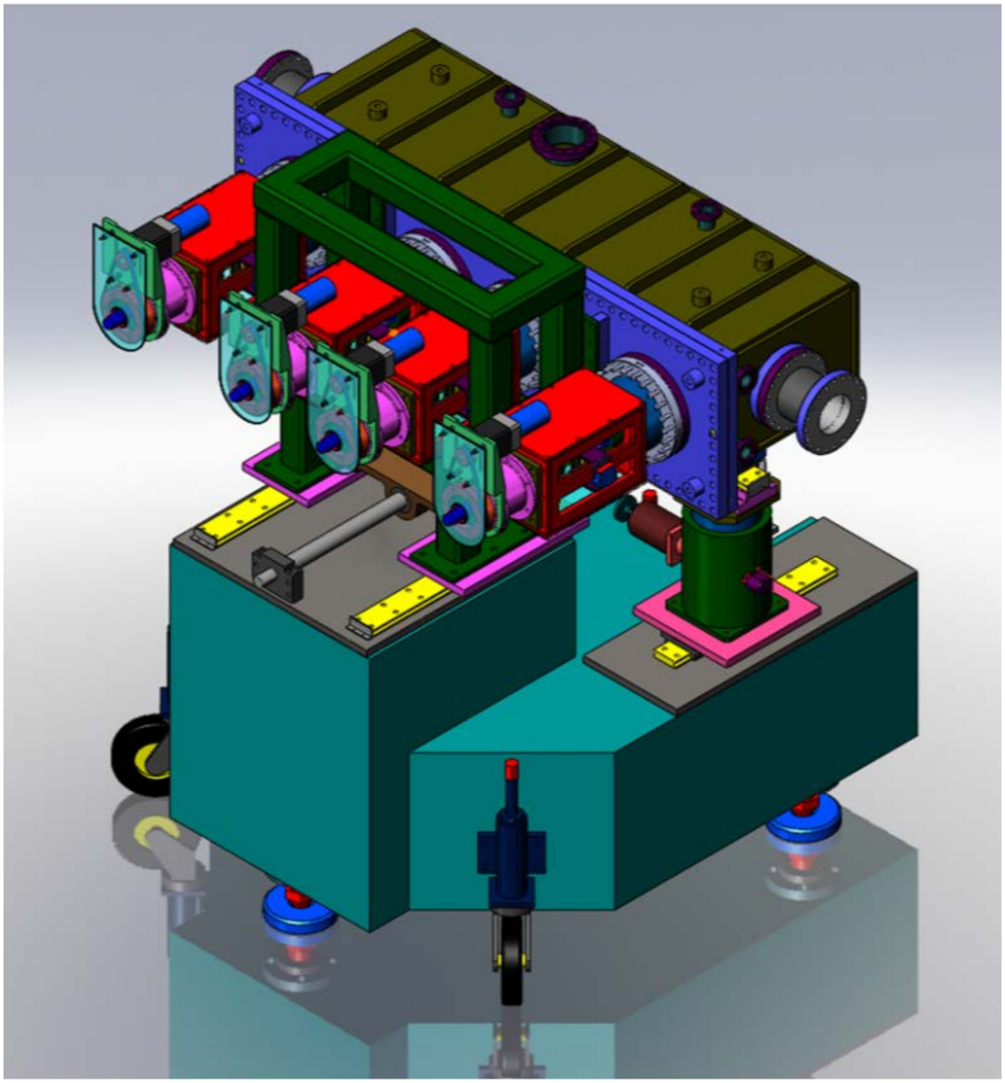

Figure 13: 3D engineering design drawing of the Double Horizontal Focusing Mirror System for the 13ID-E microprobe beamline.

mirror deflecting the beam an additional 6 mrad for a total of 12 mrad outboard deflection. This mirror combination collects and reflects more than 75% of the undulator beam below 30 keV. This mirror system has been installed in station 13ID-A (Figure 14).

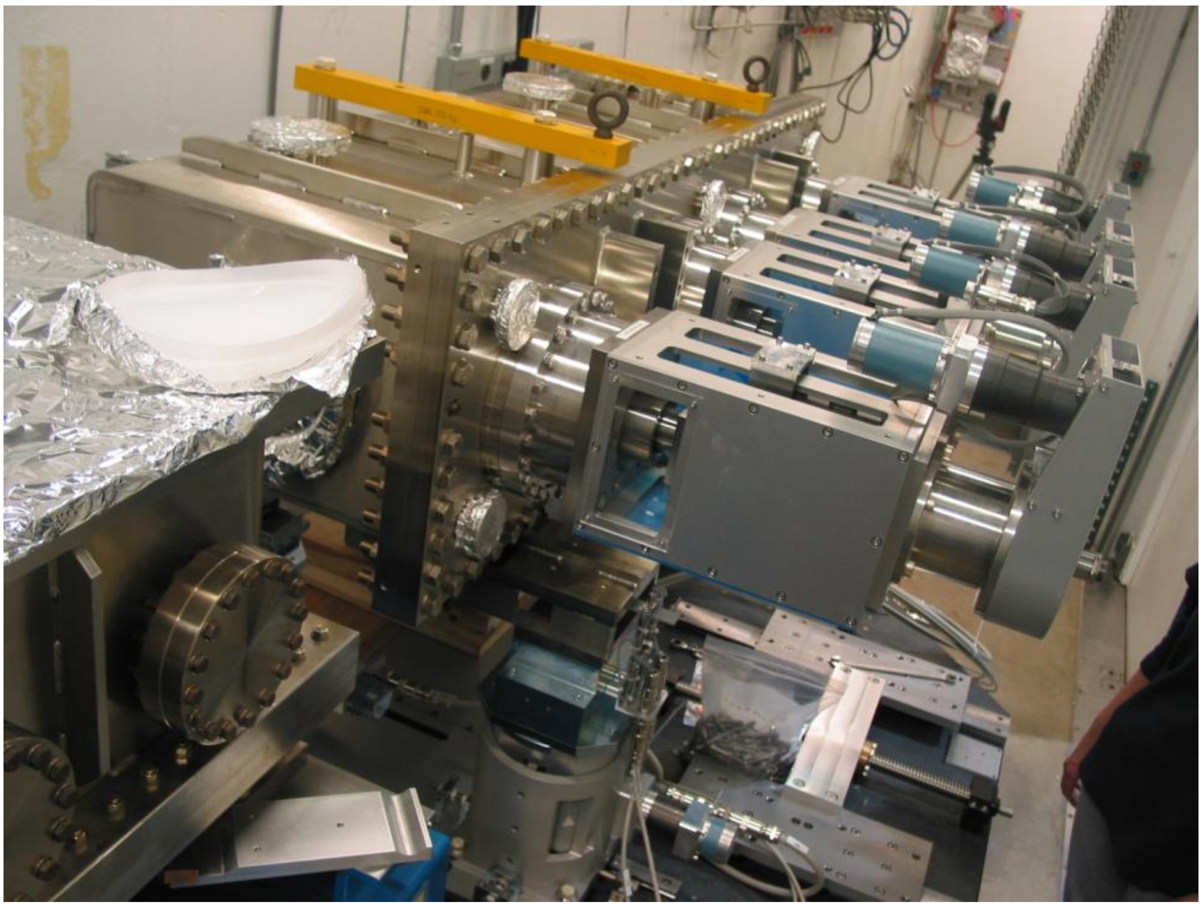

Figure 14: Double Horizontal Focusing Mirror System installed in Station 13ID-A

4.7 Secondary Source Aperture (13ID-E) and Beam Position Monitors (13ID-C/D and E)

The X-ray optics configuration provides beam sizes down to 500 nm FWHM for the X-ray microprobe in 13ID-E via a horizontal compound focusing mode. In this mode, the vertical focusing is obtained by an in-hutch, 200-mm-long KB mirror that directly images the source. The horizontal focusing is performed by combining two horizontal focusing mirrors; the first is the second in the pair of mirrors used to horizontally separate the microprobe and 13ID-C/D beams. This mirror, 590 mm long and operating at 3 mrad, is located at 31.0 m from the source and is capable of collecting 1.6 times the FWHM of the horizontal beam divergence. This long mirror creates a secondary source image 19.0 m away (demag = 1.55) with a FWHM of 341 μm. Located at this secondary source is the SSA/BPM vessel (Figure 15) which houses a secondary source aperture (SSA) and beam position monitors (Figure 16). This SSA (Figure 17) provides a convenient way of controlling the horizontal source size seen by the second horizontal focusing mirror, which is located downstream in the 13ID-E station. This second horizontal focusing mirror is 200 mm long and is located just downstream of the 200 mm KB vertical mirror and, when operated at 3 mrad, is capable of collecting 1.38 times the FWHM horizontal divergence. When operated with a 200 mm focal length, a demagnification of 24 is achieved. For the open SSA, final focus scatter plots show a spot size of 7.1 μm x 470 nm (H x V) transmitting 46% of the beam; for the 50 μm SSA, the plots show a spot size of 1700 x 470 nm (H x V) transmitting 11% of the beam. Spot sizes below 500 nm are likely to be achieved by reducing the SSA down to as low as 10 μm. The vessel housing the SSA also houses beam position monitors for both beamlines.

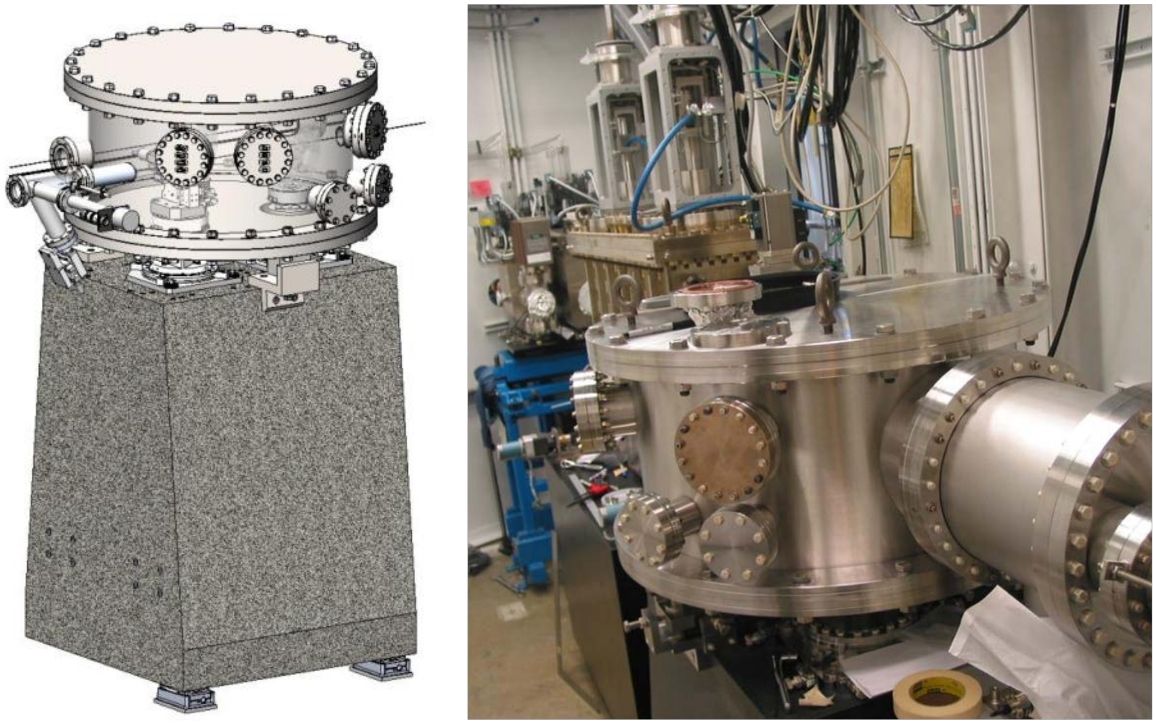

Figure 15: Rendition of the SSA/BPM Vessel (left) and photograph of the installed unit in 13ID-B (right).

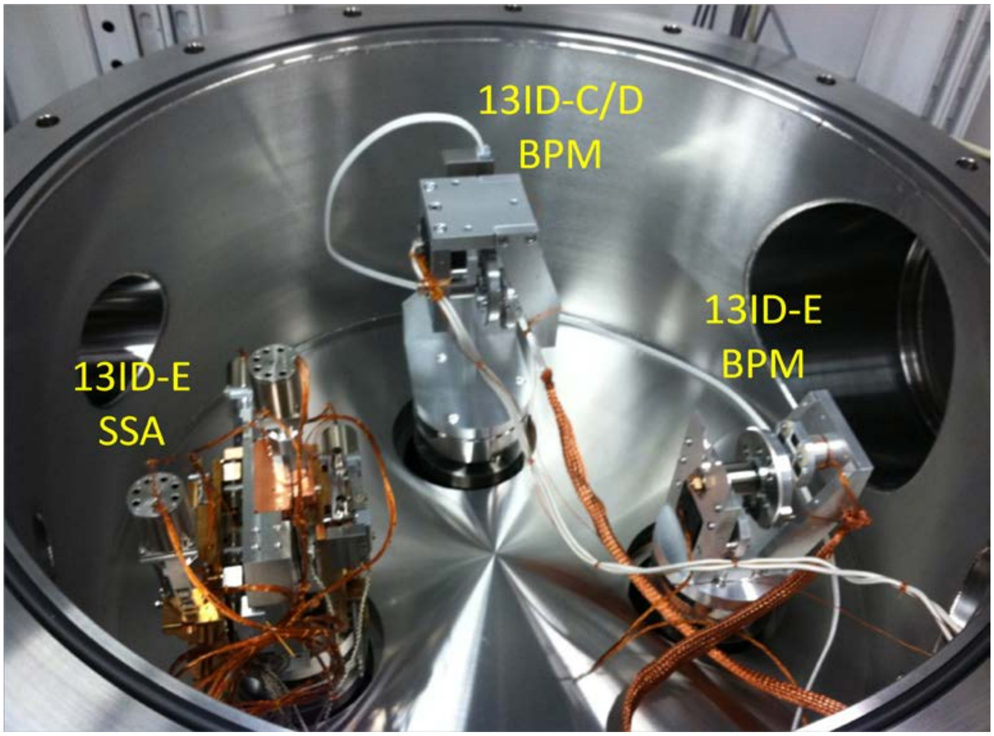

Figure 16: Photograph of the SSA/BPM Vessel with lid off showing the 13ID-E Secondary Source Aperture (SSA) and the Beam Position Monitors (BPM) for 13ID-E and 13ID-C/D.

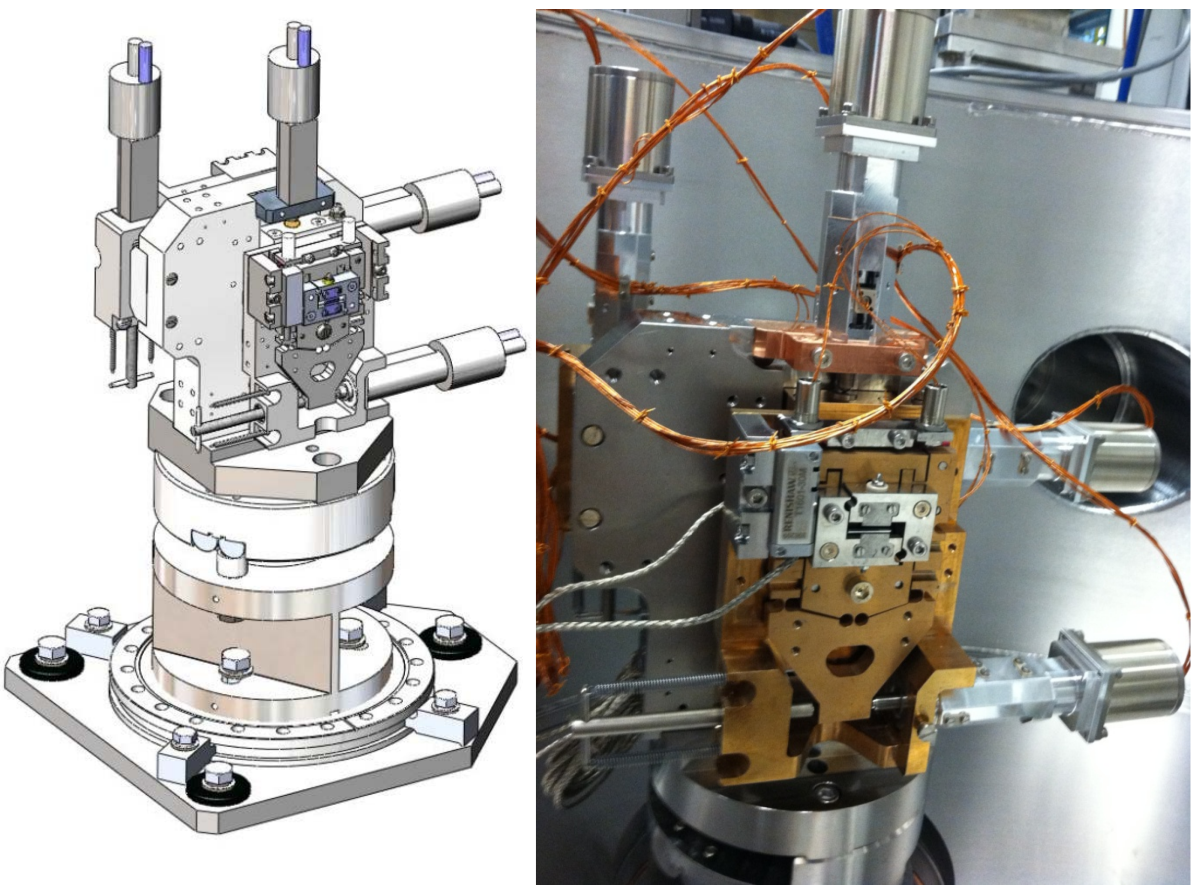

Figure 17: Detailed rendition of Secondary Source Aperture (left) and photograph of the installed unit (right) in the SSA/BPM vessel.

4.8 Kirkpatrick-Baez Microfocusing Mirror System

The previously used Kirkpatrick-Baez (KB) microfocusing mirror system employed two, 100 mm long, rhodium-coated silicon mirrors each in a bender for dynamic focusing. It routinely produced microbeams in the 2 µm range. An improved system, partially supported by the canted upgrade grants, was required for the upgraded microprobe beamline primarily to allow focusing of low energy X-rays. Also, with recent reductions in achievable mirror slope errors, larger mirrors were desired to collect more of the undulator beam without sacrificing focal spot size.

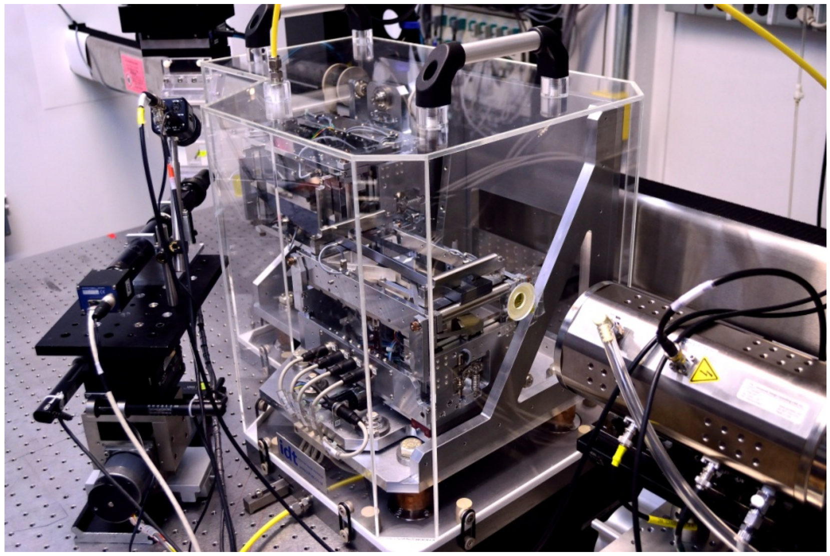

Figure 18: The 13ID-E KB microfocusing mirror system in its plastic helium enclosure and installed in the 13ID-E station. The system is designed to focus Undulator-based X-rays down to sub-micrometer spot size.

The upgraded KB system (Figure 18) consists of two, 240-mm long, highly polished, silicon mirrors each with three distinct surfaces consisting of stripes of either bare Si, Rh, or Pt. Translators running perpendicular to the beam direction allow X-ray reflections to occur on one of the desired surfaces, the optimum one depending on the specific experiment. Bare Si is valuable for low energy operations and produces the lowest energy reflection cut-off for harmonic rejection. The Pt surface is useful for high energy operations because it reflects X-ray to the highest energy (lowest angle). Rhodium is optimum for moderately high energy, particularly in the ~12 keV region which is potentially interfered by Pt absorption edges.

In conjunction with the secondary source aperture (SSA), design FWHM beam spots range from 7 µm H x 0.5 µm V with the SSA open to 1.7 µm H x 0.5 µm V with the SSA at 50 µm trading-off for flux (Figure 19). Horizontal sizes below 1 µm should be achievable reducing the SSA further.

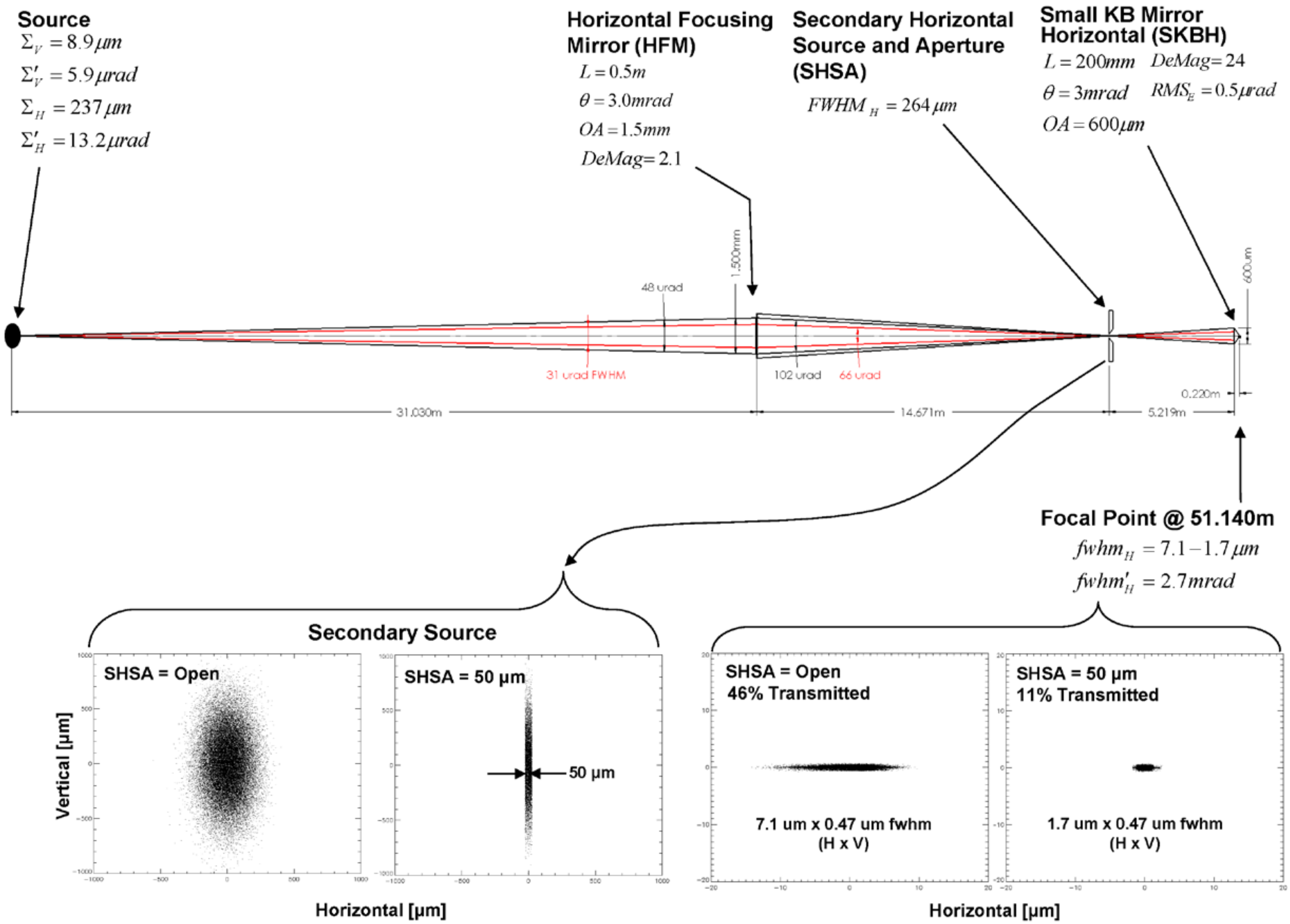

Figure 19: Horizontal ray trace for 13ID-E beamline. Beam dimensions in lower right indicate adjustability using variable SSA widths.

5. Upgraded Beamline Performance

The sections below summarize some of the initial results with the upgraded beamlines demonstrating the superb performance of the new capabilities.

5.1 13ID-C/D Branch Line

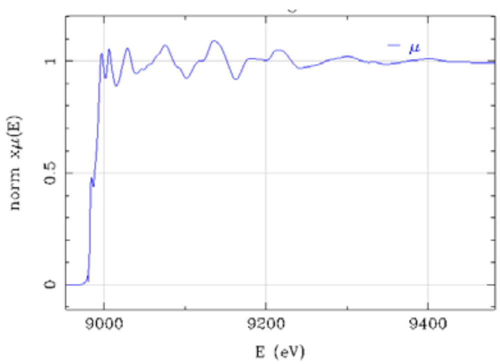

Figure 20: EXAFS scan of Cu foil using the Si(311) crystal pair on the upgraded 13ID-C/D beamline.

The upgraded monochromator is working very well and is able to work reliably at higher energy than we have in the past. While this monochromator has both Si(111) and Si(311) crystals, most of the commissioning work that has been done at the time of this report has been with the Si(111) crystal. High quality EXAFS scans have also been obtained with the Si(311) crystal set (Figure 20). Some commissioning work remains to be done on the Si(311) crystals especially at energies above 40 keV. Nonetheless, we are now in a fully operational mode and expect to run a complete schedule of General User Proposals for DAC, LVP, and Surface Scattering experiments on the 13ID-C/D branch in the first cycle of 2013 (2013-1).

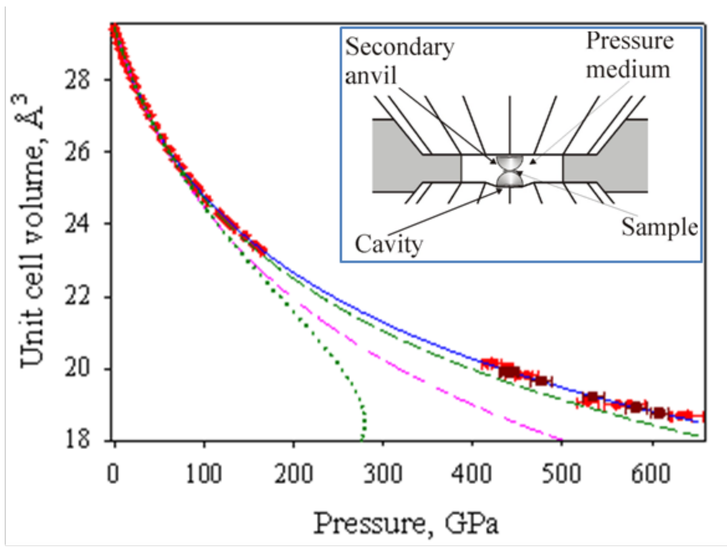

Figure 21: Inset: Schematic diagram of the double-stage diamond anvil cell. The primary anvils are made of single crystal diamonds, while second-stage anvils are semi-balls of nanocrystalline diamond. **Plot:** Pressure dependence of the unit cell volume of Re (red circles - as determined from Experiment #1 with the Au pressure standard[26]; dark-red squares – as determined from Experiment #2 with pressure obtained using the BM4 EOS of Re established in Experiment #1). Continues blue line is a result of fitting experimental data using the BM4 EOS (K_{300}=342(6) GPa, K'=6.15(15), K''= -0.029(4), V_0=29.46(1) $Å^3$/unit cell), dashed green line – BM3 EOS fitted for data collected up to 165 GPa (K_{300}= 353(3) GPa, K'=5.80(7)), dashed short-long pink line is BM3 by Vohra et al. (*Phys Rev B* 36, 979, 1987), dotted green line is BM4 by Jeanloz et al. (*Nature* 349, 687-689, 1991).

5.1.1 Implementation of Nanodiamond Anvils for High-Pressure Studies above 600 GPa

(Dubrovinsky et al., Nature Communications, 2012, DOI: 10.1038/ncomms2160)

In one of the first experiments on the upgraded 13ID-C/D branch line, we have demonstrated that double-stage diamond anvil cells (DACs) with secondary anvils fabricated from nano-crystalline diamond (NCD) allow a dramatic extension of the achievable pressure range in static compression experiments. A conventional DAC was used with second-stage anvils made of NCD semi-balls. The second-stage anvils were surrounded by a pressure-transmitting He medium allowing us to reach static pressures up to 640 GPa. A 3 µm X-ray beam at 40 keV was used for microdiffraction experiments demonstrating the enhanced sensitivity with the upgraded beamline.

We demonstrated the necessity of using the fourth order EOS in order to describe properly the behavior of Re at high pressure and also that for such a low-compressible material as rhenium static compression data in the 0.5 TPa range are essential (Figure 21).

Previous experimental and theoretical studies indicated that the c/a lattice parameters ratio of hexagonal close-packed (hcp) structured Re stays fairly constant up to about 200 GPa, while ab initio simulations predicted complex nonlinear variations (although small, within 0.33%) in the 1 TPa pressure range. Our data collected in He shows that at pressures below 120 GPa the c/a is actually constant within the measurement uncertainties, but at higher pressures up to 165 GPa the c/a slightly decreases (by 0.2%). At the much higher pressures achieved in the double-stage DACs (up to 640 GPa), the c/a ratio of hcp-Re decreases significantly, thus qualitatively supporting theoretical predictions. In the future, we are confident that achieving 1 TPa in static compression experiments with double-stage DACs is a viable goal.

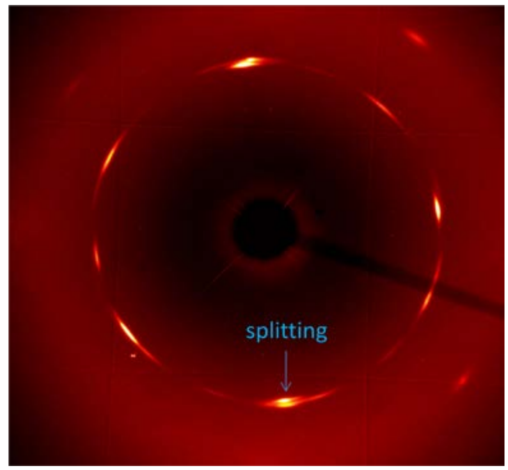

Figure 22: XRD pattern collected from H_2O in diamond anvil cell at 150 GPa in the stability field of ice-X phase. We have detected pressure induced tetragonal splitting of 110 reflection of H_2O at high pressures. Although according to theoretical calculation that phase should have cubic symmetry at this pressure.

5.1.2 High-Quality XRD Data for Low-Z Materials

After the GSECARS upgrade (optimized for high energy monochromatic undulator radiation), we are able to focus high energy X-rays (37 keV) down to less than 3 µm spot size and at the same time gain intensity by ~50 %. These new capabilities allow us to collect high quality X-ray diffraction data from low-Z materials, including H_2O, in the megabar pressure range where sample thickness is typically less than 5 µm (Figure 22). As one of the main constituents of gas giant planets, the high pressure behavior of water ice has fundamental importance in planetary science, for example.

5.2 13ID-E Branch Line

For the 13ID-E microprobe branch, the monochromator has been delivered, installed, and had first X-rays on Oct. 18, 2012 (Figure 23). The new undulator, monochromator, and large mirrors to deflect and focus the beam in the horizontal direction are working well. We have had X-rays in the 13ID-E station and have delivered beam from the Si(111) monochromator and large mirrors from 2.4 keV up to 27.5 keV. The monochromator is scanning energy quite well. Commissioning work remains especially for the Si(311) crystal set including careful alignment and energy calibration of this monochromator and the other beamline components, but so far everything is working exceptionally well. A new small K-B mirror system for the ID-E station was delivered at the end of November and commission in underway. The initial data collected to date gives us confidence that we will be able to do measurements between the S K edge (2.470 keV) and well past the Cd K edge (26.7 keV) in the 2013-1 run (Feb. – April, 2013), including micro-XRF 2-D mapping and fluorescence tomography, micro-XRD, and micro-XAS measurements. We are setting aside a few weeks during that run for further testing and commissioning but we will be running General User Proposal experiments for most of the run.

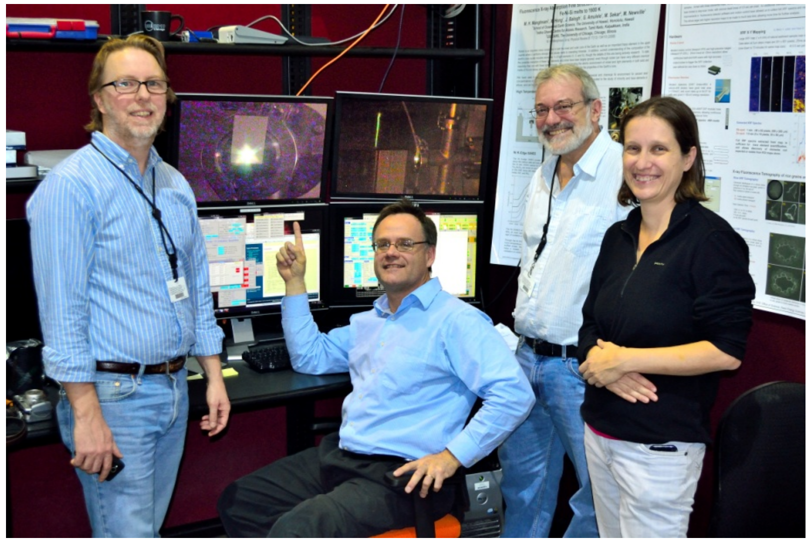

Figure 23: GSECARS beamline scientists (left-to-right: Matt Newville, Peter Eng, Tony Lanzirotti and Joanne Stubbs) celebrate first light on the new 13ID-E beamline October 18, 2012 (Peter points to X-ray beam exciting a phosphor displayed on top-left monitor).

5.2.1 Low Energy XAFS

The 13ID-E 3.6 cm period undulator allows us to produce focused beams down to the S K edge for the first time. During initial commissioning activities, a sulfur K XANES spectrum was acquired to demonstrate this capability (Figure 24 left). Also shown in Figure 24 (right) is a potassium K XANES spectrum collected from KCl. Work is ongoing to optimize analytical conditions for such measurements including minimization of absorbers in the beam (windows,

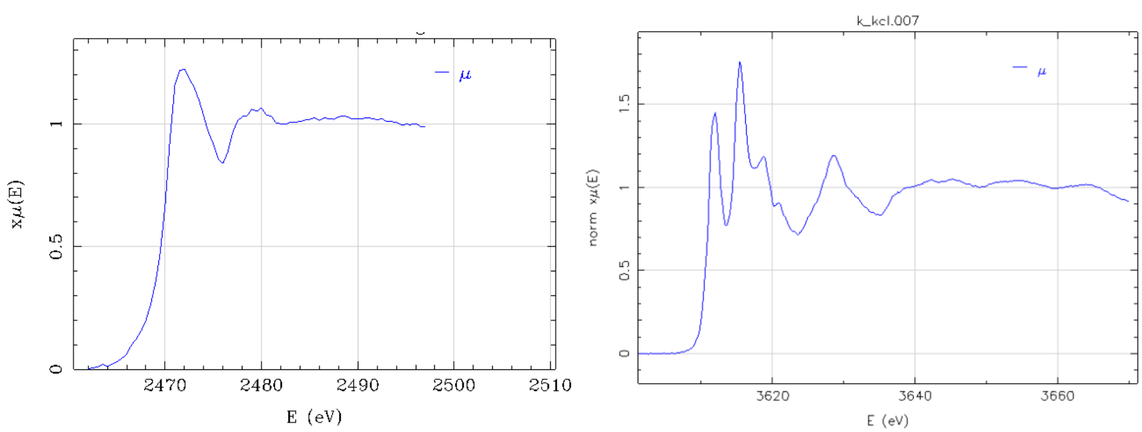

Figure 24: Low energy XAFS spectra collected in fluorescence mode (Vortex ME-4) at the new 13ID-E station. **Left:** Sulfur K XANES spectrum for elemental sulfur. **Right:** Potassium K XANES spectrum for KCl.

gases) and reduction of harmonic content. We anticipate that this low energy capability (eventually allowing full EXAFS spectra) will be under high demand by our users, especially since higher energy XAFS spectra (e.g., from heavy metals) can in principle be acquired from the same sample locations.

5.2.2 High Energy XAFS

The 13ID-E beamline was designed to operate up to 23 keV although we expect to be able to produce useful beams somewhat above that energy. To test this capability, EXAFS spectra were collected for standard metal foils in transmission mode. Figure 25 shows the excellent spectra acquired for Mo and Ag foils. The reproducibility is superb. Ag K EXAFS spectra up to 26.7 keV have been successfully acquired.

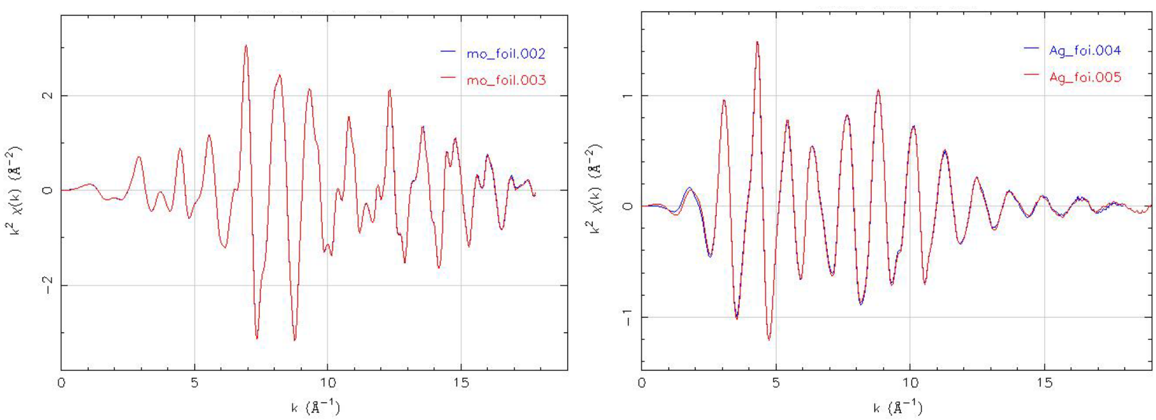

Figure 25: High energy K EXAFS spectra in $k^2\chi(k)$ collected in transmission mode at 13ID-E for a Mo foil (left; absorption edge at 20.0 keV) and Ag foil (right; absorption edge at 25.5 keV). For each plot, two scans are overlain showing the excellent reproducibility out to *k* of at least 18.

5.2.3 Microfocusing

Initial performance of the microfocusing system was excellent with vertical focal spot dimensions of 1 µm achieved in the first days of operations. Optimization of the curvature and ellipticity settings were obtained by scanning the beam over a thin (100 Å) Cr metal edge and recording the Cr K fluorescence intensity as curvature and ellipticity were varied systematically. Figure 26 shows the results of this data collection for the horizontal bender.

The first microfocused beam application was to measuring Ti K XANES spectra for phases in enstatite chondrites. Figure 27 shows one such spectrum for a Ti^{3+}-dominant enstatite grain in a type IAB chondrule within the Qingzhen EH3 meteorite. The fast-mapping capability was demonstrated with the acquisition of a superb XRF element distribution image collected with the new X-ray microprobe (Figure 28).

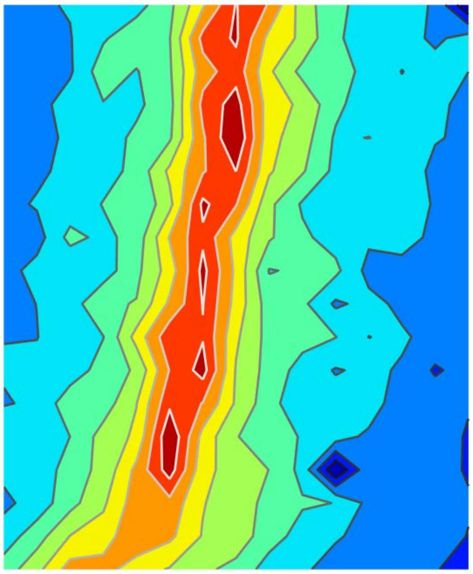

Figure 26: Curvature (vertical axis) versus ellipticity (horizontal axis) scan of the horizontal microfocusing mirror bender showing the derivative intensity (measure of tightness of focus) at each setting. The beam size at the dark red conditions was 1 µm.

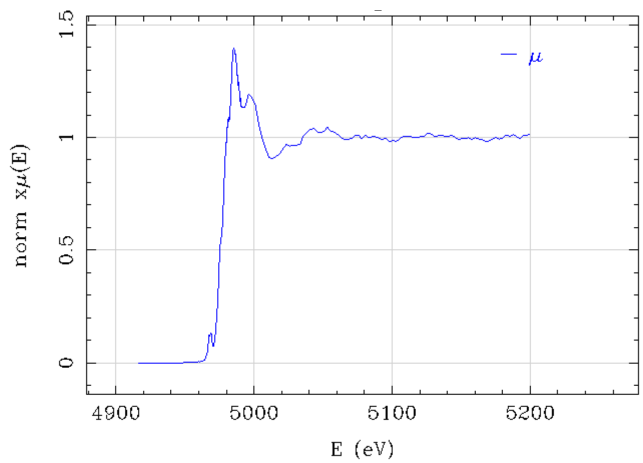

Figure 27: Ti K XANES spectra for a Ti^{3+}-dominant enstatite grain in a type IAB chondrule within the Qingzhen EH3 meteorite

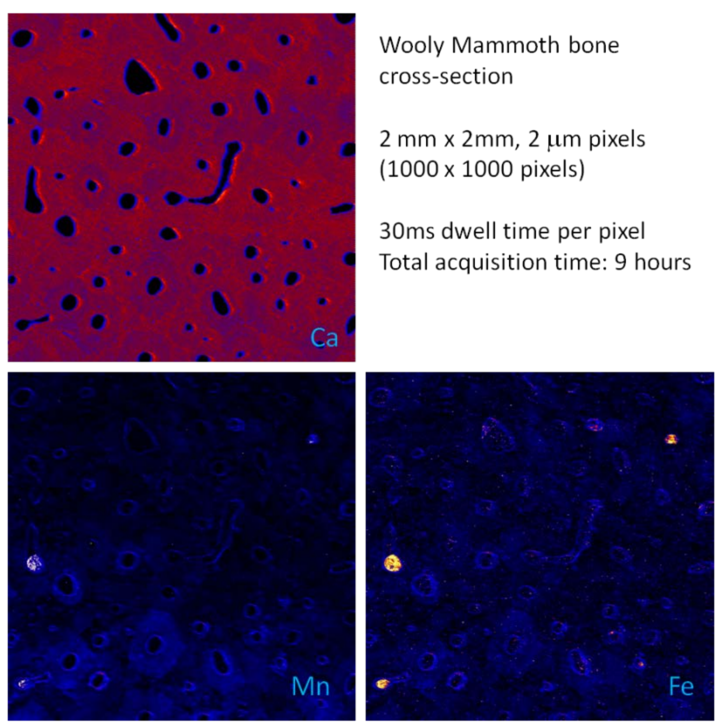

Figure 28: XRF element maps (Ca, Mn and Fe) for a cross section of a wooly mammoth bone collected with the new X-ray microprobe (13ID-E).