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Facilities for high-pressure research with the diamond anvil cell at GSECARS

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An overview of facilities for high-pressure research with the diamond anvil cell (DAC) at the GeoSoilEnviroCARS (GSECARS) sector at the Advanced Photon Source (Argonne, Illinois) is presented. There are three operational experimental stations (13-ID-C, 13-ID-D and 13-BM-D) where DAC instrumentation is installed for various types of experiments at high pressure and extreme temperature conditions. A fourth station (13-BM-C) is under construction and will be operational in 2006. While most X-ray diffraction experiments have been undertaken with powder samples so far, there is a growing demand for single-crystal diffraction (SCD) at high pressure. As one of the principal components at GSECARS, SCD is currently under rapid development. Other relevant techniques have also been developed for obtaining complementary information from powder or single-crystal samples at high pressure. For example, an on-line Brillouin system is installed and operational at 13-BM-D for acoustic velocity and single-crystal elasticity determinations. In addition, various X-ray spectroscopy techniques (*e.g.* X-ray emission and X-ray Raman) are employed for measuring electronic and magnetic properties. Future developments are discussed with the DAC program at GSECARS.

Keywords: diamond anvil cell; X-ray diffraction; X-ray inelastic scattering; X-ray emission; laser heating; Brillouin scattering; single-crystal diffraction; high pressure.

1. Introduction

High-pressure experiments at synchrotron sites have led to many new discoveries and have revealed numerous phenomena that are of fundamental importance in many fields from Earth and planetary sciences to physics and materials science. With the rapid development of the synchrotron techniques and high-pressure instrumentation, ever smaller and more complex samples at increasingly higher pressures and extreme temperatures are being studied with higher-accuracy probes for characterization of structural, electron and phonon properties. Many previous technical limitations in capability, precision and accuracy are being removed through optimization of X-ray source, X-ray optics, sample environment, experimental configuration and detectors. It is the goal of this paper to summarize developments in the diamond anvil cell (DAC) program at the GSECARS sector at the Advanced Photon Source. GSECARS is a national user facility for research in Earth, soil and environmental sciences. The layout of GSECARS and the beamline components have been described previously (Rivers *et al.*, 1998). In this paper we describe major DAC instrumentation in the GSECARS experimental stations.

The high-pressure DAC science program at GSECARS has been running since 1997 at the same time that various new techniques have undergone commissioning. Conducted experiments address key geochemical and geophysical problems in the Earth's deep interior including research in the following areas:

(i) equations of state, crystal structures and phase relations of geophysically important minerals;

(ii) high pressure–temperature studies of melts, glasses and other non-crystalline materials;

(iii) properties of light elements relevant to the outer planets;

(iv) rheology of minerals at high pressure;

(v) pressure effects on magnetic and electronic properties;

(vi) time-resolved experiments on phase transformations and chemical reactions;

(vii) ultrahigh-pressure and extremely high/low-temperature experiments.

Available X-ray techniques for research with DAC include:

(i) micro-X-ray diffraction with monochromatic radiation;

(ii) inelastic X-ray scattering (X-ray Raman);

(iii) X-ray emission spectroscopy;

(iv) X-ray fluorescence microprobe;

Table 1
Characteristics of DAC facilities at GSECARS.

	13-ID-D	13-ID-C	13-BM-D	13-BM-C
Techniques	Powder diffraction, X-ray spectroscopy, laser heating	Single-crystal diffraction, inelastic X-ray scattering, X-ray spectroscopy	Powder diffraction, single-crystal diffraction, Brillouin	Single-crystal diffraction, high-resolution diffraction
Beam size (FWHM in μm)	5–7 diameter	50 (h) × 25 (v)	7 (h) × 20 (v)	26 (h) × 28 (v)
Energy range (keV)	5–45	5–45	5–75	18, 30
Flux at sample position (photons s^{-1})	10^{11} at 37 keV	10^{13} at 10 keV	5×10^{8} at 37 keV	5×10^{11} at 30 keV†
YLF laser system	On-line	–	Off-line	–
CO_2 laser system	Under construction	–	–	–
Brillouin system	–	–	On-line	–
Raman system	On request	–	Under design	–

† The corresponding beam divergence is 10 mrad in the horizontal. By reducing the incident beam size, beam divergence can be almost linearly reduced at the expense of flux. For example, a beam divergence of 0.1 mrad can be reached corresponding to a flux of 5×10^{8} photons s^{-1} at the sample position at the energy of 30 keV, a flux still comparable with that at 13-BM-D.

(v) X-ray absorption and radiography.

In addition, other instruments have been developed specifically for DAC studies and installed in experimental stations, such as laser heating systems, Brillouin spectroscopy and ruby fluorescence systems. Currently, there are three operational stations (13-ID-C, 13-ID-D and 13-BM-D) where DAC instrumentation is installed for various types of experiments at high pressure and extreme temperature conditions. The fourth station (13-BM-C) is under construction and will be operational in 2006.

2. DAC instrumentation at GSECARS

2.1. Undulator station 13-ID-D (laser heating, powder diffraction and spectroscopy)

Station 13-ID-D is the end station of the undulator beamline. The DAC setup in 13-ID-D is primarily designed for micro-X-ray diffraction experiments at extreme pressure–temperature conditions. Combination of X-ray diffraction with other X-ray spectroscopic measurements (absorption, emission, X-ray Raman) is possible with reasonable setup time. The main characteristics are listed in Table 1. Fig. 1 shows a typical schematic of the DAC setup in the 13-ID-D station. The entire setup is on a lift table that enables precise and reproducible positioning of the entire apparatus. The lift table can be moved 'off X-ray beam' for experiments with the large volume press that shares the station at the downstream side. All controls and operations can be accessed outside the experimental station.

2.1.1. X-ray optics. The high brilliance from APS undulator-A offers extraordinary X-ray focusing capabilities. In 13-ID-D, the X-ray beam is controlled by slits to a size of ∼250 μm × 250 μm. Ion chambers (IC1, IC2; Fig. 1) before and after the slits monitor the X-ray intensity with the signal from IC2 used for feedback to the beamline monochromator for beam stability in both intensity and position. A pair of Kirkpatrick–Baez (KB) mirrors (Eng *et al.*, 1995) of length 200 mm is used for focusing the beam at the sample position to a desired size (typically around 5–7 μm in diameter at FWHM for DAC experiments). The distances between the sample position and centers of the horizontal and vertical KB mirrors are 450 mm and 650 mm, respectively. The relatively long working distances provide a well collimated beam with divergence generally <0.1 mrad, and space for laser optics and a pinhole that serves as a clean-up aperture. The clean-up aperture (20–40 μm in diameter and 200 μm thick platinum) is found to be essential for removing unwanted scattering from surrounding materials including the gasket by filtering out the tail of the X-ray beam. For example, for a sample chamber of ∼30 μm in diameter with a rhenium gasket, diffraction for a silicate mineral at 1.5 Mbar is found to be free of rhenium reflections (Mao *et al.*, 2004). All X-ray optics are enclosed in shielding material (Fig. 1), ensuring low X-ray scattering background in the station.

2.1.2. Sample positioning control. The stage package for sample positioning control is designed to have excellent stability and good resolution of motion. The sample position is 3.5 inches (87.5 mm) above a kinematic base plate (BKL-4, Newport). The sample stage has motorized *x*-*y*-*z* translations and ω rotation with its axis vertical and perpendicular to the X-ray beam. The linear translations have a position resolution of 0.1 μm, and the rotation has an angular resolution of 0.001°. The sample holders are water cooled for position stability, which is important for experiments involving variable temperatures. A microscope with zooming capability is pre-aligned to the sample position, making it easy to locate samples from experiment to experiment. The space around the sample position is relatively open, making it easy to access and also allowing for other experimental setups such as a cryostat for low-temperature experiments, on-line ruby system, or 1 m-long Rowland circle setup for X-ray spectroscopy measurements.

2.1.3. Detectors. The DAC setup in 13-ID-D is primarily designed for angle-dispersive mode with monochromatic beam. Both imaging plate (MAR345) and CCD (MAR-CCD) detectors are available and mounted on a motorized stage, making it exchangeable during the experiment. Diffraction collecting time in this station is short owing to the high-intensity X-ray beam, typically a few seconds to minutes depending on the scattering power of the samples. The sample is usually fixed at the rotation center of the sample stage. The

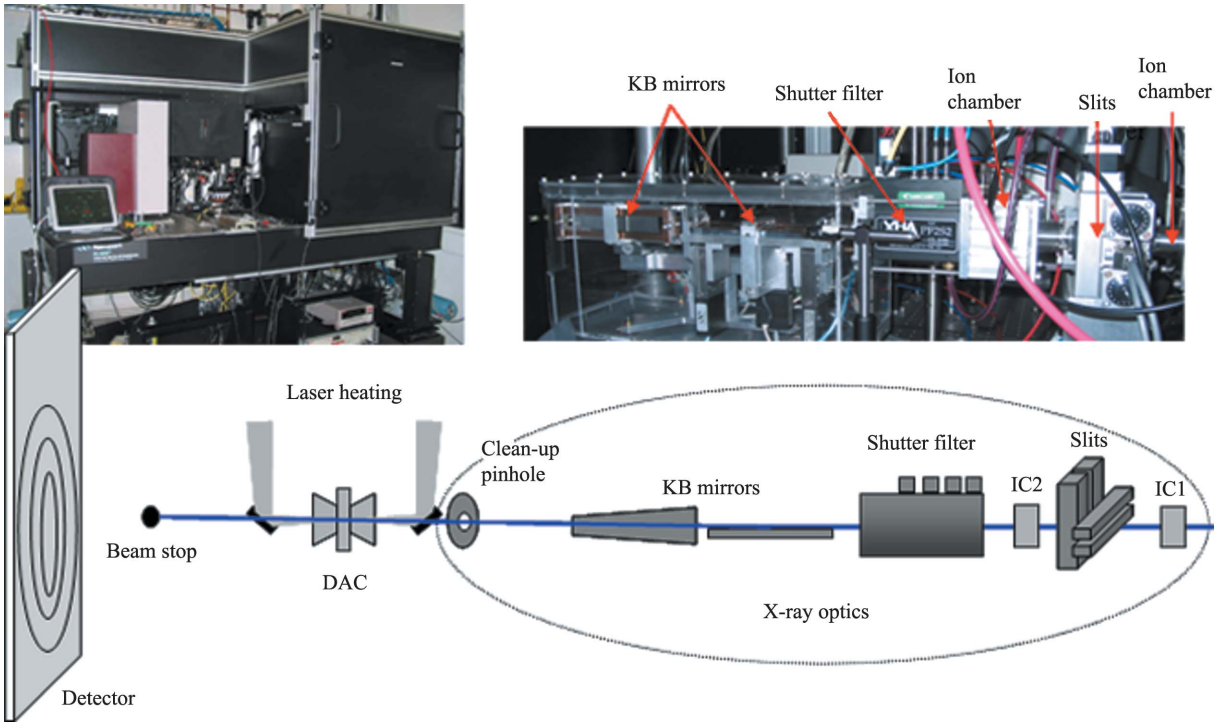

Figure 1
Schematics of the DAC setup in 13-ID-D. The inserted picture at the upper-left is an overview of the setup. The upper-right picture is a close view of the X-ray optics. IC1, IC2: ion chambers for monitoring X-ray intensity.

geometry parameters of the X-ray diffraction setup are calibrated using powder diffraction from standard materials such as CeO_2 and silicon from NIST. White beam is available but rarely used.

2.1.4. Laser heating system. A double-sided laser heating system is an integral part of the diffraction setup. The basic features of the laser heating system have been described elsewhere (Shen *et al.*, 2001). Briefly, the system consists of two lasers with one operating in donut mode and the other in Gaussian mode with maximum powers of 80 W and 65 W, respectively. The two laser beams are combined and then split from one point into two branches to heat a DAC sample from both sides. The heating area is about 20–30 μm in diameter, compared with the X-ray beam size of diameter 5–7 μm. A feedback system operates in two modes: constant laser-power mode or constant temperature (thermal intensity) mode. Temperatures can be measured simultaneously from both sides using on-line spectrometers. Sensitive CCD cameras often allow real-time imaging of X-ray-induced luminescence from the sample and/or medium, an important feature for precise alignment (the X-ray beam, the heating spot and the area where the thermal radiation is collected for temperature measurement). Recently, we have made a few modifications to improve user-friendliness and system stability (Fig. 2). Improvements include new optics for controlling the laser power on each side of the sample in the DAC for both donut and Gaussian lasers, rigid mechanical arrangements for maximizing stability of the system, and configuration changes in the temperature measurement system for better accuracy. The laser power is controlled by introducing three polarizing

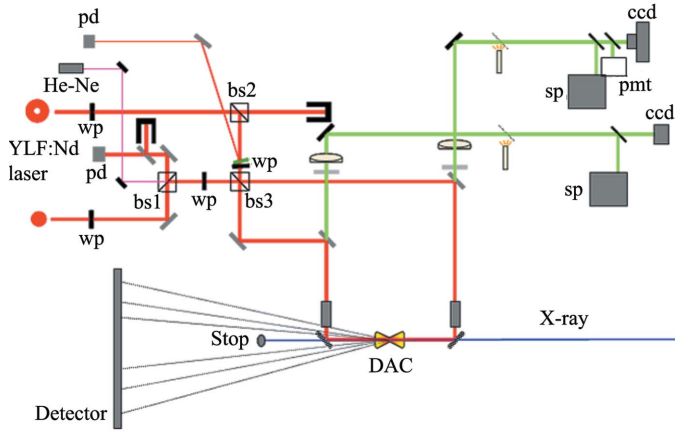

Figure 2
Double-sided laser heating system installed in the station 13-ID-D. Lines in red are laser guiding paths. Lines in green are those of thermal radiation for temperature measurements. wp, wave plate for changing polarization direction; pd, photodiode for monitoring laser power; bs1, bs2, bs3, polarizing cube beam splitters for beam splitting and power regulation; sp, spectrometer for temperature measurement; pmt, photomultiplier tube; ccd, CCD camera. The red donut symbol stands for the laser operating in TEM01* mode. The red dot is the laser operating in Gaussian mode. He-Ne is the class-II He-Ne laser for alignment purpose.

beamsplitters combined with four wave plates. By changing polarization directions, the power of each laser can be regulated (bs1, bs2), as well as the laser powers of the split beams (after bs3) to each side of the DAC.

A CO_2 laser heating system is under construction and will be operational in 2006. This is a COMPRES (COnsortium for

Materials Properties Research in Earth Sciences) sponsored project in collaboration with scientists at Princeton University and University of Chicago. The current plan is to deliver the CO_2 laser light vertically (compared with the horizontal delivery geometry of the existing YLF laser system) to the DAC sample from the downstream side. The existing temperature measurement optics at the upstream side will be used for measuring temperatures. Considering the wavelength of CO_2 laser light (10 μm) and the typical thickness of a DAC sample (5–30 μm), single-sided heating should be sufficient.

2.1.5. Other techniques. Besides primary X-ray diffraction experiments, other X-ray measurements are also performed at 13-ID-D. The L-shape of the top tier (Fig. 1, inset) provides an open area around the sample stage, which makes it possible to accommodate bulky devices such as cryostats, vacuum chambers and X-ray spectroscopy setups. X-ray emission spectroscopy (XES) apparatus, for example, is sometimes installed for studying the electronic states of the sample (Badro *et al.*, 2002). In XES, the excitation X-ray source only needs to have a higher energy than that of the absorption edge of the element of interest. Pink (undulator beam with energy bandwidth of hundreds eV) and monochromatic X-rays can be used as the source. High-resolution emission spectra are obtained by a synchronized θ–2θ scan of the analyzer and the detector on a Rowland circle. At GSECARS, XES can be combined with the on-line laser heating system for studies of electronic and magnetic states at high pressures and high temperatures (Lin *et al.*, 2005). X-ray diffraction can also be simultaneously performed if the monochromatic beam is used as the excitation source.

2.2. Undulator station 13-ID-C (single-crystal diffraction, inelastic X-ray scattering and spectroscopy)

The station 13-ID-C shares the undulator beam with the 13-ID-D station. When operating in the C station, the D station is accessible and therefore available for off-line experiments such as with laser heating and the large volume press. There are two main experimental setups in 13-ID-C. At the upstream section of the hutch is an X-ray microprobe and in the downstream location is a six-axis Kappa diffractometer. High-pressure DAC experiments have been performed at both locations. By utilizing the table-top micro-focusing KB mirrors, high-pressure XANES, EXAFS and fluorescence tomography measurements have been performed. A monochromatic undulator beam is focused to ∼1 μm × 1 μm with sufficient intensity for X-ray fluorescence mapping and extended XAFS of dilute systems at energies above 4 keV (Newville *et al.*, 1999). On the diffractometer, both high-pressure single-crystal diffraction and inelastic X-ray scattering (IXS) experiments are performed (Meng *et al.*, 2004; Mao *et al.*, 2003).

The high-pressure IXS provides insights into the high-pressure structure for samples that are unobtainable by any other method. Conventional synchrotron X-ray absorption spectroscopy (*e.g.* XANES and EXAFS) can be effective in providing element-specific atomic configurations. Application of these conventional methods to the low-Z elements at high pressure is impractical owing to the inability of low-energy (∼200 eV) X-rays to penetrate the pressure vessel. In IXS, a high-energy incident X-ray beam is used (∼10 keV) which easily penetrates the pressure vessel. The IXS signal is the modulation in the small energy loss and momentum transfer of the scattered photons which can also easily escape the pressure vessel.

The IXS setup in 13ID-C is optimized for use with a high-pressure DAC sealed with a Be gasket using a radial scattering geometry (Fig. 3). The DAC is mounted on the six-axis Kappa diffractometer where inelastic IXS spectra are collected by scanning the incident beam energy above the analyzer energy that is fixed at the elastic energy of 9.692 keV. These high-energy X-rays can easily penetrate the Be gaskets used to seal

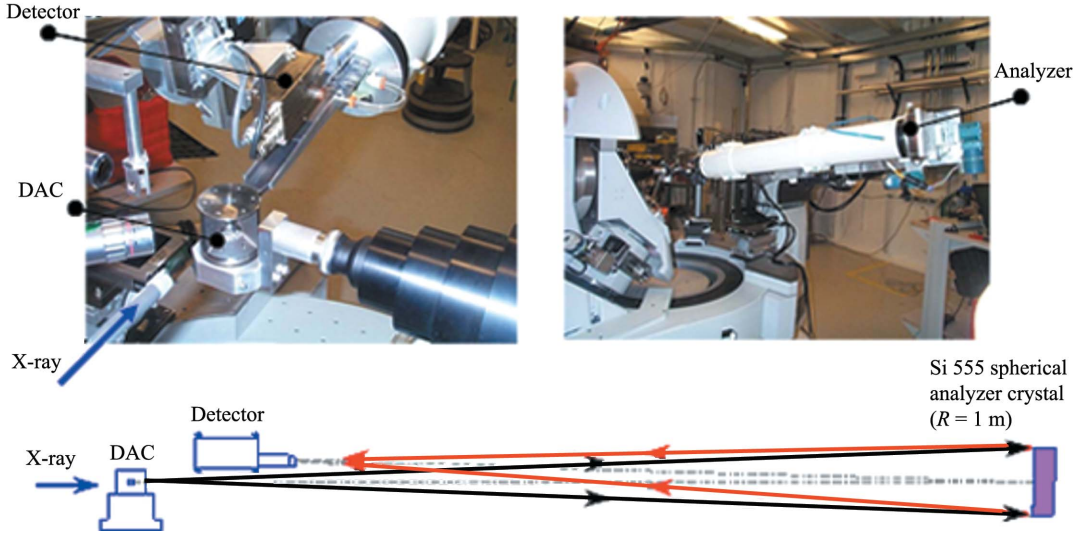

Figure 3
A backscattering geometry for inelastic X-ray scattering (X-ray Raman) experiments in 13-ID-C. The photographs are views of the DAC (top-left) and the detector arm (top-right) mounted on the six-axis Kappa diffractometer in 13-ID-C.

the DAC. The inelastic X-rays are collected over a range of scattering angles in both the horizontal and vertical plane with a linear array of six spherical Si(660) analyzers operating in a backscattering geometry (Eng *et al.*, 2005). The system resolution is ~1 eV and is limited by the resolution of the double-bounce Si(111) monochromator. The instrument routinely collects boron, carbon, nitrogen and oxygen *K*-edge spectra at pressures up to 35 GPa. The monochromatic undulator beam is double focused to a spot size at the sample of 60 μm × 20 μm (horizontal × vertical) by a pair of 1 m-long KB mirrors in 13-ID-B (Eng *et al.*, 2000).

2.3. Bending-magnet station 13-BM-D (powder diffraction, single-crystal diffraction, Brillouin scattering)

13-BM-D is the end station of the bending-magnet beamline. A beam mask in 13-BM-A divides the 6 mrad horizontal fan into two pieces, 2.5 mrad (outboard) for the 13-BM-D, and 1.5 mrad (inboard) for the side station (13-BM-C). Stations B and C are completely independent and can operate 100% of the time. The DAC setup in 13-BM-D shares a lift table with other techniques (microtomography, microprobe, EXAFS), and is based on an additional L-shape breadboard for quick installation. The design is primarily for micro-X-ray diffraction experiments at extreme pressure–temperature conditions. Some X-ray spectroscopic measurements (X-ray absorption, X-ray fluorescence, X-ray radiography) are possible with reasonable setup time. All controls and operations can be made outside the experimental station with user-friendly interfaces.

A Brillouin system is installed on the second tier with guiding optics to DAC samples (Fig. 4). An on-line Raman system is under construction. A CO_2 laser heating system is under design to be combined with the Brillouin or Raman system together with *in situ* X-ray diffraction measurements.

2.3.1. X-ray optics. 13-BM-D is served by a high-energy monochromator (Table 1). White beam is available on special request for DAC experiments. The X-ray beam at the sample position can be either unfocused or focused. Unfocused beam is mainly for radiographic images and experiments with large samples (McKelvy *et al.*, 2004). Focused beams are for various X-ray probing techniques for small samples in DAC. The flux of the focused beam at the sample position is about a factor of 200 less than that in 13-ID-D, which increases the data collection times for experiments. Typical exposure time ranges from 1 to 30 min for powder samples and less than 2 min for single-crystal zone-axis diffraction studies. In the vertical direction, a large KB mirror (1 m long) in 13-BM-B is used, which is about 7 m away from the sample position and produces a focus down to about 20 μm at FWHM. A table-top KB mirror (200 mm long) is located 400 mm from the sample position, and is used for horizontal focusing, resulting in a 7 μm × 20 μm focus spot at FWHM at the sample position. In addition to the focusing optics on the breadboard, there is a shutter/filter control box for beam control and a clean-up aperture similar to that in 13-ID-D for filtering out the tail of the X-ray beam.

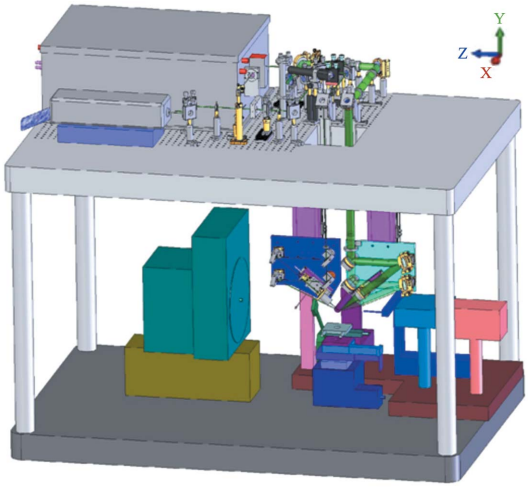

Figure 4
A Brillouin spectroscopy system integrated with the diffraction setup in 13-BM-D (Seinogeikin *et al.*, 2005). The facility allows for measuring elasticity, sound velocities and densities of materials simultaneously.

2.3.2. Sample positioning control. The sample stages are similar to those in 13-ID-D. Having the kinematic base and the same geometry in both stations, sample holders can be shared, which is very beneficial for some experiments such as off-line laser annealing. In fact, we have established a standard sample height (3.5 inch above a kinematic plate) for all DAC setups at GSECARS including the Raman system for pressure measurements and sample characterization.

2.3.3. Detectors. In 13-BM-D, an imaging plate (IP) system (MAR345) is the primary detector for its large size and high dynamic range. Collecting time is typically minutes to a few tens of minutes with a readout time of ~2 min. Compared with CCD detectors, the IP has the advantage of low dark background accumulation in relatively long exposure times. A CCD detector is also available for some experiments (chemical reactions, kinetics studies) where fast readout is critical.

2.3.4. Brillouin system. A Brillouin system has been installed as shown in Fig. 4. This project is sponsored by COMPRES and is in collaboration with scientists at the University of Illinois-Urbana. In 13-BM-D, the Brillouin system is in symmetric platelet geometry (Sinogeikin *et al.*, 2005). In Brillouin scattering, laser light interacts with phonons and is scattered with Doppler shifted frequency, where the shift (the Brillouin scattering) is directly proportional to the acoustic velocity. The Brillouin system opens a new area for *in situ* studies of materials at extreme conditions. With this 'one of a kind in the world' system, it is now possible to measure sound velocities and densities of materials simultaneously. Important materials properties (equations of state, elasticity) can be measured as a function of pressure and temperature. These data will result in new pressure scales, as the current scale still heavily relies on dynamic shock-wave data. The experimentally determined density–velocity relationships will also provide information essential for understanding seismic observations and modelling the composition and evolution of the Earth.

2.4. Bending-magnet station 13-BM-C (single-crystal diffraction, high-resolution powder diffraction)

Station 13-BM-C is under construction and will be operational in 2006. It is a side station that receives 1.5 mrad horizontal fan split from the total 6 mrad fan from the bending magnet. The other 2.5 mrad fan for 13-BM-D travels straight downstream into its experimental station. To separate the two beams horizontally, allowing enough space to accommodate the end station experimental equipment, we use a horizontally deflecting single-bounce Rowland circle monochromator with an exit angle ranging between 10 and 30°. The Rowland circle monochromator will select the X-ray wavelength as well as horizontally focus the beam. In the vertical, a dynamically bent focusing mirror will be used, which also serves to remove higher-order harmonic contamination from the beam. As shown in Table 1, the flux at the sample position is rather high, while beam divergence in the horizontal is also large (∼10 mrad). This is due to the large demagnification of the Rowland monochromator and the 1.5 mrad fan that it collects. By closing the horizontal slit before the Rowland circle, the horizontal divergence can be effectively reduced, sacrificing flux for resolution. This configuration gives flexibility for selecting optimal X-ray beams for meeting various needs in experiments. X-ray energies will be fixed at around 18 or 30 keV for DAC experiments. The DAC will be mounted onto a diffractometer already installed in the experimental station (Fig. 5), allowing both single-crystal and powder diffraction experiments to be performed at high pressure. The diffractometer has two additional degrees of freedom compared with a classic Kappa diffractometer. The detector arm can carry up to 40 kg, and has two degrees of freedom, which allows the diffraction plane to take on an arbitrary orientation and removes the need to rotate the diffraction vector of the sample onto a fixed diffraction plane. We plan to focus on two types of DAC experiments: (i) single-crystal diffraction and (ii) high-resolution powder diffraction.

Figure 5
Photograph of the six-axis Kappa diffractometer installed in 13-BM-C.

13-BM-C will be the main station for high-pressure single-crystal diffraction studies at GSECARS. The installed diffractometer can accommodate the restricted scattering geometry imposed by the DAC. The bending-magnet source is often sufficient for relatively strong scattering from single crystals. Both area detectors and point-counting detectors will be available for single-crystal diffraction. High-resolution powder diffraction measurements will be also extensively performed in this station. This will naturally offload a significant fraction of existing highly requested DAC experiments at GSECARS. The other stations will therefore be reserved for those experiments that require on-line laser heating, energy scanning or high flux. Large area detectors will be generally used for powder diffraction experiments in both axial and radial geometry. In addition, with the sophisticated detector arm, point-counting detectors will be used for higher diffraction resolution, which will be useful for studies of materials with low symmetry and samples with multiple phases.

2.5. Supporting facilities

For efficient use of beam time, it is always beneficial to have high-pressure samples prepared before the assigned beam time. However, high pressure–temperature experiments are often dynamic: samples and/or sample configuration may be subject to unpredicted changes or even failure as experimental conditions vary. It is essential to have facilities for sample preparation on site. The supporting facilities at GSECARS are designed for carrying out complete high-pressure experiments for sample preparation and characterization. The sample preparation facilities include (i) microscopes, (ii) a micromanipulator, (iii) a glove box equipped with microscope, (iv) a mechanical micro-drill machine and electric discharge machines, (v) cryogenic loading facility, and (vi) tools for routine DAC operation. A micro-Raman system is available for pressure measurement and sample characterization. The laser heating systems and the Brillouin system installed in the experimental stations can also be used as off-line facilities if hutches are accessible. A gas loading system is being designed and will be operational in 2006, allowing the loading of samples that are gases at ambient conditions. Besides these dedicated DAC facilities, there are machine shop facilities for trained users. GSECARS also provides computer software (commercial and developed by staff) for users to carry out data analysis on site.

The supporting facility plays an important role in productive experiments. It also makes it possible to carry out collaborative work between GSECARS staff and scientists who do not have high-pressure facilities at their home institutions or users from other beamlines at APS.

3. Future developments

Among the scientific goals of the DAC program is the study of materials properties across the entire pressure–temperature spectrum of the terrestrial planets. Major areas of focus will be on achieving ultrahigh pressures to multi-Mbar with powders

and single crystals, study of equations of state and phase relations, accurate structural and electron density determinations, high-pressure elasticity and rheology, studies of melts and glasses, kinetics of phase transformations, chemical reactions, multi-component systems, and high-accuracy pressure calibration at high temperatures.

Diffraction geometry will continue to be the main setup in 13-ID-D. The relatively open space around the sample stages permits other X-ray measurements to be performed, *e.g.* EXAFS that reveals element-specific local environment, IXS that reveals electronic properties, and XES (currently available) that reveals electron spin state. For laser heating experiments, we found that sample preparation plays an important role in stable and controllable heating in DAC. Introducing micro-engineering techniques in sample preparation processes will be a crucial step for future breakthroughs. Materials at high temperature often undergo numerous changes (grain growth, phase transitions, chemical reactions). Time-resolved experiments at the $\ll$1 s level will be feasible for studying kinetics of these changes with the brilliant source and the fast-developing X-ray detector technique. Minimizing background arising from diamond anvils and other surrounding materials remains a challenging task in DAC experiments, especially in liquid/amorphous scattering, thermal diffuse scattering, and low-Z materials. With the development of the collimation techniques (Mezouar *et al.*, 2002; Wang *et al.*, 2004) and the three-dimensional detector (an area detector with energy dispersion capabilities of each individual pixel), large reduction of background becomes possible either by spatial collimation and/or energy discrimination.

Inelastic X-ray scattering has opened a wide new field of near K-edge spectroscopy of the first- and second-row elements. Both energy and momentum can be scanned to obtain the full dynamic structure factor for investigation of fundamental chemical bonding, valence and conduction electronic structures, and electron correlations. To extend the pressure range, we will be pairing the large KB mirrors with a set of small KB mirrors to reduce the focal spot size to about 5 μm × 5 μm. This compound optics arrangement and the resulting spot size reduction will allow us to reach Mbar pressures without a substantial loss of signal-to-background ratio. To increase data collection efficiency, one-linear-array (six elements) detectors will be replaced by multiple arrays with corresponding multiple detectors, thus enlarging the collection solid angle and leading to one IXS scan within an hour.

With the newly installed Brillouin system in 13-BM-D, many activities are anticipated for studying high-pressure elasticity and establishing new pressure scales by simultaneously measuring sound velocities and volumes. These measurements will be extended to high temperatures, first with externally heating techniques and in the near future with the CO_2 laser heating technique.

Another area of focus in 13-BM-D will be single-crystal diffraction. Recently, a micro-X-ray diffraction has been developed by combining white and monochromatic beam capabilities (Tamura *et al.*, 2002). In this method, the polychromatic Laue pattern defines the symmetry, number of crystals, deviatoric strain/stress tensors, and their orientations. A tunable monochromator can be moved inline to deliver a monochromatic beam exactly following the original, focused, white-beam path. The monochromatic diffraction determines the d-spacing of the specific reflection and defines the unit-cell parameters of the microscopic crystal. In 13-BM-D, the energy of the monochromatic beam can be tuned from 5 to 80 keV. Instead of switching between white and monochromatic beams, it is possible to apply the monochromatic scanning Laue technique, which involves rapid scanning over a defined energy range, for defining the symmetry, crystal orientations, as well as information on specific reflections.

Single-crystal diffraction measurements with full structural determination are still largely performed with laboratory facilities routinely up to ~10 GPa (Angel *et al.*, 2000). The facility at 13-BM-C should give crystallography a major step forward by providing significant enhancement in incident beam intensity, short ranges of wavelength thus covering large reciprocal space, and the ability to select the wavelength of monochromatic radiation. Any developments made at laboratory facilities can be applied in this station, but with improved X-ray source and the scaled-up diffractometer. These changes allow reduction of the sample crystal size to less than 10 μm, and substantially shortened the data collection time.

In high-resolution powder diffraction, advances have made possible detailed structure refinements (Cox, 1991; Jephcoat *et al.*, 1999). With the flexible detector arm of the diffractometer in 13-BM-C, point-counting detectors can be used. The advantages of point detectors over area detectors include (i) the diffracted beam can be collimated so as to reduce background arising from anvils and thus maximize the signal-to-noise ratio in intensity, and (ii) the sample–detector distance can be sufficiently large for high-resolution X-ray data. These advantages are particularly beneficial for studies of materials with low symmetry or systems with multiple phases.

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