A New Facility for High-Pressure Research at the Advanced Photon Source

Mark L. Rivers, Thomas S. Duffy,[1] Yanbin Wang, Peter J. Eng,
Stephen R. Sutton, and Guoyin Shen

Consortium for Advanced Radiation Sources, The University of Chicago

The Advanced Photon Source (APS) is a third-generation synchrotron storage ring that became operational in 1996. A national user facility at the APS is being constructed for research in Earth, soil, and environmental sciences by the GeoSoilEnviroCARS (GSECARS) group of the Consortium for Advanced Radiation Sources (CARS). The GSECARS sector consists of an undulator and a bending magnet beamline, both of which have been designed to allow for a wide range of possible experiments on geological materials. An experimental station on the undulator beamline will be dedicated to high-pressure experiments using a multi-anvil press and diamond anvil cells. Energy-dispersive and monochromatic diffraction experiments will be performed in this and other stations using solid state and two-dimensional detectors. A high-pressure support laboratory is being developed concurrently. The first high-pressure experiments at the GSECARS sector were successfully conducted in December, 1996.

1. INTRODUCTION

Many advances in the study of condensed matter at high pressure have been made possible through the development of high-intensity synchrotron X ray sources. The drive to study ever smaller and more complex samples at increasingly high pressures and temperatures and with higher accuracy probes is now being aided by the development of a new generation of synchrotron facilities at locations in Europe, Japan, and the United States. Compared with existing second-generation synchrotron facilities, these third-generation synchrotron sources produce radiation with higher energy and higher brilliance, both of which are advantageous for high-pressure experiments. In this paper, we describe the high-pressure research facility that is being developed for a new third-generation synchrotron near Chicago, Illinois.

A synchrotron storage ring consists of a vacuum ring with alternating straight sections and arcs in which bunches of charged particles (e.g., positrons) travel at nearly the speed of light. Magnets are used to steer and focus the particle beam, and energy losses are replenished by radiofrequency cavities. Bending magnets are used to deflect the particles into a circular arc. The inward acceleration causes an intense, well-collimated beam of x-rays to be emitted along a tangential path from the arc. Insertion devices, consisting of arrays of pairs of alternate polarity magnets, are used to extract even higher intensity radiation from straight sections of the ring by causing the beam particles to oscillate as they travel.

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[1] Now at Department of Geosciences, Princeton University, Princeton, NJ 08544.

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TABLE 1. APS Parameters

Property	Value
Energy	7.0 GeV
Beam particle	Positron
Beam current	>100 mA
Lifetime	> 10 hrs
Fill time	3.7 min
Horizontal emittance	7 nm rad
Vertical emittance	0.7 nm rad
Undulator brilliance at 8 keV*	2 x 10^{18}
Bending magnet critical energy	19.6 keV
Positional stability	20 μm
Directional stability	2 μrad
Number of bunches	1-60
Bunch duration	73 ps
Circumference	1104 m
Number of insertion devices	35

*In units of photons/sec/mrad^2/mm^2/0.1% bandwidth.

The two types of insertion devices are called wigglers and undulators. For a given magnetic field strength, a wiggler generates radiation that adds to give a combined flux that is N times greater than a bending magnet, where N is the number of pairs of magnets comprising the wiggler. An undulator uses a weaker magnetic field to produce smaller particle displacements than a wiggler. In this case, the emitted radiation interferes to produce a spectrum of harmonic peaks. The wavelength of the harmonic peaks can be tuned by changing the magnetic gap, and hence field strength. If the undulator gap is tapered, rather than uniform, then the harmonics are broadened, and the energy distribution can approach the smooth spectrum of a wiggler.

The emitted radiation from a bending magnet or insertion device is directed down a beamline, which consists of a set of evacuated pipes transporting the beam through a series of enclosures. A shielding wall separates the storage ring from the experimental floor and beryllium windows are used to isolate separate evacuated portions of the beamline and storage ring. The radiation is first transported through one or more optics enclosures which can contain a variety of components including shutters, X ray mirrors, monochromators, and beam diagnostics equipment. The beam then reaches experimental stations where the user-controlled experimental apparatus is housed. Detailed description of the characteristics and operation of synchrotron radiation facilities can be found in *Koch* [1983], *Buras and Gerward* [1989], *Finger* [1989], *Bassett and Brown* [1990], *Brefeld and Gürtler* [1991], and *Smith and Rivers* [1995].

The Advanced Photon Source (APS) is a 7 GeV storage ring that is now in operation at Argonne National Laboratory in Argonne, Illinois. The main characteristics of the APS are summarized in Table 1 [*Moncton*, 1996]. The design goal for the facility (a current of 100 mA with a lifetime of 10 hrs) has been achieved. The eventual performance is expected to be much better than this (e.g., 300 mA current with 100 hr lifetime and fill time of less than 30 sec) [*Moncton*, 1996]. Compared to bending magnet beamlines at existing second-generation sources, the APS will deliver an X ray brilliance that is at least 10^2-10^3 times greater. The narrow, intense, short-wavelength beam at the APS is especially well-suited to high-pressure experiments which are limited by small sample volumes and absorption in the anvil materials.

The APS is a large facility situated on a 79 acre site. The storage ring has a circumference of 1100 m. The experimental floor is divided into 35 sectors, each of which consists of an insertion device and a bending magnet beamline. The sectors are being developed by independent collaborative access teams which are typically composed of members from universities, national laboratories, and industry. In the present phase of development, 20 sectors are being constructed. In addition to the beamlines on the experimental floor, each sector has 1200 ft^2 of laboratory space and an office area of 1500 ft^2. A user residence facility for housing visiting researchers is now open.

The Consortium for Advanced Radiation Sources (CARS) is a collaborative access team that is constructing three sectors at the APS. The GeoSoilEnviroCARS (GSECARS) group is designing a sector for research in Earth, soil, and environmental sciences. The other CARS sectors will be devoted to biological sciences (BioCARS) and chemistry and materials science (ChemMatCARS). The primary experimental techniques to be used at the GSECARS sector will be single-crystal and powder diffraction (at ambient and high pressure), X ray absorption spectroscopy, X ray fluorescence microprobe analysis, and microtomography.

High-pressure experiments will be conducted at the APS using two types of pressure-generating devices: the diamond anvil cell (DAC) and the large-volume press (LVP). The large-volume press is characterized by relatively large sample volumes (0.1-1 mm^3) and good pressure and temperature stability. The diamond cell, on the other hand, is compact, can easily achieve much higher pressures and temperatures and is transparent to a broad range of electromagnetic radiation.

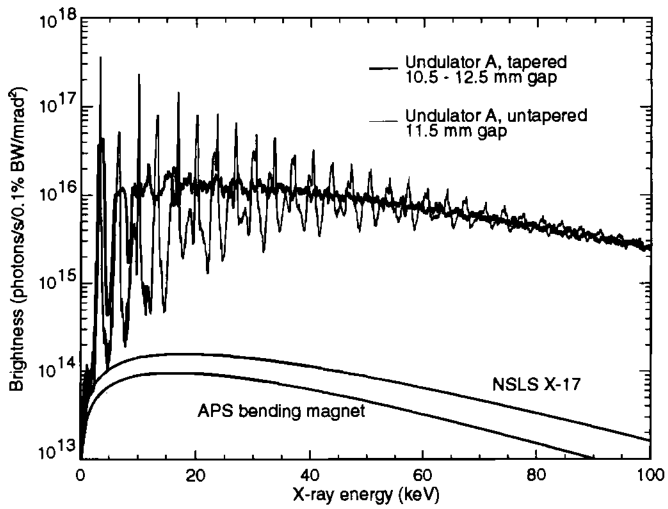

Figure 1. Brightness spectra for the APS undulator A and bending magnet. Spectra for the undulator are shown for both its normal configuration and using a tapered gap. For comparison, the spectrum of the superconducting wiggler (X17) at the NSLS is shown.

2. BEAMLINE COMPONENTS

The GSECARS sector consists of an undulator and a bending magnet beamline. The undulator is the standard APS undulator A, which is a 3.3-cm-period device. The first harmonic is tunable from 3.3 keV to 13 keV by opening the magnetic gap from 10.5 to 25 mm. In its standard configuration, the undulator produces a spectrum of harmonic peaks of approximately 1% FWHM. Harmonic peaks out to 100 keV are predicted and have been observed in the initial operation of the device. The undulator spectrum is ideal for monochromatic diffraction experiments. By changing to a tapered gap, the harmonic structure beyond 20 keV is nearly eliminated, and the spectrum resembles that from a wiggler but with higher on-axis brightness.

The spectrum from the bending magnet at the APS has a critical energy of 20 keV, which is the same at that of the superconducting wiggler at X17 of the National Synchrotron Light Source (NSLS). The bending magnet is an excellent source for energy-dispersive diffraction experiments when the brightness of the undulator is not required.

There are a number of possible figures of merit which can describe the characteristics of a synchrotron beam. The flux is the number of photons/sec/horizontal angle emitted in a relative energy bandwidth of $\delta\lambda/\lambda = 0.001$, integrated over the entire vertical angle. The brightness is flux/vertical angle which is essentially the number of photons/s per solid angle. This is relevant for experiments that use a pinhole or collimator to select a portion of the beam to interact with the sample. Finally, the brilliance is the brightness/source area. This is the appropriate measure when optics are used to focus the entire beam onto the sample. Figure 1 compares the brightness spectra for the APS undulator in tapered and untapered mode, the APS bending magnet, and the superconducting wiggler of the NSLS.

The layout of the GSECARS sector is shown in Figure 2. The two beamlines are referred to as 13-ID (insertion device) and 13-BM (bending magnet). Each beamline has 4 radiation enclosures: two optics enclosures (the A and B stations) and two experimental stations (the C and D stations). The two experimental stations of the bending magnet beamline are completely independent. The two stations on the insertion device beamline can operate independently for certain experiments and must share time for others.

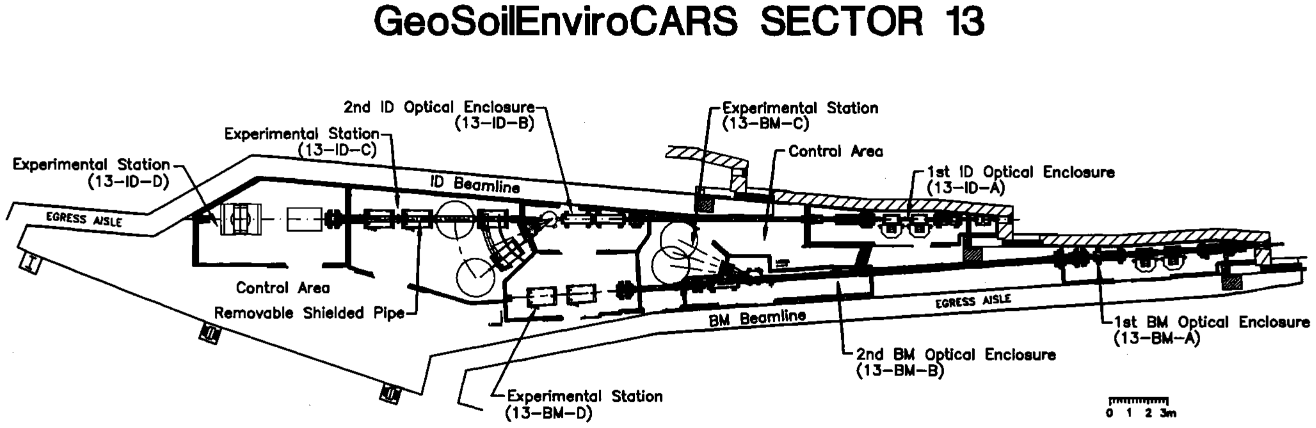

Figure 2. Layout of the GSECARS sector at the APS.

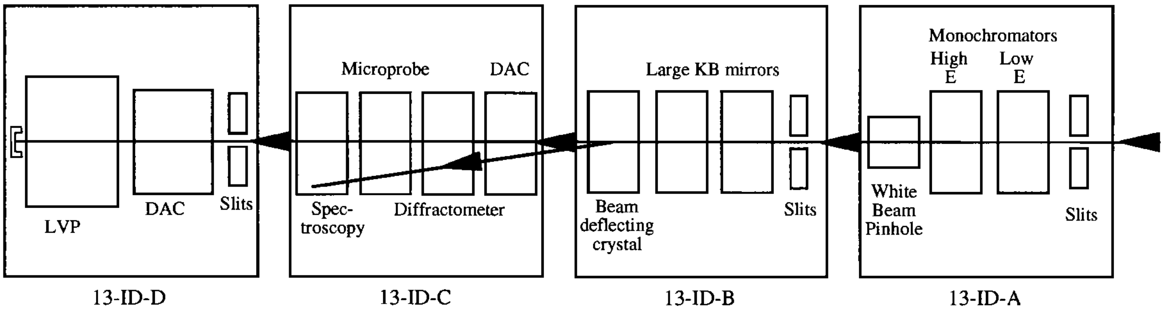

Figure 3. Schematic diagram of the main components of the insertion device beamline.

2.1 Insertion Device Layout

The first optics enclosure (FOE) of the undulator beamline (13-ID-A) will contain a set of primary slits, cryogenically cooled, high- and low-energy monochromators, a beam diagnostics tank, and a white beam pinhole. The low-energy monochromator will operate from 4.5 to 21 keV and the high-energy monochromator will be usable between 15 and 80 keV. The first crystal on the low-energy monochromator is thin, and the transmitted beam can be used directly or with the high-energy monochromator. Thus, the two monochromators can be used simultaneously. The low-energy monochromator produces a vertical +50 mm offset in the beam and the high-energy monochromator produces a -19 mm vertical offset. The pinhole is used to restrict the total white-beam power downstream of the FOE to less than 100 W (at 100 mA current) in order to simplify the power handling requirements of downstream optics. The pinhole dimensions are approximately 0.65 mm (vertical) x 1.1 mm (horizontal).

The second optics enclosure will contain additional slits, a pair of water-cooled ~1-m-long focusing mirrors, and a beam deflecting crystal. The large mirrors will be of the Kirkpatrick-Baez (KB) type: focusing is achieved by the grazing incidence reflection of a mirror bent to an elliptical shape. These mirrors will be capable of collecting the undulator beam (white or monochromatic) and focusing it to less than 100 μm. The actual demagnification ranges of the KB mirrors will be from 10:1 to 3:1 in the 13-ID-C and 13-ID-D stations. The Ge (111) beam deflecting crystal is capable of deflecting the low-energy monochromatic beam sideways into the 13-ID-C experimental station. The use of this crystal allows for simultaneous operation of the two experimental stations on the insertion device beamline. A schematic of the insertion device beamline components is shown in Figure 3.

Two experimental stations are planned for this beamline. The upstream station (13-ID-C) can be used in focused or unfocused white-beam mode for energy-dispersive experiments in the DAC. It can also be used for angle-dispersive DAC experiments. The end station (13-ID-D) will be dedicated to high-pressure experiments with a permanently installed large-volume press and an optical table for diamond cell experiments. Both monochromatic and white-beam experiments using either focused or unfocused beams will be performed here.

2.2 Bending Magnet Layout

The bending magnet beam is split into two independent beamlines (Figure 4). In 13-BM-A, the 6-mrad bending magnet fan is split by a fixed aperture plate into two components: a 2.5-mrad outboard fan for 13-BM-D and 1.5-mrad inboard fan for 13-BM-C. The first optics enclosure also houses slit tanks, high- and low-energy monochromators (for 13-BM-D), and a conical mirror for 13-BM-C. The second optics enclosure contains a bent flat vertically focusing mirror for 13-BM-D and a single bounce, horizontally focusing monochromator for 13-BM-C. The first station (13-BM-C) is a side station which is primarily dedicated to powder and single-crystal diffraction at 7.5-20 keV. The end station (13-BM-D) can use white beam, or radiation from either the high- or low-energy monochromators with sagittal focusing and a bent flat vertical focusing mirror. Both white and monochromatic diffraction experiments using either the diamond cell or the large-volume press can be carried out in the bending magnet end station.

3. HIGH-PRESSURE EXPERIMENTAL PROGRAM

High-pressure experiments generally require very small beam size, short-wavelength photons, and a high-brilliance source. Small X ray beams (~10 μm) are particularly needed for DAC experiments to minimize the effects of pressure and temperature gradients and to avoid signal from the gasket or pressure-transmitting medium. Absorption in the anvils requires the use of

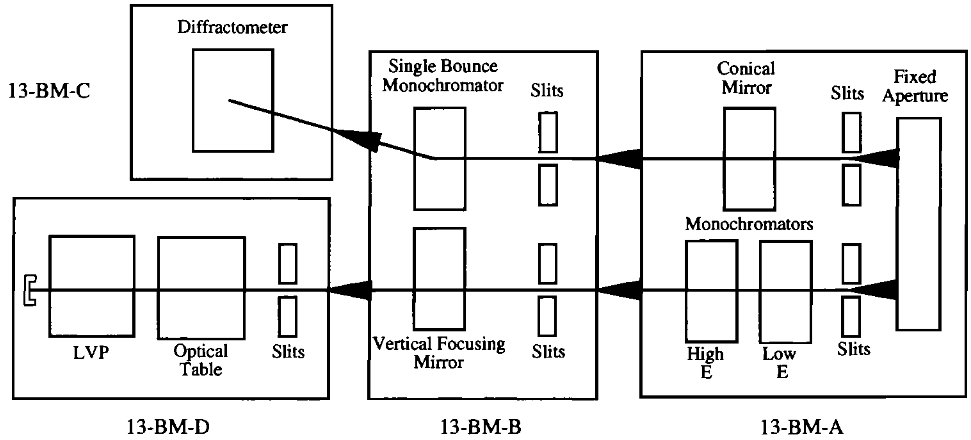

Figure 4. Schematic illustration of the primary components of the bending magnet beamline.

photons with energies above 15 keV for the diamond cell and above 20 keV for the large volume press. The extremely small volumes which can be held at high pressure (micro- to nanoliters for the large-volume press, nano- to picoliters for the diamond cell) requires the use of the highest brightness X ray source possible. The primary scientific goal of the high-pressure experimental program at the APS will be to significantly extend the range (e.g., pressure and temperature) of feasible high-pressure experiments. At the same time, there will be a major effort to improve the accuracy of the experimentally determined quantities.

3.1 Diamond Anvil Cell

Experimental stations for diamond cell research have been constructed at a number of second-generation synchrotrons [e.g., *Olsen*, 1992; *Brister*, 1992; *Hu et al.*, 1994; *Ancharov et al.*, 1995] and are currently being developed for third-generation sources at the European Synchrotron Radiation Facility [*Häusermann and Hanfland*, 1996], Spring-8 in Japan [*Shimomura et al.*, 1992*a*], and the APS. The diamond cell program at the APS will be divided into two phases. The first phase will use existing techniques (primarily energy-dispersive diffraction) to carry out frontier experiments utilizing the high brightness and coherence of the third-generation source. The second phase of experiments will use newer techniques (e.g., angle-dispersive diffraction, area detectors) for high precision studies.

A small Kirkpatrick-Baez mirror system has been developed for providing very small (<10 μm) X ray beams for diamond cell experiments [*Yang et al.*, 1995; *Eng et al.*, 1995]. These mirrors consist of 100-mm long Pt- or Rh-coated glass bent to an elliptical shape with a two-moment bender. In initial tests of this system, a 70 x 70 μm collection area was focused to a 3 x 9 μm focal spot with an efficiency of greater than 90% for wavelengths greater than 0.2 Å [*Eng et al.*, 1995].

An energy-dispersive diffractometer for diamond cell experiments has been constructed (Figure 5). This instrument consists of a two-circle horizontal diffractometer mounted on an optical table. The sample stage has both motorized and manual x-y-z translations and ω and χ rotations. A kinematic base is used for reproducibly positioning the diamond cell. An optical microscope with CCD camera is available for sample viewing. The incident X ray beam is controlled by a pair of adjustable slits made from WC cubes (from a multi-anvil device). Beam sizes down to 10 μm can be obtained in this way. The small KB mirrors are used to produce focused beams of less than 10 μm. The detector rotates

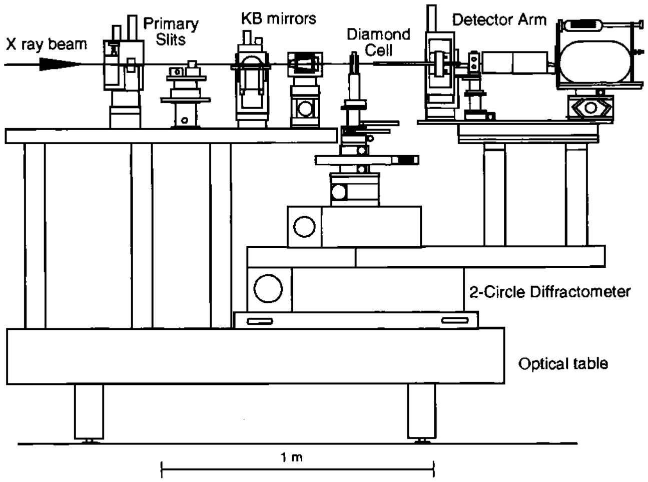

Figure 5. Energy-dispersive diffractometer for the high-pressure experimental station.

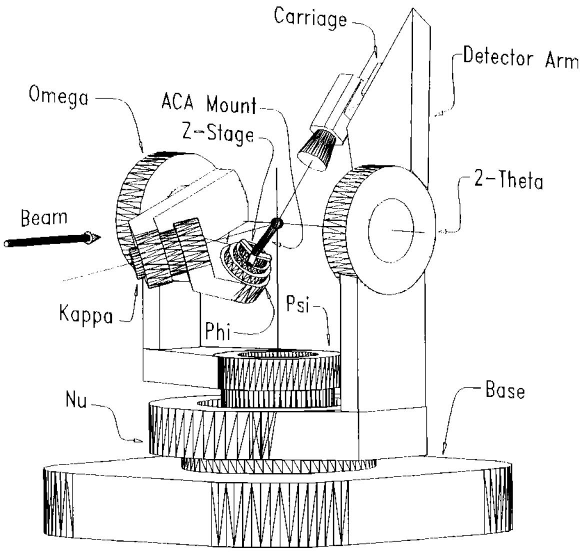

Figure 6. General layout for the 2+2+Kappa diffractometer for micro-crystal and diamond anvil cell experiments.

on a 2θ arm that has the same center of rotation as the sample stage. Two sets of slits define the diffraction angle and exclude scattered radiation from the diamonds and gasket. The diffracted intensity is recorded with an energy-sensitive solid state detector and multichannel analyzer. Overall, the system is similar to a previous one developed for beamline X17C of the NSLS [*Hu et al.*, 1994], but has been scaled up for increased stability and to handle larger and more complex sample assemblies.

The diffractometer was tested at X17B of the NSLS with a novel laser heating system [*Mao et al.*, this volume] consisting of a 100 W multimode YAG laser, optics to heat the sample from both sides, temperature stabilization, and a two-dimensional CCD for temperature measurement. This combined laser heating-energy dispersive diffraction system has been used to obtain *in situ* structural data on metals, alloys, and silicates to temperatures in excess of 1700 °C and pressures above 90 GPa (G. Shen et al., manuscript in preparation).

In addition to the two-circle diffractometer, a multi-axis diffractometer will be available for both energy dispersive and angle dispersive diamond cell diffraction. This diffractometer represents a new design, and is optimized for performing diffraction measurements on small samples contained in complex sample environments. The sample cradle consists of a fine motorized x-y-z stage capable of supporting large sample environments weighing up to 20 kg with negligible loss in sample centering over a wide angular range. The sample stage is also designed to accommodate a high-power liquid He and N_2 flow cryostat capable of rapidly cooling an object the size of a diamond anvil cell to 4.2 K. The stage is integrated into a Kappa goniometer with an inclination angle of 50°. To accommodate the restricted scattering geometry imposed by diamond anvil cells, the diffractometer has two additional degrees of freedom compared to a classic Kappa diffractometer. There is one additional horizontal ψ circle with a vertical rotation axis located below the sample cradle, and a second similar but independent ν circle located below a traditional horizontal axis 2θ detector circle. The geometry is referred to as 2+2+Kappa (Figure 6).

Having two degrees of freedom on the detector arm 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. This reduces the range of sample rotations needed, resulting in better sample centering stability for monochromatic measurements, and can completely eliminate the need to rotate the sample for energy dispersive single crystal experiments. The detector arm can carry loads up to 40 kg without loss of centering and this allows for the possibility of mounting, at the same time, both an area detector and an energy dispersive detector for micro single crystal energy dispersive diffraction.

3.2 Multi-anvil Press

Multi-anvil high-pressure devices have been used extensively at second- generation synchrotron sources such as the Photon Factory, Tsukuba, Japan [*Shimomura et al.*, 1992*a*,*b*] and the National Synchrotron Light Source [*Weidner et al.*, 1992]. A wide range of experiments have been carried out, including solid-state and melt phase equilibria, P-V-T equation of state, strength and other rheological properties, as well as crystallography using monochromatic X rays. New multi-anvil press beam lines are being developed at Daresbury and Spring 8 [*Shimomura et al.*, 1992a].

The multi-anvil press at the APS will be capable of generating pressures beyond 30 GPa and temperatures up to 2800 °C. Our design philosophy for the large-volume presses is to decouple the hydraulic presses, which are used for applying the force for pressure generation, from the pressure tooling which can have various pressure generating mechanisms. Specifically, we will utilize two major types of pressure tooling, both having a long track record in large-volume high-pressure research. The first is the so-called DIA, which consists of the upper and lower pyramidal guide blocks (bolsters) installed on the heads of the hydraulic press rams, four trapezoid end blocks (thrust blocks), and six anvil holders, as indicated in Figure 7a. The inner surfaces of the guide blocks form a tetragonal pyramid. Two of the six anvils are along the center line of this pyramid and are fixed opposite to each other on each guide block. The other four anvils are horizontally located on the midpoints of the square edges of a bipyramid. This results in the formation of a cubic nest bounded by the flat faces of six anvils. A ram force applied along the vertical axis is thus decomposed into three pairs acting along three orthogonal directions, forcing the six anvils to advance synchronously toward the center of the cube.

The second is the so-called T-Cup, recently developed at the Center for High Pressure Research and Stony Brook [*Weidner et al.*, 1996]. This is a two-stage system. The first stage is a tool steel cylinder split into six parts, each with a corner truncated into a square, enclosing a cubic cavity (Figure 7b), which contains the second-stage anvil assembly. Conic slots are made on two of the first- stage pieces to allow X rays to enter and exit. The second stage is assembled outside the press and consists of eight cubes (WC or sintered diamond) separated by preformed gaskets and spacers. Each cube has one corner truncated into a triangular face; the eight truncations create an octahedral nest in which the pressure medium is compressed. Incident and diffracted X rays can either pass through the gaps between the anvils or through the anvils directly when sintered diamond anvils are used.

A 250-ton DIA-type cubic anvil apparatus will be installed at the end station of the bending magnet beam line. This apparatus will allow most kinds of experiments currently being conducted at second-generation synchrotron sources. A 1000-ton press will be installed at the end station of the insertion device beam line and will be capable of using various high pressure tooling for much wider range of experiments. For example, an eight-cube anvil assembly may be placed within a DIA; using sintered diamond cubes, this 6/8-in-DIA system has proven capable of generating over 35 GPa and 2000 °C [e.g., *Kato et al.*, 1992]. The 1000-ton capacity also makes it possible to develop a modified DIA tooling, where the top anvil is driven independently by a smaller hydraulic jack, installed within the top guide block. This configuration allows a controlled differential stress field to be applied to the sample; with X rays as a probe, deformation experiments can be conducted at conditions of pressure and temperature.

Both the bending magnet and insertion device beam lines are capable of switching between energy-dispersive and monochromatic modes. The two large-volume ap-

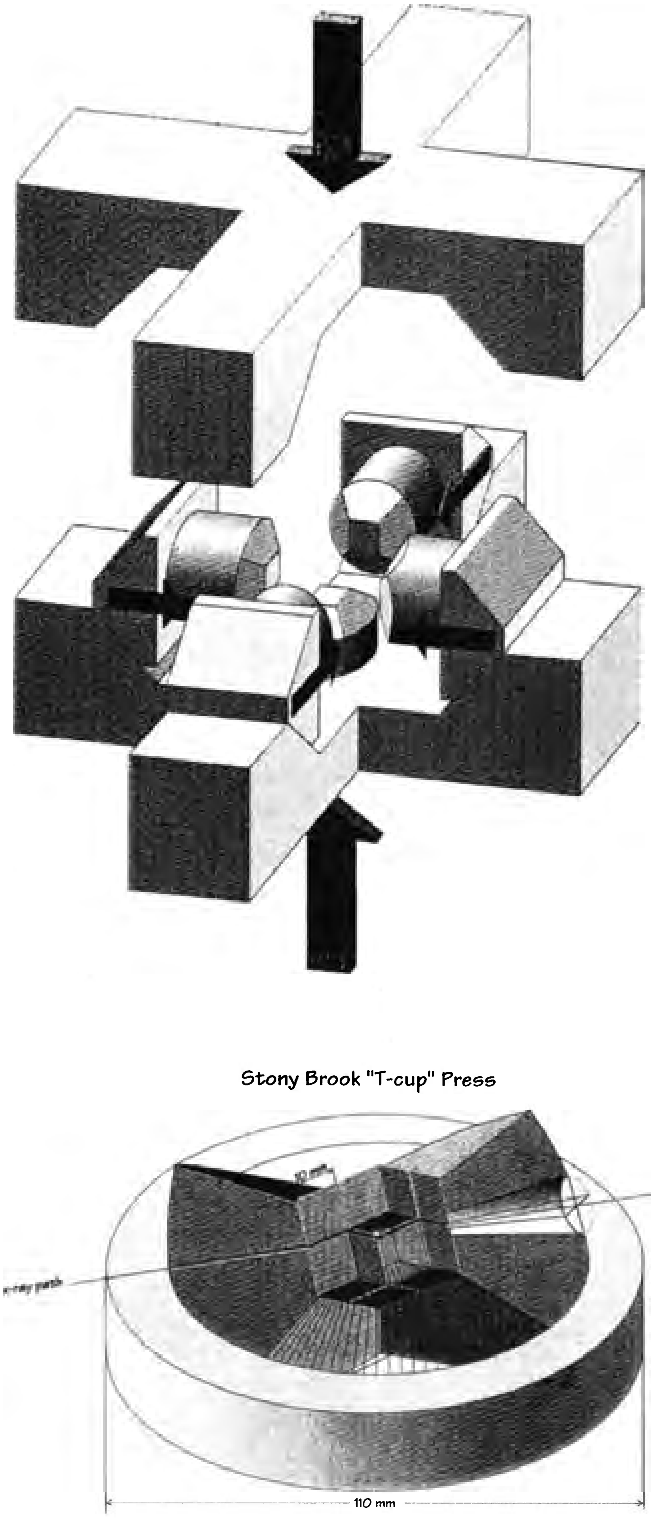

Figure 7. Pressure generating mechanisms of large volume press. (a) DIA arrangement, (b) T-cup assembly.

paratus will be able to operate with both modes. Energy-dispersive diffraction can be performed using a single or multi-element solid state detector, while angle-dispersive diffraction can be carried out using an image plate or a CCD detector.

3.3 Support Laboratory

In addition to the experimental stations, a high-pressure support laboratory is being constructed within the sector laboratory space. This laboratory will contain a variety of diamond cells including Mao-Bell, Merrill-Bassett, membrane, and inconel cells for resistive heating. Sample preparation and characterization facilities will include microscopes, a micromanipulator, glove box, mechanical microdrill and an electric discharge machine. A 3-kbar gas loading facility will allow for loading diamond cell samples which are gases at ambient conditions. A portable optical spectrometer will be used for calibrating pressure by the ruby fluorescence method. Micro-Raman spectroscopy will be carried out using a spectrograph equipped with CCD detector. Cryostats will be available for low-temperature diffraction studies.

There will also be on-site machine shop facilities for trained users and GSECARS staff. GSECARS will also provide office space and computer hardware and software for users to carry out complete on-site data analysis after their beamtime is over.

The existence of this laboratory will make it possible to carry out complete high-pressure experiments at the APS, rather than solely relying on samples prepared at the home institution. Furthermore, it makes it possible to carry out collaborative work between GSECARS staff and scientists who do not have high-pressure capabilities at their home laboratory.

4. RECENT PROGRESS AND FUTURE DIRECTIONS

First light at the CARS sector of the Advanced Photon Source occurred on September 17, 1996 in 13-BM-A. After completion of shielding verification, the first experiments within the CARS sector were carried out in this first optics enclosure of the bending magnet beamline in December, 1996. In these experiments, gold and rhenium were compressed in a diamond cell to 42 GPa. A beryllium gasket and a side-diffraction geometry were used to record the variation of d-spacing as a function of angle from the diamond cell stress axis.

The first optics enclosure of the insertion device beamline is expected to become operational in January, 1997. It is currently estimated that the first experimental stations (13-BM-D, 13-ID-C) will be in operation by summer, 1997. The endstation of the insertion device beamline (13-ID-D) is expected to be ready for white beam experiments at the end of 1997.

The GSECARS sector at the APS is a national user facility for research in Earth, soil, and environmental sciences. Beamtime will be assigned on the basis of competitive proposals. Those proposals that take advantage of the unique characteristics of third-generation sources will receive the highest priority.

Among the scientific goals of the high-pressure program at GSECARS is the study of crystal structures across the entire pressure-temperature spectrum of the terrestrial planets. Other major areas of focus will be on achieving ultra-high pressures with powders and single crystals, study of equations of state and phase transitions, high-pressure rheology, and high-accuracy pressure calibration at simultaneous high temperatures. Over the longer term, experiments will focus on complex problems such as accurate structural and electron density determinations at simultaneous high P and T, hydrothermal reactions, rheological properties, structural studies of melts and glasses, cryogenic studies, phase equilibria of multicomponent systems, and kinetics of phase transitions.

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