High Brilliance and High Pressure:
A New Diamond Anvil Cell Facility at the Advanced Photon Source

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High energy and high brilliance make the Advanced Photon Source (APS) an ideal tool for high pressure studies. In this paper, we describe the diamond anvil cell (DAC) facility that is being developed for the APS by the GeoSoilEnviroCARS group of the Consortium for Advanced Radiation Sources (CARS). The DAC program aims at studying properties of materials across the entire pressure-temperature spectrum of the terrestrial planets by a variety of experiments. Recent progress and the design concept are presented.

[diamond anvil cell, synchrotron radiation, laser heating, x-ray diffraction, high pressure and temperature]

1. Introduction

Ultrahigh pressures and temperatures in the diamond anvil cell are achieved at the expense of reducing sample volume. Characterization capability with high spatial resolution is most fundamental for probing micro-sized samples at high pressure and temperature and for minimizing the effect of gradients. High pressure experiments using x-ray techniques require the highest possible x-ray brilliance, and it is thus evident that high brilliance synchrotron radiation sources such as the APS, a new third generation synchrotron, are ideal for precise and accurate x-ray measurements. We describe the DAC program at GSECARS at the APS near Chicago and report recent progress and developments.

2. Diamond Anvil Cell Program

The DAC program is a major part of high pressure experimental programs at GSECARS [1,2]. The scientific goal of the program is to study properties of candidate materials across the entire pressure-temperature spectrum of the terrestrial planets by a variety of high pressure experiments, including

single crystal and polycrystalline x-ray diffraction,
diffraction at in situ high pressures and high temperatures,
high resolution x-ray diffraction,
hydrothermal reaction measurements,
elastic strain measurements,
x-ray diffraction of amorphous materials,
three dimensional structural microprobe,
time resolved x-ray diffraction,
x-ray absorption spectroscopy,
x-ray fluorescence microprobe, and
x-ray Raman and inelastic scattering.

Technically, the goal is to significantly extend the range (e.g., pressure and temperature) of feasible high pressure experiments. At the same time, there is a major effort to improve the accuracy of the experimentally determined quantities.

3. Facilities and Techniques

Beamline components

The GSECARS sector consists of an undulator (13-ID) and a bending magnet (13-BM) beamline. Each beamline has two experimental stations, and each of which is designed to be able to receive all kinds of beams (white, monochromatic, focused, and unfocused). The undulator is the standard APS undulator A which is a 3.3-cm-period device and is ideal for monochromatic diffraction experiments. When in tapered mode, the spectrum is smooth above 25 keV, and the on-axis brightness is greater than that derived from Wiggler A to energies beyond 100 keV. This makes the tapered undulator an excellent source for energy dispersive (ED) diffraction experiments.

To exploit the new third generation source, several kinds of focusing mirrors are employed. A pair of bent flat mirrors in a Kirkpatrick-Baez (refereed as 7:1KB) geometry are installed, which demagnify the source by 10:1 to 3:1 depending on location in the experimental stations. When a beam size greater than 10 microns is acceptable, the 7:1KB mirror produces the advantage of larger flux. For smaller beam sizes, microfocusing KB optics will be employed. The small KB mirror system consists of 100-mm long Pt- or Rh- or Au-coated glass bent to an elliptical shape with a two-moment bender. In initial tests, a 70x70 μm collection area was focused to 3x9 μm focal spot (demagnification of 180:1) with an efficiency of greater than 90% for energies below 70 keV [3].

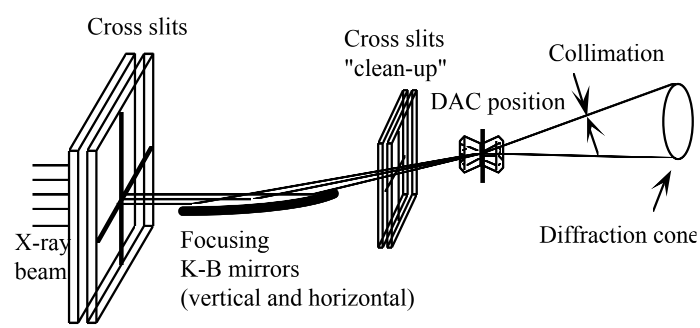


Fig. 1. A geometry for DAC x-ray diffraction experiments

Figure 1 shows a geometry for DAC experiments. The incident beam is controlled by a pair of adjustable slits made from WC cubes. Beam sizes down to 10 μm can be obtained in this way. The automated slit system also serves as the control of the incident beam to the small focusing mirrors with which beams are focused down to less than 10 μm. A “clean-up” slit system is introduced to produce “clean” focused spots required for very high pressure, low-Z material, and amorphous material experiments.

DAC stage

The DAC stage is designed to have high stability and high resolution of motion. It is based on a two-circle horizontal diffractometer (Huber 440 and 480, 0.001^o angular resolution) mounted on a lift table which is adjustable in position, height and tilt with position accuracy of 0.5 μm. The lift table is required to adopt the different positions and directions when the beam is changed from white to monochromatic or from focused to unfocused beams. The sample stage has motorized x-y-z translations and ω and χ rotations. The transitions have position resolution of 0.1 μm, and rotations have angular resolution of 0.001^o.

In addition to the two-circle diffractometer, a multi-axis diffractometer will be available for both ED and angle dispersive (AD) diamond cell diffraction. This diffractometer is optimized for performing diffraction measurements on small samples in DAC. The sample cradle consists of a motorized x-y-z stage capable of supporting DAC system weighting up to 20 kg with negligible loss in sample centering over a wide angular range. The sample stage is also designed to accommodate a cryostat capable of rapidly cooling DAC to 4.2 K. To accommodate the restricted scattering geometry imposed by DAC, the diffractometer has two additional degrees of freedom compared to a classic Kappa diffractometer. Thus it is referred as "2+2+Kappa". Having two degrees of freedom on the detector arm allows diffraction planes to take on an arbitrary orientation and removes the need to rotate the diffraction vector of the sample, resulting in better sample centering stability. The detector arm can carry loads up to 40 kg, allowing for mounting all kinds of detectors including the heavy multi-element detectors.

Detecting technique

Both energy dispersive (ED) and angle dispersive (AD) techniques are employed. An ED diffractometer for DAC experiments has been constructed [1]. ED is a well-established technique, and is fast and able to achieve excellent collimation and spatial selection. The latter is especially important to have good signal-to-noise ratios in high pressure studies because of inevitable sample surroundings. In addition, ED allows use of white beam with the highest possible flux with focusing mirrors. These make ED suitable for exploring ultra high pressures, to study low-Z materials, and to perform fast collection and time resolved measurements. The excellent spatial selection also makes a three dimensional structural microprobe possible (e.g., to study small inclusions in diamonds). Although ED is limited by the intrinsically low energy resolution of the solid state detector ($\Delta E/E$ ~1% FWHM), the peak positions are typically determined to an accuracy of $\Delta d/d<0.1\%$. Therefore, ED has been successfully used for determination of equations of state of known structures [4,5]. The peak broadness is a drawback for resolving overlapping peaks in polycrystalline diffraction. However, this is comparable or even smaller than the broadening from other sources such as nonhydrostatic stress and P-T gradients.

High resolution technique (AD) is used in the cases where it is beneficial with the intensity information and/or the intrinsic resolution. AD is a technique in development stage. The most reliable intensity data so far is from the area detector, as demonstrated by the imaging plate (IP) techniques [7]. Improving signal-to-noise ratio is a challenge, however, with area detecting techniques, especially for materials with low scattering power or at ultra high P-T conditions.

Conical diffraction geometry has been used with ED for DAC experiments [8] and it is well matched to the DAC geometry and synchrotron radiation conditions. Combining the conical diffraction geometry with a multi-element detector allows measurement of x-ray diffraction at different directions relative to the loading axis simultaneously, thus making it possible to monitor the elastic strain of a sample under uniaxial stress conditions without a need to rotate the cell to collect each direction. The conical geometry can also be combined with area detectors to reduce the background and, improve the signal-to-noise ratio.

DAC technique

DAC as a high pressure tool has been successfully coupled with different temperature techniques, with temperatures ranging from a few Kelvin to 6000 K with cryostat, external heating, internal wire heating, and laser heating techniques. At GSECARS, all these temperature techniques will be employed. For example, an in situ laser heating system is one of the major features. The system is based on double-sided laser heating technique [9] (Fig. 2) and consists of two YLF or CO_2 lasers, optics to heat the sample from both sides, temperature stabilization, and spectroradiometric temperature measurement system. With the heating system, for example, uniform temperatures of 3000 K ± 50K can be obtained in a high pressure sample of 20 μm in diameter and 10 μm thickness [9]. When combined with the fine (<10 μm) x-ray microprobe, the temperatures are uniform within the sampled region. The combined laser heating/energy dispersive diffraction system has been used to obtain in situ structural data on metals, alloys, and silicates to temperatures of 4000 K and pressures above 90 GPa.

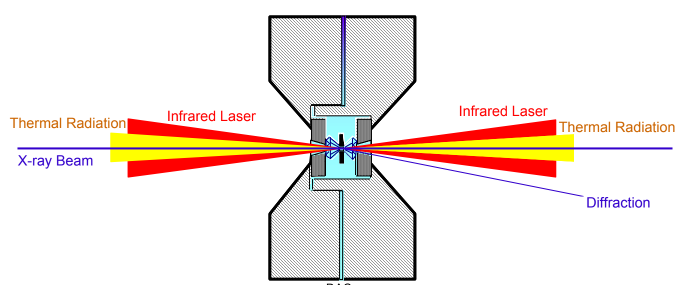
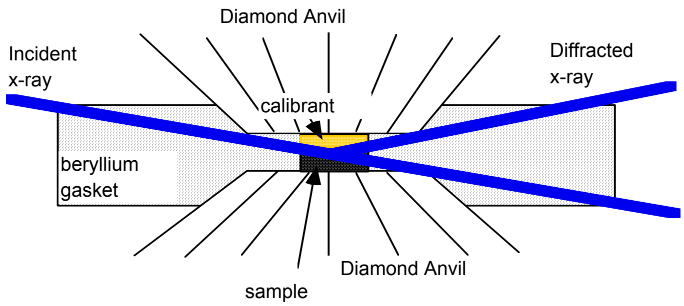
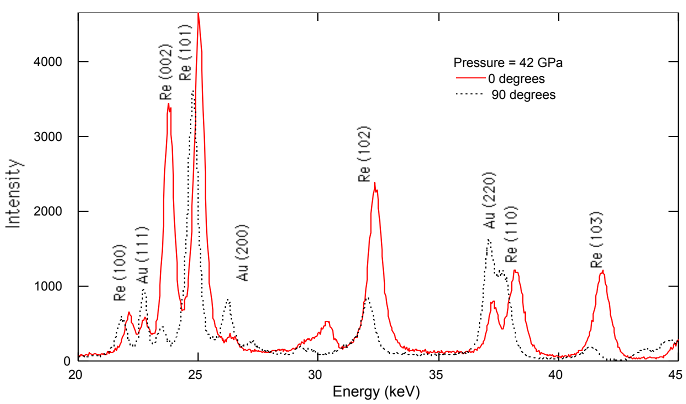


Fig. 2. Schematic illustration of double-sided laser heating with diamond anvil cell. A symmetric cell is suitable for this application.

Accurate P-V-T equations of state and crystal structures are typically measured with x-ray diffraction of samples in a hydrostatic environment. The DAC has the unique capability of being capable of containing gases and fluids as hydrostatic pressure-transmitting media. DAC has also the unique geometry of generating uniaxial stress. The orientation and magnitude of the strain ellipsoid can be mapped out by varying the x-ray diffraction direction relative to the loading axis by using high strength but x-ray transparent gaskets [10]. The determination of sample's strain/stress conditions in DAC has been currently one of major approaches at GSECARS since the first experiment in December 1996 (Fig. 3).



(a)



(b)

Fig. 3. (a) An example of side diffraction geometry. (b) Energy dispersive diffraction patterns of Re and Au at 42 GPa at the angle between the diffraction vector and the diamond cell loading axis with 0 degree (solid line) and 90 degrees (dotted line).

X-ray diffraction of polycrystalline and single crystal material at high pressures has been studied successfully with DAC. Extreme pressure conditions can only be achieved with very small sample volumes and it requires an excellent alignment and an optimized shape of anvils [11]. Single crystal diffraction requires hydrostatic media and access to a large portion of reciprocal space associated with the sample [5]. One of the goals of the DAC program at GSECARS is to extend the P-T range and explore the highest pressure for both polycrystalline and single crystal diffraction experiments.

Support laboratory

In additions to experimental stations, as described else where in this volume [2], a high pressure support laboratory is being constructed with the sector laboratory space. This laboratory contains a variety of DACs, sample preparation and characterization facilities. Major facilities include a micro-Raman spectrometer, gas loading system, glove box, mechanical microdrill and an electric discharge machine. The existence of the laboratory makes it possible to carry out complete high pressure experiments at the APS, rather than relying on samples prepared at the home institution.

4. GSECARS

GSECARS sector is a national facility. The program is actually operated and enriched by the large user community. Beam time at the GSECARS sector will be assigned on the basis of competitive proposals and those proposals that take advantage of the unique characteristics of the third generation sources will receive the highest priority.

Acknowledgment

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References

[1] M. Rivers, T. Duffy, Y. Wang, P. Eng, S. Sutton, and G. Shen, *High Pressure Temperature Research: Properties of Earth and Planetary Materials*, in press, (1997).

[2] Y. Wang, M. Rivers, S. Sutton, P. Eng, G. Shen, and I. Getting, this volume.

[3] P. Eng, M. Rivers, B. Yang, and W. Schildkamp, *Proc. SPIE*, **2516**, 41-51, (1995).

[4] H.K. Mao, Y. Wu, L.C. Chen, J.F. Shu, A. Jephcoat, J. Geophys. Res., **95**, 21737, (1990).

[5] P. Loubeyre, R.Toullec, D. Hausermann, M. Hanflank, L. Finger, R. Hemley, and H.K. Mao, Nature, **383**, 702, (1996).

[6] A. Jephcoat, L. Finger, and D. Cox, High Pressure Res., **8**, 667, (1992).

[7] Y. Fuji, K. Hose, Y. Ohishi, H. Fujihisa, N. Hamaya, K. Tekemura, O. Shimomura, T. Kikegawa, Y. Amemiya, and T Matsushita, Phys. Rev. Lett., **63**, 536, (1989).

[8] D. Hausermann, and J. Itie, Rev. Sci. Instrum., **63**, 1080, (1992).

[9] G. Shen, H.K. Mao, R. Hemley, *Advanced Material'96 - New trends in high pressure research,* 149, (1996).

[10] H.K. Mao, J.F. Shu, R. Hemley, A. Singh, American Geophysical Union, EOS transactions, **77**, f42, (1996).

[11] R. Hemley, H.K. Mao, G. Shen, J. Badro, P. Gillet, M. Hanfland, D. Hausermann, Science, **276**, 1242, (1997).

[12] William Bassett, Thomas Duffy, Dion Heinz, Russell Hemley, Ho-kwang Mao, Li-chung Ming, Guoyin Shen.