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A large-volume press facility at the Advanced Photon Source: diffraction and imaging studies on materials relevant to the cores of planetary bodies

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Abstract

A new large-volume, high-pressure facility is being utilized and developed as part of GeoSoilEnviroCARS at a third-generation synchrotron, the Advanced Photon Source. This user facility consists of two large-volume presses (LVP), a 2.5 MN (250 ton) LVP installed at the bending magnet beamline, and a 10 MN (1000 ton) LVP at the insertion device beamline. Here we report some techniques currently being developed with the 10 MN LVP and the latest scientific results obtained using the 2.5 MN LVP.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

GeoSoilEnviroCARS (GSECARS), a member of the Consortium for Advanced Radiation Sources (CARS) of the University of Chicago, is a synchrotron user facility at the Advanced Photon Source (APS) for Earth, planetary, soil, and environmental sciences. Our research goal is to advance knowledge of the composition, structure, and properties of the Earth and planetary materials as well as the dynamic processes these materials control. Techniques available at GSECARS include high-pressure experiments using both the diamond anvil cell and the large-volume press (LVP), x-ray diffraction and scattering, x-ray absorption spectroscopy, x-ray fluorescence microprobe, and microtomography. An overall description and general review of the GSECARS facility is given in [1]. The appendix gives details concerning how to apply for beamtime at GSECARS.

We reviewed the 2.5 MN LVP system, installed at the bending magnet (BM) beamline, and highlighted some of the scientific achievements at the last AIRAPT meeting [2]. In this

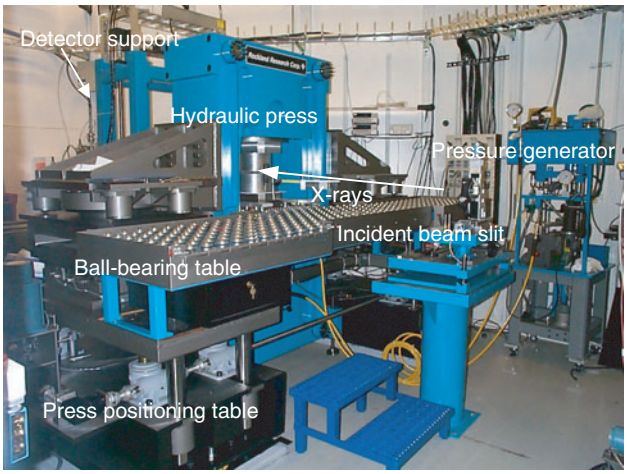

Figure 1. The 10 MN (1000 ton) LVP system at the insertion device station 13-ID-D, where either a white (by inclining magnet) or a monochromatic (to 60 keV) x-ray beam is available. Incident x-rays come from the right and are introduced to the sample through a slit system. The goniometer consists of two synthetic granite base blocks holding two columns of precision guide rails that are driven by geared stepper motors. The two columns are linked together through the press. Vertical screw jacks (four on each side) are linked together to drive the system in the vertical direction; horizontal rails in the direction perpendicular to the beam are linked whereas those in the direction parallel to the beam are independent. This allows the press to be translated in three orthogonal directions and rotated about the vertical axis. The multi-purpose detector support can be seen behind the entire LVP set-up.

paper, we report the current status of the 10 MN system on the insertion device (ID) beamline. We also introduce some of the unique techniques available on the BM beamline and highlight some results from materials relevant to the cores of planetary bodies.

2. 10 MN large-volume press on the ID beamline

In addition to the 2.5 MN (250 ton) press [2], the 10 MN (1000 ton) LVP (figure 1) was installed at the undulator beamline 13-ID-D in 2000. Currently these two presses are operational for both white and monochromatic (to 60 keV) x-ray experiments.

The 10 MN LVP, weighing 6 tons, is held by a 'goniometer' which is capable of locating the sample, which is compressed by the press, into the diffraction volume with three linear translations and one rotation degree of freedom with respect to the vertical axis. The positioning precision has been verified to be well within 5 μm for linear translation and 0.01° for rotation.

A five-axis multi-purpose detector support is able to accommodate a variety of detectors for the 10 MN LVP (figure 2): single- and multi-element solid-state detectors for energy-dispersive diffraction, scintillators for point counting, and an imaging plate and a CCD for one-dimensional and two-dimensional monochromatic diffraction. The base plate, on which each detector rests, is standardized with an X-95 rail system so that these detectors can be interchanged on a run-to-run basis. Three translations and two rotations allow any diffraction geometry to be uniquely defined; step scans with 2θ angles up to $\pm 25°$ can be performed along a great circle on any user-defined spherical surface. The detector is also able to accommodate the various diffraction geometries used for different pressure modules: DIA (a cubic anvil) [3] and T-cup (a split-cylinder) [4] apparatus.

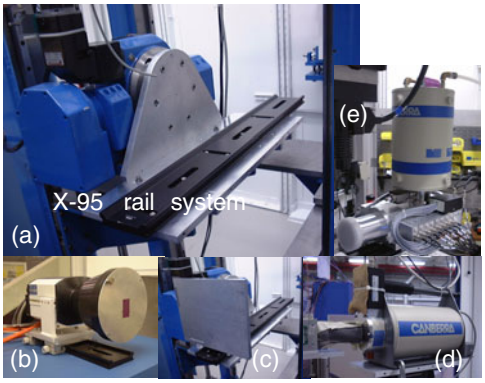

Figure 2. Variation of detectors available at GSECARS; (a) a whole view of the detector mount, (b) an x-ray CCD detector, (c) the imaging plate (holder), (d) single- and (e) multi-element solid-state detectors. The X-95 rail system enables interchanging the detectors on a run-to-run basis.

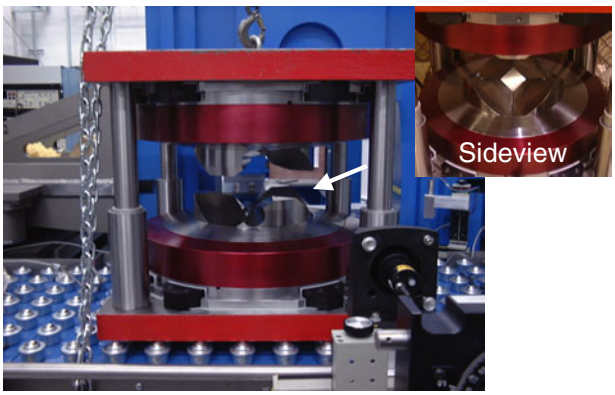

Figure 3. A split-cylinder high-pressure module T-25. The overall dimensions are approximately 560 × 560 × 410 mm (W × T × H). The truncated length of the first-stage anvils is 49.0 mm; these are designed to squeeze 25 mm cubes. The anvils along the x-ray beam have conical channels, allowing us to observe 2θ to $\pm 10°$. The arrow indicates the direction in which the 'side view' was taken.

Several pressure modules are used with the 10 MN LVP. A larger version of the T-cup (called T-25) with 25 mm WC cubes (figure 3) are used to generate up to 30 GPa and 3000 K on a 1 mm^3 sample volume. A double-stage module, with the MA8-type eight-cube assembly compressed in a large DIA is also adopted for ultrahigh-pressure experiments. A deformation DIA system [2] is currently being commissioned.

3. Research highlights

The 2.5 MN LVP has been fully functioning at station 13-BM-D since February 1998, enabling both diffraction and imaging capabilities. Over 270 experiments have been carried out by more than 35 outside users through multiple visits. The research topics pursued and experimental techniques used cover a wide range. Below are some examples of technical achievements and the new science concerning core materials.

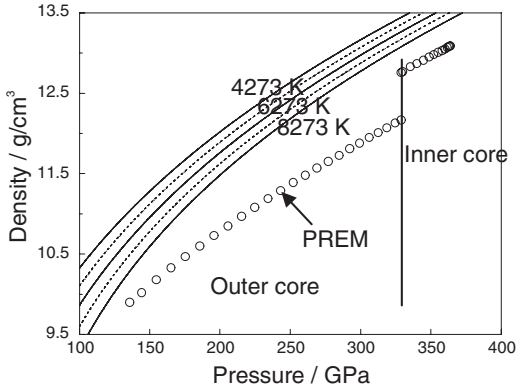

Figure 4. The density of ε-Fe at Earth's core conditions. Solid and dashed curves are obtained by extrapolation of the Mie–Grüneisen–Debye EOS. Solid curves indicate 4273, 6273, and 8273 K and dotted curves 5273 and 7273 K, respectively. Open circles are PREM densities. The ε-Fe densities are significantly higher than those for PREM, supporting the existence of light element(s) in both inner and outer core.

A double-stage split-cylinder (T-cup) apparatus with 10 mm WC cubes has been used to study phase relations and the equation of state of pure Fe [5] and FeS. Below 20 GPa, no new phase between the ε-Fe (hcp) and γ-Fe (fcc) stability fields was found and no anomalies in the c/a ratio were detected. Assuming that ε-Fe is stable at inner-core conditions, calculated densities from the extracted equation of state of ε-Fe are significantly higher than those for PREM (figure 4), supporting the notion that light element(s) must be present in the inner core, as well as the outer core [6].

Partitioning of Fe and Mg between perovskite and magnesiowüstite has been examined intensively [7–9]. The effect of light element(s) on the partitioning of Fe and Mg, however, has not been studied in depth. We simplified this model and have investigated the effect of light elements (H, S, Si, and C) on the Mg–Fe–O light-element system under lower-mantle conditions. Here we report the results obtained for the system containing S. The starting material was prepared by mixing controlled amounts of pure Fe, MgO, and pyrite (FeS_2), which is employed as the S source. Under high-pressure and high-temperature conditions, all of the d-spacings of MgO were observed to become larger at constant pressure (figure 5). Assuming that the amount of peak shift is ascribable to the Fe (not S) partitioning, the chemical composition can be estimated from the interpolation of the lattice parameters. With 9% of S (by weight), Fe metal can dissolve into MgO, forming ($Fe_{0.8}$, $Mg_{0.2}$)O. Subsequent chemical analysis using electron microprobe confirmed this conclusion. If this observation is applicable to the planetary bodies such as the proto-Earth, a core containing S may decrease its diameter resulting in an increase in Fe content in the lower mantle.

A DIA-type cubic anvil apparatus lets us collect information on the mechanism(s) by which light elements were incorporated in the core during early core formation. A typical cell assembly is shown in figure 6. A mixture of Fe (with controlled amounts of light elements, summarized in table 1) and silicate was compressed and heated. Radiographs were taken to record the Fe melt segregating from the silicate matrix or melt *in situ*. Microtomography was used on the recovered samples to examine the detailed texture of the samples non-destructively. The separation process varied depending on the light elements involved (figure 7). In the systems containing S or H, iron melt forms spherical droplets, which migrate rapidly through the silicate, forming large spheres at the bottom (figure 7, top). In the systems containing Si or C, a small channel-like structure is observed (figure 7, bottom) and no droplets are found

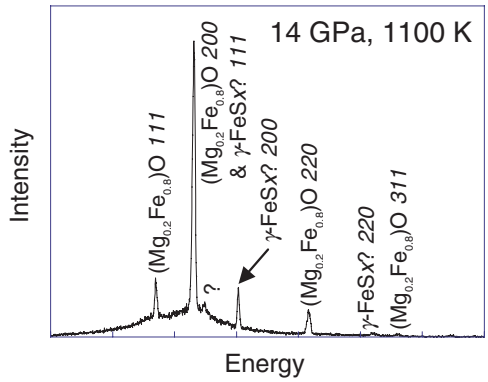

Figure 5. A representative x-ray diffraction pattern obtained at 14 GPa and 1100 K. Almost all of the peaks can be identified indicating a mixture of two fcc phases: one is similar to (Mg, Fe) O and the other to γ-Fe. Note that the intensity of the (Mg, Fe) O 111 peak is remarkably high compared to that for the end-member MgO, which is consistent with the assumption of the formation of (Mg, Fe)O. The high background indicates partial melting.

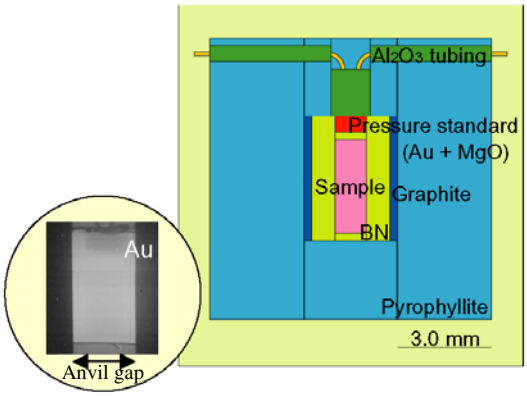

Figure 6. A cross-section of the cell assembly. A pyrophyllite cube with 9 mm edge length is employed as the pressure medium. The sample is surrounded by BN and is heated by the outer graphite tube. A radiograph at ambient condition (inset) shows homogeneous intensity before the melting occurs. The darker area in the sample chamber is an Au + MgO mixture.

Table 1. Starting materials. A mixing ratio of Fe:En = 1:1 by weight corresponds to 2:5 by volume.

Light element	Reagents	Mixing ratio (by weight)
H	Fe, $Mg(OH)_2$, SiO_2	FeH_x:En = 1:1, 2:1, 1:2, 1:4
S	Fe, FeS, $MgSiO_3$ (enstatite)	Fe 23% S:En = 1:1, 2:1, 1:2
Si	Fe 17% Si, $MgSiO_3$ (enstatite)	Fe 17% Si:En = 1:1, 2:1
C	Fe, C (graphite), $MgSiO_3$ (enstatite)	Fe 7% C:En = 1:1, Fe 15% C:En = 1:2

even at the highest temperature of 1773 K in the experimental timescale (about 1 h), although x-ray diffraction indicates that the iron alloy has been melted.

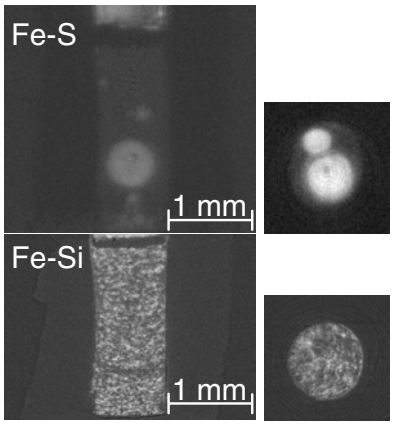

Figure 7. X-ray microtomographs of the recovered samples (top: Fe–S system recovered from 4.4 GPa and 1400 °C; bottom: Fe–Si system recovered from 3.4 GPa and 1500 °C).

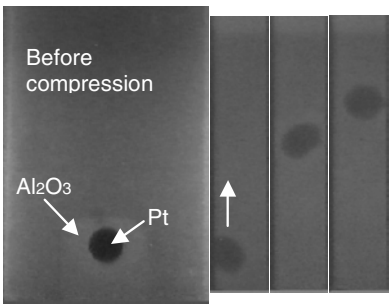

Figure 8. Representative radiographs for viscosity measurements [11]. The structure of the composite sphere [14] can be recognized by a small difference in shading. The time intervals are not identical.

The viscosity [10–13] and density of liquid Fe and its alloys [11] control the dynamics of the cores of planetary bodies. Viscosity measurements using an imaging system, therefore, providing fundamental information of the cores, which is also strongly linked to the above results. Unique and critical improvements have been made by Secco *et al* [11–14], who have developed a technique enabling a composite sphere to be used for viscosity measurement (figure 8). The composite sphere consists of an inner heavy metal core and an outer sapphire mantle, which inhibits the reaction between the core (metal) and the sample (Fe–S), and also enables us to control density precisely so that we can observe the sphere floating up (figure 8) or falling down [11], as desired.

Acknowledgments

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Appendix. The procedure for applying for beamtime at GSECARS

(a) Visit our Web site at http://gsecars.uchicago.edu.
(b) Contact a GSECARS staff scientist. The homepage lists experts for supported techniques.
(c) Apply for beamtime online through the Web page. We will e-mail in a few days, asking you to submit an experiment summary.
(d) Submit your experiment summary, a 2–3-page document.
(e) The proposal is reviewed.
(f) The beamtime award is made.
(g) The user contacts the assigned beamline scientist to make detailed arrangements.

References

[1] Rivers M, Duffy T, Wang Y, Eng P, Sutton S and Shen G 1998 *Properties of Earth and Planetary Materials at High Pressure and Temperature* ed M Manghnani and T Yagi (Washington, DC: American Geophysical Union) p 79
[2] Wang Y, Rivers M, Uchida T, Murray P, Shen G, Sutton S, Chen J, Xu Y and Weidner D 2000 *Science and Technology of High Pressure* ed M Manghnani, W Nellis and M Nicol (Hyderabad: Universities Press) p 1047
[3] Inoue K and Asada T 1973 *Japan. J. Appl. Phys.* **12** 1786
[4] Vaughan M, Weidner D, Wang Y, Chen J, Koleda C and Getting I 1998 *Rev. High Pressure Sci.* **7** 1520
[5] Uchida T, Wang Y, Rivers M and Sutton S 2001 *J. Geophys. Res.* **106** 21 779
[6] Jephcoat A and Olsen P 1987 *Nature* **325** 332
[7] Katsura T and Ito E 1996 *Geophys. Res. Lett.* **23** 2005
[8] Mao H-K, Shen G and Hemley R J 1997 *Science* **278** 2098
[9] Andrault D 2001 *J. Geophys. Res.* **106** 2079
[10] Dobson D, Crichton W, Vocadlo L, Jones A, Wang Y, Uchida T, Rivers M, Sutton S and Brodholt J 2000 *Am. Mineral.* **85** 1838
[11] Secco R, Rutter M, Balog S, Liu H, Rubie D, Uchida T, Frost D, Wang Y, Rivers M and Sutton S 2002 *J. Phys.: Condens. Matter* **14**
[12] Rutter M, Secco R, Uchida T, Liu H, Wang Y, Rivers M and Sutton S 2002 *Phys. Rev.* B at press
[13] Rutter M, Secco R, Uchida T, Liu H, Wang Y, Rivers M and Sutton S 2002 *Geophys. Res. Lett.* **29** 10.1029/2001 GLO 14392
[14] Secco R, Tucker R, Balog S and Rutter M 2001 *Rev. Sci. Instrum.* **72** 2114