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Laser Heated Diamond Anvil Cell at the Advanced Light Source Beamline 12.2.2

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Abstract

The laser heating system for the diamond anvil cell at endstation 2 of beamline 12.2.2 of the Advanced Light Source in Berkeley, CA has been constructed and is available for *in-situ* high-pressure high-temperature x-ray experiments. The endstation couples a high-brilliance synchrotron x-ray source with an industrial strength laser to heat and probe samples at high pressure in the diamond anvil cell. The system incorporates an 80 watt Nd:YLF (cw) laser operated in TEM01* mode. Double-sided heating is achieved by splitting the laser beam into 2 paths that are directed through the opposing diamond anvils. X-ray transparent mirrors steer the laser beams coaxial with the x-ray beam from the superconducting bending magnet (energy range 6-35 KeV) and direct the emitted light from the heated sample into two separate spectrometers for temperature measurement by spectroradiometry. Objective lenses focus the laser beam to a size of 25 micron diameter (FWHM) in the sample region. An x-ray spot size of 10 micron diameter (FWHM) has been achieved with the installation of a pair of focusing Kirkpatrick-Baez mirrors. A unique aperture configuration has produced an x-ray beam profile that has very low intensity in the tails. The main thrust of the program is aimed at producing *in-situ* high-pressure high-temperature x-ray diffraction data, but other modes of operation, such as x-ray imaging have been accomplished. Technical details of the experimental setup will be presented along with initial results.

Introduction

The laser heated diamond anvil cell (LHDAC) is a powerful technique for investigating the properties of materials at extreme temperatures and pressures in a static environment. Coupling this instrument with synchrotron-based x-ray diffraction enables the *in-situ* study of crystal structure (crystal-crystal phase changes, solid-liquid phase change, lattice parameter determination, texture, etc.) not possible with a standard laboratory x-ray source.

Synchrotron x-rays, graced with high flux, are able to collect a suitable diffraction pattern in a few minutes, which is the same order of time scale that one can hold a sample at a temperature of thousands of Kelvins. In addition, advances in focusing optics have allowed the tailoring of the x-ray spot size so that it fits neatly within a region of relatively constant temperature inside the laser heated hotspot. Implementation of superconducting bending magnets have made possible the generation of

hard x-rays (E > 2keV) at the Advanced Light Source in Berkeley, which started out as a soft x-ray source. These hard x-rays are crucial for scattering from the LHDAC sample, as they are sandwiched between diamonds that have an opaque region in the x-ray regime below ~10 keV.

The synchrotron based LHDAC can thus give insight into the behaviour of materials at extreme high temperature and pressure and has made significant contributions in the fields of geophysics and materials science. Recent research with this technique has given insight on the structure of the Earth's inner liquid core [1] and discovered an important new phase in the Earth's lowest-most mantle. [2,3].

Experimental

X-ray Generation, Conditioning and Detection

Endstation 2 (ES-2) of beamline 12.2.2 is situated approximately 1 meter downstream of Endstation 1 (ES-1) and uses an x-ray optic arrangement slightly modified from that of ES-1. Description of the primary (beamline) x-ray optics as well as that of ES-1 are described elsewhere [4]. Here we focus our attention on production of an approximately 10 x 10 micron x-ray spot situated at the at the ES-2 sample position.

The primary focus spot of the x-rays at ES-1 (0.06 mm (v) x 0.02 mm (h)) is re-imaged downstream onto a pair of Kirkpatrick-Baez (K-B) mirrors that demagnify 6:1 vertically and 2:1 horizontally, producing an approximately 10 x 10 micron spot at the sample position. The K-B mirror pair are made of a silicon substrate coated with 4 nm Rh and 25 nm Pt.

A unique aperture configuration of offset slits defining the virtual x-ray source for the K-B mirrors succeeded in producing a narrower focused x-ray beam than standard opposed slits as shown in Fig. 1; however, the beam profile was best modelled as a gaussian, rather than the flat top profile that was the design goal. The gaussian tails of the beam are attributed to scatter producing surface irregularities of the mirror on the order of 1 mm to 1 μm [5]. To prevent the beam tails from hitting the highly scattering gasket that surrounds the sample, a 1 micron Ta pinhole is placed approximately 10 cm upstream of the sample position. Traces of unfocused straight through x-ray beams (most noticeable at energies above 25 keV) were blocked by means of a 1 mm Pb pinhole placed at the exit of the K-B mirror tank.

A retractable pin diode is used for sample alignment. A sensitive large area detector (Mar 345 image plate) collects the x-ray diffraction pattern. A diffraction pattern from a typical silicate mineral can be collected on the order of 2 minutes, while a high-Z material like a metal can give a suitable pattern in 30 seconds. The image plate has a readout time of ~135 seconds.

Laser

We use a high-power infrared laser (Quantronix Falcon 217D, Nd:YLF, 50 W maximum power in TEM01*), commonly used in industry for cutting and welding, as the heating source for the high-temperature experiments. Our design goals for laser heating are large hotspots with small radial and axial temperature gradients. When these goals are met the x-ray beam will diffract from regions of the sample at relatively uniform high temperature and high pressure.

To achieve as uniform a temperature as possible we use the laser in TEM01* mode, which when combined with double sided heating, produces the lowest temperature gradients [6]. As shown in Fig.

2, the vertically polarized laser beam is first brought to a liquid crystal variable waveplate (Bolder Optik, USA) which rotates the polarization of the beam according to the applied voltage. Next the beam enters a polarizing beamsplitting cube, which passes the component of beam that maintains vertical polarization and dumps the remainder into a beamtrap (Kentek, USA). The liquid crystal has a fast response time and can be used in a feedback loop to stabilize the temperature of the sample. Because of the long distance (~4m) the laser beam travels to get to the sample, we have installed a beam expander to control the divergence of the beam. Next the beam is split into two paths that get reflected downwards onto the focusing objective lenses (80mm focal length, OptoSigma) and finally reflected along the x-ray beam path from both upstream and downstream direction via thinly Ag-coated amorphous carbon mirrors (Alfa Aesar). In this manner we achieve a hotspot of typical dimension of 25 micron in diameter, although this can be varied by adjusting the focus of the objective lens.

Temperature Measurement

Light from the sample (reflected, transmitted, and emitted) is reflected off of the same amorphous carbon mirrors used for laser delivery, collimated through the objective lenses, reflected off of total reflection mirrors, then sent through a 1 meter focal length lens and reflected onto a beamsplitter, sending light into a camera and onto an entrance slit of an imaging spectrometer (Acton 150i, Hammamatsu detector). The ratio of focal length of the objective and focusing lens (1m/0.08m) gives a total magnification at the detector of 12.5X, which results in the image of a 25 micron hotspot illuminating slightly more than 12 pixels of the detector (detector pixel size is 25 microns).

Accuracy and precision in determination of the temperature of the heated region of the sample is of utmost importance. Diamonds are the best conductor of heat known and thus the hotspot within the DAC is subject to tremendous temperature gradients. In order to minimize the gradients and heat effectively, care must be taken during sample preparation to adequately thermally insulate the heated material from the diamond anvils with the use of a non-reactive insulating material such as Al_2O_3 or NaCl.

Much attention has been paid to the validation of temperatures determined with this system. Temperatures are determined for each row of pixels in the detector by performing a weighted least-squares fitting of the observed signal from the spectrometer to the Planck radiation formula, after making corrections for the system response and wavelength dependent emissivity (when known) [7]. In this manner we are able to measure temperature profiles from the sample corresponding to the portion of the hotspot incident on the entrance slit of the spectrometer (typical slit width is 25 microns, corresponding to 2 microns of the sample). The spectrometer receives light from 450 – 900 nm, but the system response is highest from 570-800 nm. In order to minimize the effects of chromatic aberration of the long-working distance optics we typically limit our Plank fits to a wavelength range of 600-800nm and close down the f-stops directly in front of the refocusing lenses (I2 in Fig. 2) [8].

We used a variety of methods of validation of our measured temperatures against known standards of temperature: (a) Resistance heating to melt 5 pure metal (Ni, Fe, Pt, Mo, and W all >99.9%, Alfa Aesar, USA) filaments in order to verify ambient pressure melting temperatures gave an average discrepancy of 22K after applying intensity correction for wavelength dependent emissivities; (b) Double-sided laser heating of ambient pressure Fe and Pt foils (using visual and x-ray indicators as a check) gave a discrepancy of 40K for the Fe foil and 29K for the Pt foil; (c) Comparison of the high-

pressure melting temperature of Pt with a study done using visual observation of melt [9] is in progress. Data shown in Fig. 3 shows that the diffraction intensity of a sample of Pt sandwiched between two insulating layers of Al_2O_3 drops at high temperature and rebounds when quenching to room temperature. The drop in intensity is greater than can be accounted for due to the increased thermal vibration of the atoms (Debye-Waller factor), indicating that the x-ray beam was sampling regions of molten Pt. However, as no definitive melt signal (i.e. broad amorphous peaks) was present, more study is needed.

Applications

This system is optimized for reliable x-ray data collection from samples that are simultaneously at many GPa of pressure and with temperatures of many thousands Kelvin. Major applications will be in experimental mineral physics as well as physics of condensed matter.

Summary

The synchrotron-based laser heated diamond anvil cell installed at beamline 12.2.2 of the Advanced Light Source has been verified to produce *in-situ* x-ray data at simultaneous high-pressure and high-temperature (T>1500K, P>10GPa). The x-ray spot size is focused to approximately 10 x 10 micron spot and centered within a ~25 micron laser heated spot. Primary applications include experimental geophysics and novel materials synthesis.

References

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Figure Captions

FIG. 1. Micro-focused x-ray beam image taken with a $CdWO_4$ scintillator, shown with vertical and horizontal cross-sections taken along the dashed white lines. A two-dimensional gaussian fit to the two-dimensional image yields FWHM of 11.0 microns horizontally and 12.5 microns vertically. Profiles

indicated with filled circles; gaussian fits shown with solid lines; open circles in horizontal profile show best beam size for non-offset slits.

FIG. 2. Schematic of double-sided laser heating system at Beamline 12.2.2, ES-2 of the Advanced Light Source. trm1, trm2: total reflection mirror (OptoSigma); wp: liquid crystal waveplate (Bolder Optik); pbc: polarizing beamsplitting cube (Newport); be: beam expander (Zeiss); irbs1: 50/50 infrared beamsplitter (OptoSigma), irm1, irm2: infrared mirror (Optosigma); l1: 80 mm focal length objective lens (OptoSigma); trm3: silver coated (Optics for Research) x-ray transparent amorphous carbon mirrors (Alfa Aesar); DAC: diamond anvil cell; trm4: total reflection mirror (Optosigma); spf: short pass filter (Edmund Optics); f: f-stop/iris aperture (Optosigma); l2: 1 meter focal length lens (Edmund Optics); trm5: total reflection mirror (Optosigma); bs1: 50/50 visible beamsplitter (Edmund Optics); trm6: total reflection mirror (Optosigma); Sp: spectrometer (Roper Scientific)

FIG. 3. Angle dispersive x-ray diffraction data collected during simultaneous *in-situ* laser heating and x-ray diffraction data from a sample containing Pt and Al_2O_3 at 9 GPa. X-ray diffraction patterns show the diminishing intensity at high temperature and rebounding intensity upon room temperature quench. Vertical bars show expected peak positions for Pt and Al_2O_3 at 9 GPa and 298 K. Time sequence is from bottom to top. Inset shows thermal radiation data and fit, normalized to system response and plotted according to the linearized Wien construct, for the highest temperature x-ray diffraction pattern (3250 K).

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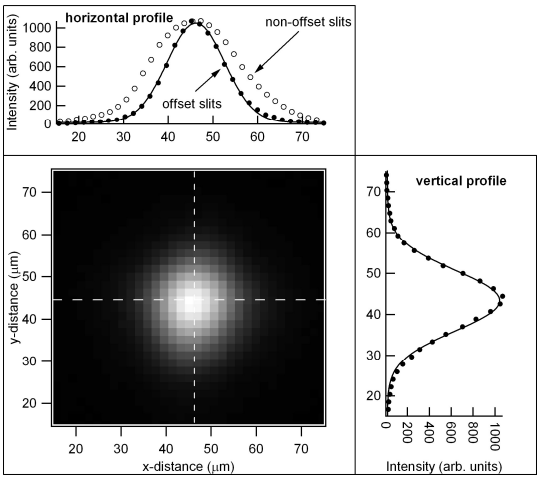
Fig. 1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non-offset slits
offset slits
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30
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50
60
70
y-distance (μm)
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vertical profile
Intensity (arb. units)

Double Sided Laser Heating System at Beamline 12.2.2, ES-2 at the Advanced Light Source

Fig. 2

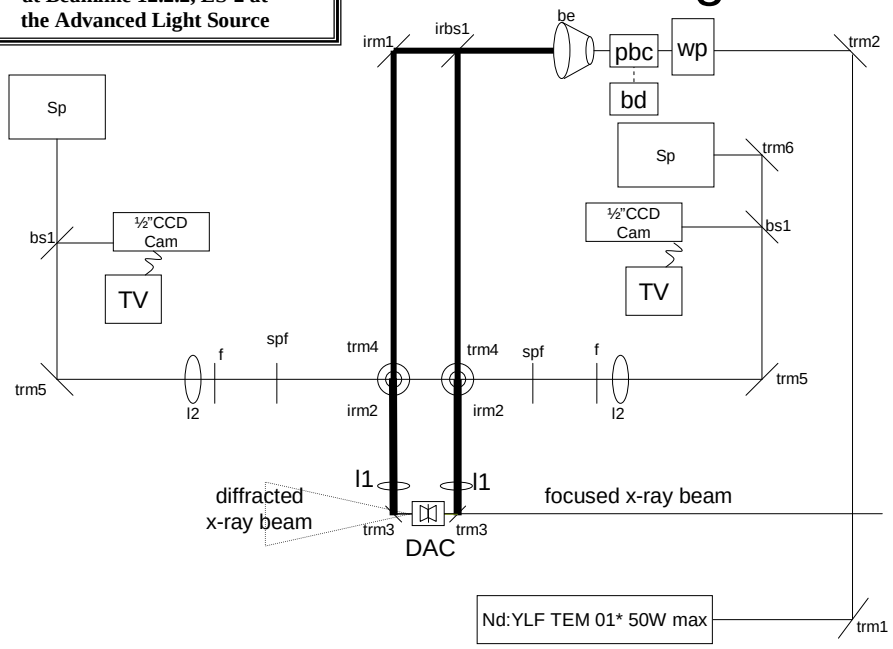
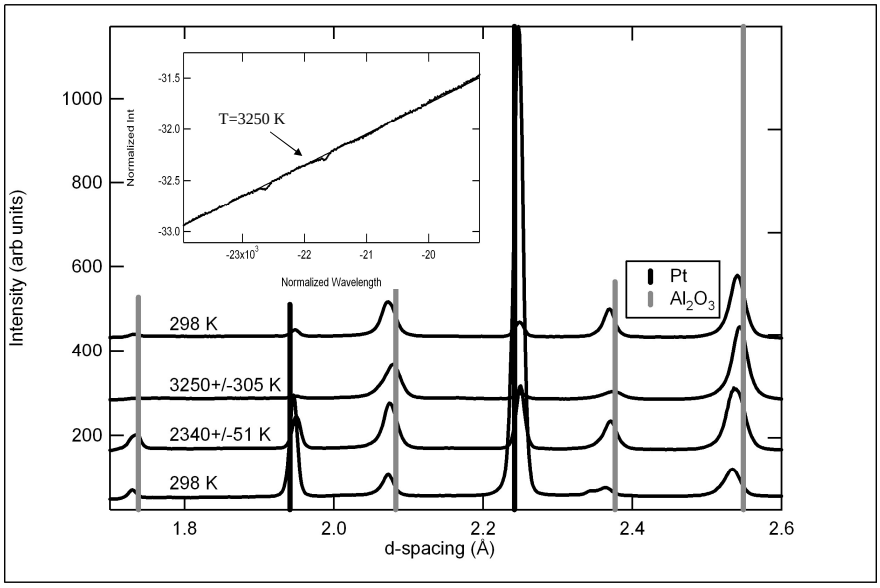


Fig. 3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Normalized Wavelength