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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 50 W Nd:YLF (cw) laser operated in TEM01* mode. Double-sided heating is achieved by splitting the laser beam into two 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 μm diameter (FWHM) in the sample region. An X-ray spot size of 10 μm 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.

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1. Introduction

The laser-heated diamond anvil cell (LHDAC) offers a powerful means of investigating the properties of materials at sustained high temperatures and pressures. Coupling this instrument with synchrotron-based X-ray diffraction enables the *in situ* characterization of crystal structures (crystal–crystal phase changes, solid–liquid phase change, lattice parameter determination, texture, etc.) not possible with a standard laboratory X-ray source.

Due to the high flux available from a synchrotron, it is possible to collect a suitable X-ray diffraction pattern in a few minutes; in comparison, samples can be held at temperatures of thousands of Kelvin and pressures of hundreds of thousands of atmospheres for minutes to hours. In addition, advances in focusing optics allow tailoring the X-ray spot size so that it fits neatly within the central region of relatively constant temperature inside the laser-heated hotspot. Installation of superconducting bending magnets at the Advanced Light Source in Berkeley has allowed generation of hard X-rays ($E > 2$ keV): crucial for scattering from the LHDAC sample, as it is sandwiched between much larger diamonds and these are highly absorptive to X-rays with energies below ~10 keV.

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The synchrotron-based LHDAC can thus give insight into the behavior of materials at extremely 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 into the structure of the Earth's liquid outer core [1], and revealed an important new phase in the Earth's lowermost mantle [2,3].

2. Experimental

2.1. X-ray generation, conditioning and detection

Endstation 2 (ES-2) of beamline 12.2.2 is situated approximately 1 m 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 $10 \times 10\,\mu m^2$ X-ray spot situated at the ES-2 sample position.

The primary focal spot of the X-rays at ES-1 (0.06 mm (v) $\times$ 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 $10 \times 10\,\mu m^2$ spot at the sample position. The K–B mirror pair is made of a silicon substrate coated with 4 nm Rh and 25 nm Pt.

A unique aperture configuration of offset slits defines the virtual X-ray source for the K–B mirrors, and succeeds in producing a more narrowly focused X-ray beam than can standard opposed slits (Fig. 1); however, the beam profile is best modeled as a Gaussian, rather than the flat-top profile that was the design goal. The Gaussian tails of the beam are attributed to scatter produced by surface irregularities in the mirror on the order of 1 mm–1 μm [5]. To prevent the beam tails from hitting the highly scattering gasket that surrounds the sample, a 100 μm Ta pinhole is placed approximately 10 cm upstream of the sample position. Traces of unfocused X-ray beams passing straight through the mirror pair (most noticeable at energies above 25 keV) are 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 within about 2 min, while a high-Z material can give a suitable pattern in 30 s. The image plate has a readout time of ~135 s.

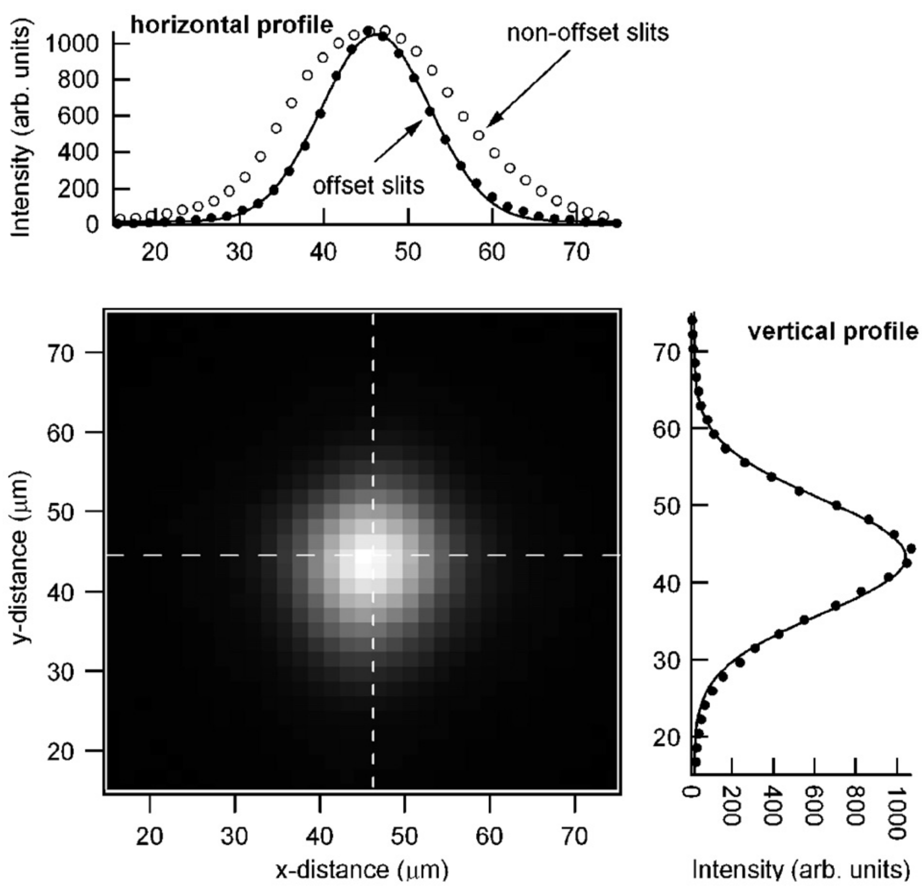

Fig. 1. Image of a micro-focused X-ray beam taken with a $CdWO_4$ scintillator, shown with vertical and horizontal cross-sections taken along the dashed white lines. A two-dimensional Gaussian fit to the image yields FWHM of 11.0 μm horizontally and 12.5 μm vertically. Profiles indicated with filled circles; Gaussian fits shown with solid lines; open circles in horizontal profile show beam size for non-offset slits.