A Laser-Heated Diamond Cell System for *In Situ* X-ray Measurements at High Pressure and High Temperature

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Introduction

As a unique method to reach ultrahigh static pressure-temperature conditions (P>100 GPa, T>1500K), the laser-heated diamond anvil cell (LHDAC) has been widely used for high P-T studies.[1-5] Major advances have been made on the LHDAC technique in the last two decades in heating capability, temperature measurement, temperature control, and the minimization of temperature gradients within sampling region. The latter is especially important for the application of this technique to x-ray measurements on a heated sample at high pressures. Recently the LHDAC has been combined with *in situ* x-ray diffraction at high P-T.[5,6] The high brilliance of synchrotron radiation allows control of the x-ray beam to a size smaller than of the laser heating spot but still with enough photon flux to accurately measure x-ray diffraction and to conduct a variety of x-ray spectroscopy measurements.

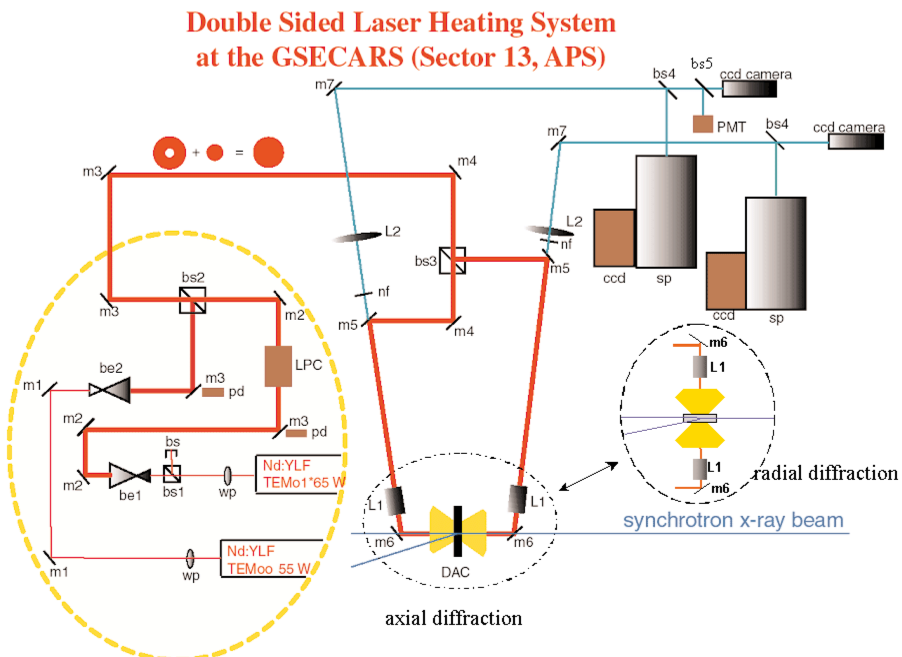

FIG. 1. Schematics of the double-sided laser heating system at the GeoSoilEnviro-CARS sector.

Techniques and Applications

There are several critical issues in LHDAC experiments with synchrotron radiation. (1) The heating spot must be much larger in size than the x-ray beam; (2) temperature gradients in both the radial and axial directions should be minimized to be less than other experimental uncertainties, e.g., diffraction accuracy; (3) steady temperature is required for the x-ray measurement; (4) the micro x-ray beam should be well collimated to avoid contamination from strong scattering by surrounding materials (e.g., gasket); and (5) the pressure and temperature of the sampling area should be well controlled and determined.

A double sided LHDAC system (Fig. 1) has been developed and is operational at the GeoSoilEnviroCARS sector. The laser heating system allows the generation of a large heating volume compared to the x-ray beam size, allowing for *in situ* x-ray measurements at simultaneously ultrahigh pressures (to > 160 GPa) and ultrahigh temperatures (to > 4000K). Typical heating areas of 10-25 µm in diameter from both sides result in minimum temperature gradients in the sampling volume both radially and axially in the diamond anvil cell and the minimum error arising from the chromatic aberration in temperature measurement. A feedback system permits steady heating of a diamond anvil cell (DAC) sample over minutes to hours. Temperatures can be controlled remotely by software. When combined with the x-ray microbeam (3-10 mm) technique, a temperature variation of less than 50K can be achieved within the x-ray sampled region over 10 min. The LHDAC is the coupling of two parts: the laser beam and the heating object in the DAC. Proper sample configuration design and preparation, including effective use of insulation layers, are crucial for a successful experiment.

This system has been widely used for simulating the pressure-temperature conditions of the deep Earth's interior. Crystal structures,[7] phase boundaries,[8] equations of state,[9] phase stability 10, and electronic structures 11 have been studied at high pressures and high temperatures for a variety of Earth's candidate materials.

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