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Double-sided laser heating system at HPCAT for *in situ* x-ray diffraction at high pressures and high temperatures

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Abstract

An overview of a YLF:Nd laser heating system at the undulator x-ray diffraction station (16ID-B) of the high-pressure collaborative access team (HPCAT) of the Advanced Photon Source is presented. Based on the double-sided laser heating technique, the system is designed with considerable effort on the mechanical and optical stabilities, features for user-friendly operation, and the capability of accommodating diamond anvil cells of various heights up to 68 mm. This system has been used for x-ray diffraction studies of a wide range of materials to over 150 GPa and above 3000 K. Applying the laser heating technique to radial x-ray diffraction studies at simultaneous high-pressure and high-temperature (PT) conditions requires heating to be conducted at variable angles relative to the x-ray direction. A rotation laser heating design is discussed.

1. Introduction

Invented more than 30 years ago [1], the laser-heated diamond anvil cell has been a unique static technique for reaching ultrahigh-PT conditions ($P > 100$ GPa, $T > 1500$ K) and has been used for research in physics, chemistry, materials science, geoscience and planetary science, leading to numerous important discoveries and novel phenomena of fundamental importance [2–12]. Over the past three decades, major technical progress has been made in the areas of heating capability and stability, temperature control, and temperature measurement [13–15]. The issue of an axial temperature gradient in the sample layer has been resolved by introducing the double-sided laser heating technique [15–17]. With the rapid development of synchrotron x-ray diffraction and scattering in the study of high-pressure problems, laser-heated diamond anvil cell has been becoming an increasingly powerful tool for high-pressure research.

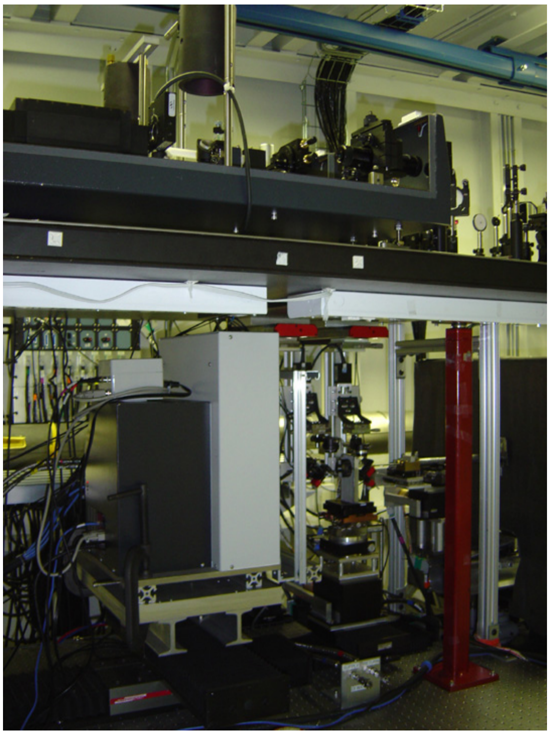

Figure 1. An integrated laser heating and micro-focused x-ray diffraction setup at the undulator beamline (16ID-B) of HPCAT.

Integrating a laser-heated diamond anvil cell with synchrotron radiation research represents far greater challenges than those applications in laboratories. Only in recent years, this application has been beginning to evolve into a routine method for *in situ* synchrotron x-ray studies at simultaneous high-PT conditions. Key challenges include system stability to less than 1 to 2 μm and accurate coupling between the x-ray beam and heating spot. Although there has been significant progress in recent years, further improvement is needed to achieve high accuracy in x-ray diffraction, pressure and temperature measurements at ultrahigh-PT conditions. Furthermore, current laser heating design limits its application to the conventional diffraction geometry; radial x-ray diffraction studies under simultaneously high-PT conditions are not feasible.

As a dedicated high-pressure synchrotron x-ray facility at the Advanced Photon Source, HPCAT has developed a double-sided YLF laser heating system coupled with its undulator x-ray diffraction beamline. In addition to developing state-of-the-art techniques, much of our effort has been on improving system stability and developing features for user-friendly operation. The purpose of this article is to present an overview of the system. A rotation laser heating design for extending the technique to radial x-ray diffraction studies at simultaneous high-PT conditions will be discussed.

2. System outline

Figure 1 shows the laser heating system as part of an integrated high-PT x-ray diffraction setup. The major portion of the heating optical components is located on the top experimental

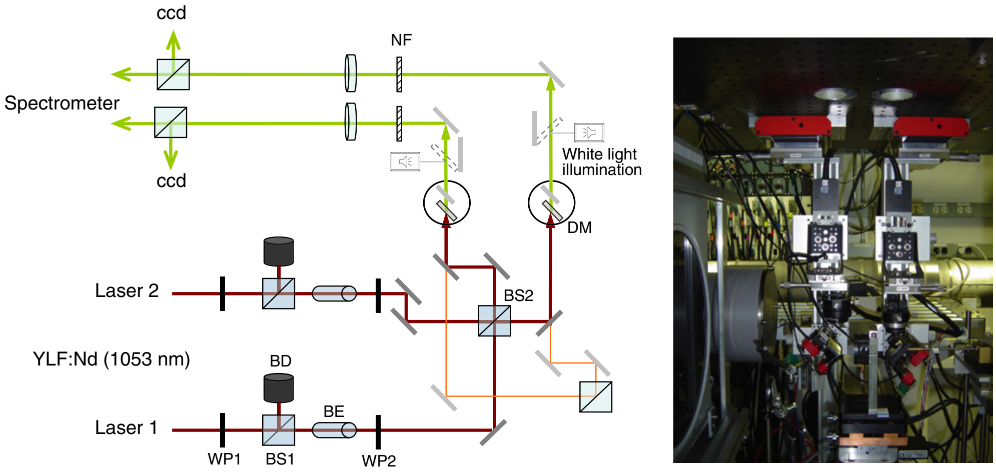

Figure 2. Left: laser heating optical components on the top experimental table, shown schematically. WP denotes wave plate; BS, beam splitter; BE, beam expander; BD, beam dump; DM, dichroic mirror; ccd, CCD camera. The red lines represent the YLF laser path, orange lines the He–Ne alignment laser, and green lines the thermal radiation from the heating spot. Right: heating optics below the top experimental table.

table, shown schematically in figure 2 (left). This double-sided laser heating system has two identical YLF:Nd lasers (Photonics GS40, wavelength = 1053 nm) operating in horizontally polarized donut mode (TEM_{01} mode) and providing a total maximum output of 170 W with a power stability >99%. The heating laser beam, which is a combination of the two YLF:Nd lasers, is split into two beams that pass through the opposing diamond anvils to heat the high-pressure sample simultaneously from both sides, as shown in figure 2 (right). Temperatures are measured separately from both sides with an imaging spectrograph and equalized by adjusting the power ratio of beam splitting. With centralized remote control, heating and x-ray diffraction measurement can be conducted simultaneously; furthermore, the heating spot on the sample, as well as the coupling between the heating spot and the temperature measurement, can be monitored from both sides using CCD cameras and adjusted as needed.

3. System optics and components

As shown in figure 2 (left), each laser beam passes through a power regulator (section 3.1) and a beam expander, which expands the beam to about 10 mm in diameter. The expanded beams are then combined and re-split into two for double-sided heating (section 3.2). The heating optics for each heating beam includes a dichroic mirror, which reflects infrared (IR) laser light but allows visible light to pass through, an apochromatic objective lens (with a 70 mm focal length for the up-stream side and 100 mm for the down-stream side) and a 4 mm-thick carbon mirror. The heating lasers are reflected by the dichroic mirrors vertically down through the openings in the top experimental table, focused with the apochromatic objective lenses, and re-directed by the 4 mm-thick carbon mirrors to pass through the opposing diamond anvils for sample heating. The visible portion of the black-body radiation from the heated spot is collected separately from both sides by the same carbon mirrors and apochromatic objective lenses used for heating, passes through the dichroic mirrors to the top experimental table, and is focused by the achromatic lenses (with a focal length of 1000 mm) to a spectrometer for temperature measurement (section 3.3) and to the CCD cameras for sample viewing.

3.1. Laser power control

The YLF:Nd lasers need to be operated at a working current defined by the laser manufacturer for optimal beam profile and stability. Therefore, for the purpose of heating control, a separate power regulator is needed for each laser. Utilizing the polarized nature of the laser beam, the power of each laser is controlled by a power regulator that consists of a quarter wave plate (WP1), a cube beam splitter (BS1) and a beam dump (BD). As the quarter wave plate rotates from 0° to 45°, it continuously changes the polarization of the laser beam from vertical to horizontal. Accordingly, the proportion of the laser beam passing through the beam splitter varies from 0% to 100%. The beam dump is orientated at 90° to the laser beam direction on the reflection side of the beam splitter to collect the unused laser power (vertically polarized beam). By using a Newport SR50PP rotation stage, the rotation of the wave plate can be made accurately at an angle of 0.004° per step, providing fine and stable control of the laser power.

3.2. Laser beam combination and re-distribution for double-sided heating

This step provides the useful purposes of combining two different laser modes [15], splitting one laser for double-sided heating, and varying the power ratio of the two heating laser beams. As shown in figure 2 (left), the components for this step include two quarter wave plates (WP2)—one for each laser beam to be combined—and a cube beam splitter (BS2). To combine the two laser beams and split the combined beam evenly, the polarization of both lasers should be set at an angle of 45° from the vertical direction using the quarter wave plates; with the horizontal component passing through the cube splitter and the vertical component reflected, each of the two output beams from the cube splitter contains 50% of the horizontally polarized beam from one laser and 50% of the vertically polarized beam from the other laser. The power ratio of the two split beams can be changed by simultaneously increasing the rotation angle of one wave plate and decreasing the other by the same amount with a pseudo motor control. The angle combination of the two wave plates (0°, 45°), (45°, 0°) and (22.5°, 22.5°) correspond to single up-stream side heating, single down-stream side heating and double-sided heating, respectively. The contribution of each laser to the combined beam is controlled by the individual power regulators.

3.3. Temperature measurement

Temperatures are determined by fitting the visible portion of the black-body radiation (600–800 nm) from the heating spot to the Planck radiation function:

$$I_\lambda = \frac{c_1 \varepsilon(\lambda)\lambda^{-5}}{\exp(c_2/\lambda T) - 1}, \tag{1}$$

where I_λ is the spectral intensity, ε is the emissivity, λ is the wavelength, T is the temperature, $c_1 = 2\pi hc = 3.7418 \times 10^{-16}$ W m^2, and $c_2 = hc/k = 0.014\,388$ mK. This spectral radiometry method has been widely used for temperature measurement in laser heating systems and has been discussed in detail elsewhere [3, 14, 17, 18]. In our system, we use an Inspectrum 300 imaging spectrograph equipped with a thermoelectric-cooled back-illuminated Hamamatsu CCD (1024 $\times$ 250 pixels). This imaging spectrograph covers a wavelength range of 260 nm and gives a wavelength resolution better than 1 nm with a 50 μm entrance slit. A two-leg fibre-optic bundle, coupled to the double-entrance slit of the spectrograph, allows us to collect the thermal radiation simultaneously from both sides of the sample. With a fibre size of 100 μm in diameter and a 50 μm entrance slit, thermal radiation from a 5 μm $\times$ 10 μm hot spot is collected for temperature measurement. The system response is calibrated by a tungsten ribbon lamp (OL550, Optronic Laboratories) with radiance calibrated according to National Institute for Standards and Technology (NIST) standards. The temperature measurement is verified by the known radiation point at 2000 K provided by the manufacturer of the standard lamp.

3.4. In situ high-PT x-ray diffraction with laser heating

The laser heating system at HPCAT is a part of an integrated high-PT x-ray diffraction setup. The heating optical components for up-stream and down-stream heating, shown in figure 2 (right), can be moved into and out of the x-ray path separately, allowing setup switching between the double-sided heating, one-side heating and regular diffraction modes. Typically, the heating area is about 30 μm in diameter compared with the x-ray beam size of 5–10 μm focused by a pair of 200 mm-long KB mirrors. A 40 μm clean-up pinhole is positioned behind the up-stream heating mirror; the diffracted x-rays are collected with a MAR imaging plate or a MAR CCD detector. For accurate diffraction measurements, an x-ray fluorescence spot from a sample material is used to locate the x-ray beam position for precise coupling between the x-ray beam and the heating spot. Common issues associated with any *in situ* x-ray diffraction with double-sided laser heating involve (1) a reduction in x-ray intensity through the absorption of x-rays by the two carbon mirrors and (2) the strong amorphous carbon diffraction pattern from the down-stream mirror overlapping with the diffraction pattern of the sample. The absorption of a 4 mm carbon mirror in our system introduces an intensity reduction of about 18% at 35 keV. The amorphous diffraction pattern can be completely blocked out by placing a piece of lead on the back of the down-stream carbon mirror.

4. Radial diffraction study with *in situ* laser heating

High-pressure radial x-ray diffraction has been used for rheology and elasticity studies [19–21]. It is also a useful method for studying samples that develop a strong preferred orientation or evolve into a single crystal at high pressure [22]. In radial diffraction experiments, the DAC needs to be rotated to allow x-ray diffraction measurements to be carried out at different angles relative to the DAC axis. Such a diffraction geometry is clearly in conflict with the conventional laser heating optics and with most resistive heating setups. As a consequence, high-pressure radial x-ray diffraction studies have been conducted at ambient temperature or on temperature-quenched samples.

In figure 3, we present a design of a rotation laser heating system for radial x-ray diffraction measurements at simultaneous high-PT conditions. In this design, the heating unit—consisting of a cube beam splitter to split a laser beam for double-sided heating, a wave plate for balancing the power distribution between two heating beams, dichroic mirrors, heating laser focusing lenses, and reflecting carbon mirrors—is attached to a rotation stage mounted to the bottom of the top experimental table. The heating direction relative to the x-ray beam can be changed by rotating the whole heating unit. To maintain the same heating spot at different angles, the rotation axis of the heating unit, the vertical laser beam from the top experimental table, and the rotation axis of the DAC need to be co-axial, and the rotations of the DAC and the heating unit synchronized. One advantage of this setup, in comparison with the conventional laser heating arrangement, is that the x-ray diffraction path is separated from the heating path and, therefore, it eliminates the issues introduced by heating delivering mirrors. The radial temperature gradient can be an issue when x-ray diffraction is measured at large angles near 90° from the heating direction. Increasing the heating laser beam size and reducing the sample size (a ratio of $\sim$3:1) should minimize or completely eliminate the effect of a radial temperature gradient.

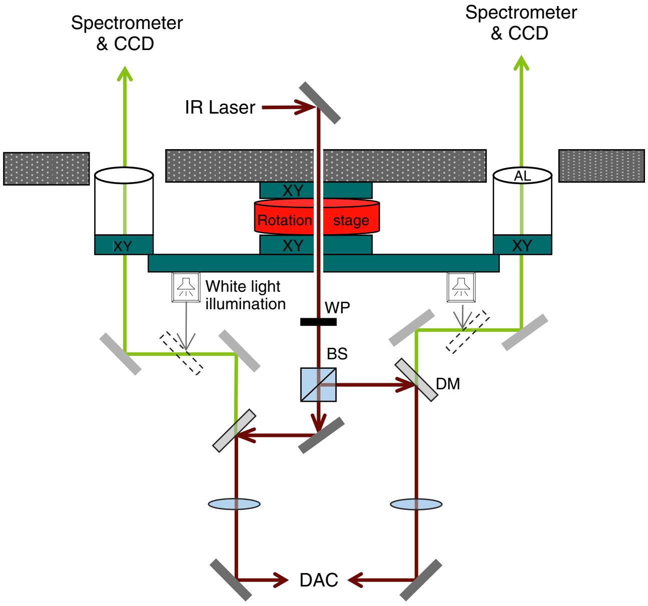

Figure 3. A rotation laser heating design for radial x-ray diffraction studies at simultaneous high-pressure and high-temperature conditions. WP denotes wave plate; BS, beam splitter; DM, dichroic mirror; xy, linear xy stage; and AL, achromatic lens. The red lines represent the YLF laser path and green lines the thermal radiation from the heating spot.

5. Future outlook

The scientific goal of HPCAT is to study the properties and novel behavior of materials subjected to high pressures and high temperatures from cryogenic states to thousands of degrees. Considering the laser heating technique, achieving this goal involves pushing the limit of the technique to reach new record high temperatures at high pressure, as well as continuously improving the accuracy of x-ray diffraction measurement and PT calibration. In addition to further enhancing the capability of the current system, we look into the prospect of extending the laser heating application to *in situ* radial x-ray diffraction studies, adding the CO_2 laser heating capability, and exploring the pulse laser heating technique. High-pressure sample loading for laser heating is a critical but overlooked area in practice. Implementing existing micro-positioning techniques for precise sample loading, developing new gaskets, and exploring new medium materials for better thermal insulation and x-ray beam positioning are important aspects for achieving high-quality measurements.

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