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A portable laser heating microscope for high pressure research

A portable laser heating microscope for high pressure research

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Abstract. We report the progress of the construction of a portable laser heating microscope for a broad range of materials studies at extreme pressure-temperature conditions. The essential features are portability, a broad temperature range, and a modular design making it flexible for a variety of applications in high pressure research for different environments, including in synchrotron and neutron applications and optical spectroscopies. The integrated instrument functions like a microscope, containing an infinity-corrected microscope head, fiber lasers, IR and UV spectrograph modules, IR and visible CCD cameras, and a control system. It provides stable laser heating on an area greater than 30 μm in diameter. Temperature can be controlled and reliably measured down to 500 K. Extending temperature measurement over 10,000 K with the short wavelength optics is discussed.

1. Introduction

In the laser heated diamond anvil cell (DAC) technique, first introduced by Ming and Bassett[1], a laser beam passes through the transparent diamond anvil and only locally heats the laser-absorbing samples without heating the gasket and other DAC components, thus avoiding interference with the DAC operation. The laser heating (LH) technique has now been widely used in heating DAC samples to thousands of degrees. Temperatures of a heated spot are usually measured by monitoring thermal radiation through the so-called radiospectrometry technique[2, 3]. In order to reach a uniform volume temperature in three dimensions, the double sided laser heating technique was developed by shaping laser beam profile, optimizing sample configuration, and refining optical arrangement [4, 5]. The LH technique has been successfully integrated with a variety of micro-probing techniques, including synchrotron, neutron, and optical lasers (e.g., Raman, Brillouin).

Conventional LH systems with their typical meter-length lasers, >100 kg power supplies, cooling systems, rigid optical trains and optical table, are difficult to move and time-consuming to align, and must be regarded as fixed instrument at individual facilities. High-pressure research programs are

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greatly enhanced at a handful of facilities where dedicated on-line LH systems are installed (e.g.,Refs. [6-10]). Numerous facilities with powerful analytical techniques, however, have not yet been able to install LH system due to technical and budget constraints. A key issue is the portability. The high-pressure DAC itself is a portable and flexible component. Making LH systems portable and available to all these analytical facilities will open new opportunities in high pressure-temperature science and technology.

With the advancement in the laser technology, the available fiber laser now has the size of a small flashlight yet available with hundreds of Watts power and better collimation than the large fixed YAG or YLF laser. It has been successfully used in the LH DAC [11]. Using fiber laser solves the major obstacle in designing a portable LH system. Recently, a compact laser heating system based on a fiber laser has been constructed for synchrotron applications [12]. The reported bench top system can be pre-aligned and minimizes the setup time for specific synchrotron applications. However, the system is still based on a fixed DAC position located almost in the middle of the bench, limiting the flexibility of integrating with various probing instruments. Here, we report a system that is as portable and flexible as a microscope with the following features:

Portability – The heating microscope can be installed and integrated at a facility and optimized for high pressure-temperature DAC experiments within a short time period (1-2 hours). For double sided heating, two systems can be employed.

Co-axial alignment: The heating laser and the imaging path are co-axially aligned within the microscope. Thus, the visual object, the heating target and temperature monitoring point are engineered to be at the same location, without any additional alignment.

Modular design – The system is based on an infinity corrected microscope, making it possible for different geometries for objective lenses to maximize the flexibility. The IR and UV-VIS spectrographs are independent, pre-aligned modules that can be put together with minimal adjustment or can be added to other existing fixed LH systems.

Extended medium temperature range – The system covers the important temperature range of 500 – 1200 K with an IR detector and optics.

Extended maximum temperature range – With ampleness of fiber laser power (2 x 100 W) and UV detector and optics, the system can extend temperature measurements above 5000 K.

2. Optics layout

A typical optical layout for a system is shown in figure 1. The infinity corrected optics allows the insertion of auxiliary devices, such as beam splitters and intermediate tubes, into the optical pathway between the objective and the zoom body without introducing spherical aberration, requiring focus corrections, or creating other image problems. The heating laser delivers into the microscope pathway by a polarizing cube beam splitter (*bs2*). The image signal is divided by a 50/50 beam splitter (*bs3*), with one branch feeding into an optical fiber for temperature measurement and the other to a camera (InGaAs or CCD) for visual observations. Depending on applications, an objective with a suitable working distance may be selected. The supporting tubes for an objective can be extended or shortened as needed. Together with additional mirrors, the system can be used for various types of DACs in different geometries. All the optics are mounted on a rigid breadboard for stability (figure 2).

2.1. Heating laser optics

The fiber laser (*IPG Inc.*) delivers polarized, well-collimated light at a wavelength of 1.064 μm up to 100W in continuous mode. Typically, we set a fixed output power of the laser and change the power by a power regulator consisting of a wave-plate (*wp1*), a polarizing cube (*bs1*) and a beam stop (*bs*). By rotating the wave-plate driven by a piezo-stage, the laser power can be regulated remotely from 0 to the maximum of 100W. The laser beam is about 7 mm in diameter with a gaussian beam profile. In order to have a uniform heating spot, a laser beam shaper (Mol Tech) is used, which effectively changes the beam profile from a gaussian shape to a flat-top like profile[13]. A reversed beam expander (*be*) maybe used when we need to enlarge the heating spot to its maximum. Typical focal spot size is 30-50 μm in diameter, depending on the choice of an objective. With the reverse beam expander controlled by a pneumatic device, the focal size can be further expanded by a factor of 1-2. Finally, through a polarizing beam splitter (*bs2*), the laser beam enters the microscope path in a co-axial manner. The co-axial arrangement ensures the alignment of the laser with the visual imaging path even when a mirror is added or the tube extended/shortened.

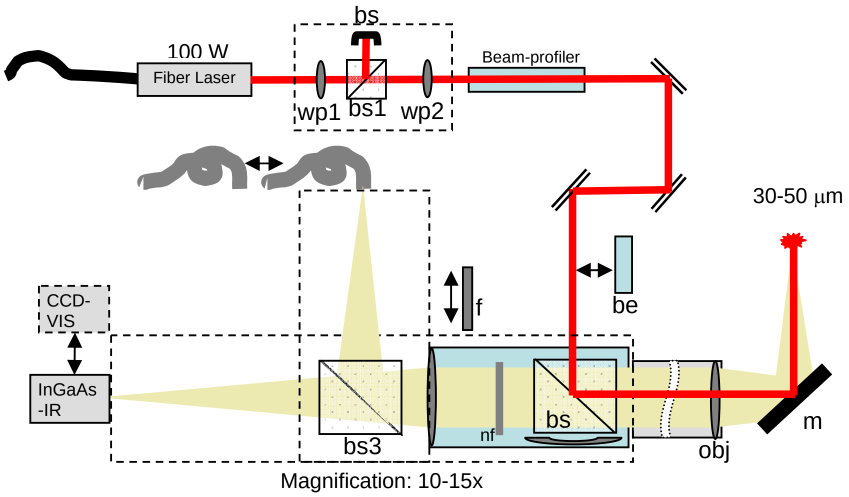


Figure 1. Optical layout of the laser heating system. *Fiber laser*: 100W polarized laser from IPG Inc; *wp1*, *wp2*: wave plates; *bs1*: polarizing beam cube; *bs*: beam stop; *beam-profiler*: pi-shaper from Mol Tech; *be*: beam expander; *bs2*: polarizing beam cube; *f*: glass filters; *nf*: notch filter; *bs3*: 50/50 beam splitter; *obj*: apochromat from US Laser; *m*: carbon mirror coated with internally protected silver. The curly objects represent optical fibers for delivering thermal signals to a spectrograph for temperature measurement. The CCD-VIS and the InGaAs-IR denote a CCD camera and an InGaAs camera for visual observation, respectively.

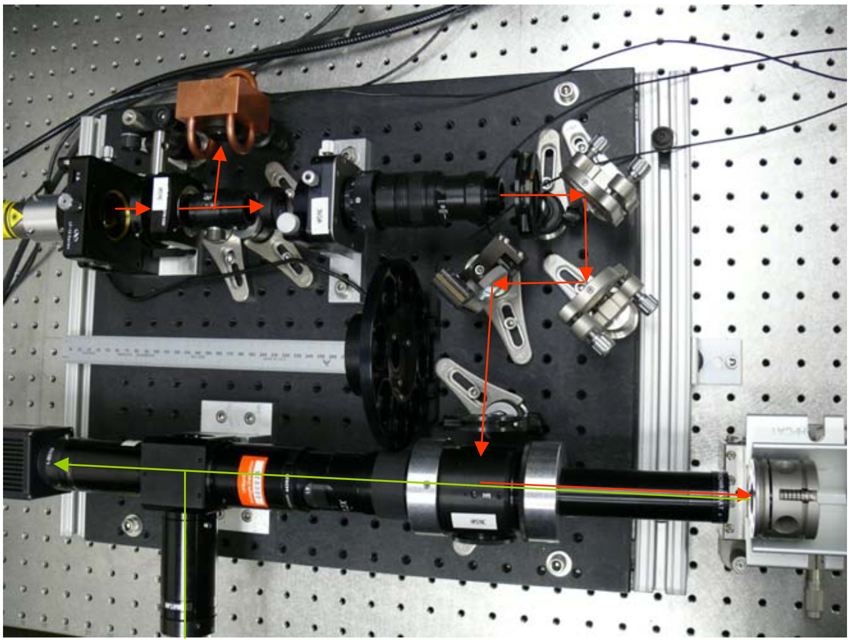
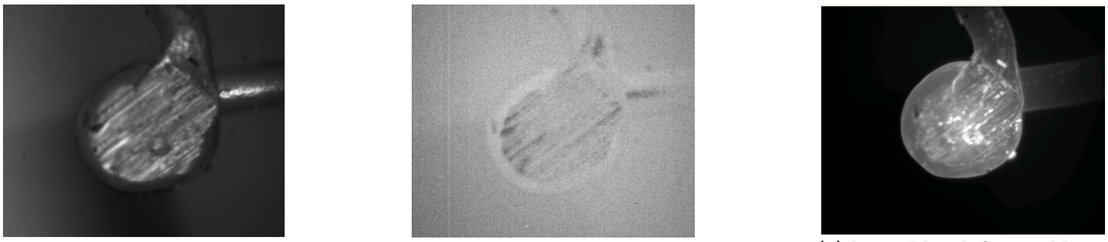


Figure 2. A picture of the optical layout with the cover open. A symmetric DAC is at the lower-right corner.

2.2. Infinity corrected microscope

The Navitar 12x zoom body is the main frame of the microscope. Between the zoom body and the objective, the beam path is parallel, allowing the insertion of optics (beam splitters, filters, tube extensions). Two sets of objectives with focal length of 37 mm and 77 mm are currently in use, which gives an overall magnification of 10x and 15x, respectively. A 50/50 beam splitter (*sp3*) is used to divide the image path into two branches: one for visual observation by a CCD or an InGaAs camera, the other for temperature measurement through an optical fiber. The optical fiber has a core size of 80 μm in diameter, thus monitoring an area of about 5-8 μm in diameter at the sample position.



(a) illuminated by light (b) thermal image at T=473 K (c) heated by defocused laser

Figure 3. Thermocouple images recorded by an InGaAs camera (Goodrich). The thermocouple, with the bead size of ~300 μm, was mounted in a small furnace. Images are compared at different conditions.

One feature of the system is that all optics are selected to be usable for short wavelength IR, i.e., for the wavelength range up to 1800 nm. The InGaAs camera (Goodrich) is sensitive to light radiations at 900-1800 nm, matching the spectrograph wavelength used for temperature measurements in the medium temperature range (500-2000K). The use of the short IR camera avoids any focus corrections arising from wavelength mismatch, and also allows for visualizing thermal objects as low as 450 K (figure 3b).

For applications at higher temperatures (>2000 K), shorter wavelength ranges (200-900 nm) are used for temperature measurements. A CCD camera is thus used for visual observation to match the covered wavelength range. All these changes are based on modular designs and can be made in a "plug-in" manner.

Figure 4a shows a picture of the laser heating microscope with all shielding panels covered. The infinity corrected optics allows for different configurations for mounting the objective lenses (figure 4b). These configurations make the system flexible for a variety of applications in high pressure research for different environments, including axial and radial DAC geometries (see Section 4 below).

(a) (b)

Figure 4. (a) A picture of the constructed laser heating microscope. It weighs ~15 kg, with dimensions of 54x38x20 cm^3. (b) Various configurations for mounting an objective to maximize the flexibility in integrating other analytical techniques.

3. Temperature measurement

Temperatures are measured by spectroradiometry [2, 3], i.e., by fitting thermal radiation signals in a given wavelength range to the Planck radiation function. Thermal radiations are delivered by an optical fiber and dispersed by an Acton spectrograph. For a temperature range of 500 – 2000 K, an InGaAs OMA detector (Princeton Instruments) is used for collecting thermal radiation at wavelength of 1300-1600 nm. For higher temperatures, we collect thermal radiations in a wavelength section, spanning 200-300 nm, from a total range of 200-900 nm by using a CCD detector (Princeton Instruments), with shorter wavelength section used for higher temperatures.

3.1. Selection of wavelength range

In spectroradiometry both temperature and emissivity values are derived by fitting to the Planck radiation function:

$$I_\lambda = c_1\varepsilon_\lambda\lambda^{-5}/[\exp(c_2/\lambda T)-1], \qquad (1)$$

where I_λ is observed spectral intensity, λ is wavelength, c_1=3.7418x10^{-16} Wm^2, c_2=1.4388x10^{-2} mK, T temperature, and ε_λ emissivity. In literatures, the most reported temperatures with the LH DAC have been obtained with the grey body assumption, i.e., with wavelength independent emissivity. From the limited available data, however, emissivity is a function of wavelength. To the first order approximation, ε_λ may be expressed as a linear function of wavelength:

$$\varepsilon_\lambda = \varepsilon_0[1+\varepsilon_1(\lambda-\lambda_0)], \qquad (2)$$

where λ_0 could be a central wavelength of a selected range. Under the grey body approximation, ε_1=0. Therefore, the emissivity ε_0 in Eq. (1) can be treated as a normalization factor in intensity. In this case, both temperature and emissivity ε_0 values can be obtained from thermal radiation signals fitting to the Planck radiation function (1).

The challenge arises when we consider the wavelength dependence emissivity, even if we only consider the linear dependence (Eq. 2). It requires the radiation data in the entire wavelength range to reliably obtain temperature, emissivity and its wavelength dependence. In all the laser heating systems, however, only a limited wavelength range is used, leading to that the two parameters (T, ε_1) are correlated with each other and cannot be uniquely derived by the curve fitting. Often times, we need to fix a wavelength dependence value ε_1 to constrain temperatures, with ε_1 either assumed to be zero (the grey body assumption) or a value from the data at ambient pressure. Because of the unavailability of the emissivity data at high pressures and high temperatures, the assumed ε_1 values inevitably involve errors. Therefore, for a reasonable temperature determination, the fitted temperature values should not be too sensitive to the choice of ε_1 values.

To check the sensitivity at different wavelength range, we used the radiation data from a standard lamp, which covers a large wavelength range of 300–2500 nm. To have a good fit in the entire wavelength range, it is necessary to introduce wavelength dependence of emissivity (ε_1). We find that all three parameters (T, ε_0, ε_1) can be reliably obtained if we use radiation data in a wavelength range covering both sides of the main peak. If a wavelength range of only one side is used, the fitted temperature values are tied to the choice of ε_1 values. When the shorter wavelength side (500-800 nm) is used, the resultant temperatures show weakly dependence to the choice of ε_1, within 60 degrees for different ε_1 values from zero to the maximum in literature[14]. If we use the longer wavelength side (1200 – 1600 nm), the fitted temperatures are strongly sensitive to the choice of ε_1 values, with differences in temperature more than 500 degrees for only small changes in ε_1 values. The exercise above illustrates that a proper selection of wavelength range is critical for minimizing the effect of ε_1 in temperature determinations. Because of the limited wavelength range in the current spectrometer systems, we should keep a wavelength range at the shorter side of the main radiation peak.

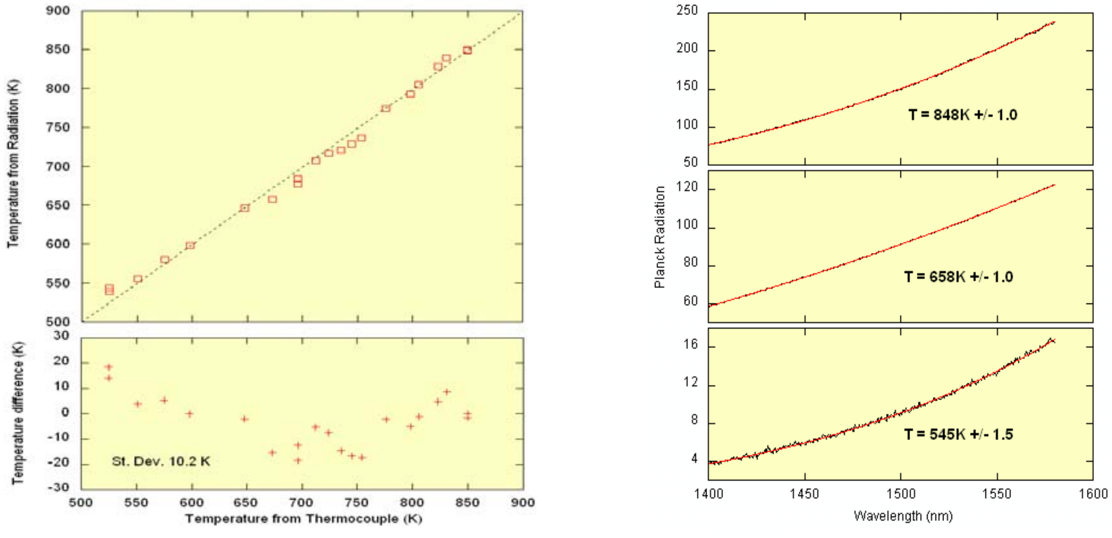
In our portable laser heating system, the thermal radiation is delivered by an optical fiber to the entrance slit of an Acton spectrograph which holds two detectors, an InGaAs OMA detector and a CCD detector, covering wavelength ranges of 1200–1800 nm and 200-900 nm, respectively. This broad wavelength coverage allows us for selecting optimal wavelength ranges for different temperature ranges (Table 1).

Table 1. The wavelength selections for different temperature range

Temperature range (K)	Wavelength range (nm)	Detector type
500 – 1700	1300 – 1600	InGaAs
1200 – 4000	500 – 800	CCD
3000 – 5000	300 – 600	CCD
> 5000	200 – 500	CCD

3.2. Temperature measurement down to 500 K

The system can measure temperatures down to 500 K by adopting short wavelength IR optics. To cross-check the measurement accuracy, we have tested the system with a thermocouple (R type) mounted in a furnace. The thermal radiations directly from the thermocouple bead were recorded at different temperatures controlled by the furnace. Figure 5a compares the temperatures from thermal signals with those from thermocouple readings. It can be seen that these two measurements are in reasonable agreement with a standard deviation of about 10 degrees in the covered temperature range. We find that the fitting quality to the Planck radiation is very good, with small statistics errors (<2 degrees) (figure 5b) in the wavelength range of 1400 – 1600 nm. At a given temperature, the temperature measurements by thermal signals are highly reproducible, within 2-3 degrees over multiple measurements. Below 500 K, the thermal signal in the measured wavelength range becomes too weak.



(a) (b)

Figure 5. (a) Temperatures measured by thermal signals directly from a thermocouple bead are compared to thermocouple readings (upper), with a standard deviation of 10.2 degrees (lower). Errors are within the symbol size. (b) Examples of the thermal radiation fittings to the Planck radiation function.

3.3. Extending maximum temperatures

The 500-900 nm is the most commonly used range in LH systems. This range is suitable for temperature determination between 1200 K and 4000 K. To extend maximum temperature range, shorter wavelength range should be used (Table 1). To avoid detector saturation, a set of glass filters mounted on a motorized wheel are often used for signal attenuation. In the temperature range of 10,000 K, the radiation maximum peaks at around 250-300 nm and becomes narrower. A range of 200-500 nm can in fact cover signals from both sides of the main radiation peak, measuring the shape of the radiation function. It is then possible to obtain both temperature and wavelength dependence of emissivity information simultaneously. We are still in the process of establishing a reliable reference source of >7000 K for cross-checking the temperature determination. This is a subject of research and the result will be reported elsewhere.

4. Applications

The flexible laser heating microscope is highly portable, with a weight of ~15 kg and dimensions of 54x38x20 cm^3, within the airline carry-on luggage allowance. It has wide applications for high pressure researches in different environments. Here, we present examples of mostly used geometries in synchrotron high pressure experiments. The heating spot size of 30-50 μm in diameter matches well the probing x-ray beam size, which is typically less than 5 μm in many beamlines at the third generation synchrotron sources. By combining two heating microscopes, it is easy to realize double sided heating.

4.1. Axial geometry

In on-axis geometry, incident x-ray passes through diamond anvils (figure 6). By using x-ray transparent mirrors (e.g., amorphous carbon substrate), x-ray measurements in the forward direction can be performed *in situ* at high pressures and high temperatures. Such measurements include angle dispersive x-ray diffraction, nuclear forward scattering, inelastic x-ray scattering among others. Using x-ray transparent gasket, scattering signals through the gasket can be measured, such as in nuclear resonant inelastic x-ray scattering and x-ray emission spectroscopy.

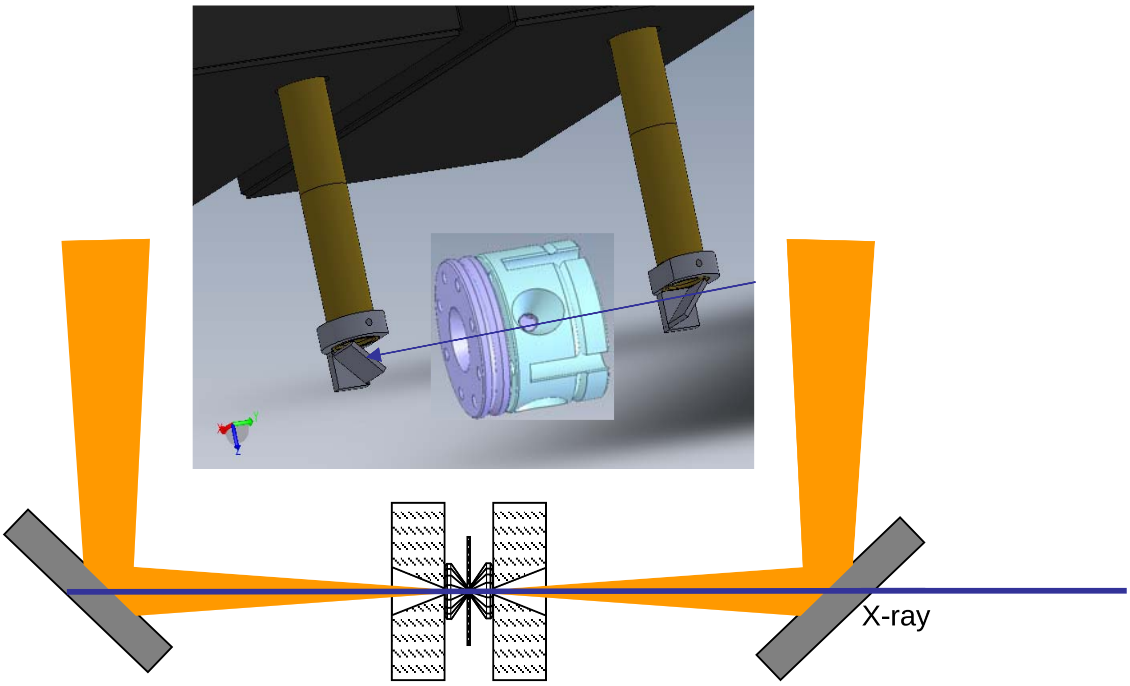
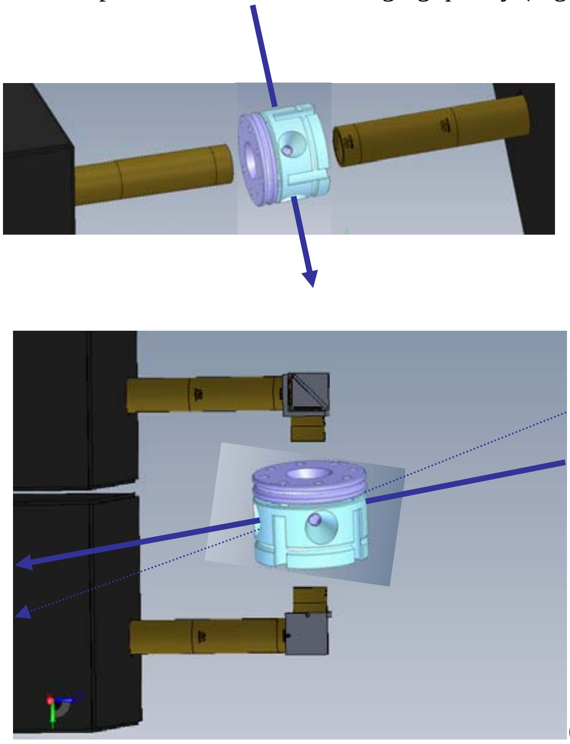


Figure 6. Schematics of an axial geometry. The insert is a configuration for the double sided heating in the axial geometry using two heating microscopes.

4.2. Radial Geometry

In radial geometry, the loading axis of a DAC is perpendicular to the x-ray beam. This geometry is now more and more widely used in synchrotron high pressure experiments. Applications include radial x-ray diffraction for rheology and elasticity, and various x-ray scattering and spectroscopy techniques which often use beryllium gasket for relatively low energy x-rays to pass through. In this geometry, laser beam can be applied from the side. Depending on whether the geometry is horizontally (Fig. 7a) or vertically (Fig. 7b) perpendicular to the x-ray beam, additional mirrors may or may not be necessary. If no mirror is needed, then objectives of short working distance can be used for a larger numerical aperture and for better imaging quality (Fig. 7a).



(a)

(b)

Figure 7. Radial geometry with the heating laser horizontally (a) and vertically (b) perpendicular to the x-ray beam shown in solid lines. The dashed line in (b) shows that the loading axis may be tilted to the x-ray beam.

4.3. Other applications

The above geometries are just a few typical examples. There are other geometries for specific x-ray techniques and measurements. For example, in radial geometry, the loading axis may be tilted to the x-ray beam (figure 7b). With the heating microscope, it is flexible enough to follow the tilting and realize x-ray measurements at *in situ* high pressures and high temperatures.

The heating microscope can also be used as a thermometer for temperature determinations in both externally and internally resistively heated DAC experiments, covering temperatures above 500 K. The region between 300 and 500 K can be easily reached by heating tapes, hot plates and many other simple methods, and can be considered as small perturbation of ambient temperature. The portable system should be applicable to neutron DAC experiments, and various optical spectroscopy techniques, such as Raman and IR spectroscopy and Brillouin scattering.

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