

A laser heating diamond anvil cell system for high-pressure nuclear resonant scattering at 3-ID, APS

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ABSTRACT

Nuclear resonant scattering (NRS) provides the basis for powerful synchrotron-based techniques, including synchrotron Mössbauer spectroscopy (SMS) and nuclear resonant inelastic x-ray scattering (NRIXS), which probe the elastic, magnetic, thermodynamic, and vibrational properties of iron-bearing materials and are, therefore, well suited for studying the structure and composition of Earth's and planetary interiors. Here, we report the development and commissioning of a double-sided laser heating system for diamond anvil cell experiments at the 3-ID beamline of the Advanced Photon Source (APS). A key feature of the system is a 7° offset between the x-ray and laser-delivery paths, which positions the near-sample mirrors outside the x-ray path. This design minimizes x-ray absorption, enhances the NRS signal rate during laser heating, improves imaging quality, and significantly reduces thermal drift during extended data acquisition periods. The setup is optimized to match the reduced x-ray beam size available after the APS upgrade, making NRS measurements under extreme pressure-temperature conditions more practical. In addition, the system supports complementary online ruby fluorescence and x-ray diffraction measurements for characterizing samples before and after laser heating. We demonstrate the performance of the system through representative SMS and NRIXS measurements conducted under simultaneous high-pressure and high-temperature conditions.

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I. INTRODUCTION

Understanding the behavior of materials under extreme pressure (P) and temperature (T) conditions is essential for advancing knowledge across a wide range of scientific fields. In geosciences, experimental access to conditions of Earth's deep interior with synchrotron-based probes provides critical insights into the structure, composition, and evolution of planets.^{1,2} Among these techniques, nuclear resonant scattering (NRS) offers a unique view into the elastic, magnetic, thermodynamic, and vibrational properties of iron-bearing materials, which are key components of Earth's and planetary interiors.

Over the past decades, NRS experiments using diamond anvil cells (DACs) conducted at 3-ID of the Advanced Photon Source

(APS) have produced many scientific breakthroughs that have advanced our understanding of iron-bearing materials at high pressures. These experiments include both nuclear resonant inelastic x-ray scattering (NRIXS), which provides a direct probe of the phonon density of states (PDOS) of the resonant ^{57}Fe isotope, and synchrotron Mössbauer spectroscopy (SMS), which reveals hyperfine interactions and probes electronic and magnetic properties of ^{57}Fe -bearing samples.^{3–5} High-pressure ^{57}Fe NRS measurements at 300 K have provided important constraints on the electronic, magnetic, elastic, and vibrational properties of Earth- and planetary-relevant materials, including Fe-bearing silicates, oxides, hydrous phases, Fe, and Fe alloys.^{6–9} Furthermore, the development of a dedicated laser heating system at beamline 3-ID-B¹⁰ with laser-heated diamond anvil cells (LHDACs) has enabled high P - T NRIXS

measurement of Fe up to 73 GPa and 1700 K¹¹ and of Fe₃C up to 47 GPa and 1450 K¹² to constrain sound velocities and vibrational properties at extreme conditions, as well as using SMS to constrain the high-*P* melting of Fe¹³ and Fe alloys,^{14,15} and FeO.¹⁶

Despite these significant successes, high *P*-*T* NRS studies remain limited compared with high-*P* experiments at 300 K. The scarcity of high *P*-*T* NRS data, particularly for NRIXS, stems mainly from the fundamental conflict between the technique's requirements and the limitations of traditional laser heating systems. NRS is inherently a photon-hungry technique because it probes nuclear resonances with natural linewidths on the neV scale, requiring the incident x-ray beam to be monochromatized to a meV-scale bandwidth near the resonance energy, which severely limits usable photon flux. Depending on the ⁵⁷Fe concentration in the sample, for instance, collecting a high-quality SMS time spectrum usually takes minutes to hours, while obtaining a high-quality NRIXS energy spectrum often requires hours to days of continuous data acquisition for a single *P*-*T* point. Most existing laser heating systems were designed for x-ray diffraction, where exposures are measured in seconds or even shorter at many advanced synchrotron light sources.^{17–20} Maintaining a stable alignment between the x-ray beam, the laser spots, and a potentially drifting sample for the long duration of an NRS measurement is, therefore, an immense challenge. In addition, radial temperature gradients can complicate both NRIXS and SMS measurements: they broaden the temperature distribution sampled by the x-ray beam and, particularly for SMS, can affect the time-domain response through temperature-dependent changes in the Lamb-Mössbauer factor. Furthermore, mechanical instabilities and thermal drift in the optical path often introduce significant temperature uncertainties, posing a major challenge for long-duration NRIXS and SMS experiments under extreme conditions.

The APS recently completed a major upgrade,²¹ which provides a substantial reduction in source size, facilitating experiments at scales previously inaccessible. Within this framework, the ⁵⁷Fe NRS program has transitioned from the 3-ID-B station to the newly commissioned 3-ID-C station. A central upgrade of this transition is the integration of high-performance Kirkpatrick-Baez (KB) focusing mirrors, which reduce the focused monochromatic beam to the micrometer scale and, making high-pressure NRS studies at multi-megabar pressures more practical. Although the transition to the APS-U 48-bunch timing mode (from the previous 24-bunch mode) leads to a shorter timing window between x-ray pulses, the enhanced bunch purity provides an accessible time window of ~12 to 65 ns, and both SMS and NRIXS remain operational. For high *P*-*T* NRS experiments using laser heating, however, maintaining a micrometer-level alignment between the laser-heated region and the x-ray focus throughout extended data acquisition places unprecedented demands on the mechanical and thermal stability that the legacy laser heating system at 3-ID¹⁰ could not provide. To fully exploit these advances for research at extreme *P*-*T* conditions, a new generation of laser heating systems is required that can maintain μ m-level stability over the extended timescales required for NRS data collection.

In this study, we present the development of a new integrated double-sided laser heating system for DAC experiments as part of the high-pressure platform dedicated to the ⁵⁷Fe NRS program at the 3-ID-C station. Compared with the previous 3-ID-B laser-heating setup,¹⁰ the present system is designed to enhance thermal and

mechanical stability, reduce x-ray absorption by near-sample optical components, improve sample imaging and spectroradiometric temperature measurement, and maintain reliable overlap among the x-ray focus, laser-heated region, and temperature-measurement area during long data-acquisition periods. The system is optimized to match the reduced x-ray beam size available after the APS upgrade and accommodates complementary diagnostics, including online ruby fluorescence pressure measurements and x-ray diffraction (XRD) using an EIGER2 X 500K detector (Dectris). We demonstrate its application through representative SMS and NRIXS experiments at beamline 3-ID-C of the APS, Argonne National Laboratory.

II. LASER HEATING SETUP AT 3-ID-C, APS

Double-sided laser heating systems are widely used in university laboratories and synchrotron facilities for high *P*-*T* studies of materials, with common design requirements, including stable laser delivery, sample imaging, temperature measurement, and coupling to *in situ* probes, such as x-ray diffraction^{17–20} and x-ray absorption spectroscopy.²² In addition, a portable double-sided pulsed laser-heating system has been developed for time-resolved experiments.^{23,24} The present system differs from these more general or portable implementations in that it is a dedicated double-sided laser-heating platform designed specifically for ⁵⁷Fe NRS experiments at 3-ID-C, where the limited usable photon flux, long SMS and NRIXS acquisition times, high-resolution monochromator thermal-stability requirements, and need for avalanche photodiodes (APDs) access impose additional constraints on the optical layout.

The principal design of a laser heating diamond anvil cell system is defined by three main components: (1) laser beam delivery and focusing, (2) sample imaging and temperature measurement, and (3) coupling of the setup with the desired experimental technique.²⁵ The latter often places specific constraints on the components that can be used and on the space available to build the system. In addition to these constraints, the overall optical height plays a critical role in mechanical stability. At beamline 3-ID-C, high-pressure NRS experiments are carried out on a dedicated (8 ft × 4 ft) optical table, on which the laser heating system is permanently installed together with other NRS-related components. The focused x-ray beam is positioned 30 cm above this tabletop. In our design, the major portions of the laser heating system optical paths were kept below this height to enhance stability and then steered upward to intersect the sample precisely at the focused x-ray position with a 7° angular offset on each side. To further improve mechanical and thermal stability, the optical components are mounted on 1.5-in.-diameter stainless-steel posts and thermally compensated ZeroDrift mirror mounts (Newport) throughout the system. An overview photograph of the installed laser heating system at beamline 3-ID-C is presented in Fig. 1(a). Sections II A–II D describe how these considerations were addressed in each part of the system.

A. Laser delivery path

Two continuous-wave ytterbium fiber lasers (IPG Photonics), each operating at a wavelength of 1064 nm with a maximum output power of 100 W, are used to heat the sample loaded in a DAC from both sides independently. Both infrared (IR) lasers deliver an unpolarized TEM₀₀ (Gaussian) beam. Laser operation is controlled

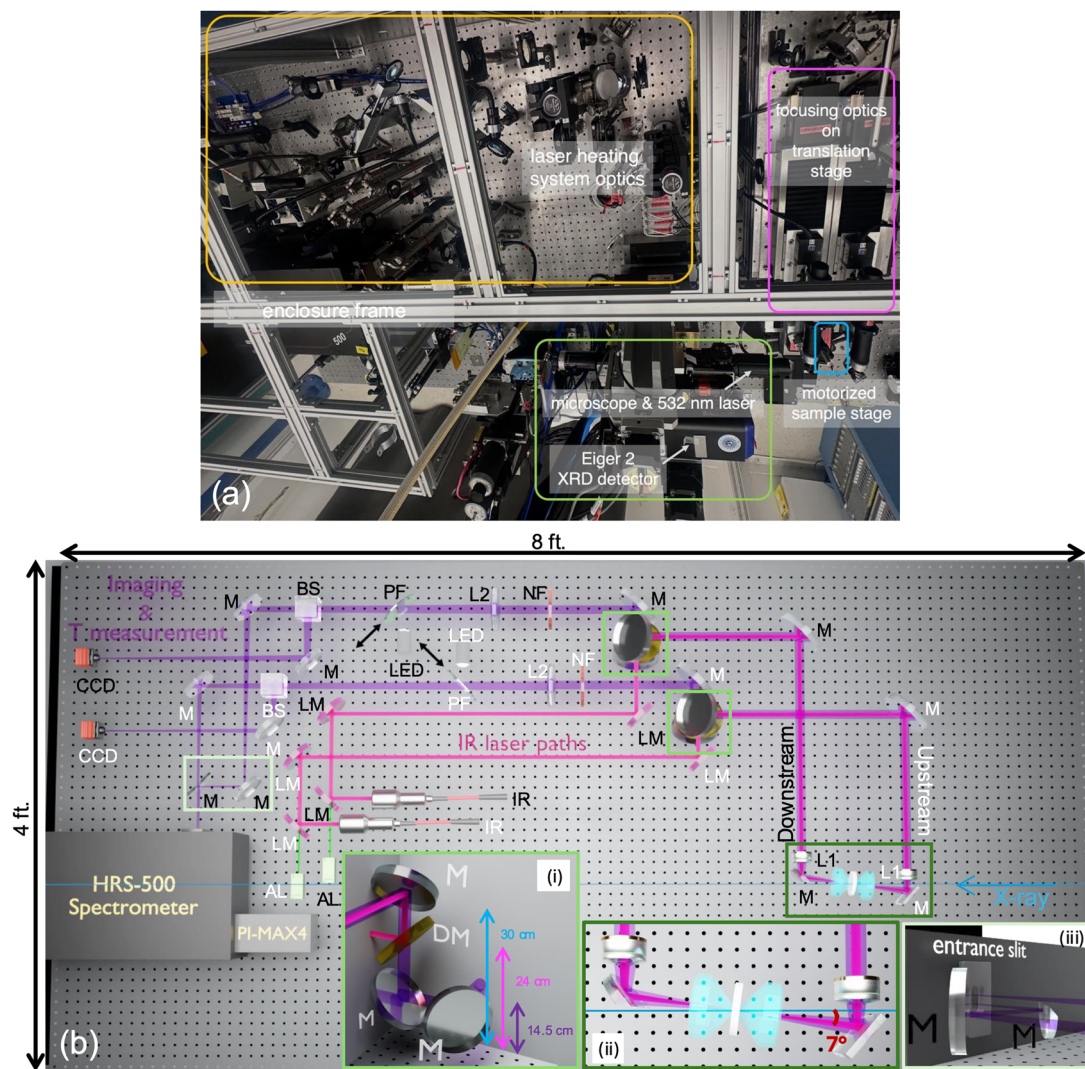


FIG. 1. Double-sided laser heating system at beamline 3-ID-C of APS. (a) Top-view photograph of the setup. (b) 3D schematic layout of the laser heating system. The infrared laser delivery paths are shown in pink, the imaging and temperature-measurement paths are shown in purple, and the x-ray beam path is shown in blue. Upstream and downstream components are labeled in white and black, respectively. The insets show (i) the mirror set used to steer the IR laser to the focused x-ray beam height, with the dichroic mirror reflecting the 1064 nm laser toward the sample while transmitting visible light for imaging and temperature measurement, (ii) the near-sample geometry with the 7° offset between the laser-heating path and the x-ray path, and (iii) the D-shaped mirror used to offset the upstream and downstream thermal-emission signals before the spectrometer entrance slit. IR: 100 W continuous wave ytterbium fiber laser, 1064 nm (IPG Photonics). AL: 1 mW 532 nm alignment laser (Lasermate). LM: 1064 nm Nd:YAG laser mirrors with UV fused silica substrates (Newport). DM: 1064 nm dichroic mirrors (Semrock). M: silver-coated mirrors with Zerodur substrates (Newport). L1: geoHEAT apochromatic objective lens with 61.6 mm focal length (AdiOptica). NF: 1064 nm notch filter (Semrock). L2: achromatic lens with 1000 mm focal length (Thorlabs). LED: 18 W LED illuminators (Schott). PF: 8/92 pellicle beam splitters (Thorlabs). BS: 50/50 non-polarized cube beam splitters (Newport). CCD: cameras for sample imaging (Teledyne FLIR). HRS-500 spectrometer: $f/6.5$ triple-grating spectrometer (Teledyne Princeton Instruments). PI-MAX4: ultrafast, intensified CCD with a 1024×253 pixel chip (Teledyne Princeton Instruments).

remotely using the RS-232 serial communication protocol and external analog inputs integrated in the EPICS (Experimental Physics and Industrial Control System) database and is accessed through a user-friendly graphical interface. Each IR beam is co-aligned with a 532 nm green alignment laser (Lasermate, 1 mW output), which is used for laser and imaging path alignment and provides a visual reference for the invisible IR beam positions. High-energy Nd:YAG

laser mirrors with UV fused silica substrates (Newport) are used to direct both laser beams. Laser delivery paths are shown as pink lines in Fig. 1(b). The major portions of the delivery paths are situated at a height of 24 cm above the optical tabletop. Near the sample, laser beams are steered to a height of 30 cm to match the focused x-ray beam and are aligned with the temperature-reading/imaging axes using two dichroic mirrors (Semrock), as shown in inset (i)

of Fig. 1(b). These dichroic mirrors reflect the 1064 nm IR radiation toward the sample while transmitting visible light from the heated sample for imaging and spectroradiometric temperature measurements.

IR laser beams are focused using two sets of geoHEAT 60-VIS apochromatic objective lenses (L1, $f_{1064\text{ nm}} = 61.6\text{ mm}$, AdlOptica), which are optimized for high-pressure laser-heating experiments. The laser spot size at the best focus, measured by analyzing the intensity distribution along the diameter of the circular heating spots imaged on both surfaces of a Pt foil, has a Gaussian shape with a full width at half maximum (FWHM) of $\sim 25\text{ }\mu\text{m}$. A critical feature of our optical geometry is a 7° offset for the laser delivery path relative to the x-ray axis. By introducing this angular offset, the near-sample mirrors that guide IR lasers into the DAC are positioned outside the x-ray path [as shown in inset (ii) of Fig. 1(b)]. As a result, x-ray absorption is minimized, which improves the signal rate for NRS measurements during laser-heating. This geometry also allows high-performance silver mirrors with Zerodur substrates to be used near the sample, improving image quality and thermal stability without the need for lower-performance, thin amorphous-carbon or beryllium mirrors, which were used in the previous setup at 3-ID-B. The remote tilting of near-sample mirrors is achieved using specially made aluminum holders mounted on motorized goniometers (Newport), with the vertex located at the intersection of the IR laser beam and temperature-reading/imaging axes. This assembly is adapted and modified from the 13-ID-D (GSECARS) design²⁰ to accommodate the specific mirror geometries of our system (a rectangular mirror for the upstream path and a D-shaped mirror for the downstream path). On both the upstream and downstream sides, the geoHEAT lens and near-sample mirror assemblies are mounted on high-precision, high-speed, encoded linear translation stages (Newport) with a 100 nm resolution, enabling precise translation into and out of the sample environment.

B. Sample imaging and temperature measurement

Sample imaging and thermal emission are collected through the same geoHEAT objectives (L1) used for IR laser focusing and are then directed along a common optical path fixed at a height of 14.5 cm above the tabletop (shown as purple lines in Fig. 1). Two 1064 nm notch filters (NF, Semrock) are used to block IR lasers and prevent potential damage to sensitive optics. Both upstream and downstream paths are split after the second achromatic focusing lens (L2, $f = 1000\text{ mm}$, Thorlabs) by a 50/50 cube beam splitter (BS, Newport) into two separate components. The transmitted branch is focused onto the spectrometer entrance slit, while the reflected branch is focused onto the CCD camera (Teledyne FLIR) for sample imaging from both sides. The optical magnification of the system is $\times 16.2$, a substantial improvement over the $\times 10$ magnification of the previous 3-ID-B laser heating system. The illumination and visualization of the sample from both sides using transmitted fiber LED light are achieved by inserting thin-film pellicle beam splitters (PF, Thorlabs) mounted on pneumatic stages. The pellicle beam splitters are fully retracted from the optical path for the T measurement. This configuration enables sample chamber visualization at both 300 K and during laser heating, as demonstrated in Fig. 2.

Thermal emission from the laser-heated sample on both sides is guided to the entrance slit of the SpectraPro HRS-500 spectrometer (Teledyne Princeton Instruments). As shown in inset (iii) of

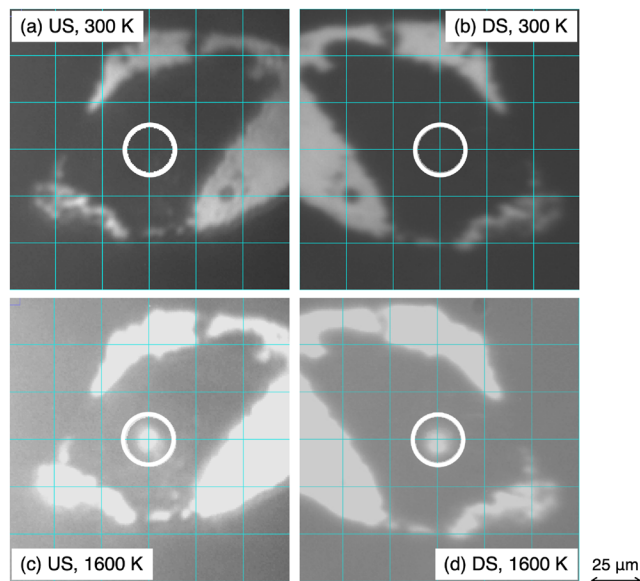


FIG. 2. Representative downstream (DS) and upstream (US) images of a DAC sample acquired (a) and (b) at 300 K and (c) and (d) during laser heating, demonstrating the image quality available for monitoring the sample chamber and heated region during high P - T measurements. The white circle marks the center of the CCD camera field of view and is aligned with the x-ray beam position.

Fig. 1(b), the upstream and downstream paths are merged before the entrance slit using a D-shaped mirror, which reflects the downstream signal, while the upstream signal propagates over the mirror edge. This geometry allows images from both sides of the sample to be centered simultaneously on the PI-MAX4 detector when the HRS-500 spectrometer is operated in imaging mode.

The final alignment between the x-ray beam, sample image, heating spot, and spectroradiometric collection path is established using a $\sim 15\text{ }\mu\text{m}$ ruby sphere placed at the sample position. The x-ray beam can be visualized directly from x-ray-induced ruby fluorescence and aligned to the center reference mark of each imaging CCD camera field of view. With the ruby image centered on the reference mark of those cameras, the same ruby sphere is simultaneously imaged on the PI-MAX4 detector, while the HRS-500 spectrometer is operated in imaging mode [Figs. 3(a) and 3(b)], thereby co-aligning the imaging CCDs and the spectrometer-detector system. After the x-ray position and spectroradiometric collection path are aligned, the IR laser beams are brought to the same position by adjusting motorized IR steering mirrors. The laser position is further verified by heating a Pt foil and confirming that the hotspot is centered on the same reference mark [Fig. 3(c)].

This procedure ensures that the spectroradiometric temperature measurement probes the same region as the x-ray beam and the heating spot from both sides throughout the experiment. The entrance slit of the spectrometer is typically set to $100\text{ }\mu\text{m}$, corresponding to a projected collection width of $\sim 6\text{ }\mu\text{m}$ at the sample position. This collection width is comparable to the focused x-ray beam size of ($\sim 4 \times 13\text{ }\mu\text{m}^2$) and is smaller than the heating spot size of $\sim 25\text{ }\mu\text{m}$ FWHM, so the measured thermal emission is collected from the central portion of the x-ray-probed and

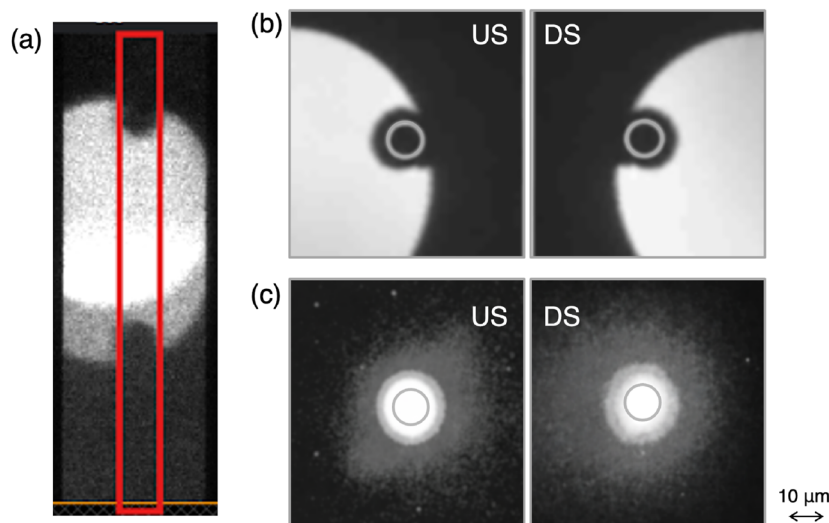


FIG. 3. Alignment of the x-ray beam, imaging system, spectrometer, and heating spot. (a) A $\sim 15 \mu\text{m}$ ruby sphere at the sample position imaged on the PI-MAX4 detector with the HRS-500 spectrometer in imaging mode, used to co-align the spectrometer-detector system with the imaging CCDs. The red box marks the center of the entrance slit. (b) Ruby sphere aligned to the gray reference mark on the both US and DS imaging CCDs using x-ray-induced ruby fluorescence. (c) US and DS hotspot images on a Pt foil, confirming that the alignment of the IR laser-heated spot is aligned with the same reference position.

laser-heated region. During temperature measurements, the upstream and downstream thermal-emission signals are vertically offset by 3 mm, which allows two separate regions of interest (ROIs) to be used for independent upstream and downstream temperature determination [Fig. 4(a)]. Within the spectrometer, the incoming light is collimated by the spectrometer's first mirror and subsequently energy-dispersed by either 150 grooves/mm (800 nm

blaze wavelength) or 300 grooves/mm (500 nm blaze wavelength) gratings. The resulting spectra are captured by the PI-MAX 4 time-gated intensified CCD detector (Teledyne Princeton Instruments), which can be electronically gated to enable fast and reliable temperature measurements during laser heating experiments.

In order to obtain a reliable T measurement, the system response is calibrated by collecting a thermal radiation spectrum

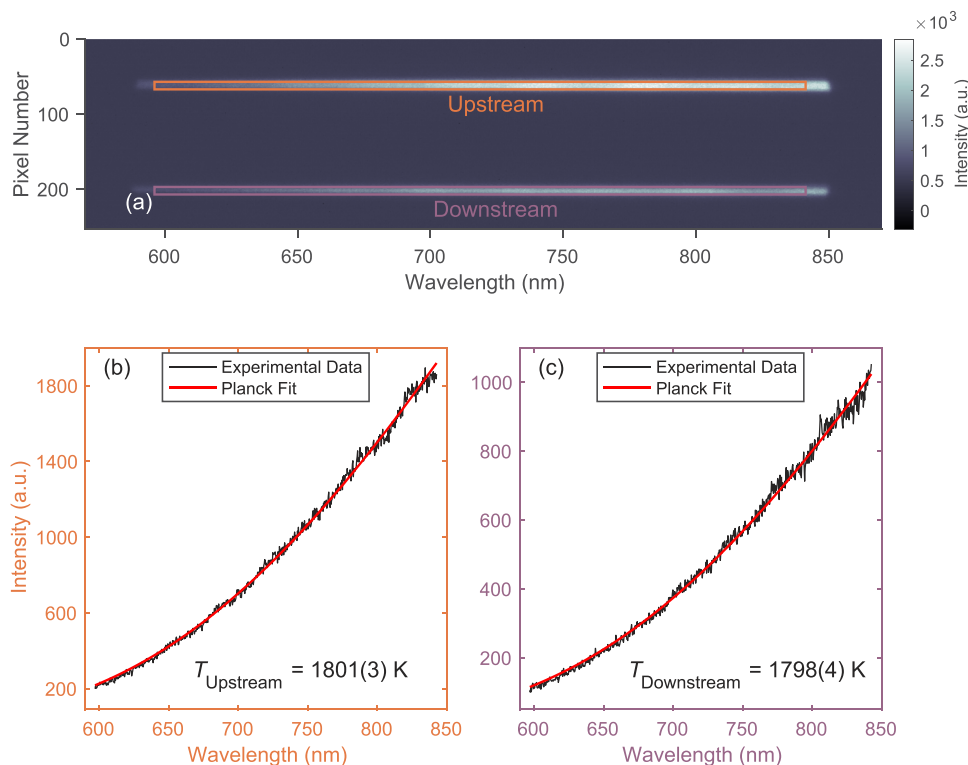


FIG. 4. Example of temperature measurements on a heated DAC sample at around 68 GPa. (a) Data collected with an exposure time of 20 ms. The orange and purple boxes indicate the ROIs used in the T-Rax software for temperature fitting.²⁶ Thermal radiation spectra (black curves) of (b) upstream and (c) downstream are fitted using the gray body approximation of Planck's law (red curves). The reported uncertainties only represent fitting uncertainties and do not include systematic contributions from calibration, alignment, wavelength range, and the gray body approximation.

from a tungsten (W) lamp (Gigahertz-Optik) at 3162 K. The W lamp is placed at the sample position by verifying that the filament is in focus on the imaging CCD, and the reference spectra are taken from upstream and downstream separately. The final T is calculated by fitting a Planck function in the gray body approximation to the obtained intensity profile (background-subtracted and response-calibrated) using the T-Rax software.²⁶ The version deployed at 3-ID-C is integrated with EPICS and can directly access process variables, enabling automated and real-time temperature readout during experiments (implemented by C. Skordas, GSE-CARS). Representative temperature measurements collected with the 150 grooves/mm grating, which provides a broader spectral window for spectroradiometric fitting, are shown in Figs. 4(b) and 4(c).

C. Laser heating combined with nuclear resonant scattering

A schematic of the NRS setup at 3-ID is presented in Fig. 5. The incident x-ray beam is tuned to 14.4 keV using a liquid-nitrogen-cooled silicon high-heat-load monochromator (HHLM). The beam for NRS experiments is then further monochromatized by a silicon high-resolution monochromator (HRM) located in the 3-ID-B

hutch to achieve the ~ 1 meV energy bandwidth required for ^{57}Fe NRS.²⁷ The recent transition of the ^{57}Fe NRS program to the 3-ID-C station allows the 3-ID-B hutch to remain isolated during experimental operations. This eliminates the frequent thermal disturbances caused by hutch access for sample changes. Consequently, the 3-ID-B environment maintains exceptional thermal stability with fluctuations of less than 5 mK per h and 50 mK over days,¹⁰ which is critical for the HRM during long-duration experiments. The meV x-ray beam is focused by a pair of bendable KB mirrors to ~ 4.3 (horizontal) $\times$ 13.4 (vertical) μm^2 , measured by knife-edge scans using tungsten rods (Fig. 6); the larger vertical size is mainly associated with the asymmetric crystal reflections of the HRM in the vertical diffraction plane. Clean-up slits can be positioned between the KB mirrors and the sample to reduce the tail intensity of the spatial x-ray distribution during the experiment.

For high P - T NRIXS measurements, miniature panoramic DACs (mini-pDACs, DAC Tools) with two wide side-openings, adapted from earlier designs for NRIXS studies at high- P and low- T ,^{28,29} are used for their compatibility with the laser heating system [shown in Fig. 7(a)]. Two avalanche photodiodes (APDs) are positioned ~ 10 mm from the sample to collect the NRIXS signals. A third APD, located in the forward-scattering direction, is used to measure the instrumental resolution function during NRIXS scans

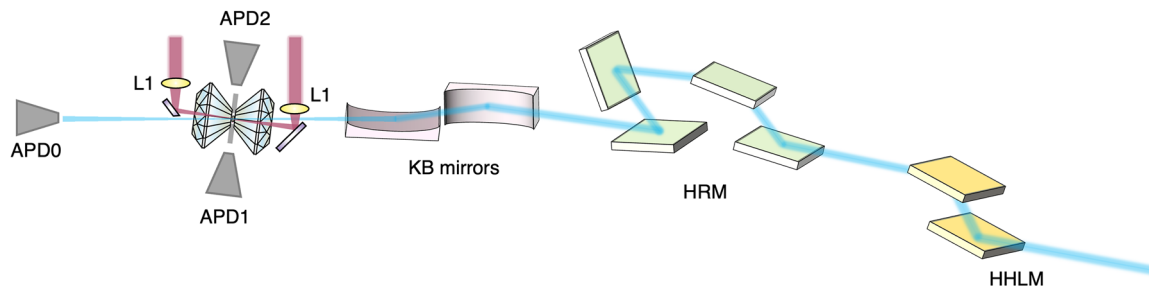


FIG. 5. Experimental setup for NRS under high pressure and high temperature. HHLM: liquid-nitrogen-cooled silicon high-heat-load monochromator. HRM: silicon high-resolution monochromator. KB mirrors: Kirkpatrick–Baez mirrors for x-ray focusing in both horizontal and vertical directions. APD: avalanche photodiode detectors for SMS (APD0) and NRIXS (APD1 & 2). Eiger2: detector for *in situ* XRD measurements (Dectris). 532 nm Laser: 200 mW laser for ruby fluorescence measurements (Lasermate).

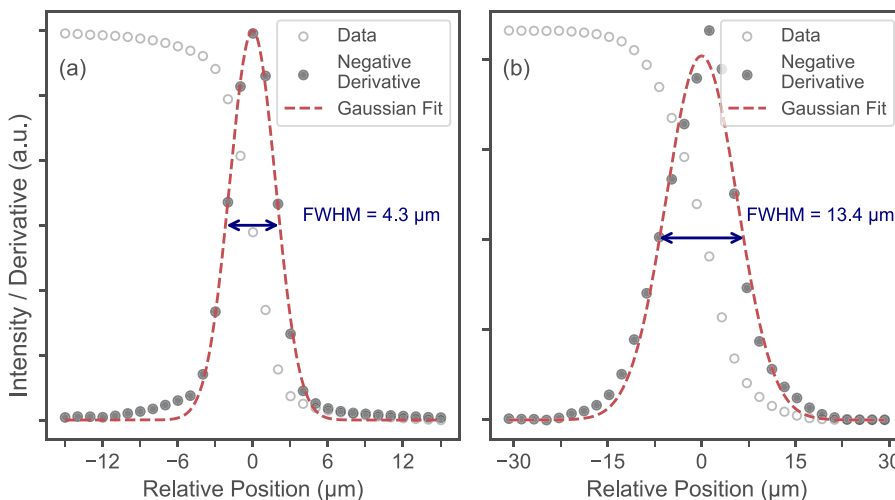


FIG. 6. Size of the focused x-ray beam measured by knife-edge scans along (a) horizontal and (b) vertical directions. The open and solid circles represent the raw prompt x-ray intensity and negative derivatives of the raw profile, respectively. The Gaussian fit (red dashed curves) of the derivatives yields a spot size of 4.3 (horizontal) $\times$ 13.4 (vertical) μm^2 .

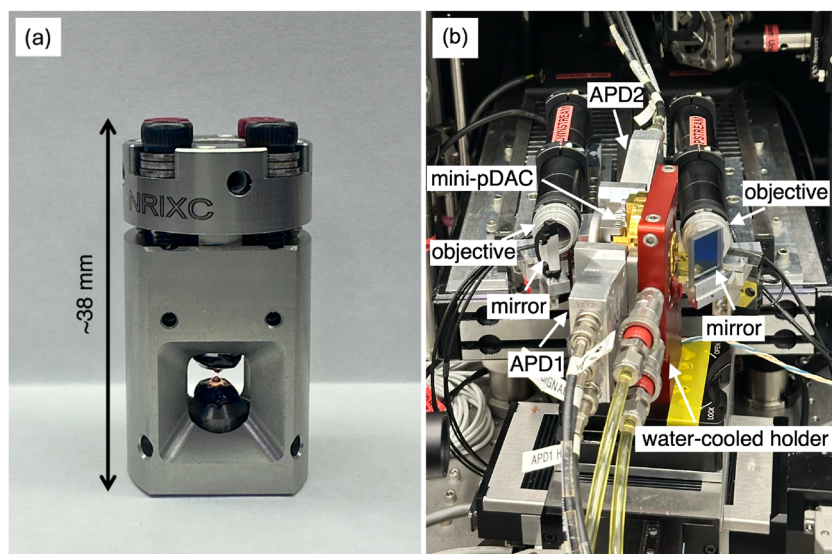


FIG. 7. Photographs of the mini-pDAC and experimental setup for high P - T NRXS measurements. (a) Miniature panoramic diamond anvil cell (mini-pDAC, DAC Tools) with a total height of ~ 38 mm. (b) Mini-pDAC mounted in a water-cooled holder to dissipate heat from the DAC body during laser heating. Near-sample laser heating optics are moved into position. Two APDs are placed close to the DAC side-openings to collect NRXS signals.

simultaneously. In the high P - T SMS configuration, symmetric and 3-pin DACs are used, with a single APD positioned ~ 1 m downstream of the sample along the x-ray path to capture the forward scattering signal. The sample loaded in a DAC is positioned at the rotation center, as confirmed by x-ray scans over a $\pm 5^\circ$ rotation range. The DAC is then rotated to 7° for the measurements so that the sample surface is approximately perpendicular to the incident IR lasers, while keeping the x-ray beam close to the DAC compression axis. This geometry also orients the DAC openings more directly toward the near-sample mirrors, improving the collection of thermal emission from the heated sample and enhancing the quality of T measurements. Water-cooled holders are used to minimize the heating of the DAC body [Fig. 7(b)], thereby enhancing mechanical stability and reducing progressive misalignment during data acquisition.

In addition, *in situ* XRD can be performed using an EIGER2 X 500K detector (Dectris). In the current implementation, this capability is primarily used for room-temperature, high-pressure measurements to determine unit-cell volumes of the sample and pressure medium, thereby constraining *in situ* sample pressure before and after laser heating. The detector is positioned to maximize the accessible 2θ range within the constraints imposed by DAC openings and x-ray energy ($E = 14.4125$ keV, $\lambda = 0.8603$ Å). The extension of this capability to high P - T measurements is under development, as the accessible diffraction angle is further limited during laser heating by the 7° DAC rotation required for the laser heating and thermal-emission collection geometry. Online ruby fluorescence pressure measurements using a 200 mW, 532 nm DPSS Laser (Lasermate) are available for room-temperature pressure gauging. The resulting fluorescence signal is dispersed by a 1200 grooves/mm grating (500 nm blaze) and recorded using the PI-MAX 4 detector.

D. Improvements relative to the previous 3-ID laser-heating setup

Compared with the previous laser heating implementation at 3-ID-B,¹⁰ the present system provides a dedicated and substantially

more stable laser-heating platform for high P - T NRS experiments at 3-ID-C. In the previous implementation, parts of the laser-heating optics were built on an upper tier above the optical table where NRS experiments were performed. In contrast, the present system is built directly on the same optical table as the sample stage, detectors, and other NRS-related components, eliminating the two-tier structure. The major portions of the optical paths are kept at a lower height and steered upward only near the sample position to intersect the focused x-ray beam. Together with 1.5-in.-diameter stainless-steel posts and thermally compensated ZeroDrift mirror mounts throughout the system, this layout reduces mechanical drift and improves thermal stability during extended SMS and NRXS measurements.

The 7° offset between the x-ray axis and the laser-delivery paths is a key feature of the near-sample optical geometry. This offset positions the final IR steering mirrors outside the incident x-ray path, thereby reducing x-ray absorption by near-sample optical components and improving the NRS signal rate during laser heating. It also removes the need for thin amorphous-carbon or beryllium mirrors near the sample, allowing high-performance mirrors with thermally stable substrates to be used instead. The reduced x-ray absorption further enables direct visualization of x-ray-induced fluorescence from KCl or ruby inside a DAC sample chamber, which is now routinely used to confirm the x-ray position and align it with the reference mark of the imaging cameras during experiments.

Improved imaging quality and faster, more reliable spectroradiometric temperature measurement are achieved through several optical and detector upgrades. The use of geoHEAT 60-VIS objectives improves the imaging quality and increases the optical magnification from the previous $\times 10$ to $\times 16.2$, allowing more reliable visualization and alignment of the laser-heated region relative to the smaller x-ray focus. In addition, all metallic mirrors in the present system are silver-coated, whereas parts of the previous system used aluminum-coated mirrors. The higher and smoother reflectivity of silver over the visible-to-near-infrared range improves optical throughput and spectral response for both imaging and

spectroradiometric temperature measurements. Temperature measurement has also been upgraded with an HRS-500 spectrometer and a PI-MAX4 detector, together with the T-Rax fitting program integrated into EPICS, allowing fast and reliable temperature readout during experiments.

For high P - T NRIXS, the present system is compatible with mini-pDACs adapted from previous high- P , low- T NRIXS designs. This compatibility is important because the 61.6 mm working distance of the geoHEAT objectives leaves limited space around the sample for the DAC and holder, while NRIXS measurements require wide access for side APD collection. The mini-pDAC geometry, together with water-cooled DAC holders, provides the optical access, detector access, and thermal stability needed for hours of high P - T NRIXS measurements.

In addition, the system accommodates complementary diagnostics, including online ruby fluorescence pressure measurements and x-ray diffraction using an EIGER2 X 500K detector. Installation at the downstream 3-ID-C experimental hutch also provides an additional stability benefit by spatially separating operations from the HRM in 3-ID-B. Sample changes and laser-heating operations can now be performed without accessing the HRM hutch, thereby eliminating frequent thermal disturbances to the HRM environment during experiments. Because the HRM stability is critical for reducing energy or intensity drift relative to the ^{57}Fe nuclear resonance over long data-acquisition periods, this separation provides an additional advantage for both SMS and NRIXS measurements.

The reduced x-ray beam size after APS-U, together with the smaller heating spot achieved with the new optical design, also improves the spatial matching between the x-ray-probed volume, laser-heated region, and temperature-measurement area. This improved overlap is particularly important for measurements above 100 GPa, where small sample drift or misalignment can otherwise affect measured temperature, signal intensity, and data quality. The present system, therefore, directly addresses the main limitations of the previous setup and provides a more stable platform for both SMS and NRIXS experiments under simultaneous high P - T

conditions. The routinely achievable temperature depends more on DAC sample preparation, including sample size, thickness, laser-absorber properties, and thermal insulation. During commissioning, the system reached temperatures up to ~ 4500 K for approximately 1 min at ~ 70 GPa, and sustained heating below 2000 K for several hours during high- P NRIXS measurements.

III. EXAMPLES OF APPLICATIONS

Examples of successful applications of the developed system are presented in Secs. III A–III C, illustrating its capabilities with NRS techniques. High P - T NRS experiments are conducted at 3-ID-C of APS in swap-in/swap-out mode with 48 bunches that are separated by 76.7 ns. Sections III A–III C highlight the system performance for both SMS and NRIXS experiments, including stable measurements under laser-heating, reliable temperature determination, and high-quality data collected at high P - T .

A. Synchrotron Mössbauer spectroscopy of ^{57}Fe at high P - T

As a demonstration of SMS measurements with the laser heating system, SMS time spectra were collected from ^{57}Fe at $P_{300\text{K}} = 55 \pm 2$ GPa. Two type-I diamond anvils with 300 μm flat culets were mounted in a symmetric DAC. One 250 μm thick rhenium gasket was pre-indented to 50 μm thick, and a 125 μm -diameter sample chamber was drilled using the laser drilling system at HPCAT.³⁰ The sample was prepared from a 95% isotopically enriched ^{57}Fe foil, thinned to 7 μm , and cut into a $40 \times 55 \mu\text{m}^2$ section using the Ge focused ion beam at the Center for Nanoscale Materials, Argonne National Laboratory. The sample was sandwiched between two dehydrated KCl layers, which served as both the pressure-transmitting medium and thermal insulation. Pressure at 300 K was constrained from *in situ* XRD measurements collected at 3-ID-C using the Eiger 2 detector [Fig. 8(a)].

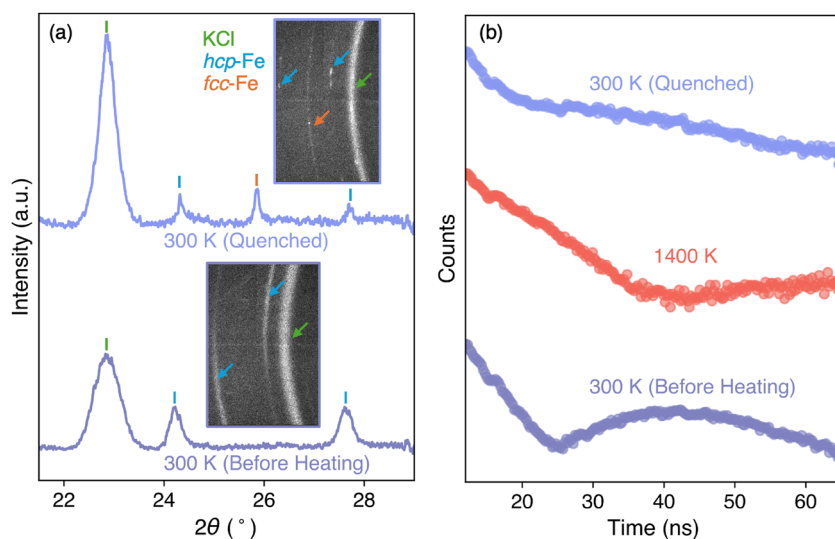


FIG. 8. SMS and XRD measurements of ^{57}Fe at $P_{300\text{K}} = 55 \pm 2$ GPa. (a) Integrated XRD patterns collected at 300 K before heating and after quenching from 2700 K. The insets show corresponding detector images. The quenched XRD pattern indicates recrystallization and formation of *fcc*-Fe sample after heating. (b) SMS time spectra collected at 300 K before heating, at ~ 1400 K during laser heating and at 300 K after quenching. The SMS spectra are vertically offset for clarity.

A 300 K SMS time spectrum was first collected before heating. The sample was then heated to ~ 1400 K for ~ 3 min, during which a high- T SMS spectrum was acquired at this temperature. Afterward, the laser power was increased from both sides to heat the sample to ~ 2700 K, followed by rapid quenching to room temperature. A final 300 K SMS spectrum and an XRD pattern were collected after quenching from 2700 K. The three SMS time spectra are shown in Fig. 8(b). The spectrum collected at ~ 1400 K differs noticeably relative to the initial room-temperature time spectrum, reflecting the temperature dependence of the nuclear resonant response from the laser-heated ^{57}Fe sample. The post-quench spectrum differs from the initial room-temperature spectrum, indicating that the x-ray-probed sample volume was modified during the high-temperature excursion; this is consistent with post-quench XRD observations confirming sample recrystallization and formation of *fcc*-Fe after heating and quenching from 2700 K [Fig. 8(a)]. These results demonstrate the SMS compatibility under high P - T conditions.

B. Melt detection using synchrotron Mössbauer measurements

The laser heating system was further tested in an SMS melting experiment on $^{57}\text{Fe}_{0.8}\text{Ni}_{0.1}\text{Si}_{0.1}$ at $P_{300\text{K}} = 63 \pm 2$ GPa. One 3-pin DAC equipped with two 300 μm flat culet diamond anvils was used for high-pressure generation. A 250 μm thick rhenium gasket was pre-indented to a thickness of 55 μm , and a 120 μm sample chamber hole was drilled using an electron discharge machine (EDM). A 95% isotopically enriched $^{57}\text{Fe}_{0.8}\text{Ni}_{0.1}\text{Si}_{0.1}$ foil with a thickness of 10 μm was cut into a $65 \times 80 \mu\text{m}^2$ section and sandwiched between dehydrated KCl layers that served as both the pressure-transmitting medium and thermal insulation. One ruby sphere was loaded without contact with the sample and used as the pressure marker. Ruby fluorescence and Raman spectroscopy of the diamond anvils (diamond edge) were collected using the offline Raman spectroscopy system at GSECARS of the APS,³¹ and the pressure at 300 K was determined to be 63 ± 2 GPa.

The SMS heating run followed a standardized experimental sequence as described in previous studies to detect the melting of Fe,¹³ $\text{Fe}_{0.9}\text{Ni}_{0.1}$,¹⁴ $\text{Fe}_{0.8}\text{Ni}_{0.1}\text{Si}_{0.1}$ ¹⁵ and FeO.¹⁶ The approximate positions of the x-ray beam and laser-heated spot were first aligned visually on upstream and downstream camera images using the x-ray-induced fluorescence of KCl. The alignment was further verified by performing x-ray absorption scans in both horizontal and vertical directions to map the topography of the sample and avoid parallax errors. Before heating, the sample was annealed at ~ 1500 K for ~ 3 min, and the laser power ratio is adjusted to balance the upstream and downstream temperatures. A high- T SMS time spectrum collected during annealing was used to constrain the sample effective thickness, a key parameter for the subsequent fitting procedure. The sample was then heated through an incremental laser-power ramp controlled by a programmed acquisition sequence, while the time-integrated delayed counts were recorded for every 3-s interval. Sample temperatures were monitored from both sides by spectroradiometry and fit with gray-body radiation curves. Simultaneous monitoring of transmitted x-ray flux via ionization chambers confirmed that sample thinning remained below 2% prior to the onset of melting.

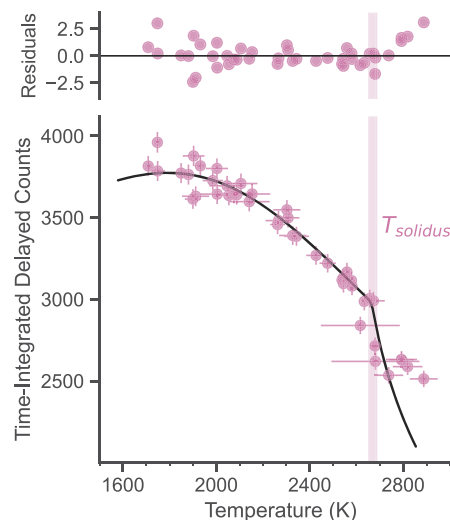


FIG. 9. Determining the solidus temperature of $^{57}\text{Fe}_{0.8}\text{Ni}_{0.1}\text{Si}_{0.1}$ at $P_{300\text{K}} = 63 \pm 2$ GPa. The time-integrated delayed counts, collected in 3 s acquisition intervals, are plotted as a function of temperature. The solid black curve results from fit using the SIMX module of the MINUTI software package.³² The best-fit solidus temperature and uncertainty are indicated as the shaded bar.

The solidus temperature is quantitatively determined by fitting the temperature dependence of delayed counts with an experiment-specific scattering intensity model using the SIMX module in the software MINUTI 2.4.0³² (Fig. 9), resulting in a solidus temperature that is in very good agreement with the reported solidus curve of $\text{Fe}_{0.8}\text{Ni}_{0.1}\text{Si}_{0.1}$ ¹⁵ under similar pressure conditions using the same technique. This consistency provides an essential validation of the newly developed system, demonstrating that the high P - T SMS data quality achieved at 3-ID-C is fully sufficient for quantitative melting determination under extreme conditions.

C. High-temperature nuclear resonant inelastic x-ray scattering

Iron powder, isotopically enriched to 97% in ^{57}Fe , was used as the starting material and lightly pressed between two opposing diamond anvils to form 10 μm -thick flakes. One mini-pDAC with 300 μm flat culet anvils was used. A beryllium gasket was pre-indented to about 50 μm , and a 180 μm hole was drilled using the laser drilling system at HPCAT.³⁰ To strengthen the gasket, the hole was filled with amorphous boron epoxy. This filled gasket was then laser-drilled again at its center to form the final sample chamber with a diameter of 120 μm . One ^{57}Fe flake was sandwiched between two dried KCl layers that served as both the pressure-transmitting medium and thermal insulation. Pressure at 300 K was constrained by *in situ* XRD measurements performed at both 3-ID-C and GSECARS' beamline 13-BM-C and was determined to be 52.8 ± 0.9 GPa using equations of state of Fe³³ and KCl.³⁴

High P - T NRIXS energy scans were collected from the laser-heated sample. The sample surface temperature was maintained at ~ 1600 K, with fluctuations characterized by a standard deviation of 12 K over 3.5 h based on spectroradiometric measurements [Fig. 10(a)], providing sufficiently stable conditions for NRIXS data

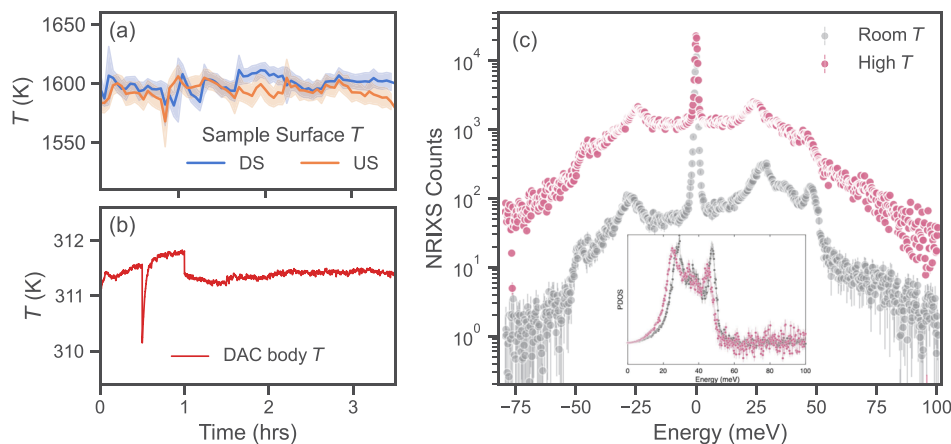


FIG. 10. NRIXS measurements of ^{57}Fe at $P_{300\text{K}} = 52.8 \pm 0.9$ GPa during 3.5 h of continuous laser heating. (a) Sample surface temperatures measured from the US and DS sides by spectroradiometry. (b) DAC body temperature monitored by a thermistor during heating. (c) NRIXS energy scans collected at high- T , with the room-temperature data included for comparison. The data are vertically offset for clarity. The inset shows the corresponding PDOSs derived from the high- T and room- T NRIXS data.

collection. A thermistor is attached to the DAC body to monitor its temperature rise during extended data collection [Fig. 10(b)]. The energy scan range was -80 to $+100$ meV around the ^{57}Fe nuclear resonance energy, with a step size of 0.25 meV and a counting time of 2 s per step. Each scan required ~ 30 min, and seven spectra were collected and summed to improve counting statistics. For comparison, room-temperature NRIXS energy scans were also collected from the same sample before laser heating at $\sim 1600\text{K}$, with 13 scans acquired and summed. XRD patterns collected before and after the heating sequence showed no diffraction peaks attributable to iron carbide.

High- T and room- T NRIXS energy scans collected in this study are plotted in [Fig. 10(c)]. The detailed-balance principle, reflected in the ratio of probabilities for the phonon creation and annihilation of an NRIXS spectrum, can be used to determine the mean temperature of the sample volume probed by the x-ray beam.^{35,36} The higher symmetry of the high- T data provides clear evidence that the sample was maintained at an elevated temperature during data acquisition. The mean temperature derived from the detailed-balance principle is 1480 (30) K, about 120 K lower than the spectroradiometric surface temperature of ~ 1600 K. This level of agreement is reasonable given the expected thermal gradients of laser-heating.²⁵ Meanwhile, the DAC body temperature remained stable within several degrees during heating owing to efficient water cooling, which is critical for both mechanical stability and APD operations. The high- T data are of sufficient quality to derive the phonon density of state (PDOS), which shows a shift toward lower energies relative to the room- T PDOS, and the initial slope in the low-energy regime increases noticeably, consistent with thermal phonon softening.

IV. CONCLUSIONS

We have developed and commissioned a high-performance laser heating system at beamline 3-ID-C of APS for diamond anvil cell experiments, providing an improved platform for stable, long-duration NRS measurements under simultaneous high P - T conditions. The stability of the new system at 3-ID-C and the 7° angular offset design are marked improvements compared with the

former setup at 3-ID-B. High-level system performance has been demonstrated through representative high P - T SMS and NRIXS experiments that validate its thermal and mechanical stabilities, reliable temperature determination, and high data quality. With this new capability now operational, 3-ID provides a platform for high P - T NRS measurements that will enable new studies of materials under extreme conditions, with broad applications in deep Earth geophysics, planetary science, and high-pressure physics. Future experiments at even higher pressures and temperatures will further expand the accessible P - T range and the scientific scope of this capability.

Looking ahead, the implementation of pulsed laser-heating will be essential for NRIXS measurements at ultrahigh temperatures, which can potentially reduce chemical reactions and sample instabilities within the DAC.³⁷ In addition, the availability of a smaller, 1 – 2 μm -scale x-ray beam will motivate further development of the laser heating system,³⁸ including laser beam expansion and shaping optics for variable heating spot sizes and flat-top laser profiles, to fully exploit the reduced beam size and maintain optimal spatial overlap with the x-ray probe under ultrahigh P - T conditions.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Cijin Zhou: Conceptualization (supporting); Data curation (lead); Formal analysis (lead); Investigation (lead); Methodology (supporting); Visualization (lead); Writing – original draft (lead); Writing – review & editing (lead). **Barbara Lavina:** Conceptualization (supporting); Data curation (supporting); Methodology (supporting); Supervision (supporting); Writing – review & editing (supporting). **Thomas S. Toellner:** Data curation (supporting); Methodology (supporting); Supervision (supporting); Writing – review & editing (supporting). **Vitali B. Prakapenka:** Conceptualization (supporting); Methodology (lead); Resources (supporting); Supervision (lead); Writing – review & editing (supporting). **Stella Chariton:** Methodology (supporting); Resources (supporting); Supervision (supporting); Writing – review & editing (supporting). **Dongzhou Zhang:** Data curation (supporting); Methodology (supporting); Resources (supporting); Supervision (supporting); Writing – review & editing (supporting). **Mark L. Rivers:** Methodology (supporting); Resources (supporting); Software (supporting); Supervision (supporting). **Wolfgang Sturhahn:** Conceptualization (supporting); Data curation (lead); Formal analysis (equal); Investigation (supporting); Methodology (supporting); Supervision (supporting); Validation (equal); Writing – review & editing (supporting). **Jennifer M. Jackson:** Conceptualization (supporting); Data curation (lead); Formal analysis (equal); Funding acquisition (supporting); Investigation (equal); Resources (supporting); Supervision (supporting); Validation (equal); Writing – review & editing (supporting). **Jiyong Zhao:** Conceptualization (lead); Data curation (supporting); Funding acquisition (lead); Methodology (lead); Project administration (lead); Resources (lead); Supervision (lead); Writing – review & editing (supporting).

DATA AVAILABILITY

The data that support this study are available from the corresponding authors upon reasonable request.

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