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ABSTRACT

Combination of *in situ* laser heating with single-crystal X-ray diffraction (scXRD) in diamond anvil cells (DACs) provides a tool to study crystal structures and/or chemistry of materials at simultaneous high pressures and high temperatures. Here, we describe the first dedicated single-crystal X-ray diffractometer coupled with double-sided laser heating (dsLH) system. The scXRD/dsLH setup was developed for the P02.2 Extreme Conditions Beamline at PETRA III and became available for general users in 2017. It enables the collection of reliable scXRD data at simultaneous high pressure and high temperature. We demonstrate the performance of the setup on example of studies of iron and chromium nitrides.

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I. INTRODUCTION

Diamond anvil cell technique provides a tool to generate pressures, which can exceed those in the center of the Earth (360 GPa), and to collect *in situ* spectroscopy (Raman, Mössbauer, X-ray absorption, etc.) and/or X-ray diffraction data. However, the analysis and interpretation of the data obtained at multimegabar pressures is extremely difficult, especially due to multiple phase transitions and/or chemical reactions, formation of unusual crystal structures and/or chemical compounds unknown at ambient conditions.

Single-crystal X-ray diffraction (scXRD) in DACs opens a way to determine not only the crystal structure of a material but also to

refine its chemical composition for even very complex multiphase and/or multigrain samples. Due to significant progress in the field of high-pressure technology, since a decade scXRD became available at pressures above ~15 GPa. Novel gas-loading systems^{1–6} allow one to use soft pressure transmitting media (helium or neon) in DACs, thus preserving single crystals to megabar pressures. New types of diamond anvils and DACs with wide X-ray opening up to 90°⁷ enlarge the coverage of the reciprocal space and thus improve the data redundancy which leads to the higher quality of the final structural models.

Heating is an important parameter for high-pressure experiments, especially for studies dedicated to modeling of processes in

deep Earth's interior. Double-sided laser-heating (dsLH) systems are widely used at synchrotron facilities.^{8–11} However, until recently, all of them were stationary and did not allow DAC rotation during simultaneous heating, thus making *in situ* high-pressure high-temperature scXRD impossible. A portable laser-heating system, first developed in Bayerisches Geoinstitut (BGI),¹² can be mounted directly on a goniometer stage and rotated along with the DAC, providing the opportunity to collect scXRD simultaneously during laser heating.

Here, we describe the first dedicated scXRD/dsLH setup consisting of a single-crystal X-ray diffractometer coupled with double-sided laser heating system. The setup was developed for beamline P02.2, the Extreme Conditions Beamline (ECB), at PETRA III and became available for the general users in 2017.

II. SETUP OVERVIEW

The scXRD/dsLH setup is mounted on the General Purpose experiment of beamline P02.2¹³ (Fig. 1). The major components of the setup (from the top to the bottom) are listed below and shown in Fig. 2:

- *sample holder* equipped with water cooling;
- *two portable universal laser heating heads* (UniHeads, PrecitecTM) equipped with 4 mm-thick glassy carbon mirrors; one UniHead is mounted horizontally (the upstream h-UniHead), the other one, vertically (the downstream v-UniHead); and
- *multiaxis positioning systems* realized through the combination of translation positioning stages:
 - motorized Newport X- and Y-stages and motorized Huber Z-stage for DAC alignment;
 - motorized Huber XY- and Huber Z-stages for upstream h-UniHead; and

- manual Standa Z-stage for downstream v-UniHead;

- *stabilization elements* (aluminum frame with steel pillars and supporting corner elements, clamp for h-Unihead, counterbalance for the dsLH system, etc.) to decrease vibrations and improve the stability of heating during rotation; and
- *4-axis positioning system* for alignment of dsLH system composed of the following translation stages:

- *Huber motorized XY-stage;*
- *rotational positioning device, 2-circle (ω -2 θ) Huber goniometer; and*
- *heavy-load Huber motorized Z-stage;*

- *heavy-load Huber motorized XY-stages* for the alignment of the ω rotation axis to the focus of the X-ray beam;
- *aluminum base plate* (50 mm thick) integrating the entire setup, including an X-ray diode and a beamstop; and
- *additional components of the setup*, which are not shown in Fig. 2:

- *two SPI RedPower R4 fiber lasers* (50 W, excitation wavelength of 1070 nm);
- *two Ocean Optics QE65000 spectrometers* for collection of the thermal radiation; and
- *(optional): 2 θ -movable detector arm* mounted on ω -stage (for Lambda or Pilatus detectors).

The scheme of the optical components in the dsLH system is adopted from Kuppenko *et al.*¹² (see Fig. 3). Two UniHeads¹² are used to focus the laser beams on the sample from both the sides (Figs. 2 and 3). Each of them includes a compact set of optical components. The 90° arrangement of the two UniHeads serves to minimize the shadows cast on the detector by various components of the system to reduce possible vibrations of the optical components and to achieve the largest possible ω rotation range. The glassy carbon

FIG. 1. Overview of the scXRD/dsLH setup mounted on the General Purpose experiment at PETRAIII beamline P02.2 (ECB). The primary X-ray beam passes through the set of Compound Refractive Lenses (CRLs) focusing the beam down to $\sim 5 \times 5 \mu\text{m}^2$. Lead shielding covers X-ray optics in order to reduce parasitic scattering. During rotation of the scXRD/dsLH setup as a whole, diffraction data are collected using a Perkin Elmer XRD1621 flat panel detector. To allow rotation, the X-ray pinhole is located at a distance of about 80 mm from the sample. The intensity monitor (diode) and the beamstop are attached to the mounting plate of the scXRD/dsLH setup. Platinum-foil (Pt-foil) absorbers can be optionally used to attenuate the primary X-ray beam intensity.

FIG. 2. ScXRD/dsLH setup: view from the downstream side (a); from the upstream side (b); and perpendicular to the X-ray beam (c).

mirrors are mounted at an angle to the axes of the UniHeads [Figs. 2(c) and 3] and serve as a reflector of the laser light in order to direct it to the sample. The upstream mirror is mounted precisely at 45°, which aligns the laser beam parallel (coaxial) to the X-rays that penetrate the mirror. The downstream mirror is placed at a larger angle, between 50° and 55°, to avoid primary X-ray beam interaction and prevent strong unwanted diffuse scattering. Both the mirror holders are connected to the Newport piezo mounts for fine tuning of the final laser beam position.

For the sample illumination during alignment, the UniHeads are equipped with built-in light emitting diodes (LEDs) controlled externally. In the system, the sample is visualized using μ EYE (downstream) and Manta G-125 (upstream) CCD cameras equipped with IR-filters. Laser focusing and sample observation use the same

optical path. We installed GeoHEAT 40_NIR objective (AdlOptica GmbH) that provides a working distance of 80 mm at a focal length of 40.8 mm.¹⁴ A long working distance is necessary to keep optics away from the primary beam, yet a short focal length provides higher magnification, which in this case is 20 $\times$ for the system in total.

The SPI fiber lasers used for sample heating are capable to operate in continuous and modulated modes with a maximum modulation frequency of 100 kHz. Each fiber laser is equipped with a pilot laser, which emits light in the range of 630–680 nm and may be used for prealignment. Electrically tunable lenses (OptotuneTM) are used for focusing of the laser spots; they are installed directly on the end of the laser fibers. Laser fibers are mounted on the UniHeads with lightweight aluminum holders designed and

FIG. 3. Schematic diagram of the optical components of the dsLH system (based on the system described in Ref. 12). The UniHeads are aligned mutually perpendicular.

constructed specifically to reduce vibrations of the laser beam during ω -rotation.

The scXRD/dsLH setup contains all the components required for data collection and laser heating in one module. It can be mounted/unmounted on the General Purpose experiment at P02.2 (Fig. 1) within a few hours.

III. SETUP OPERATION

A. Preparation of the beamline

In comparison with the standard sample stack at the General Purpose experiment, the scXRD/dsLH setup demands additional space between the sample and the lead shielding (which encapsulates X-ray optics) to allow for its rotation (Fig. 1). This requires a longer X-ray focusing distance. The focusing distance of 1477 mm is achieved by using 91 Compound Refractive Lenses (CRLs) at an X-ray energy of approximately 42.7 keV (Fig. 1). The position of the X-ray focus is modified by moving either the CRLs or the heavy-load Huber motorized X-stage along the beam direction. A standard tungsten round edge is used to estimate the focus size (typically $\sim 5 \times 5 \mu\text{m}^2$). A thin tungsten cross hair is used to calibrate the beam position and bring the rotation ω -axis to the beam by using the heavy-load Huber motorized Y- and Z-stages.

The sample-to-detector distance, coordinates of the beam center, and tilt angle and tilt plane rotation angle of the detector images are calibrated using CeO_2 standard powder. In addition, a single

crystal of orthoenstatite $[(\text{Mg}_{1.93}, \text{Fe}_{0.06})(\text{Si}_{1.93}, \text{Al}_{0.06})\text{O}_6]$, *Pbca*, $a = 8.8117(2)$, $b = 5.18320(10)$, and $c = 18.2391(3)$ Å] is used to calibrate the instrument model of the CrysAlisPro[®] software (sample-to-detector distance, the detector's origin, offsets of the goniometer angles, and rotation of the X-ray beam and the detector around the instrument axis).

B. Alignment of the lasers and temperature measurements

The initial alignment of the lasers is accomplished via a piece of platinum foil by centering both guiding lasers on the observation area using adjusting screws on the UniHeads. The final adjustments are made using the Pt thermal glow appearing when a sufficient near-infrared radiation (NIR) laser power is achieved.

Thermal radiation from both sides of the sample is collected using two optical fibers with a core diameter of $400 \mu\text{m}$, which are connected to the Ocean Optics QE65000 spectrometers. Each optical fiber is aligned to the heating spot and focused on the image plane. To do so, the end of the optical fiber leading to the spectrometer is connected to a strong visible light source which illuminates the Pt foil. The position of the light spot on the foil is then adjusted by moving the screws which mount the optical fiber to the UniHead. In this way, due to the magnification of the system, the sample temperature is probed from an area of $20 \mu\text{m}$ diameter, centered at the heated spot.

The thermal radiation spectra are analyzed using spectro-radiometry¹⁵ and fitted using the gray body approximation of

Planck's law in the wavelength range of 650–800 nm with software developed for this purpose. Temperatures are calibrated with tungsten halogen lamp (OPTEMA Engineering GmbH, OL-245M-K3) placed on the sample stage and precalibrated at 2200, 2500, and 2900 K.

For temperature measurements during the ω rotation of the sample, thermal spectra are collected every second with exposure time of a few hundred milliseconds. The stage rotation and the temperature data collection of the detector are synchronized manually. The setup allows maintaining the laser heated sample under stable temperature during the entire data collection with a temporal variation that is well below the typical temperature measurement uncertainty of ± 100 K (Fig. 4).

C. Installation of a DAC

After a DAC is placed in the holder, the sample alignment is accomplished in three steps. In the first step, the upstream and downstream lasers are aligned to the center of the gasket. Using a video camera and the motorized X-, Y-, and Z-stages right below the DAC, the sample is first positioned from the downstream v-UniHead observation side. Then, the upstream h-UniHead is moved into position using the motorized XY- and Z-stages below it. In the second step, using the motorized XY-stage, the heavy-load Z-stage and the ω -2 θ goniometer below the dsLH system, the DAC together with the aligned lasers is moved into the X-ray beam as well as to the center of the rotation axis using the standard procedure of the sample centering by X-ray absorption.¹⁶ Finally, the exact point of interest on the sample can be chosen by moving the X-, Y-, and Z-stages below the DAC while the sample is observed through the video camera. Currently, the motors allow the DAC movement by

± 25 μm with an accuracy of 0.003 μm in horizontal (X and Y) directions, while in the vertical (Z) direction, it is possible to move by ± 15 μm with an accuracy of 0.003 μm .

The scXRD/dsLH setup provides a possibility to work with various types of DACs. However, the appropriate thickness of a DAC (including a membrane cup, if used) is limited to 50 mm to enable correct positioning of the mirrors.

D. Data collection

The scXRD data collection is realized in an *oscillation* (=narrow slicing) mode, i.e., when the diffraction is recorded while rotating the sample about a single ω -axis in small steps of 0.5° – 1° . During the data collection combined with the laser heating, it is possible to collect the frames in ranges of ω from -38° to $+25^\circ$. Diffraction data are measured using a Perkin Elmer XRD1621 flat panel detector in a shutterless mode that reduces the data collection time to 1 or 2 min for the entire ω rotation range. Platinum-foil (Pt-foil) absorbers of the beamline may be used to decrease the intensity of the primary beam in order to avoid oversaturated diffraction peaks on the detector.

E. Known problems and limitations

At the moment, the scXRD/dsLH setup for X-ray diffraction experiments provides an even power distribution within the laser beam with a diameter of 10–30 μm , since the size of the homogeneously heated area should be significantly larger than the diameter of the X-ray beam of $\sim 5 \times 5$ μm^2 . Rotation of a DAC results in vibration of the sample around the aligned position with a magnitude of about 1–2 μm during the entire data collection (from -38° to $+25^\circ$). In experiments up to about 100 GPa, in which the size of a sample is of about 10 μm , such vibrations are acceptable, as the sample still remains irradiated. However, in experiments above 100 GPa, the size of a sample drastically decreases and the size of the laser beam should be reduced down to 1–10 μm . This might require significant modifications of optical schemes and components as well as mechanics of the system to improve the stability further.

IV. EXAMPLES OF THE SETUP APPLICATION

We have conducted a number of experiments to demonstrate capabilities of the scXRD/dsLH setup for *in situ* studies of materials, during which high pressures and high temperatures are applied simultaneously. Two examples are presented below.

A. High-pressure synthesis and crystal structure of FeN

Iron nitrides were discovered in the mid-19th century and have been studied intensively since then due to their refractory properties and potential applications as magnetic recording materials. In addition, due to the abundance of nitrogen deeper in Earth's regions, assuming iron nitrides as components of the Earth's core can lead to an improved description of the core's elastic properties.^{17,18}

Until recently, the iron-nitrogen system was only known for its iron-rich phases α'' -Fe₁₆N₂, α' -Fe₈N, γ' -Fe₄N, ϵ -Fe₃N_{1-x}, β -Fe₇N₃, and ζ -Fe₂N.¹⁹ The application of pressure leads to the formation of nitrogen-rich compounds with potentially high hardness and high

FIG. 4. Example of time-dependence of the temperature during laser heating and simultaneous scXRD data collection (step-scan) recorded from both sides of the heated sample of CrN; green dots designate the upstream side temperature and red dots designate the downstream side temperature. Thermal radiation is collected every second with an exposure time of 800 ms. Solid lines connecting the dots are guides for the eye. Dashed lines show the mean temperature values for each side over the entire rotation. Shaded areas represent the margins of one standard deviation.

energy density.^{20–24} In particular, very recently, Fe₃N₂, FeN, FeN₂, and FeN₄ were synthesized due to high-pressure high-temperature reactions between iron and nitrogen.^{25–27}

In this study, we have synthesized NiAs-type FeN^{25,26} and characterized its crystal structure at high-pressure and high-temperature conditions using the scXRD/dsLH setup described above. Three pieces of Fe were placed into a BX90 DAC²⁸ equipped with 250- μ m diamond anvils. The cell was then loaded with N₂, serving as both a pressure-transmitting medium and reaction agent. A small ruby ball was placed in the pressure chamber for pressure determination. The cell was pressurized to about 50 GPa. Laser heating at 1900(200) K provoked a chemical reaction between iron and nitrogen resulting in the synthesis of iron nitride FeN as single crystals.

The scXRD data were collected both during and after laser heating. In each experiment, we managed to collect 36 unique reflections with intensities $I > 3\sigma(I)$, which enabled the refinement of anisotropic displacement parameters. As expected, the unit cell parameters obtained during and after laser heating [$V = 29.853(7)$ and $28.86(4)$ Å³, respectively] differ due to the thermal expansion of the crystal. The thermal parameters acquired during laser heating are about twice as large as those at room temperature (Table I and Fig. 5). The difference in the quality of the structural refinements for the room- ($R_1 = 0.0594/wR_2 = 0.0692$) and high-temperature data ($R_1 = 0.0772/wR_2 = 0.0793$) is insignificant, proving the technical feasibility of scXRD for *in situ* studies of materials at high pressures and simultaneous high temperatures in laser-heated DACs.

B. High-pressure synthesis and crystal structure of CrN

Chromium nitrides are an attractive class of materials with a number of excellent physical and chemical properties. For example, chromium nitride CrN with a cubic rock salt structure plays a

FIG. 5. Crystal structure of FeN at 293 K (a) and 1900 K (b). Orange and blue balls show the positions of Fe and N atoms, respectively. Thermal ellipsoids are shown at the 90% probability level.

fundamental role in the steel hardening process²⁹ and is often used as a component in advanced coating systems.³⁰

Theoretically predicted nitrogen-rich chromium nitrides CrN₂ and CrN₄ were suggested to become thermodynamically stable at pressures above 7 and 15 GPa, respectively,^{31,32} and are expected to be superhard compounds.^{31,32} The high-pressure reaction between chromium and nitrogen, studied by Hasegawa and Yagi at 10 GPa and 1800 K in a laser-heated DAC, yielded CrN as the only product.³³ Here, we studied the same reaction but at a pressure of 47 GPa in order to search for predicted nitrogen-rich chromium nitrides.^{31,32}

A piece of Cr was loaded into a BX90 DAC equipped with 250 μ m Boehler-Almax type diamond anvils. The cell was loaded with N₂ as pressure-transmitting medium and reaction agent.

TABLE I. Details of crystal structural refinements of FeN and CrN.

	FeN	FeN	CrN	CrN
Pressure (GPa)	49.6	49.6	47	47
Temperature (K)	293	1900(200)	293	1300(200)
Symmetry	$P6_3/mmc$	$P6_3/mmc$	$Fm\bar{3}m$	$Fm\bar{3}m$
Lattice parameters				
$a = b$ (Å)	2.6299(11)	2.6689(4)	3.960(6)	3.9819(5)
c (Å)	4.819(7)	4.8394(6)	3.960(6)	3.9819(5)
V (Å ³)	28.86(4)	29.853(7)	62.10(15)	63.135(14)
Z	2	2	4	4
Refinement				
No. of measured, independent, and observed reflections	154/40/36	112/47/36	67/20/19	59/18/16
R_1/wR_2	0.0594/0.0692	0.0772/0.0840	0.0886/0.0870	0.0705/0.1061
U_{iso} (metal) (Å ²)	0.0116(10)	0.0247(16)	0.0081(15)	0.036(7)
U_{iso} (N) (Å ²)	0.014(4)	0.026(4)	0.004(3)	0.030(6)

FIG. 6. The crystal structure of CrN at 293 K (a) and 1300 K (b). Blue and gray balls show the positions of Cr and N atoms, respectively. Thermal ellipsoids are shown at the 90% probability level.

FIG. 7. Time-dependence of the temperature during laser heating and simultaneous scXRD data collection for CrN. Green dots designate the upstream side temperature, and red dots designate the downstream side temperature. Solid lines connecting the dots are guides for the eye. Dashed lines show the mean temperature values for each side over the entire rotation. Shaded areas represent the margins of one standard deviation.

Laser-heating of Cr in nitrogen to 1300 K at 47 GPa resulted in the formation of CrN, the same phase, which was observed in Ref. 33 (Table I). The predicted CrN₂ and CrN₄ phases were not detected.

The CrN crystal showed thermal expansion upon laser heating [the unit cell volumes $V = 63.135(14)$ and $62.10(15) \text{ \AA}^3$ were obtained during and after laser heating, respectively]. The thermal parameters acquired during laser heating are larger than those at ambient temperature (Table I and Fig. 6). As shown in Fig. 7, no significant variations in temperature were observed during the entire data collection.

V. CONCLUSIONS

We have developed the first dedicated single-crystal X-ray diffractometer coupled with double-sided laser heating system. The scXRD/dsLH setup encompasses all the components required

for scXRD data collection and simultaneous double-sided laser heating in a single module which can be mounted/unmounted on the General Purpose experiment of beamline P02.2 (ECB) at PETRA III within a few hours. The alignment of the optical components and preparation of the beamline takes approximately one working day. The setup has been tested in the course of several high-pressure high-temperature experiments aimed at studying chemical reactions between nitrogen and two metals, iron and chromium. It enabled to obtain reliable results in the structure solution and refinement of iron and chromium nitrides.

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