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Diamond anvils with a round table designed for high pressure experiments in DAC

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ABSTRACT

Here, we present new Diamond Anvils with a Round Table (DART-anvils) designed for applications in the diamond anvil cell (DAC) technique. The main features of the new DART-anvil design are a spherical shape of both the crown and the table of a diamond and the position of the centre of the culet exactly in the centre of the sphere. The performance of DART-anvils was tested in a number of high pressure high-temperature experiments at different synchrotron beamlines. These experiments demonstrated a number of advantages, which are unavailable with any of the hitherto known anvil designs. Use of DART-anvils enables to realise *in situ* single-crystal X-ray diffraction experiments with laser heating using stationary laser-heating setups; eliminating flat-plate design of conventional anvils, DART-anvils make the cell alignment easier; working as solid immersion lenses, they provide additional magnification of the sample in a DAC and improve the image resolution.

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Diamond anvil cell technique; high pressure generation; DART-anvil; single-crystal X-ray diffraction; laser heating

1. Introduction

Single-crystal diamonds were introduced as 'Bridgman anvils' in 1959 (Figure 1). Two major types of diamond anvil cells (DACs) – opposite plate and piston-cylinder – were designed, and their different modifications have been dominating in the scientific practice hitherto [1]. Subsequent decades of development turned a DAC into a universal high-pressure device providing the capability for a wide range of *in situ* measurements of properties of matter at pressure–temperature conditions corresponding to the entire Earth interior and beyond [2]. Already in the beginning of the 1970s, the DAC technique demonstrated broad opportunities for high-pressure research dealing with Mössbauer, infrared

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Authors contributions: L.D. and N.D. proposed the DART-anvil design, planned and led the research, and wrote the manuscript; E.K. made technical design, A.L. provided optical engineering expertise; M.B., E.B., E.K., G.A., A.P., V.P., E.G., K.G., and L.D. performed test experiments.

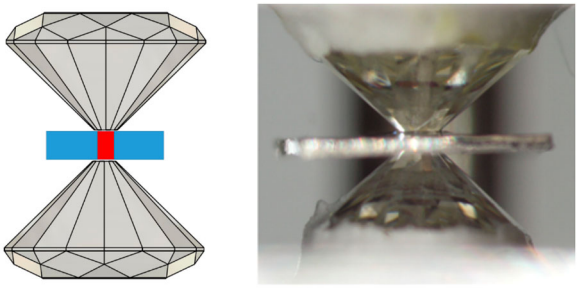

Figure 1. A schematic (a) and a microscope image (b) of an opposed diamond anvil assembly, the main part of a DAC. Two gem quality diamonds squeeze a sample (red) placed into a hole in a metallic gasket (blue).

and Raman spectroscopies, resistivity measurements, X-ray diffraction (XRD), and inelastic scattering.

Diamond anvils are obviously the most important components of a DAC, and with the development of new analytical techniques used to study materials at extreme conditions, anvil design evolved as well. Rapidly growing areas of DAC applications – single-crystal and powder X-ray diffraction, *in house* and synchrotron-based spectroscopic techniques (Brillouin spectroscopy, inelastic X-ray scattering, nuclear inelastic scattering, etc.) – require diamond anvils with a large optical aperture. Widely available on the market over decades are classical diamond anvils of 'standard Drukker' or 'modified Brilliant' cut (Figure 2(a)). They have, however, a number of limitations, especially in modern applications. For example, in order to achieve pressures in the range of 100 GPa, the diamonds should be rather thick (of about 2 mm or more), which leads to a significant absorption of X-rays (particularly those of relatively low energies, below 25 keV). The optical opening larger than 60° is difficult to achieve with such thick diamonds. A solution was found due to making crowns of diamond anvils conical [3,4] (Figure 2(b)), and later spherical [5] (Figure 2(c)). In these cases, anvils are not anymore glued on the surface of a flat supporting plate (Figure 1(b)), but instead, their crowns of a conical or spherical shape are fixed inside a conical (spherical) cavity of identical size drilled in the hard metal or tungsten carbide (WC) support. Such anvils are usually thin (of about 1.4–1.5 mm) and can provide up to about 90° optical opening (85° X-ray aperture) [5]. Note that the table of all currently used diamond anvil designs is

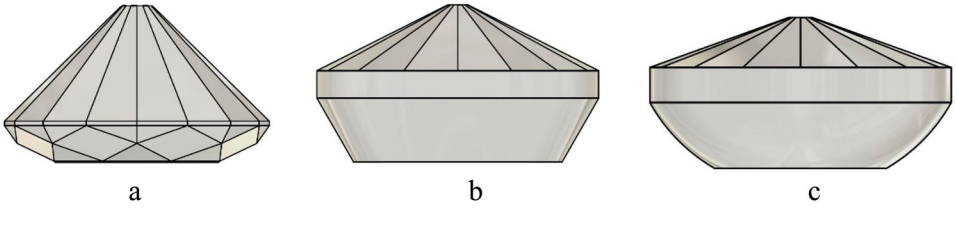

Figure 2. Examples of diamond anvils of different designs: (a) modified Brilliant, (b) Boehler Almax, and (c) diamond anvil with a spherical support.

flat. In [6] it was suggested that truncated sapphire balls could serve as anvils in a high-pressure cell and as focusing optical elements.

Most of the novel and quickly developing methods of *in situ* investigations of pressurised materials (single-crystal X-ray diffraction, inelastic X-ray scattering, Brillouin spectroscopy, etc.) require moving DACs with respect to the beam of electromagnetic radiation (e.g. X-ray and laser), which is used for heating, exciting, or probing materials' properties at extreme pressure and variable temperature conditions. Conventional anvils have 'flat-plate' geometry, and rotation of a cell at any angle with respect to the stationary beam leads to strong refraction, as diamond is a material with a high refractive index ($n = 2.425$ at 532 nm). This destroys the alignment of the sample with respect to the optical beam. Special procedures (sometimes quite complicated and time and labour consuming) are required to maintain the alignment with respect to X-rays and optical systems, but still certain types of experiments, which involve DAC rotations, cannot be performed while keeping lasers or optical components stationary. For example, immobile laser heating of a sample in a DAC with conventional (flat-table) anvils during single-crystal X-ray diffraction data collection is impossible, as the sample is getting out of the focus of the laser beam upon rotation of the cell.

In this work, we present a new design of diamond anvils, the Diamond Anvil with a Round Table (DART), which enables to eliminate a number of problems associated with the use of conventional anvils in DACs. The major feature of the new DART-anvil design is a spherical shape of both the crown and the table of a diamond with the centre of the culet located exactly in the centre of the sphere.

2. Diamond anvil with a round table (DART-anvil)

To realise the new DART-anvil design, one should polish a diamond as shown in Figure 3. The crown and the table are not distinguished anymore, and the anvil has a spherical surface below the girdle circle. The lower girdle circle (as shown in Figure 3(a)) is the latitude of the sphere, whose centre is at the culet of the diamond (Figure 3). To distinguish

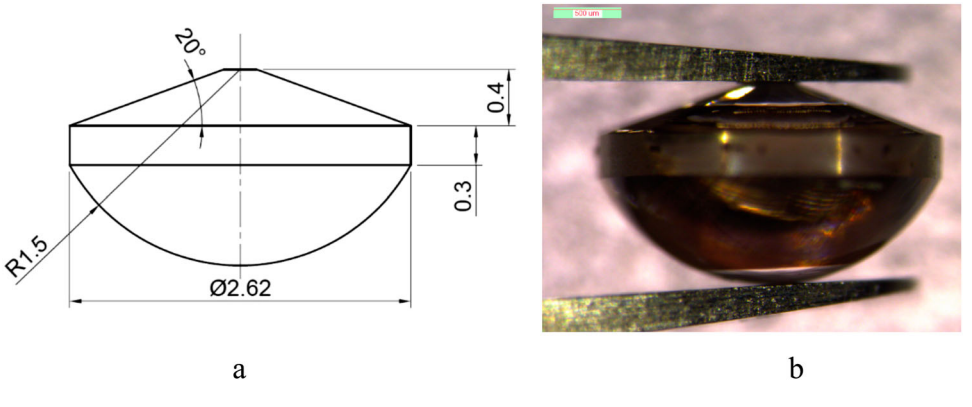

Figure 3. A schematic (a) and a microscope image (b) of the DART-anvil. (Dimensions may be scaled/adjusted in accordance with experimental needs.)

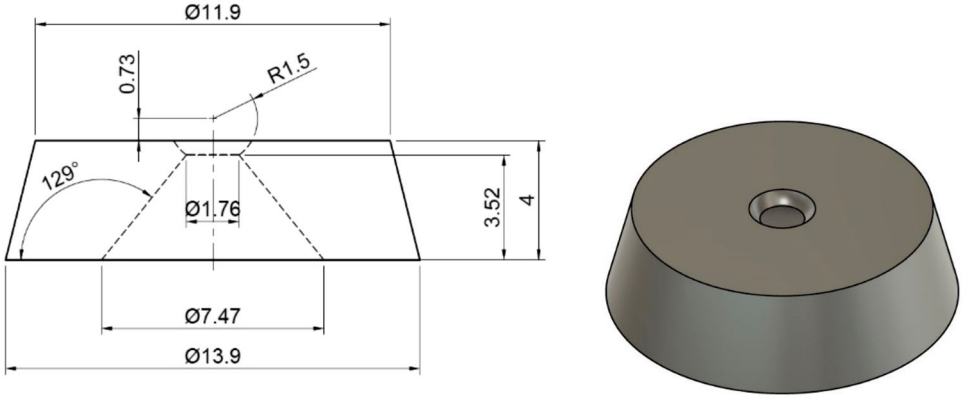

Figure 4. A schematic of a seat for the DART-anvil and its three-dimensional image.

the design of our anvil from previously known ones, we call it a 'DART' to underline that the table has a convex shape.

The DART-anvils, to be mounted in a DAC, require special seats. A schematic of a seat for the DART-anvil, suitable for the BX90-type cells [7], and its three-dimensional image are shown in Figure 4.

DART-anvils provide a number of advantages, which are unavailable with any of the hitherto known types of anvils. First, they enable ~2.4 times enlargement of the image of a sample in a DAC. Indeed, according to Abbe invariant [8],

$$n\, y \sin(\omega) = n'\, y' \sin(\omega'),$$

where n and n' are the refractive indexes of media, y is the object size, y' is the image size, ω and ω' are the slope angles, i.e. angles between a ray and the optical axis. In case of a DART-anvil, which effectively acts as a lens, $n = \sim 2.4$ (diamond), $n' = 1$ (air), $\omega = \omega'$ since the centre of the spherical surface coincides with the object plane. Consequently, the magnification is $y'/y = n/n' = \sim 2.4$. Thus, a DART-anvil acts as a solid immersion lens [8,9] and significantly magnifies the image of a sample in the pressure chamber. It also enhances the physical resolution of the imaging which is proportional to the numerical aperture, NA $= n \sin(\omega)$, of the observation channel [8] being essentially increased due to high refractive index of the lens material (diamond).

Imaging from the centre of a spherical lens is also characterised by a remarkable feature from the point of view of aberrations – for the practically used NA the image is free of not only spherical aberration, but also from coma and astigmatism in a zone around the surface centre. This simplifies drastically the alignment procedure and provides high-quality imaging even in case of certain misalignments or de-focusing. In contrast to diamond anvils of known design, which have two parallel optical surfaces and thus the image of an object observed through the anvils does not coincide with the objects' physical position along the optical axis, the DART is free of that problem. As a result, the optical and X-ray alignment of the DACs equipped with DART-anvils (e.g. with respect to the goniometer axes) becomes much more simple and accurate; no refraction correction is needed once the sample is aligned to the optical focal point, thus simplifying the procedure of

aligning to the X-ray focus. Providing this advantage in combination with the higher magnification of the objects confined in the pressure chamber and enhanced physical resolution, DART-anvils have a great potential for applications in different spectroscopies, including Raman, IR, and Brillouin.

The major effect that DART-anvils promise is applications requiring laser heating. In particular, DART-anvils allow performing *in situ* high-pressure high-temperature (HPHT) single-crystal XRD experiments in DACs using *stationary lasers*. With the design we propose, a laser beam remains focused on the sample even if the cell is rotating: due to the spherical shape of the anvil, the direction of the incoming laser beam is maintained perpendicular to the anvil's surface, while the DAC is rotated during the XRD data collection (Figure 5).

3. Examples of applications of DART-anvils

We have conducted a number of HPHT experiments with DACs equipped with DART-anvils aimed at demonstrating their possible applications and advantages. The XRD experiments we describe below were performed using existing laser-heating setups. In all of the experiments Re gaskets with the initial thickness of 200 μm were used. The gaskets were indented to the thickness of 20–25 μm. Gases (He, Ne, or N_2) were loaded at a pressure of 1.3–1.4 kbar [10]. In all cases, the culet size of DART-anvils was 250 μm.

3.1. Performance of DART-anvils on compression at ambient temperature

To check the stability of DART-anvils, we assembled a DAC with one DART-anvil and one Bohler-Almax-type anvil (250 μm culet, 4.0 mm outer diameter). In this cell, a powder of iron was compressed up to ~70 GPa at ambient temperature in a He pressure-transmitting medium. After decompression, we opened the cell and inspected the anvils under a microscope. Both anvils remained intact; no problems in the course of compression or decompression appeared.

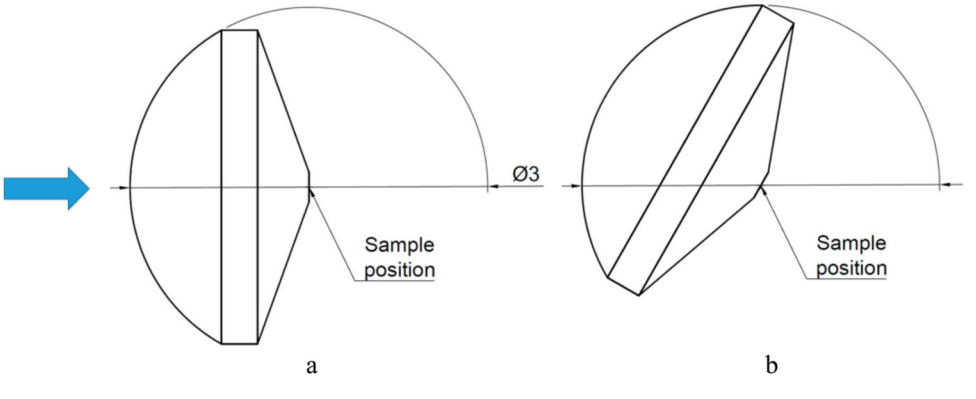

Figure 5. A schematic view of a DART-anvil aligned with respect to the incoming beam before rotation (a) and after rotation (b). The arrow shows the direction of optical observation and/or the direction of a laser beam. The sample remains in focus upon rotation.

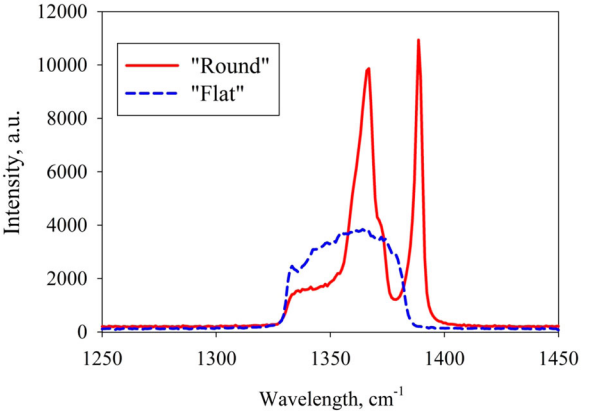

Figure 6. Examples of Raman spectra collected from the diamond-to-sample interface of the DART- (red continuous line) and Bohler-Almax- (blue dashed line) anvils at 24(1) GPa.

In this experiment, pressure was measured not only using ruby fluorescence, but also on a Raman signal from the centres of the culets of both of the diamond anvils, employing the existing correlation between the high-frequency edge of the Raman band with the normal stress at the culet face [11]. For this purpose, a LabRam spectrometer (He–Ne laser, x50 long working objective, 200 µm confocal hole) was used. Interestingly, the signals from the DART and the standard 'flat' Bohler-Almax anvils have a different shape (Figure 6). The position of a sharp peak observed in the spectrum from the DART-anvil indeed corresponds to the most stressed part of the anvil, so that the pressure estimation on a Raman signal from the centre of the culet [11] appears to be more accurate for DART-anvils when compared to conventional flat ones.

3.2. Single-crystal XRD data collection during laser heating at 13-IDD at GSECARS (APS, USA)

The experiment described below was performed at 13-IDD (GSECARS) at the APS. We used the flat-top laser-heating setup standard for this beamline [12]. X-ray beam with a wavelength of ~0.295 Å was focused to about 2×3 µm^2 FWHM. Data were collected using a Pilatus 1 M CdTe detector (DECTRIS). A mini-BX90 cell (30 mm outer diameter) [7] was equipped with two DART-anvils. This cell was mounted in a standard water-cooled holder, which was not yet optimised for the actual laser optical system and allowed rotation in the range of −4° to 20° during laser heating (while opening of the cell was 68°). The three-layer sample consisted of an iron foil (about 3 µm thick and 50 µm in diameter) compressed between two layers of powdered bridgmanite $(Mg_{0.88}Fe_{0.12})(Si_{0.96}Al_{0.04-})O_3$ pre-synthesised in a large volume press. This 'sandwiched' sample was loaded into a hole of 125 µm in diameter and 20 µm thick made in a Re gasket. Pressure was measured using the thermal equation of state of iron [13].

The cell was aligned with respect to the rotational (ω) axis using an X-ray beam and applying the standard for the 13-IDD beamline procedure. After the alignment, the cell was rotated about the ω-axis, while the sample was observed using the optics of the laser-heating system; no visible changes in the position of the sample were detected

upon rotation (Figure 7). Upon laser heating, no shift in the position of the heated spot was observed (Figure 8), and the temperature, measured spectroradiometrically, was reasonably stable (for example, in one run it was maintained within 1600–1850 K, and in another run, within 2200–2400 K).

Several crystallites of bridgmanite formed already after the first annealing of the sample at temperatures of 1300–1700 K. Unfortunately, the X-ray diffraction from individual crystallites was rather weak, and, in combination with a relatively small rotational angle (24°), it was not easy to collect data sets suitable for single-crystal structure refinement. Still, after collecting ω-step scans (0.25° step with 0.25 s/step), we were able to find a single-crystal domain, which gave about 90 unique reflections (about 50 of them with $F_o > 4\sigma(F_o)$). Integration in *CrysAlis*^{Pro}© gave $R_{int} \sim 9$ %, and the structure was refined using SHELXL© [14] with $R_1 \sim 10$ % (14 parameters to refine; Table 1). It is remarkable that the quality of the diffraction data collected during heating (1700(150) K) was practically similar to that collected at ambient temperature (Table 1).

3.3. Single-crystal XRD data collection during laser heating at extreme conditions of beam line (ECB P02.2) at PETRA III (DESY, Germany)

A small (about 10 μm in diameter and 5 μm thick) piece of comercially available polycrystalline chromium (Sigma Aldrich Inc.) was placed inside a hole drilled in a Re gasket. A

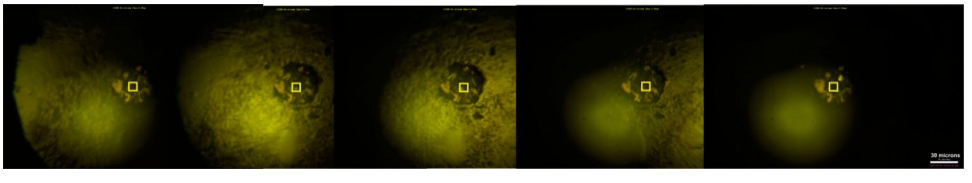

Figure 7. A series of photographs of a sample in a DAC (an iron foil in silicate perovskite at ~35 GPa) taken with a five-degree interval upon rotation of the DAC about the ω-axis. Beforehand the cell was aligned with respect to the rotational centre of the goniometer at 13-IDD beamline at the APS (USA). The position of the X-ray beam is marked by the yellow square. No visible changes in the position of the sample were detected upon rotation. The scale is 30 μm.

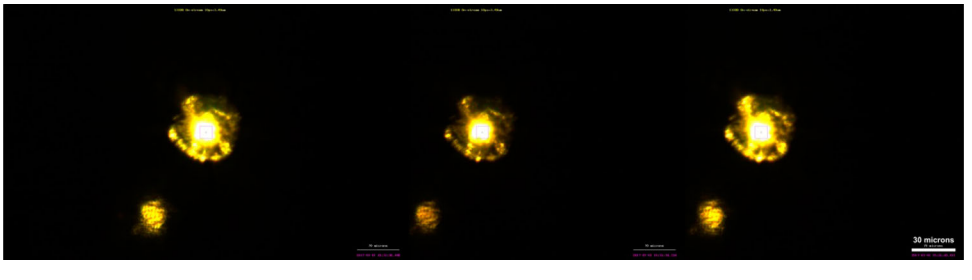

Figure 8. A series of photographs of a sample in a DAC (an iron foil in silicate perovskite at ~35 GPa) taken with an eight-degree interval upon rotation of the DAC about the ω-axis and simultaneous laser heating at 2300(150) K using a stationary laser. Beforehand the cell was aligned with respect to the rotational centre of the goniometer at 13-IDD beamline at the APS (USA). The position of the X-ray beam is marked by the square. No shift in the position of the heated spot with respect to the sample was detected upon rotation. The scale is 30 μm.

Table 1. Results of the single-crystal structure refinement of bridgmanite $(Mg_{0.88}Fe_{0.12})(Si_{0.96}, Al_{0.04})O_3$ (silicate perovskite ABO_3, where $A = (Mg_{0.88}Fe_{0.12})$ and $B = (Si_{0.96}Al_{0.04})$; space group P*bnm*, #62) at different pressures and temperatures.

			Lattice parameters				
P, GPa	*T*, K	$R_{int}/R_1/R_1$all, %	*a*, Å	*b*, Å	*c*, Å	*V*, Å^3/unit cell	Atomic parameters (*xyz*U)
35(1)	298	8.6/9.6/12.7	4.578(8)	4.764(4)	6.692(9)	145.95(37)	A −0.026(4) 0.4381(14) 0.25 0.039(3) B 0.0 0 0.0 0.041(2) O1 0.105(5) 0.028(2) 0.25 0.026(3) O2 0.205(8) 0.306(2) −0.057(2) 0.049(4)
43(1)	1700(150)	9.6/8.2/9.4	4.606(9)	4.759(7)	6.708(13)	147.0(5)	A −0.024(2) 0.442(1) 0.25 0.024(2) B 0.0 0 0.0 0.019(1) O1 0.105(5) 0.040(2) 0.25 0.015(2) O2 0.174(4) 0.299(1) −0.046(1) 0.017(2)

mini-BX90 DAC equipped with DART-anvils was used. The sample chamber was loaded with nitrogen, which served as both a pressure-transmitting medium and a possible reactant. A ruby sphere was also placed into the chamber as a pressure gauge. The sample was compressed to the desired pressure of 38(1) GPa.

Figure 9 shows the sample in the pressure chamber after compression. The DART-anvil with its spherical surface acts as a solid immersion lens and all visible objects are magnified. The microscope scale of 50 µm corresponds indeed to the value of ~21 µm (Figure 9) due to additional magnification by x2.4 of the diamond spherical lens, which also improves optical resolution, so that individual grains (crystals) of solid nitrogen become visible (Figure 9).

Single-crystal X-ray diffraction experiments on this sample were performed at the ECB beamline (P02.2) at PETRA III (DESY, Hamburg) ($\lambda = 0.2904$ Å, Perkin Elmer XRD1621 flat panel detector) [15]. All operations at the beamline were performed 'as usual', without any specific modifications for the DART-anvils' implementation. X-ray diffraction images

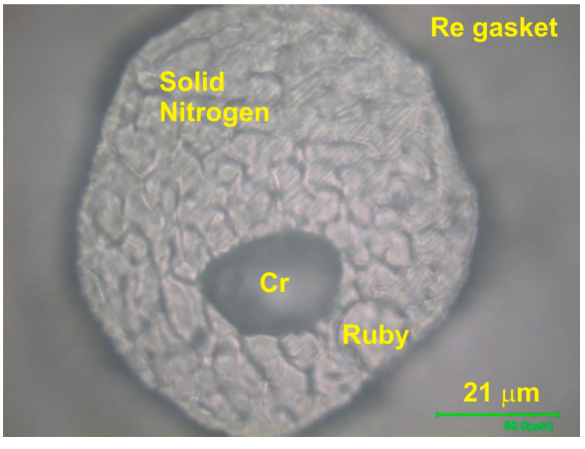

Figure 9. A sample of Cr compressed in N_2 medium to 38(1) GPa. Sizes of the ruby ball and the Cr particle were measured before compression and compared with 'apparent sizes' in the DAC. All objects in the pressure chamber are additionally magnified by ~2.4 times. The corrected scale (21 µm) is designated. Due to improved resolution, individual grains (crystals) of solid nitrogen (of a few microns in dimension) are clearly visible.

Table 2. Crystallographic data for Cr before ('cold') and during ('hot') laser heating at 38(1) GPa.

	'Cold'	'Hot'
Chemical formula	Cr	Cr
Pressure (GPa)	38(1)	
Temperature (K)	293	2350(150)
Space group	$Im\bar{3}m$	$Im\bar{3}m$
a (Å)	2.7622(3)	2.7672(2)
V (Å^3)	21.075(5)	21.190(3)
Calculated density (g/cm^3)	8.194	8.149
Observed reflections	54	56
Unique reflections	15	14
Unique reflections [$I > 2\sigma(I)$]	15	14
Parameters	2	2
R_{int}	18.06	19.70
R_1	0.0623	0.0409
wR_2	0.0756	0.0506
U_{iso} (Å^2)	0.0075(11)	0.0111(9)

were collected upon continuous rotation of the cell from −20° to + 20° about the ω-axis (wide scans) and with a narrow 0.5° scanning step in the range from −38° to + 38° ω (step scans). Due to the limitations implied by the mirrors of the laser-heating system, upon heating the cell was rotated in the angular ω range from −24° to + 35°. Data integration and semi-empirical absorption correction were performed using *CrysAlis^{Pro}*©. The crystal structures were solved and refined using the computer program JANA2006 [16].

Before, during, and after laser heating we could observe single-crystal domains of Cr. The crystal structure of Cr was refined against single-crystal X-ray diffraction data (Table 2). Cr maintains its *bcc* structure up to the temperature of 2800(100) K at 38(1) GPa. We could clearly observe the linear thermal expansion of chromium manifesting in the increase of the unit cell parameter by ca. 0.55% and enlargement of thermal ellipsoids (Table 2).

4. Conclusions

We have developed a new design of diamond anvils, 'diamond anvils with a round table' (DART-anvils), in assembly with a spherical support (anvil's seat). Their performance was tested in a number of HPHT experiments at different synchrotron beamlines. Use of DART-anvils provides a number of advantages, which are unavailable with any of the hitherto known anvils designs. In particular, they allow considerable (∼x2.4) enlargement of the image of a sample in a DAC and improve its resolution, as a DART-anvil works as a solid immersion lens. Use of the DART-anvils improves the precision of the pressure measurement in DACs on a Raman signal from the centre of the culet of a diamond anvil. The procedure of the sample alignment on the rotational stage is greatly simplified for the cells with DART-anvils. *In situ* single-crystal XRD experiments with laser heating can be realised with stationary laser-heating setups, i.e. the laser beam need not be moved simultaneously with the rotating sample. Due to the spherical shape of the DART-anvil surface, the angle of incidence of the incoming laser beam remains normal to this surface and focused on the sample, while the DAC is rotated during the XRD data collection.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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