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Online remote control systems for static and dynamic compression and decompression using diamond anvil cells

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The ability to remotely control pressure in diamond anvil cells (DACs) in accurate and consistent manner at room temperature, as well as at cryogenic and elevated temperatures, is crucial for effective and reliable operation of a high-pressure synchrotron facility such as High Pressure Collaborative Access Team (HPCAT). Over the last several years, a considerable effort has been made to develop instrumentation for remote and automated pressure control in DACs during synchrotron experiments. We have designed and implemented an array of modular pneumatic (double-diaphragm), mechanical (gearboxes), and piezoelectric devices and their combinations for controlling pressure and compression/decompression rate at various temperature conditions from 4 K in cryostats to several thousand Kelvin in laser-heated DACs. Because HPCAT is a user facility and diamond cells for user experiments are typically provided by users, our development effort has been focused on creating different loading mechanisms and frames for a variety of existing and commonly used diamond cells rather than designing specialized or dedicated diamond cells with various drives. In this paper, we review the available instrumentation for remote static and dynamic pressure control in DACs and show some examples of their applications to high pressure research. © *2015 AIP Publishing LLC.* [http://dx.doi.org/10.1063/1.4926892]

I. INTRODUCTION

High Pressure Collaborative Access Team (HPCAT) is a dedicated synchrotron facility for high-pressure research in multidisciplinary fields. It has been highly instrumental in the development of third-generation synchrotron radiation technology for extreme conditions science for addressing fundamental questions in physics, chemistry, materials sciences, geosciences, and biosciences.[1] Since the beginning of its operation over a decade ago, HPCAT has developed and integrated an arsenal of novel X-ray diffraction and spectroscopic high pressure and high/low temperature synchrotron radiation techniques, as well as complementary optical and electromagnetic probes to advance cutting-edge high-pressure science and technology. For a user oriented facility such as HPCAT, the ability to accurately and consistently control sample pressure remotely at various temperatures is crucial for effective and reliable operation. Development of new experimental techniques, that will be presented here, further contribute to expanding the available pressure-temperature (P-T) range of experimental conditions, increasing the efficiency and productivity of the beamlines, and improving the quality of experimental data. Over the last several years, we have invested considerable effort into developing instrumentation for remote and automated static and dynamic *in situ* pressure control in diamond anvil cells (DACs). For HPCAT to remain a versatile facility, a variety of remote pressure control options need to be available to users. These capabilities make it possible for users to achieve their scientific goals and allow for development of next generation experimental capabilities.

Since the invention of the DAC,[2] various means of remote pressure control have been designed and implemented. These include both mechanical[3–8] and pneumatic[9–18] mechanisms for controlling pressure in specialized DACs used first for application in cryogenic environments. Later, pneumatically controlled DACs (membrane DACs or MDACs) gained popularity because of their versatility and usability at a wide range of P-T conditions[19–21] and became commercially available. Other pressure control devices, such as a motorized gearbox for Mao-Bell type DAC[22] or piezoelectrically driven DACs,[23,24] are of more limited use at specific P-T conditions.

Even though there exists an array of devices for remote pressure control in DACs, these devices are typically integrated with specific types of DACs for a restricted variety of experimental conditions and geometries. Because HPCAT is a user facility, our strategy is to keep a user friendly approach and concentrate our effort on developing universal pressurizing-depressurizing frames for common types of DACs rather than design specialized diamond cells. As a result, users are not required to bring DACs with integrated pressure control mechanisms, but rather bring DACs most suitable for their experimental needs, load them at their convenience with appropriate pressure medium, and use pressure control devices provided by HPCAT. Other design considerations for developing a collection of pressure control devices also include the following:

(a) simplicity and ease of manufacturing, with possibility of in-house modifications,

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(b) compatibility with temperature control devices-cryostats and whole cell heaters,
(c) reliable bidirectional pressure control,
(d) programmability and ability to synchronize DAC pressure control with data collection systems for semi-automatic data collection,
(e) ability to perform fast, accurate unidirectional (fast ramp compression and decompression) and cyclic pressure change during time-resolved experiments.

With aforementioned considerations in mind, a collection of devices for remote and automated pressure control in DACs has been developed at HPCAT over the last several years. These pressure control devices fall into three major categories: pneumatic (double-diaphragms), mechanical (gearboxes), and piezoelectric. Various modular drives can be combined into versatile yet user friendly assemblies for accurate control of sample pressure and compression/decompression rate. In this paper, we review the available pressure control devices and their combinations and show some examples of their applications to high pressure research. More examples can be found elsewhere in this volume.[25]

II. PNEUMATIC REMOTE PRESSURE CONTROL

The pneumatic means of remote pressure control in a DAC (bellows and diaphragms/membranes) remain most popular, especially at cryogenic conditions, because of their simplicity and efficiency. Typically, helium gas (which has lowest freezing temperature) inflates either a bellows,[11] a double-diaphragm,[18] or a single-sheet membrane[19] which produces axial displacement and applies force to the diamond anvils. The first pneumatic control devices in high-pressure experiments were used before diamond anvil cells became popular. For example, Ward[9] used phosphor bronze bellows to power piston-cylinder apparatus at 4.2 K and 15 kbars, while Endo[10] controlled pressure in a pressure vessel with Bridgman anvils to 10 GPa at liquid helium temperature. After the DAC gained popularity due to its small size, ease of operation, and ability to obtain high pressure, various modifications were combined with bellows pressurization mechanisms and widely used for different types of cryogenic experiments down to mK temperatures.[11–16] While mechanical systems (worm gear intensifier[3–5] or lever arm type DAC with either direct drive[6] or wedge mechanism[7,8]) can provide larger forces and potentially result in higher pressures, the massive mechanical linkages introduce significant heat leaks as well as thermal and mechanical disturbances during operation, thus requiring realignment of the sample after each pressure change. These disturbances are usually not encountered with pneumatic systems. The relative displacement of the diamonds necessary in DAC experiments is typically within several tens of micrometers, which led Daniels[18] to simplify bellows into a pair of thin disks welded together around all free edges to make an inflatable double-diaphragm structure. It is worth noting that even though bellows may require more gas for inflation, thus increasing heat impulse during pressurization, the efficiency of a bellows drive can be slightly higher than that of a double-diaphragm because the contact surfaces do not deform during pressurization, thus retaining maximum area. Because of this, bellows expanders are still used in specific cryogenic experiments.[17] Moreover, in devices with constrained environmental chambers (e.g., cryostats), it is possible to stack multiple bellows in such a way that the force adds without increasing the diameter of the apparatus.[26]

In 1988, LeToullec, Pinceaux, and Loubeyre[19] introduced a multipurpose MDAC which was not specifically designed for cryogenic work, although there are several reports of its use at cryogenic conditions.[20,21] Unlike bellows of double-diaphragm type, this is a single-sheet annular membrane of 0.2 mm thick welded with an electron beam or a laser to a rigid support. This type of membrane combined with various DACs is currently commercially available. Even though MDACs based on single-sheet membrane are popular due to commercial availability, the membrane efficiency is lower than that of double-diaphragm design.[18] In addition, double-diaphragms are much easier to manufacture, are significantly less expensive than a single-sheet rigid support design, and are proven to perform exceptionally well.[27–29]

Most commercial DACs with pneumatic drives are designed for specific purposes and special P-T conditions—either cryogenic, room temperature, or high temperature, and often are prohibitively expensive for general users. We concentrated our efforts on designing universal, user-friendly pneumatic pressure control systems which can be used with a wide variety of user-supplied DACs. An overview of the pneumatic pressure control systems designed and used at HPCAT is given in Sec. II.

A. Double-diaphragm design

The double-diaphragm design at HPCAT is similar to that of Daniels.[18] Unlike the European style,[19] where the membrane drive consists of a rigid frame with a single-sheet flexible membrane, the double-diaphragms (also commonly called membranes or double-membranes[30]) are made of two identical thin steel sheets, both of which plastically and elastically deform during expansion. This design makes the system more efficient and much less expensive to manufacture. HPCAT double-diaphragms are typically laser welded from 250 μm thick 304 type stainless steel. The gas supply capillary has a compound design that consists of a rigid 1/8 in. diameter stainless steel tube 0.15-0.25 in. long with an annealed 1/16 in. diameter, 5-6 in. long 304 stainless steel capillary brazed to it with silver-nickel brazing alloy (this limits the maximum service temperature of the double diaphragm to 1000 K; a rigid single-piece capillary can be used if higher temperatures are required). The typical and most popular HPCAT double-diaphragm has outside diameter (OD) of 2.0 in. and the inside diameter (ID) of 0.94 in. The ID of 0.94 in. was originally chosen to provide 60° (4Θ) symmetric opening for diffraction and other X-ray and optical experiments with "standard symmetric" DACs of Carnegie design.

In certain cases, the "standard" size is not satisfactory and double-diaphragms of different sizes can be made to accommodate specific experimental conditions. For example, double-diaphragms with a larger ID (1.2 in.) were made to provide full symmetric opening of 90° or more in BX90

DAC's[31] or "short symmetric" diamond cells. Also smaller membranes (e.g., 1.5 in. OD) made of thinner stainless steel sheets (200 μm thick) can be used anvils for more compact cells (e.g., mini BX90[31]). Note that these modified double-diaphragms typically have much smaller surface area than the standard design and therefore are less efficient and generate lower forces on the anvils. Alternatively, for cases where larger forces are required (e.g., in DACs for neutron diffraction), the double diaphragms with much larger diameter (i.e., 100 mm) can be used to provide loads exceeding 10 metric tons even with standard gas tank pressure.[30]

Typically, the double-diaphragms are very resilient and can inflate up to 2-4 mm (depending on the weld quality and anneal state of the diaphragm material) without failing, although the practical limit for re-usability is about 1-1.5 mm. Thus, individual double-diaphragms can serve many tens if not hundreds of cycles if used with care. The only instances of failure have occurred when one or both diamonds break during an experiment and the double-diaphragm catastrophically expands beyond its safe limit (if safety setscrews are not engaged), or if the double-diaphragm was not constrained properly during initial DAC setup and assembly.

B. Sample and diaphragm pressure

Double-diaphragms of this design, if constrained between rigid DAC parts, are capable of holding (and working) at gas pressures of several thousand PSI (several hundred atmospheres).[18] At HPCAT, we use commercially available high pressure helium tanks; therefore, the maximum pressure in a double-diaphragm is typically 2200 PSI (150 atm). The ideal pressure amplification is the ratio of the effective area of the double-diaphragm to the diamond culet size. Nevertheless, in reality, the pressure amplification factor is reduced by the double-diaphragm properties, preload, deformation of double-diaphragm material, friction in the DAC, diamond shape and side support outside the culet area, as well as other factors. Note that at non-ambient temperatures, the pressure amplification factor can be further reduced due to higher friction between the piston and cylinder caused by freezing liquid contaminants (water and grease) if special care was not taken during loading and assembly or due to oxidation/distortion of DAC parts in resistively heated DACs. Also, this factor will be reduced in the Boehler-type deflection DAC[32] where significant force is required to deform the DAC plates.

Our typical double-diaphragms with 2.0 in. (50.8 mm) OD and 0.94 in. (23.9 mm) ID have a total area of ~1580 mm^2 and effective area of 50%-80% (depending mostly on the degree of engagement, preload, and initial inflation/deformation). Thus, the typical force applied to the DAC piston can reach 12-20 kN (1.2-2.0 tons) at 2200 PSI (150 bars) gas pressure. When the pressure amplification factor is taken into account, this force is sufficient to produce sample pressure in excess of 100 GPa (1 Mbar) in a DAC with 300 μm culets. With smaller culets, it is possible to reach multi-megabar pressures (for example, 208 GPa with 100 μm diamond culets[25]), while larger diamond culets and sample volumes may require a larger double-diaphragm to reach maximum pressure (for example, to reach 94 GPa with 1 mm beveled culets in a neutron diffraction cell, 11 tons of load was produced by a 100 mm diameter double-diaphragm[30]).

C. Pressure control

1. Fine pressure controller

Over the last several years, we have designed and continuously improved upon a manual fine pressure control unit. Despite its active use over the last several years, it has several drawbacks which prevent it from being an optimal pressure control device. (1) The high pressure, high precision needle valves used to finely regulate pressure wear quickly and fail if not used properly, thus requiring costly maintenance. (2) It cannot be programmed or controlled remotely. (3) Due to the absence of active feedback, it is most effective only in static temperature conditions, as any change of temperature (in cryostat or at high temperature) on the double-diaphragm causes pressure drifts due to thermal pressure, and the gas pressure has to be constantly adjusted manually. (4) Poor stability for long experiments in the event of even the smallest leak in the system.

HPCAT recently acquired GE Druck PACE 5000 modular pressure controllers which solved the above mentioned maintenance and stability problems while adding the capability to make gas pressure a computer controlled process variable. For example, the controller maintains a constant gas pressure in double-diaphragms or single-sheet membranes for arbitrarily long periods of time, preventing pressure drifts due to small gas leaks and/or due to thermal contractions/expansion of gas during temperature change. The controller allows programmable on the fly data collection by either setting the gas pressure to follow a unique pressure-time path with external control signal, or simply by internally setting an appropriate pressure change rate (up to 3000 psi (200 bars) per s). Currently, each of HPCATs' four experimental stations is equipped with PACE5000 controllers. Generally, they are placed outside experimental stations and pressurized gas is supplied to experimental setups via a long 1/16 in. OD steel capillary. Typically, they are operated in manual mode via the controller's touch screen, although the controllers can be operated remotely using an internet connection and a simple graphical user interface. The controllers are compact (440 $\times$ 90 $\times$ 320 mm) and lightweight (10.1 kg) which allow for portability.

The PACE 5000 controller has a pressure range of 0-210 bars (0-3000 psi), an exceptional precision of better than 0.02% (including linearity, hysteresis, repeatability, and temperature effects for the gauge), and pressure stability of 0.003% full scale. Compact size, remote control availability, and a user friendly intuitive touch screen interface make it a versatile controller which can be used in any experimental station as well as in off-line instruments.

2. Fast release pressure control

Even though the PACE5000 controller is capable of very fast (up to 3000 psi/s) pressure increase rates, these

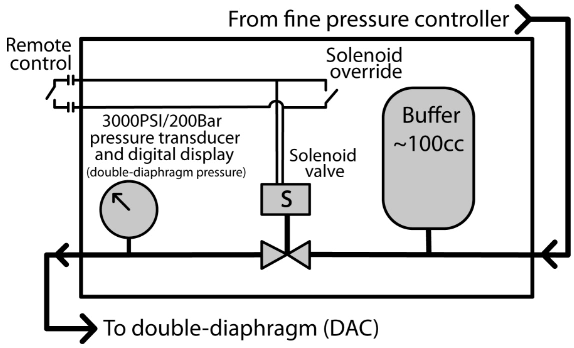

FIG. 1. Fast release attachment for instantaneous increase in membrane pressure.

rates are still not sufficient for some types of jump/ramp compression time-resolved experiments[33] or fast pressure release experiments.[25] To allow for faster pressure changes in a pneumatically controlled DAC, we have designed a fast pressure release attachment to the gas pressure controller. The fast release box (Fig. 1) is based on a gas buffer of ~100 cm^3 and a solenoid valve capable of handling 3000 psi. The gas buffer is filled to a desired pressure using a conventional pressure controller. The solenoid can be controlled either remotely (i.e., from outside the experimental station) or locally with an override switch. The fast release box is typically placed close to the DAC to decrease the double-diaphragm inflation time, which depends on a number of factors including length of capillary, differential pressure, inner diameter of the connecting capillary, and original deformation (zero pressure volume) of the double-diaphragm. The typical pressure increase rate in a DAC with attached double-diaphragm is on the order of megabars per second (i.e., 400 GPa/s reported by Velisavljevic *et al.*[33]). This fast release controller setup is also typically used in pressure quench experiments with typical decompression rates up to 6-10 Mbars/s.[25]

D. Membrane/double-diaphragm containment cans for DACs and double-diaphragms

At HPCAT, we developed a variety of containment cans for different DACs and various pressure/temperature conditions. The cans are either of cap-can design when the double-diaphragm diameter is greater than the DAC OD or plug-can design when double-diaphragm diameter is comparable with DAC OD.

1. Cap-can assembly

The most common way of pneumatically engaging the DAC is a double-diaphragm cap-can as shown in Fig. 2. The assembly consists of externally and internally threaded caps made of hardened 440C stainless steel. A 1 mm thick steel washer (pusher) with ID and OD corresponding to that of the double-diaphragm is typically inserted between the double-diaphragm and the DAC to prevent dimpling of the double-diaphragm plates over DAC holes. The typical confining cap-can assembly is designed for use with thin 2 in. diameter double-diaphragm and 1.875 in./48 mm symmetric type DACs up to 40 mm long. It provides 60° symmetric x-ray or optical opening on both ends. Four side access holes (0.5 in. diameter) can be used for 90° diffraction/scattering experiments as well as for accessory wiring (thermocouples, wires for resistivity measurements, etc.). In the case of short symmetric DACs, double-diaphragms with 2.0 in. OD and 1.2 in. ID can be used if a symmetric 90° opening is required.

The bottom of the externally threaded can has four holes on a 1.5 in. bolt circle so that pressure can also be adjusted using the DAC's pressurizing screws. Typically, this is done in cryogenic work when the load is transferred from the screws to double-diaphragm before the experiment thus preventing runaway pressure increase during cool-down.

Variations of this type of constraining can are also used with various types of non-symmetric cells. For example, Fig. 3 shows a compact panoramic DAC of HPCAT design with and without the double-diaphragm loading mechanism.

2. Plug-can assembly

Another modification of double-diaphragm constraining cans is based on a plug-can design. The standard type of this assembly ("universal can") can accept DACs of different designs up to 2.0 in. diameter, including various piston-cylinder type DACs, BX90 type DAC,[31] or plate-type DACs[32] and their modifications. In this case, the same plug-can assembly can be used to accommodate various DACs using simple adapter plates. Fig. 4 shows a plug-can assembly with BX90[31] and Boehler design plate DACs.[32]

This type of assembly typically has a relatively loose fit for the DAC. It can be viewed as an advantage in certain cases, such as whole cell mild resistive heating for temperatures below 450 K. Fig. 5 shows a symmetric DAC with a

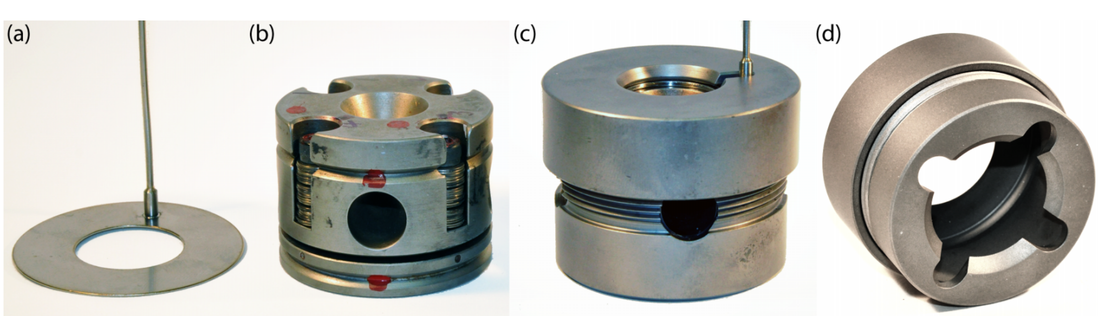

FIG. 2. Double-diaphragm (a), symmetric piston-cylinder type DAC of CIW design (b), cap-can assembly for symmetric DAC and membrane (c), and membrane cap-can for short symmetric DAC (d).

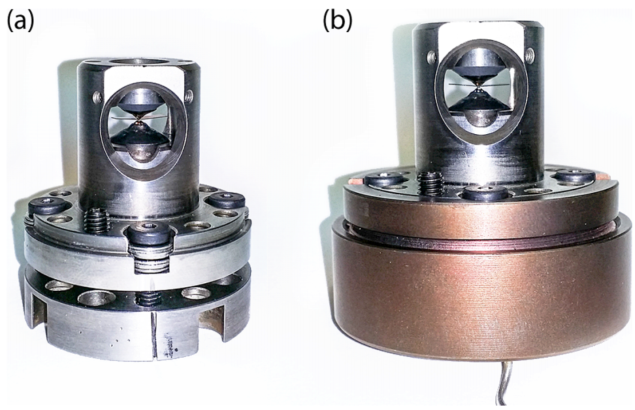

FIG. 3. Mini-panoramic DAC without (a) and with (b) double-diaphragm can attachment.

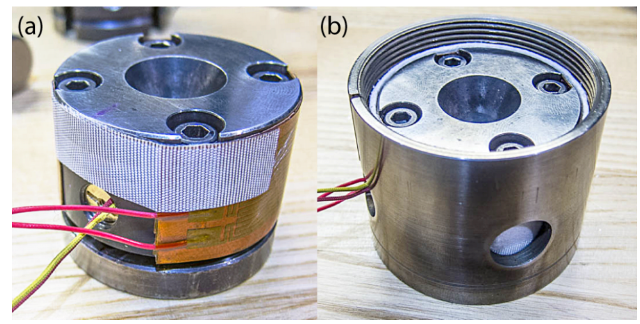

FIG. 5. Symmetric DAC with Kapton tape heater outside (a) and inside (b) plug-can pressurizing frame.

Kapton tape heater and thermocouple. The DAC is isolated from the can with a layer of mica (bottom) and a cylinder of Cotronics thermo-insulating ceramic paper. This assembly can be coupled with a second (decompression) double-diaphragm or a piezoelectric driver, as will be discussed in Secs. II H, II I, and IV B of the article.

E. Tie rod frames in cryostat

Can-type pressurizing frames are good for room-temperature, mild (<450 K) resistive heating, and laser heating experiments. However, due to poor thermal contact between the can body and the DAC, they are not effective for either cryogenic temperatures or whole cell external heating above 500 K. If whole cell conductive heating or cooling is required, we use a tie-rod pressurizing frame design similar to the first Paris-Edinburg cell design,[34] which is flexible and works effectively even with larger DAC assemblies.[30] In this design, the heating/cooling block is mechanically independent of the pressurizing frame. This makes it possible to make clamp-type conductive heaters/coolers from soft copper and the load-bearing frame from parts capable of handling the tensile forces generated by the double-diaphragm. Shown in Fig. 6 is a symmetric DAC in a cryostat DAC holder. A copper block clamping the DAC is cooled by direct flow of cryogen (helium or nitrogen) through the top part of the holder, which allows cooling of the DAC to 7-8 K even in a compact cryostat with a large (1.5-2.5 in.) optical/x-ray opening and without radiation shield. The pressurizing frame is loose with respect to the copper holder, but constrains the DAC—double-diaphragm assembly along the loading axis. The constraining plates are made of hardened steel (440C) to prevent bulging of the plate and breakage of the inner weld/seam of the double membrane.

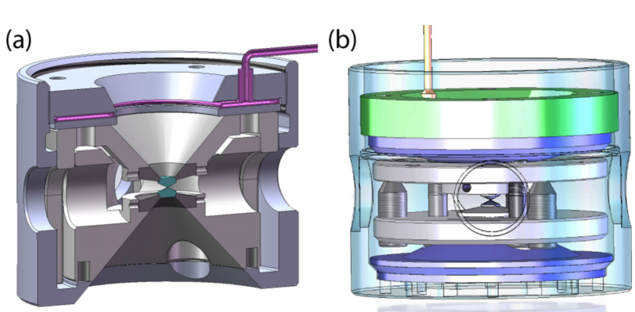

FIG. 4. Plug-can assembly with BX90 DAC (a) and plate DAC (b).

F. Tie rod frames with whole-cell heating

The same principle of independent clamps (thermal and pressurizing), as described in Sec. II E, can also be used for whole-cell resistive heating of a DAC. Typically, resistive heating in a DAC is performed either with a small single or double heater around the sample and diamonds (e.g., Refs. 27, 31, and 35) or in combination with a whole cell heater (e.g., Ref. 36). While using small resistive heaters has a number of advantages (i.e., higher sample temperature and faster temperature change), it also has a number of disadvantages such as large temperature gradients between cell body, heater, and anvils/sample, difficulty in placing the thermocouple near the sample in good contact with the diamond resulting in uncertainties in sample temperature, as well as time consuming DAC preparation. The whole-cell heater assembly allows easy and convenient high temperature high pressure measurements in common types of DACs without tedious preparation. A regular DAC can be clamped into a copper or a brass block which is heated by several standard cartridge heaters. Because the cell is heated uniformly, there is a very small thermal gradient in the DAC and a thermocouple can be placed on any part of the DAC.

The whole cell heater designed to operate in atmospheric conditions (without inert gas or vacuum shroud) can safely heat a DAC to 450-550 K, after which the DAC begins to oxidize and pressure control via double-diaphragm becomes very problematic due to increased piston-cylinder friction. To prevent oxidation of the DAC and retain its functionality at high temperature, the DACs are placed either in vacuum chambers[27,37] or into an inert atmosphere.[35] At HPCAT, we use a rectangular stainless steel vacuum shroud, a water-cooled aluminum base, and a copper heater block with cartridge heaters, as shown in Fig. 7. The vacuum approach has an advantage in that it helps to keep the vacuum shroud at relatively low temperatures. An aluminum radiation shield grounded to a water-cooled base further limits radiative heating of the outer vacuum shroud. The upstream Kapton or fused silica/sapphire window has a size of up to 1.5 in. and allows rotation of the DAC/chamber assembly by ±20° for single

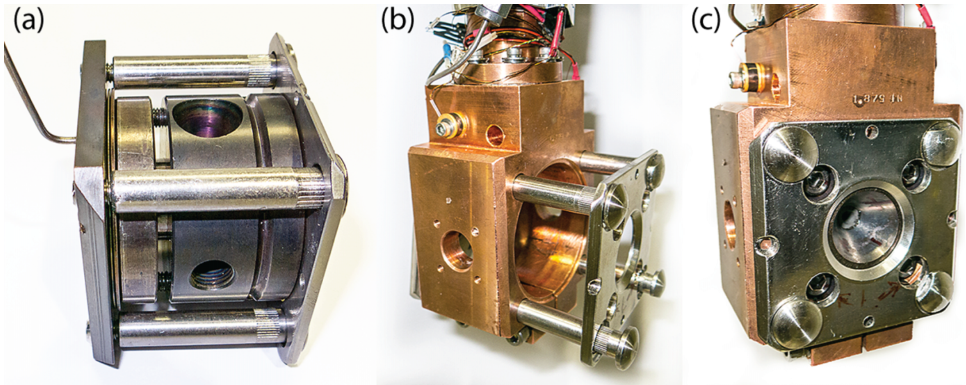

FIG. 6. Tie-rod frame with symmetric DAC (a) and cryostat DAC holder ((b) and (c)).

crystal diffraction measurements. The 2.5 in. downstream window is typically made of Kapton or polyester (in case pressure measurements via ruby fluorescence are required) and allows collection of diffraction (or other) signal in solid angle exceeding 60°. To date, this whole cell heating assembly has been used up to 900 K. It is also being used as a first-stage heater for preheating the DAC, while the sample is being heated by a compact heater close to the diamonds and the sample similar to Ref. 36.

G. Decompression double-diaphragm setup

While there are many ways of controlling pressure increase, decreasing pressure in DACs has always been problematic mainly because of two reasons: plastic deformation of the gasket and friction of the DAC between piston and cylinder. The pressure in the sample chamber of a gasketed DAC is increased due to compression of the pressure medium while the gasket is plastically deformed and elastically compressed. Typically, soft pressure media (i.e., condensed gases) are significantly more compressible than the gasket and in order to generate appreciable sample pressure, the gasket needs to be plastically deformed. Thus, when gas or liquid pressure medium is used, the only way to appreciably reduce the pressure (past elastic limit of the gasket) is to release some pressure medium from a sample chamber. This procedure is extremely difficult to perform in a controlled fashion. Decompression experiments are easier to perform with either solid pressure media or no pressure media at all. Still, controllable pressure decrease is problematic because of the friction between the DAC parts. This is especially problematic at cryogenic temperatures partially due to freezing of grease and possibly trapped moisture and air. This problem can be partially mitigated by adding passive spring elements, which would act against compression force, if decreases in the efficiency of the pneumatic drive (double-diaphragm or membrane) can be tolerated. Nevertheless, some experiments require the ability to actively and reliably control the magnitude and rate of decompression. This can be done by a second double-diaphragm applying opening force to the DAC.

Fig. 8 shows a symmetric DAC with a pneumatic decompression attachment which allows pressure decrease in a controllable fashion. The attachment consists of a low-profile, double-can setup (parts A and C) similar to that shown on Fig. 2. The other parts of the assembly are a double-diaphragm (F), pusher piston (G), two setscrews (B), and two pushing pins (D). The assembly is rigidly attached to the piston part of a DAC (E) with two screws (H). During the assembly, the position of the setscrew (B) is adjusted in such a way that when the pushing pins (D) touch the cylinder side of DAC, there is some (a few hundred micrometers) gap between the can (C) and the pusher piston (G). Thus, when the double-diaphragm (F) is inflated, it pushes the pusher piston (G) toward the DAC. Because the distance between the piston (G) and the cylinder part of the DAC is constrained through the pins (D),

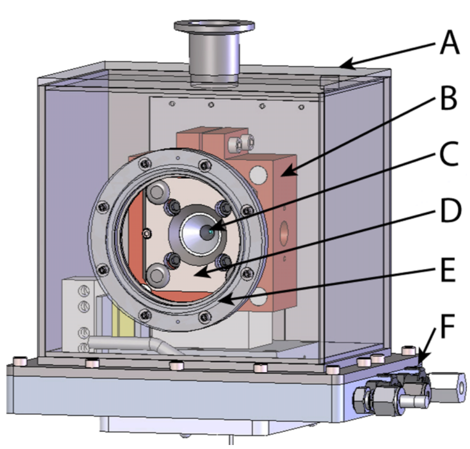

FIG. 7. Vacuum shroud assembly for resistive whole-cell heating. A–stainless steel shroud, B–copper block with cartridge heaters, C–DAC, D–tie-rod pressurizing frame, E–Kapton/polyester window, and F–aluminum base with feedthroughs.

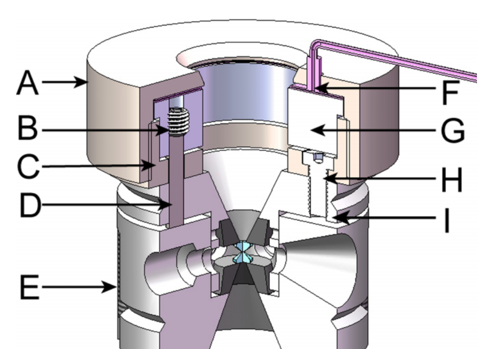

FIG. 8. Symmetric DAC with double-diaphragm decompression attachment.

inflating the membrane is actually pulling the piston side of the DAC away from its cylinder side, thus opening the DAC and decreasing sample pressure.

The setup shown in Fig. 8 is useful when controlled decompression, especially fast decompression, is essential. Initially, the DAC pressure can be increased with the standard pressurizing screws and then, the double-diaphragm can be inflated at a controlled decompression rate. The typical use of this configuration is for pressure quench experiments in synthesizing amorphous and crystalline metastable phases. For example, this particular decompression setup in combination with the fast release pressure control box (Fig. 1, Sec. II C 2 of this paper) was used to synthesize the amorphous form of silicon by pressure quenching the high-purity metallic silicon phase from 20 GPa to ambient pressure at a pressure decrease rate of ~0.6 – 1.0 TPa/s (more details are given in Ref. 25).

H. Dual ("push-pull") double-diaphragm setup

The decompression attachment can be easily combined with simple double-diaphragm can assemblies, i.e., cap-can assembly shown in Fig. 2 and described in Section II D 1. Such combination provides significant flexibility in pressure paths by allowing multiple compression and decompression cycles with controlled amplitude and at predefined pressure slew rates. This type of setup (Fig. 9) was used to study targeted nucleation of metastable polymorphs of germanium during decompression experiments[38] and is now commonly used for other experiments requiring bidirectional pressure control.

I. Dual ("push-pull") double-diaphragm setup for cold-finger type (sample in vacuum) cryostat

The dual ("push-pull") double-diaphragm setup is found to be especially useful in cryogenic experiments. The majority of cryogenic experiments are very time consuming and it can take several hours to prepare a cryostat with a DAC, cool the DAC down, and stabilize at a temperature of interest. Therefore, the ability to change pressure bi-directionally (i.e., for mapping phase diagrams) in order to optimize the use of the experimental time (beamtime) is highly desirable. Also, during cooling, the pressure in the DAC typically increases uncontrollably (usually by several GPa) due to differences in thermal expansion of diamonds and metal parts (metal shrinks several times faster than diamond) and an increase in spring constant of the spring washers. Many high-pressure experiments require very low (sub-GPa) initial sample pressures at cryogenic temperatures. One way of minimizing sample pressure increase during cool-down is by transferring the force from the DAC pressurizing screws (which cannot be changed at low temperature) to the double-diaphragm before cooling. While this approach often works, maintaining low sample pressure during cool-down is not guaranteed due to the absence of a controllable counter-force.

At cryogenic temperatures, the friction between DAC parts is usually significantly higher than at room temperature, partially due to the freezing of impurities trapped in between the piston and cylinder. Because of this, the efficiency of the double-diaphragm decreases significantly and it becomes impractical to use passive decompression springs. To solve this problem and increase the efficiency of high-pressure experiments at cryogenic conditions, we have designed a dual double-diaphragm clamp mechanism (Fig. 10) based on a tie-rod frame design (see Sec. II E). As in Sec. II E, the copper cooling block is mechanically decoupled from the steel pressurizing assembly with two double-diaphragms. Direct contact with the copper block allows a sample temperature of 7-8 K. Also, the dual ("push-pull") double-diaphragm pressurizing frame allows accurate bidirectional sample pressure control in the DAC from a few tenths of GPa to maximum pressure allowed by the experimental setup.

III. MECHANICAL REMOTE PRESSURE CONTROL (GEARBOX)

Even though pneumatic/membrane control remains the most common means of remote pressure control, in certain cases, motor driven mechanical devices (gearbox) have a

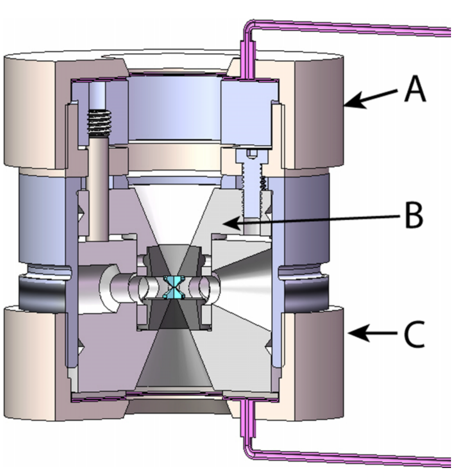

FIG. 9. Dual double-diaphragm setup for compression-decompression experiments at room temperature. A–DAC decompression attachment, B–DAC, and C–double-diaphragm cap-can assembly.

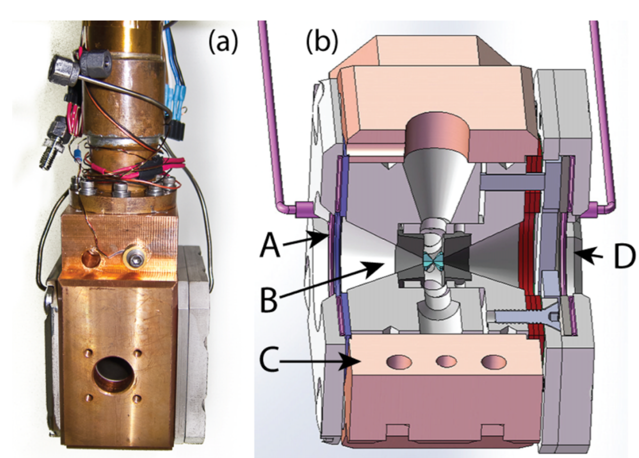

FIG. 10. Dual ("push-pull") double-diaphragm pressure control frame for compact cryostat. A–compression double-diaphragm, B–DAC, C–directly cooled cryogenic clamp, and D–decompression attachment.

number of advantages. The major advantage of the motor-driven system is smooth and efficient pressure change and the possibility of super fine pressure and temporal resolution. Theoretically, pneumatic drives should provide identical fine resolution, but in practice are less advantageous when super fine pressure increments are required. This is partially due to creep and relaxation in the membrane or double-diaphragm itself. This becomes very important at low and moderate pressures for studying soft materials such as clathrates[39] or other systems where precise and fine increments in pressure are important.

Another advantage of a gearbox drive is their independence from the pressure screws. Unlike pneumatic (membrane or double-diaphragm) or piezoelectric systems which apply an incremental dynamic load over and above the preload supplied by the screws, the mechanical gearbox system is rotating the pressure screws themselves and can be engaged or disengaged at any point without changing pressure in the DAC. This allows one to pause experiments at any time and continue the same or different measurements on the DAC without disturbing the sample pressure. Yet another advantage of the gearbox is the possibility of using it at low cryogenic temperatures, below 5-6 K, when pneumatic drives cannot be used effectively.

At HPCAT, the gearbox motor is driven remotely from outside the experimental station by the same Experimental Physics and Industrial Control System (EPICS) control system used to operate other beamline apparatus (although it can be easily driven independently). Therefore, it is relatively easy and straightforward to synchronize the DAC pressure with the rest of the experimental controls for automatic sequencing and data collection.

The first version of motorized DAC gearbox used at HPCAT was based on a Mao-Bell piston-cylinder DAC with a double lever arm pressurizing mechanism.[22] The gearbox system allowed changing pressure in the DAC remotely from outside the synchrotron x-ray hutch while collecting a dense dataset of diffraction images to multi-megabar pressures (230 GPa) in a few hours. While the system showed excellent performance, it was only restricted to Mao-Bell type DACs combined with a bolt-spring-lever arm assembly. Below is the description of a newer gearbox designed to control pressure in symmetric (and compatible) DACs.

A. HPCAT gearbox design

The HPCAT motor driven pressure control system (gearbox) was specifically designed to control pressure in four screw actuated "symmetric type" piston-cylinder DAC or any other compatible DAC (i.e., four screw version of Merrill-Bassett DAC[40] or Mao-Bell DAC[41] which has two right-handed and two left-handed loading screws on 1.50 in./38 mm diameter). Fig. 11 shows the HPCAT gearbox coupled with a "standard symmetric" DAC of Carnegie design. The DAC is rigidly held in the kinematic DAC holder and thus, the position of the sample with respect to the beam remains stable (within a few micrometers) while the pressure is being changed; therefore, there is no need for realignment of the sample at every pressure point. The gearbox is normally positioned upstream of the x-ray beam, allowing the downstream opening

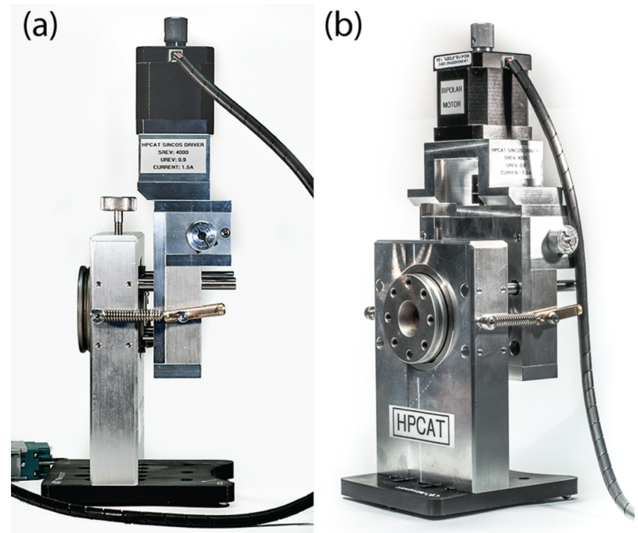

FIG. 11. HPCAT gearbox coupled with "standard symmetric" DAC of Carnegie design.

of the DAC to be fully utilized for x-ray diffraction and scattering or other measurements. For single crystal diffraction measurements, there is a horizontal slot in the casing of the gearbox for X-ray passage (Fig. 12) that allows the DAC-gearbox assembly to be rotated by ±30° without blocking the x-ray beam.

The gearbox portion of the assembly is designed to have freedom of movement along the axis of the DAC. This allows the assembly to follow the axial movement of the advancing pressure screws. Another benefit of this design is that it accommodates various screw lengths and Belleville washer combinations. Positive engagement of the gearbox is made possible by a linear guide and extension spring arrangement.

A view of the inner workings of the gearbox is shown in Fig. 12. The device is based on five matching pairs of right handed stainless steel worm shafts and bronze worm wheels of 48 pitch mounted on hardened steel shafts supported by ball bearings. The top pair transfers the rotation of the vertical shaft

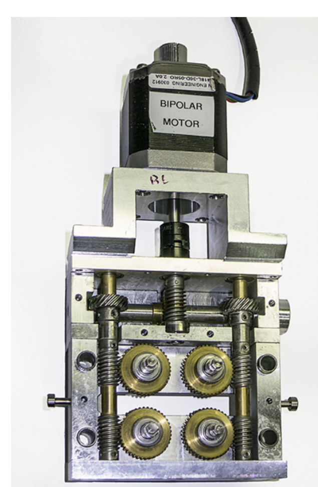

FIG. 12. Open view of HPCAT gearbox for room and high temperature experiments.

from the high torque stepper motor to the horizontal distribution shaft with a 20:1 gear ratio. The rotation of the horizontal shaft is translated into rotation of two vertical shafts with two pairs of helical gears. These two vertical shafts rotate four horizontal shafts aligned with the DAC pressurizing screws via four pairs of worm gear assemblies. The four horizontal shafts have either permanent or removable hexagonal terminations which are used to rotate the socket head screws on the DACs. Due to semi-symmetric arrangement, left and right hex shafts rotate in the opposite directions.

The worm shaft and wheel have a double thread (pressure angle of 20°) with 20:1 gear ratio. The theoretical gear ratio of this gearbox assembly is 400:1; thus, one full rotation of the motor shaft corresponds to a 0.9° rotation of the DAC pressurizing screws. Due to friction, the efficiency of each pair of gears, both two worm and one helical, is about 70% each;[42] therefore, the effective torque amplification is approximately 135 times.

One of the biggest advantages of the gearbox is its use at cryogenic temperatures below 5-6 K. Controlling pressure in a piston-cylinder DAC at these temperatures can be very challenging because of significant friction in the DAC, freezing of helium, or insufficient helium pressure in pneumatic (membrane, double diaphragm) devices. Thus, one version of the HPCAT gearbox is a modification specifically designed for use in a helium flow cryostat. With proper dry lubrication (Teflon, dry moly, graphite, or tungsten disulfide), the gearbox remains operational even when submerged in liquid helium.

Due to the heat generated by a motor, it was removed from the cryo-gearbox design. The cryogenic version of the gearbox has only one gear train (four pairs of worm—worm gears with 20:1 gear ratio) close to the DAC (Fig. 13). The gearbox is driven either manually with wrenches or by a geared stepper motor from outside the cryostat. Torque is transmitted by two tie-rods coupled through two spur gears that rotate in opposite directions in order to minimize the net torque on the DAC-gearbox assembly. This canceling effect minimizes motion of the sample during sample pressure changes.

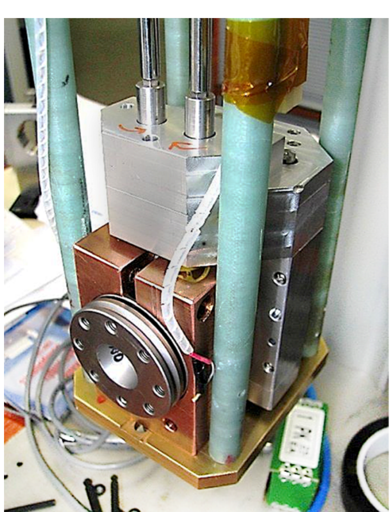

FIG. 13. 20:1 gear ratio cryostat type gearbox with engaged symmetric DAC.

B. Examples of gearbox use

The gearbox is in use at HPCAT in a variety of diffraction and spectroscopy experiments at various pressures. Due to the popularity of the gearbox, HPCAT has multiple copies of the system which can be used simultaneously. The maximum pressure which can be achieved in a gearbox-driven DAC mostly depends on the diamond anvil cell configuration (diamond culet size, tightness of pressurizing screws, etc.) and in majority of cases, it is in the megabar regime. For example, Kalita[43] and Souza-Neto[44] studied phase transitions and equations of state (EOSs) of TiH_2 and EuO, respectively, to pressures exceeding 90 GPa where the maximum pressure was determined by the stability of the sample rather than gearbox performance. The cryogenic version of the gearbox is also frequently used with a He flow cryostat for angle dispersive x-ray diffraction (often combined with electrical resistivity) measurements,[45] typically when pressures exceeding 100 GPa are required.[46]

One of the major advantages of the gearbox is the ability to collect high-pressure diffraction data in a semi-automated mode (i.e., when the pressure increase can be programmed to be performed between x-ray exposures). This function is especially useful in cases such as single crystal diffraction where the sample has to be rotated around a vertical axis during the x-ray exposure. Fig. 14 shows integrated diffraction patterns of single crystal NaCl through B1-B2 phase transition in helium pressure medium[47] and the evolution of relative volumes of B1 and B2 phases of NaCl. On-the-fly diffraction data were collected while the sample was rotated by 30° about the vertical axis. Pressure was increased automatically between diffraction collections in pressure increments of about 0.25 GPa.

IV. DYNAMIC PRESSURE CONTROL

While there are many mechanisms for controlling pressure, such as double-diaphragm or mechanical gearbox, these

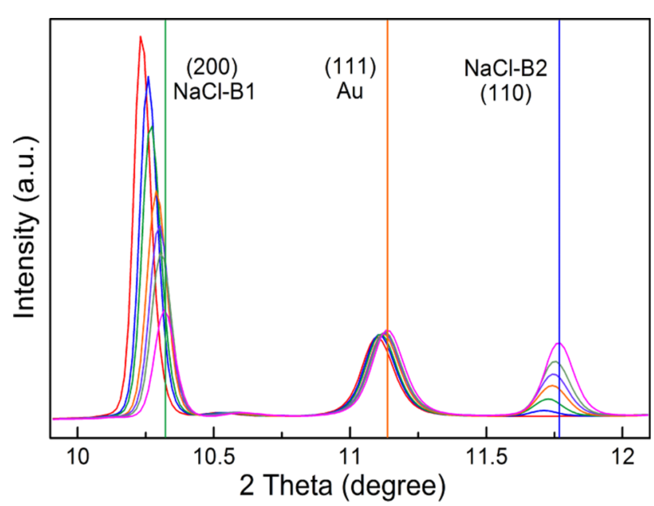

FIG. 14. Integrated diffraction patterns of single crystal NaCl through B1-B2 phase transition. The peaks from left to right are (200) peak of NaCl in B1 phase, (111) peak of gold, and (110) peak of NaCl in B2 phase. The vertical lines correspond to peak positions of NaCl and Au at 31 GPa according to EOS parameters from Refs. 48 and 49.

devices lack the ability to perform modulated or very fast pressure changes at the fast compression/decompression rates required for studying kinetics of phase transitions and metastable phases. The piezoelectric actuator is the preferred drive mechanism for such time-resolved experiments.

The first attempt to use a piezoelectric actuator to control DAC pressure in a cryostat was undertaken by Tozer in 1993.[50] These first attempts were unsuccessful due to cracking of the transducer under load. The first successful piezoelectrically driven DAC (dynamic DAC or dDAC) was presented by a group at Lawrence Livermore National Laboratory.[23,24] They adapted piezoelectric actuators to a conventional diamond anvil cell design, which enabled a repetitive and precise time-dependent load/pressure profile to a sample. This dynamic DAC was successfully used to study compression rate dependent kinetics of solidification, crystal growth, and various phase transitions under variable compression rates of water/ice,[51–54] as well as other elements and compounds.[55–57]

Although piezoelectric actuators typically have a limited stroke, they have the advantage of very fast modulation (sub kHz) rates, as well as excellent precision and repeatability. At HPCAT, we have developed piezoelectric drives which can be adapted to a variety of DACs, combined with pneumatic pressure control, and be used in compression and decompression modes. Some of these attachments along with examples of experimental capabilities are described below.

A. Piezoelectric drive attachment for DACs

As discussed above, our approach was not to design a specialized dynamic DAC, but rather make a flexible piezoelectric pressure control system which can be used with various types of common DACs. Depending on the experiment, the piezoelectric drive can be used in various modes: decompression, direct compression, and reverse compression. The decompression mode is used either for single jump pressure quench experiments or for cyclic compression with different amplitudes and shapes of pressure paths. The direct compression mode is used for fast pressure increase at maximum compression rates of tens of TPa per second (see Section III C in Ref. 25). The reverse compression mode is used for rapid compression experiments but with a more controllable starting pressure point and pressure ramp rate.

Fig. 15 shows the schematics and a photograph of a dynamic piezoelectric drive attached to a symmetric DAC in decompression (panels (a) and (b)) and direct compression (panel (c)) modes. The assembly for decompression uses the same principle of operation as the double-diaphragm decompression system (Fig. 8), where the piezoelectric actuator housing (I and J) is bolted with two screws (G) to the piston part of the DAC. The piston (F) and cylinder (H) parts of the DAC are pushed apart by pushing pins (K and L) due to expansion of the piezoelectric actuator (C). We use Physik Instrumente (PI) PICA high-load through ring actuators (25 mm OD $\times$ 16 mm ID) with relatively long (60 and 80 μm) displacements. Note that the actual displacement of the DAC diamonds is much smaller due to elastic deformation and contact gaps between different parts of the assembly. The inner hole allows for passage of the x-ray beam, thus requiring only one actuator

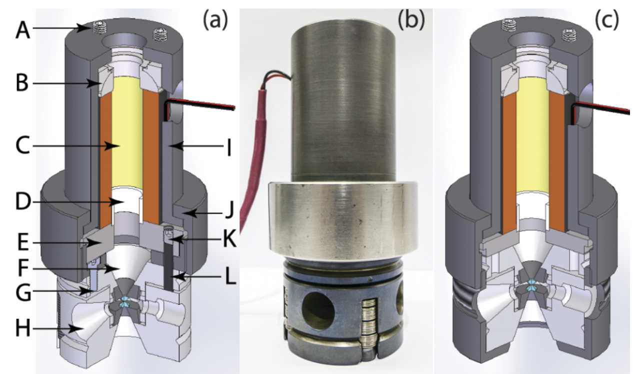

FIG. 15. Dynamic piezoelectric drives (actuators) attached to a symmetric DAC. (a) Schematics and (b) photograph of the assembly in decompression mode, (c) piezoelectric drive–DAC assembly in compression mode.

(instead of 3 actuators in previous designs[24]) which can be placed concentrically with the DAC load axis. The actuator has a blocking force of 9 kN, which is sufficient to increase sample pressure in a DAC to almost 3 Mbars using a rhenium gasket between beveled anvils of 250 μm culets and 50 μm flats (i.e., Section III C of Ref. 25). Due to the brittleness of the ceramic actuator, special care should be taken to make sure that the actuator and all adjoining parts have a maximum contact area. Using a spherical washer (B) with preloading swivel screws (A) and an insulating alignment ring (D) minimizes the stresses experienced by the piezoelectric actuator (C) in contact with the piston-pusher (E).

In the current setup (Figs. 15(a) and 15(b)), the initial sample pressure in the DAC is generated by the pressurizing screws through Belleville washers. Increasing the voltage on the piezoelectric actuator results in a decrease of sample pressure. Typically, a few cycles of increasing-decreasing voltage brings the gasket into an elastic regime and allows cyclic experiments with pressure paths controlled by various waveforms (trapezoidal, square, and sinusoidal functions) over a typical pressure range of several GPa. The displacement of the piezoelectric actuator is controlled by a PICA piezoelectric high-power amplifier/controller with 0-1100 V output, which in turn is controlled by a standard function generator capable of various waveforms. The response time of piezoelectric actuators primarily depends on their capacitance. The maximum sinusoidal frequency of our current piezoelectric actuators is about 400 Hz and the fastest, full displacement time is about 250 μs (when step or square function is applied).

The piezoelectric-actuator–DAC assembly can also be used in compression mode (Fig. 15(c)) to provide step and ramp type increases of sample pressure. As of this writing, it provides the fastest mechanism for large pressure increase in a DAC. So far we have been able to achieve a sample pressure increase rate of 34 TPa/s (43 GPa in 1.25 ms) (see Section III C of Ref. 25).

The initial sample pressure in the DAC/piezoelectric actuator combination is controlled by both the DAC pressurizing screws and the actuator preloading screws. The pressure increase rate is controlled by the amplitude and rate of change of voltage applied to the actuator. The pressure/voltage relations cannot be precisely calculated beforehand and require calibration for each set of experimental conditions. Note that the assembly in compression mode cannot be effectively used for cyclic pressure modulation due to the absence of an opposing, decompressive force, unless the DAC is specifically equipped with a respective counter force mechanism (springs or double-diaphragm setup in decompression mode).

The piezoelectric drive can be easily combined with a pneumatic double-diaphragm compression mechanism. Fig. 16 shows a symmetric DAC in a double-can compression assembly similar to that in Fig. 2 with attached piezoelectric drive assembly (Fig. 15) in decompression mode. Such combination allows remote control of base pressure (i.e., the pressure prior to engaging the piezoelectric actuator) with a double-diaphragm and further pressure modulation, of several GPa, with the piezoelectric actuator.

B. Application examples

In this section, we present two examples of diffraction measurements with pressure modulation by the piezoelectric drive.

In the first example, we studied the melting and crystallization processes of gallium as a function of compression and decompression rate. Pure metallic gallium shows an unusual phase diagram and properties. Even though gallium is solid at ambient pressure (phase I), the gallium melt can be easily supercooled, so that gallium can remain in a liquid metastable state for long periods of time, which is not typical of any metal.[58] Between ~0.5 and 2.5 GPa at room temperature, the stable phase of gallium is liquid.[59] Thus, compression of Ga(I) at room temperature first results in pressure melting followed by pressure-induces crystallization into Ga(II) phase.

Liquid gallium readily reacts with metals. To prevent possible reaction with the rhenium gasket, the sample chamber was first filled with NaCl, then a ~50 μm hole was laser-drilled in the sodium chloride, and a piece of Ga was placed into this hole. Thus, NaCl served as pressure medium and pressure marker[48] as well as a chemical barrier. The angle dispersive diffraction measurements were performed in a symmetric DAC inserted into a double-diaphragm cap-can assembly and coupled with a piezoelectric drive in decompression mode (Fig. 16). For diffraction measurements, we used a monochromatic x-ray beam with an energy of 20.000 keV focused to $\sim 5 \times 7\ \mu m^2$ (V × H FWHM). The diffraction spectra were collected with a prototype Dectris Eiger 1M detector with a rate of 800 full frame images per s (maximum for that prototype). 1.25 ms exposure time (with ~20 μs readout time) was sufficient to clearly see the appearance and disappearance of liquid and solid phases of gallium (even though the crystalline phase was always either single crystal or extremely textured). Note that despite short exposure time, we could clearly see diffraction from liquid Ga, and this was the base for phase boundary positioning. Fig. 17 shows sinusoidal modulation of the pressure in the Ga sample from ~1.7 to ~2.8 GPa at 20 Hz frequency as a function of time. Black squares represent solid phase and red triangles represent liquid Ga. Our data suggest that at this compression/decompression rate (43.7 GPa/s based on the equation of state of NaCl[48]), there is a hysteresis between crystallization and melting, and the liquid phase can be over pressurized (analogous to supercooled) resulting in different melting and crystallization pressures of Ga. Thus, this new technique provides a route of gaining further insight into material behavior, such as possible hysteresis and kinetics of solid-solid and solid-liquid transitions.

The use of a piezoelectric drive attachment is not restricted to symmetric type DACs. It can be used with virtually any type DAC for various types of experiments. For example, high-frequency radial diffraction measurements in a panoramic DAC combined with fast, precise cyclic loading/unloading by a piezoelectric drive can be performed over very short time periods. This short time scale is necessary for studying the rheology of minerals from the elastic response and lattice relaxation as a function of pressure, temperature, and compression rate.

Fig. 18 shows a compact panoramic DAC, identical to that shown in Fig. 3, with an attached piezoelectric drive. The base sample pressure in this configuration is generated

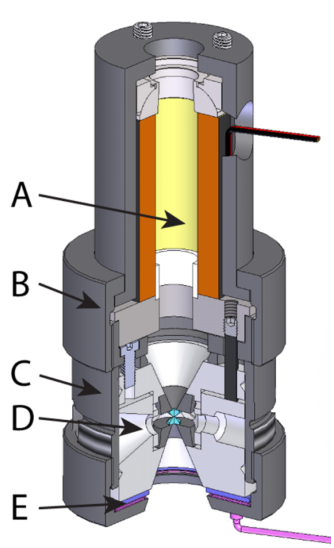

FIG. 16. Dynamic piezoelectric drive in decompression mode and double-diaphragm assembly in compression mode coupled with symmetric DAC. A–piezoelectric actuator, B–actuator housing, C–double-diaphragm can assembly, D–DAC, and E–double diaphragm.

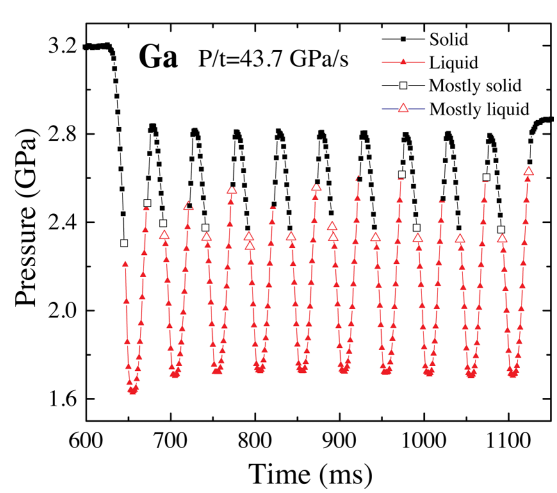

FIG. 17. Modulation of pressure in Ga sample as a function of time. The modulation frequency is 20 Hz and data collection rate is 800 Hz.

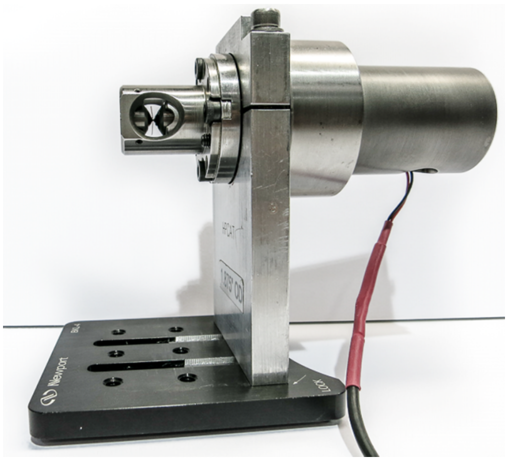

FIG. 18. Compact panoramic DAC for radial diffraction measurements with piezoelectric drive attachment.

by the Belleville washers placed on the DAC pressurizing screws, while the pressure modulation is performed by the piezoelectric actuator in decompression mode.

Shown in Fig. 19 are results of cyclic radial diffraction measurements on the B2 phase of KCl. The KCl sample was placed into a sample chamber made in a Kapton-boron epoxy x-ray transparent gasket,[60] and the pressure was manually increased to about 10 GPa before the piezoelectric actuator was engaged. In this experiment, the actuator was modulated with a trapezoidal waveform of different amplitudes.

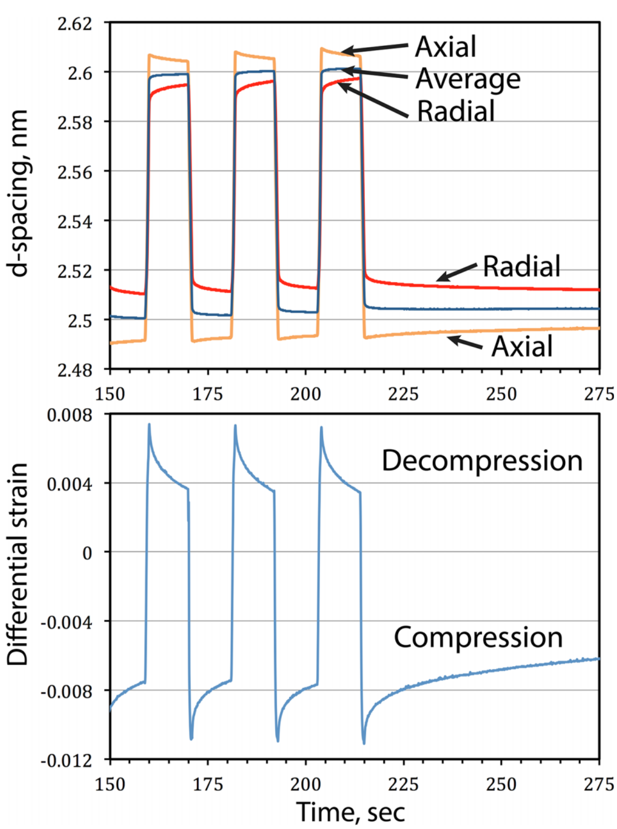

FIG. 19. (Top) d-spacing of (110) reflection of KCl in B2 phase 3.5–9.6 GPa in radial diffraction geometry during cyclic compression-decompression. (Bottom) Corresponding differential strain (normalized difference between d-spacings in radial and axis directions).

The typical period was 10 s with a rise/fall time of 1 s. Due to the good elastic response of the boron-epoxy gasket and a reasonably good match in the compressibility of the gasket and KCl, the average maximum pressure amplitude was ~6 GPa (from ~3.5 GPa on decompression to ~9.6 GPa on compression). The radial diffraction images were collected using a Dectris Pilatus 1M with 100 ms exposure.[25] For each diffraction image, we performed three individual integrations: horizontal sector of 10° (axial direction), vertical sector of 10° (radial direction), and integration of the complete image (average). Fig. 19 (top) shows the d-spacing of the (110) reflection of KCl from axial, radial, and average integrations. As expected, the maximum compression (minimum d-spacing) during compression and maximum decompression (maximum d-spacing) during decompression are observed along the axial direction, while the d-spacing in the radial direction changes the least. Fig. 19 (bottom) shows the corresponding differential strain (normalized difference between d-spacings in radial and axis directions). During both fast compression and decompression, the magnitude of the differential strain rapidly increases at first, then decreases with time, asymptotically approaching an equilibrium value due to the lattice relaxation. The analysis of such time-dependent stress-strain relations can provide a wealth of information about strength, elastic, and rheological properties of materials.[61,62]

V. SUMMARY

In this paper, we presented an overview of instrumentation for remote and automated DAC pressure control designed at HPCAT over the last several years. These instruments represent loading-unloading devices used with various commonly used DACs. This concept eliminates the need for specialized or dedicated diamond cells and allows reliable remote sample pressure control at various temperature conditions from 4 K in cryostats to several thousand K in laser-heated DACs. The pressure control devices fall into three major categories: pneumatic (double-diaphragms), mechanical (gearboxes), and piezoelectric. Various modular drives can be combined into versatile yet user friendly assemblies (e.g., compression-decompression assembly with two double-diaphragms, piezoelectric—double-diaphragm assembly, and so on) for accurate control of sample pressure and compression rate. This allows both time and pressure change rate to become experimental variables. In addition to stable bidirectional pressure control during static experiments, these new devices allow unidirectional (fast ramp) compression and decompression, as well as cyclic pressure modulation in DACs with pressure change rates of tens of TPa/s, thus making possible a variety of novel time resolved experiments with DACs.

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