Standard Operating Procedure – Diamond Anvil Cell

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- Special thanks to - Qi Li, Arturas Vailionis and Angela Hwang
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Contents

1 Diamond Anvil Cell (DAC)

1.1 Applications/uses of DAC research

It is important to understand if DAC research is the appropriate tool for you to use as it requires significant investment of time, money and lab space. The learning curve to be able to load cells successfully is sharp and takes at least 3-6 months of rigorous practice to be able to do experiments.

Initial investment –

1) Diamond anvil cell – $5,000-10,000 depending upon the type and measurement to be done
2) Diamonds – $1,000 – 2,000 each and can often break/get damaged during the learning cycle
3) Stereo microscope - $7,000 – 10,000
4) Miscellaneous – Ruby powder, supplies and materials - $2,000-3,000

Diamond anvil cell is a valuable tool to study materials under high pressures and mechanical stresses. Diamond anvil cells can be heated as well to access high pressure and high temperature phases of materials. Both hydrostatic and non-hydrostatic stress states can be generated which can show different material responses.

Diamonds are transparent to a UV-vis light and x-rays which form the basis of several characterization techniques. Near visible light techniques like UV-vis spectroscopy, Raman scattering, Photoluminescence, pump and probe studies and life-time measurements as well as x-ray techniques like x-ray diffraction (XRD), x-ray absorption techniques and tomography are widely used. Combination with secondary techniques like electrical measurements, NMR and magnetic studies under pressure are also performed.

Research areas where DAC is used widely are – nanomaterial deformation and mechanical studies, simulating interior of earth's core, phase transformations, bulk modulus calculations, thermodynamics and others.

1.2 List of DAC geometries that are compatible with XRD

Requirements –

1) High angular opening (above 60 deg. Two theta)
2) Required pressure range achievable
3) Diamond culet size above 300 μm (gasket hole 250-300 μm)
4) Should be able to fit on a goniometer head

Fig. 1.2.1: Support design to fit larger DAC onto the goniometer head. Image: Bruker

List of suppliers for DAC –

1) Almax-Easy Lab – https://www.almax-easylab.com/
2) DESY DAC – http://photon-science.desy.de/facilities/on_site_infrastructure/laboratories_technical_infrastructure_shift_service/sample_environment_and_ecsi/high_pressure_instrumentation/diamond_anvil_cells_dac/index_eng.html

List of compatible DACs

1) Merrill Basset cell (*1*)

Fig. 1.2.2: Merrill Basset cell. Image: DESY and reference *(1)*

Available for order from DESY DAC
Notes – Opening window is small (though enough), maximum attainable pressure is low and best use for single crystal XRD work

2) Diacell Bragg-Mini

Fig. 1.2.3: Diacell Bragg Mini. Image: Almax easy lab

Available for order from Almax easy-lab
Notes – Opening is 85º and maximum pressure attainable is 20 GPa

3) Plate DAC

Fig. 1.2.4: Plate DAC. Image: Almax easy lab

Available for order from Almax easy-lab and DESY
Notes – Opening is 85º

4) One20DAC

Fig. 1.2.5: One20DAC. Image: Almax easy lab

Available for order from Almax easy-lab

Notes – Opening is 120^{o} and maximum pressure 50 GPa

5) Bragg-(S) and Bragg-(S)Plus

Fig. 1.2.6: Bragg-(S) cell. Image: Almax easy lab

Available for order from Almax easy-lab

Notes – Opening is 90^{o} and maximum pressure 100 GPa

1.3 Components of DAC

Components of diamond anvil cell –

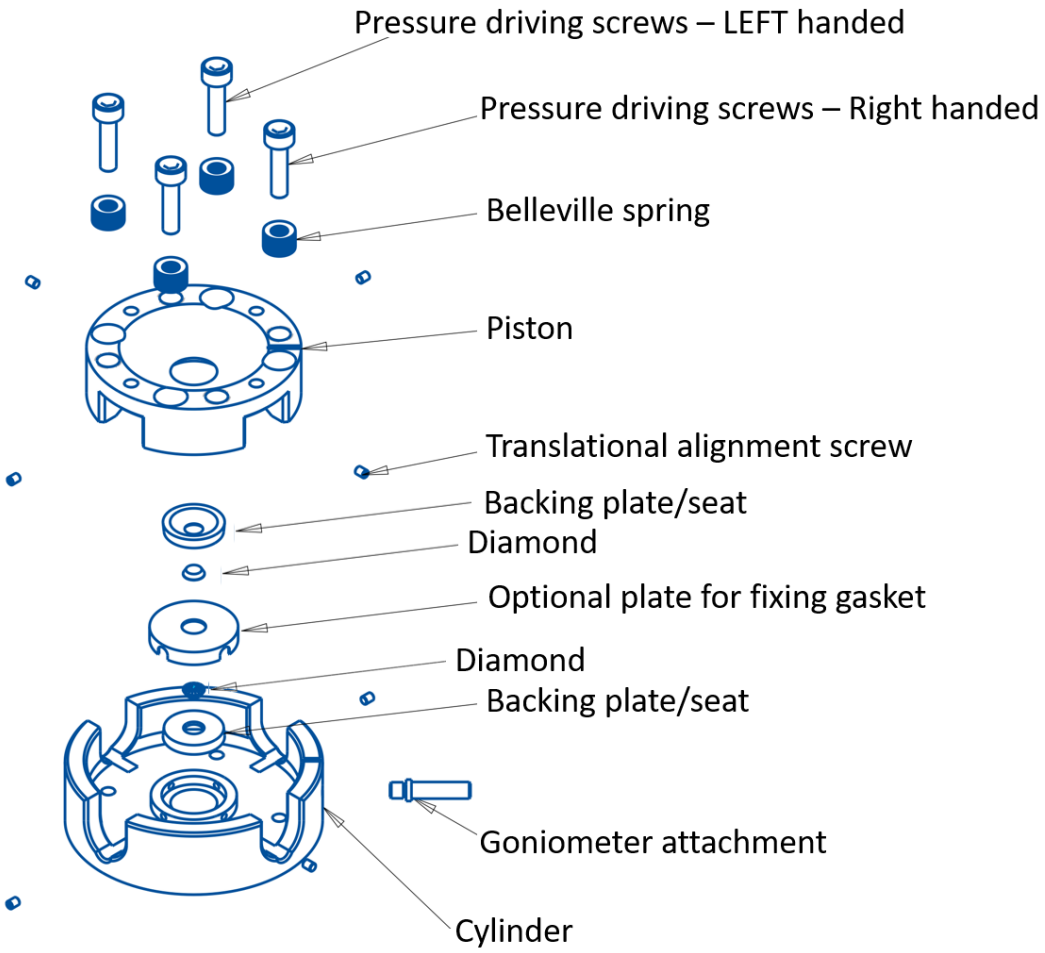

Fig. 1.3.1: Diamond anvil cell components.
Image: Almax-easy Lab.

Diamonds –

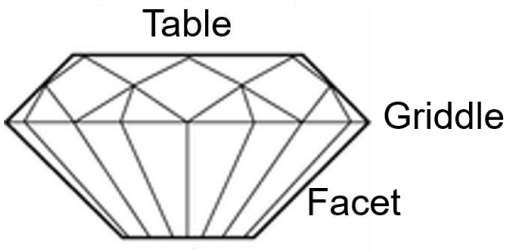

Fig. 1.3.2: Diamond. Image: SERC – Carleton College

1.4 Theory for DAC construction and pressurization

Cell design: DAC consists of two opposing anvils sitting on WC backing plate/seat and a metal gasket between them (see Fig 1.4.1 (*2*, *3*)). The metal gasket has a hole in the center which is the sample chamber. The space between the diamonds and metal gasket is filled with a pressure transmitting medium (PTM) (solid, liquid or gaseous). The pressure is applied by applying small

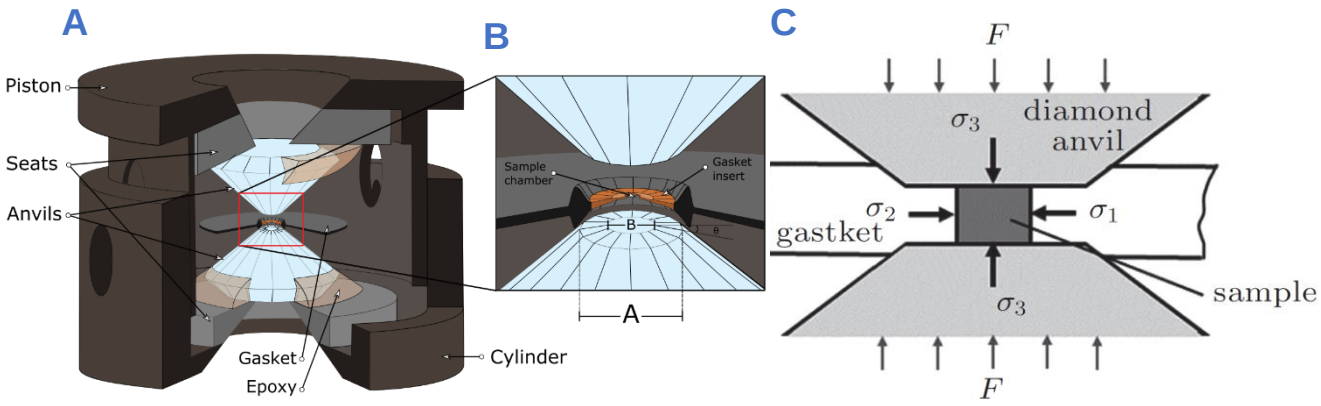

Fig. 1.4.1: Diamond anvil cell design. A) Complete diamond anvil cell. B) Diamonds and sample chamber in the gasket. C) Different stresses in the sample chamber. Image reference *(2,3)*.

load on the diamonds using pressure driving screws. The small culet of the diamond applies pressure on the PTM and the metal gasket surrounding the sample chamber. The gasket shrinks (few microns) and applies pressure on the PTM from the side. The pressure state in the sample chamber depends upon the state of the PTM. If the PTM is liquid or gaseous then the pressure is hydrostatic or quasi-hydrostatic, and if the PTM is solid then the pressure is non-hydrostatic and uniaxial along the diamond axis.

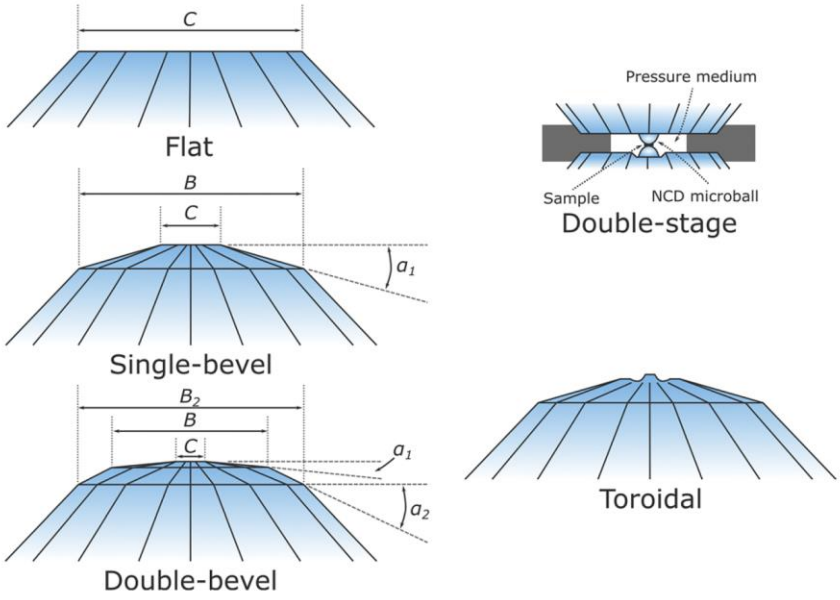

Fig. 1.4.2: Different types of diamond anvils. Image reference *(4)*.

Diamond design: Different types of diamonds are available (see Fig. 1.4.2). Different designs have different maximum pressures achievable. Flat design is the most basic, then bevels are designed on the flat anvil to extend the pressure range. Double stage and toroidal designs push the maximum range even further reaching pressures more than 1000

GPa. See reference (*4*) for details regarding the maximum achievable pressure for each type of diamond. The maximum pressure is also dependent on the culet diameter of the diamond, smaller the culet higher the pressure achievable.

X-ray diffraction through DAC: XRD is a powerful technique to get atomistic information from the sample under DAC compressions. The most common direction of incident x-ray beam is along the axis of the diamonds. In this setting the x-ray beam must have high enough energy to pass through the diamonds and still give strong diffraction signal from the sample. Bruker D8 venture on campus has Mo x-ray source which has high enough energy to pass through the diamonds. The diffraction signal is collected from the planes which are aligned at θ angle along the axis so, the diffraction planes experience high shear forces (when using non-hydrostatic and to an extent quasi-hydrostatic pressure mediums) (see Fig. 1.4.3 (*5*)). The diffraction is collected on a 2-D detector and 1-D XRD plots can be generated by radial averaging.

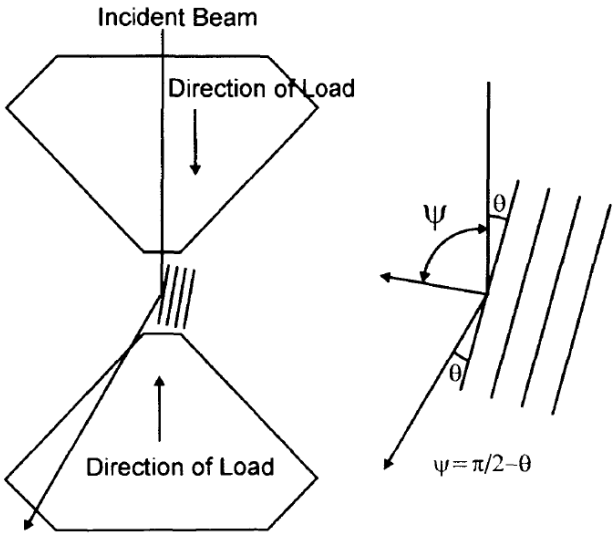

Fig. 1.4.3: Diffraction plane in diamond anvil cell versus loading direction. Image reference *(5)*.

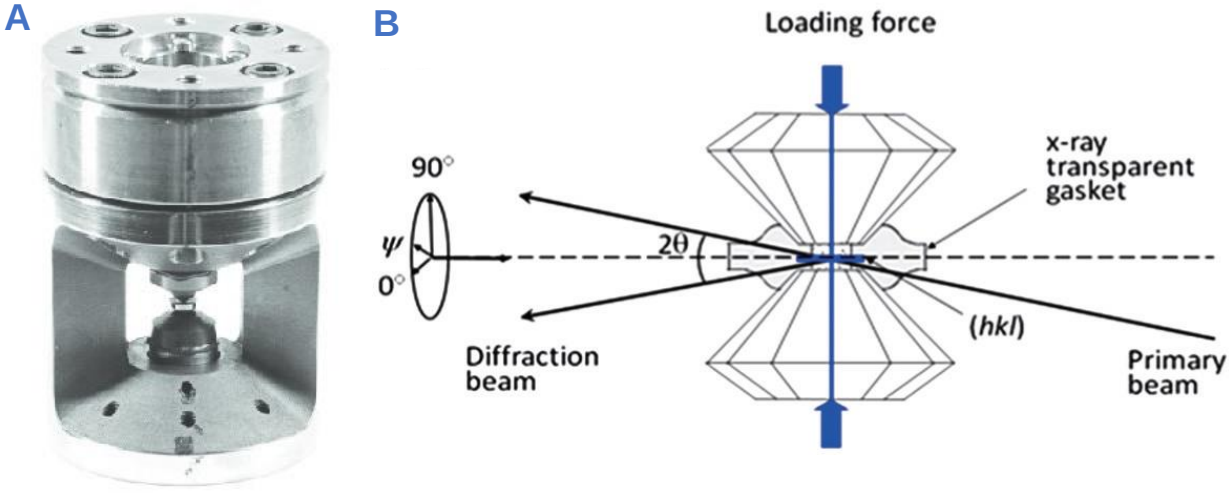

Fig. 1.4.4: Radial diffraction. A) Panoramic DAC. B) Radial diffraction geometry. Image reference *(3-6)*.

Radial diffraction is the other geometry in which diffraction signal from the sample can be collected. This geometry is currently not compatible with Bruker D8 venture machine on campus. To use this diffraction geometry, one needs special DAC called “panoramic DAC” (see Fig. 1.4.4 (*3*, *6*)). This DAC allows one to align the diffraction beam parallel to the gasket of the DAC and get a diffraction pattern from the sample. The gasket must be x-ray transparent to allow the incident beam to pass through the gasket. Researchers use Be gaskets or Boron-epoxy-Kapton gasket. These are discussed further in “Choosing gasket material” section.

1.5 Steps for gluing and aligning diamonds

Setting up the DAC requires perfect alignment of the diamonds to minimize the shear forces experienced by the diamonds. There are two major alignment procedures to be done – horizontal alignment and tilt alignment (see Fig. 1.5.1 (*5*)). Most DACs do not have the option for tilt alignment – the manufacturer makes sure that the backing plate and diamonds are perfectly flat when fixed properly. Horizontal alignment is required for most DACs.

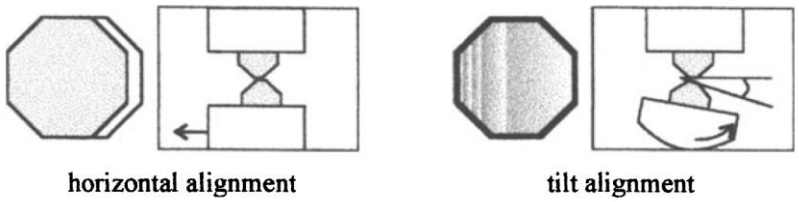

Fig. 1.5.1: Diamond alignment. A) Horizontal. B) Tilt alignment. Image reference *(5)*.

Glue: STYCAST 2850FT – Catalyst 9 or 24LV is used to glue the diamonds to the backing plate.

Newton fringes: This is the main phenomenon used to make sure the diamonds are aligned. Newton fringes/rings are observed due to interference of the reflected light between two surfaces. When the surfaces are not perfectly flat and have point/line contact (tilted flat surfaces) rather than planar contact between the two flat surfaces Newton fringes are observed. Fig. 1.5.2 shows the types of fringes that can be seen where the point x is in contact between the surfaces. The Fig. 1.5.3 (*7*) shows the typical observed fringes when the flat diamonds touch each other that are not aligned for tilt.

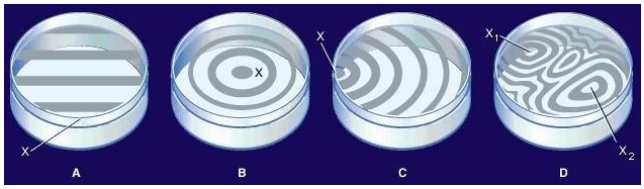

Fig. 1.5.2: Newton rings when two surfaces are in contact. Image: Encyclopedia Britannica, Inc.

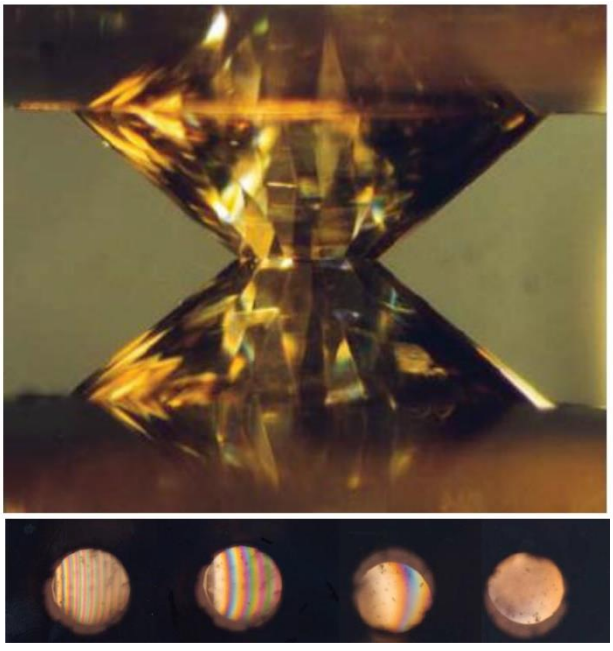

Fig. 1.5.3: Diamonds in contact and Newton fringes. Image reference *(7)*.

No-tilt alignment possible: In this case the diamonds are carefully placed on the backing plate rough horizontal alignment is first done and then the cell is assembled. The diamonds are slowly brought in contact by gently pushing them closer to each other by fingers (see Fig. 1.5.3). When the diamonds are close enough, they are observed in transmission through the top diamond under a microscope. Newton fringes are observed (see Fig. 1.5.3).

Then the diamonds are pushed further so that they adjust themselves on the backing plate/seat until the fringes disappear. The tilt alignment is done and can be observed from the side of the DAC (shown in Fig. 1.5.3). Then the cell is disassembled, and the glue is applied around the diamond using a needle. Care must be taken to avoid glue from seeping between the diamond and the

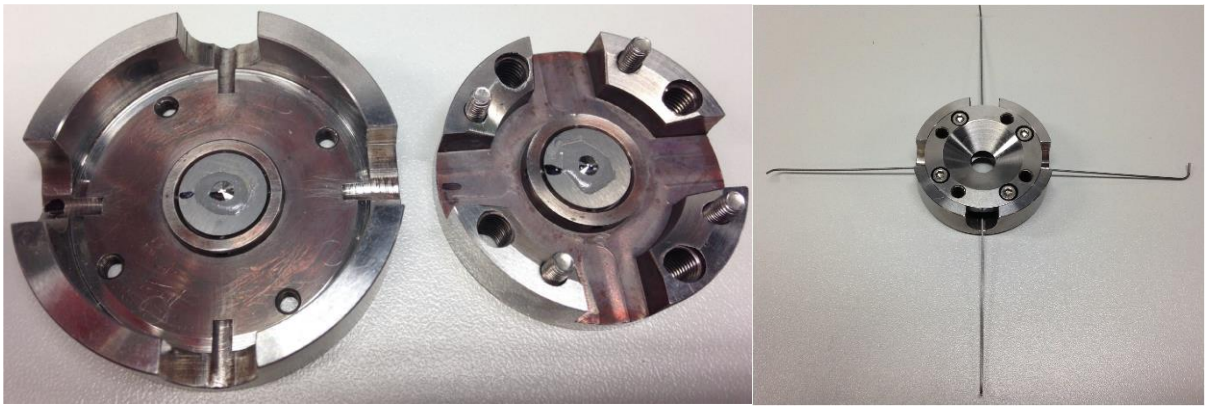

Fig. 1.5.4: One20DAC diamond anvil cell and accessing lateral screws for horizontal alignment. Image: Almax-easy Lab.

backing plate (or else the diamonds will be tilted) also, the glue must not cover the diamond culet and the portion of diamond where the gasket touches the diamond.

Horizontal alignment: This must be done after gluing the diamonds using the epoxy glue and letting it dry. Horizontal alignment is done by controlling the four lateral screws that move the backing plate (see Fig. 1.5.4). The cell is assembled and then the two faces of diamonds are brought close (don't let the diamonds touch each other) till you can see both the faces by changing the focus of the microscope. Then the screws are adjusted till the faces are aligned perfectly to each other.

Rotational alignment: This is only required for diamonds which have less than 8 sides. Generally, diamonds are cut to have 16 or 8 sides which makes the culets almost circular, so rotational alignment is not required. However, if using diamonds with less sides, the culet is not spherical then one needs to do rotational alignment. This procedure is tedious and can be done by rotating the backing plate or the cylinder/piston by hands itself until the alignment is done and marking the cylinder/piston to assemble the cell in the right way always.

1.6 List different pressure mediums and their hydrostatic limits

Pressure Medium	**P_{max} (GPa) of quasi-hydrostatic limit**
Silicon Oil	<2.0/0.9
Toluene	1.7
Water	2.2
Isopropyl alcohol	4.2
Glycerine:water (3:2)	5.3
Petroleum ether	6
Pentane-isopentane (1:1)	7.4
Methanol	8.6
4:1 Methanol:Ethanol	9.8
16:3:1 Methanol:Ethanol:H_2O	14.5
Glycerol	1.4
H_2	177
He	60-70
Ne	16
Ar	9
Xe	55
N_2	13

1.7 Choosing gasket material

Choice of gasket material and thickness depends on the maximum pressure desired for the experiment.

1) Common gasket materials include –
    a. Stainless steel
    b. Rhenium
    c. Tungsten
    d. Beryllium (dangerous)
    e. Ni(Cr 39-41%)(Al 3-4%) (Russian Alloy)
    f. CuBe
    g. Kapton/boron/epoxy
2) Researchers use rhenium and tungsten gaskets if they want to go to very high pressures more than 50 GPa.
    a. Drilling sample chamber in Re and W gaskets is not possible using a microdrill (the only campus facility to drill holes).
    b. Point contact EDM machine must be bought to drill holes, if one wants to use Re and/or W gaskets. Almax-easy lab sells one for around ~19,000 USD.
3) Stainless steel is used for lower pressures up to 40 GPa.
    a. T-301 stainless steel of 250 μm thickness from McMaster is used.
4) Maximum pressure reached during DAC compression depends on the initial indentation thickness of the gasket – thinner gasket for higher pressures.
5) Beryllium or Kapton/boron/epoxy gasket is used if one wants an x-ray transparent gasket.
    a. Machining beryllium is dangerous and can cause terminal disease called “Chronic Beryllium Disease”.
    b. Kapton/boron/epoxy gasket is an easy, cheap and safe alternative to Be. Please see reference (*8*) to know how to prepare Kapton/boron/epoxy gasket
6) Currently, the x-ray spot size (Bruker D8) is about 400-500um so we are getting very intense diffraction signal from the gasket. We are working on reducing the spot size down to remove gasket signal. With a 300um hole the gasket (stainless steel) gives 1 strong diffraction peak. If it is not possible to shrink the beam down, then using an amorphous gasket will be the only solution to avoid the gasket signal.

1.8 Preparation of different samples including nanomaterials

XRD requires one to load a lot of sample volume – the more sample the better the signal. Generally, the sample chamber is packed with sample. This however, depends upon the sample itself – chemical composition, grain size, and degree of crystallinity. Heavier metals are easier to get diffraction signal than light elements. Larger grain sizes and more crystalline regions with higher symmetries give stronger diffraction signal. There are multiple ways to load samples depending upon the sample and the technique –

Hydrostatic measurement – This stress state is used when researchers want to determine bulk modulus and other elastic constants using complicated equations of state. It is generally very tough to conduct a truly hydrostatic pressure condition as one must load only very few pieces of sample to surround the sample by pressure medium. This significantly drops the XRD intensity and one must go to synchrotron sources to get reasonable signal.

The requirements to conduct hydrostatic measurement are -

- PTM should remain liquid or become a very soft solid (quasi-hydrostatic) under pressure.
- PTM must be compatible and has favorable interaction (dissolves) with the sample.

These conditions are important because they ensure that the liquid surrounds each particle and applies a truly hydrostatic pressure on each particle. XRD collected is an average of large number of particles so, for true hydrostatic pressure medium each particle must be surrounded by PTM.

Following things can make the system non-hydrostatic –

- Particles are not soluble in the PTM and so they are clumped together and sitting at the bottom.
- Loading a lot of sample which increases particle-particle contact.

In this case, the particle is not surrounded by the PTM and particles are touching each other or the gasket/diamond. This changes the stress state on each particle and so we cannot use a hydrostatic pressure medium approximation.

Non-hydrostatic measurement – The stresses along the axis of diamond is much higher than the lateral stresses which creates a non-hydrostatic pressure state. This stress state is used by researchers when they want to induce plastic deformation or shear stresses in the system. The XRD geometry and diffraction planes observed become critical to understand. This is much easier experiment to conduct however, getting meaningful data from XRD becomes a challenge.

The requirements to conduct non-hydrostatic measurement are –

- PTM should freeze at low pressure to apply non-hydrostatic pressure state.
- Loading a lot of sample increases the non-hydrostatic pressure state.

1.9 Preparing gasket and loading DAC

Gaskets are made from sheets of materials listed in the previous section of around 250 μm thickness. The sheet is cut into squares or circles (1 cm) to cover the diamond and extend a little bit more onto the backing plate for easier placement (see the indentation video tutorial). Gasket must then be indented using the diamond anvil cell to take the shape of both the diamonds and thin down to 30-100 μm between the diamonds (depending upon pressures that one wants to reach).

Indentation

Requirements for indentation –

1) Gasket sheet – T301 SS used https://www.mcmaster.com/301-stainless-steel
2) Punch to get a ~1cm circular sheet – Amazon – https://www.amazon.com/gp/product/B00V9KAK8C/ref=ppx_yo_dt_b_asin_title_o02_s00?ie=UTF8&psc=1
3) Sharp blade to make a marking on the gasket
4) Q-tips and cleaning solvents like ethanol https://www.amazon.com/TecUnite-Pieces-Cotton-Precision-Pointed/dp/B07CWNB56X/ref=pd_nav_b2b_ab_bia_t_1?_encoding=UTF8&psc=1&refRID=DWAT0MKDRD6HYHMJ34H4
5) Clay – any modeling clay (make sure it is not soluble in solvents)
6) Hex screw set
7) Preferably stereo microscope with camera attached for distance measurements.

Fig. 1.9.1: Clay.

Procedure for indentation – Please see the video – [link]

Drilling (only for stainless steel)

Sample chamber must then be drilled into the center of the indentation. This is done on campus using the microdrill machine available in SNF (contact Elmer Enriquez). Sample chamber size should be kept between 1/2 to 1/3 of the diamond culet size however, to do XRD on campus the sample chamber must be at least 300 to 350 μm in size (to minimize gasket signal). The large gasket size introduces anisotropic stress state in the sample chamber which must be accounted for.

Requirements for drilling –

1) Flat stage (micrometer) –

https://www.amazon.com/gp/product/B012FWKRKS/ref=ppx_yo_dt_b_asin_title_o04_s00?ie=UTF8&psc=1

2) Sharp tip drill bit – PMT micro tool (as per order) –

Fig. 1.9.2: Sharp drill bit.

3) Oil
4) Acrylic flat block – https://www.amazon.com/Sunday-Int-AH08-Krystal-Acrylic/dp/B002PI8ZCU
5) Magnification eye piece – https://www.amazon.com/gp/product/B078JPRH2Y/ref=ppx_yo_dt_b_asin_title_o04_s00?ie=UTF8&psc=1
6) Only "Solid Carbide" micro drill bits – Grainger https://www.grainger.com/product/M-A-FORD-Solid-Carbide-Micro-Drill-42CT35

Procedure for drilling –

1) Indented gaskets are taped to (the bottom diamond side facing down) on the acrylic flat block.
2) Acrylic flat block is then taped to the micrometer stage
3) This is taken to the SNF microdrill and mounted
4) Sharp tip is installed in the microdrill and the center alignment is done using the side view camera and the magnification eye piece. The drill bit should look aligned from both sides.
5) The drill is switched on and then the sharp tip is used to make a small indent (20-30 um deep) in the center of the gasket. This is useful for reducing the walk of the drill bit.
6) Then the drill is replaced with the actual carbide drill bit and then the alignment is redone.
7) Some oil is dropped on the gasket and then the drill is switched on.
8) The drill is moved down in 10 μm steps till a through hole has been made.
9) Then the gasket is taken out and observed under a microscope.
10) Only gaskets with a roughly centered hole will be used further.
11) Gasket is thoroughly cleaned and sonicated for at least 10 min in acetone.
12) If there are any stuck metal pieces, then they are removed using a tweezer or needle.

Loading

Requirements for loading –

1) Dried sample on a glass slide or substrate.
2) Ruby powder (Almax-easy lab) for pressure measurement
3) Preferably stereo microscope
4) Sharp needles –

https://www.amazon.com/gp/product/B072JJ7H5P/ref=ppx_yo_dt_b_asin_title_o01_s00?ie=UTF8&psc=1

5) Anti-static gun – https://www.amazon.com/gp/product/B0033SHDSS/ref=ppx_yo_dt_b_asin_title_o05_s00?ie=UTF8&psc=1

Procedure for loading – Please see the video – [link]

1.10 Raman microscopy – checking pressure through Ruby

Ruby is used as pressure calibrant. Ruby has characteristic fluorescence when excited with <650 nm laser light. The Ruby fluorescence has peaks around ~694 nm and this peak red shifts with increasing pressure and the shift is calibrated. Online pressure calculator is a useful website to measure pressure using ruby fluorescence - http://kantor.50webs.com/ruby.htm.

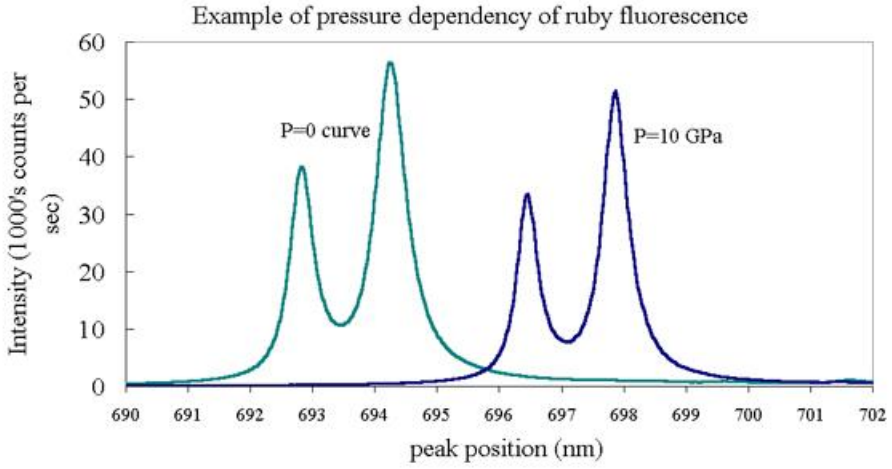

Fig. 1.10.1: Ruby fluorescence change with pressure. Image: Tomas Duffy

Ruby fluorescence can be measured using Raman system available on campus using the x10 objective with the 532 or 633 nm laser excitation. The pressure that is determined using Ruby is the effective hydrostatic pressure in the sample chamber with no orientational dependence. Different positions in the sample chamber can give different pressure values so, Ruby particles near the sample or the center of the chamber should only be used as pressure calibrant.

2 Powder x-ray diffraction using Bruker D8 Venture

More than 3,000 DAC – XRD papers have been published over the years (data from scopus.com) out of which bulk modulus calculations have been performed in over 1,200 papers and high-pressure phase transformation have been performed in over 800 papers. These two methods are the focus of this SOP however, a lot of additional studies can be performed using high pressure XRD like plastic deformation and defect density determination, microstructural changes, and high-pressure synthesis.

2.1 Setup

1) Bruker D8 venture is used to collect high pressure XRD of materials on campus.

2) Small size collimator and longer beam stop is used to fit the DAC on the goniometer.

3) Mo Kα (17 keV) is used as the x-ray source. Mo is used as it has high enough energy to penetrate the diamond and the sample to get good XRD signal.

4) The collection time is kept as 600 seconds to get a good XRD pattern.

5) Different sample to detector distance is used to improve resolution of small and large angle peaks.

Fig. 2.1.1: Bruker D8 venture setup.

6) The collected 2-D XRD pattern is circularly integrated using the Apex software available on the XRD machine to generate 1-D XRD patterns.

Users are trained by Arturas Vailionis to use the XRD machine, setup the beam stop and collimator, align the DAC with the beam, and use the Apex software to collect the XRD patterns and integrate them.

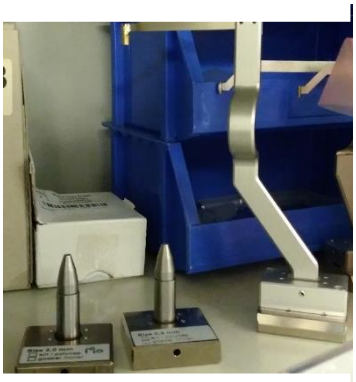

Fig. 2.1.2: Short collimator and longer beam stop.

2.2 Bulk modulus calculations

Bulk modulus determination can be done using hydrostatic compression of materials. Tracking XRD peaks with pressure and then using it to determine precise lattice parameter to give you the bulk modulus of materials. Several equations of states have been developed to study the lattice parameter change with pressure. The essential requirement of this method is to be able to track the XRD peaks with pressure.

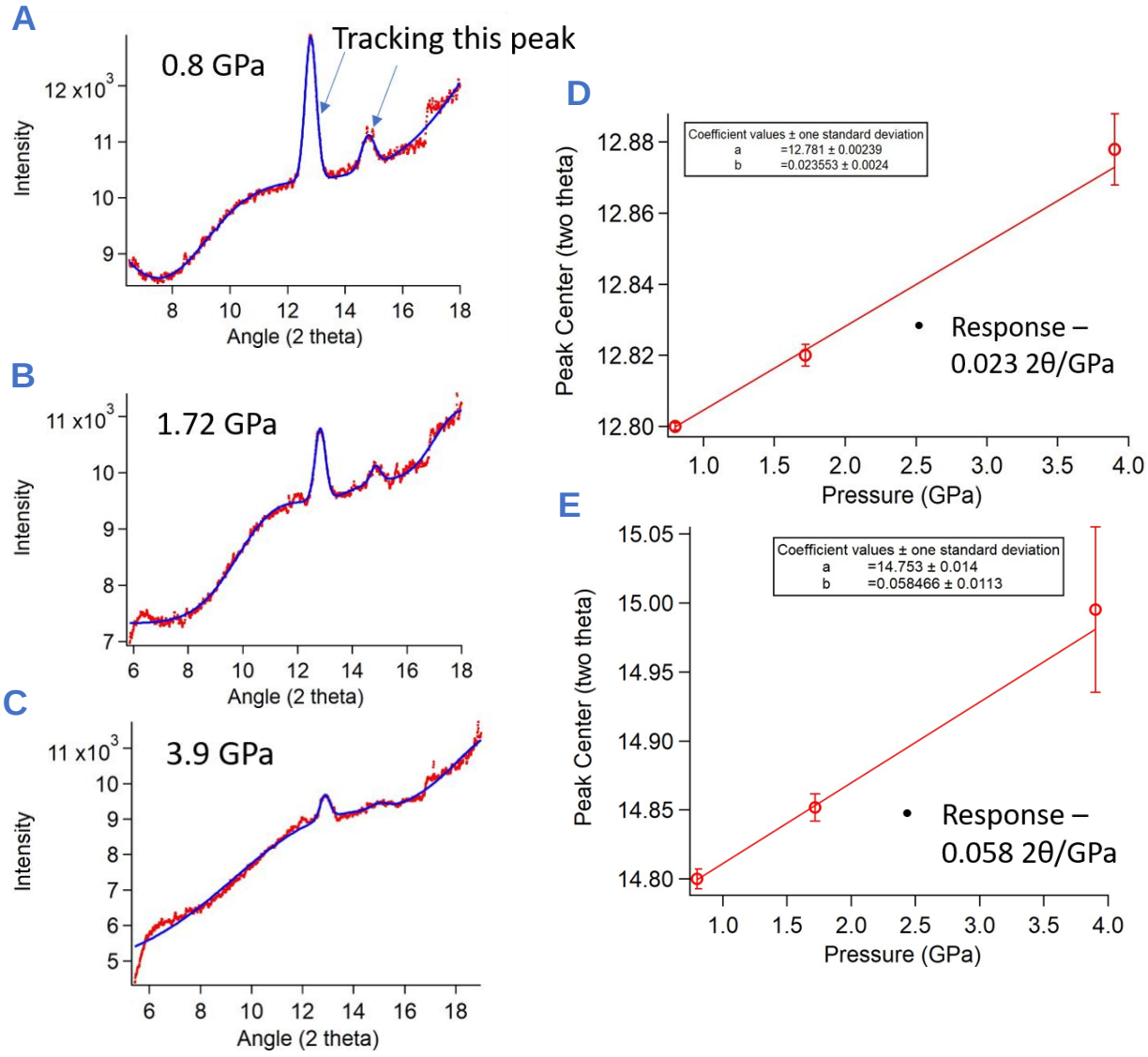

Fig. 2.2.1: Hydrostatic high pressure XRD at lower angles. A) XRD pattern at 0.8 GPa. B) XRD pattern at 1.72 GPa. C) XRD pattern at 3.9 GPa. D) Tracking first peak with pressure. E) Tracking second peak with pressure.

We tested out the material response under pressure using a nanomaterial (identity kept confidential) under pressure. We tracked the XRD peak shift with pressure. Silicon oil was used

as quasi-hydrostatic pressure medium to apply a uniform pressure on the particles. Particles were loaded into a sample chamber of 300 μm diameter in a T-301 stainless steel plate. XRD patterns were collected on the Bruker D8 machine on campus using the Mo radiation source.

2.3 Phase change studies

High pressure and temperature phase transformation studies are performed in diamond anvil cells. XRD can give you structural transformations with pressure. Complicated analysis like Rietveld analysis can be used to determine atomic positions and crystal structural information from XRD. Several interesting phases have been determined using diamond anvil cell techniques. Complete phase diagrams of several metals have been determined like Fe, Si and others. Many times, metastable phases can be formed and be recovered using high pressure techniques.

We tested out the structural transformation under high pressure using a material (identity kept confidential). We tracked the XRD peaks to observe the evolution of structural changes. Clear formation of new XRD peaks show that the structure had transformed under pressure. 4:1 Methanol:Ethanol mixture was used as quasi-hydrostatic pressure medium to apply a uniform pressure on the particles. Particles were loaded into a sample chamber of 300 μm diameter in a T-301 stainless steel plate. XRD patterns were collected on the Bruker D8 machine on campus using the Mo radiation source.

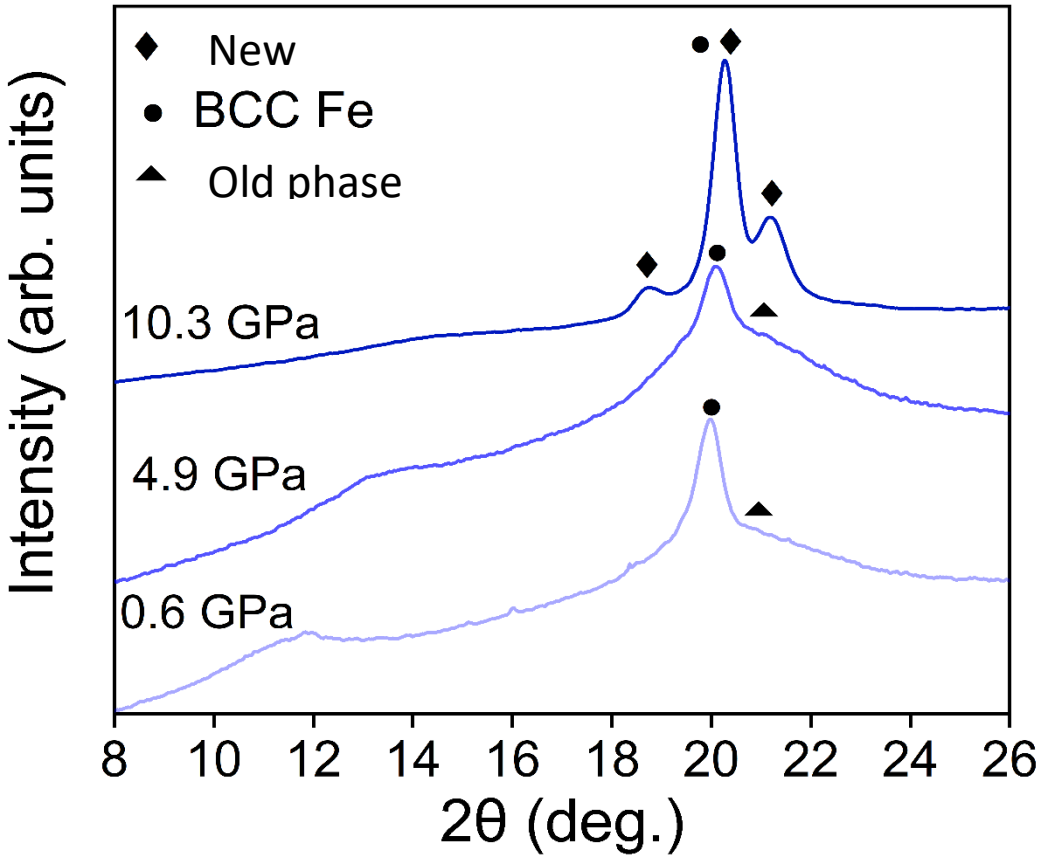

Fig. 2.2.2: Hydrostatic high pressure XRD at higher angles. Phase transformation with pressure is clearly visible. BCC Fe peak is from the gasket.

3 Frequently Asked Questions

Q1.How to determine the maximum pressure that we can go to?

This is critical to determine as when you exceed the maximum pressure the diamonds will break. The maximum pressure is dependent upon the gasket thickness, the culet diameter, the sample chamber size and position, and gasket material. Never use the manufacturer maximum pressure limit as they report that for the most ideal conditions so, the maximum pressure limit is different for each experiment. Two easy ways to stop pressurization before reaching the maximum pressure limit is – tracking the gasket hole deformation, and pressure increase with each incremental screw tightening. Former is covered in the next question. For the latter, pressure increase is exponential and with smaller and smaller incremental turns the pressure shoots up higher and higher. When with an incremental turn the pressure doesn't increase or doesn't increase as much as expected then you must stop. The diamonds have reached the maximum pressure limit.

Q2.What if the gasket hole deforms?

Gasket hole must deform under pressure. Deformation of the gasket hole is dependent upon the thickness of the gasket, sample chamber size and position. Initially, the sample chamber shrinks in size upon pressurization if this initial decrease in size is large (>50 um) then stop as you may not have any pressure medium and the force is directly impacted on gasket. If the decrease is small, then you can proceed forward. With increasing pressure, the deviatoric stresses increase in the sample chamber and you can observe that the gasket hole becomes an ellipse. If the gasket hole increases in size too rapidly or if the hole is about to reach the edge of the diamond culet then you must stop as this the maximum limit of pressure your gasket can handle.

Q3.Are diamonds forever?

It is very easy to break diamonds! and very expensive. Diamonds can break when the maximum shear stresses exceed a limit or when diamonds touch each other. It is easy to break diamond with another diamond. One must take extreme care when bringing diamonds in contact like when gluing or aligning the diamonds. Even if the diamonds touch with the slightest force the diamonds shatter. Small scuffs on the surface is still okay to be used for high pressure experiments but a crack that goes through or is internal then the diamonds will break when pressurized.

Q4.Do diamonds deform?

Diamonds deform at high pressures. Culets deform and form cups, it has been widely studied and reported (*9*). This generally happens at very high pressures > 50 GPa and care must be taken when pressurizing samples at such high pressures.

Q5.Any other pressure calibrants?

Ruby is the most widely used pressure calibrant. However, several XRD based pressure calibrants are also used. In this case a small chip of a metal like Pt or Au is added in the

sample chamber which has a well-known equation of state. XRD peaks from these pressure calibrants interfere with the sample peaks however, they can be more accurate measure of pressure from the sample chamber.

Q6. How to control deviatoric stresses in the sample chamber?

Deviatoric or nonhydrostatic pressures are always present in the sample chamber. However, if one wants to perform ideal hydrostatic experiments then they need to minimize the deviatoric stresses. This can be done by first, positioning the sample chamber at the center of the diamond culet. Second, making smaller sample chambers as the pressure is maximum at the center and decreases with distance from the center. Thirdly, right choice of PTM which is compatible with the particles (dissolves them as we want each particle to be surrounded by PTM) and doesn't solidifies under pressure.

Q7. How to combine high pressure with other measurements like electrical or high/low temperature?

DAC can be combined with other measurement techniques like electrical and high/low temperature. Electrical measurements require electrodes to be put inside the sample chamber and must not come in contact under pressure. Reference (*10*) details the method for conducting electrical measurements.

High temperature measurements can be done by using resistive heating to heat the whole DAC. A thermocouple is placed inside the sample chamber or in contact with the diamonds. Reference (*11*) details the method for resistive heating of DAC.

Low temperature measurements can be done by using the cryo-stream in the Bruker D8 venture machine and temperature can be monitored using a thermocouple.

Q8. Is gas loading possible on campus?

Gas loading generally requires high pressure gas to fill a sealed thick-walled vessel where DAC is placed and then sealed inside once the gas fills the sample chamber. However, gas loading can be done using liquid nitrogen. In this method the DAC is assembled with a small gap between the gasket and the top diamond. Then the DAC is dipped inside a vessel filled with liquid nitrogen. Liquid nitrogen fills the DAC and the sample chamber. Then the DAC is completely sealed while inside the liquid nitrogen. Special care must be taken while handling liquid nitrogen like wearing appropriate personal protective equipment and the glue that holds diamonds in place should be safe at cryogenic temperatures. Additional techniques detailed in Reference (*12*, *13*) have been developed to produce liquid argon and load in the DAC.

4 References

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