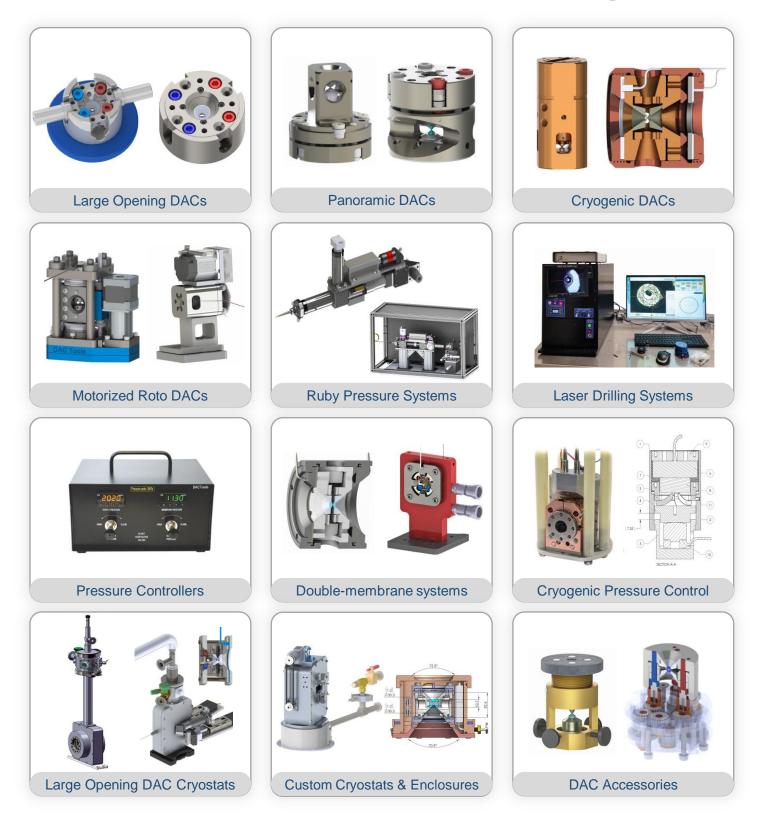




# **DAC Tools LLC – Product Categories**





# About DAC Tools

DAC Tools LLC is an engineering company which specializes in design and manufacturing of state of the art conventional, customized, and highly specialized equipment for research at extreme conditions (high pressure at high and cryogenic temperatures), preliminary with diamond anvil cells. Located in Chicago area, DAC Tools is the only company in the USA which designs and manufactures such equipment.

DAC Tools was started in 2009 by Stas Sinogeikin who has more than 30 years of experience in high pressure scientific research and instrument development in the major US University (UIUC) and DOE National Laboratory environment (APS, ANL). It started a sole proprietorship side business aimed to provide researchers in High-pressure field with custom equipment for high pressure high/low temperatures not available elsewhere. Over the years we have provided US National labs and multiple Universities in USA, as well as research groups all over the World, with conventional and specialized custom Diamond Anvil Cells and supporting equipment for controlling and measuring pressure and temperature.

Currently DAC Tools is a full-time fast growing company both in size and product line, which provides complete range of products for high pressure research, including unique custom and customized state-of-the-art equipment designed and optimized for specific experiments and constrained environments.

Main product categories include, but are not limited, to the following:

- Complete Diamond Anvil Cells (DACs) for different experimental techniques and broad range of pressure-temperature conditions;
  - Regular steel DACs for X-ray diffraction, optical and X-ray spectroscopy, other experiments;
  - o Beryllium-coper non-magnetic DACs for various experiments at cryogenic temperatures;
  - o Inconel 718 high-temperature spectroscopy and x-ray diffraction DACs;
  - Motorized rotational Shear and Tomography DACs;
- Uni- and bi-directional (compression / decompression) static and dynamic pressure pneumatic and electro-mechanical pressure control systems for various DACs;
- Portable / online and stationary optical (e.g. Ruby Fluorescence) systems for pressure measurements;
- Laser Drilling / Micromachining Systems for sample and sample environment / gasket preparation;
- Custom HP cryostats with remote bi-directional pressure control for optical and X-ray spectroscopy and X-ray diffraction;
- Resistive heaters and protective enclosures for DACs;
- Tools and accessories for Diamond Anvil Cell work;

Contact us to find out how we can apply our experience to your routine or unique situation.





# LABORATORY TABLE-TOP LASER DRILLING AND MICROMACHINING SYSTEM

#### Models LDMS-23FS / LDMS-23SM

The laser drilling / micromachining system is used for drilling / micromachining holes in gasket material and

preparing samples and sample environments for Diamond Anvil Cell and other experiments. The system is based on a pulsed laser with <500 ps pulse duration, maximum pulse energy of ~80 microjoules up to 1,000 Hz (option to 5,000 Hz for heavier and cleaner micromachining). Short laser pulse duration allows ablating materials without thermal melting, thus leaving a clean edge after drilling (with proper post-processing). With optics designed for a tight focus (down to 7-8 micrometers), the machine can drill holes down to ~8 micrometers in diameter and larger (in thin gaskets). The system can be provided with either fixed magnification 10x camera or with optional 12x zoom Navitar microscope for better flexibility and magnification range.



Figure 1. LDMS-23FS system components: A - laser machining system enclosure / main unit; B – All-in-One control computer; C - low magnification overview monitor; D - laser power supply.

The system is provided with an environmental chamber allowing drilling / micromachining operations in active flow or sealed inert gas atmosphere, or in vacuum. The

laser drilling / micromachining operations in deuter now laser drilling / micromachining system allows drilling nonconductive materials such as amorphous boron and silicon carbide gaskets, diamond, oxides and many other materials including organic materials (e.g. kapton). The machine can also be used for sample cutting and for making engineered samples and sample environments (e.g. insulation layers for laser heating). The system is easy to operate, fully remote-controlled, enclosed as a Class-I laser for safe operation, and has a precise fully motorized sample alignment and positioning system with user friendly interface.

Figure 2. LDMS-23SM system in compact frame.

LDMS-23FS (easier to upgrade) and compact LDMS-23SM (takes less desk space). In basic configuration the performance of these systems is identical.

• User friendly Class 1 laser system

The system is available in two frame sizes - full sized

- Sturdy and low maintenance, very easy to operate
- Sub-micron fully motorized sample position and trajectory control
- Versatile can micro-machine any material
- Fast drilling of an average gasket hole typically takes about a minute or less
- Environment control chambers (sealed and active flow operations)
- Short pulse ablates, does not melt
- Very large dynamic range of laser power and laser frequency





# SPECIFICATIONS OF THE LASER DRILLING AND MICRO MACHINING SYSTEM

Models LDMS-23FS / LDMS-23SM

Laser

#### **Main System specifications**

Size (W-D-H, approx.):	~61 x 46 x 60 cm (FS) ~31 x 61 x 48 cm (SM)	Manufacturer: Model:	Teem Photonics (France) PNP-M08010-120
Weight (approx.):	~73 / 68 kg (FS/SM)		PNP-M05550-120 (opt.)
Frame:	~25 mm anodized	Wavelength:	1064 nm
	aluminum	Energy per pulse:	Lμ 08<
Protective Panels:	~3 mm thick black	Average power:	>80 mW (>400 mW opt.)
	anodized aluminum	Repetition rate:	1 - 1000 Hz (5,000 Hz opt.)
Sample compartment:	Motorized IN/OUT	Pulse length:	<500 ps
Number of cameras:	( 0	Peak power:	>160 kW (>800 kW opt.)
	12x zoom optional.)	Short-term stability:	< 1%
Operating voltage:	110-250 V, 50-60 Hz	Long-term stability:	3%
Additional equipment	- Laser power supply	Beam profile:	Gaussian TEM00
(included):	<ul> <li>Control computer</li> <li>Low-magnification</li> </ul>	Beam divergence:	$2.0\pm0.5$ mrad
	overview monitor	Beam ellipticity:	<1.3
Positioning stages		Beam focus:	<10 µm (7-8 typ.)
		Objective lens:	Mitutoyo 10x PlanAPO NIR
X-Y-Z stages:	Newport MFA stages	Objective WD:	30.5 mm
Travel range:	25x25 mm (MFA)	Operating	
Minimum step:	~300 nm (0.3 μm)	temperature:	20-35 °C
Driver/controller:	AllMotion EZ4AXIS	Maximum power	
Control Software:	DAC Tools in-house	consumption:	<120 W
	custom software	Warranty:	1 year

Note: The exact specifications and dimensions are subject to change. Possible changes are aimed to optimize and improve the system performance.

WARRANTY – This product is warranted to be free of defects in material and workmanship for one (1) year from date of purchase. This warranty is limited to the correction of any such defect, or the replacement of any such defective item, provided that: (a) item was/were purchased from DAC Tools LLC; (b) we are properly notified and consent to the return of the item(s) in question; (c) the item(s) is(are) returned with proof of purchase date; and (d) it is found upon inspection by us that the item(s) is(are) defective as noted above. This warranty does not cover labor costs, consequential damages, nor does it apply to any item(s) that have been improperly installed, overloaded, altered, or otherwise abused by the customer, its agent(s) or employee(s).





# LARGE OPENING DIAMOND ANVIL CELLS

DACTools manufactures a variety of Diamond Anvil Cells optimized for different high-pressure experiments. While "Standard" Symmetric DAC remains popular and we do supply different versions of this DAC (i.e. Imperial 1.875", Metric 48 mm, Metric Mini 40 mm, regular and shortened), there are several modifications of larger openings symmetric DACs which are gaining popularity.

The Spherical Seat DACs (SSDAC) with 70 and 80 degrees real symmetric X-ray opening were specifically designed for single crystal x-ray diffraction at synchrotron facilities and on a laboratory-based (i.e. Rigaku) diffractometer and



for use with Boehler-Almax type conical seats where diamond tilt alignment can be critical. The design allows the cell to be used with two membranes (compression and decompression) and still keep up to 70-80 degrees real symmetric x-ray opening. SSDAC-70 has ample space inside for small resistive heaters.



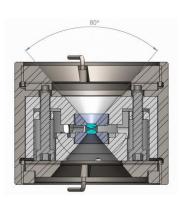
SSDAC-70/80 is a single crystal diffraction DAC, but it can be used with a variety of experimental techniques where large symmetric opening is required such as X-ray total scattering measurements, Brillouin and other optical spectroscopies as well as powder x-ray diffraction. If 80 degrees symmetric opening is not absolutely crucial, a longer (and thus a bit more stable) 70 degrees configuration (SSDAC-70) is preferrable, especially for Megabar range.

The version of such DAC without diamond tilt control is called iBX-70/80 ("imperial BX" DAC) and is an optimized upgraded version of BX-90 making the DAC more practical and functional compared to the original BX-90 DAC. The optimized 50 mm metric version (mBX-60/70/80) is under active development.

SSDAC-70/80 and iBX70/80 have the same diameter (although slightly different height) and hole pattern and are compatible with equipment designed for this DAC family (DAC holders, single and double membrane cans and pressurization frames, gearboxes for gas-loading systems, etc.)

With proper diamond culet size (<250  $\mu$ m), diamond alignment, and sample preparation the iBX and SSDAC cells can be routinely used in sub-Megabar and Megabar pressure range.

All above mentioned DACs allow for multiple ways of pressure control – either with screws, mechanical gearboxes, or with membranes (see *e.g. Sinogeikin et al., Rev. Sci. Instruments 86, 072209, 2015*). The DACs can be preloaded to starting pressure with four screws and then engaged with remote



pressure control devices. The DACs can be supplied as stand-alone versions, with a single membrane canister, or in double-membrane symmetric configuration which provides up to 80 degrees real symmetric X-ray and optical opening with proper choice of diamond anvils, diamond seats and membranes.

A new addition to large opening / single crystal DACs is Mini-BX80 Diamond Anvil Cell. This is a modification of a compact (39mm OD) *Mini-BX90 DAC* optimized for better stability, convenience, and versatility: it

> can accept a variety of diamond seat combinations while remaining very compact and stable, can be easily integrated with remote membrane pressure control, and can be used in exotic sample environments such as a compact cryostat for double-sided laser heating.

> All these DACs can be supplied with pins for mounting on conventional goniometers and other custom furnishings for easier DAC handling.







# PANORAMIC DIAMOND ANVIL CELLS

#### Two-fold Panoramic DAC for Inelastic Scattering, Radial Diffraction, and Tomography

Old Style Two-fold panoramic DACs provide 140 degrees symmetric radial opening in horizontal direction and up to 68 degrees opening in vertical direction. In many experiments they are still a primary choice for radial x-ray diffraction measurements, X-ray tomography, inelastic X-ray scattering measurements with or without polycapillary optics, NRIXS measurement with large APD detectors, and other techniques requiring large angle panoramic view in radial geometry and easy access to the sample. The DACs can be integrated with compression and / or decompression membrane systems.



#### Compact DACs for Inelastic scattering, Radial diffraction, Tomography, and Time-resolved experiments

DACTools offers a family of compact panoramic DAC, both 2-fold and 4-fold, which provide up to 90



degrees radial opening (both vertical and horizontal) and can be used for a variety of techniques such as radial diffraction, X-ray tomography, X-ray inelastic scattering, and other techniques, including non-X-ray ones. The DAC can be easily combined with a compression / decompression membrane drive or a piezoelectric drive in either compression or decompression mode and used for x-ray diffraction measurements in radial geometry in static or dynamic (e.g. cyclic pressure variation) regime.

#### Wide opening (150°) Diamond Anvil Cell for Tomography and Radial diffraction

TOMO150 compact panoramic 2-fold Diamond Anvil Cell was primarily designed for x-ray tomography but is widely used for a variety of other experiments. It provides 150 degrees of unobstructed symmetric radial opening in horizontal direction and up to 50 degrees in vertical direction (depending on the diamond + seat configuration). It has the same diameter and screw pattern as the popular GL-CIW Standard Symmetric DAC which makes integration of

this DAC into experimental infrastructure (DAC holders, gearboxes, membrane systems, etc.) for Symmetric DACs relatively easy and straightforward. The DAC is suitable for multiple experimental techniques such as X-ray diffraction



in radial and axial scattering geometry, inelastic X-ray scattering, X-ray tomography, as well as optical measurements such as Raman spectroscopy, among others. Properly prepared DACs can readily reach and exceed megabar pressures. If hydrostatic conditions are required – the DAC can be loaded with inert gases as pressure medium in GSECARS / APS type and compatible gas loading systems.







# LARGE OPENING CRYOGENIC BeCu DACs

A large number of physical phenomena, such as superconductivity, magnetic ordering, quantum critical phenomena, and others often appear only at extremely low cryogenic temperatures (e.g. below 5-10 K). Therefore there is a lot of interest in studying various materials at simultaneously low temperature and high pressure by different experimental methods and thus a significant demand for a variety of different diamond cells designed for low temperature studies. While DACs made of hard steel (SS 440C, Vascomax C300/350, etc.) can be used in low temperature experiments, especially in "wet" cryostats, the material of choice for cryogenic DACs is Beryllium Copper alloy (e.g. C17200 / Alloy 25) which has a unique combination of properties: non-magnetic, high strength, high thermal conductivity.

Currently our most popular BeCu DAC is Shortened Symmetric DAC. It is based on a Metric Symmetric DAC (48 mm OD) but with cylinder shortened by 5 mm to decrease working distance (for e.g. Raman spectroscopy) to ~12 mm and reduce thermal mass. The DAC can be easily integrated into a number of cold-finger cryostat and has built-in features such as socket for mounting temperature sensor in CU package, holes and threads for bolting the DAC to cold finger base, and so on.



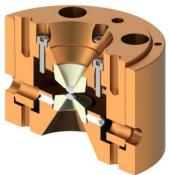
Another modification of the Standard Symmetric DAC is a Mini-Symmetric DAC with 40 mm Diameter. This DAC has smaller outside dimensions and is ~50% lighter than the full sized BeCu DAC. Nevertheless, the inner diameter and height of the diamond / seat area is the same and allows using "standard" sized diamond anvils and seats.

The DAC can be easily integrated with single or double membrane BeCu canister (compression and decompression) and fit into 47 mm (all versions) and <59 mm (shortened version) cryostat bores along and normal to the bore axis respectively.

Another large opening cryogenic DAC recently developed by DACTools has 70 degrees symmetric opening and diamond tilt mechanism so that the DAC can be easily used with Boehler-Almax type diamond anvils and seats. The DAC was initially developed as a part of cryogenic system for single crystal Brillouin scattering (can continuously rotate about the DAC axis inside the cryostat) but can be used for a variety of other studies such as single crystal x-ray diffraction.

Virtually any type of DACTools' DACs (e.g. split-style SSDAC 70/80 and Mini-BX80, BX-60/70/80, or custom DACs) can be made of BeCu alloy providing that there is sufficient demand (e.g. 4-5 DACS) to justify production.

If the DACs are intended to be used in strong magnetic fields, they can be supplied with non-magnetic screws (e.g. BeCu, Inconel 718, Titanium5) and spring washers (Inconel 718) or split cylinder springs (BeCu), as well as nonmagnetic diamond seats made of either WC with Ni binder, BeCu, Pascalloy (non-magnetic NiCrAl alloy) or cemented cubic boron nitride.



To minimize tear and wear and improve the performance, stability and lifetime of BeCu DACs the pistons can be coated with Hard Chrome (magnetic) or the whole DAC can be coated with a layer of WS<sub>2</sub>.

The variety of BeCu DACs and special diamond seats stocked by DACTools is constantly increasing – please contact us to check for available new models and lead times.

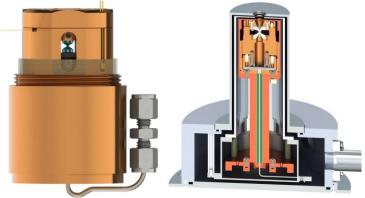




# PANORAMIC AND SPECIALIZED BeCu DACS

While large opening DACs can be used for a variety of experiments – X-ray scattering, optical spectroscopy, electric measurements and such, often they are not suitable for specific environments either because of limited geometry / opening or large size not appropriate for certain types of cryostats or experimental setups and conditions. Below are few examples of such DACs which have been designed and manufactured by DACTools.

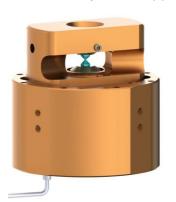
The compact (38 mm OD, ~56 mm long) BeCu DAC with diamond tilt alignment rocker and integrated membrane pressure control was designed as nonmagnetic DAC for use with Janis ST-500 or Physike Scryo-S500 cryostats and strong magnetic fields (superconducting magnets with small bores) for Raman and other spectroscopy measurements requiring short working distance (WD <= 12mm). The current version was designed for use with "blind"



membrane thus the sample can be accessed optically from only top / cylinder side.

The DAC was initially designed to have an integrated membrane pressure control. Nevertheless it can be loaded and operated independently of the membrane. The DAC can be closed and pressure can be controlled with 4x M4 screws and a set of Belleville spring washers.

The Beryllium Copper version of DACTools TOMO150 DAC can be used in a variety of primarily x-

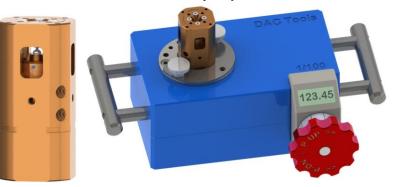


ray based experiments were large (150 degrees) opening in the sample plane (perpendicular to DAC axis) is essential. It can be used in different X-ray spectroscopy / inelastic scattering measurements where the x-ray beam can be focused on the sample either through the diamond anvil or through the gasket, and the signal can be collected typically from a side through the x-ray transparent gasket. The DAC can also be used in a regular axial geometry although the angular opening is limited. The DAC can be easily integrated with a special membrane drive and radiation shield for improved temperatures.

The non-magnetic DT-25/28-55-BeCu DAC is designed for a variety of experiments at cryogenic conditions in confined spaces (28 mm min. diameter

of the cryostat bore). The DAC is comparable with HMD PPMS 25 mm DAC (DAC-SRr-25-55) while has a lot of modifications to improve performance, stability, reliability, and ease of operations. The DAC has a rocker / spherical seat so that the tilt of one diamond anvil can be easily adjusted.

The pressure in the DAC is increased by rotating one pressurizing screw (1 mm pitch) with integrated ceramic thrust ball bearings. The pressure in the DAC is typically increased with an optional 100X gearbox (which can provide piston / pressurizing screw advancement with 100 nm = 0.1 um resolution), although other options including membranes) are possible.







# ROTATIONAL / TORSIONAL DIAMOND CELLS FOR AXIAL X-RAY DIFFRACTION

There is a rapidly growing interest in studying materials under high pressure and severe deformation conditions / shear strain. The ability to generate large plastic shear under high pressure and study the process with in-situ x ray diffraction provides means to study a very large array of related phenomena, such as rheological properties of materials in the Deep Earth, dynamic microstructural evolution during extreme shear deformation for manufacturing new alloys and material with unique properties, pressure reduction in shear-induced phase transitions and formation of new phases, pressure self-focusing effect and others.

Therefore one of the ongoing developments of DACTools are custom dynamic rotational / torsional

(shear) Diamond Anvil Cells (DACs) for axial x-ray diffraction measurements. The project was originated by PNNL to study the behavior of materials under fast shear strain with relative diamond rotation rates up to 1000 RPM and now the development continues to expand the velocity range of the DAC (e.g. from 0.001 to 4,000 RPM) and add high temperature capability.

Initially the DAC was designed in such a way that it could operate in two modes – fast (> 10-100

RPM) and slow (< 10-100 RPM) where fast mode requires a precision bearing of other frictionless mechanism to keep the alignment of the piston during fast rotation and in slow mode the piston is rotated within a split cylinder / clamp with adjustable tension attached to the body of the DAC.

The specifics of the design is that the diamond anvils are not glued to the WC seats but are rather pressed into square shaped bronze holders to prevent diamond rotation and detachment from the seats. Also, an integral part of the design is presence of diamond alignment rockers / hemispheres on both sides of the DAC so that the tilt of both diamonds can be adjusted to make sure that the diamond culets are always parallel during the rotation, which is extremely important at fast rotation speeds.



Over the last few years DACTools have made a lot of modifications to the original design aimed to improve the stability, performance, and ease of operation. In addition to creating a set of alignment tools (laser stand, microscope, etc.) and modifying the DAC body for easier alignment



and handling, we also have redesigned the adjustable piston clamp, changed original gear drive with belt drive, added high-temperature capability (to 300°C so far), added possibility of using speed reducing gearbox for continuous rotation down 0.001 RPM, and created software for integrating / synchronizing the system with synchrotron beamlines. Currently we are working on improving the alignment and stability of the system at high rotational speeds (e.g. 4,000 RPM) by testing frictionless piston / cylinder combinations such as air bearings.





## **MOTORIZED ROTARY DIAMOND ANVIL CELL (RotoTomo-DAC)**

Rotary Diamond Anvil Cell (RotoTomo-DAC) is designed for X-ray tomography, radial diffraction, and (high energy) 3D X-ray microscopy in radial geometry. The Cell provides about 70 degrees of x-ray opening in horizontal plane (determined by the position of the force posts) but can be increased to 80-90 degrees if required.

The Roto-DAC can operate in two modes: panoramic with simultaneous rotation of both pistons (and anvils), and shear mode where only one piston / anvil is rotating.

In synchronous rotation / panoramic mode two coaxial pistons rotate within a single cylinder; thus both diamonds with the sample rotate simultaneously over the 360° angular range. The cylinder has split nature and the tightness can be adjusted using screws with spring washers.





In shear mode one

piston / diamond can be locked in place and the other can rotate, causing the sample to shear between diamond culets in circular fashion.

The Roto-DAC is driven by a gear motor which rotates the pistons through a series of spur gears. There are two built in gear combinations in the gearbox: single with force amplification / speed reduction factor of 2.8 and double with force amplification / speed reduction factor of 7.84.

The Roto-DAC is typically driven by a stepper motor (either NEMA 11 or NEMA 17) with built-in planetary gearboxes. The gearmotor of proper size and gear ratio can be selected to provide the required rotational speed and torque.

The gear motor can be driven by the computer-controlled motor driver, manufactured and supplied by DACTools and

operated through its dedicated software.

The pressure in the DAC can be controlled either manually (with pressure screws) or remotely with a standard double-diaphragm membrane.

Typically the pistons and the split cylinder, as well as the force frame, are made of hardened stainless steel SS440C. To reduce the weight of the device for synchrotron applications the frame can be made of strong but light Titanium 5 alloy.







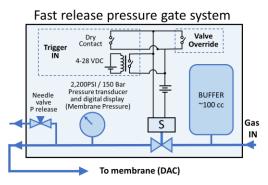
# MANUAL HIGH-RESOLUTION DAC MEMBRANE PRESSURE CONTROLLER

While computerized automatic pressure controllers (i.e. GE PACE 5000) become progressively more popular, the Manual high-resolution DAC Membrane pressure controller remains in high demand because of its simplicity, reliability, and reasonable price. The main specifications of the controller include:

- Pressure rating 3,000 psi / 200 bar with resolution of 1 psi (0.07 bar).
- Fine needle valves for increasing and decreasing pressure.
- <sup>9</sup> Built-in buffer (~100 cm<sup>3</sup>) for high and low temperature experiments.
- Digital readout of supply and output pressure, can display in PSI or Bars.
- Remotely controlled version is under development.
- The membrane fine pressure controller requires a coarse pressure regulator which is attached to a gas tank. Can be provided for domestic (USA) use.

# PRESSURE GATE SYSTEM FOR FAST PRESSURE CHANGE IN MEMBRANE DAC

Pressure gate system is designed for fast (ms level) pressure change (increase or decrease) in



membrane-driven DACs for time-resolved studies of the effect of fast compression and decompression on materials properties and phase transitions. The system features ~100cc gas buffer, manual valve for controlling pressure in the buffer, and electric solenoid valve which can be synchronized with other equipment. Although membrane-based pressure jump can be a little slower than that generated by a piezo transducer, membrane system is capable of generating significantly larger pressure changes due to larger force and displacement.

## **MEMBRANES**

DACTools provides a variety of different laser welded membranes made of 304 and 316 (less magnetic) stainless steel, as well as non-magnetic Inconel 600/625 membranes. While the most common membrane diameter is 2.0 inch (50.8 mm) for regular DACs, DACTools can manufacture any custom-sized membranes of reasonable diameter (e.g. from 30 to 100 mm, with or without central opening). We also provide composite membranes with steel frame as replacement membranes for LLNL-type and Diacell-type DACs and multi-convolution bellows for specific applications.

We can also design a custom lever amplified membrane / bellows compression system which allows generating significant forces on the diamond anvils in constrained space (e.g. small cryostat bores).









# **MEMBRANE PRESSURE CONTROL IN DACS**

DACs with integrated membranes are popular in Europe, and while we do offer some DACs with

integrated membranes, DACTools' specialty are single and double membrane canisters and tie-rod systems for various popular and exotic Diamond Anvil Cells made by DACTools and other companies. The canisters and tie rod systems provide required x-ray and optical opening and flexibility in setting up a desired pressure control configurations, for example a compression membrane drive combined with a decompression piezo drive for controlling pressure increase / decrease rate in time-resolved experiments, or pressurizing / depressurizing frames for cryostats.

Typically the canisters are made from 440C stainless steel or Beryllium Copper (for cryogenic applications) heat treated to a maximum yield strength.

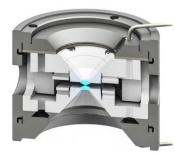
Most canisters are designed to work with 2.0" (50.8 mm) OD and either 0.94" or 1.20" ID membranes, although canisters for custom DAC and membrane sizes (both Imperial and Metric) are often designed and manufactured on request.

While most of membrane canisters are used for pressure increase, for specific cases decompression canisters and tie-rod systems can be designed and manufactured for a majority of Diamond Cell types.





## **DOUBLE MEMBRANE PRESSURE CONTROL IN DACS**



In many cases a single compression membrane is sufficient for highpressure experiments in compression mode. Nevertheless, controlled decompression of DAC with a single membrane can be very challenging. Also accurate control of sample pressure at cryogenic conditions or in resistively heated DAC with just a single compression membrane is often close to impossible. Therefore the addition of a second (decompression) membrane can often be the only way to reach desired experimental conditions both at room and low/high temperatures. Double membrane systems provide significant flexibility in pressure paths by allowing multiple compression and decompression cycles

with controlled amplitude.

DACTools offers a variety of double membrane systems for accurate pressure control. We offer decompression attachments for various DACs with integrated membranes as well as "DAC + compression canister" combinations. For many DAC types DACTools offers combined single-canister double membrane compression / decompression devices. For special cases, such as cryostats and whole-cell resistive heaters, we offer customizable tie-rod double membrane systems where both membranes can be positioned either on the opposite sides of a DAC, or on the same side for special cases requiring short working distance such as Raman spectroscopy or IR laser heating. In most cases such systems preserve the optical / X-ray opening of the DAC at least on one side, or both with proper/optimized DACs.







# **PORTABLE / LAB RUBY PRESSURE MEASUREMENT SYSTEM**

The portable ruby pressure measurement system is designed for online Ruby fluorescence pressure measurements in various Diamond Anvil Cells in the pressure range from ambient to sub-megabar at various temperatures (from cryogenic to 700K). The system can be provided in a custom configuration tailored for a particular application, space constraints, and/or experimental requirements. The system can be provided with a laser enclosure, a mounting stand and X-Y-Z manual sample stage, effectively turning the Portable system into an advanced Laboratory system with 12X zoom capability.

#### Specifications of the Portable / Lab Ruby pressure measurement system

The Portable (online) Ruby system is based on Navitar 12X Motorized Ultra-zoom infinity corrected microscope, DPSS 532nm laser with adjustable intensity and >300 mW of maximum power, and fiber coupled Ocean Optics spectrometer (typically HR2000+ or a newer HR-4VIS spectometer, or high-sensitivity low background Peltier-cooled Ocean Optics QE-Pro spectrometer for more demanding applications).

The Ruby signal is delivered to the Ocean Optics spectrometer via a multi-mode optical fiber. DACTools typically provides 105 µm Ultra and 200 µm diameter patch optical fibers. They can be easily conf replaced by fibers with other diameters for better throughput / resolution.



Figure 1. Portable Navitar 12X Ultrazoom Ruby system in 90 degrees configuration.

The custom DAC Tools software for measuring / automatic fitting and tracking / recording the Ruby spectrum is provided with the system. We also offer a Windows PC all-in-one computer or a laptop with preinstalled software for sample observation (through a 3.2 MP Flir Chameleon-3 digital camera) and Ruby spectra processing software.

The system is infinity corrected. It is supplied either with Mitutoyo LWD 5X objective or Mitutoyo compact objectives with working distance of ~70 mm (2X) OR ~60 mm (3X). The working distance of the system can be easily extended to 100-200 mm using achromatic lenses.

The zoom ratio is controlled with a dedicated DACTools Zoom controller through DACTools computer software (an additional manual zoom control is optional). With 5X Mitutoyo objective the vertical field of view on the monitors ranges from ~180 µm to ~2.0 mm.

In standard configuration the system is equipped with Flir Chameleon-3 3.2 MP digital camera with DACTools camera software. The digital camera can be replaced with HR analog video camera.

The system can be provided with an optional "docking station" so it can be used as a compact laboratory system when not needed online.



Figure 2. Ruby system with laser protective enclosure (doors not shown) and manual translation stages.

Lab version of the system is provided with the XYZ sample positioning stage stack and sample / DAC holder. The stack is based on Newport high precision manual stages. The system support is designed to accommodate the height and position of the bare "Standard Symmetric" or other DAC and can be easily customized. The manual XYZ stack can be replaced with a motorized version.

The Lab version of the system is provided with 12" x 30" (30 x 90 cm) or comparable mounting breadboard with cover / protective enclosure ( $\sim$ 12" x 32" x 16" = 31 x 81 x 41 cm).





# STATIONARY / LAB RUBY PRESSURE MEASUREMENT SYSTEM

The Lab ruby pressure measurement system is designed for offline Ruby fluorescence pressure measurements in various Diamond Anvil Cells in the pressure range from ambient to sub-megabar. It is a fixed magnification system with typical magnification of 5X-10X. Depending on specific demand, the system can be provided in a custom configuration, and various elements can be replaced / upgraded both before ordering and after delivery with special arrangement.

### Specifications of the Lab Ruby pressure measurement system

The Ruby system is powered by a green Lasermate DPSS 532 nm laser with adjustable intensity and >300 mW maximum power. The ruby spectra are collected with Ocean Optics HR2000+ or a newer HR-4VIS spectrometer, which can be upgraded with thermoelectrically cooled QEpro spectrometer for more demanding applications.



The Ruby signal is delivered to the Ocean Optics spectrometer via a multi-mode optical

fiber. DACTOOLS will provide 105 µm and 200 µm patch optical fibers. They can easily be replaced by fibers with different diameter for better throughput or resolution.

The spectrometer is controlled by a custom open access DACTools software with automatic ruby spectrum fitting and pressure tracking function. The software allows using the system in time-resolved mode with burst data collection at a rate up to 200 spectra per second.

The system is infinity corrected. It is typically supplied with 10x Mitutoyo objective lens with ~34 mm working distance. The objective can be replaced with other objective lenses if higher / lower magnification is required.

The system frame is modular and is based on Thorlabs (as well as custom) opto-mechanical parts. Thus the system can be relatively easily modified / upgraded should the need arise.

The XYZ sample positioning stage stack and sample / DAC holder are provided. The stack is typically based on Newport high precision manual stages. Motorizes stages can be provided on demand. The microscope support will be custom designed to accommodate the height and position of the typical sample / DAC.

The illumination system consists of two LED illuminators – built in coaxial for reflected light and behind-the-sample flexibly mounted LED for transmitted light.

 The system is typically provided with 12"x24" (300x600 mm) or comparable mounting breadboard with solid cover / protective enclosure.

The system comes in several parts and requires some minor assembly in the lab.



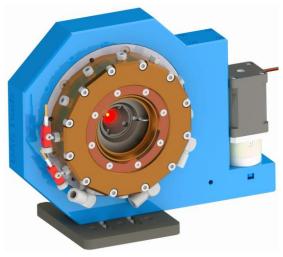




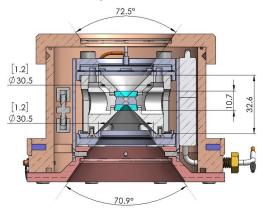
## LARGE OPENING WATER-COOLED CONTROLLED GAS ENCLOSURES FOR SINGLE CRYSTAL BRILLOUIN SCATTERING AND X-RAY DIFFRACTION IN RESISTIVELY HEATED DACS

The original bronze enclosure was developed by DACTools in collaboration with Dr. Bin Chen (U. Hawaii) and Dr. Vitali Prakapenka (GSECARS / U. Chicago). The purpose of the enclosure is to provide a working environment with protective atmosphere and single or double membrane compression / decompression capability for a variety of large opening High-Temperature Diamond Anvil Cells.

The prime design goal was an enclosure for BX-90 type DAC with large (up to 70 degrees) symmetric x-ray opening for single crystal diffraction plus single crystal Brillouin spectroscopy which requires rotation of the sample around the DAC axis. Thus the original enclosure has round shape and bore OD of 90 mm (central part) so it can be integrated with Newport URS-150 rotational stage compatible with GSECARS beamline infrastructure. The recent versions have larger diameter for



easier handling of wires and membrane tubing, and can be provided with a custom motorized rotational



stage.

With proper Diamond Anvil Cell selection and preparation, and suitable high temperature setups the enclosure allows high pressure measurements at temperatures up to and exceeding 1,000°C (as proven by recent experiments at GSECARS).

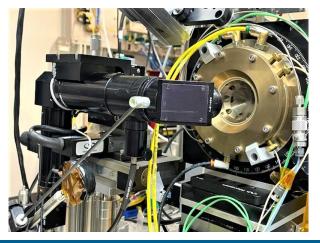
The original enclosure had one compression membrane, but the pressure frame was recently redesigned to allow doublemembrane (push-pull) pressure control while keeping up to 70 degrees of real symmetric X-ray and optical opening. The actual openings depend on the enclosure windows size and type, DAC type, and the Diamond Anvil / Diamond Seat configuration.

The enclosures are typically used with one of two types of hard windows – fused Silica and single crystal Sapphire windows. The custom flanges with other types of window material such as Mylar / Polyester, Kapton, KBr, etc. can be made on demand.

The enclosures are water cooled for stability and to protect the surrounding equipment. The design allows to cool down not only the main body of the enclosure but also the front and back flanges separately.

The enclosures have multiple vacuum tight feedthroughs for easy connections between the DAC and outside equipment. The connections / feedthrough include main power for DAC heater and two to four thermocouples (K-type and/or R-type). The enclosure can operate either in vacuum or in inert atmosphere as it has feedthroughs for pumping vacuum and supplying an inert gas.

The original bronze enclosure was designed for BX-90 type DACs. Currently DACTools makes multiple versions of double-membrane enclosure with Be Bronze and Aluminum bodies, with larger inner space, and compatible with multiple DACs such as DACTools' iBX-70 and HT SSDAC-70, Standard Symmetric DAC and other large opening DACs.







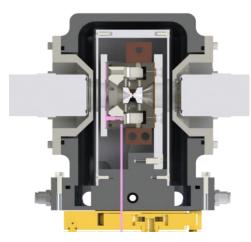
## CONTROLLED ATMOSPHERE ENCLOSURE WITH INTEGRATED MEMBRANE PRESSURE CONTROL FOR RESISTIVELY HEATED DIAMOND ANVIL CELLS

Currently DACTools does not provide High Temperature (HT) Diamond Anvil Cells (DACs) with small resistive heaters around diamonds as ready to go plug-and-play solution (although we are rapidly developing this area, including manufacturing of "off-the-shelf" heaters). Nevertheless, we have been actively involved in development of equipment for High Pressure Research at HT, including "whole cell" DAC heaters and controlled atmosphere / vacuum enclosures.

One of the examples is the development of the controlled atmosphere modular enclosure with integrated membrane pressure control for resistively heated Diamond Anvil Cells. The base of this aluminum enclosure is water cooled and has



a number of feed-thoughts for membrane pressure lines, heater and thermocouple wires, and inert gas or vacuum in accord with specific experimental requirements.

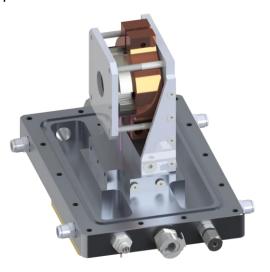


The enclosure has modular nature and can be used with different sets of flanges and window materials depending on the type of measurements and required experimental conditions. For example, for optical measurements (Raman scattering, reflectivity, etc.) where the objective lens has to be close to the sample (e.g. ~30 mm) the recessed flanges with fused silica or sapphire windows can be used. If larger opening is required, e.g. for X-ray diffraction measurements, the enclosure can be equipped with large opening flanges with Kapton or Mylar, or large fused silica or sapphire windows.

The enclosure can be used with a whole cell heater

(typically Copper clamp of appropriate inner diameter with pressed-in standard off-the-shelf heating cartridges). With proper DAC insulation such heater can heat the whole DAC to 500-600°C. If higher temperature is required – the sample can be heated with small resistive heater around the diamond anvils, with the whole cell heater ("double stage heating") or without it.

Typically enclosures have reinforced stainless steel DAC holder and a single or double membrane (compression + decompression) tie rod pressure control system with membranes on the same (for shorted working distance) or opposite sides of the DAC. The pressure frame and the shroud / flanges can be individually designed depending on the type of the DAC and specific experimental requirements.







# **COMPACT MODULAR VERSATILE NO-DRIFT CRYOSTATS**

The design concept of the modular combination cryostats is based on the following requirements:

Stable sample position: the sample moves only a few tens of micrometers while the temperature is changing. This is achieved by fixing the copper sample / DAC holder in place and adding flexible cryogen transfer links.

Flexibility in shape and volume: The volume and shape of the cryostat can be easily changed by choosing the correct flanges and radiation shields. For example, it is possible to use flanges with large round openings and Kapton windows for single crystal diffraction and total scattering or silica / sapphire windows positioned only a few millimeters from the DAC for Raman spectroscopy measurements or IR laser heating. The modular design also allows using bulky attachments for remote pressure control (e.g. piezo drive for time-resolved measurements).

Variable shroud shape and size: Depending on experimental requirements, the main shroud can be of different shape; for example, a rectangular shroud can be used where side flanges (x-ray emission / inelastic scattering in 90 degrees geometry) or multiple electrical connections are essential.

Versatility: The cryostats can accommodate various types of DACs: symmetric, various BX types, modifications of SSDAC, etc. (with different DAC clamps / canisters).

The cryostat can have various combinations of pressure drives: single membrane, two membranes on a

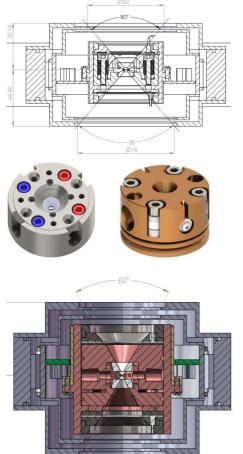
single or opposite sides, compression membrane plus decompression piezo drive, external mechanical / gearbox drive, etc.

Coaxial configuration: The cryogen transfer line is inserted into the cryostat vertically and the sample is typically positioned on the transfer line axis. This allows rotating the cryostat around vertical axis with minimized

drift, making it suitable for single crystal x-ray diffraction.



The first version of compact cryostat was designed by DACTools in 2015 for Jilin University and was made for several facilities resulting in a significant amount of high profile research. Recently DACTools worked in close collaboration with Physike Technology Inc. to design lighter and more efficient versions of the modular cryostat Scryo-S300 and its modifications. This cold-finger cryostat has interchangeable double-membrane BeCu canisters for different types of DACs, as well as various flanges, windows, and radiation shields. Current maximum angle configuration allows 80 degrees of symmetric x-ray opening (e.g. with DACTools' SSDAC-80), making this cryostat ideal for single crystal x-ray diffraction. The first tests conducted in 2022 demonstrated that with this cryostat even with large opening (60 degrees) the temperature on the sample in a DACs can reach below 6K. We believe that with further optimization we can reach 4-5K with 80 degrees opening.







## CUSTOM EXOTIC CRYOSTATS

DACTools has designed and manufactured a number of unique compact cryostats with integrated pressure control systems. These cryostats were designed in close collaboration with (beamline) scientists and are intended for specific experiments with severe geometry / environment restrictions for integration into experimental infrastructure.

One of the examples are two compact cryostats designed specifically for NRIXS measurements where

the APD detector (in air) must be 12 mm away from the cold sample in DAC (in vacuum), although it can also be used for X-ray diffraction, NFS, and other measurements. For details see Zhao et al. (2017) Rev. Sci. Instrum 88, 125109. Another example is a compact cryostat for Resonant Inelastic X-ray scattering integrated with online Ruby pressure measurement system. The cryostat system was designed for specific (radial) geometry and is fully integrated into an existing hutch instrumentation. For details see Kim (2017) High Pressure Research, 36:3, 391-403.





Recently collaboration with Vitali in Prakapenka & Co. (GSECARS) we have designed a slim cryostat for use with GSECARS double sided laser heating system which has very short working distance of <21 mm on each side. For our design we used DACTool's Mini-BX80 DAC with membrane-based remote pressure control.

This new system allows to utilize high resolution x-ray diffraction techniques including single crystal and non-crystalline scattering (amorphous solids and molten fluids), as well as optical spectroscopy to study materials properties in super-wide temperature range (10 - 6,000 K). But most importaintly, keeping diamonds at cryogenic temperatures during laser heating experiments solves a lot of problems with unwanted chemical reactions and element diffusion such as carbon diffusion from

diamond anvils and reaction with the sample; mass transport during laser heating; sample reaction with pressure medium or/and diamond anvils; premature

breakage of diamond anvils in experiments with alkali metals and hydrogen, and many others.

Another recent development (in collaboration with Physike Technology Inc) is design and construction of a custom motorized cryostat for single crystal Brillouin spectroscopy for Jilin University. The key point of the design is that the Diamond Cell can rotate inside the cryostat between Brillouin scattering measurements to sample different crystalographic directions. We are using a spring-loaded cold finger clamp which can be opened with outside motor for smooth rotation and closed for good thermal conduction between the cold finger and the specially designed 50mm OD Diamond Anvils Cell with 70 degrees symmetric opening. The pressure can be controlled using moveable hex wreches (which protrude into the cryostat) and a manual gearbox with rotation timing. The first tests have shown that the temperature in the sample can reach 10K and below.

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# EXAMPLES OF DAC PRESSURE CONTROL IN LARGE FLOW CRYOSTATS

#### **Compression-decompression dual membrane pressure control**

- Double-membrane compression / decompression assembly is designed for symmetric DAC with diameter of 1.875 inch (47.625 mm), but it can be adapted to majority of common and custom DACs of different sizes.
- Typically uses two identical 2.0 inch OD membranes for compression and decompression.
- Minimum operating temperature ~5K. The DAC can be brought to 1.8K, but membranes will not work below 4-5K (helium will freeze).
- Depending on cryostat and DAC design, the minimum working distance can be brought down to below 30 mm.

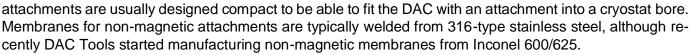
#### **Gearbox pressure control**

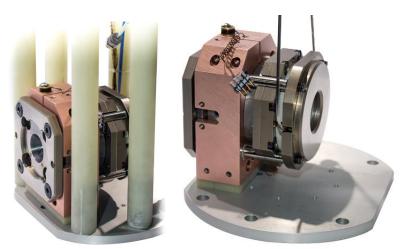
- Original version is designed for symmetric DAC with diameter of 1.875 inch (47.625 mm).
- Works with two right-handed and two left-handed #10-32 screws on 1.5 inch (38.1 mm) Bolt Circle Diameter.
- The pressure can be increased / decreased manually or by a motor by rotating tie-rods from outside the cryostat. The rods are linked to minimize sample motion (rotation).
- The gearbox was proven to successfully work even submerged into liquid helium.
- Can be modified for use with other DAC types and "wet" cryostats.

# **DECOMPRESSION ATTACHMENTS FOR MEMBRANE DACs**

Controlling pressure at cryogenic conditions can be quite challenging due to multiple factors such as unequal contraction of diamonds and metal parts, strengthening of spring washers, freezing of contaminants (e.g. finger grease), etc. An addition of a second (decompression) membrane can often be the only way to reach desired experimental conditions (especially in a low pressure region below 5-10 GPa). Thus DACTools offers custom membrane-based decompression attachments made of alloy / hard stainless steel or Beryllium copper (for cryogenic / non-magnetic DACs) and designed for use with Diacell and other integrated membrane DACs.

Because many commercial cryostats have very limited flexibility and a relatively small diameter cryostat bore (e.g. 2.7 inch or smaller) decompression









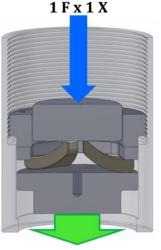




## COMPACT FORCE-AMPLIFIED PNEUMATIC REMOTE PRESSURE CONTROL MECHANISM FOR CRYO DACS

A large number of physical phenomena such as superconductivity, magnetic ordering, and quantum critical phenomena, often appear only at very low temperatures (e.g. below 5 K). There is an immense interest to investigate these phenomena at high pressure as a means of tuning interatomic distances, and thus the interaction parameters controlling these phenomena, in a continuous and well controlled fashion.

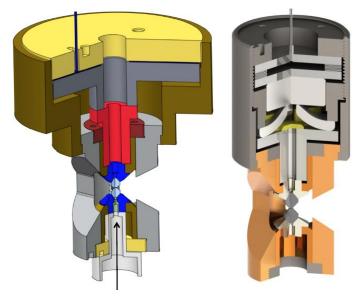
The simultaneous generation of tunable high pressure and ultra-low temperatures is a well-known problem. While some cryostats have relatively large bores or space inside the shroud and allow to use large membranes which generate significant forces, the majority of cryostats (e.g. top loaders) have limited bore diameters, especially when it comes to studying materials in high magnetic fields. Also at Neutron facilities, where the loads applied on the diamond anvils are an order of magnitude larger than those in regular DACs (e.g. 1-10 tons vs 0.1-1 tons), regular neutron membrane DACs cannot fit into top loading cryostats capable of reaching temperatures of 4 K and below.



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In collaboration with the High Pressure group from SNS / ORNL and with the <u>DOE SBIR grant</u> DACTools has developed a compact (<70 mm OD) force-amplified pneumatic pressure control mechanism for DAC which can generate remotely controllable load in the order of 10-12 tons with up to 1 mm displacement and provide smooth pressure change at base temperatures without the need for significant thermal cycling of the system, which can be problematic at Neutron facilities due to many factors.

The design is based on a combination of pneumatic bellows (which provide significantly larger displacement than double-leaf membranes) and several (three in this case) levers which amplify the force by 3-4 times at the expense of displacement. In the shown setup 60 mm OD bellows in combination with levers can generate the same 12 tons of load as a single double-leaf membrane with 100 mm diameter. This



Left: Large volume neutron diamond cell with double-diaphragm type membrane drive with 120 mm OD and 100 mm membrane (Boehler at al., 2017). Right: The compact pneumomechanical attachment to the same neutron DAC capable of generating the same force and displacement as the large membrane on the left (canister OD = 69 mm, bellows OD = 60 mm). allows to reduce the OD of the membrane canister from 120 mm to 68 mm and also decrease thermal mass allowing faster cooling / heating of the DAC system.

This development allows to significantly decrease minimum experimental temperatures in remotely controlled nDAC from current 5-10 K down to 2 K while the sample is at high pressures of several tens of GPa.

The new developments allow to create a wide range of compact DACs for various neutron scattering experiments which can reach sub-Megabar pressures at 2-4 K while preserving accurate remote pressure control capabilities.

The new concepts can be used for developing high-pressure instrumentation outside the neutron scattering field (e.g. compact DACs) in sample environments with very limited space (e.g. cryostats withs strong magnetic field) but with requirement of relatively large remotely controlled forces.

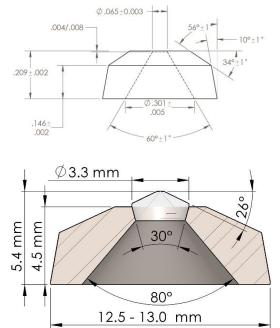




## **DIAMOND ANVIL CELL ACCESSORIES**

DACTools' goal is to be able to provide high-pressure community with a full range of tools for doing High Pressure research with Diamond Anvil Cells. This includes DAC accessories and consumables from various diamond anvils and diamond seats through non-magnetic seats/gaskets (made out of exotic materials) and to various diamond alignment jigs and other small tools essential for high pressure research.

The current inventory includes a variety of pressed and cut C2 tungsten carbide *flat diamond seats of popular and custom shapes and sizes*. For example, we currently stock common 5.3 mm high flat seats with 50-60 degrees opening and 1.12 mm and 1.65 mm holes. We also have multiple *conical support seats for Boehler-Almax* type anvils. The stock of various seats, from 3.2 mm to 11 mm high, is growing at fast pace and will follow the demand. We can also supply custom seats of virtually any size and shape from WC (including non-magnetic WC with Ni binder), cBN, BeCu and other suitable materials.



DACTools has a limited stock of Rhenium gaskets and virtually unlimited stock of annealed Ruby spheres and stainless steel gaskets.

In collaboration with Dr. Reini Boehler we provide Diamond Anvils (typically Type 2A HPHT) with 30 degrees conical support mounted in Vascomax seats. Smaller support angle for the same x-ray opening results in thicker and stronger Diamond Anvils leading to higher anvil stability and higher maximum pressure.

We are also starting collaboration with Loto-eng and planning to market *micro EDM / spark eroder machines* which provides convenient and economical way to drill gaskets for DAC work.

## **DIAMOND MOUNTING JIG**

The diamond mounting jigs are used for fixing diamonds to tungsten carbide, boron nitride, and others diamond seats with epoxy. Currently we offer spring loaded stainless steel jigs. The universal design allows working with both flat table diamonds and conical support (Boehler type) diamonds.

The jigs accept seats from 9 to 18 mm in diameter and large range of seat thicknesses from 3 to 12 mm. The seat position is adjusted by M3 (0.5 mm pitch) thumb screws. A typical plunger has a



through hole, which allows lighting the diamond culet for alignment (critical when aligning diamonds with flat table). Plunger force can be up to 5-10 kg depending on washer configuration or type of spring plunger. The force on the diamond can be easily calibrated and adjusted by rotating the threaded piston. Custom configurations are available.

The jig system with Class 1 laser for controling diamond tilt in conical support diamond anvils and seats is coming soon.





# **GEARBOXES FOR GAS LOADING SYSTEMS**

Since 2009 DACTools has designed and manufactured multiple custom gearbox adapters for closing various DACs in different types of highpressure gas loading systems such as GSECARS, CIW, Top Industrie, and some other. The current collection includes gearboxes for DAC designed and manufactures by DAC Tools and other DAC "Standard" suppliers: Imperial and Metric Symmetric DACs, Mini-BX80 and variants of BX90, iBX and SSDAC series, Diacell DAC, different Panoramic DACs, Almax-Boehler DAC, and 4-pin GL DAC, and can be designed for virtually any DAC



with which will fit into a gas chamber of a particular gas loading system leaving about 0.5-1.0" (or more) space at the bottom.



Except for the gearbox for Boehler-Almax DAC which has three screw drivers, almost all earlier gearboxes have two pairs of hex screw drivers which rotate independently of each other – this enables to control pressure in DACs which have either left-handed (LH) and right-handed (RH) screws, or only right-handed screws. Recently we have designed several gearboxes for gas loading systems which have only one hex drive (such as CIW-type system and Edinburgh systems) and these gearboxes can handle either DACs with all RH screws (e.g. some versions of BX90 DAC and SSDACs), or DACs with left and right screws.

Most of our recent gearboxes have spring loaded changeable hex bits (e.g. 3 mm and 5/64") for DACs with the same screw spacing but different sizes (e.g. modifications of BX90 DACs have either #8-32 or M4 screws depending on the manufacturer). All gearboxes are supplied

with 3-D printed fillers (e.g. nylon plugs) which minimize empty volume inside the gearbox and help to reach the highest gas pressure.

All gearboxes allow measuring pressure during gas loading (provided that the gas loading system has an integrated Ruby system).

Using the gearboxes and integrated Ruby systems the DACs can be closed and retrieved at minimum holding pressure of 0.3-0.5 GPa.

