High-Pressure Techniques and Neutron Scattering

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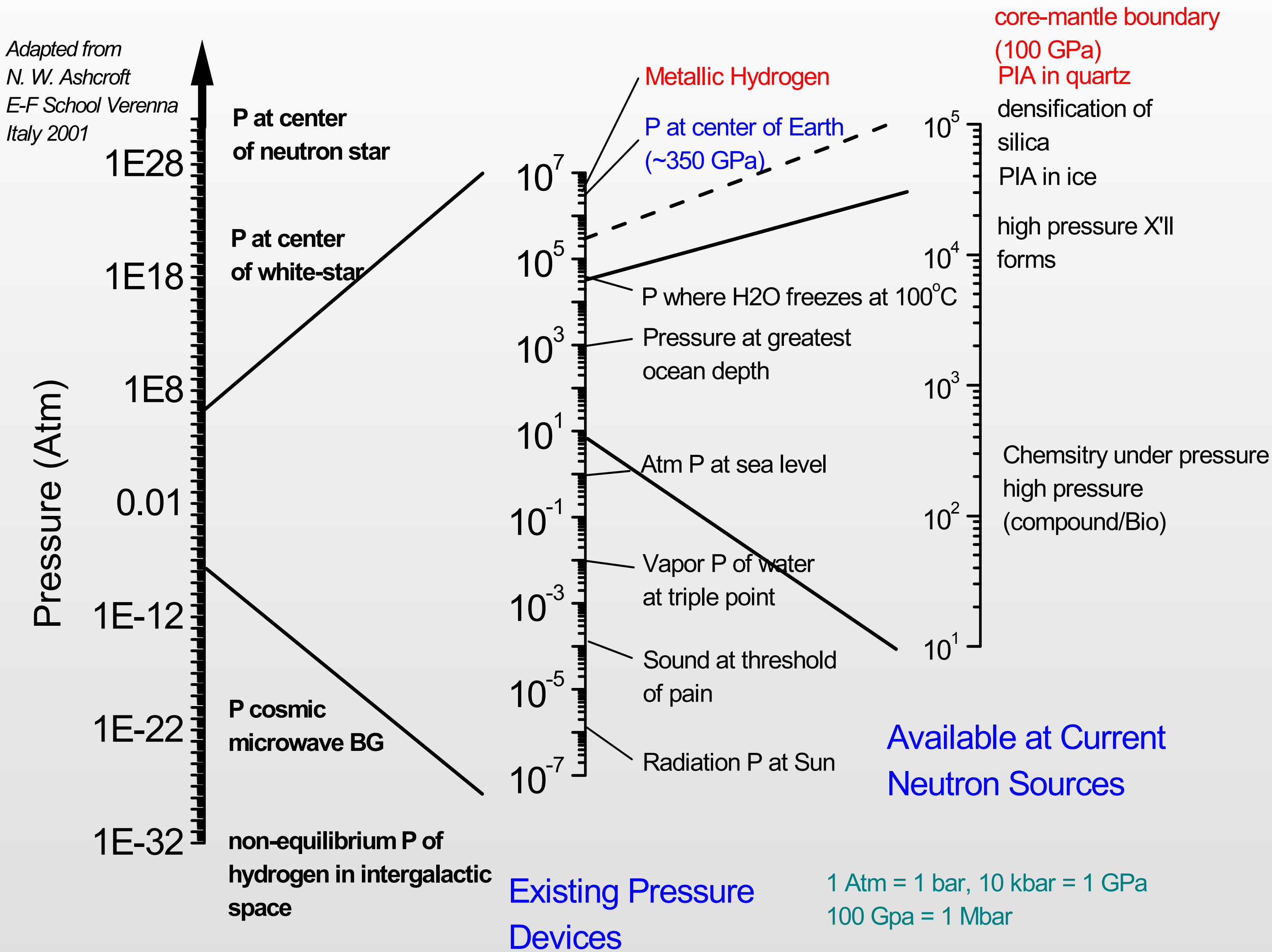
Pressure Ranges



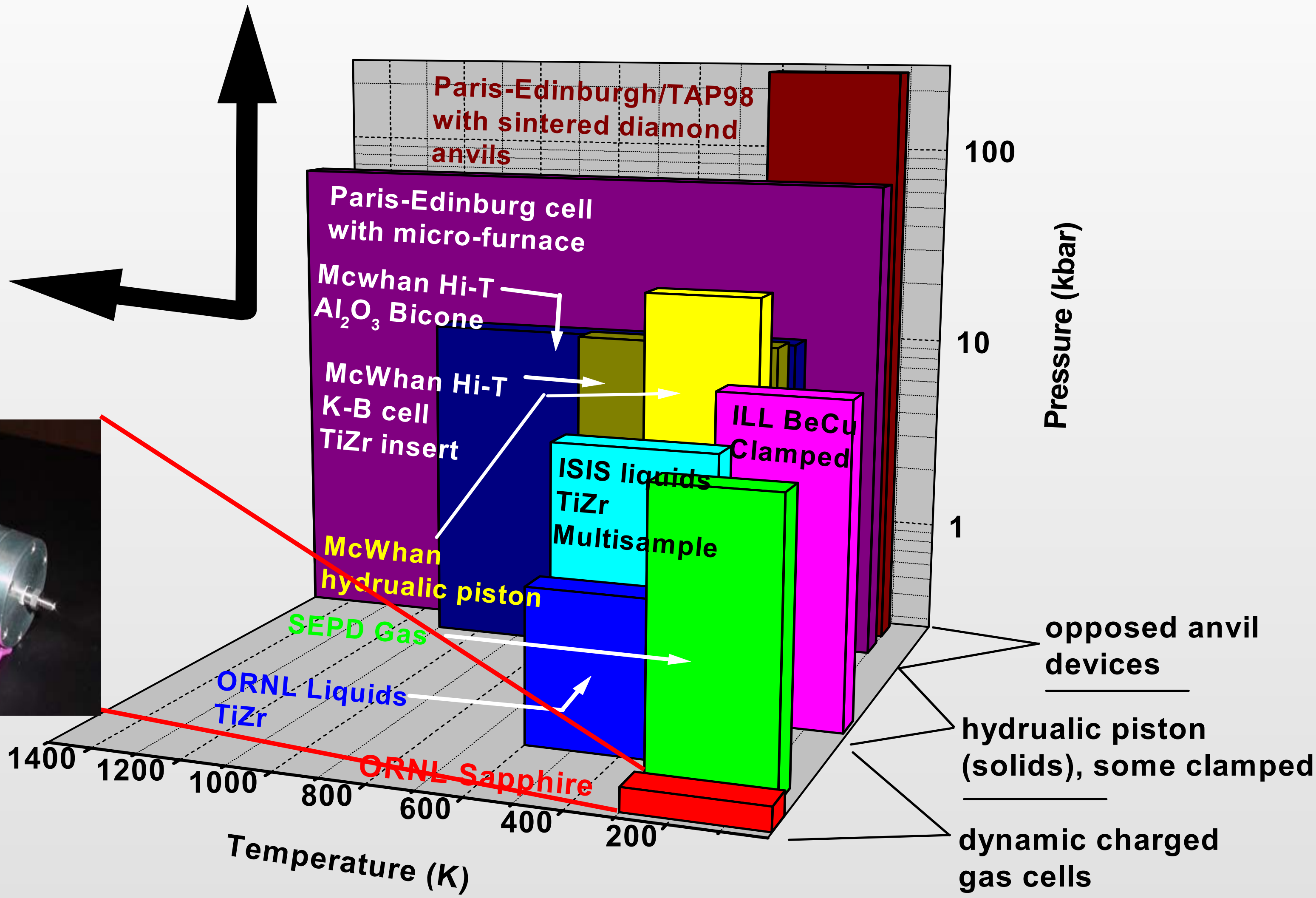
Milestones in HP Techniques

- ~ 60 years since initiation of dynamic (shock wave) methods (remarkable expansion of PVT range → nuclear shocks)
- Percy Williams Bridgman wins 1946 Noble Prize, "for the invention of an apparatus to produce extremely high pressures, and for the discoveries he made therewith in the field of high pressure physics"
- ~ 40 years since Lawson-Tang paper on possibility of diamond anvil static high-pressure cells
- ~ 17 years since Mao-Bell ultra high-pressure (1 megabar) experiment. Mao-Bell piston cylinder DAC.

1. Of the thermodynamic variables, temperature plays by far the most prominent role in probing material properties.
2. Bridgman (1926): pressure offers a route to breaking down the electronic structure of atoms—new bulk properties of matter. Very high pressures allow the study of truly basic problems of condensed matter physics (nuclei and <u>all</u> electrons)

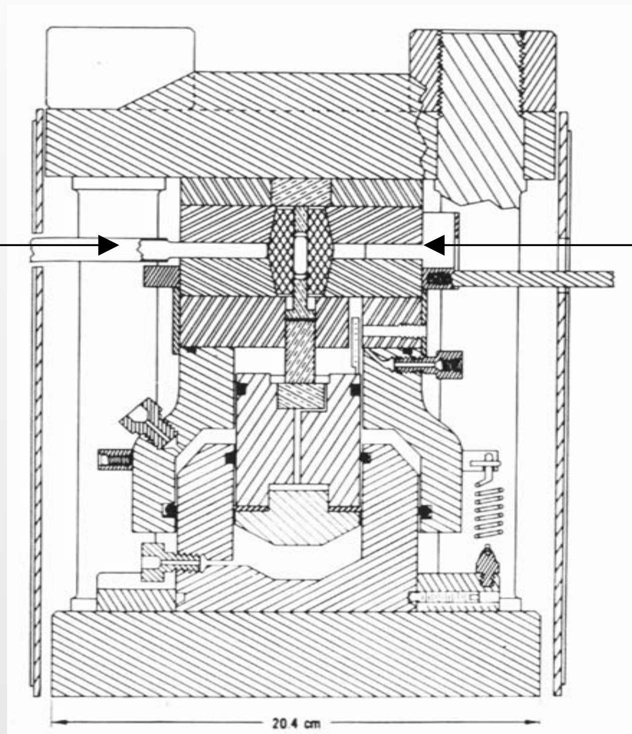
Adapted from N. W. Ashcroft E-F School Verenna Italy 2001

Survey of Neutron HP cells

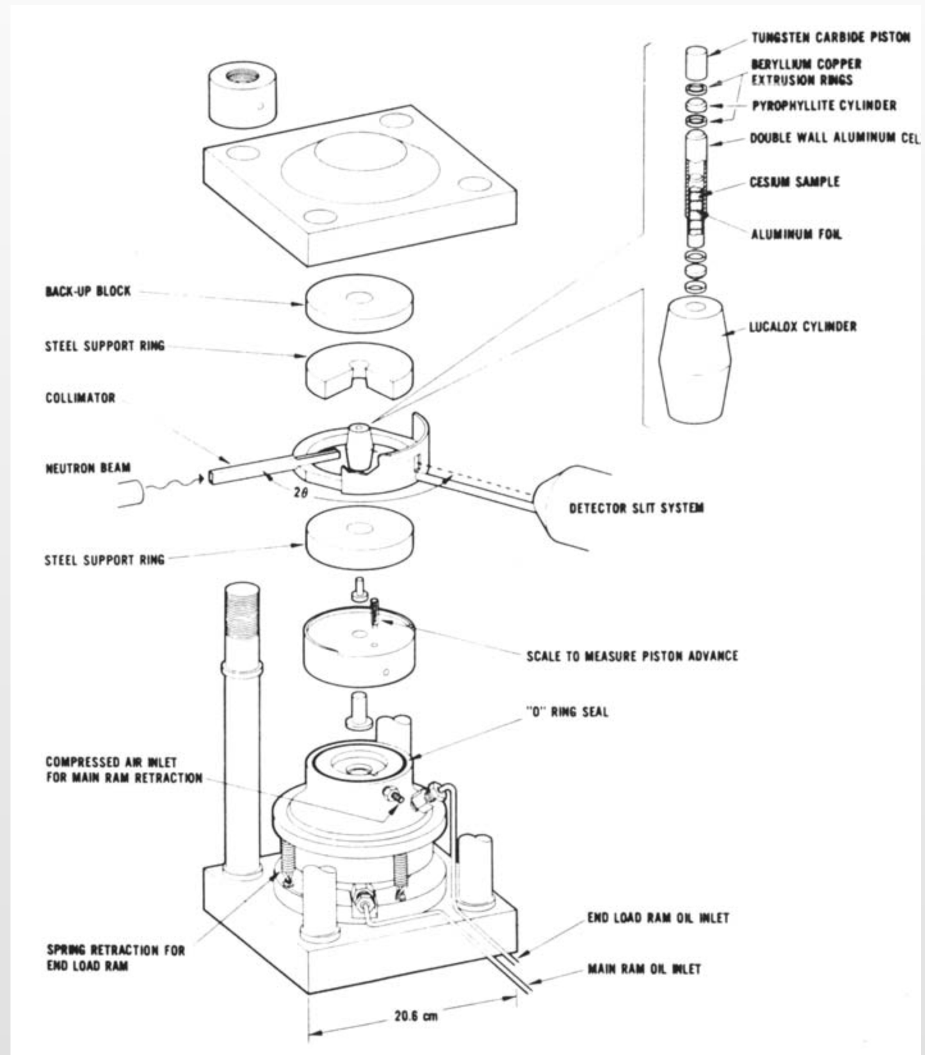


A.J.Rondinone, private comm.

McWhan High Pressure Neutron Cell, 30-40 kbar



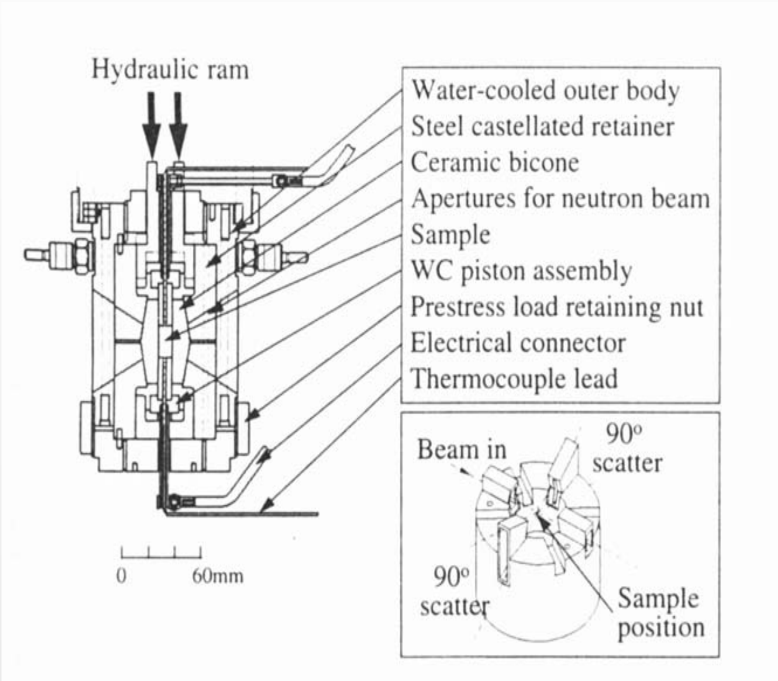
6 mm neutron window

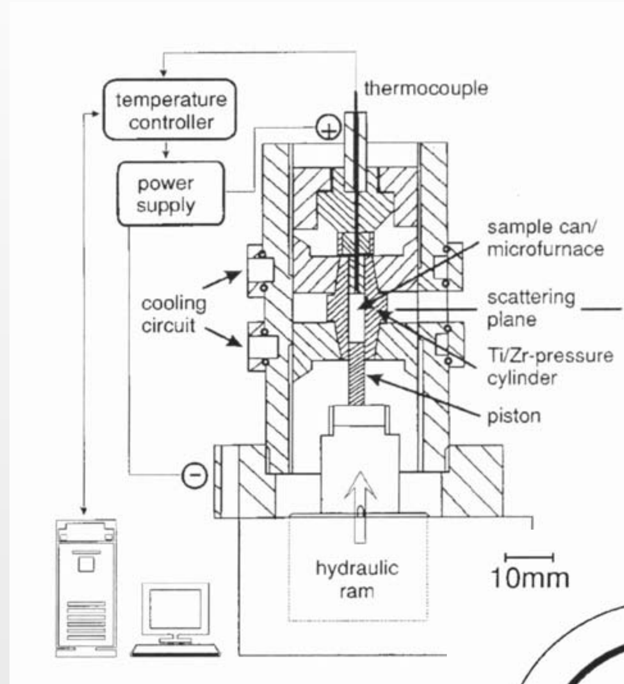


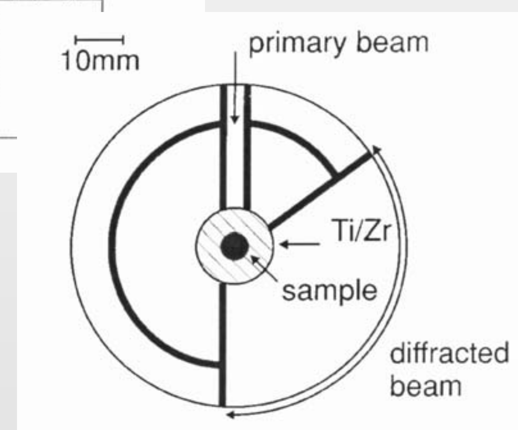
- Design first reported in 1974, represented the first “jump” in pressure capability
- two press design simultaneously provides radial support to binding ring and piston thrust
- polycrystalline alumina sample camber, complicate data somewhat, most other parts are tool steel
- used primarily for compression of powdered solids
- 0.1 cm^3 samples, 6.5 mm piston

D. B. McWhan, et al., Rev. Sci. Instrum. 1974

Variations on the McWhan Cell



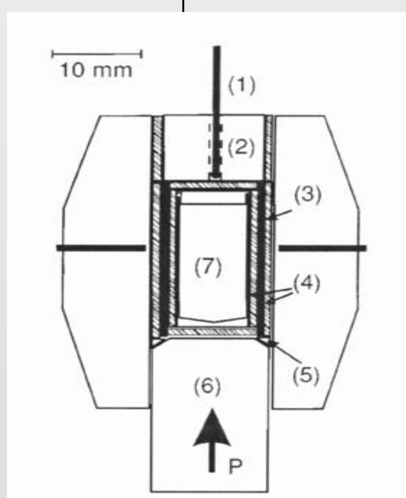




1997

- High temperature cell for neutron scattering at pulsed sources, fixed scattering geometry, ISIS
- used to 1100 K and P ~ 12 kbar
- primarily powdered solids (diffuse to 7 kbar, alumina bicone peaks)
- heated by a carbon tube around sample, high current with ~ 0.02 Ω

S. Hull, et al., Nucl. Instr. Meth., (1997)

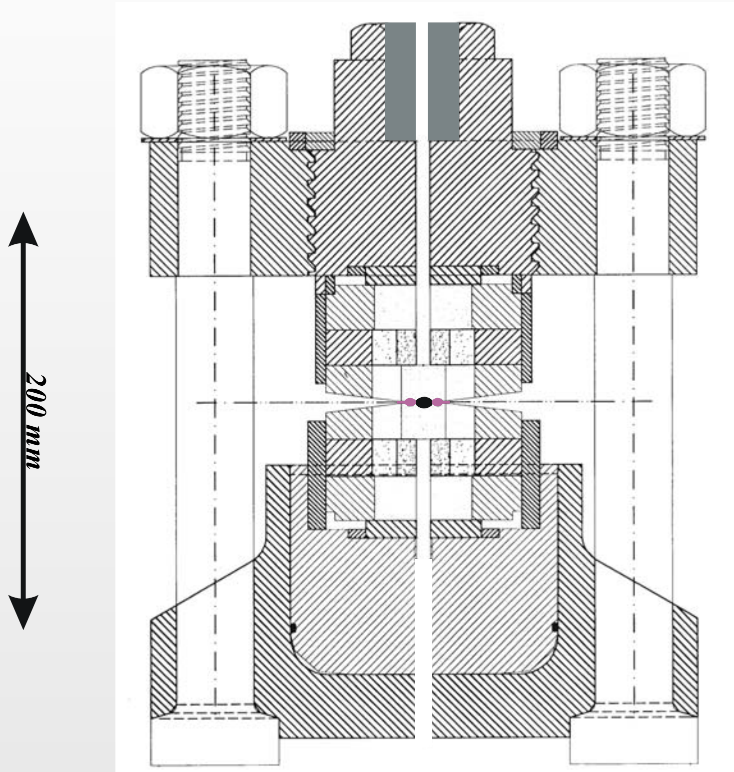


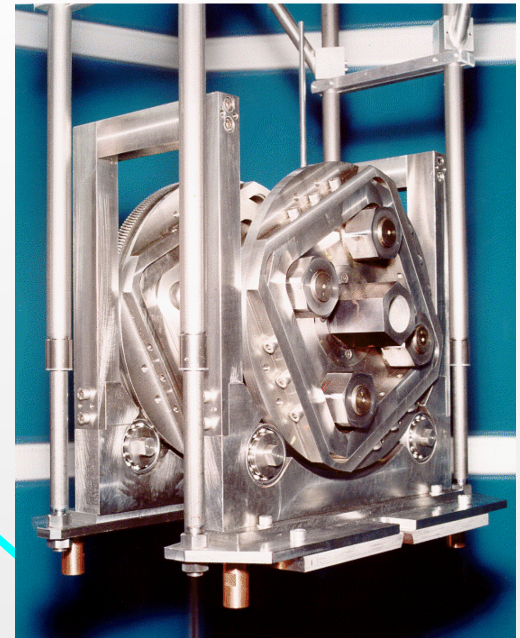
1997

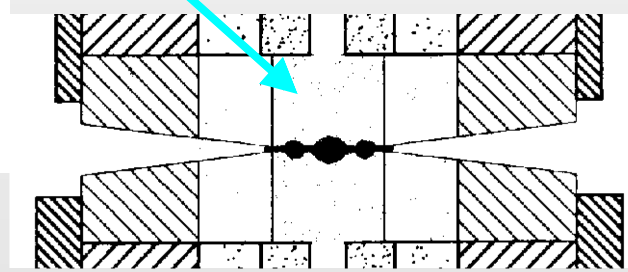
- Known as Kiel-Berlin cell, HMI-Berlin
- 700 K carbon heater, P ~ 15 kbar
- designed for monochromatic incident beam, TiZr bicone used to reduce cell Bragg scatter

K. Knorr, et al., Rev. Sci. Instr., (1997)

Paris-Edinburgh HP Cell







- First opposed anvil (WC or sintered diamond) device designed specifically for use at pulsed neutron source, ISIS. Relies on compression of gasket to reduce sample cell vol. (200-400 tonne press)
- Temperature ranges from < 100 - 1200 K with pressures up to ~ 25 GPa (250 Kbar)
- Primarily used for solid powdered crystals, but has been used for single crystals and amorphous solids
- Typically TiZr gasket material cone angle (i.e. detector coverage) of 14^{o}
- This cell also in use at Los Alamos, and one is currently being shipped to IPNS

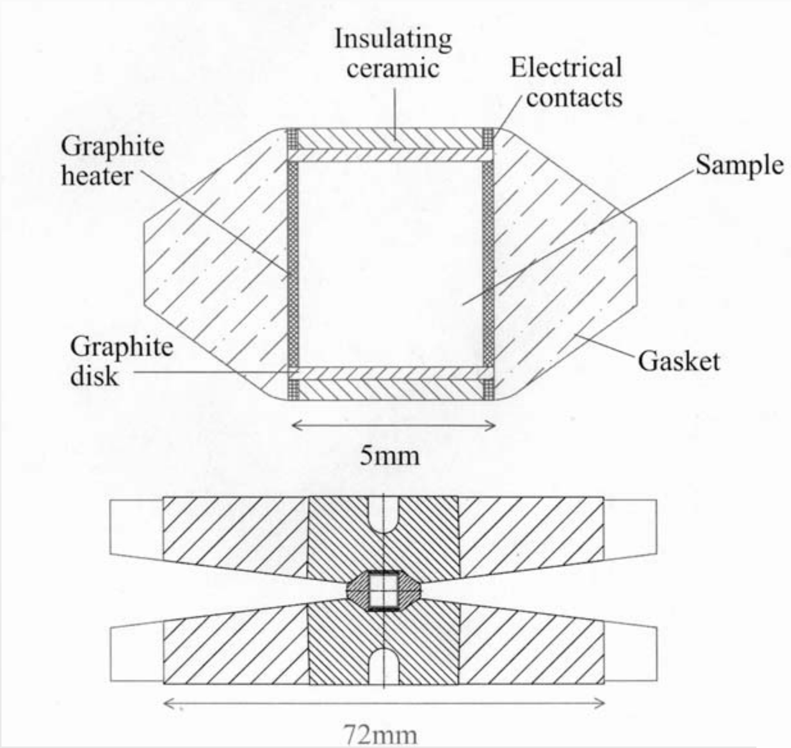
Adapted from J. Loveday, private comm. and M. Guthrie Ph. D. Thesis

Hi-Pr Pearl: A Neutron Diffractometer

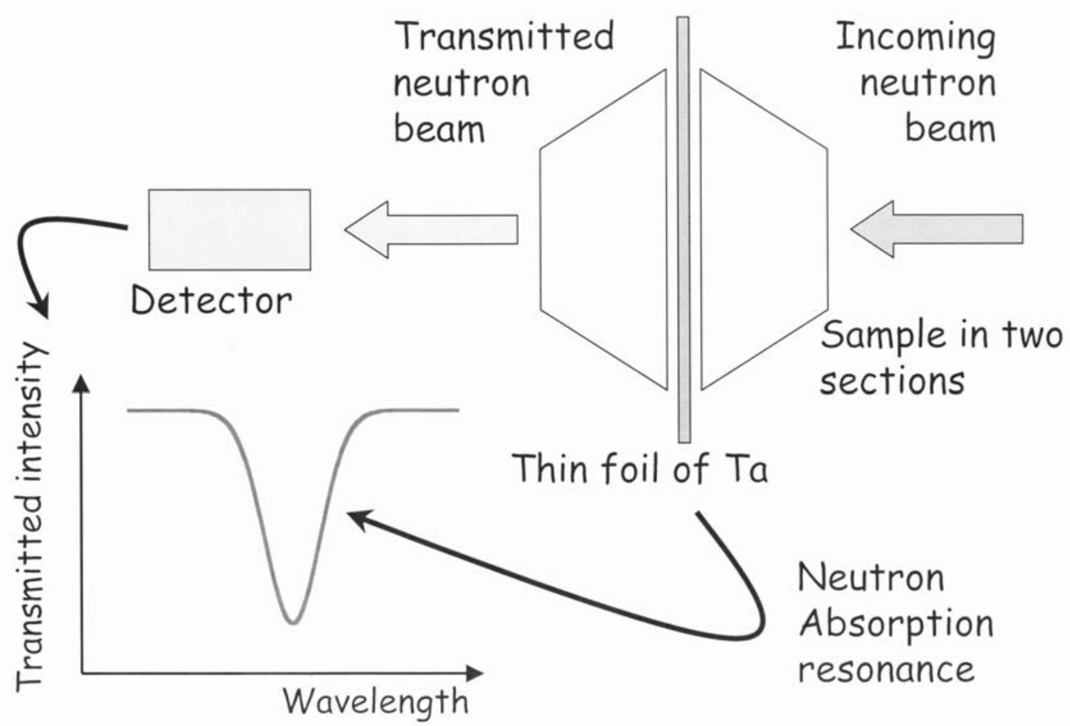
- First Beamline designed specifically for HP- neutron scattering studies, designed around the P-E cell entire assembly (with cradle and tank) is between 150-190 Kg. $\Delta d/d \sim 0.8$ % at 90^o
- Large out-of-plane 90 ^{o} coverage with options for use with lower angle scattering banks, $20 \leq 2\theta \leq 160$
- One single-crystal detector
- Plans underway for the next generation of high-pressure instrument at ISIS, including single crystal capability

Adapted from M. Guthrie Ph. D. Thesis

Heating the P-E Cell



1100 K achieved using this technique
With Pressures up to 7 GPa (70 kbar)



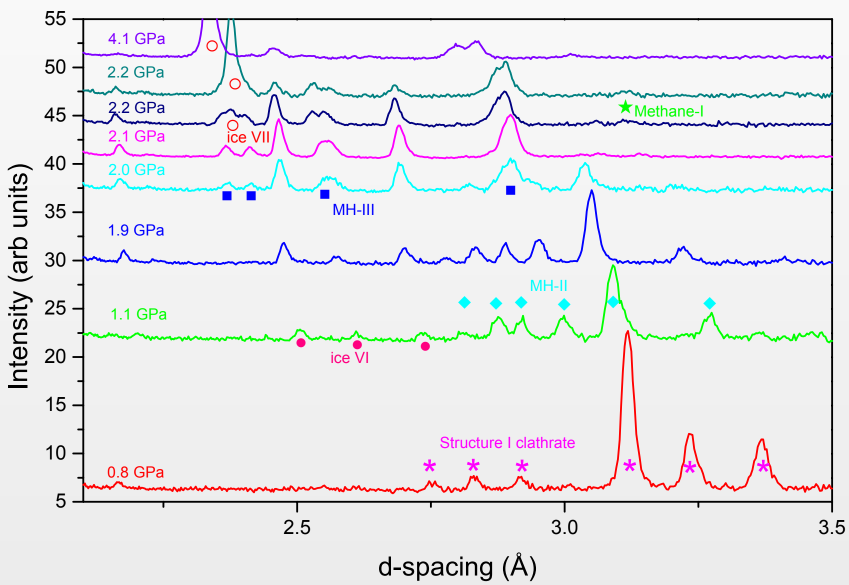
Line width of absorption resonance is calibrated to temperature.

Laser heating not possible due to opaque anvils, WC and sintered diamond.

Adapted from M. Dove Cambridge University 2002

Examples of Recent Science with the P-E Cell

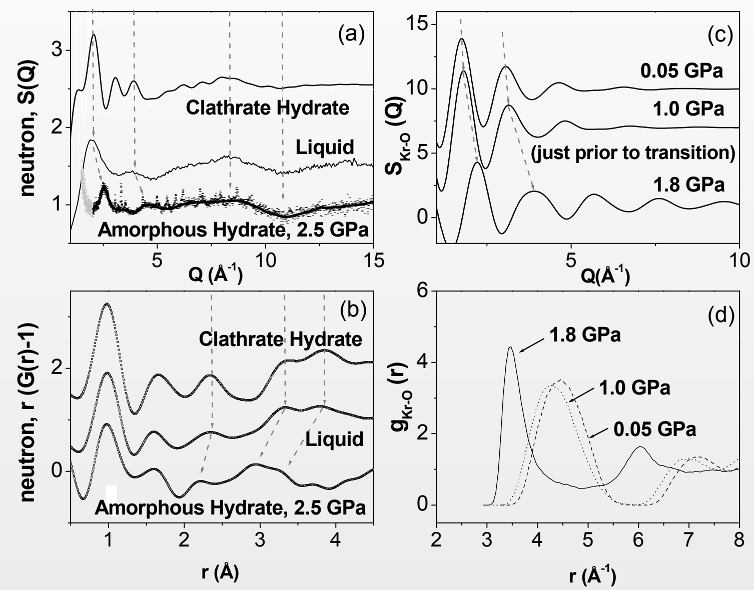
High-Pressure forms of CH4 hydrate



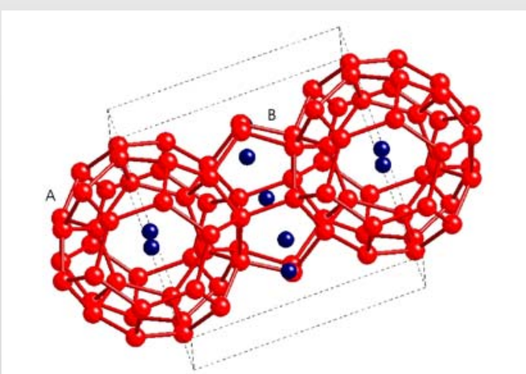
Loveday, Nelmes, Guthrie, Klug and Tse, PRL 2001

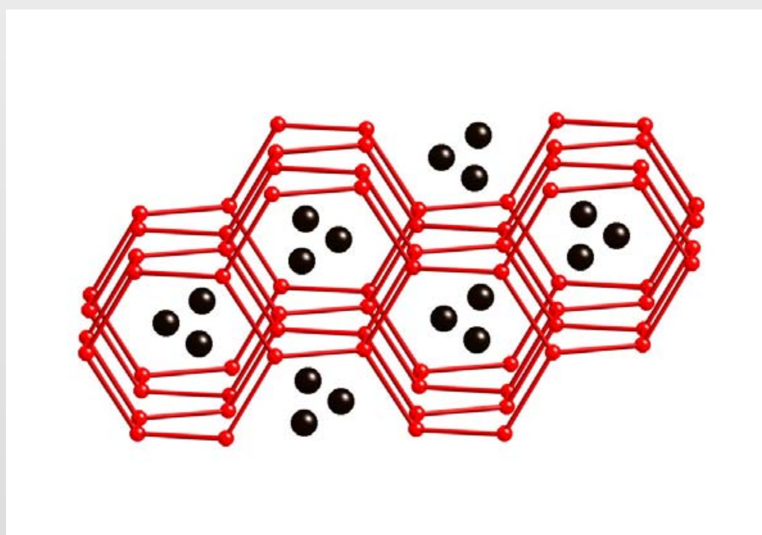
Loveday et al Nature 2001

Amorphization of clathrate hydrate, 77K



C.A. Tulk, et al., submitted May 2002





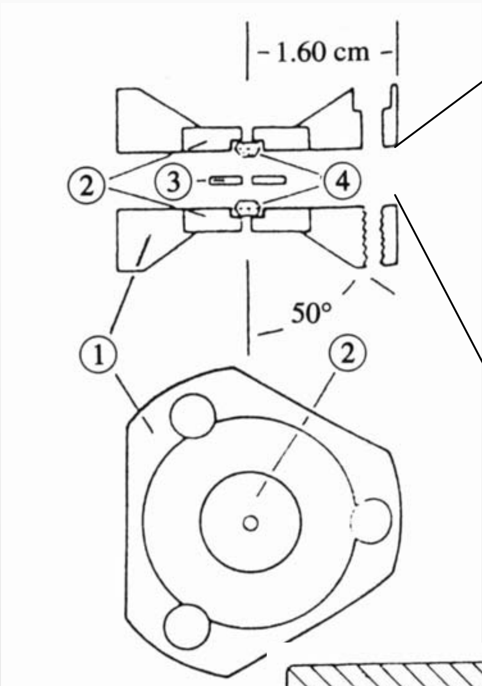
With major advances in source performance comes the opportunity for significant advances in techniques → new science (not just smaller and faster), e.g. synchrotron x-ray sources and multi-mega- bar pressures, opened new regions of phase space!!

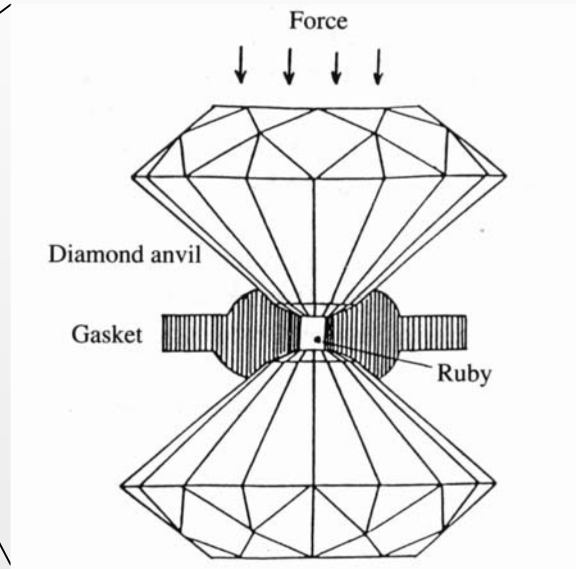
What's the most exciting science, and how do we plan to adapt/advance high pressure technology to the unprecedented flux level to be provided by the SNS?

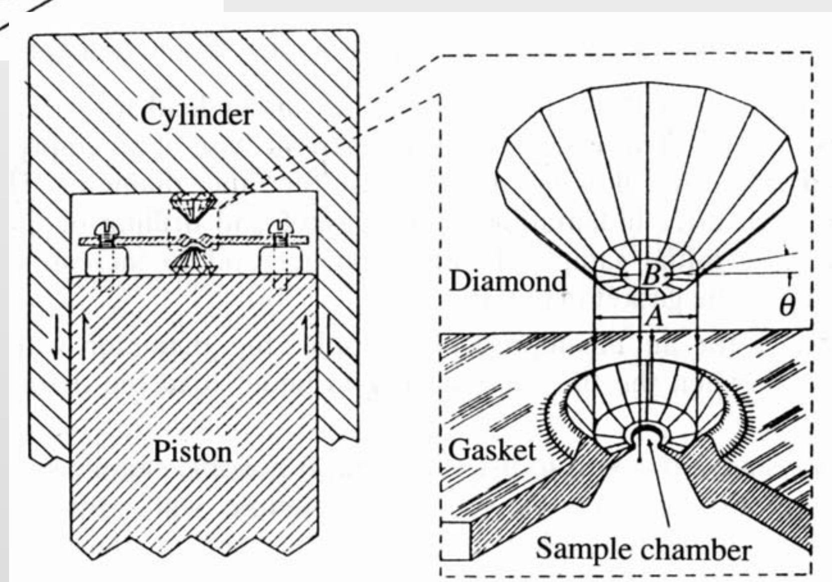
Science Motive

- Nature of dense hydrogen, including the metallic state (From cryogenic to brown dwarf conditions) A problem made for neutrons to solve!
- Hydrogen Bonding, symmetric hydrogen bonds in the water system! Planetary ices (hydrogen disorder)!
- State of the Earth's core, complex alloys to core P-T conditions!

Typical off-the-shelf Diamond Anvil Cell





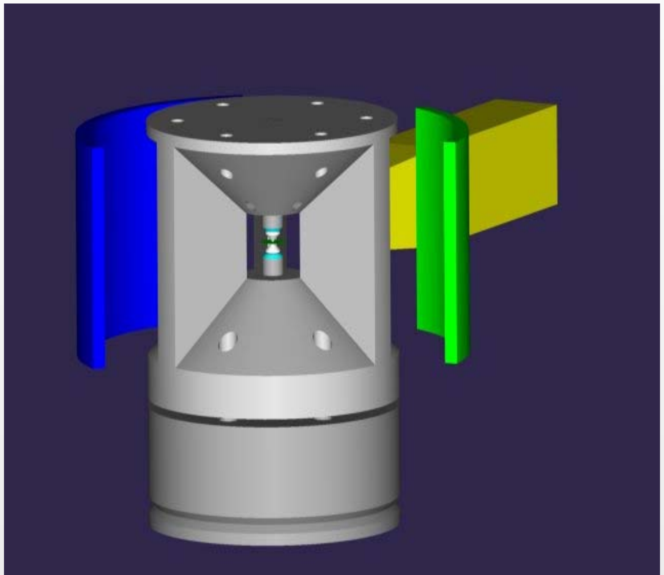


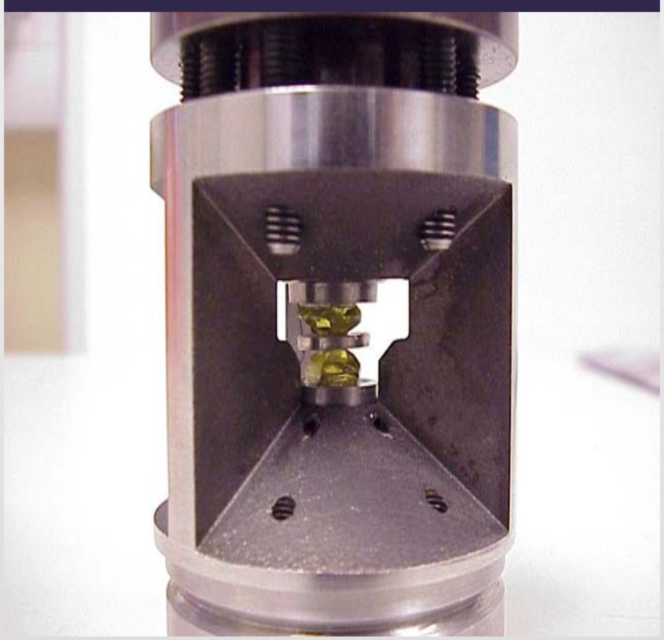
- The most widely used device for pressure > 1 GPa (10 kbar), has generally not been applied to neutron scattering (very successful with x-rays).
- Sample sizes are very small, culet ~ 400-700 μm (20-50 GPa)
- Sample sizes are 1/3 x culet size ~ 133x15 μm disk.

- Gasket typically of stainless steel or beryllium copper, but TiZr is an option, they must provide high-pressure chamber and support the anvil culet.
- Very stable piston-cylinder system with beveled culets are capable of multi-mega bar pressures (few physical probes)

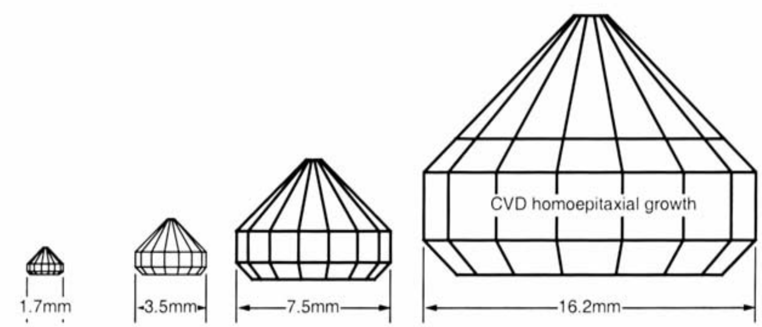
J.M. Besson, in High-Pressure Techniques in Chemistry and Physics, 1997

Open cylinder (panoramic) design, large volume DIA



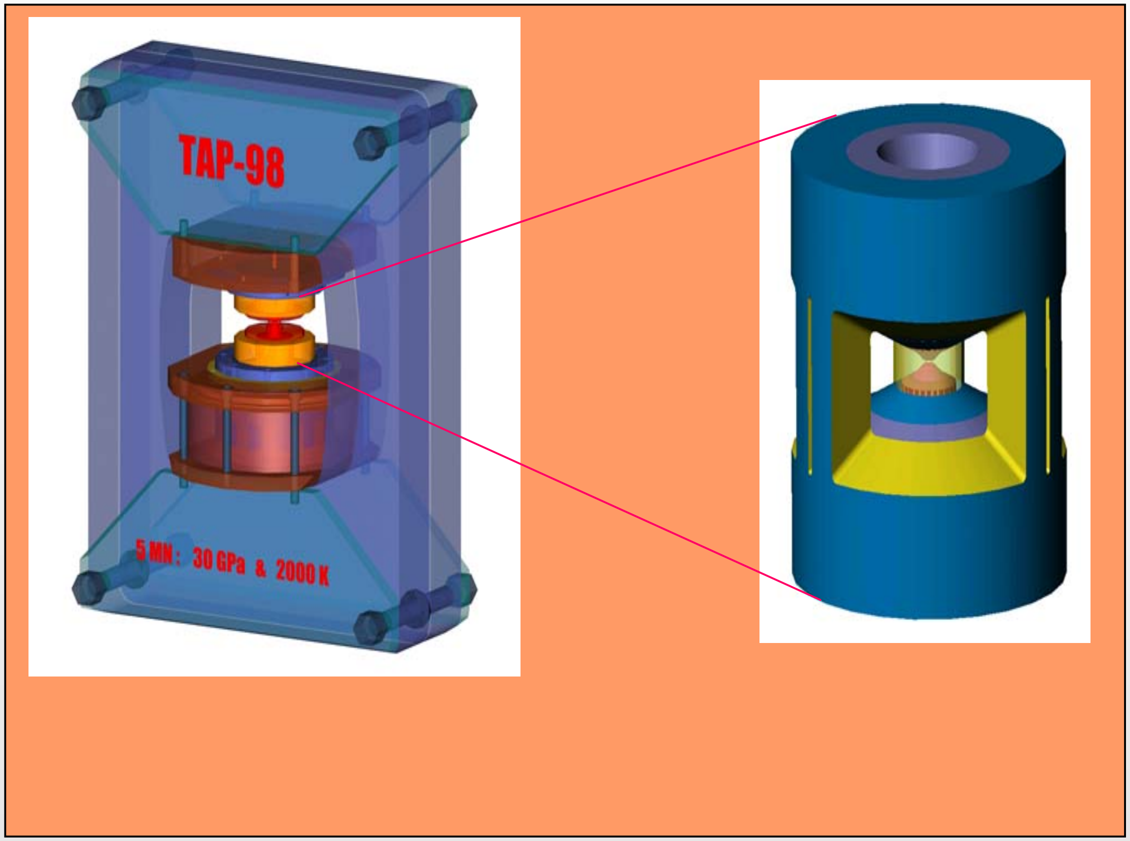


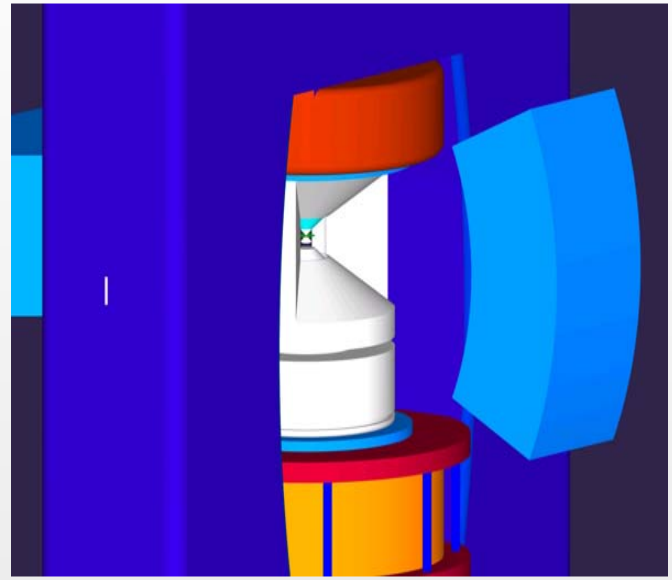
- Large open cylinder design, very stable along the thrust axis, removed sections each measure 105^o in the equatorial and 68^o in the vertical. **Very Large Detector Coverage**
- Large anvil required for the goal of 1 mm^3 samples (3-4 mm culet and 25-30 mm tables. Currently, synthetic sapphire, moissanite (hexagonal SiC), or CVD diamonds are candidates. Incident beam through the gasket.
- Natural diamonds far too expensive and likely not defect free (cost of natural stones increase quadratically with size while CVD diamonds are linear). Currently ~ 2.5 ct synthetic diamonds are available.
- Current prototypes 30-40 GPa



weight, carat	0.025 ct	0.25 ct	2.5 ct	25 ct
Natural diamond	$200	$1,200	$25,000	$2,000,000
Synthetic diamond	$400	$1,500	$5,000	----

Achieving Pressures > 50 GPa with Large Volume Samples

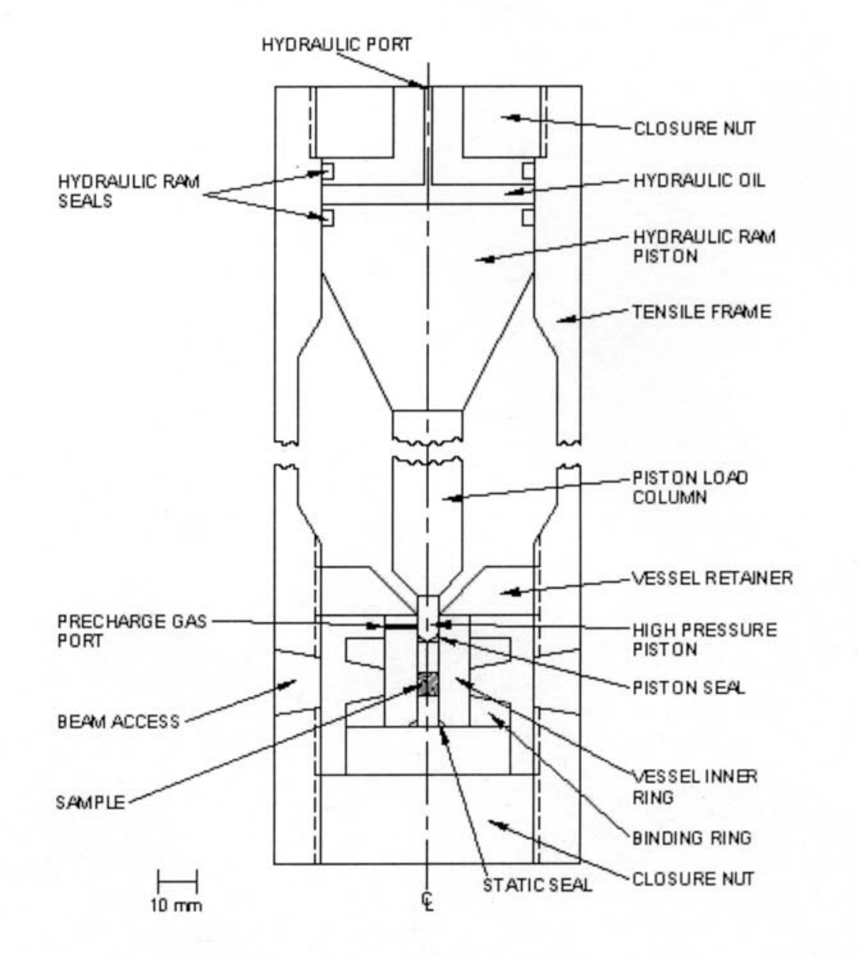




Panoramic cell in frame press with detector coverage provided by the vertical detector bank, other bank curved in the horizontal plane.

Frame press ~ 500 ton (e.g. TAP-98 with toroidal anvils currently entering service at Los Alamos) provides the required clamping force with superior stability. The panoramic cell, or more traditional toroidal anvils, can be incorporated with the press and positioned in the beam. A clamping mechanism may also be used to facilitate removal of the HP cell assembly for easy handling.

Higher not always better, the gas-pressure initiative



- No opposed anvil cell ever provides truly hydrostatic pressure. However, often this is a requirement for particular experiments.

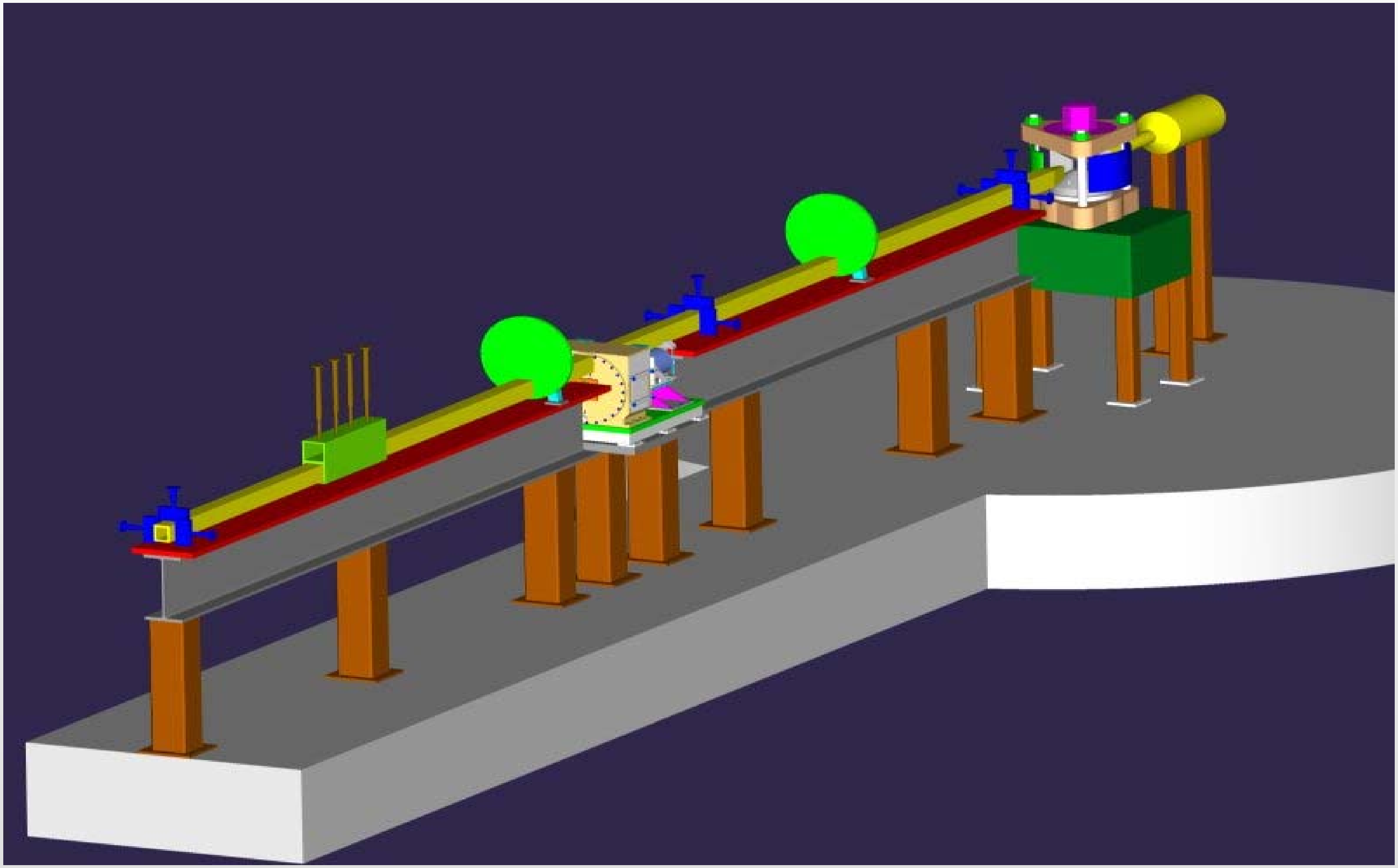
- 3 GPa (30 kbar) gas cell is precharged with an intensified gas to 6-8 kbar, advancing the piston with the hydraulic ram then seals the gas inlet port, and further compresses the gas and sample volume. Insert may be made of TiZr as in the McWhan cell.

- Such cells are used to 60 kbar (with heat treated maraging (350) steel, I. Getting)

- Can be made quite compact for heating/cooling

More modifications of the classic McWhan design

Proposed SNS Beamline Layout



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Thank-You