

# High Pressure Techniques



M. Guthrie, Geophysical Laboratory

# High pressure: a tool for material science

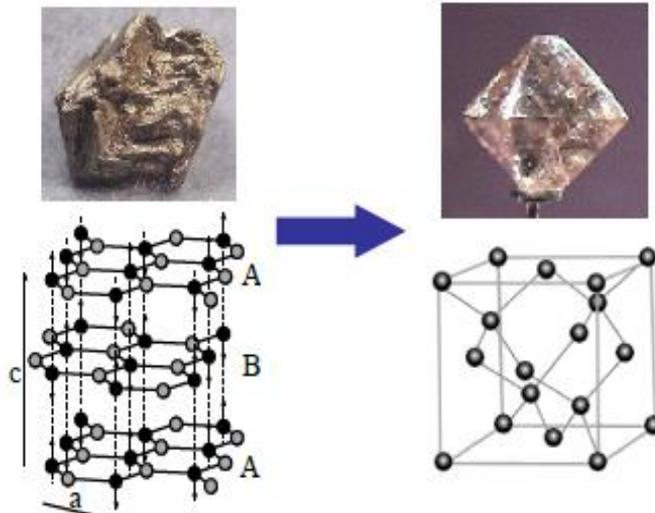
- Without varying external parameters, chances of understanding materials is rather limited (e.g. SC...).
- Popular variables are  $T, X$  (less common are  $H, E, \Phi$ ...)



P is an underused but uniquely powerful variable

# High pressure: a tool for material science

- P is essential to explore full versatility of inter-atomic bonds

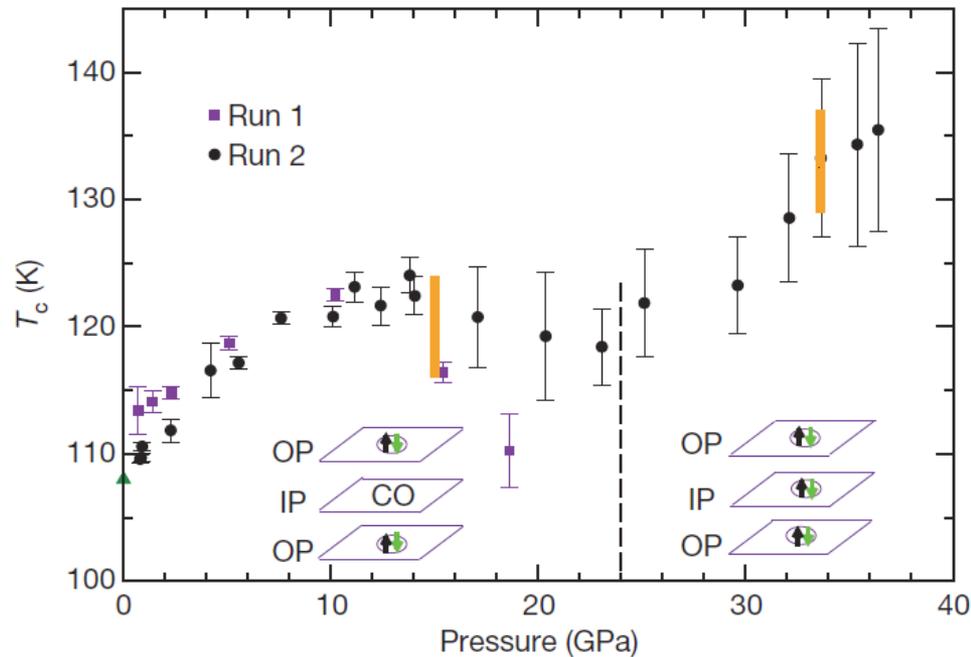


Vital to synthesise diamond

- Contemplate new spectrum of  $sp^3$  C allotropes e.g. mesoporous diamond, amorphous diamond, hydrogenated carbons...)

# High pressure: a tool for material science

- P Can directly modify electronic properties (subtle)

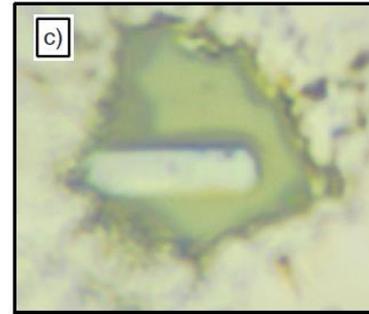
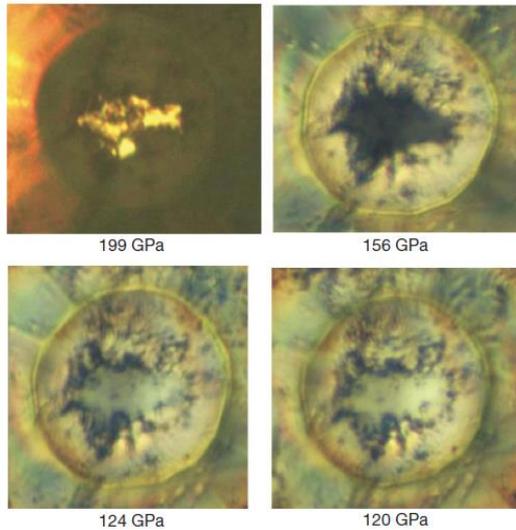


Can tune superconducting transition  $T_c$

# High pressure: a tool for material science

- P Can directly modify electronic properties (dramatic)

**Transparent sodium**  
(at 199 GPa)  
[2] Ma et al Nature 458  
182 (2009)



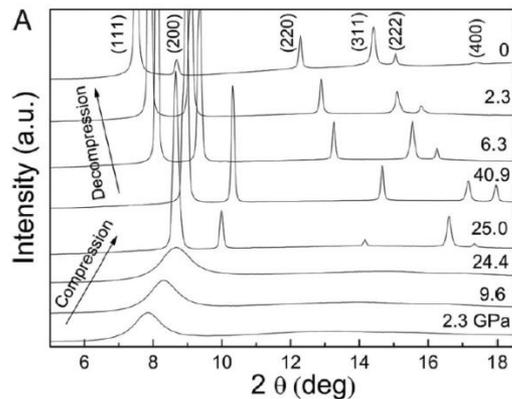
**Metallic oxygen**  
(at 133 GPa )  
[G. Weck et al PRL 102 255503  
(2009).]

Metal  $\longleftrightarrow$  Insulator  
transitions

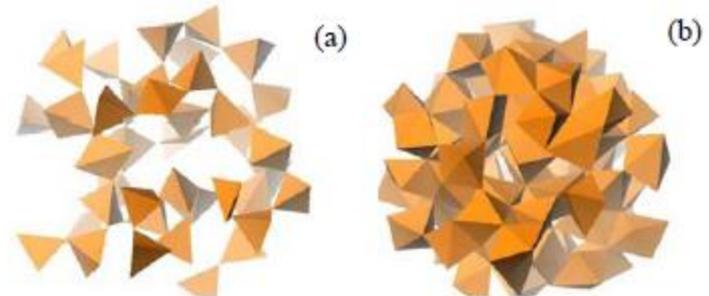
# High pressure: a tool for material science

- P can provide direct access to metastable states

- Pressure induced amorphisation (ice,  $\text{SiO}_2$ ,  $\text{ZrW}_2\text{O}_8$ ...)



- Pressure induced Crystallisation (ice, Si, Ge, BMG...)



- Pressure amorphous-amorphous transitions (ice,  $\text{SiO}_2$ ,  $\text{GeO}_2$ , Si...)

# High pressure in biology

## Pressure can also denature proteins...

INSTITUTE OF PHYSICS PUBLISHING JOURNAL OF PHYSICS: CONDENSED MATTER  
J. Phys.: Condens. Matter 18 (2006) L107–L113 doi:10.1088/0953-8984/18/7/L01

### LETTER TO THE EDITOR

## On the physics of pressure denaturation of proteins

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Online at [stacks.iop.org/JPhysCM/18/L107](http://stacks.iop.org/JPhysCM/18/L107)

### Abstract

We show that the entropic effect originating from the translational movement of water molecules plays critical roles in the pressure-induced denaturation of proteins. In our statistical–mechanical method, the partial molar volume



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- \* Fresh, untreated fruit juice transported over 1500 km
- \* A shelf life of three weeks, thanks to new technologies: Pascalisation and PurePulse®
- \* Huge logistical benefits, and more fresh products for the consumer



## Also claims (controversial) that some bacteria can survive extreme pressures (in excess of 1.6 GPa)

## Microbial Activity at Gigapascal Pressures

Anurag Sharma,\* James H. Scott,\* George D. Cody,  
Marilyn L. Fogel, Robert M. Hazen, Russell J. Hemley,  
Wesley T. Huntress

We observed physiological and metabolic activity of *Shewanella oneidensis* strain MR1 and *Escherichia coli* strain MG1655 at pressures of 68 to 1680 megapascals (MPa) in diamond anvil cells. We measured biological formate oxidation at high pressures (68 to 1060 MPa). At pressures of 1200 to 1600 MPa, living bacteria resided in fluid inclusions in ice-VI crystals and continued to be viable upon subsequent release to ambient pressures (0.1 MPa). Evidence of microbial viability and activity at these extreme pressures expands by an order of magnitude the range of conditions representing the habitable zone in the solar system.



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### fresh facts...

Why does guacamole turn brown? Avocados are very sensitive to oxygen, and just begin to turn brown after being exposed to the air.

# Pressure scale?

**SI unit for pressure: Pascal, Pa ( 1 Nm<sup>-2</sup> )**  
**i.e. Force/Area**

**Research at neutron and x-ray facilities is routinely conducted at pressures measured in GigaPascals, GPa\*.**



## **Reference**

*Atmospheric pressure ~ 0.0001 GPa*  
*Deepest point of the ocean ~ 0.1 GPa*  
*Stability field of diamond > 5 GPa*  
*Center of the Earth: ~350 GPa*



(\*I may slip into kbar = 1000 bar during talk...conversion is easy 1 GPa = 10 kbar)

# How do you generate high pressures in the lab?

Mechanical compression of gases possible since early in the industrial revolution.

Gas pressures up to  $\sim 200$  bar ( $0.02$  GPa) are common.



200-300 kPa (2-3 bar)



1.5 MPa (15 bar)



20 MPa (200 bar)

Higher gas pressures of up to  $\sim 0.5$  GPa in oil & gas industry

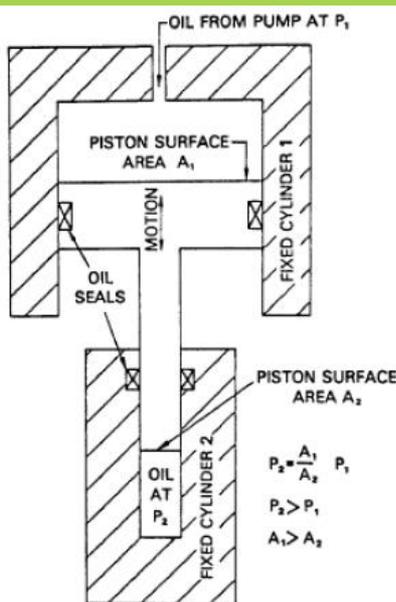
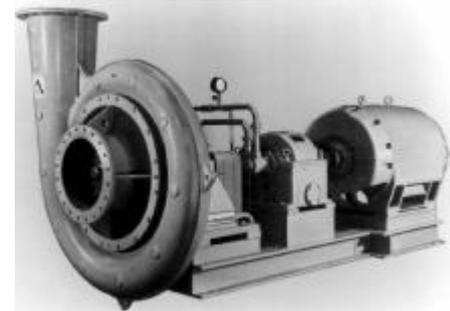
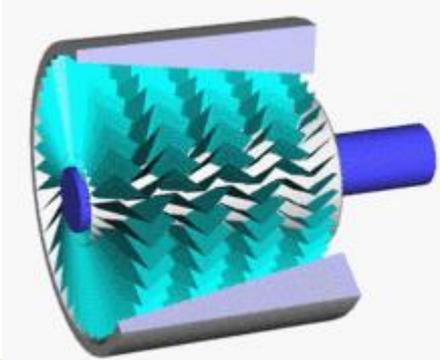
Compressing solids and liquids is much harder, and was considered impossible until early 20<sup>th</sup> century.

***What's the difference between compressing a gas and compressing a solid?***



# How do you generate high pressures in the lab?

Wide range of gas compressors (see e.g. [http://en.wikipedia.org/wiki/Gas\\_compressor](http://en.wikipedia.org/wiki/Gas_compressor))



For highest gas pressures - one dominant technique: **the piston cylinder.**

$$P = F/A$$

- Pressure,  $P_1$  applied to Area,  $A_1$
- This generates force,  $F = P_1 * A_1$
- This force is applied to smaller area,  $A_2$
- Generating pressure  $P_2 = F/A_2 = P_1 * A_1/A_2$

**The greater the pressure, the simpler the device**

# Going beyond the piston-cylinder

How about solids? Can they be compressed using a piston-cylinder?

Yes...Maximum pressures of  $\sim 2$  GPa are relatively routine (max  $\sim 5$  GPa)...this is already enough to compress some solids (e.g.  $\sim 15$  ice phases below 3 GPa – by rearranging molecules)

**But a radically different design was required to go to higher pressure.**

This came courtesy of Percy Bridgman in the early 1900's  
(and subsequently earned him a Nobel Prize)



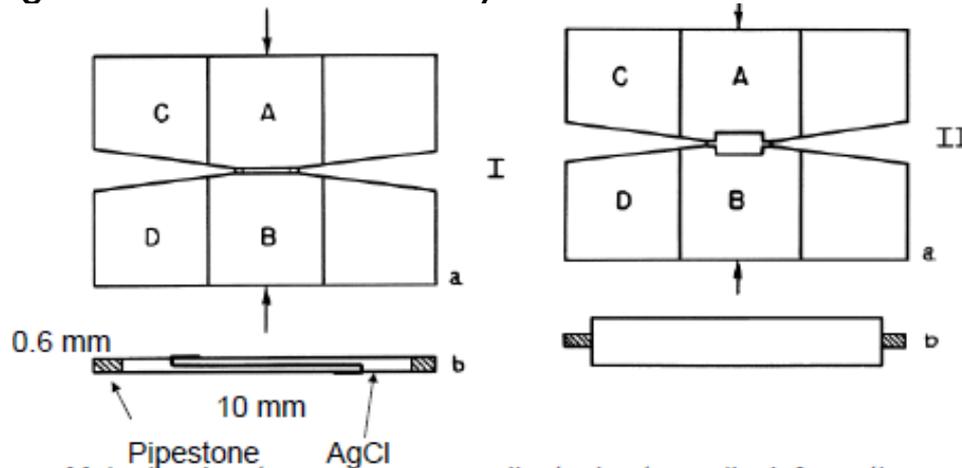
P. W. Bridgman  
1882-1961

*“You, Mr. Bridgman, have succeeded in doing what was once considered impossible. By the use of new alloys and by other ingenious devices you have been able figuratively speaking, to bring into your laboratory parts of the interior of the earth or of other places where no human being is able to exist, and you have been able there to examine the physical and chemical properties of a quantity of different substances under the enormous pressures you have created. You have thus been able to reveal a number of strange phenomena in the behaviour of matter under other circumstances than those which we consider to be normal.”*

- Sigurd Curman, President Royal Academy of Sciences,  
Prior to presenting Bridgman's Nobel Prize in physics 1946

# “Stuck between a hard place and a hard place”

Bridgman’s insight was a technique based around an opposed anvil design – with it he eventually reached  $\sim 40$  GPa



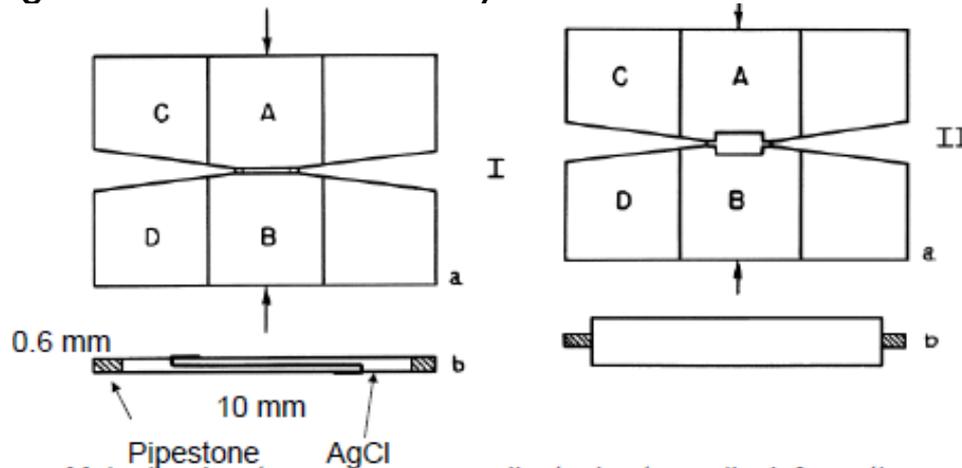
## Three elements of the opposed anvil technique:

- 1) Two anvils made of a hard material
- 2) A force to push these anvils together
- 3) A gasket made of a material that is strong, but able to flow

**These same principles apply to the majority of high-pressure cells operating today above  $\sim 2$  GPa at synchrotron and neutron sources.**

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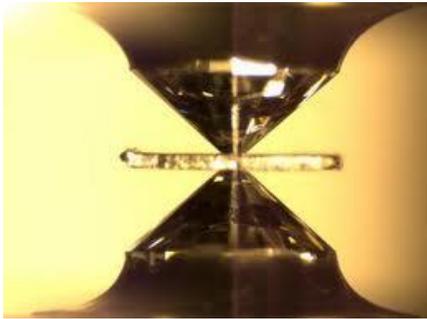
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# “Stuck between a hard place and a hard place”

What is a hard material? Bridgman used a composite of WC and cobalt. Other materials used are pure WC, sapphire ( $\text{Al}_2\text{O}_3$ ), moissanite ( $\text{SiC}$ ), c-BN...



But in almost all cases, the best material is diamond.

Diamond anvils are either single-crystal or poly-crystalline. PCD available (sintered, typically with Co binder). Also in last 10 years ultra-hard nano-PCD (HIME-DIA)



**Three elements of the opposed anvil technique:**

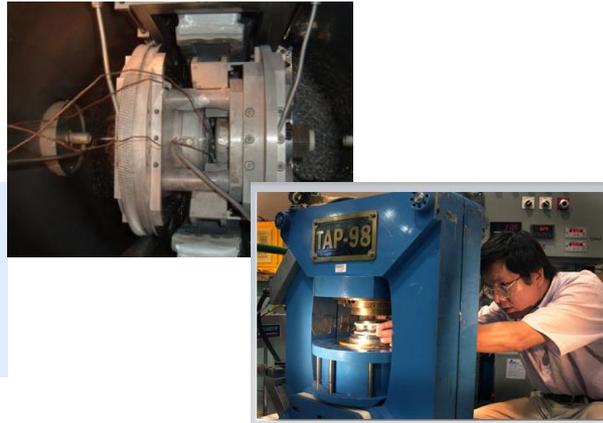
- 1) Two anvils made of a hard material**
- 2) A force to push these anvils together
- 3) A gasket made of a material that is strong, but able to flow

# “Stuck between a hard place and a hard place”

The amount of force (and how it's applied) depends on the area of the sample and the required pressure



X-ray cells (<1 tonne)  
(screw, membrane,  
piezo actuator)



“conventional” neutron cells 150-500  
tonnes (hydraulic presses)



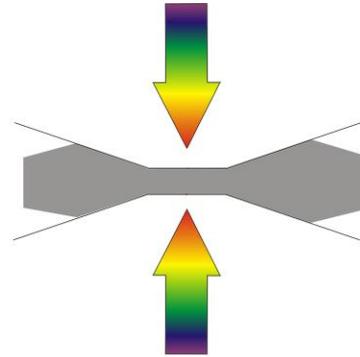
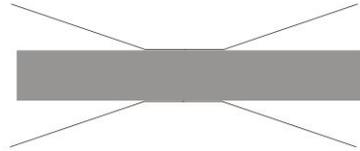
Multi-anvil 1000-6000  
tonnes

## Three elements of the opposed anvil technique:

- 1) Two anvils made of a hard material
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- 3) A gasket made of a material that is strong, but able to flow

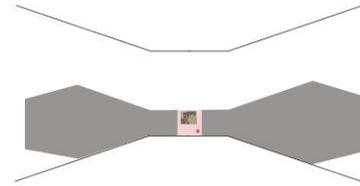
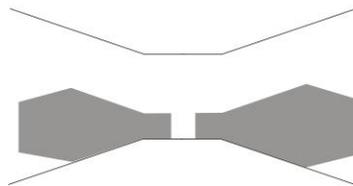
# “Stuck between a hard place and a hard place”

1) Gasket (typically metal, but can also be composite material )



2) Apply force to ‘indent’ gasket:  
•Work hardens gasket  
•Forms support for diamond tips  
•stable geometry (thin)

3) drill hole for sample  
(for DAC’s need EDM or laser as hole is very small)



4) Load sample,  
pressure calibrant\* and  
pressure medium\*

## Three elements of the opposed anvil technique:

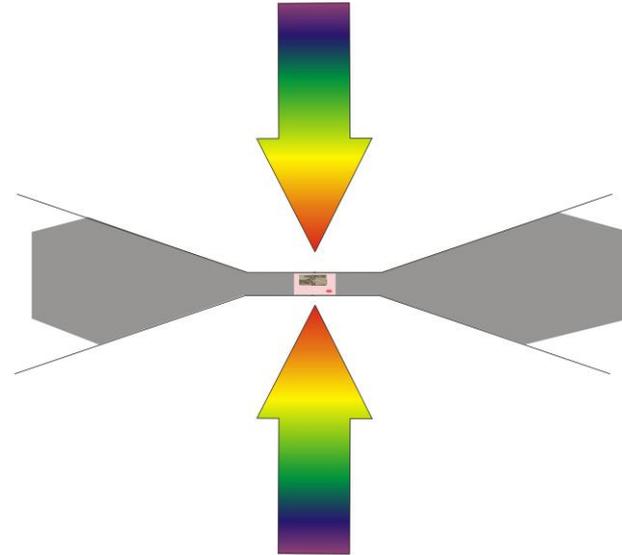
- 1) Two anvils made of a hard material
- 2) A force to push these anvils together

**3) A gasket made of a material that is strong, but able to flow**

# “Stuck between a hard place and a hard place”

Seal cell by applying further force.  
As gasket can flow, it follows pressure gradient, moving away from sample.

In process, thinning and reducing volume available to sample – increasing pressure.



## Three elements of the opposed anvil technique:

- 1) Two anvils made of a hard material
- 2) A force to push these anvils together
- 3) **A gasket made of a material that is strong, but able to flow**

# Pressure measurement

As with any experiment, accurate knowledge of the variable you control is very important.

Pressure is measured the same way any other variable is:

**Calibrate something with a physical response to variable of interest**

Example 1) **Ruby fluorescence.**

- Probably the most ubiquitous pressure sensor above 2 GPa
- Under laser light, ruby fluoresces with particular spectrum
- The wavelength (colour) shifts in a known way with pressure

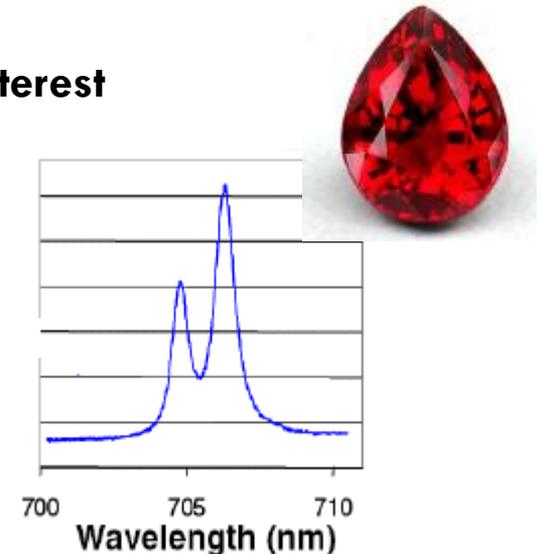
Example 2) Known equation-of-state of calibrant

- If ruby isn't an option (opaque anvils, high temperature, reactivity)
- Can load a secondary sample with a known pressure-volume relation. Use diffraction to determine volume – and, therefore, pressure.

Others... raman shift of  $C^{13}$ , pressure-load curves, ...

**How are the calibrants calibrated?**

Typically shock wave data (discussed later) can give a direct equation of state.



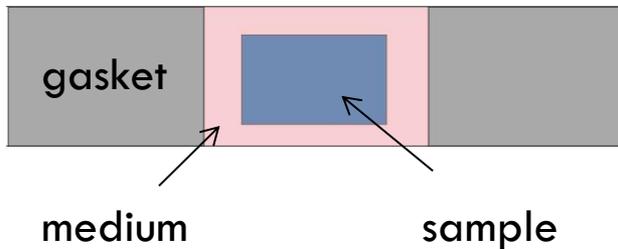
# Pressure media

Imagine hard sample directly squeezed between two diamonds...



Results in enormous strain (often many GPa)

Solution is to surround the sample with a medium that is very soft...



- Because medium is soft, it can't sustain a P gradient
- Sample feels equal pressure on all sides
- Fragile single-crystals, bio-samples can be compressed
- Best media are the inert gases: He, Ne, Ar
- Methanol:ethanol, silicon oil, fluorinert also used  
(Need medium that doesn't react with sample)

# Beyond two opposed anvils

For large volumes, an alternative technique uses multiple (typically 6-8) anvils.

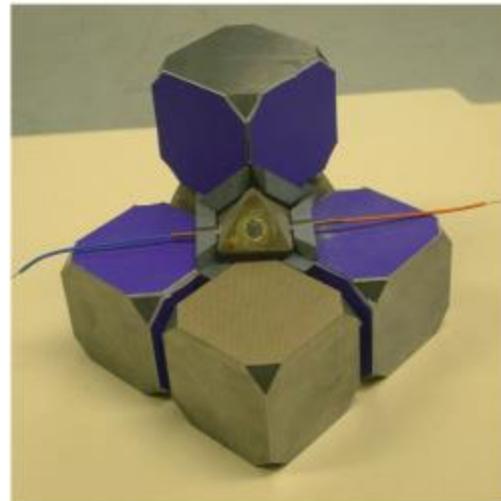
Well suited for liquid/glass diffraction studies, tomography, element partitioning studies...

Elements:

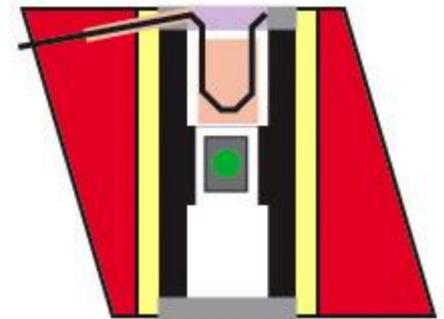
- Usually uniaxial force (from very large capacity press (+1000 tonnes) but 6 axis presses exist
- 6 anvils with square faces come together to form a cubic sample volume
- 8 anvils – cubes with corner cut off - form octahedral sample space. This assembly can be pressed inside 6 regular anvils (double-stage design)

Sample space is typically filled with:

- Gasket
- Thermal insulation
- Graphite Heater
- Contacts for thermocouples/heater
- Pressure medium/sample encapsulation



8 cubes (5 shown) press on octahedron  
Gaskets are made of pyrophyllite



Cross section of octahedron shows:  
MgO pressure medium (red)  
ZrO<sub>2</sub> insulator (yellow)  
stepped LaCrO<sub>3</sub> heater (black)  
Mo rings (grey)  
MgO insulating parts (white)  
Sample (grey/green)  
Thermocouple wire

2-stage design with PCD anvils, can reach ~80 GPa

# Dynamic compression

Completely different route to achieve highest pressures is via dynamic techniques:

Shockwaves can generate exceptionally high P & T over short time period:

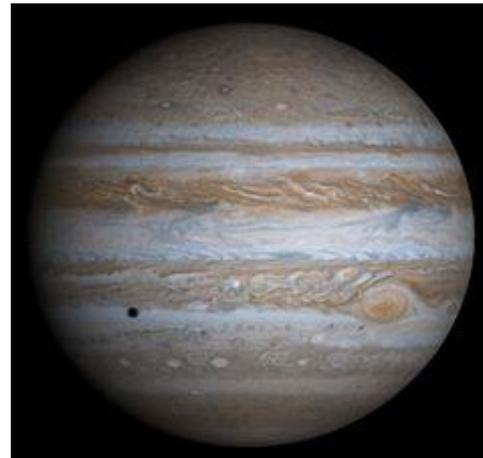
- Nuclear
- Gas gun
- Lasers (NIF)



Under shock, samples experience conditions that lie on a locus in PT space called “Hugoniot”

**At NIF expect to reach TPa and  $10^4$  K regime (Centre of Jupiter)**

Alternative techniques using Piezo actuators can look at dynamic phenomena.



# Science at high pressure

Have looked at ways to generate, control and measure extremely high pressures.  
In order to conduct *science*, need way to probe effect of pressure on sample material

Great variety of probes:

## Visual observations

Phase transitions (solid-solid, melting, conductivity), single-crystal growth

## Laser-based

Raman, UV & IR spectroscopy  
Brillouin

## Transport

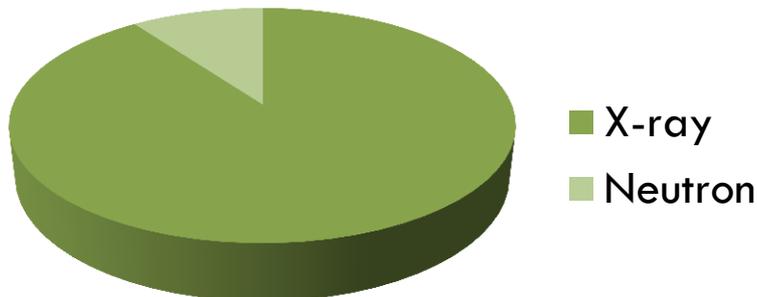
**measurements**  
Electrical conductivity, magnetic susceptibility

## Others...

sound velocity, DTA...

**Focus here on synchrotron x-ray and neutron based probes**

## Variety of techniques



- Above 0.6 GPa, neutrons limited to diffraction, phonon measurements, tomography.
- In contrast, huge (and rapidly expanding) range of synchrotron x-ray techniques: (XRD, XAS, XMCD, XRS, XES, IXS, NRIXS, transmission density, tomography...)

# Neutrons have many unique capabilities

## 1) Scattering length is not linearly dependent on atomic number

- neutron diffraction is a great tool for studying light atoms. It's the only technique that can precisely locate protons (deuterons), Be, B<sup>11</sup>, C, N, and O are strong scatterers

- possibility of negative scattering lengths (e.g. H) means specific pair correlations can be removed

-isotopic substitution can greatly enhance contrast and can also simplify analysis of non-crystalline matter.

## 2) Absorption cross sections also not linearly dependent on atomic number:

Li<sup>6</sup>, H, B<sup>10</sup> are strong absorbers. Pb is transparent.

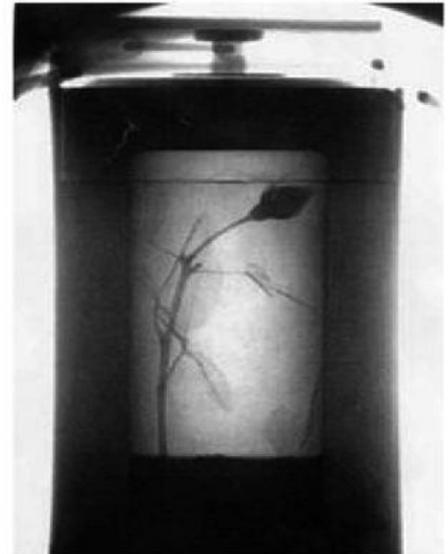
## 3) Neutrons have an intrinsic magnetic moment

- They are scattered by nuclear spins and sensitive to magnetic order.

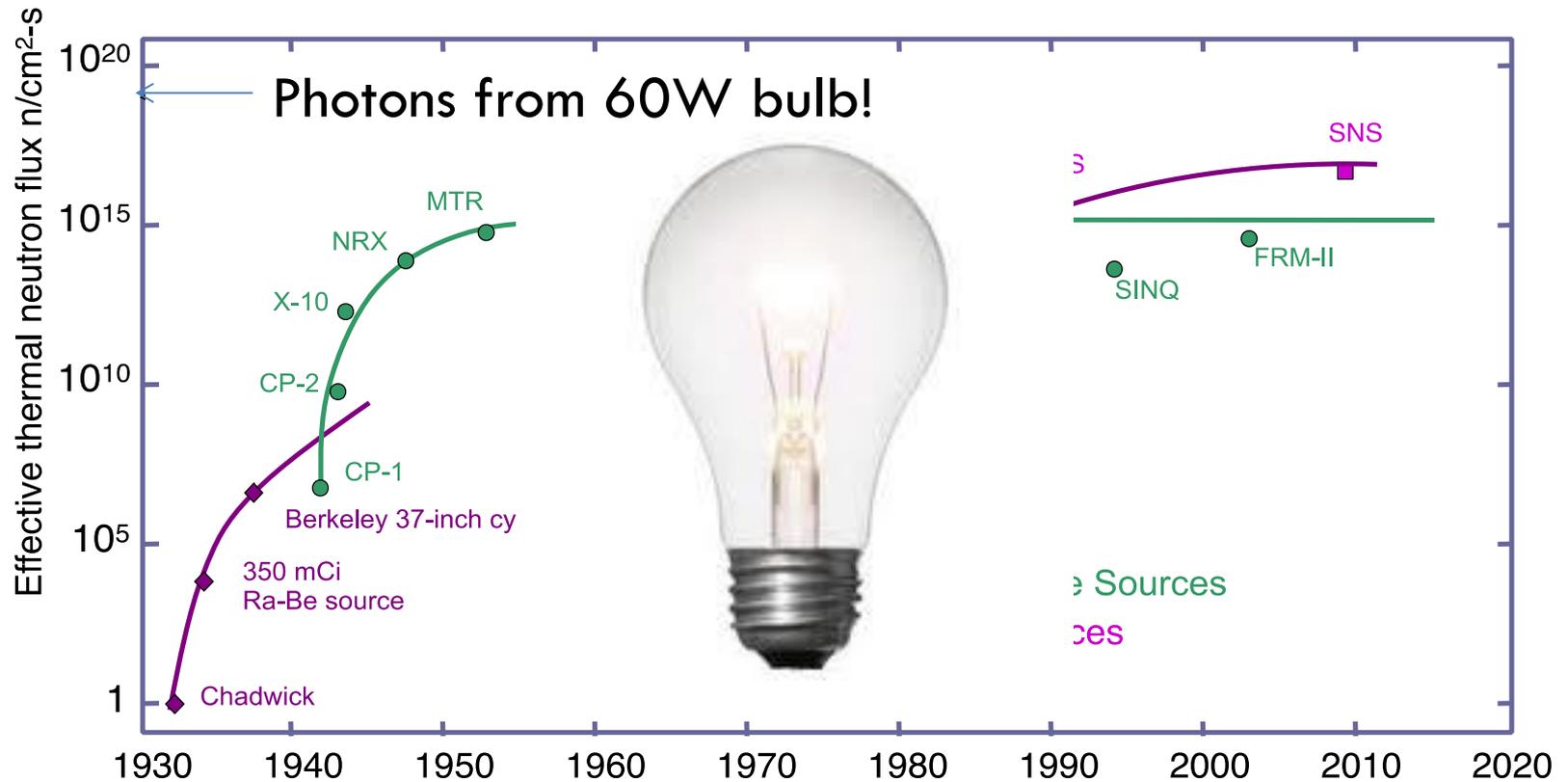
## 4) Scattering is via inter-nuclear interactions. Pointlike.

- No atomic form factor, so high Q-vectors are accessible.

Leading to exceptional real-space resolution.



# Neutrons have one major disadvantage



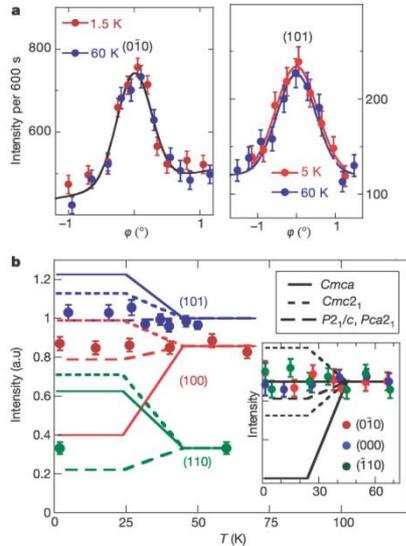
(Updated from *Neutron Scattering*, K. Skold and D. L. Price, eds., Academic Press, 1986)

This is a challenge for high pressure because samples are small...

# Examples of High-Pressure Neutron science

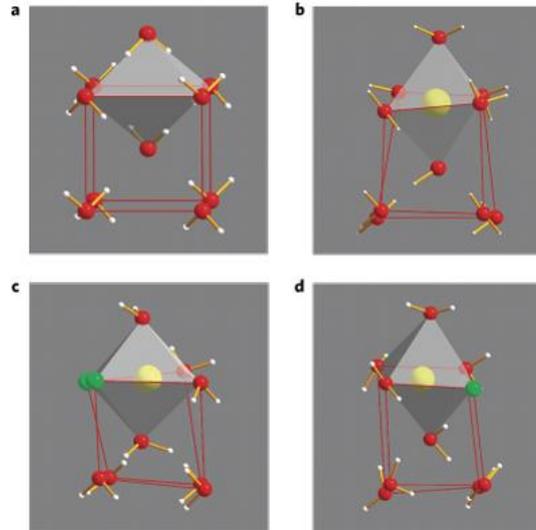
## Broken symmetry in hydrogen

Goncharenko & Loubeyre Nature (2005).



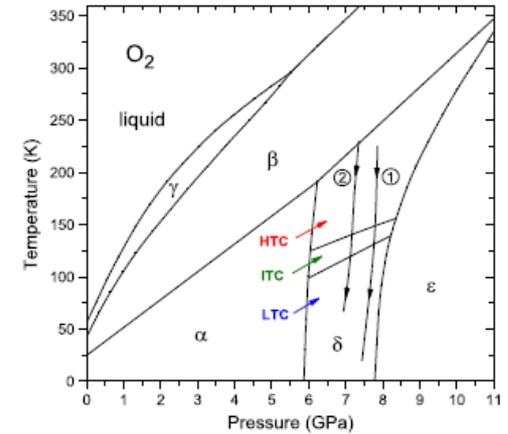
## Salty Ice VII

Klotz et al Nature Physics (2009).



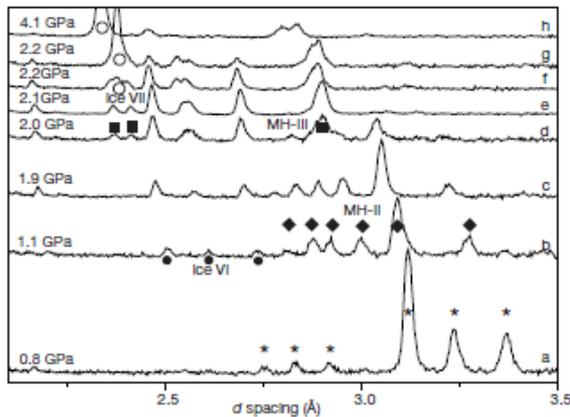
## Magnetic ordering in solid O<sub>2</sub>

Klotz et al PRL (2010).



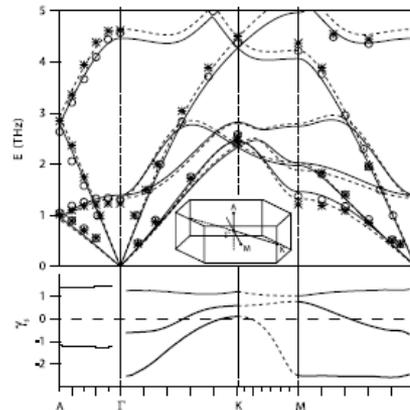
## Stability of methane hydrate

Loveday et al Nature (2001).



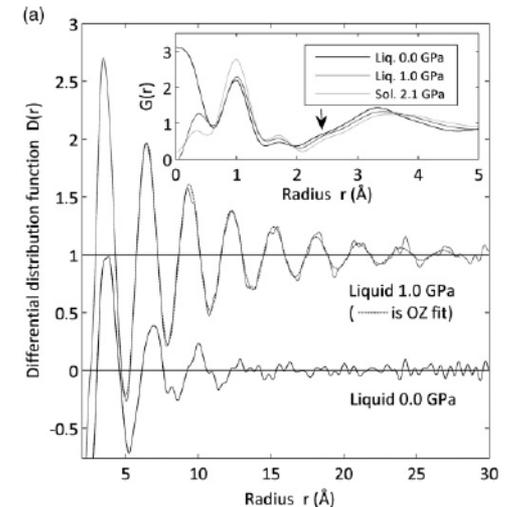
## Phonon dispersion in ice Ih

Strassle et al PRL (2004).



## Structure of liquid ammonia

Guthrie et al PRB(2012).



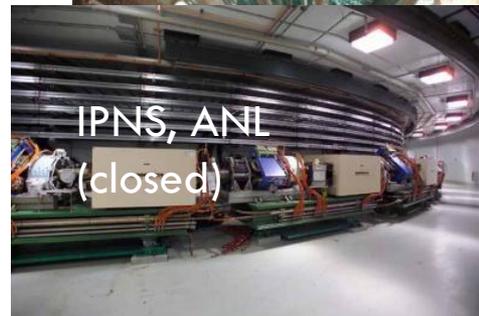
# Neutrons science at high pressure

Mature neutron facilities with HP programmes:

## Europe



## US



- Typical max P ~ 25 GPa
- Max T ~ 1500 K
- Min T ~ 4 K

# Neutrons science at high pressure

New neutron facilities with HP programmes:



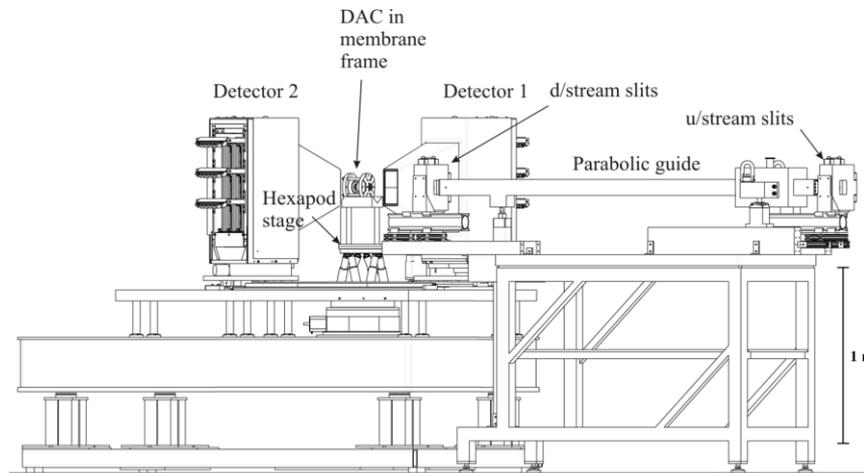
JPARC, Japan



SNS, Oak Ridge

- Typical max pressure ~ ? GPa
- Max temperature ~ ? K
- Min temperature ~ ? K

# SNAP – high pressure at the SNS



- Highly pixelated area detectors (Anger cameras) give simultaneous access to large volumes of reciprocal space.
- Movable detectors mean wavelength coverage can be swept from low to high Q-vectors (or high to low d-spacing)
- Moveable flight tube can be replaced with different focusing optics (Elliptical or KB).
- Precise (1  $\mu\text{m}$ ) stage permits alignment of very small samples.
- Highly versatile diffractometer: can study single-crystals, powders or even liquid structure

# SNAP – high pressure at the SNS

Conventional HP neutron sample volumes are  $\sim 100\text{mm}^3$  and require 200-500 tonne presses



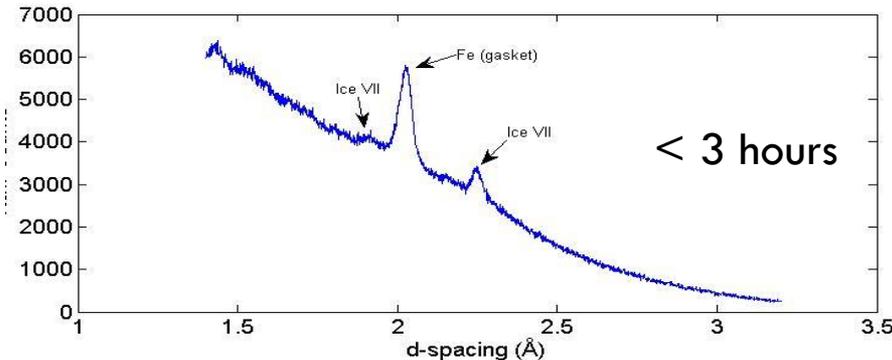
X-ray cell to scale



But the intense flux at SNS means samples can be small

Is it possible to do neutron work with a DAC?

At NXS 2011,  
showed this  
dataset from  
SNAP.



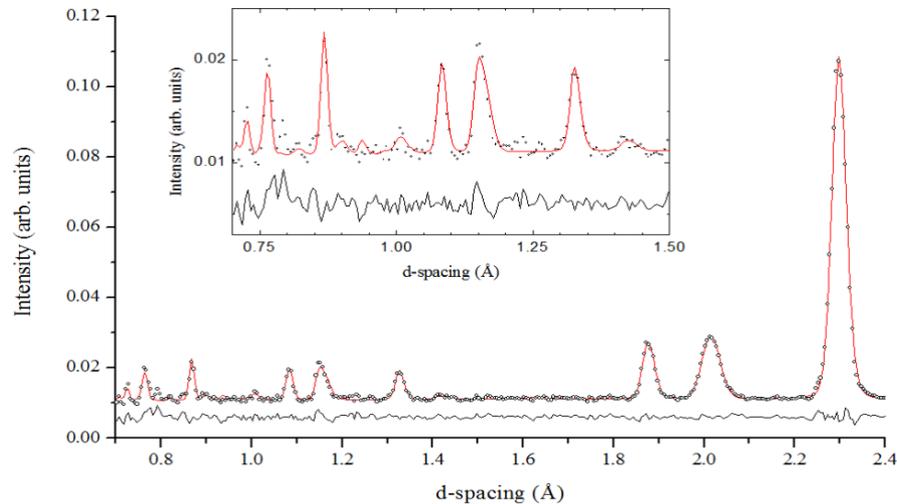
$\text{D}_2\text{O}$  Ice VII (8 GPa)  
 **$0.01\text{mm}^3$**   
(in diamond anvil cell)

And claimed...

“high-quality diffraction data up to 60 GPa in 1-2 years”

# SNAP – high pressure at the SNS

After 2 years of development, have huge improvement in data quality...



$$\chi^2 = 1.49$$

First real structural measurements with neutrons at pressures up to 50 GPa...

ASPNAS

## Neutron diffraction observations of interstitial protons in dense ice

Malcolm Guthrie<sup>a,1</sup>, Reinhard Boehler<sup>a</sup>, Christopher A. Tulk<sup>b</sup>, Jamie J. Molaison<sup>b</sup>, António M. dos Santos<sup>b</sup>, Kuo Li<sup>a</sup>, and Russell J. Hemley<sup>a,1</sup>

<sup>a</sup>Geophysical Laboratory, Carnegie Institution of Washington, Washington, DC 20015; and <sup>b</sup>Neutron Sciences Directorate, Oak Ridge National Laboratory, Oak Ridge, TN 37831

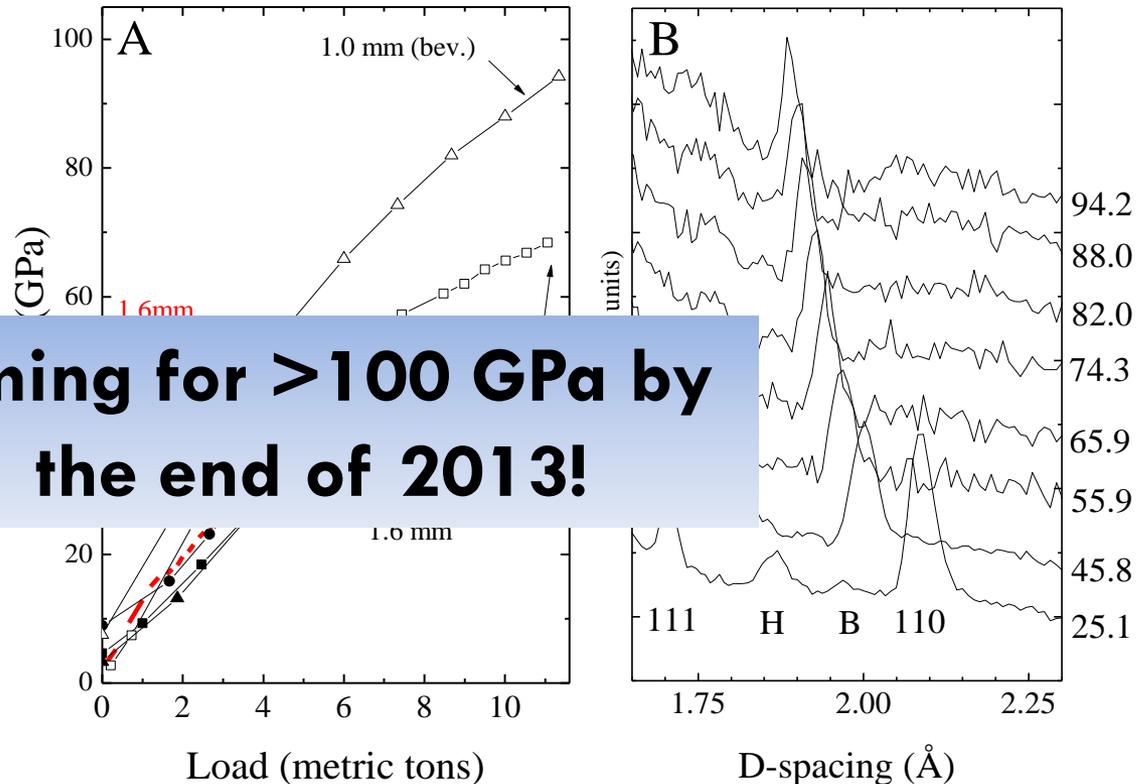
Contributed by Russell J. Hemley, May 16, 2013 (sent for review April 22, 2013)

The motif of distinct H<sub>2</sub>O molecules in H-bonded networks is believed to persist up to the densest molecular phase of ice. At even higher pressures, where the molecule dissociates, it is generally assumed that the proton remains localized within these same networks. We report neutron-diffraction measurements on D<sub>2</sub>O that reveal the location of the D atoms directly up to 52 GPa, a pressure

in the molecular geometry up to a reported 20 GPa (19) (see comment in *Materials and Methods*). A lack of in situ neutron-diffraction data above this pressure has precluded direct measurement of the D locations on the approach to symmetrization. By developing a supported diamond-anvil cell (S-DAC) (12) and coupling this with the intense radiation flux at the dedicated high-

# SNAP – high pressure at the SNS

As of Aug 2013 got to ~94 GPa!



**Aiming for >100 GPa by  
the end of 2013!**

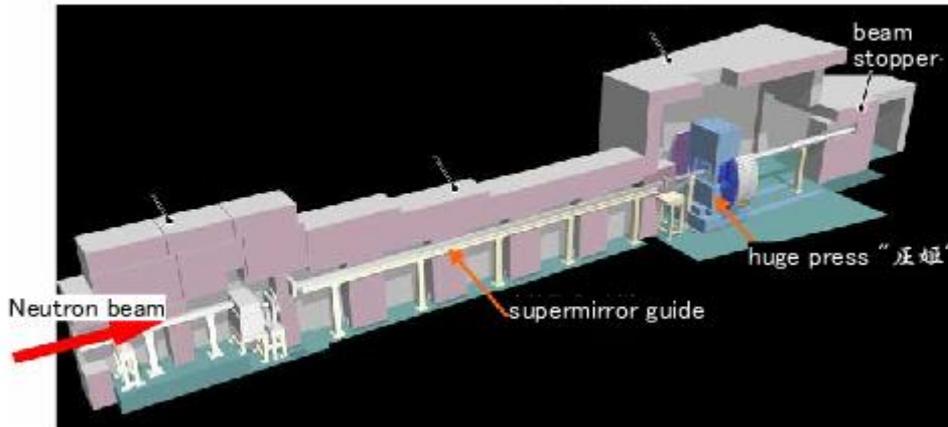
This breakthrough is not only about high pressure.

Low T, High T, gas loading, *in situ* spectroscopy,...a more synchrotron-like neutron experience.

# PLANET and the “Pressure Princess”

J-PARC Japanese spallation source (design spec of 1 MW) *is operational*

Multi-anvil based HP neutron beamline: PLANET



Beam scientists, Dr. Sano (left) and Dr. Hattori in front of the high-pressure device "ATSUHIME". "ATSU" means "pressure" and "HIME" means "princess" in Japanese.

Bad luck...

Mar 2011 – Fukushima earthquake set back construction

May 2013 – Accident at Hadron Facility resulted in shut-down

Once fully operational will be competitive with SNS

# Future Neutron Sources



**Construction start 2014**  
First neutrons <6 years



**Begin construction 2020?**  
“Cold” neutron source

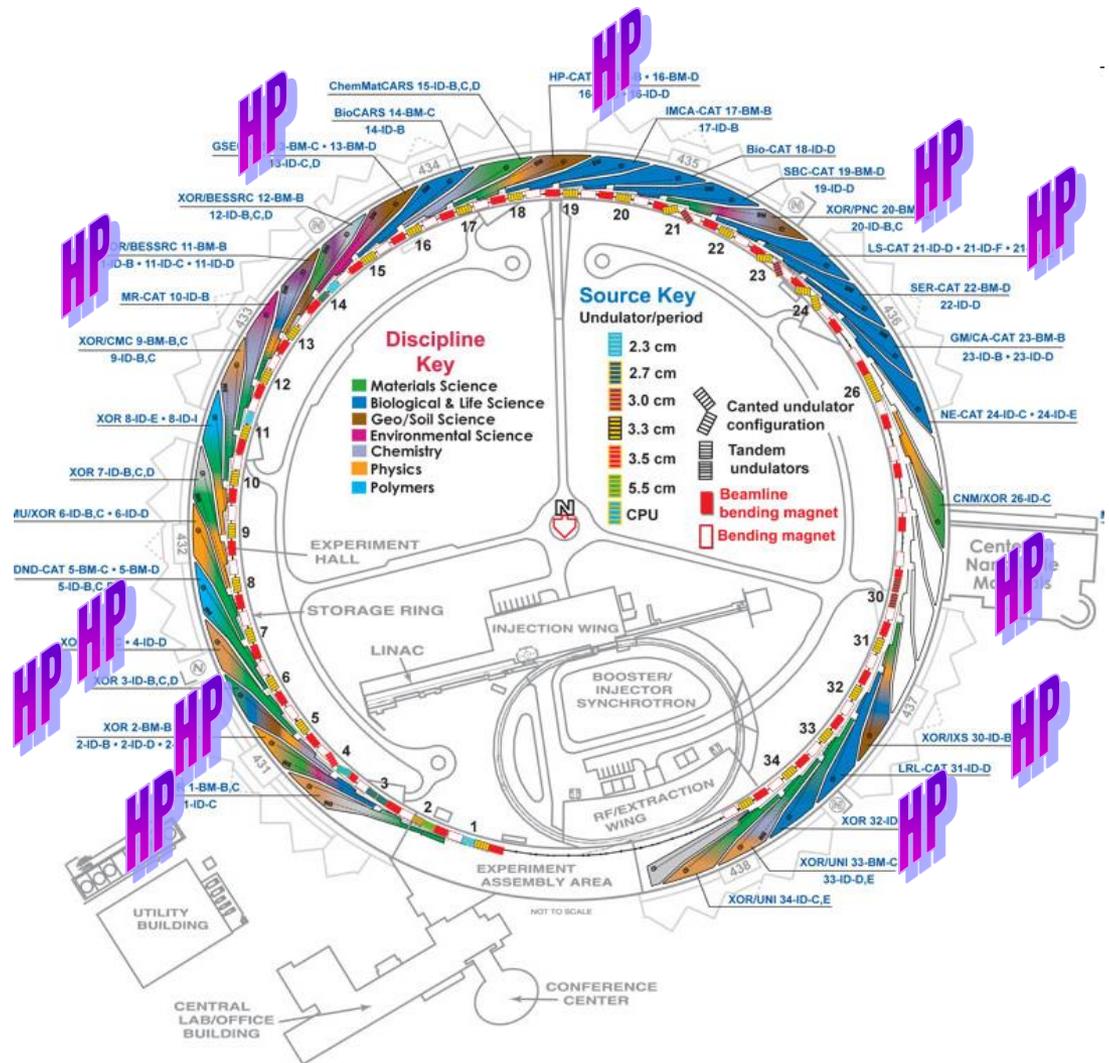
**Bright future for high pressure neutron science!**

# X-ray science at high pressure

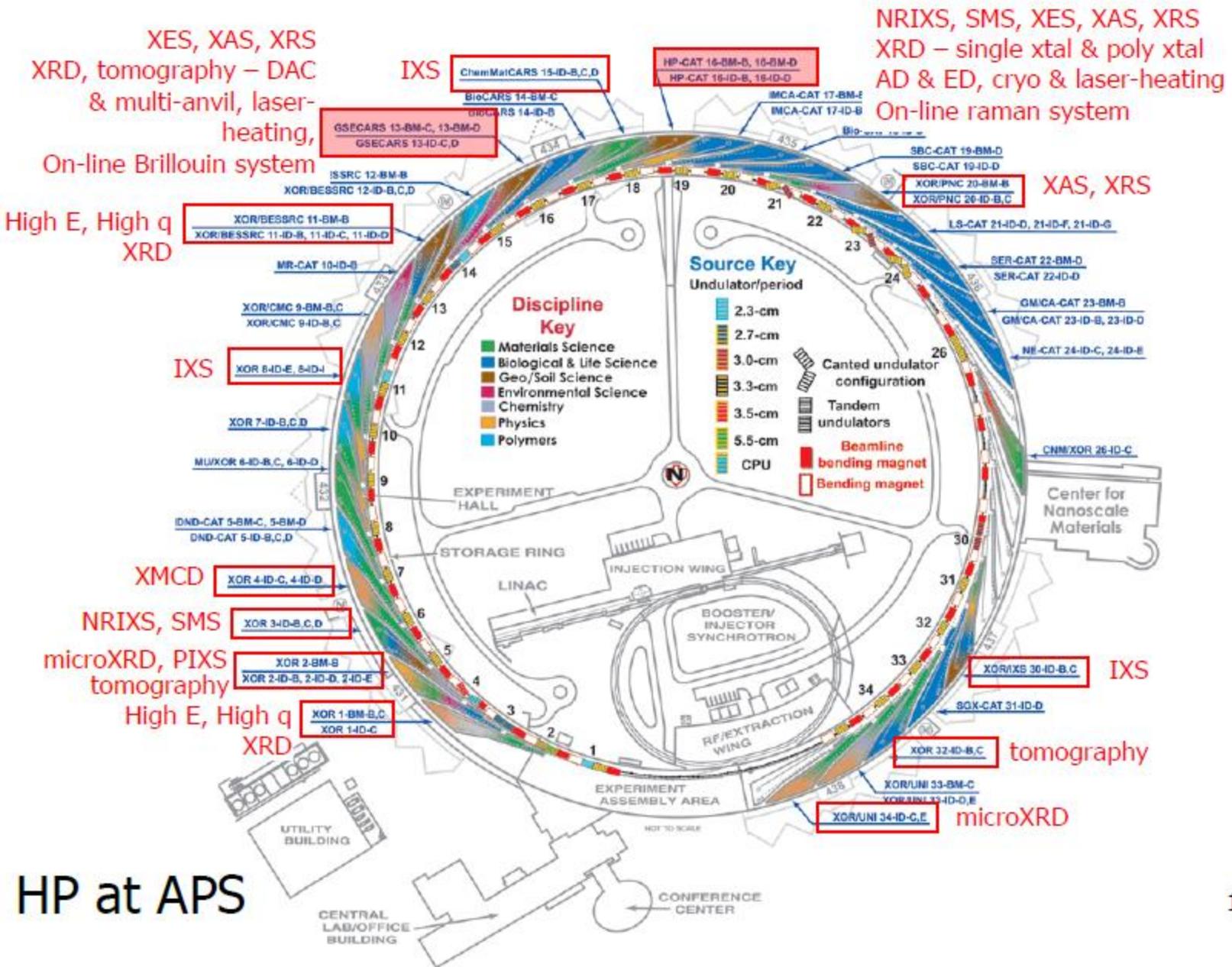
Access to high pressure at synchrotron sources has exploded in last 10 years

All major synchrotrons have dedicated high pressure beamlines (e.g. ESRF, APS, SPring-8, Petra-III, NSLS, NSLS-II (proposed). Extreme conditions are an integral part of the (ongoing) APS upgrade.

Beyond dedicated beamlines...portable high pressure apparatus are extremely wide-spread.



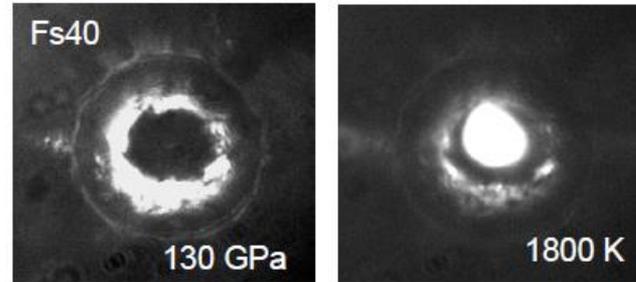
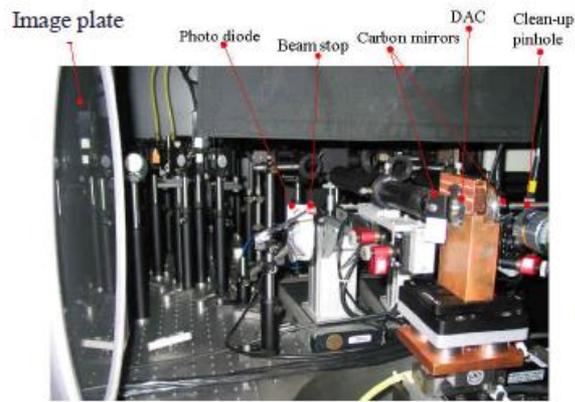
With few exceptions almost all synchrotron techniques you'll hear about in NXS2013 can be combined with high pressure



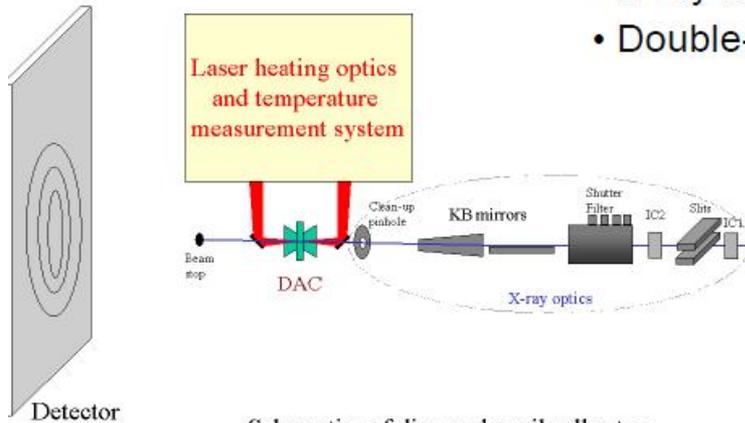
# High-pressure diffraction with x-rays

Modern HP beamlines deliver extremely intense, low divergence beams 2-5 $\mu\text{m}$

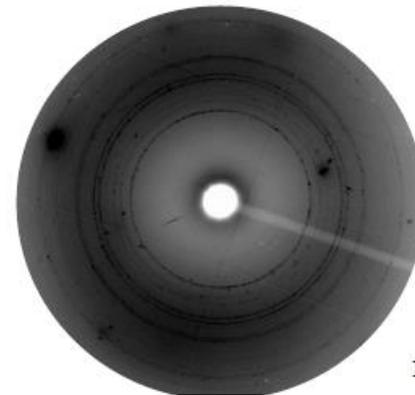
Coupled with laser heating – can reach  $>300$  GPa and  $>3000$  K



- Gasket hole  $\sim 60$   $\mu\text{m}$  (culet =  $150$   $\mu\text{m}$ , bevel diameter =  $300$   $\mu\text{m}$ )
- X-ray beam  $\sim 6 \times 7$   $\mu\text{m}$
- Double-sided Nd:YLF laser heating



Schematics of diamond anvil cell setup at 13ID-D at the Advanced Photon Source

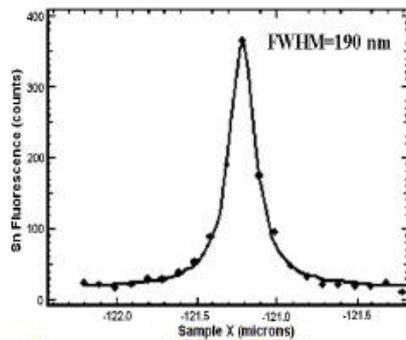


# Nano-probe diffraction at high P

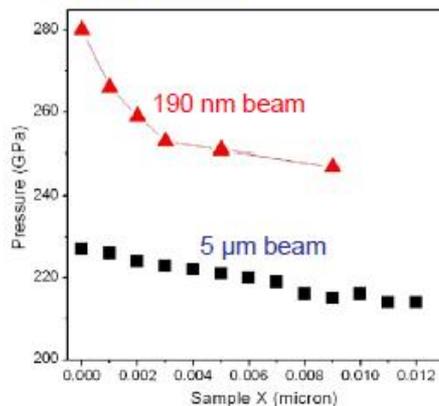
Beams orders of magnitude smaller than neutrons permit sub-micron studies  
(could be route to TPa pressures?)

Using 200 nm focused x-ray beam we can...

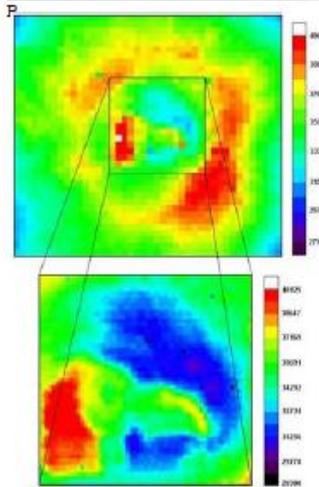
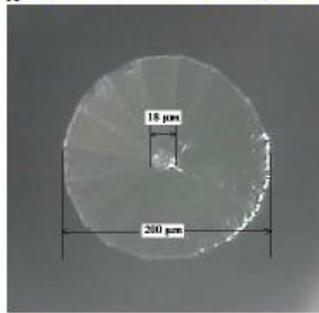
L Wang , PNAS (2010)



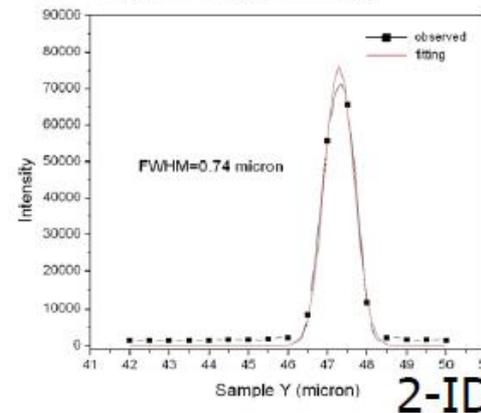
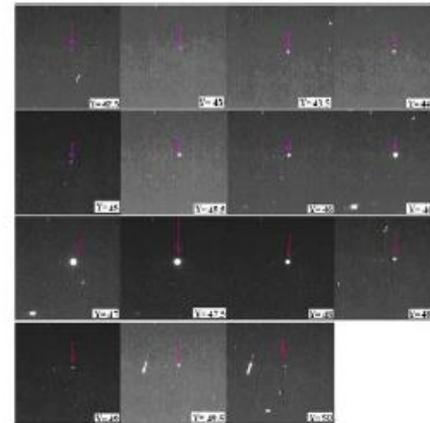
Observe 20 GPa/ $\mu\text{m}$  gradient & peak-pressure in 1- $\mu\text{m}$  area



Separate submicron Pt, Re, Fe samples



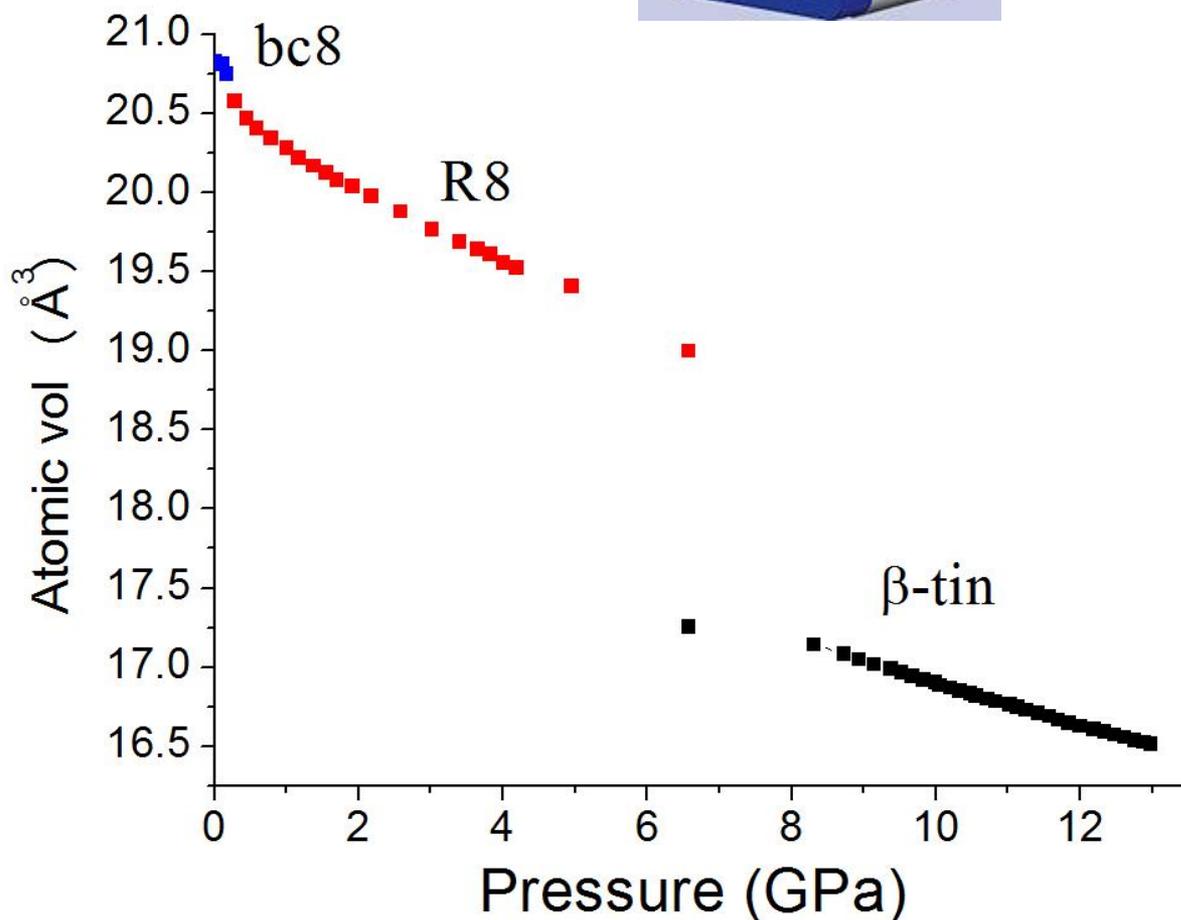
Conduct single-crystal XRD on submicron powder



# Time-resolved diffraction at high P

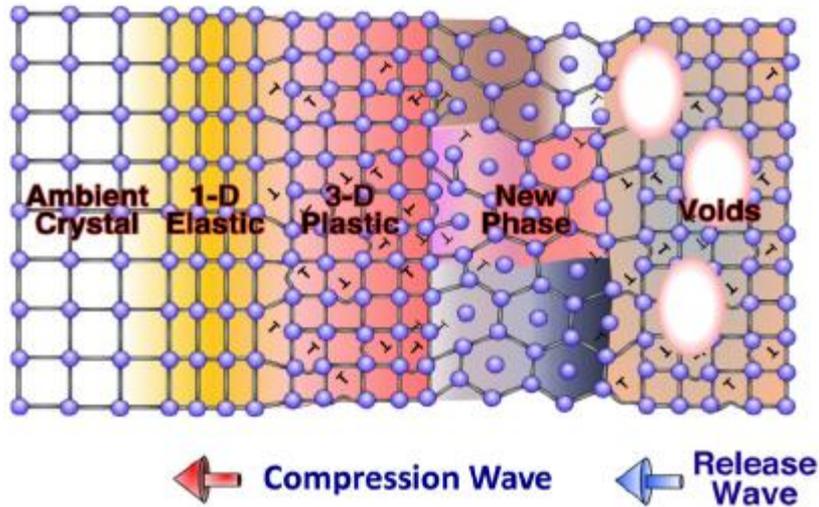
Add small “t” to variables

- Continuous exposures
- Rietveld refinement every 7 ms
- Structural information throughout transitions



# Shocking Diffraction

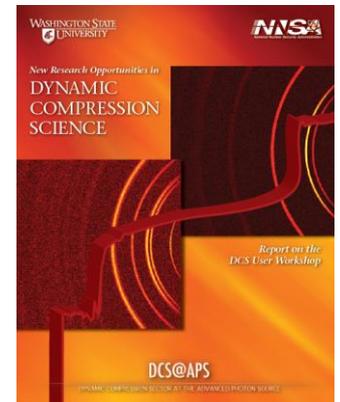
Very rapid acquisition (ns) facilitates measurements *during shock wave*.



- Phase transitions
- Deformation processes and fracture dynamics
- Dynamics of chemical reactions

DCS is a facility at the APS to study structure and dynamics in these thermo-mechanical extremes.

MOU signed late 2012 for construction at Sector 35

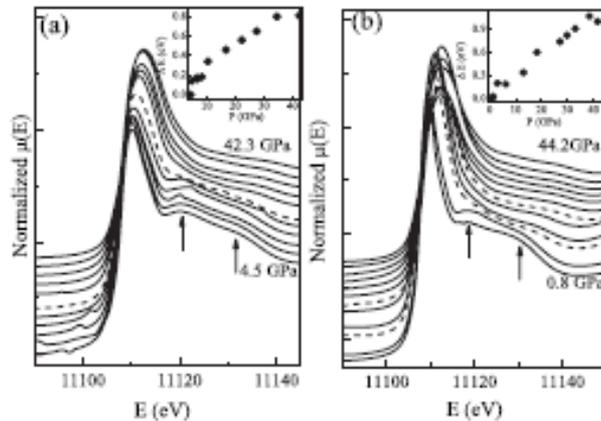


<http://www.dcs-aps.wsu.edu/>

# X-ray absorption spectroscopy (XAS)

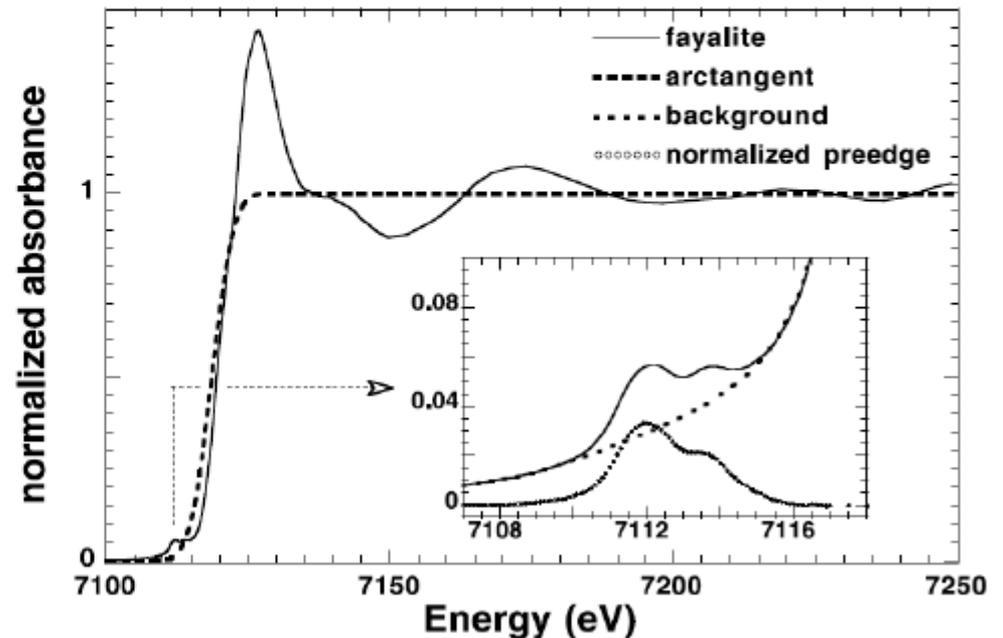
Direct insight into local structure and bonding environment

- Pre-edge position and intensity: oxidation state
- Edge height: concentration
- XAFS: coordination & structure



Coordination change in GeO<sub>2</sub> glass measured with XANES

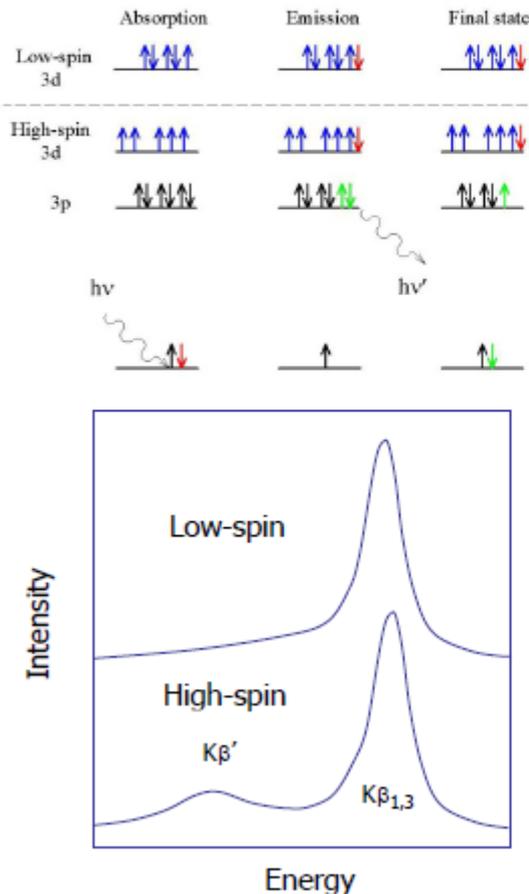
Baldini et al PRB (2010).



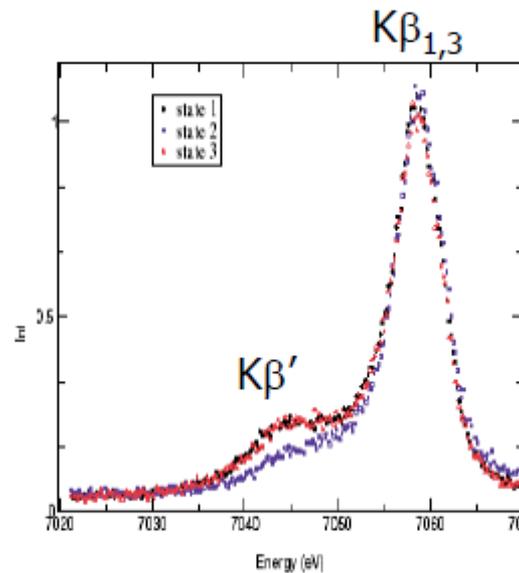
Wilke et al, Amer. Min. 2001

# High-pressure x-ray emission spectroscopy (XES)

Observations of high spin-low spin transitions in 3d elements

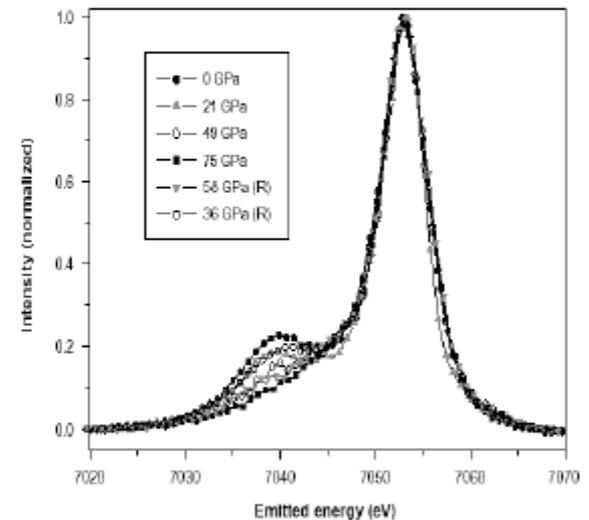


FeO & Fe<sub>2</sub>O<sub>3</sub>



Badro *et al*, PRL 1999  
Badro *et al*, PRL 2002

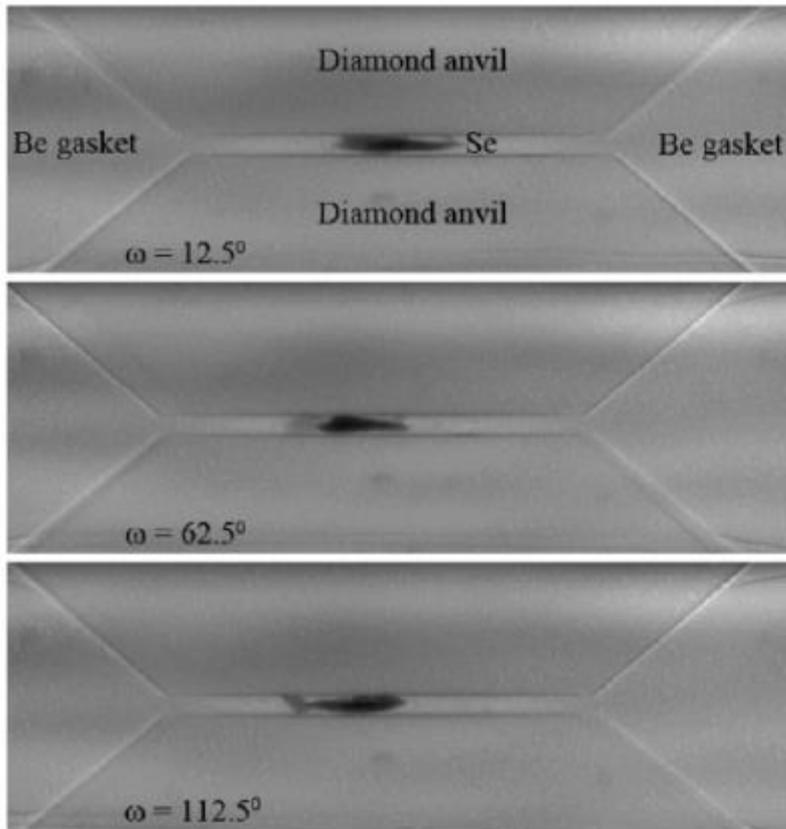
(Fe,Mg)O & (Fe,Mg)SiO<sub>3</sub>



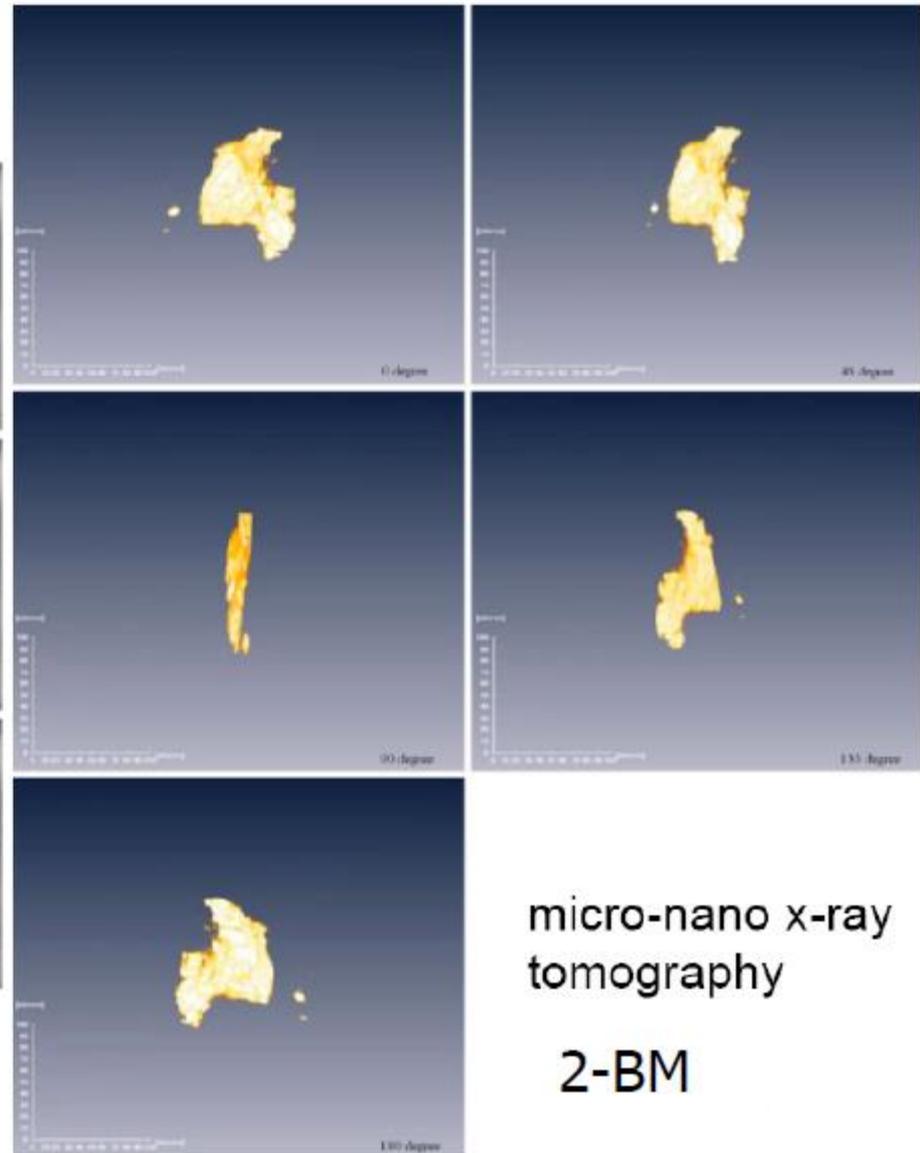
Badro *et al*, Science 2003  
Badro *et al*, Science 2004  
Li *et al*, PNAS 2004  
Lin *et al*, Science 2007  
Lin *et al*, Nature Geo. 2008

# Micro-tomography

Accurate volume measurement of amorphous Se at high pressures



Liu *et al*, *Proc. Nat. Acad. Sci.*  
**105**, 13229 (2008)



micro-nano x-ray  
tomography

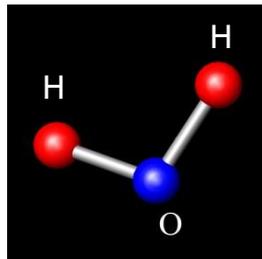
2-BM

Resolution above is  $\sim 1 \mu\text{m}$ . Using TXM techniques 20nm is possible.  
Also, coherent diffraction imaging has been used to image strain dist. In gold nano-particles

# Combining X-rays and Neutrons



Example: H<sub>2</sub>O

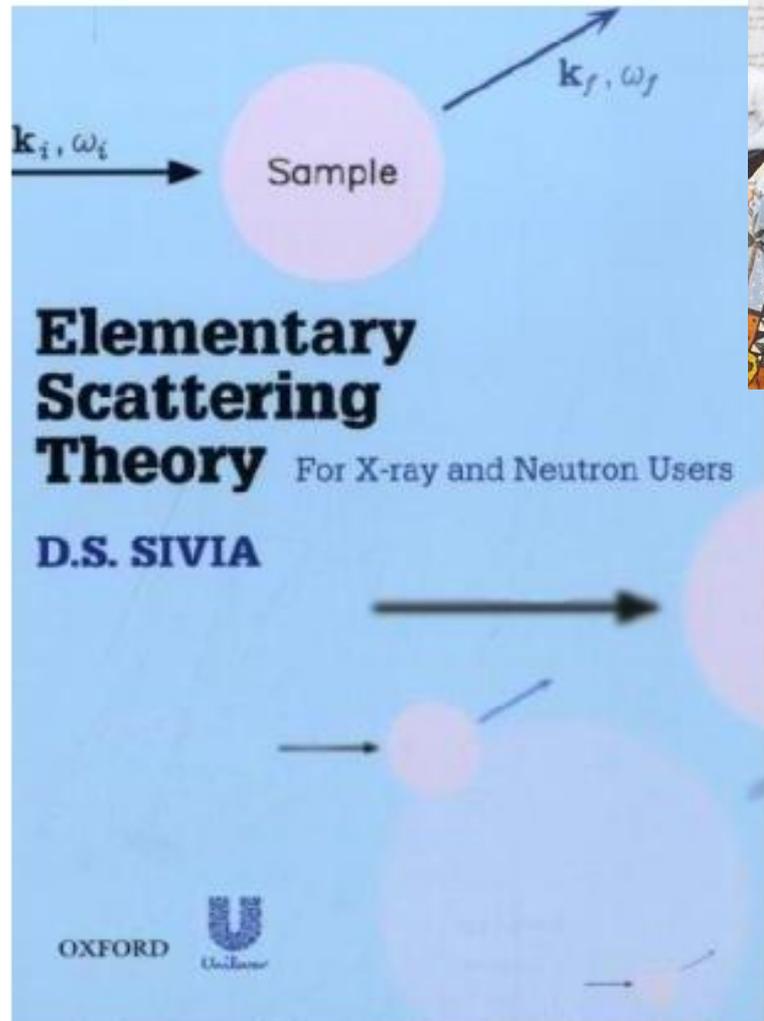


Partial	Neutron	X-ray (Q = 0)
O-O	9%	64%
O-H*	42%	32%
H-H*	49%	4%

As neutron capabilities increase, there's great scope for complementary experiments

# Summary

- Pressure is a powerful modifier of the physical properties of matter
- In the lab, we are able to achieve static pressures and temperatures approaching the centre-of-the-Earth (and dynamic pressures approaching centre of Jupiter – DCS)
- Scientific capabilities are ‘technique-driven’, demanding materials with most extreme properties of strength and hardness.
- Synchrotron HP diffraction and XAS techniques are mature, with a huge diversity of x-ray techniques continually being developed.
- Neutrons can make a powerful contribution to HP science, especially in diffraction.
- Now is beginning of new period of growth in neutron capabilities based at new generation of intense sources, such as SNS.
- Combination of x-ray and neutron science will become increasingly important as scope of neutron capabilities improves in next several years



“Although mathematics cannot be avoided...the aim has been to write a book that most scientists will still find approachable. To this end, the first two chapters [provide] a background in the mathematics and physics that are implicitly assumed in other texts. **Thereafter, the philosophy has been one of keeping things as simple as possible.**”

Published Jan 2011