some technical news

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Efree *highlight*
Feb.11, 2015

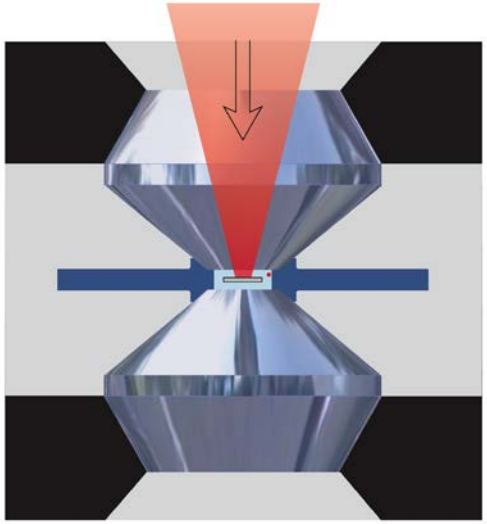
Outline

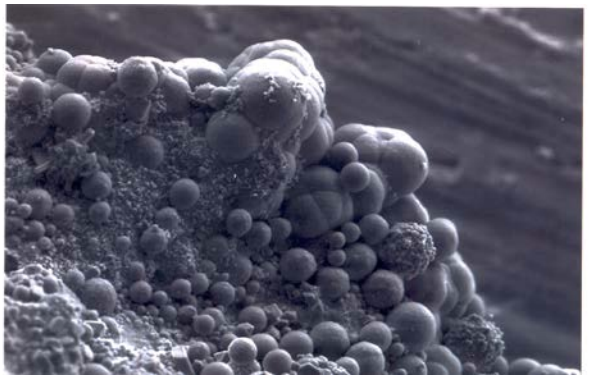
- good and bad instruments for material synthesis
- some new diamond cell techniques (flash heating, rapid compression/decompression)
- Neutron diffraction at SNAP (ORNL)
- PE cells or not

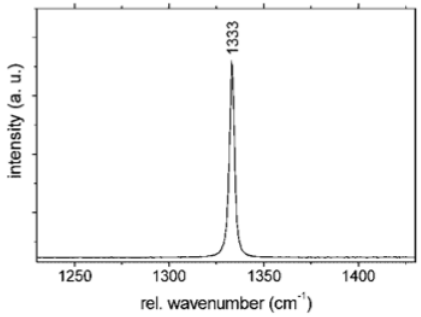
CO_2 laser heating for material synthesis

λ 10 μm, (oxides, organics just about anything)

synthesis of of new materials using IR lasers

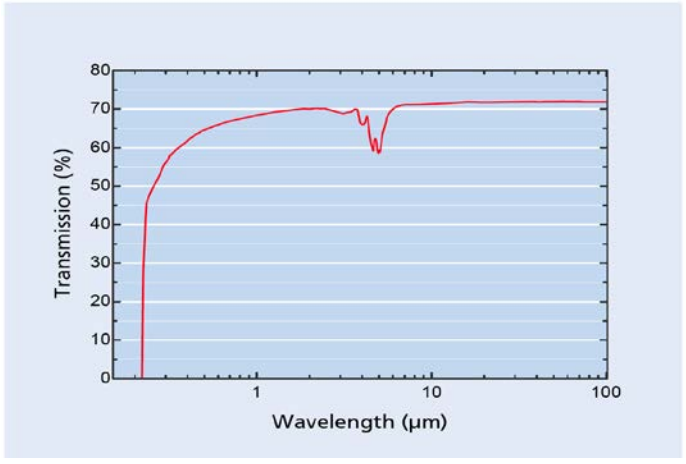




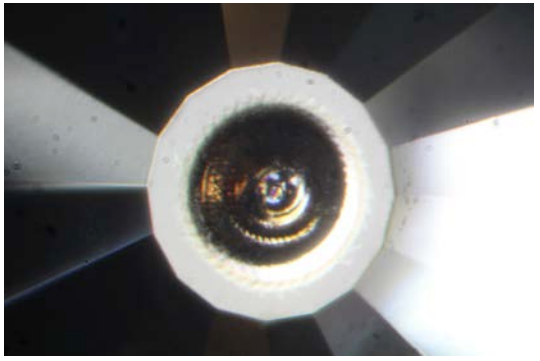


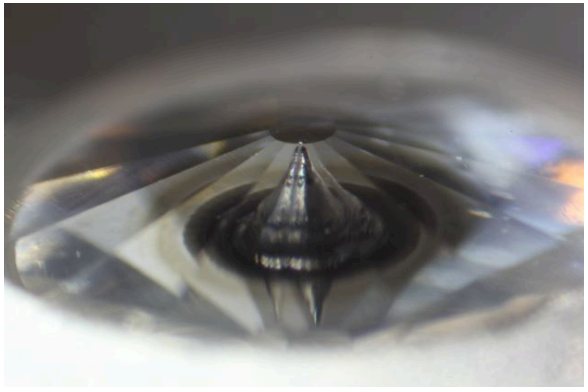
example: synthesis of micron-sized diamond sheres from methane

UV (193 nm) laser cutting system

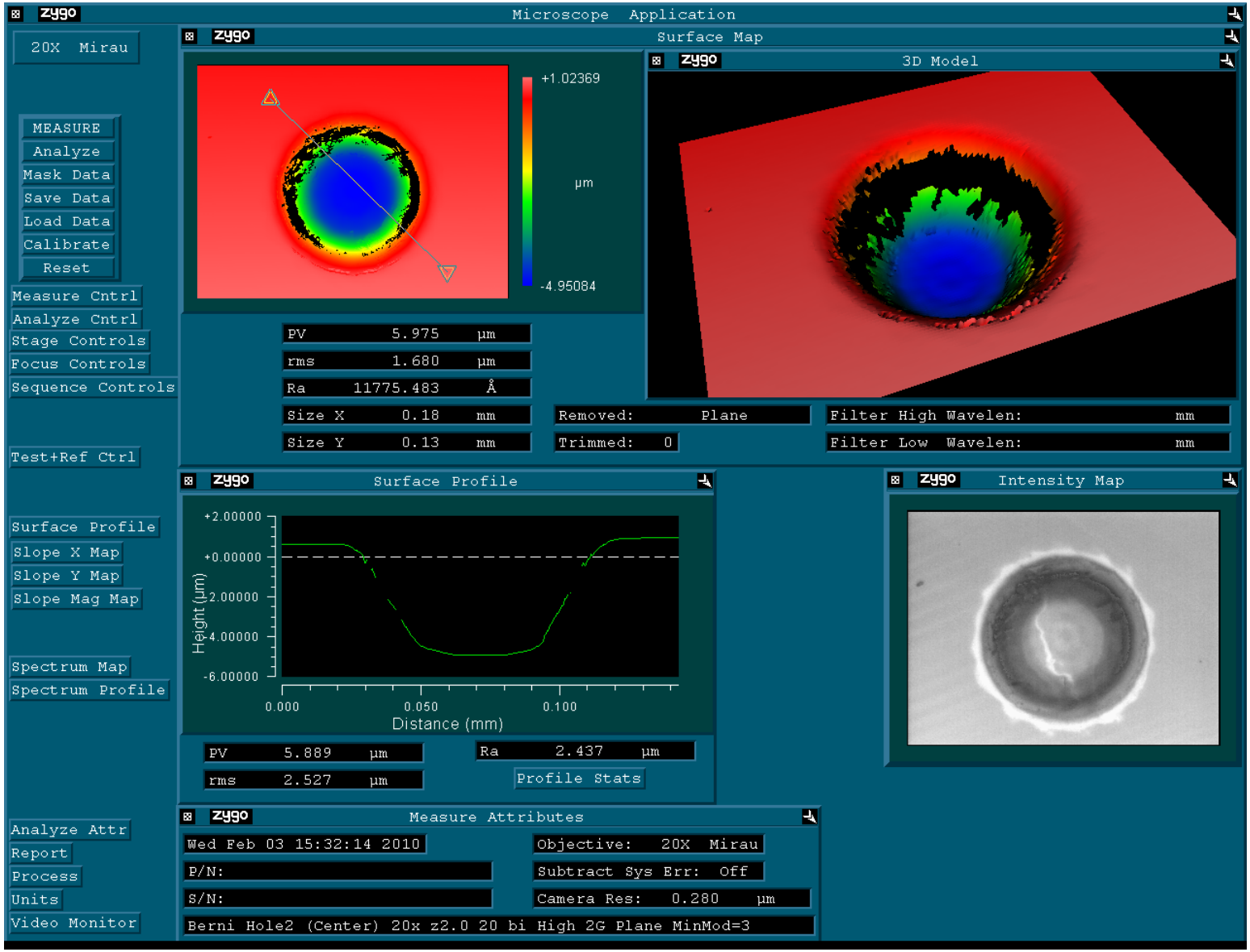


diamond machining



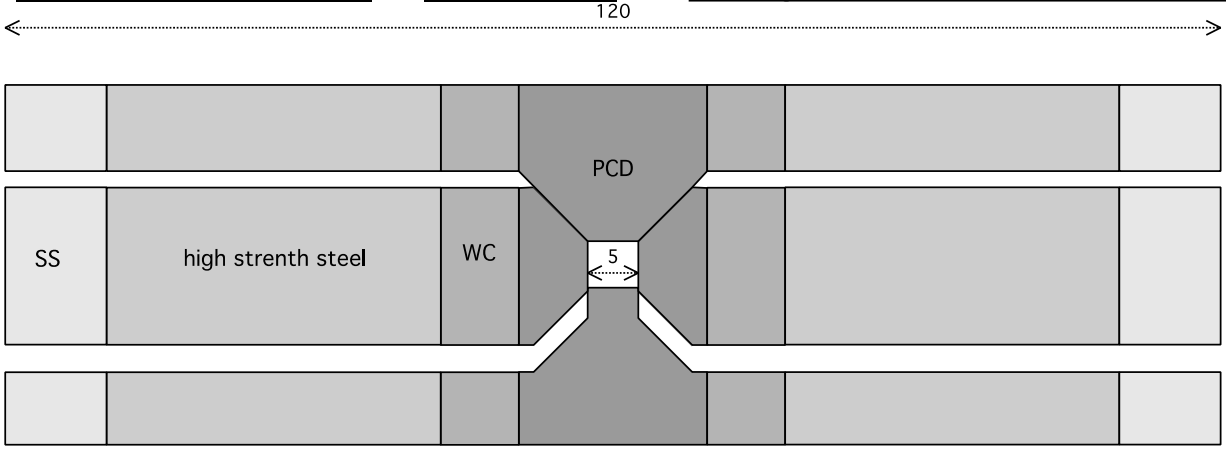


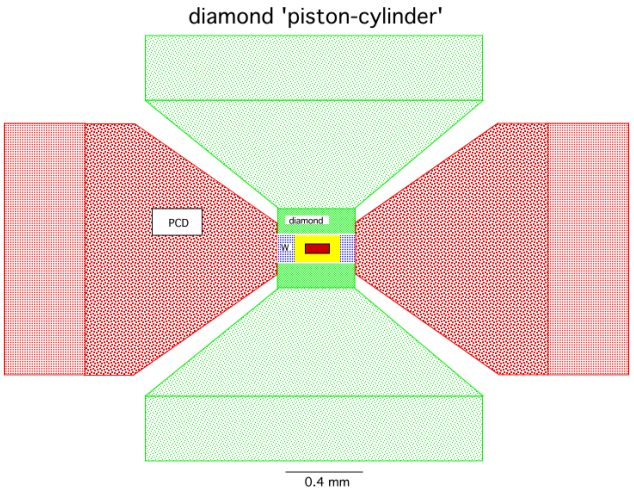
Profilometer



Large volume cells for synthesis

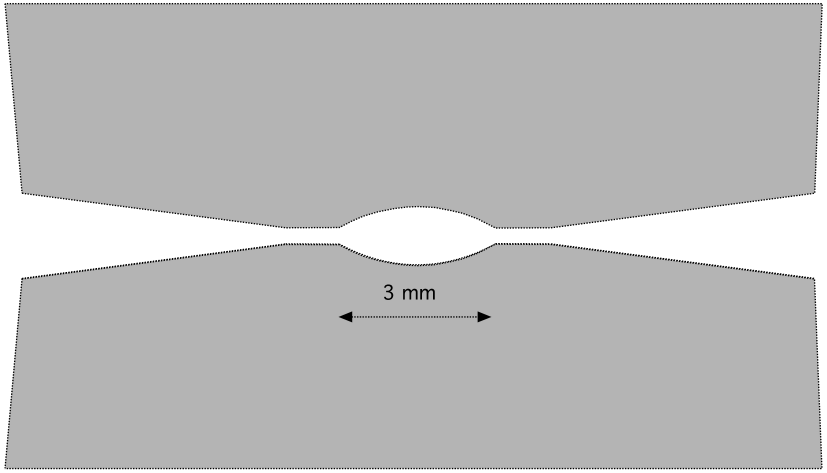
piston-cylinder **> 20 GPa** **sample volume > 20 mm^3**





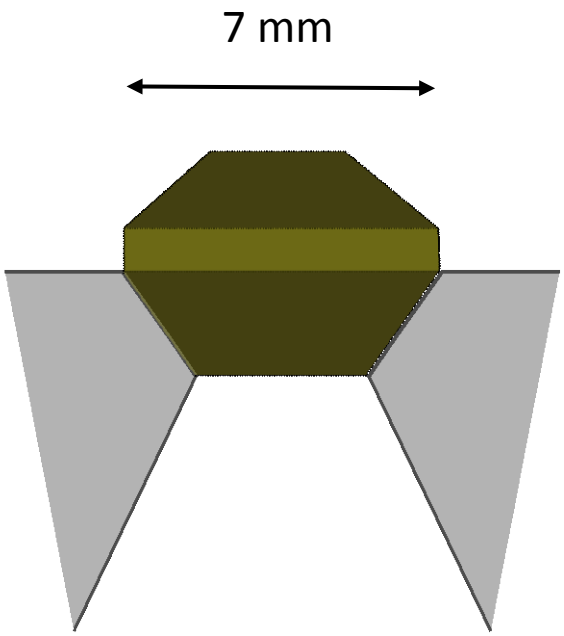
double toroid

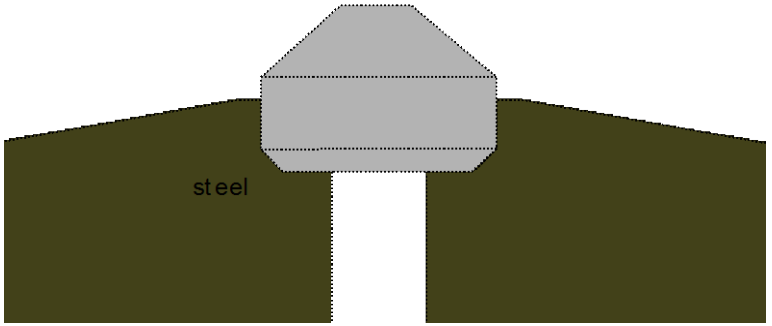
simple dish



reliable to 25 GPa

LARGE **CVD** DIAMONDS !

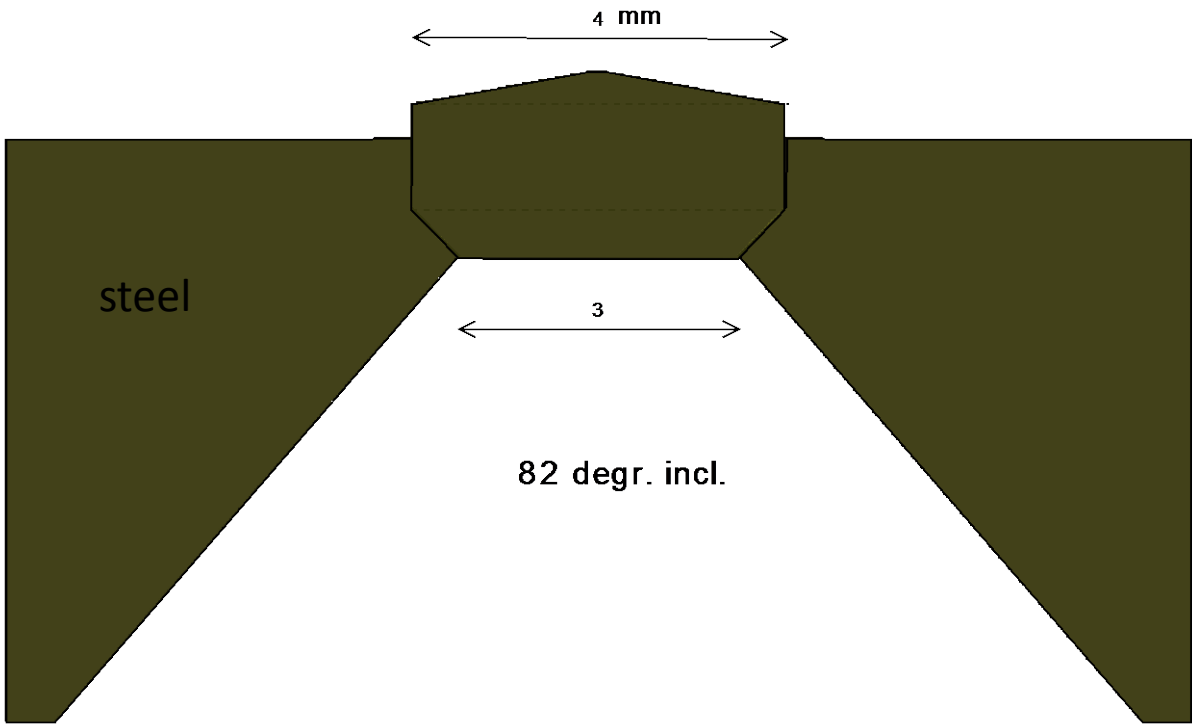




sample volumes up to 2 mm^3

other benefits of large CVD diamonds

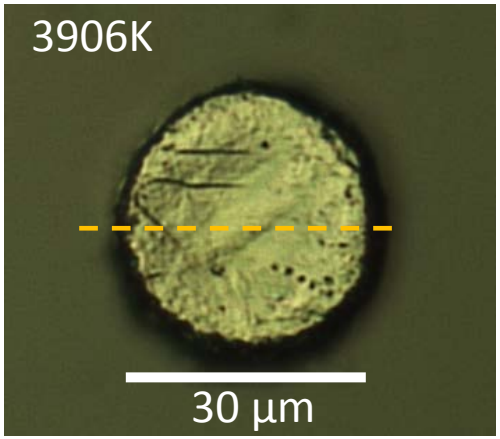
new cells for single crystal X-ray diffraction



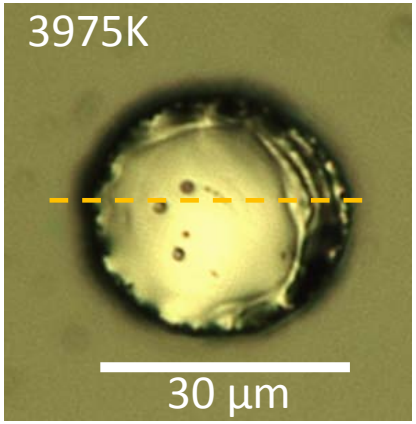
82 degr. 200 micron culet 90 GPa

Some new experimental techniques

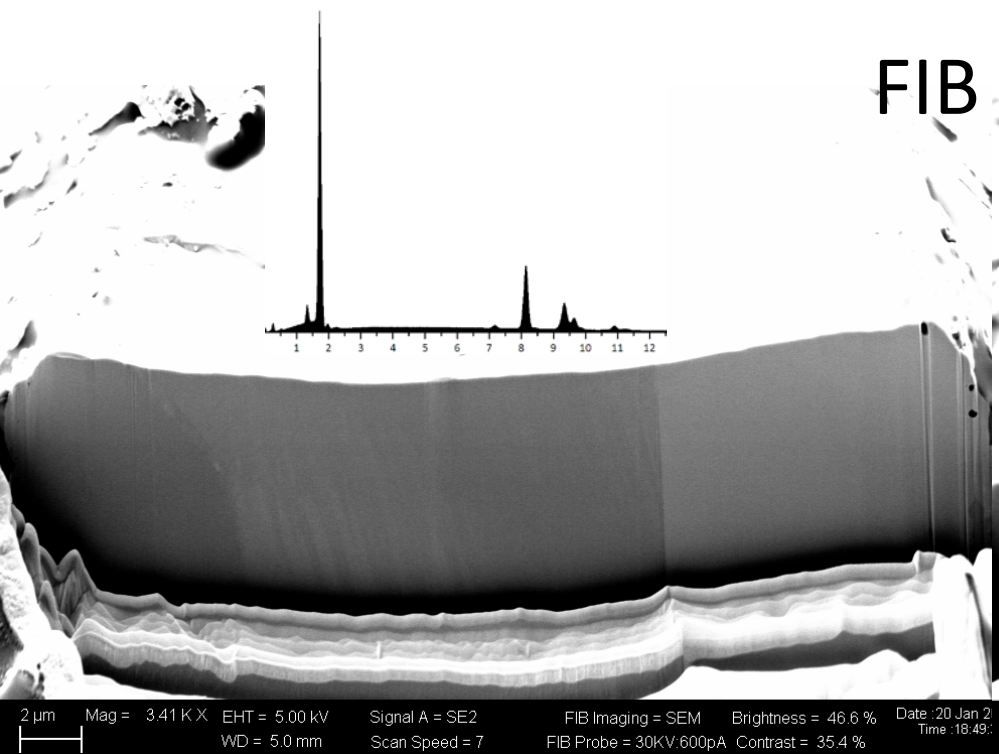
Flash heating

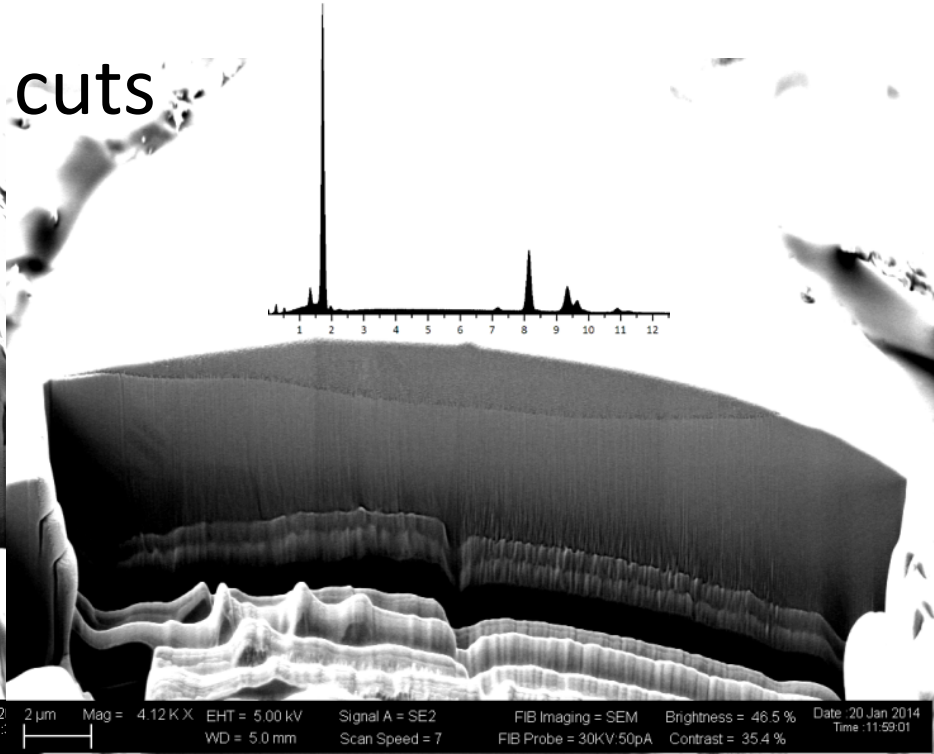


Recovered samples
(50 Gpa)



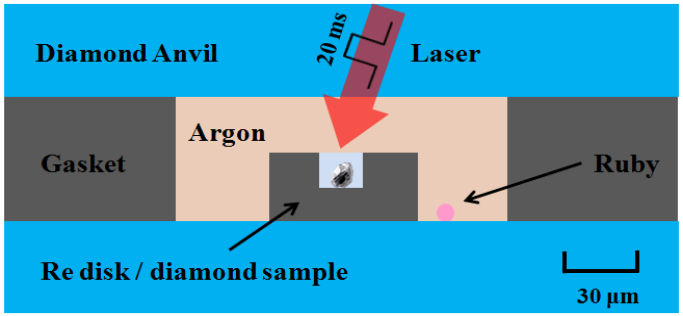
FIB cuts

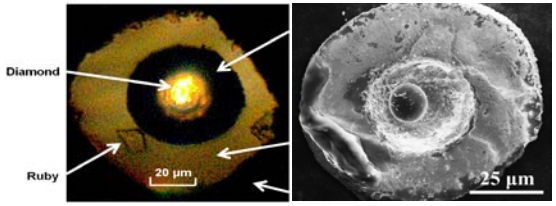




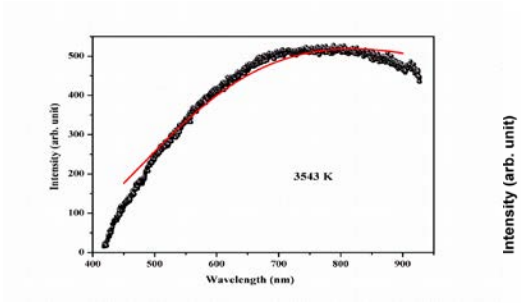
diamond melting

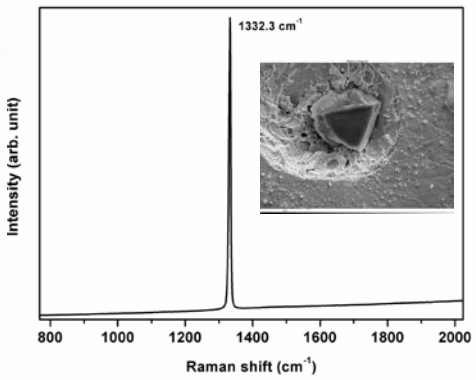
20 ms flash-heating in Re cavity



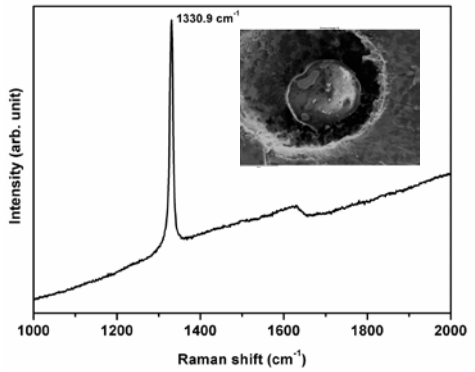


30 GPa, 3203 K

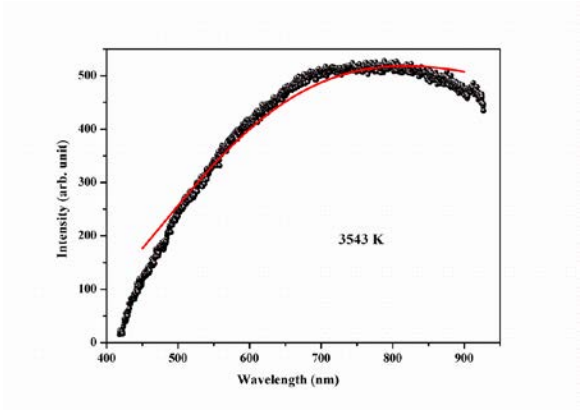


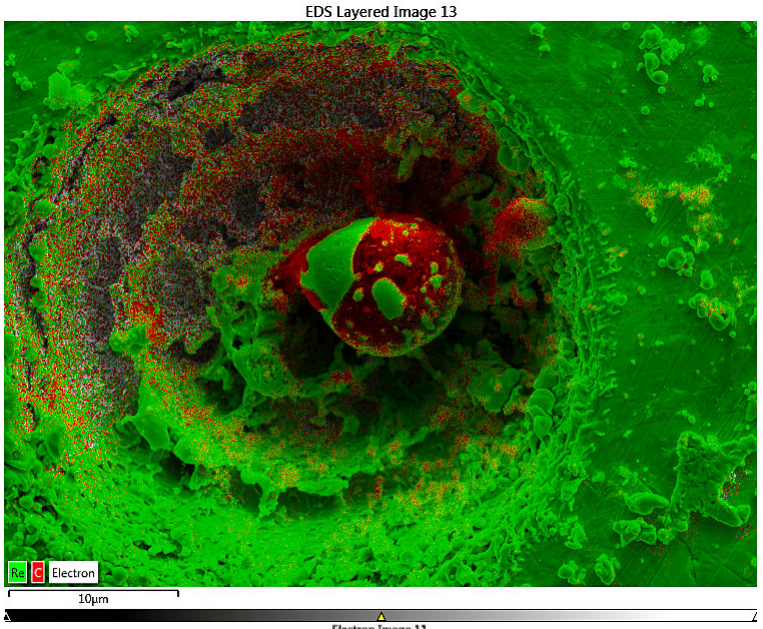


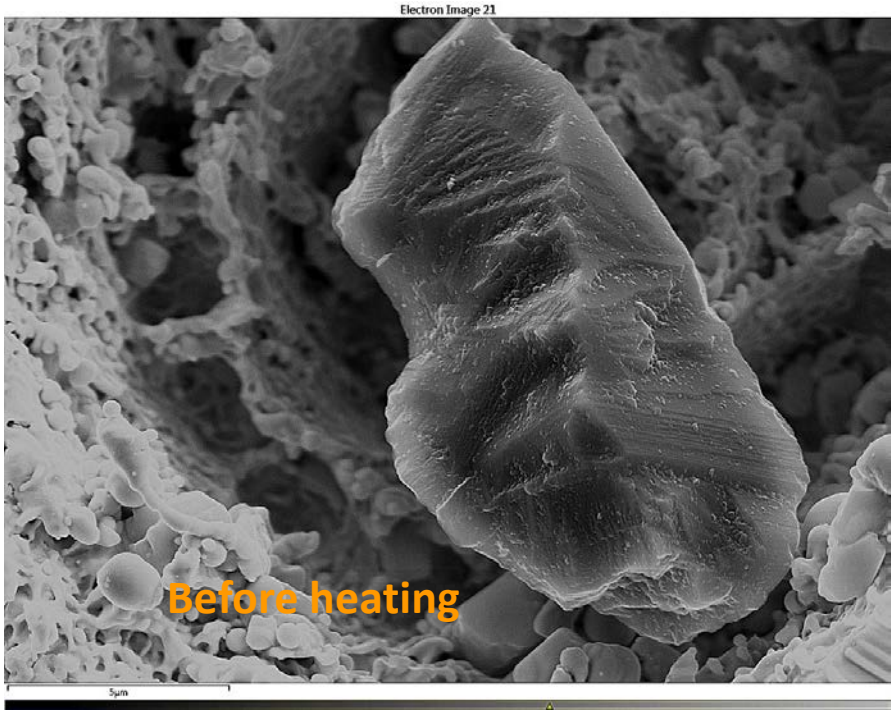
25 GPa, 3340 K



3543 K

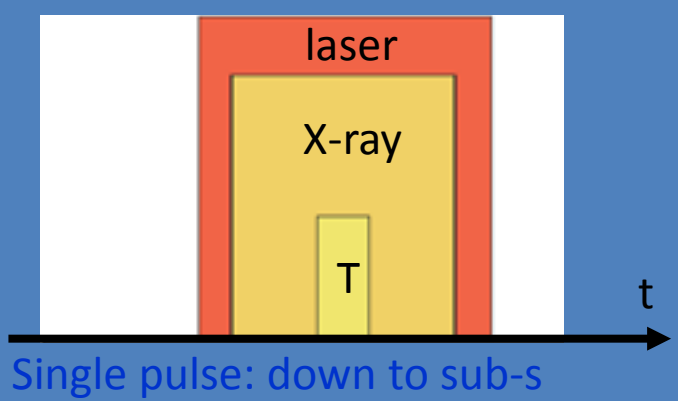

Intensity (arb. unit)
Wavelength (nm)
3543 K
0
100
200
300
400
500
400
500
600
700
800
900


EDS Layered Image 13
Re
C
Electron
10µm

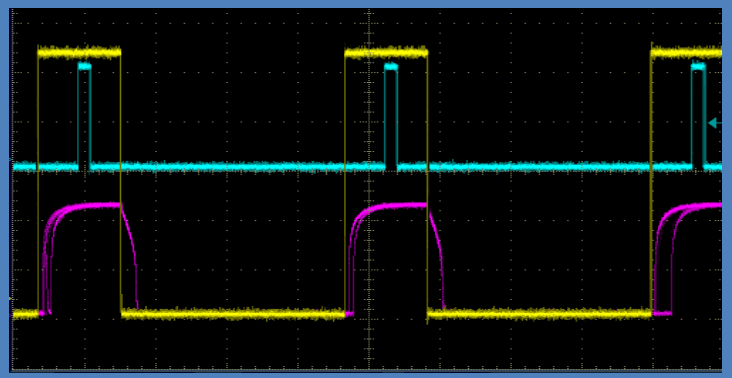

Electron Image 21
Before heating
5µm


Electron Image 11
After heating
5µm

Flash heating at HPCAT



Single pulse: down to sub-s

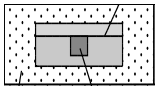


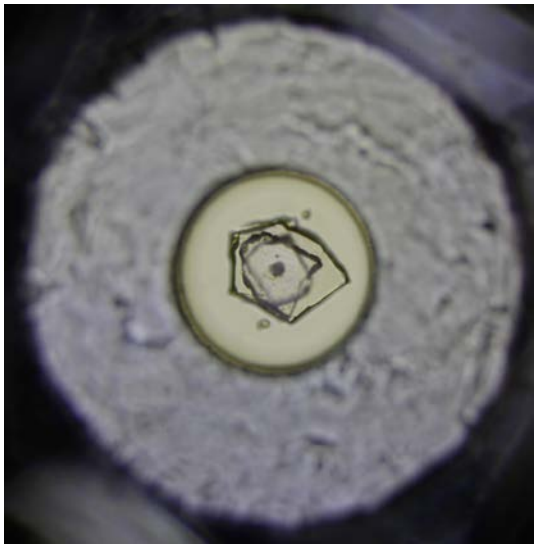
Multiple events: µs-ms

Pulsed laser heating

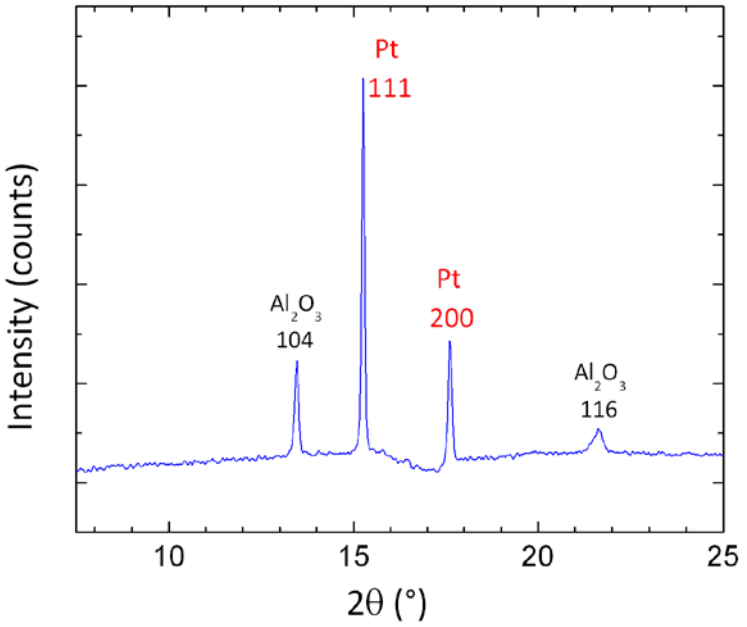
synchronizing laser, X-ray, temperature measurement

sample encapsulation

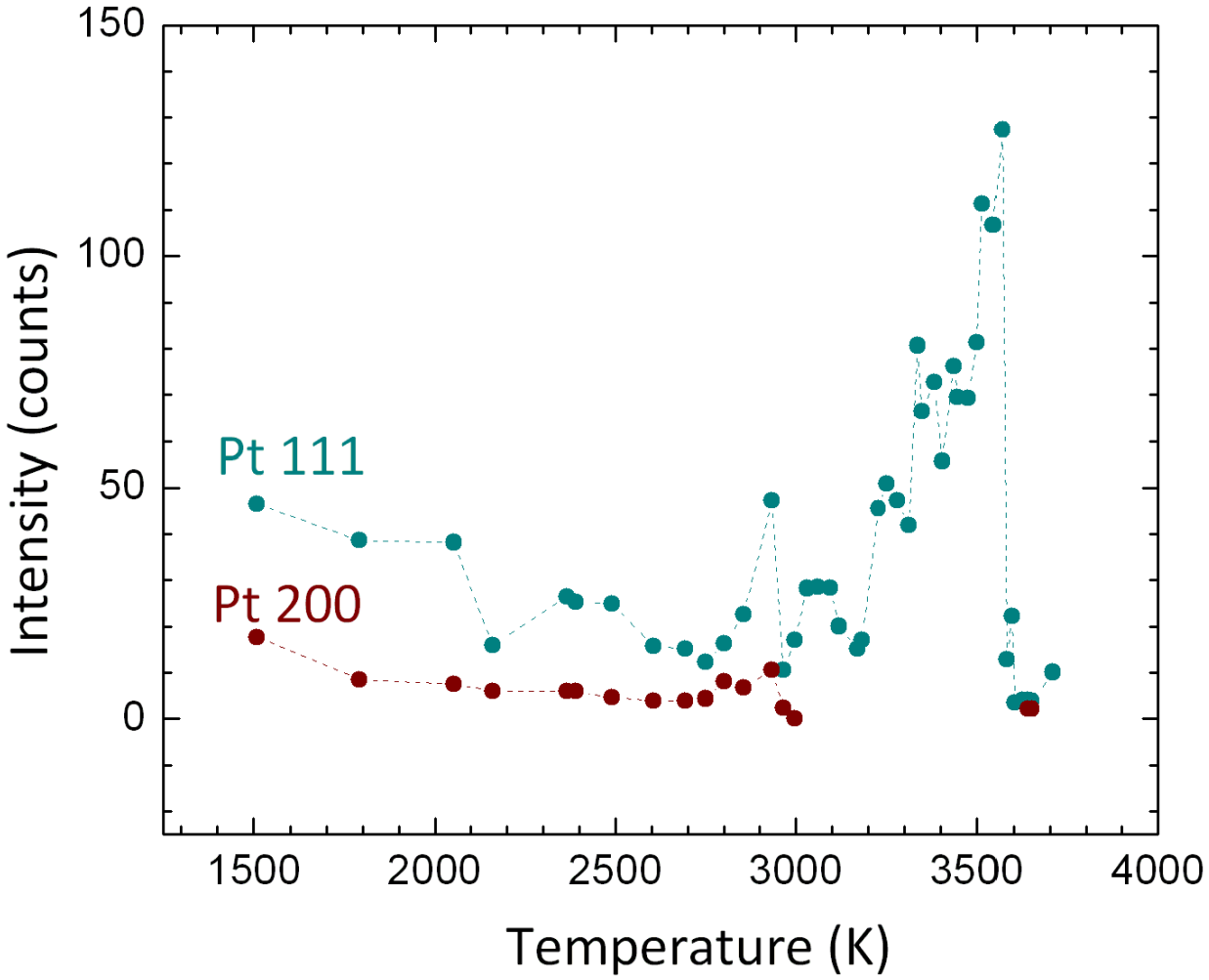




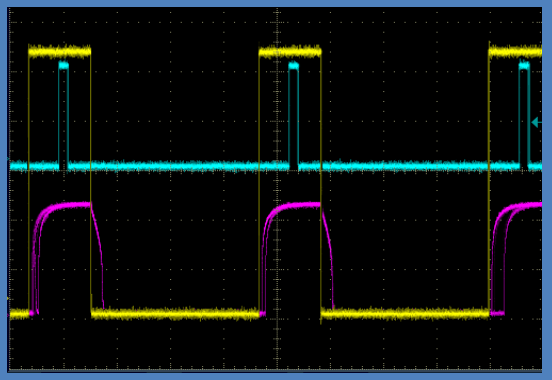
Pt 50 GPa 1 millisecond (single shot)



melting

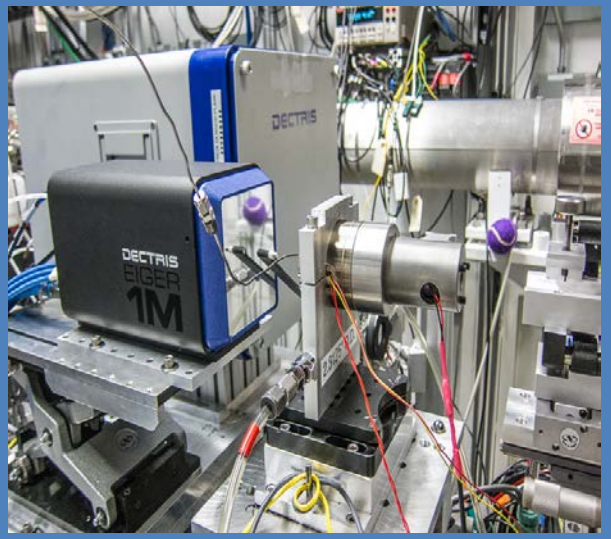

150
100
50
0
Intensity (counts)
Pt 111
Pt 200
1500
2000
2500
3000
3500
4000
Temperature (K)

P-T(*t*)



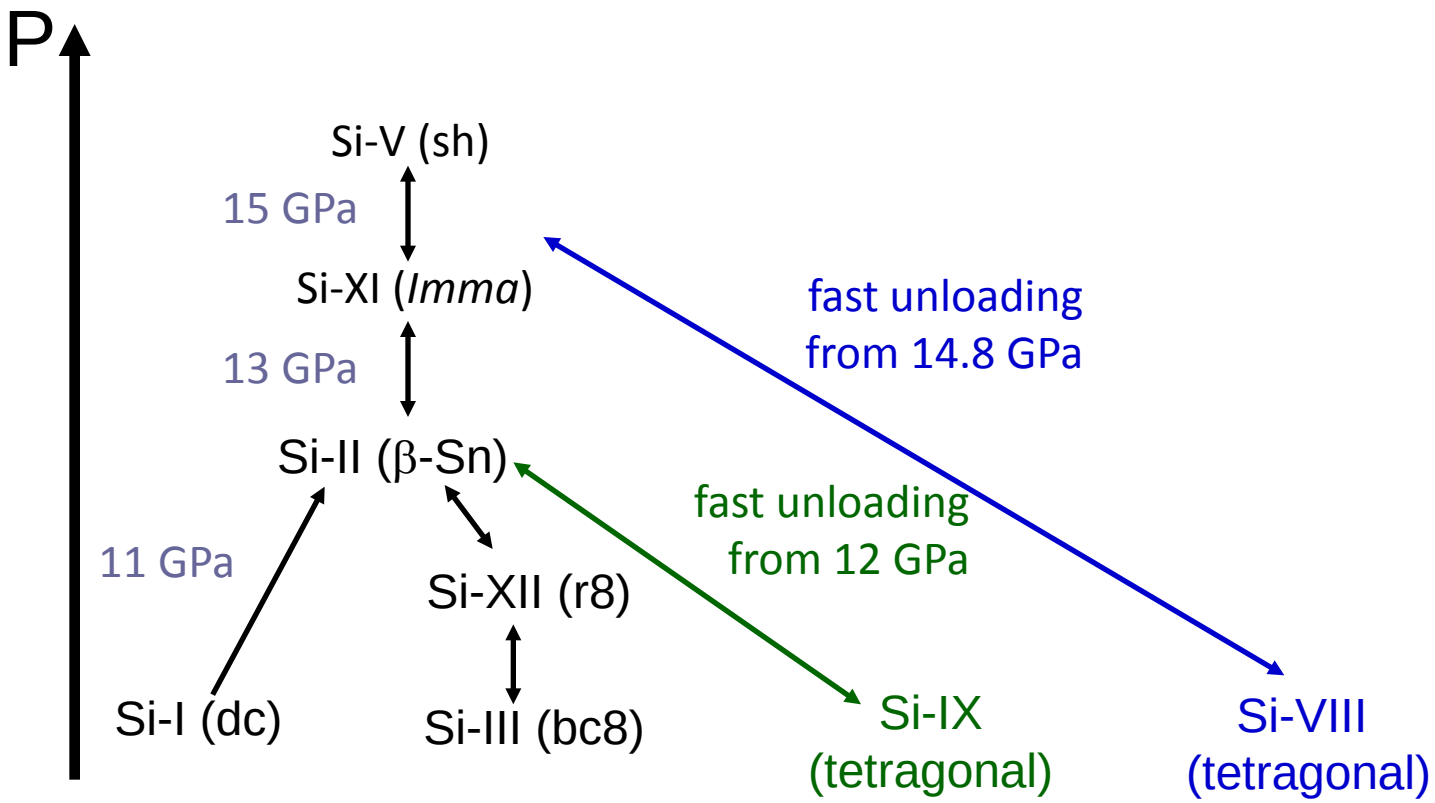
Modulated Laser Heating

P(*t*)-T



dDAC – Fast Detectors

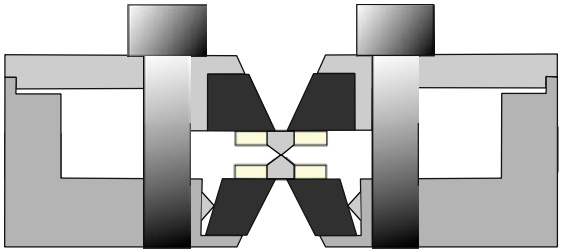
Si: Fast unloading in the DAC



fast unloading yields different crystalline phases.

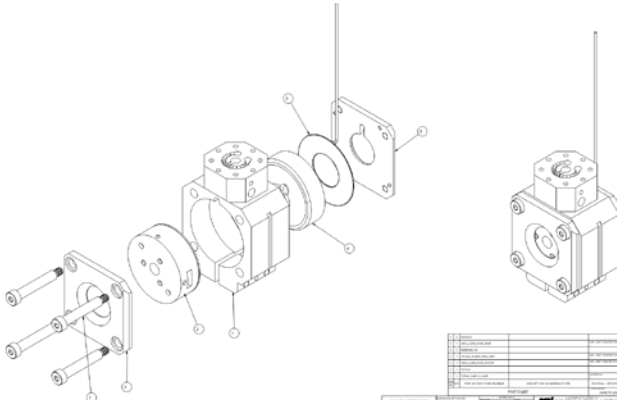
rapid loading / unloading

piezo cell



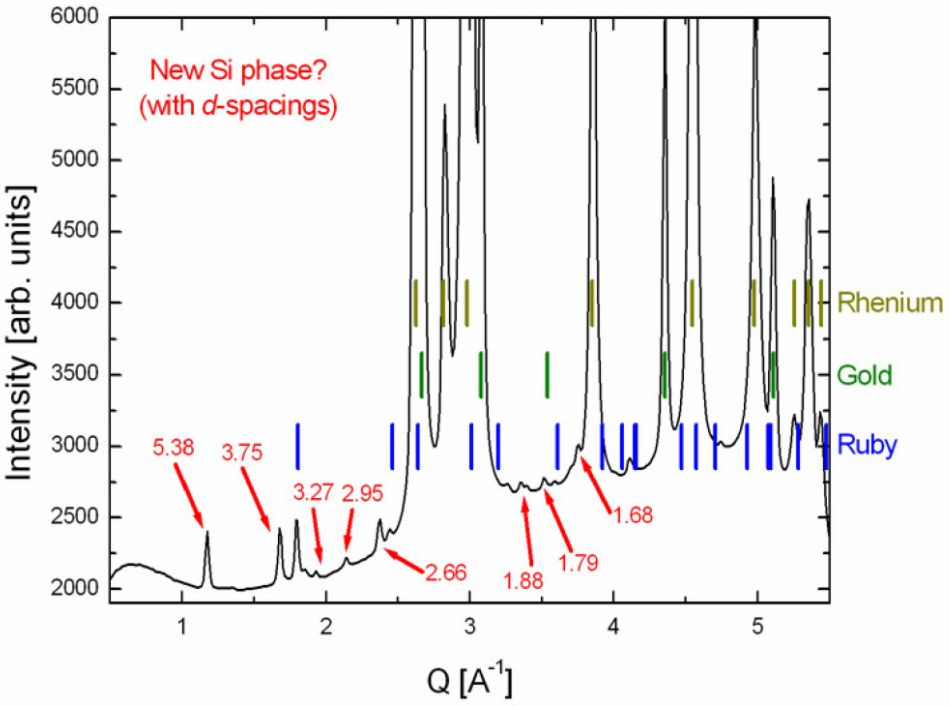
“pot-cell”

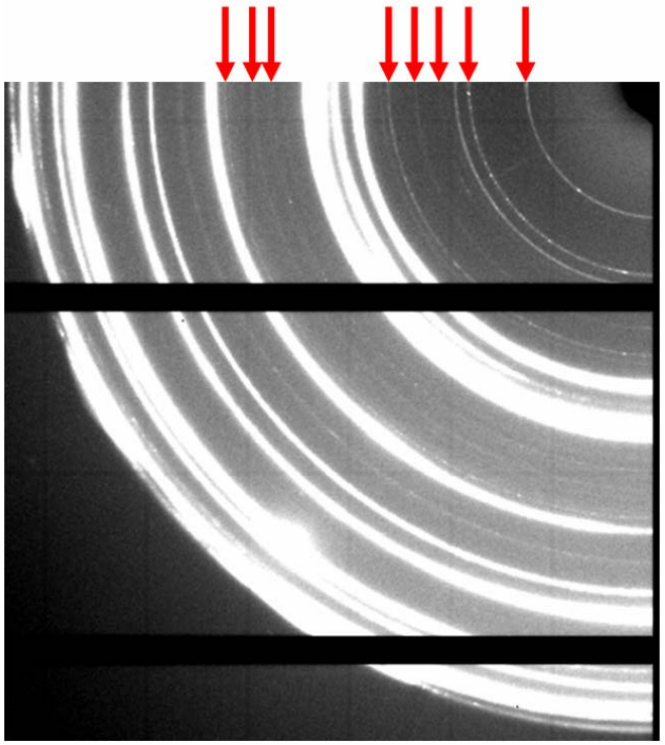
Cryo-compression of Si



Si was metallized in a cryostat and then decompressed.
resulting in a new metastable phase.

1D XRD of new phase





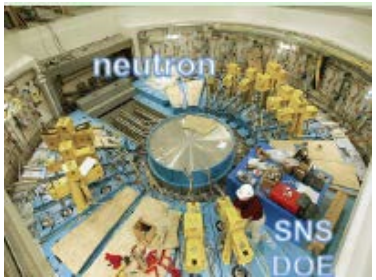
2D XRD of new phase

Haberl et al.

Neutron Diffraction

light atoms, liquids, magnetic order

Neutron diffraction



SNAP

Spallation Neutrons and Pressure

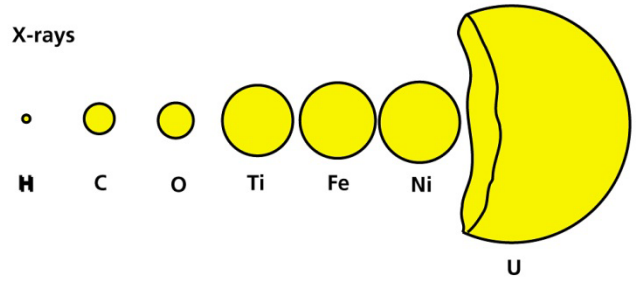
since 2010

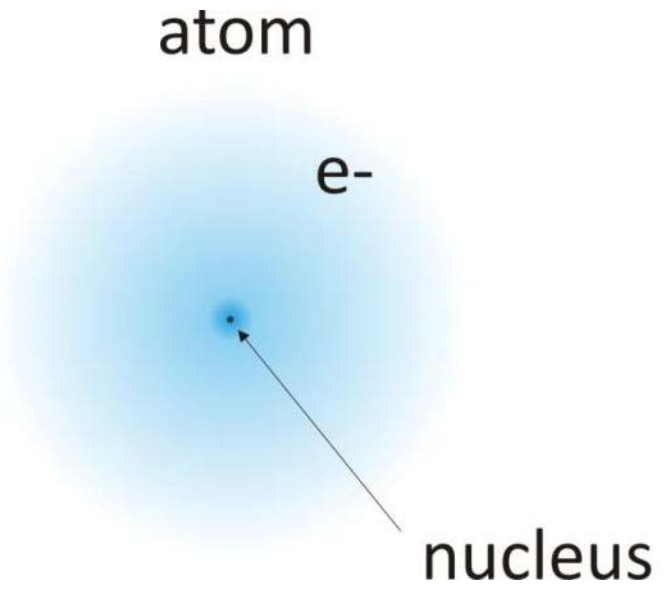
Instrument Development Team (IDT)

Why Neutrons?

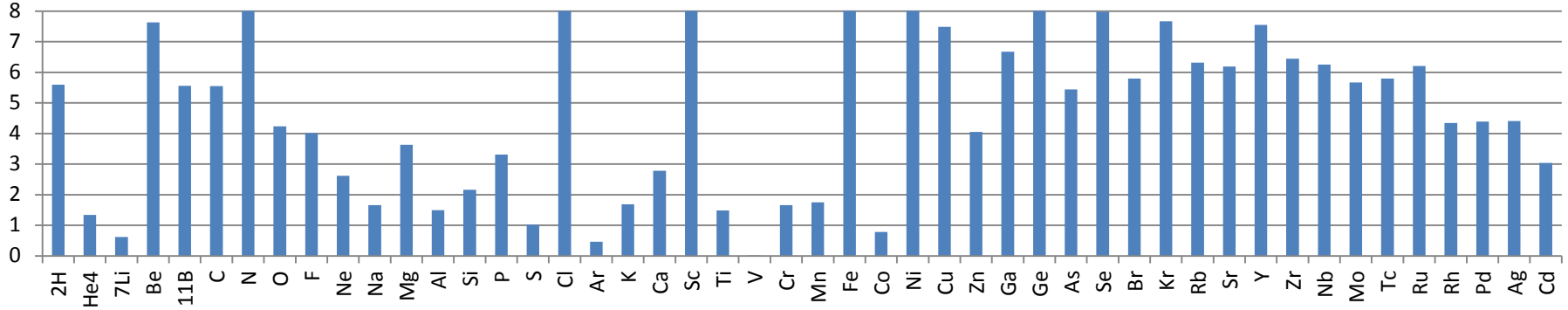
- light atoms
- magnetic order, subtle structural distortions
- non-crystalline/ amorphous structures
- neutron imaging (diffusion processes)

different from X-ray diffraction



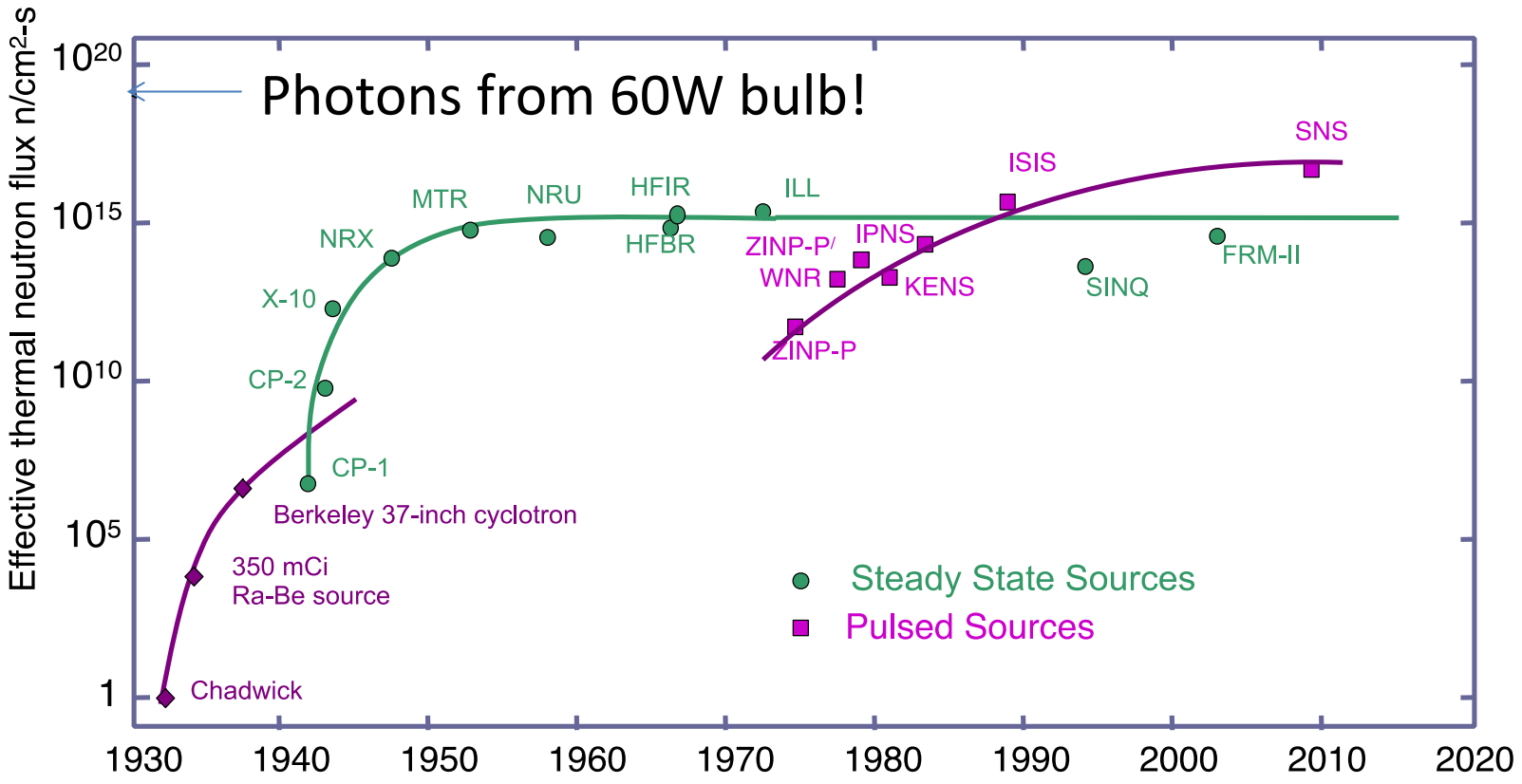


Neutron Coherent Cross-Section (Barn)



Scattering strength is isotope dependent

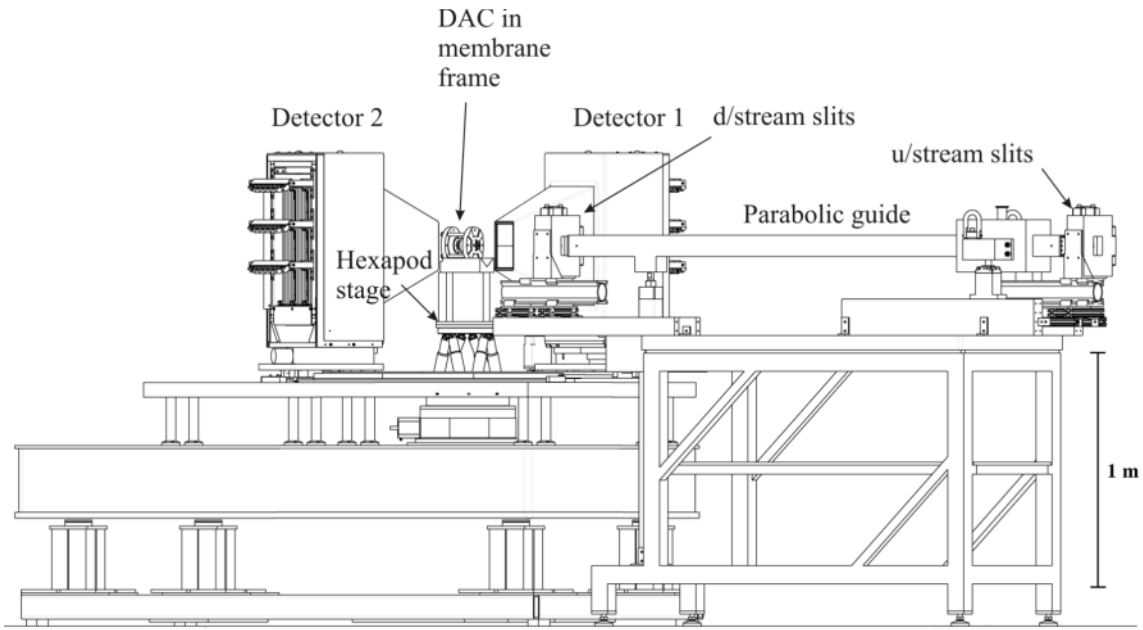
Neutrons have a major disadvantage

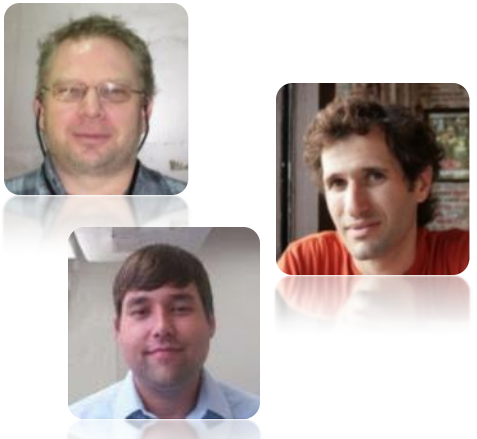


(Updated from *Neutron Scattering*, K. Skold and D. L. Price, eds., Academic Press, 1986)

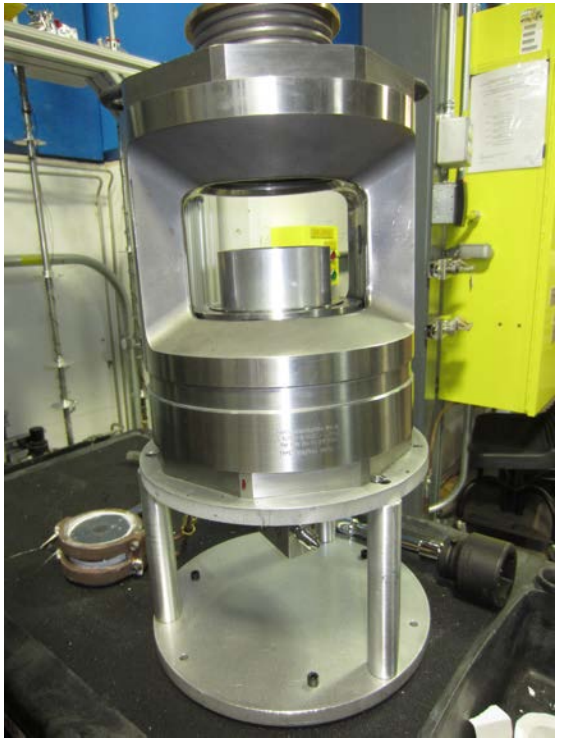
The Instrument

SNAP – a dedicated high pressure diffractometer, came online 2006

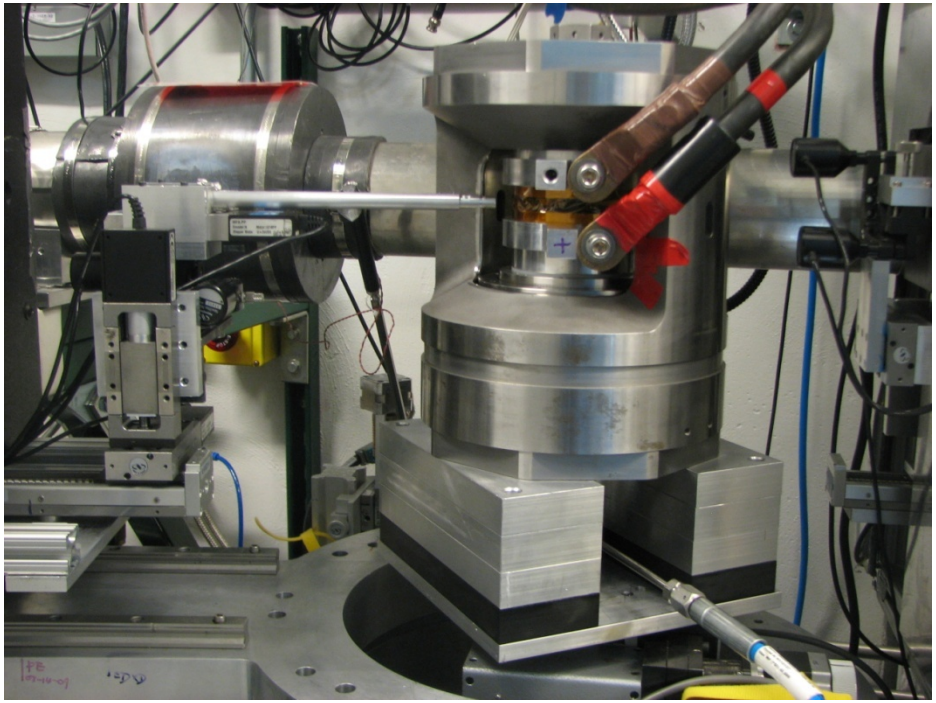


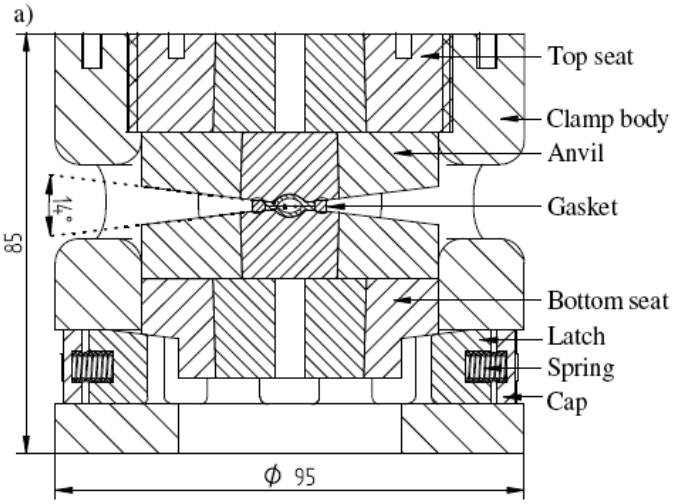


Traditionally : big (expensive) equipment



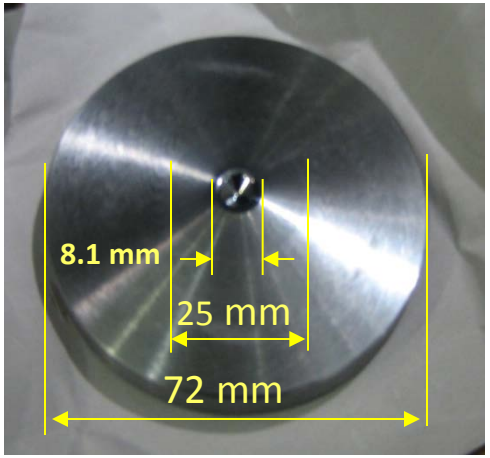
Conventional High Pressure Neutron Diffraction





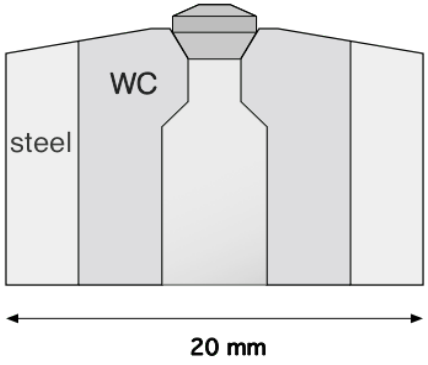
sample volumes ≈ 25 mm^3

pressures up to 25 GPa

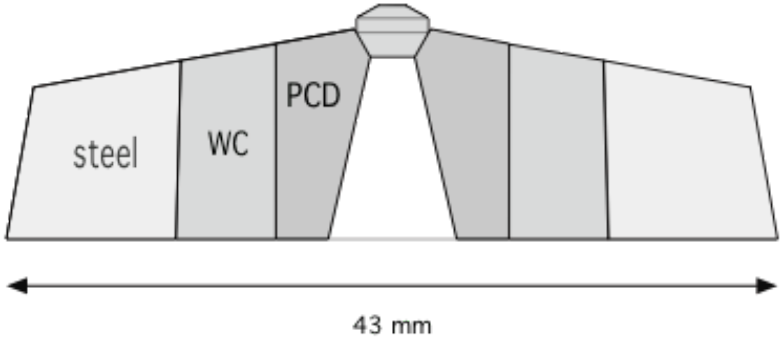


New approach is to use diamond anvil cells

Large culets (1.0 to 1.5mm) make diamond support critical:

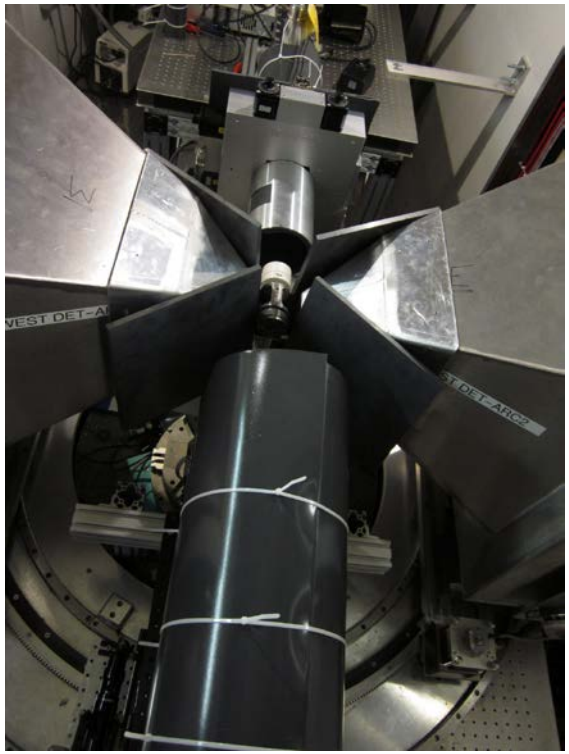


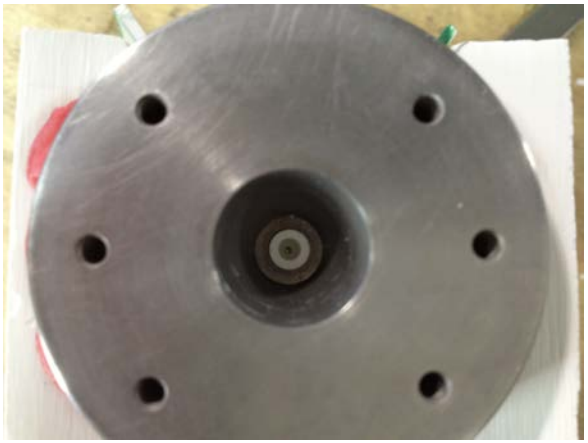
40 GPa



95 GPa

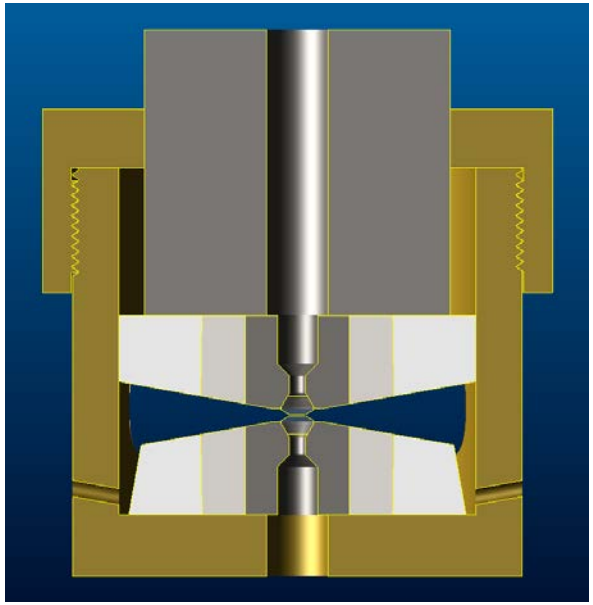
reducing background

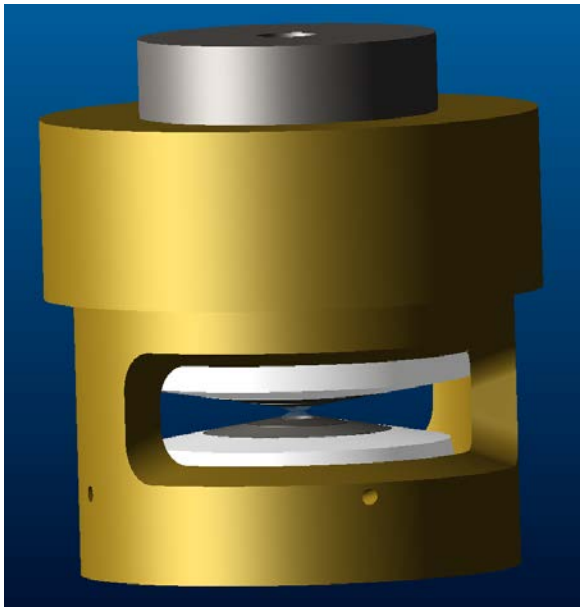




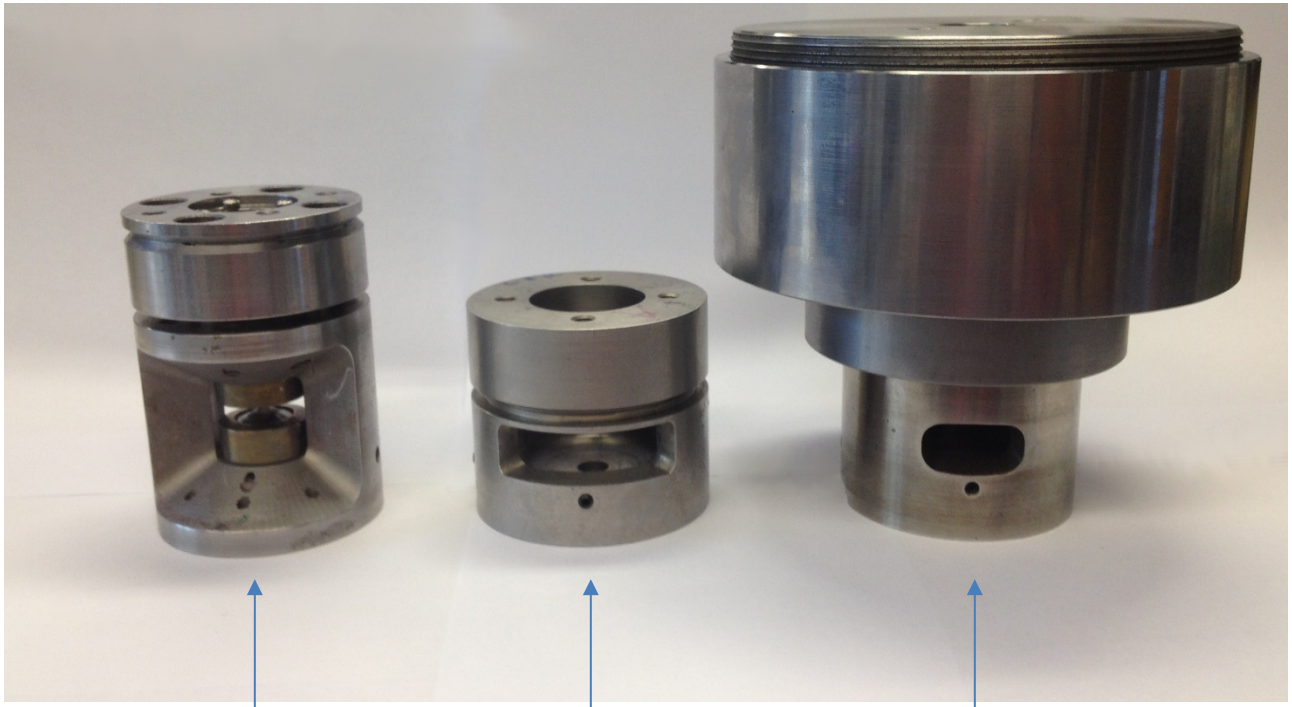
BN collimator

New Large Volume Diamond Cells for Neutron Diffraction and Material Synthesis

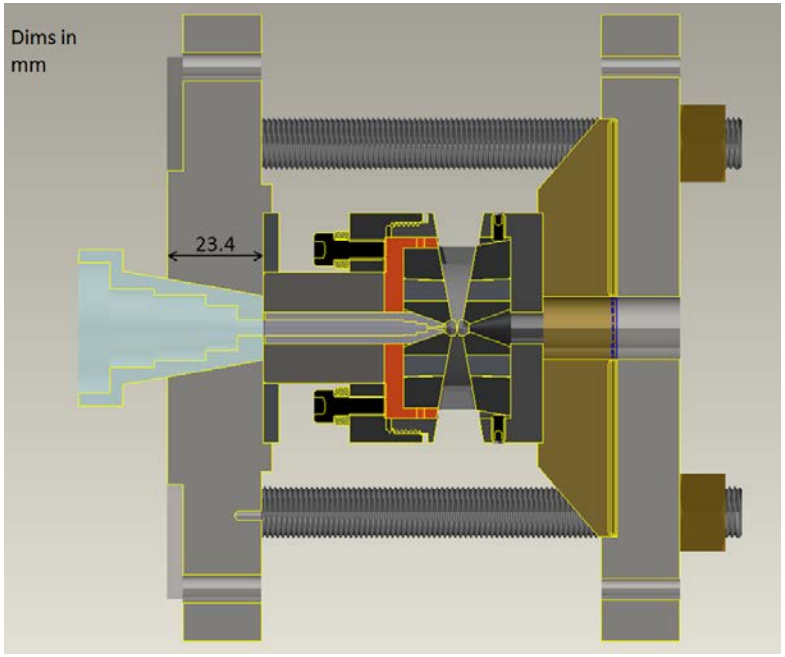


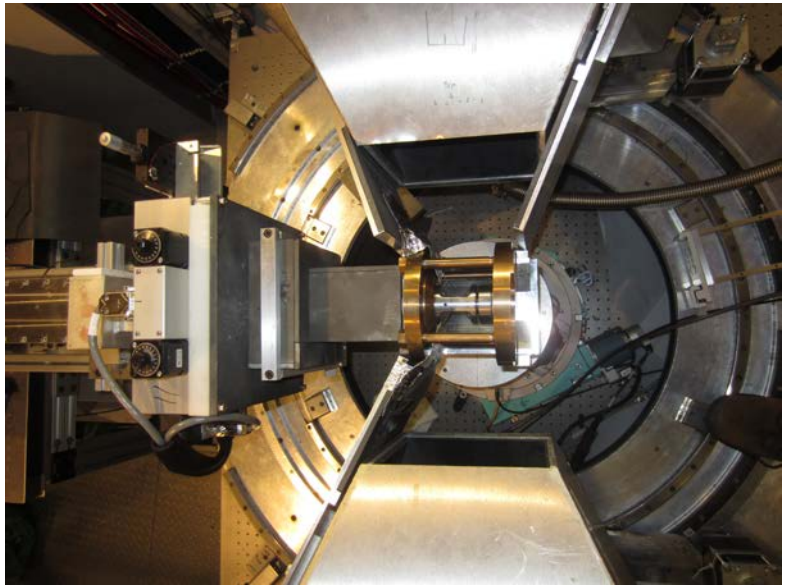


The Cells

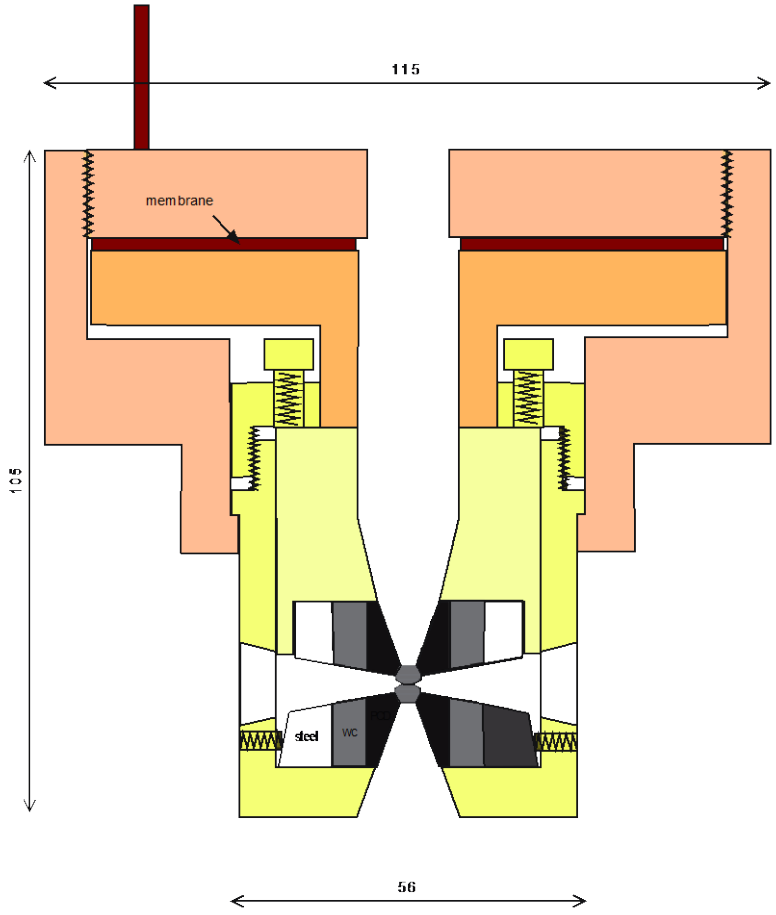


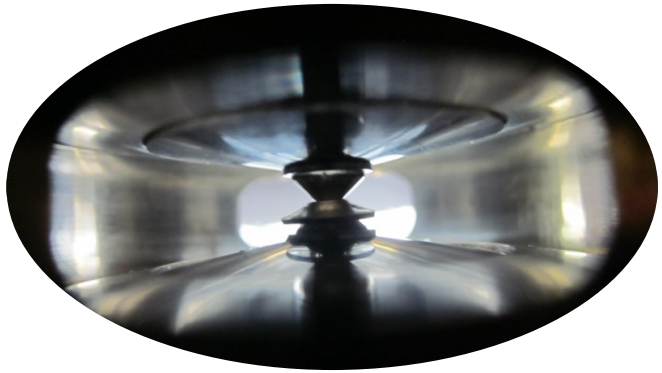
Early 2000s 2013 2014


Dims in
mm
23.4



New, (two part) press designs for > 10 tons using gas membranes
Stage 1 (yellow) allows loading of compressed gas samples

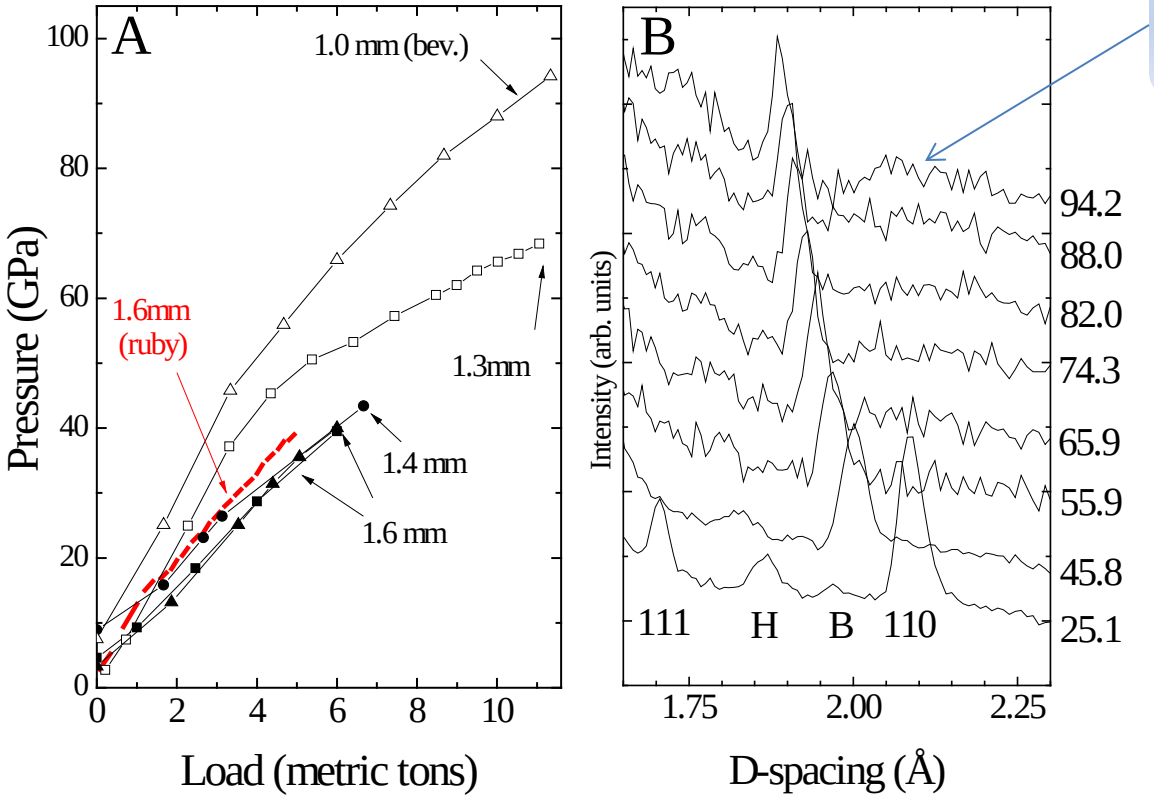




Pressure limit

PCD seats Fails at ~12 tons

achieved **94 GPa** with 1.0mm beveled culets.



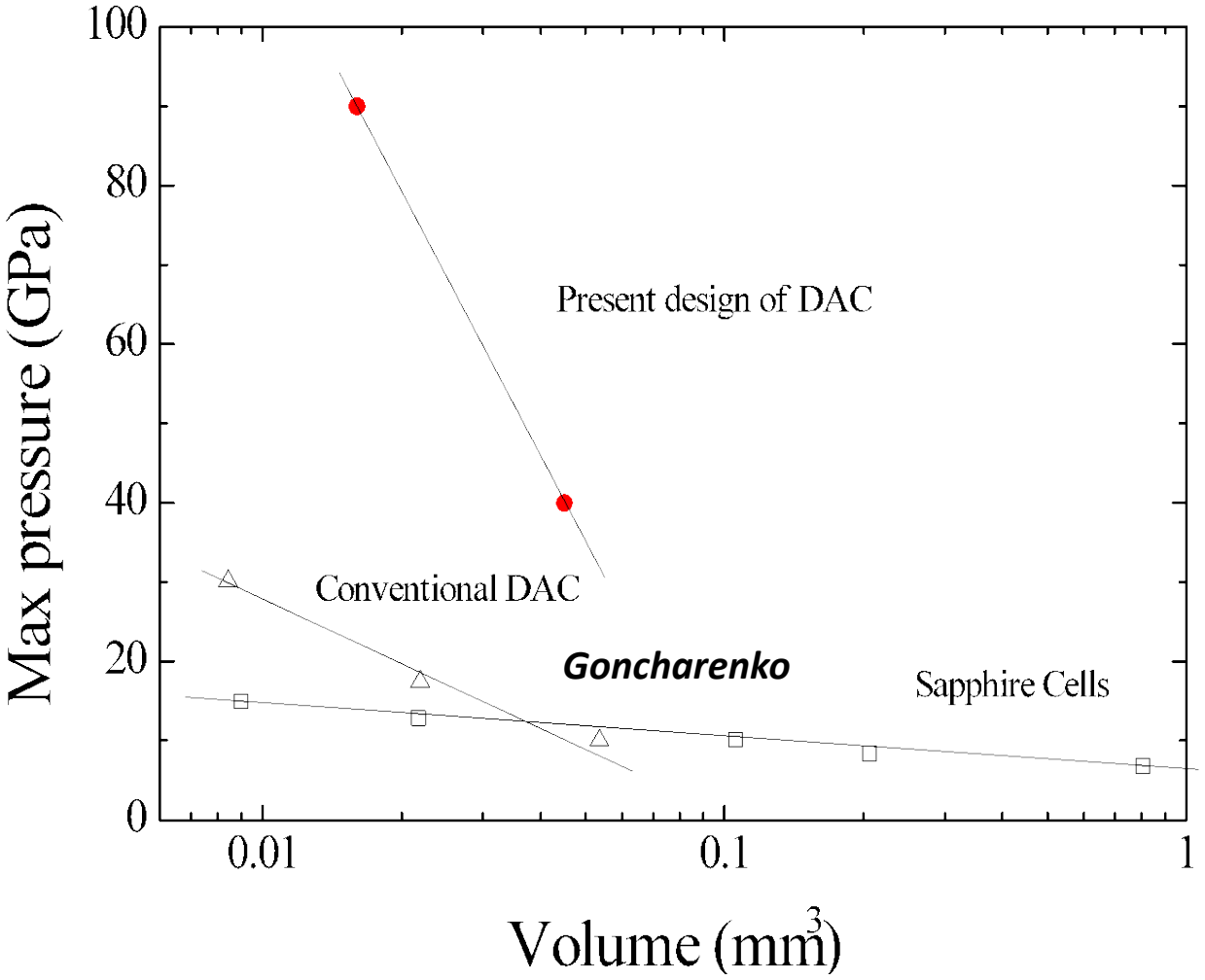
10 min data set!

A: pressure-load (open symbol PCD; closed symbol WC)

B: 110 diffraction peak from ice VII up to 94 GPa

R. Boehler, M. Guthrie et al, HPR http://dx.doi.org/10.1080/08957959.2013.823197 (2013).

a new dimension

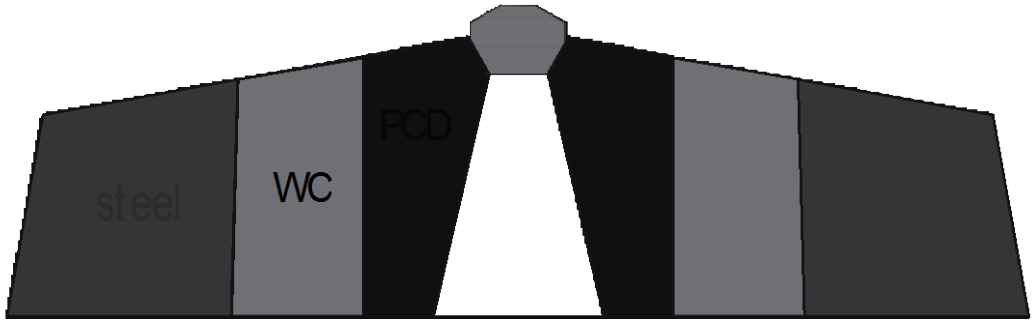


PNAS
June 2013

HPR
July 2013

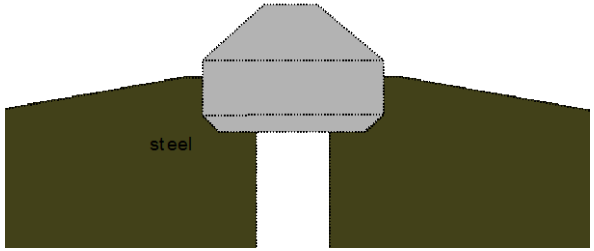
still problems

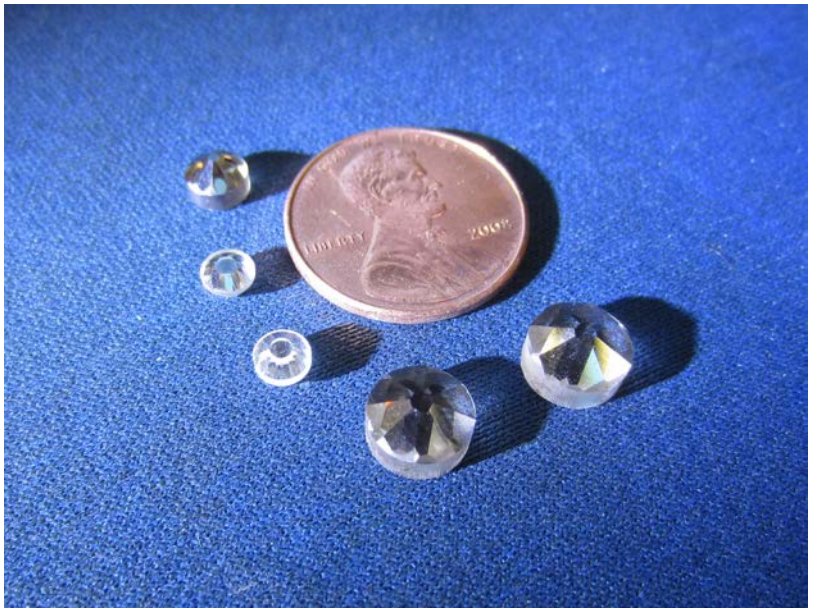
new approach: larger, simpler anvils (CVD), simpler seats



8 mm anvils

pending



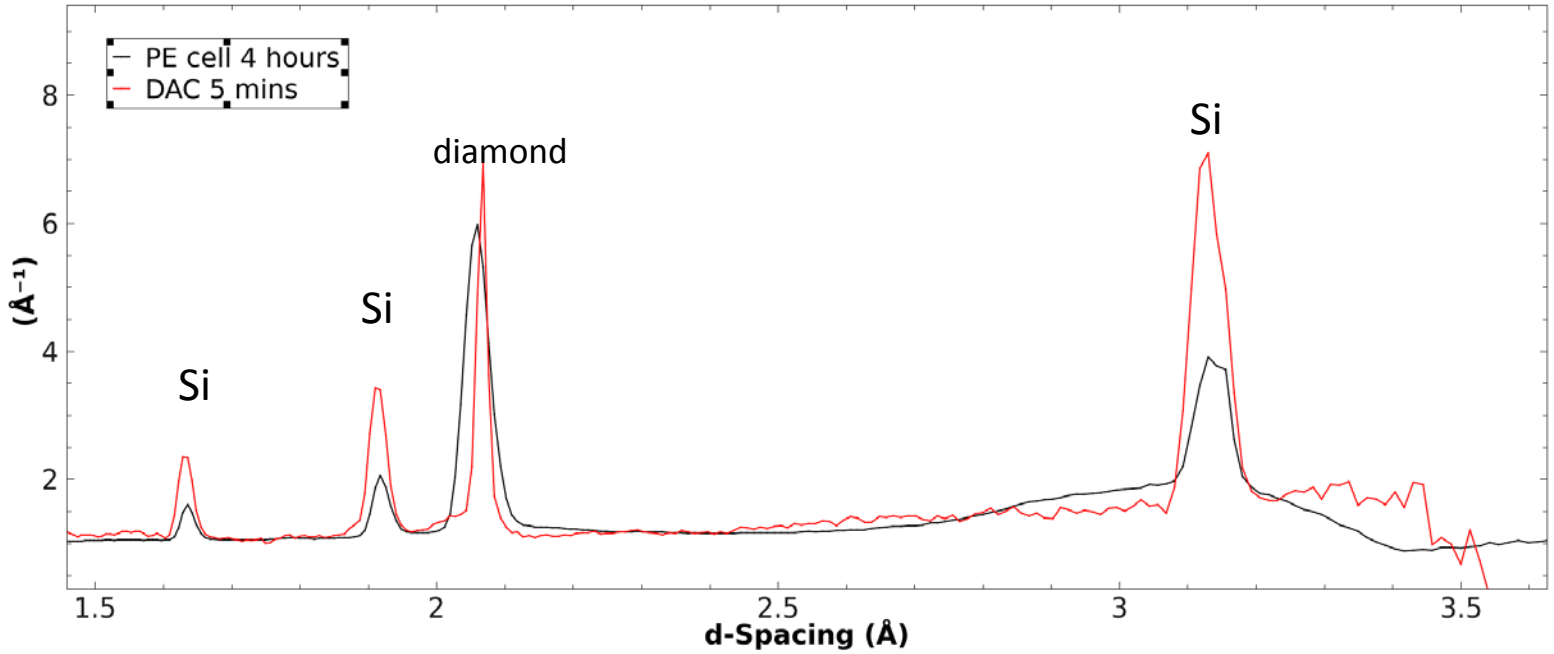


Comparison PE cell and DAC

PE cell double toroid: 5.5 mm^3, 4 hrs loading, 4hrs exposure, 4 hrs unloading,
frequent blow-outs 15-20 Gpa

DAC 2.5 mm culets: 1.5 mm^3, fast loading, short exposure, optical access,
pressure range likely significantly higher **(1/4 cost, fraction of time)**

test: **Si** ambient pressure



to be discussed…