X-Ray Diffraction under Extreme Conditions

Gaston Garbarino
European Synchrotron Radiation Facility
Grenoble, France

Outline

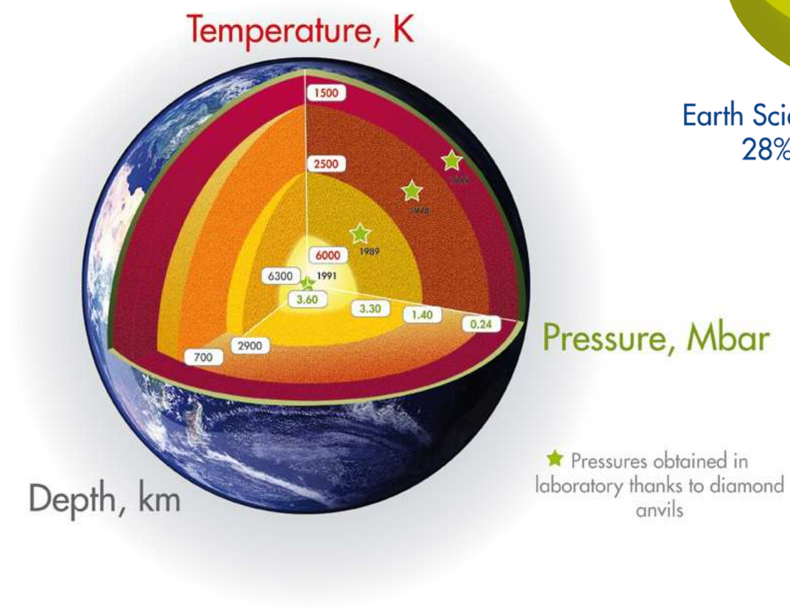
- Introduction
- What - Why - How is high pressure?
- Example of a high pressure beamline (ID27 @ ESRF)
- Scientific examples of high pressure experiments

Introduction

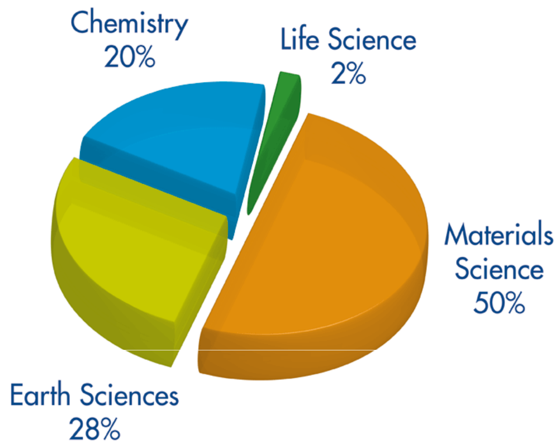
‘Science at Extreme Conditions’: multidisciplinary research

Biology → Geophysics

RP,RT	3.5 Mbar T<6000 K



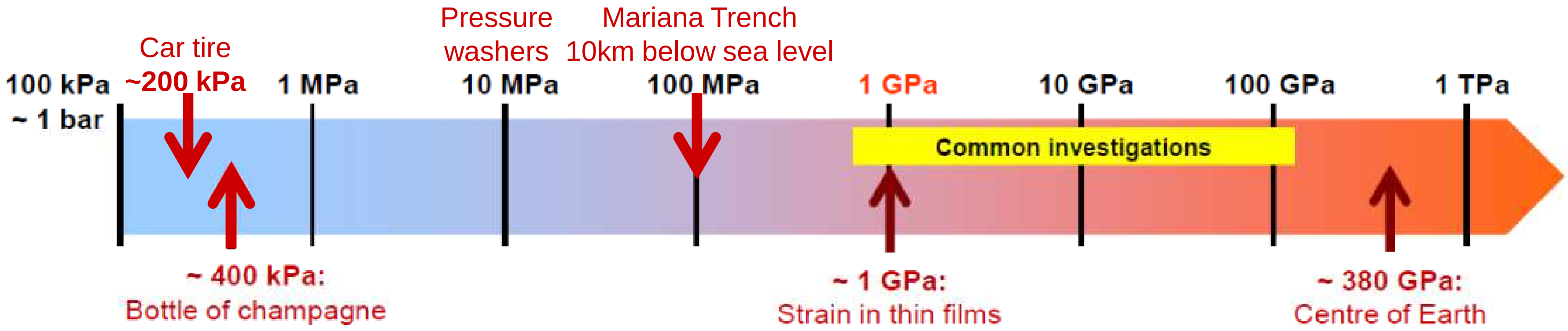
Percentage breakdown of beamtime per scientific domain (2008)

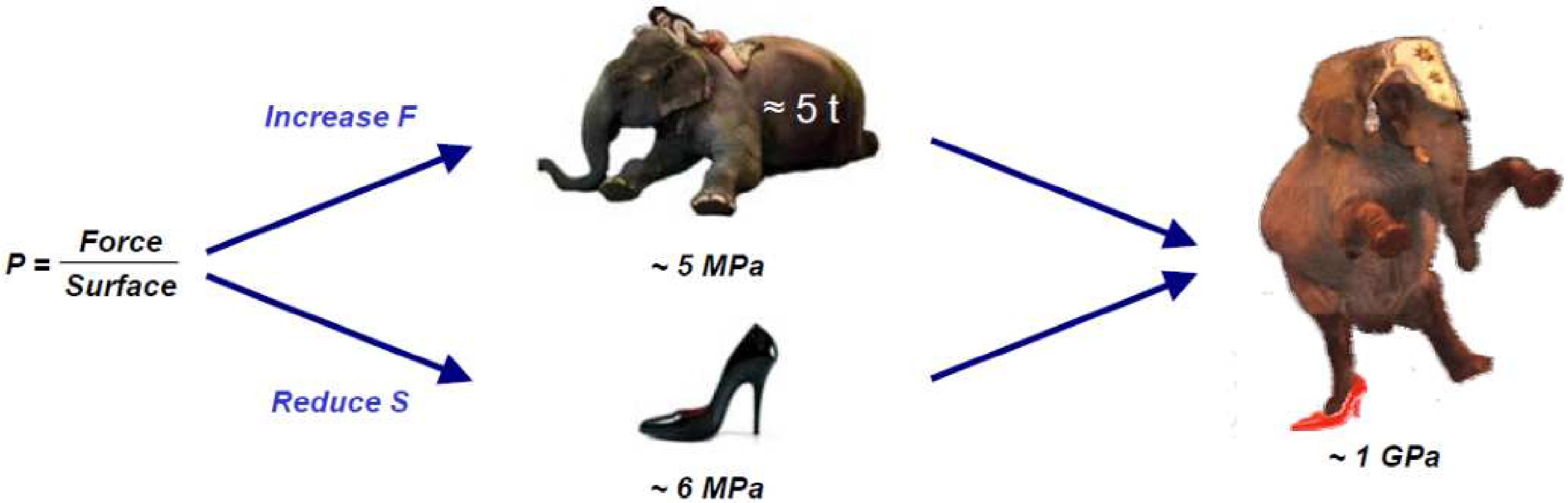


What is high-pressure?

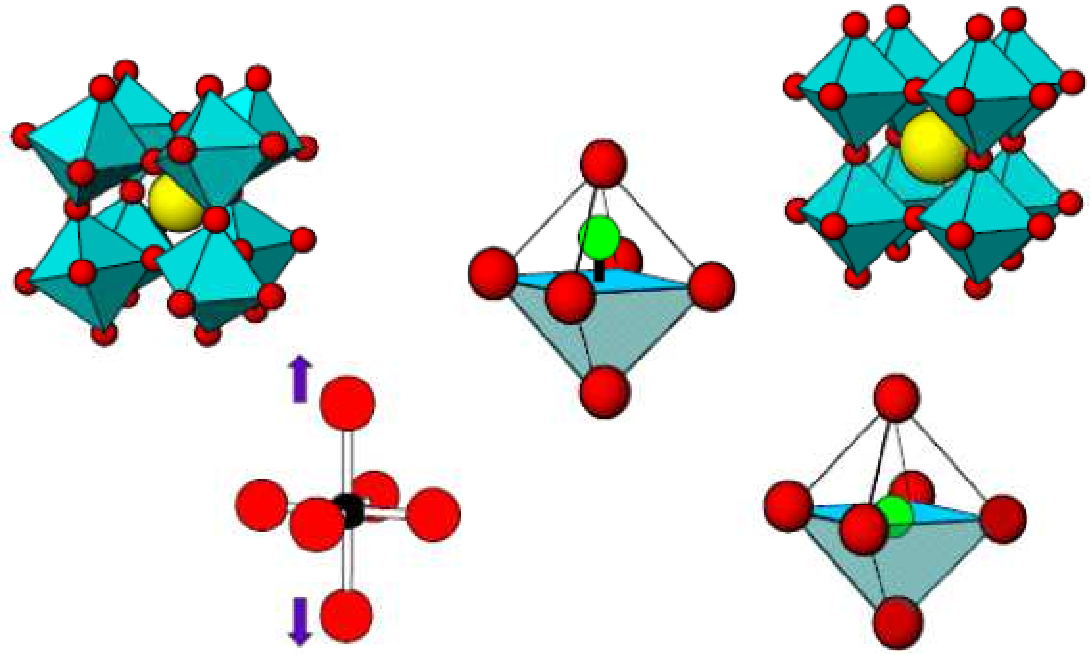
High pressure units

1 **bar** = 1 kg/cm^2 = 1013 hPa = 10^2 kPa = 10^{-1} MPa = 10^{-4} **GPa**
50 GPa = 500 kbar = 500 t/cm2





What is the effect of high pressure?



<u>High pressure:</u>

Strong modification of interatomic distances and bond angles
Modification / Tuning of coupling and physical properties
"Clean" parameter because it acts only on interatomic distances
Important changes in volume (chemical bonding)

0 to 700 K $\rightarrow \Delta V/V0 \sim 1\ \%$

1 bar to 100 GPa $\rightarrow \Delta V/V0 \approx 25\ \%$

~ 1 GPa

More elegant ways to apply high-pressure …

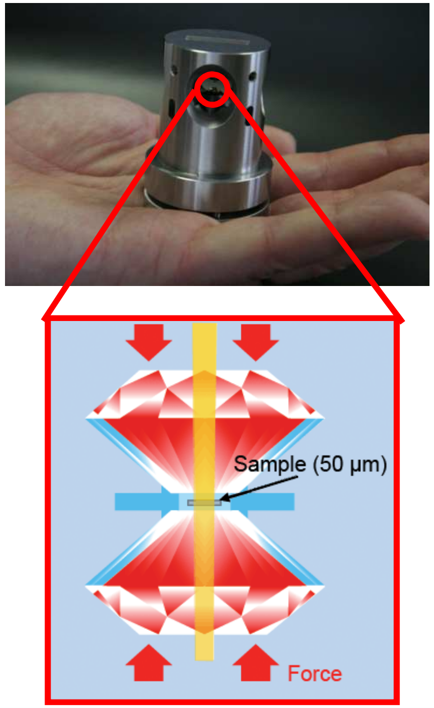
1) Large volume cells
2) Diamond anvil cells
3) ….

$$P = \frac{Force}{Surface}$$

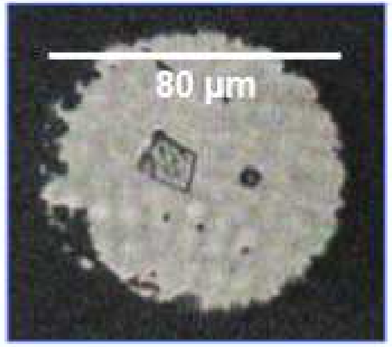
1.1) Multi anvil cell

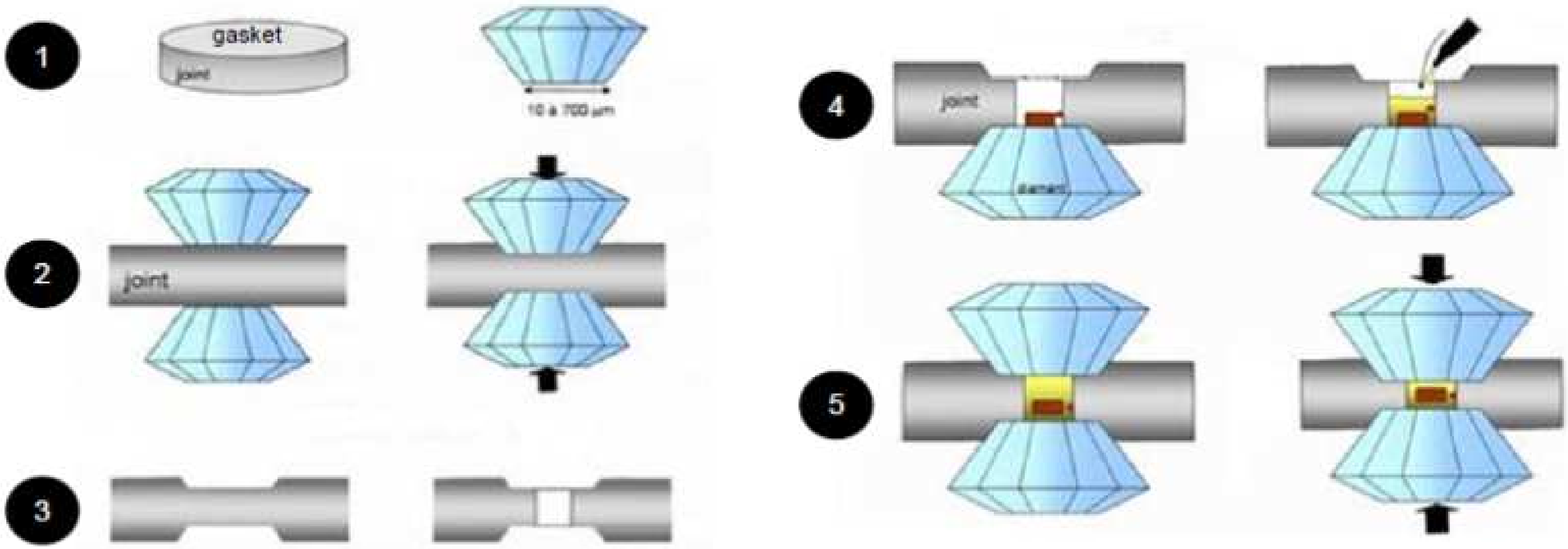
1.2) Paris-Edinburgh cell

2) Diamond anvil cell

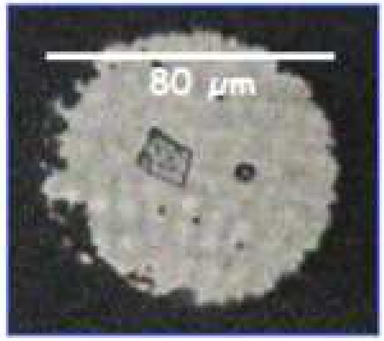


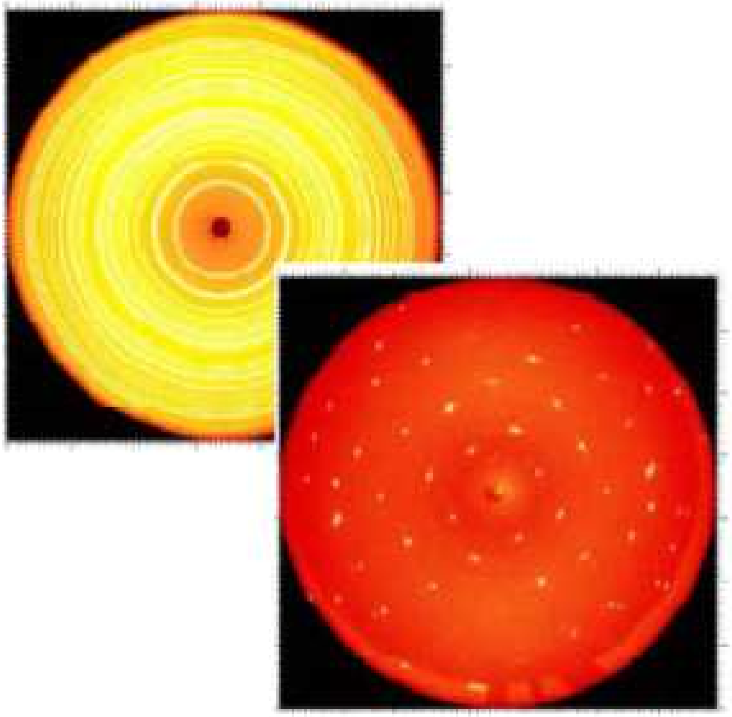
Diamond Anvil Cell



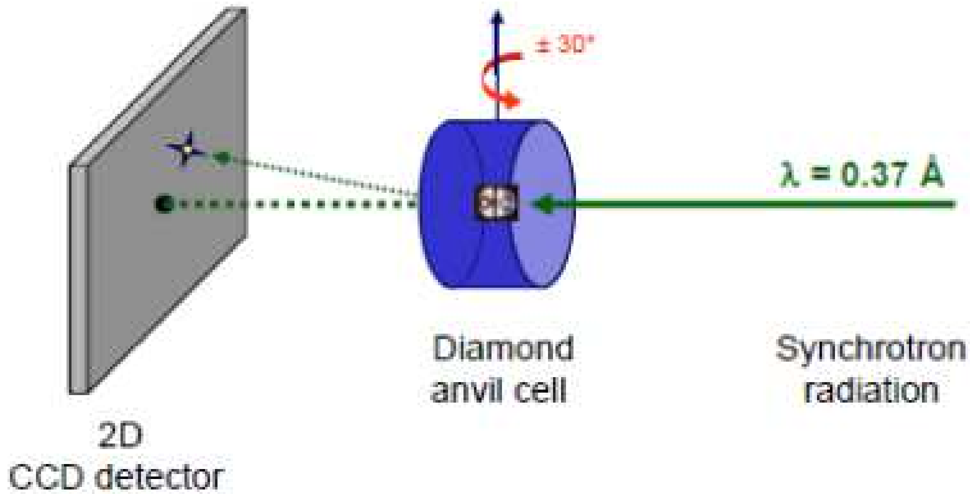


XRD using a DAC

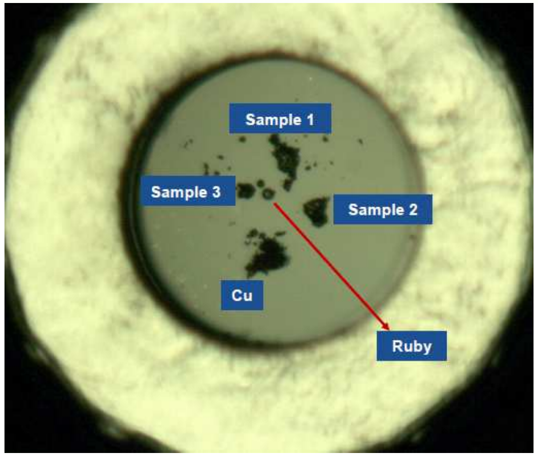




Synchrotron sources , ex. diffraction

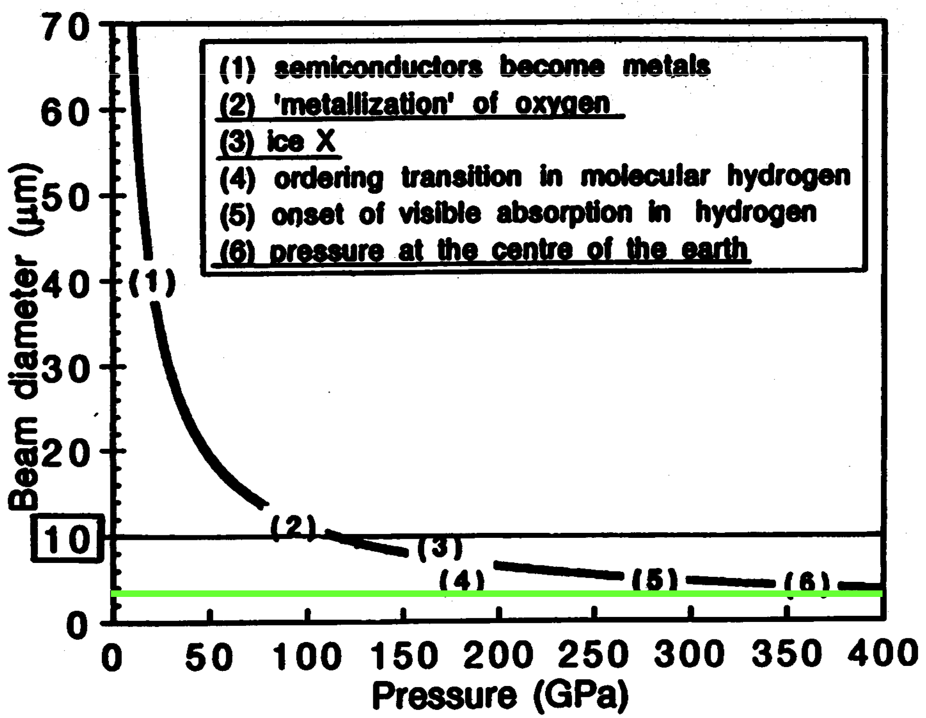


Diamond Anvil Cell + X rays



<100µm = 0.1mm

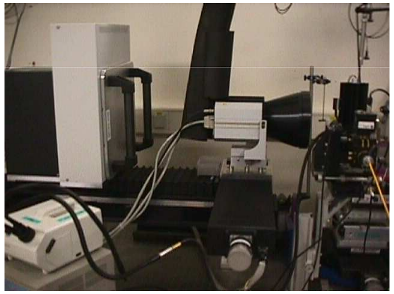
Need of very intense and very small X-ray beam.

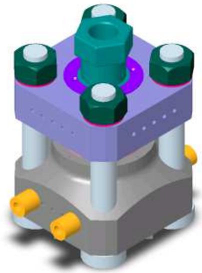


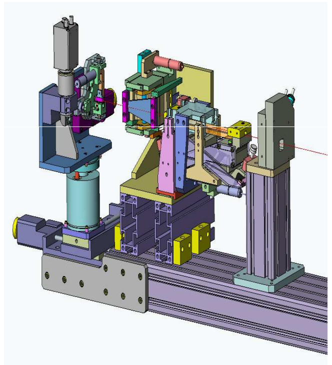
ID27 at ESRF (Grenoble, France)

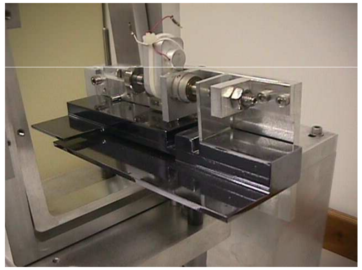
Very intense micro-focused beam (2 microns) using two KB mirrors at short wavelengths: $0.15<\lambda<0.4$ Å

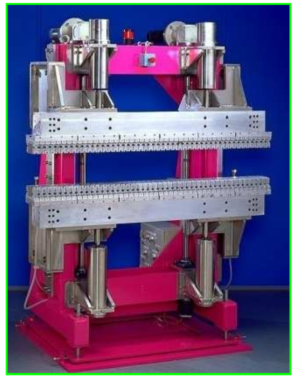
ESRF
6 GeV











Detectors

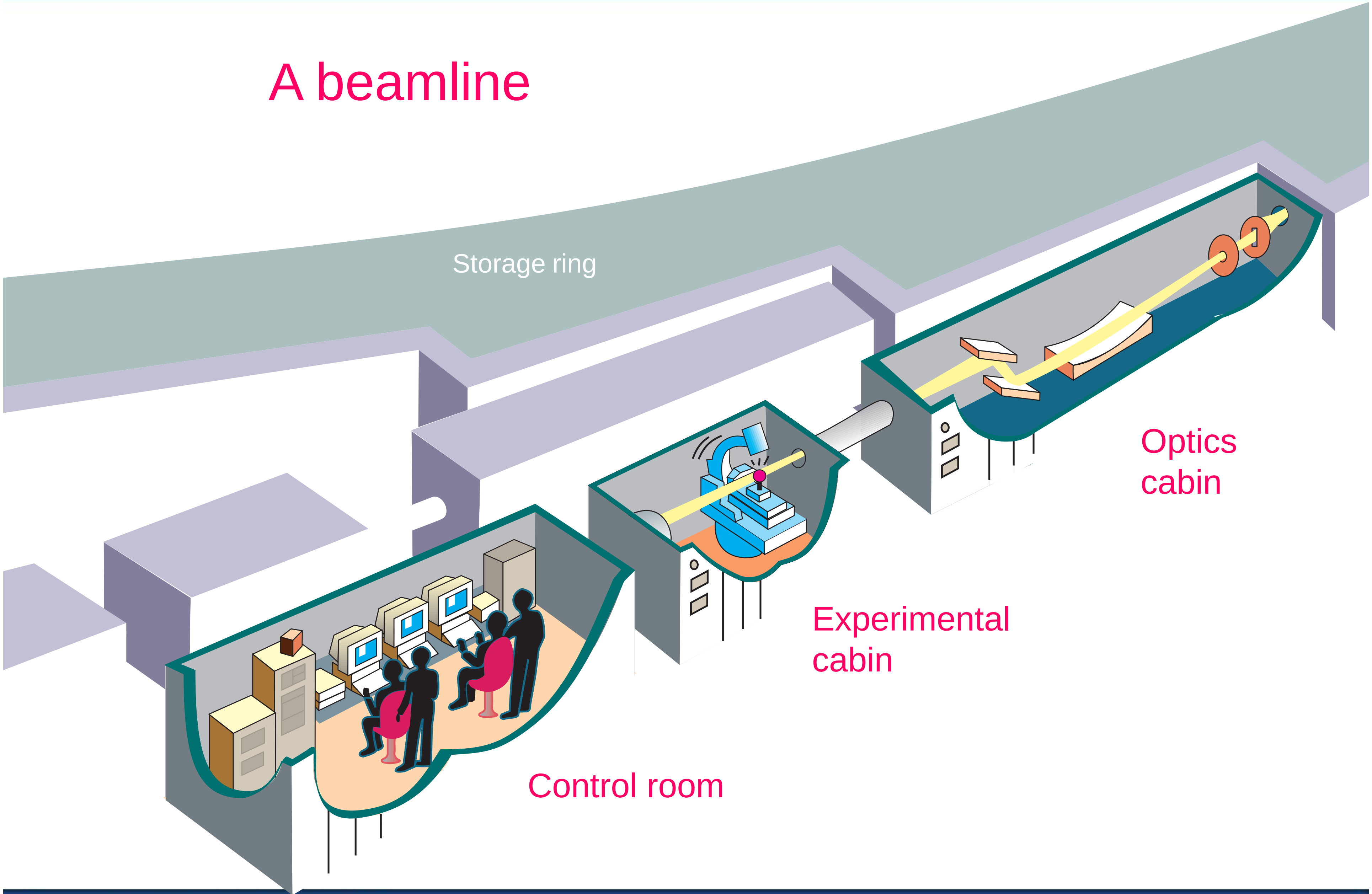
Sample environment

Mirrors

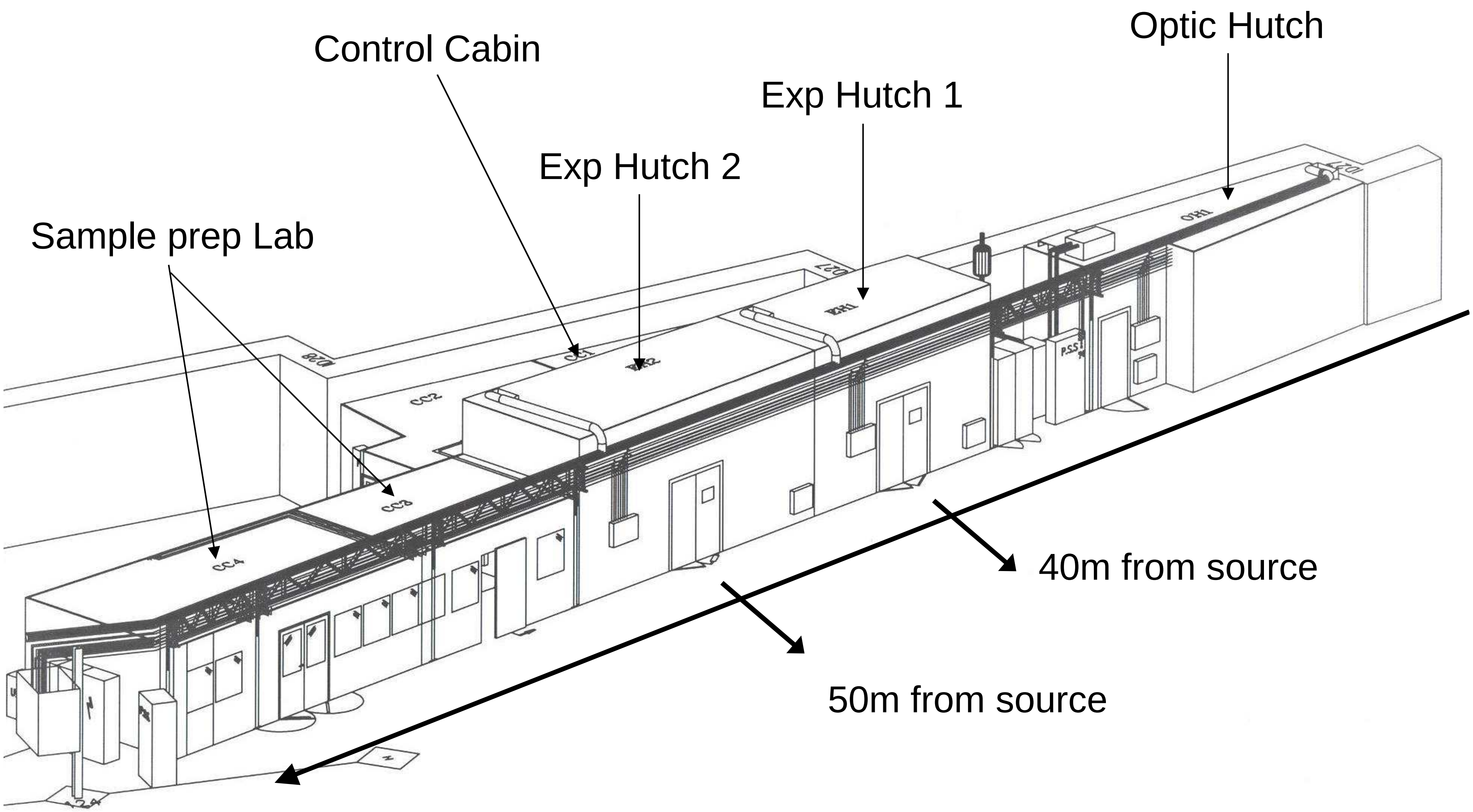
Monochromator

X-ray Source

A beamline

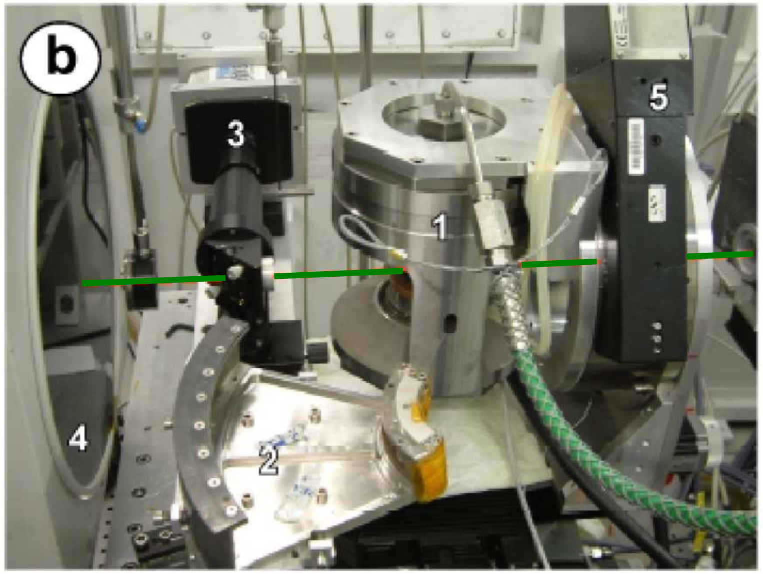

Storage ring
Optics cabin
Experimental cabin
Control room

ID27 at ESRF (Grenoble, France)



ID27 – EH1

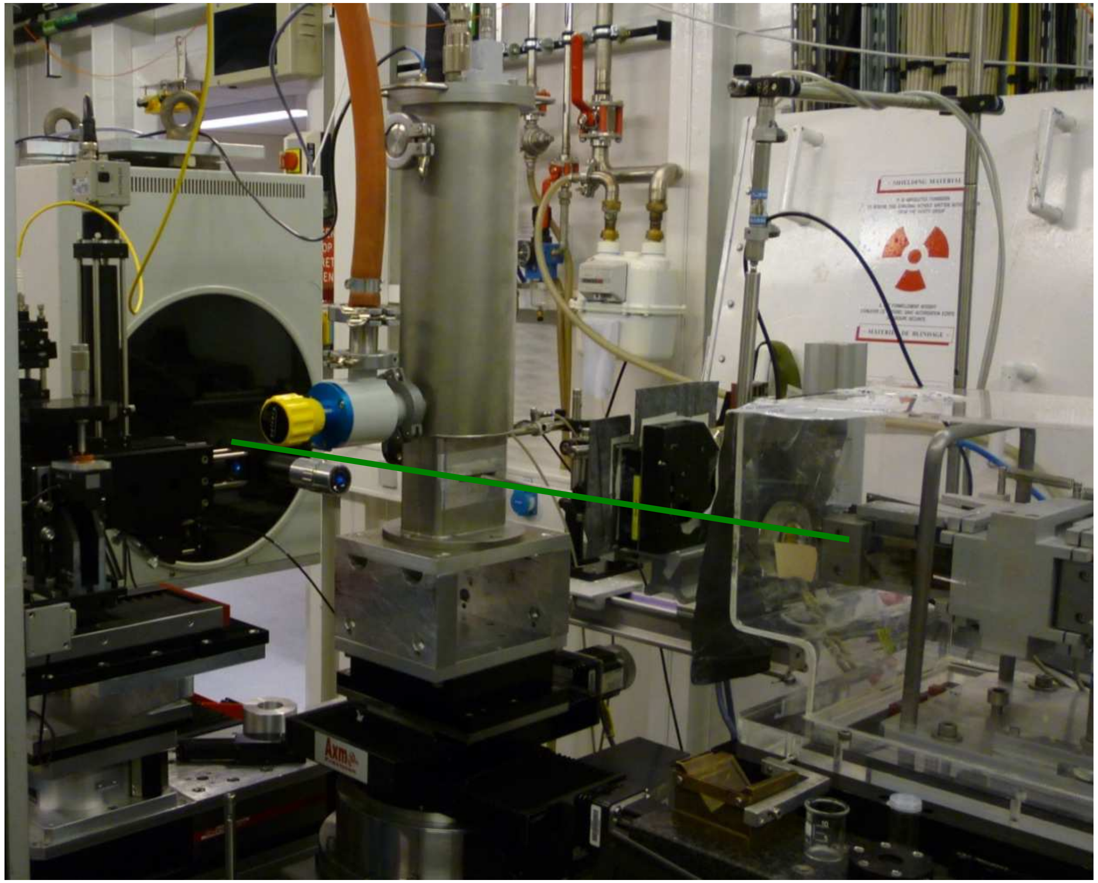
- **X-Ray Diffraction/Viscosity/Density/Tomography on powders-liquids-amorphous samples**
- **Paris-Edinburgh large volume cell**
- The only monochromatic large volume cell
- Pressure up to **17 GPa** on **5mm³** sample volume
- High temperature **T<2300 K**



RoToPEC

ID27 – EH1

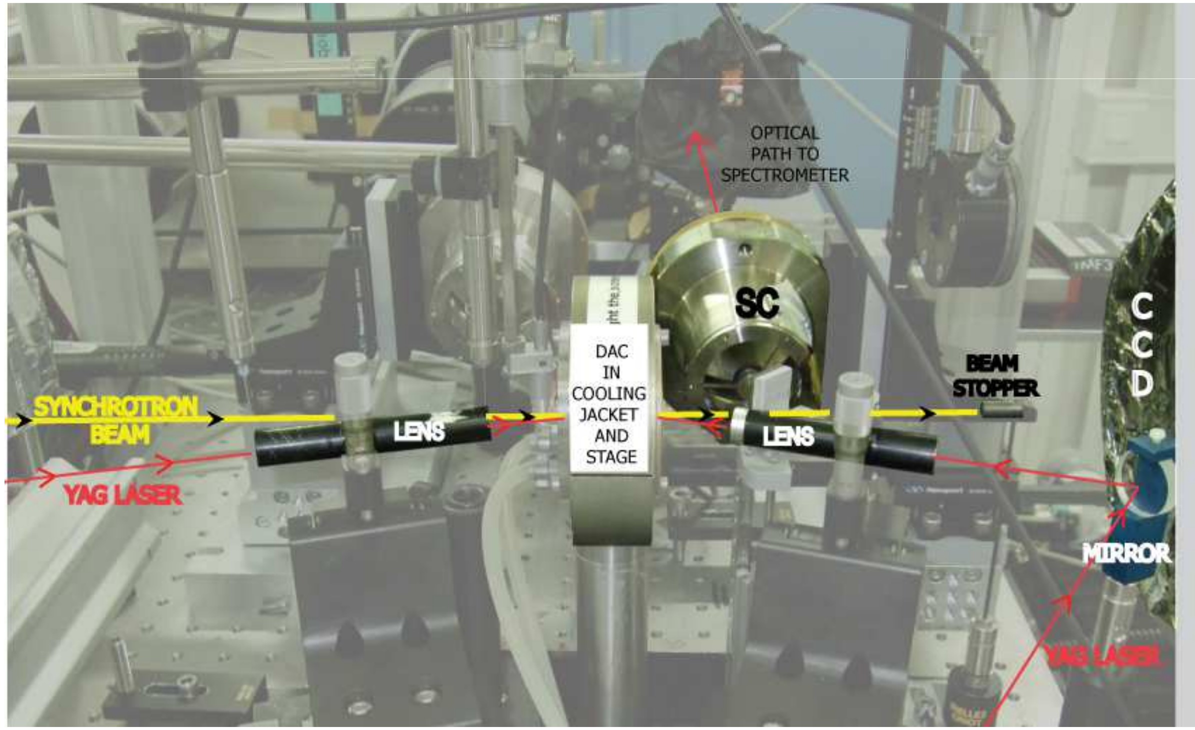
- **X-Ray Diffraction on single crystals and powders with DAC**
- **Diamond anvil cell**
- Accessible PT domain for in situ powder XRD: **P>300GPa; T>4K**
(helium flow cryostats)



ID27 – EH2

- **X-Ray Diffraction on single crystals and powders with DAC**
- **Diamond anvil cell**
- Accessible PT domain for in situ powder XRD: **P>300GPa; T>5000 K**

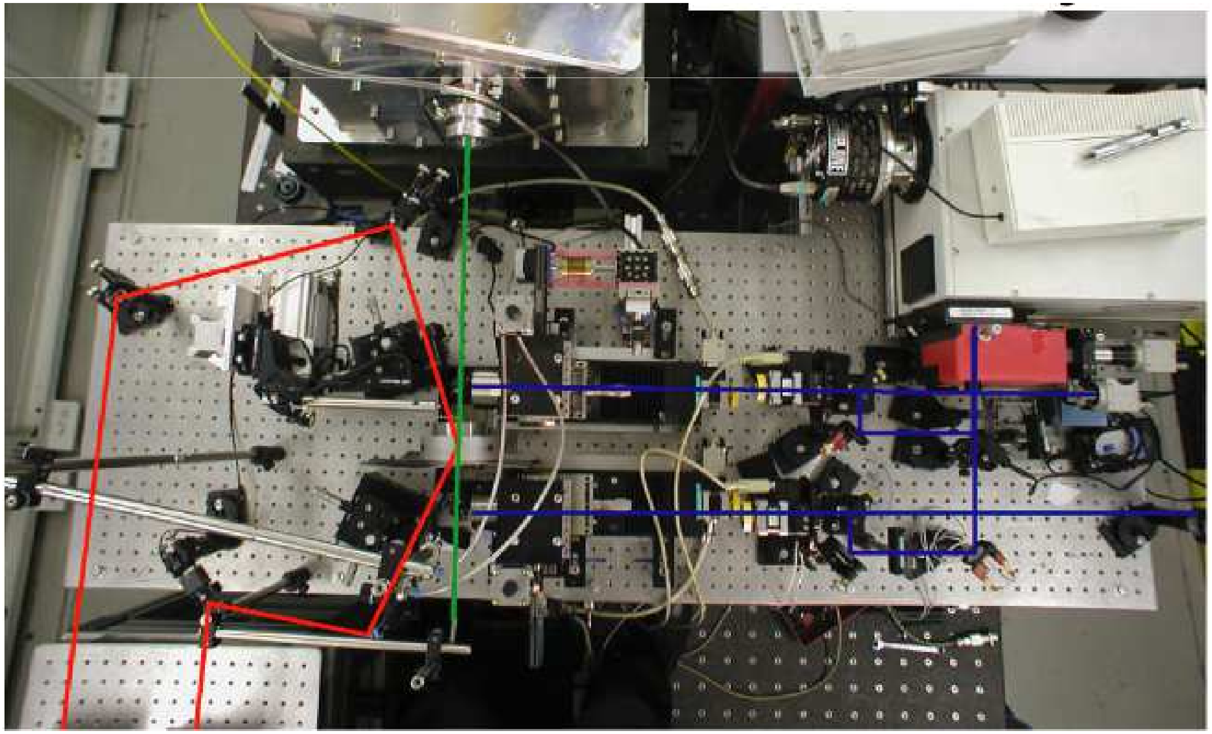
(YAG, CO_2 lasers)



ID27 – EH2

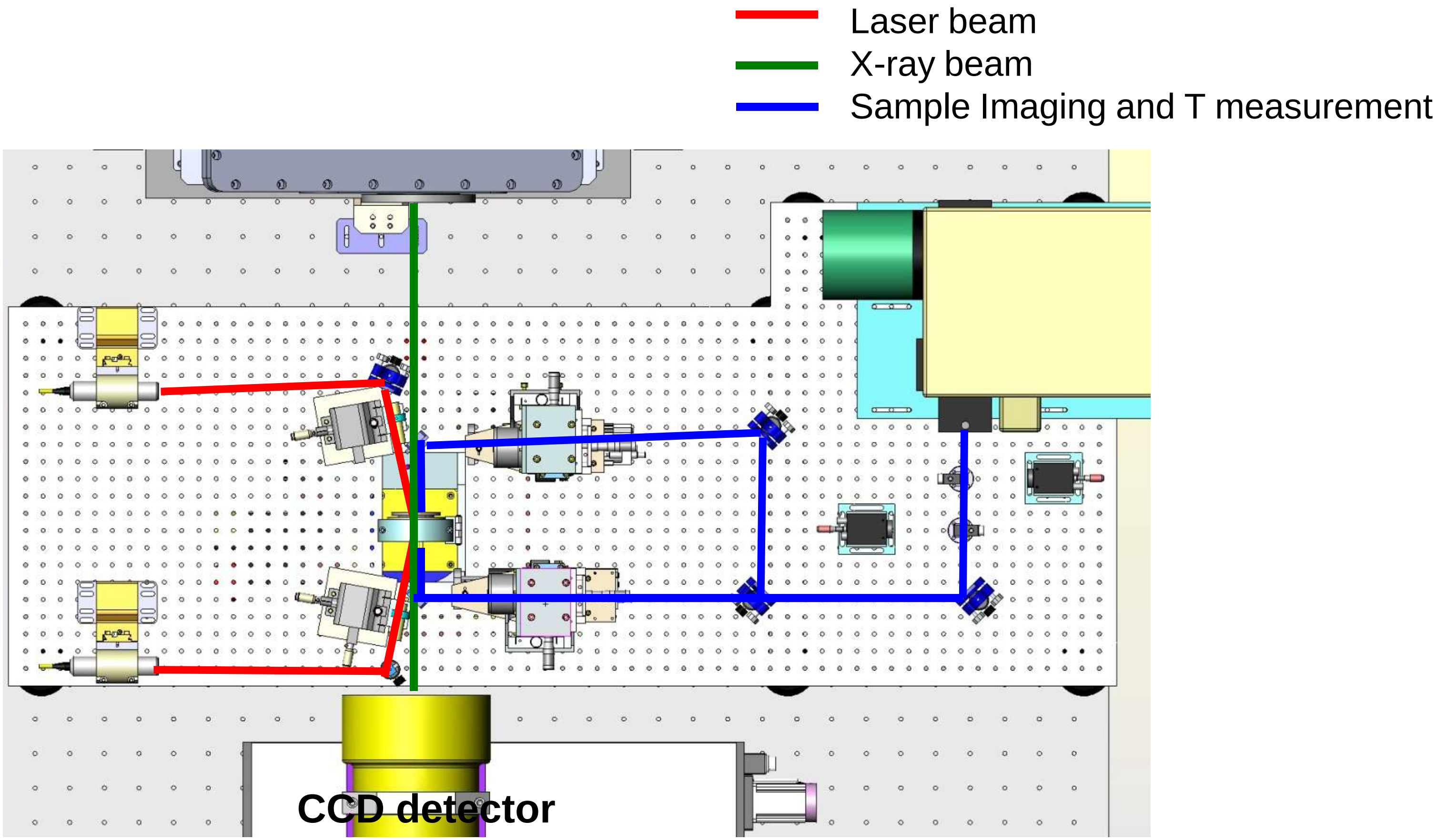
- **X-Ray Diffraction on single crystals and powders with DAC**
- **Diamond anvil cell**
- Accessible PT domain for in situ powder XRD: **P>300GPa; T>5000 K**
(YAG, CO_2 lasers)

Laser beam
X-ray beam
Sample Imaging and T measurement



ID27 – EH2

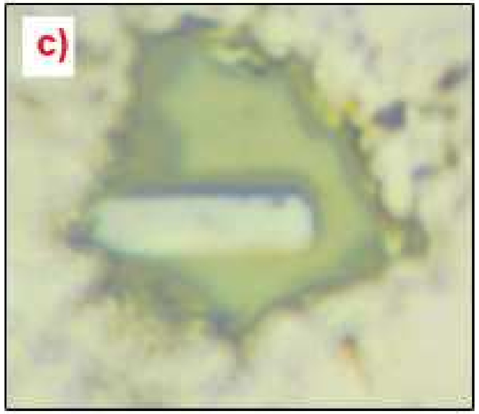
- **X-Ray Diffraction on single crystals and powders with DAC**
- **Diamond anvil cell**
- Accessible PT domain for in situ powder XRD: **P>300GPa; T>5000 K** (YAG, CO_2 lasers)



Example of high pressure experiments

Structure of metallic oxygen

P=138 GPa



5 microns single crystal
of oxygen in
a 20µm gasket hole

O_2 becomes metallic at P~96 GPa

Structure of the metallic phase?

Liquid and glassy states at HP

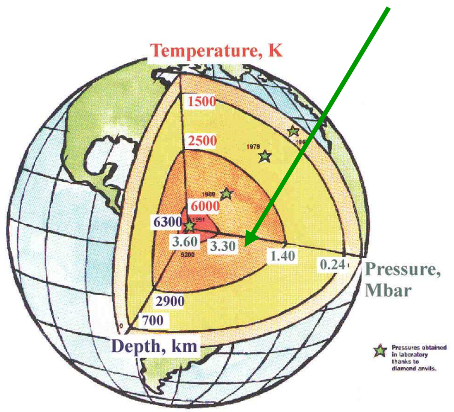
Physics, chemistry and biology

•Effect of pressure on chemical bonds: neighbors distances, coordination number, angles...

•Structural relations between polymorphs in the solid and liquid states at high pressure are poorly understood.

•Melting curves

Liquid Fe



Geophysics

•Determination of planets cores structures

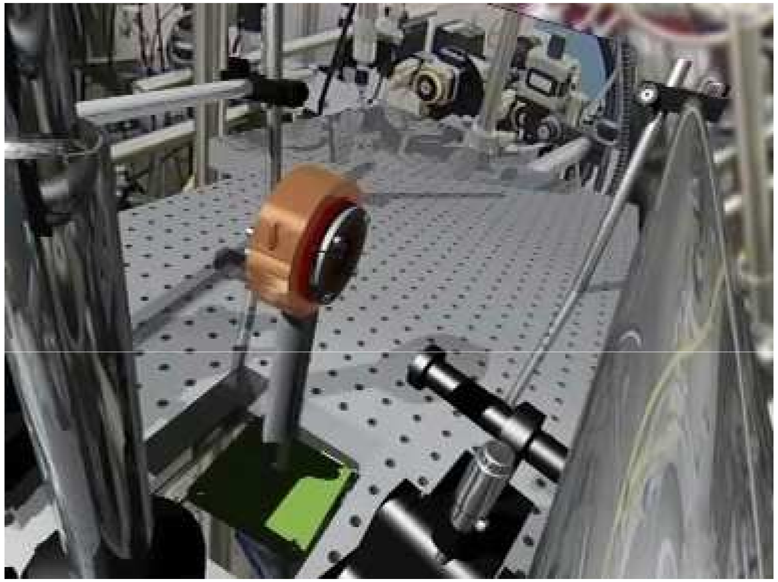
•Effect of light elements

•Water in the Earth's upper mantle

•Magmas...

Melting at HP

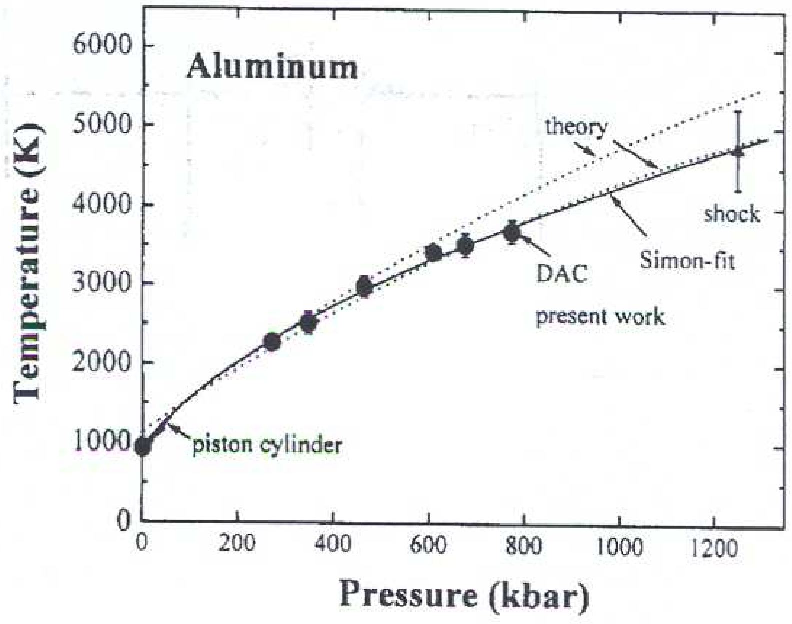
One can better know the structure of matter at the center of the Earth …

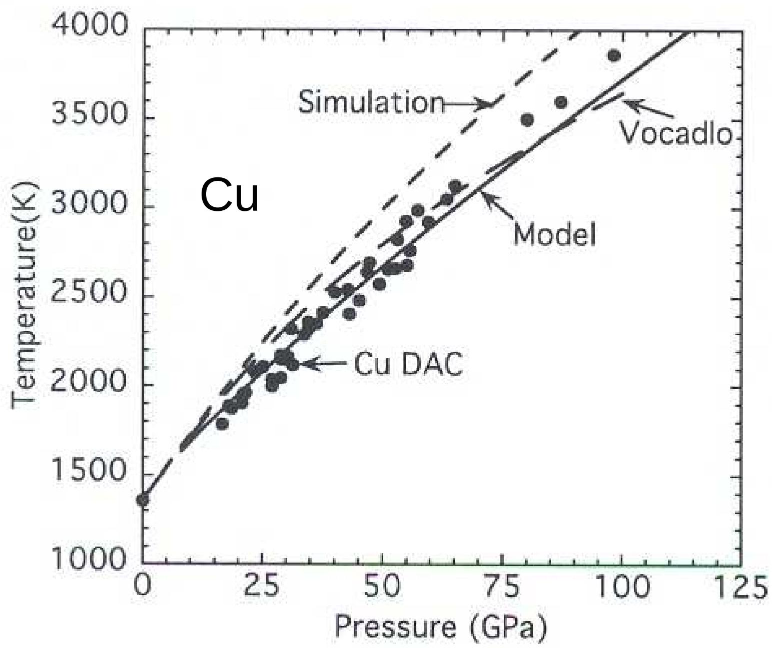


… by studying samples put under extreme conditions of pressure and temperature.

Melting at HP

Good agreement between DAC, shock compression and theory for many systems: i.e. Al, Cu



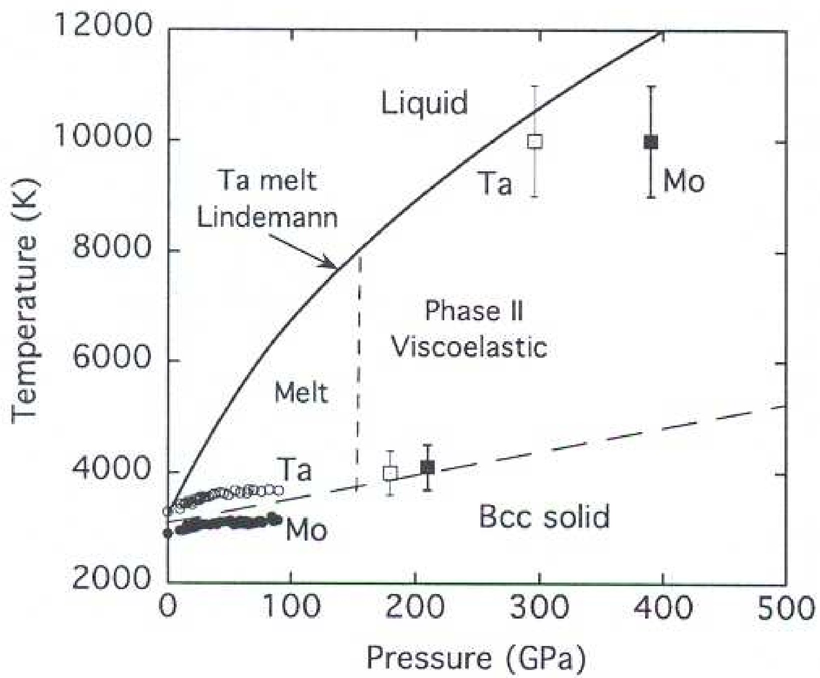


Ref. : Al: R. Boehler, M. Ross, EPSL, 153, 223(1997)
Cu: M. Ross, R. Boehler, D. Errandonea, PRB, 76, 184117(2007)

Melting at HP

But also some fundamental disagreements:

Most famous: Iron but also other transition metals such as Ta, W, Mo, Fe…



Ref.: M. Ross, D. Errandonea, R. Boehler, PRB, 77, 184118 (2007)

Why???

↓

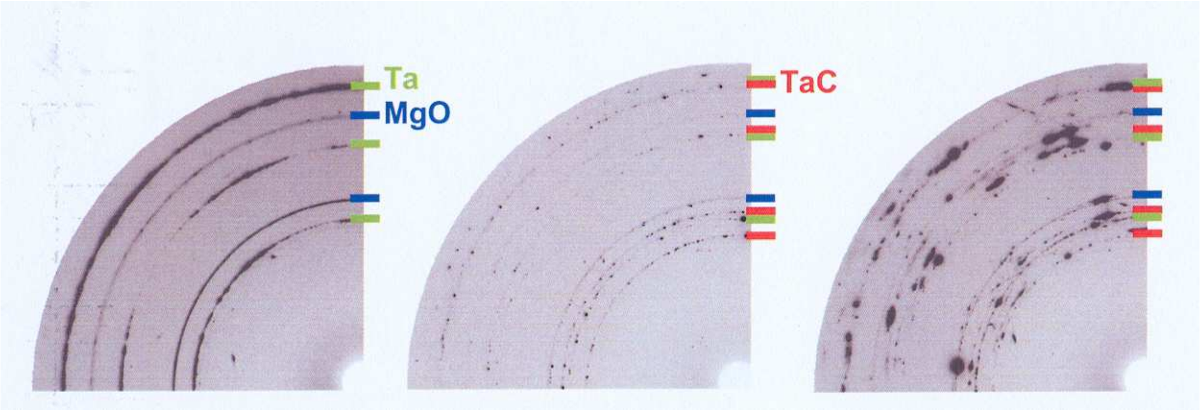
Reaction with diamonds

Synthesis of carbides

….

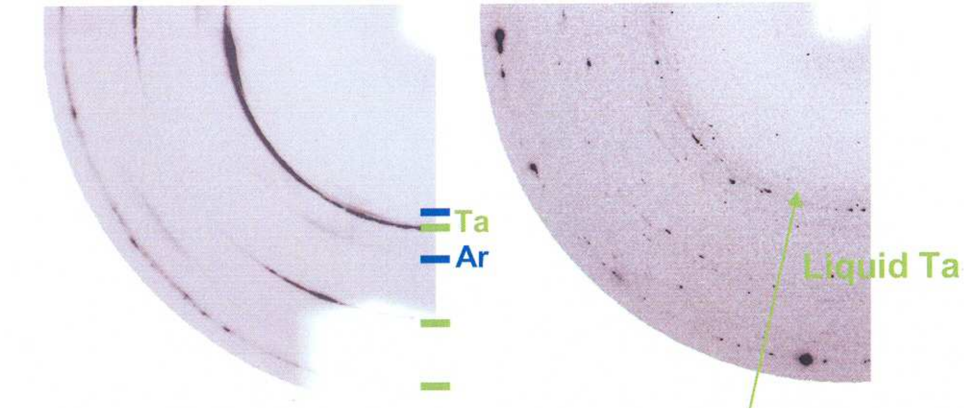
Melting of Ta @ HP

Ta in MgO (pressure medium)

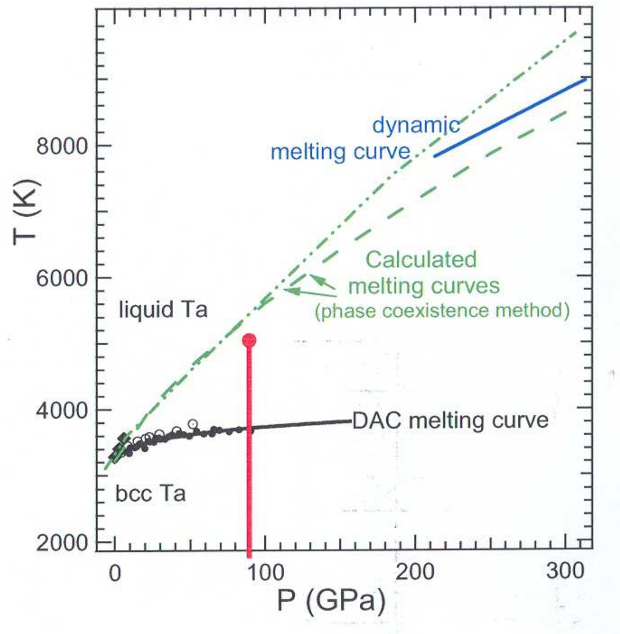


Before heating | During heating | After heating: ~ 50% of Ta transformed into TaC

Ta in argon (pressure medium) Al_2O_3 coated diamonds
Still some traces of TaC but much less than without coating

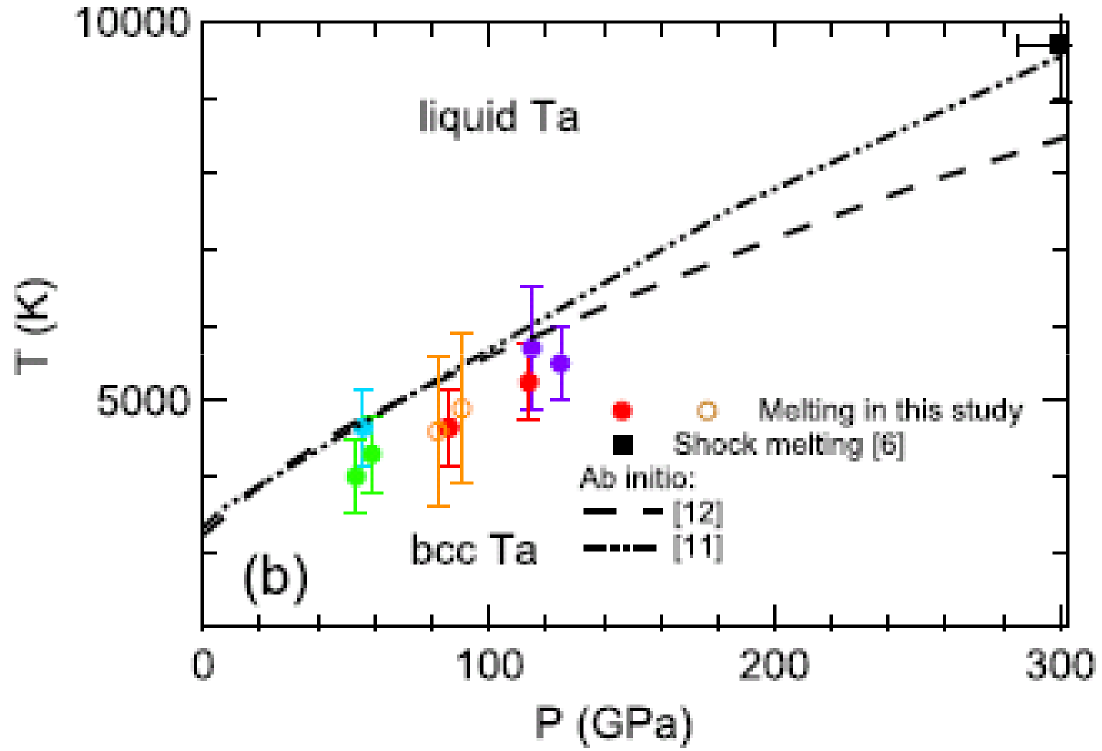


Before heating | During heating



Dewaele et al, Phys. Rev. Lett. 104, 255701 (2010)

Melting of Ta @ HP

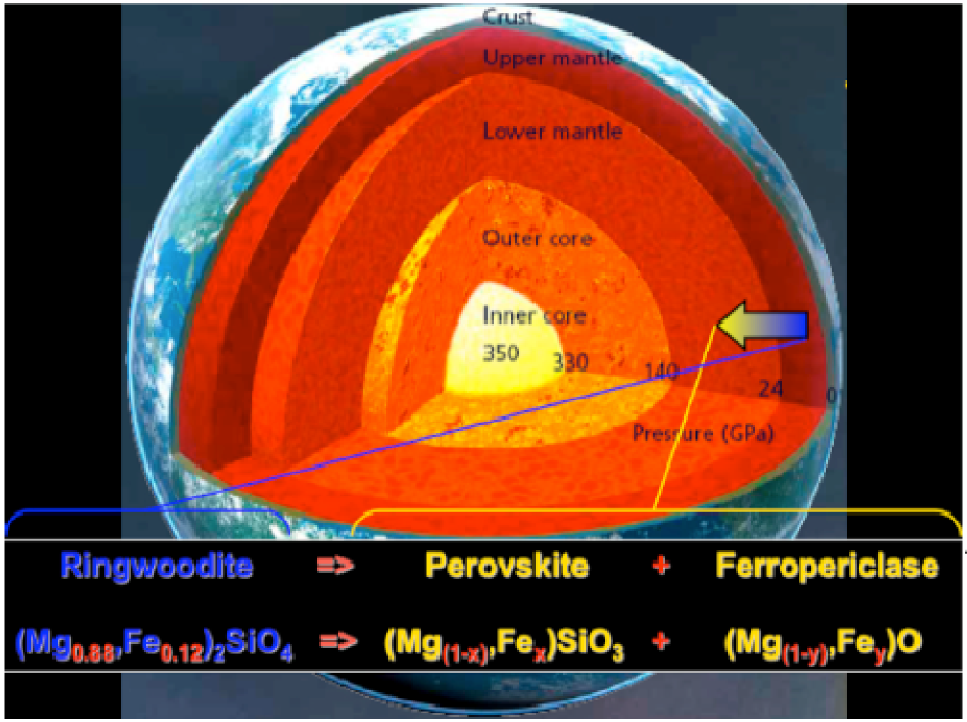


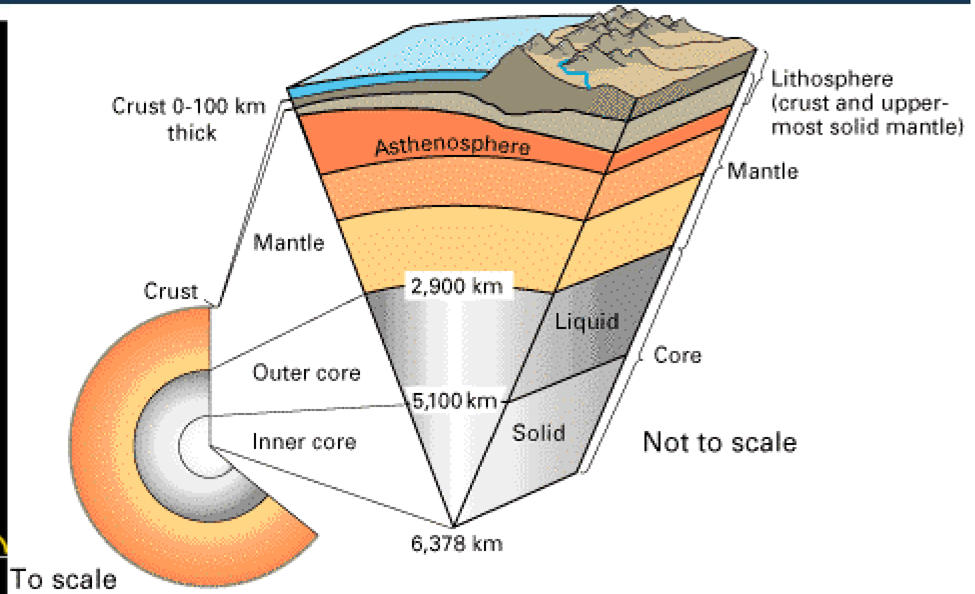
Melting curve of Ta in good agreement with theory and shock data

Used pressure medium: MgO, Ar, Ne, NaCl, Al_2O_3, KCl

Dewaele et al, Phys. Rev. Lett. 104, 255701 (2010)

Melting of peridotite





Peridotite is the dominant rock of the upper part of the Earth's mantle. + X-ray diffraction at high pressure – temperature

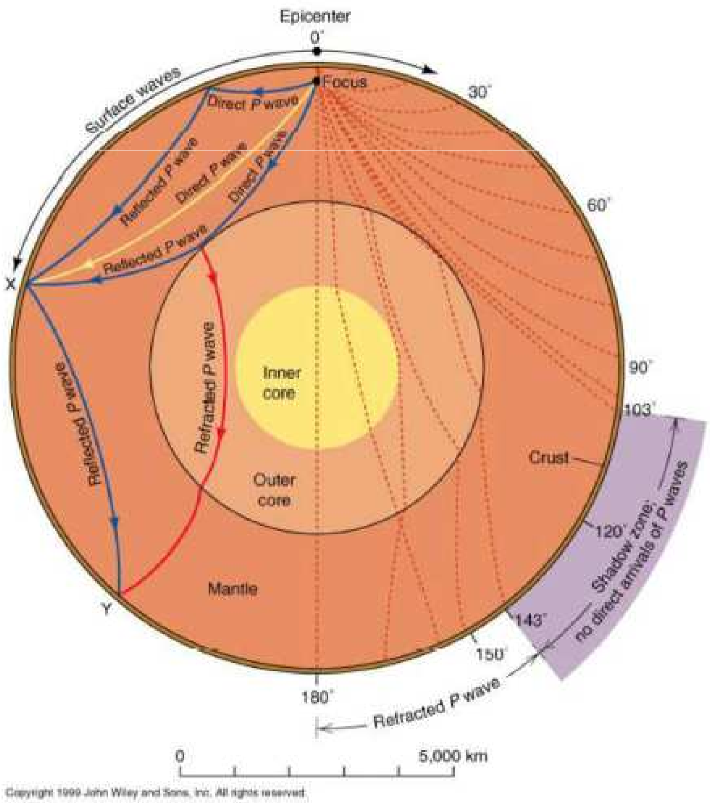
Reproduce the Earth interior in a laboratory is possible!!!

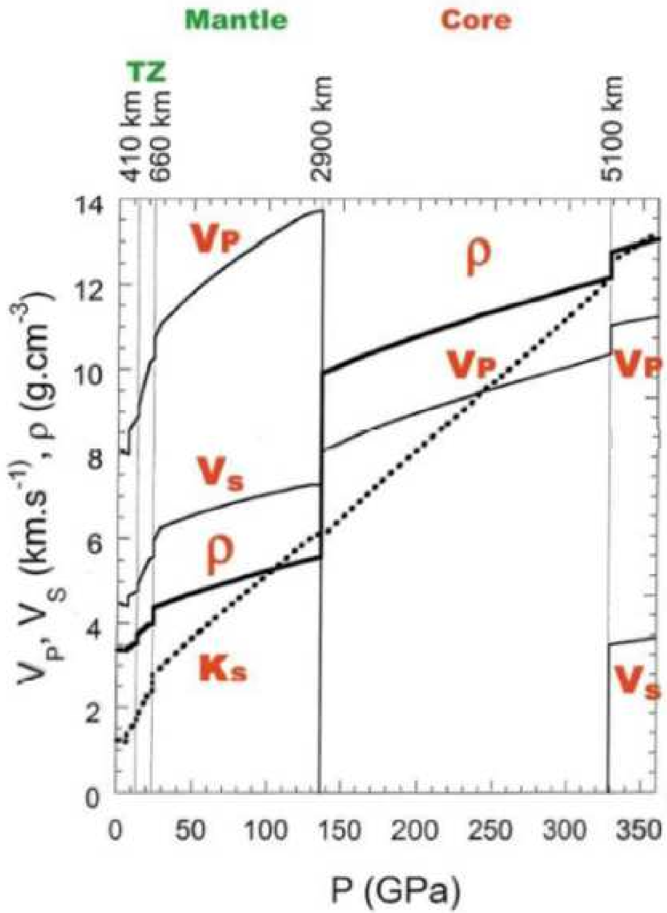
Melting of peridotite

Geophysics

Determination of planets cores structures
Effect of light elements
Water in the Earth's upper mantle
Magmas...

Seismic waves



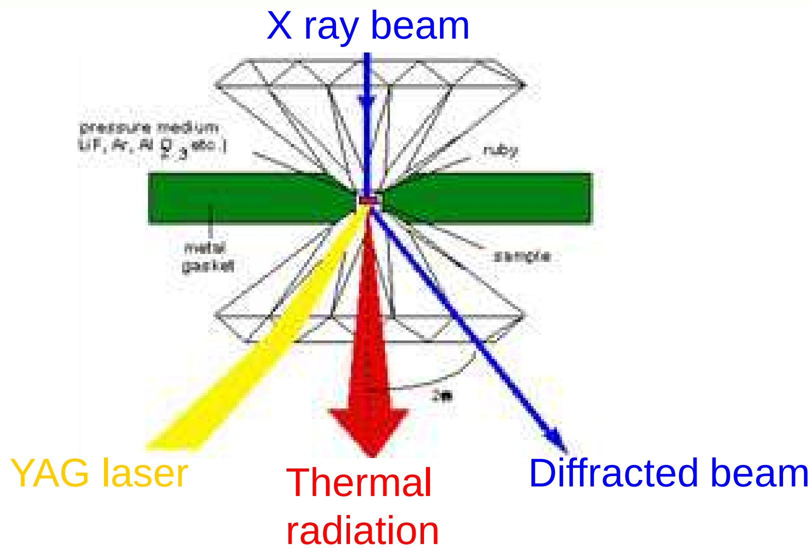


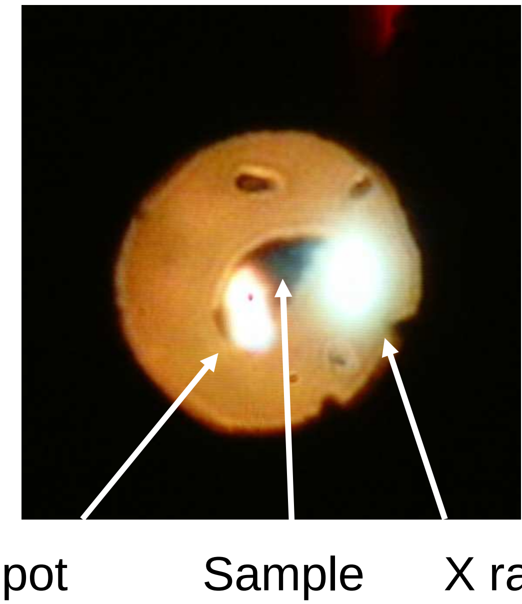
Preliminary Reference Earth Model
Dziewonski and Anderson (1981)
Phys. Earth and Planetary Interiors, 25 297

Melting of peridotite

Reproduce the Earth interior in a laboratory is possible!!!

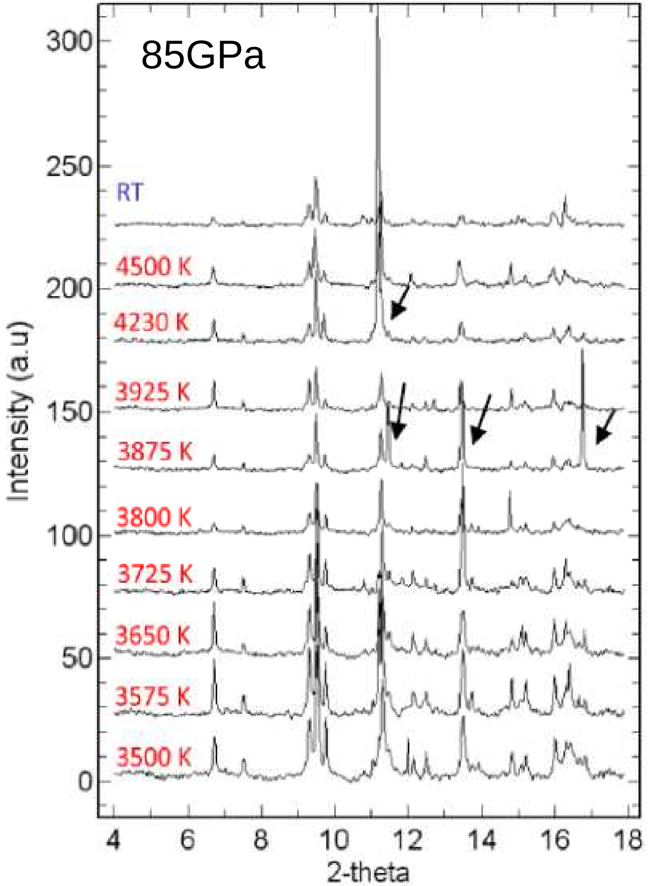
Natural fertile peridotite + DAC (P<140GPa) + X ray diffraction + laser heating up to 5500K

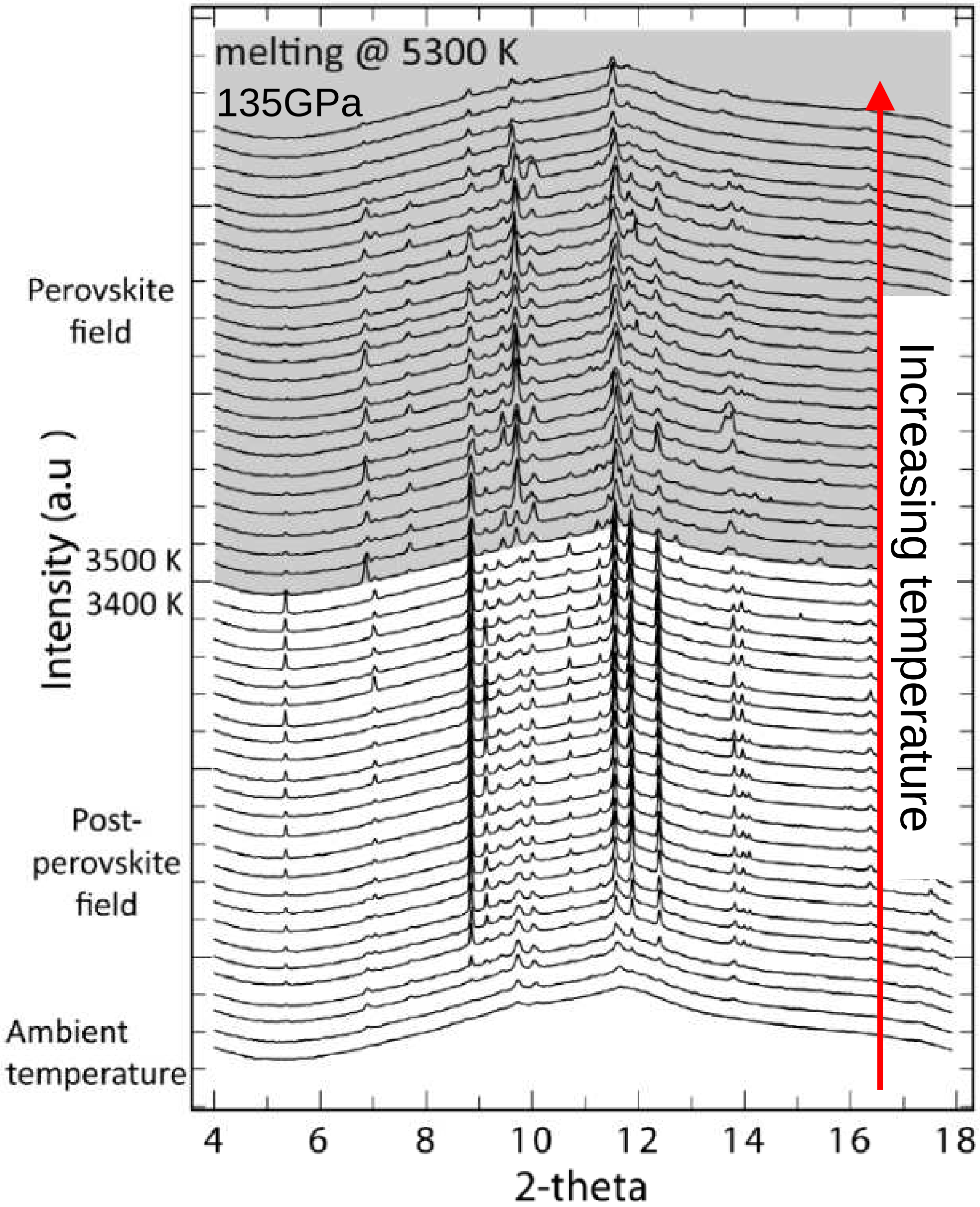




Melting of peridotite

Natural fertile peridotite +
DAC (P<140GPa) +
X ray diffraction +
laser heating up to 5500K

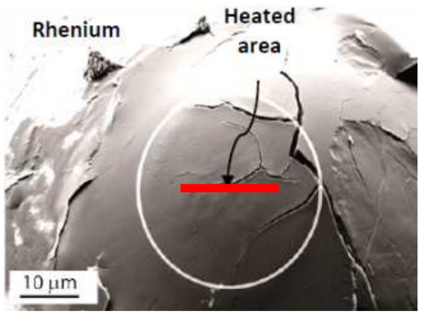


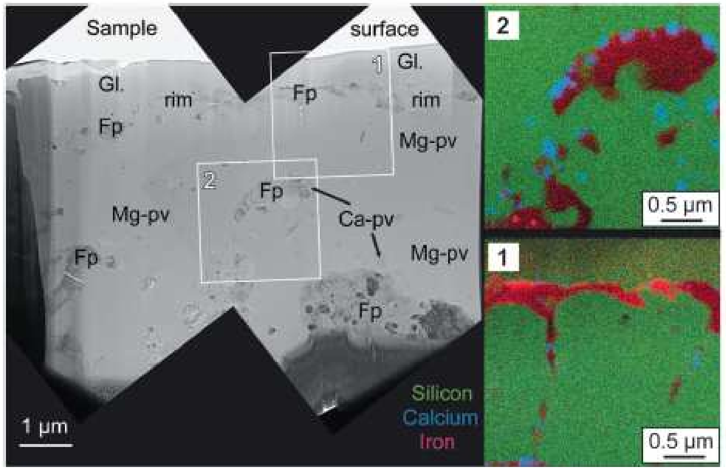


Fiquet et al, Science 329 p1516 (2010)

Melting of peridotite at 140GPa

Natural fertile peridotite +
DAC (P<140GPa) +
X ray diffraction +
laser heating up to 5500K +
FIB analysis on recovered samples

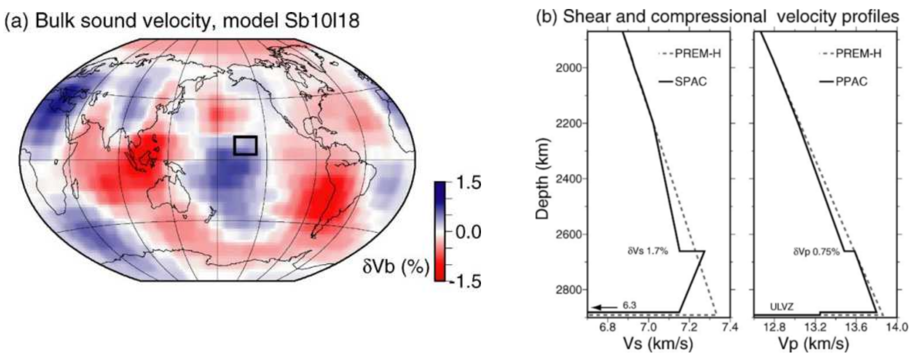




Fiquet et al, Science 329 p1516 (2010)

Melting of peridotite at 140GPa

Deep mantle melting

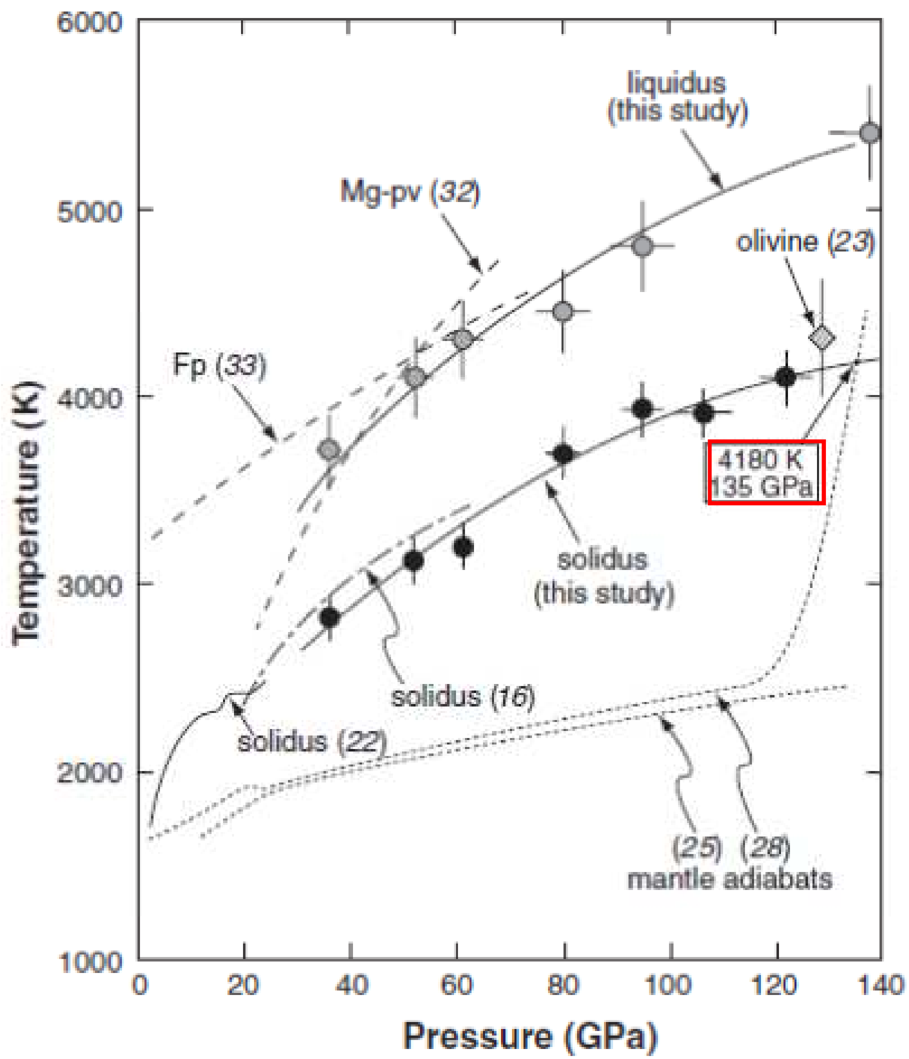


Melting of Peridotite to 140 Gigapascals

G. Fiquet,[1*] A. L. Auzende,[1] J. Siebert,[1] A. Corgne,[2,3] H. Bureau,[1] H. Ozawa,[1,4] G. Garbarino[5]

Partial melting in the deepest part of the mantle is highly plausible and explain the presence of ULVZs.
Match between the average geotherm and mantle solidus at the CMB may also explain why ULVZs are not observed as a continuous layer in the deep mantle.

Natural fertile peridotite

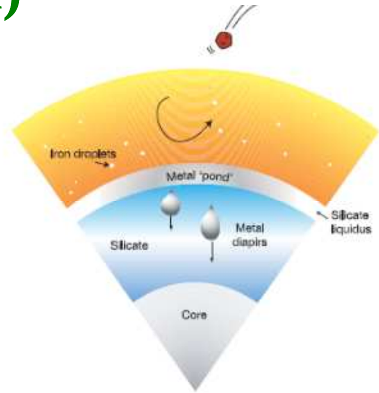


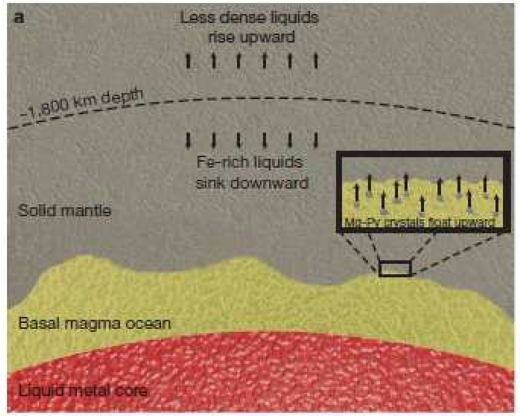
Fiquet et al, Science 329 p1516 (2010)

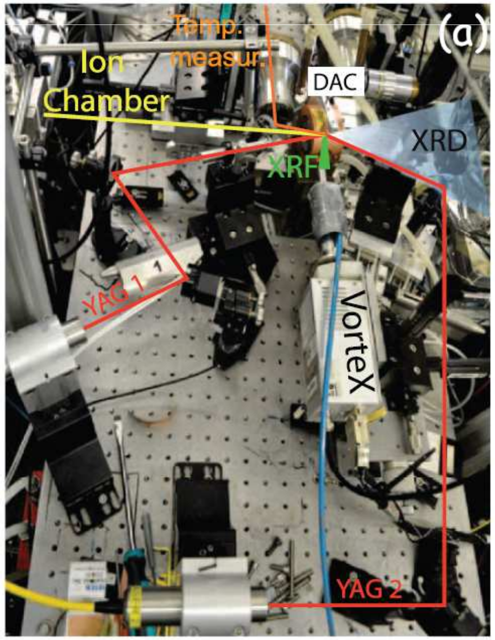
Iron partitioning

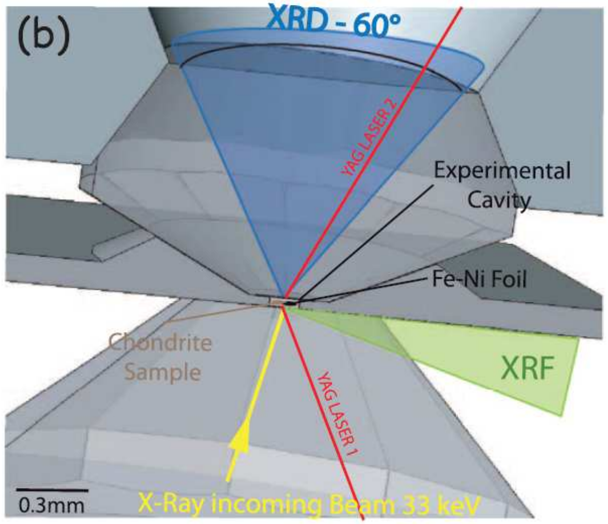
DAC: X ray diffraction (P<100GPa)
+ laser heating up to 5000K
+ X ray fluorescence

Deep mantle melting









Andrault et al, Nature, 487, 354 (2012)

Iron partitioning

DAC: X ray diffraction (P<100GPa)
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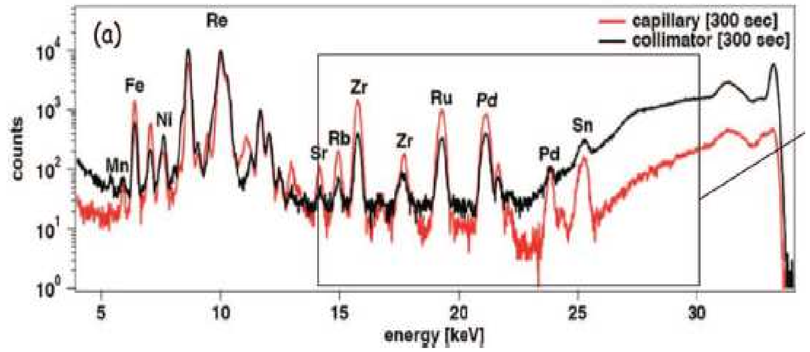
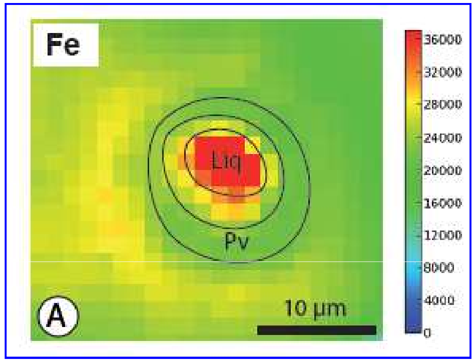


Chondritic mantle glass in LH-DAC at ID27

XRF mapping

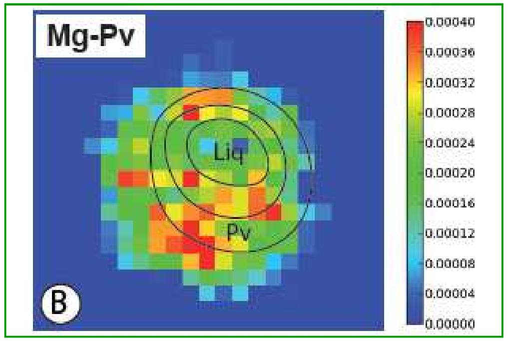


Chemical

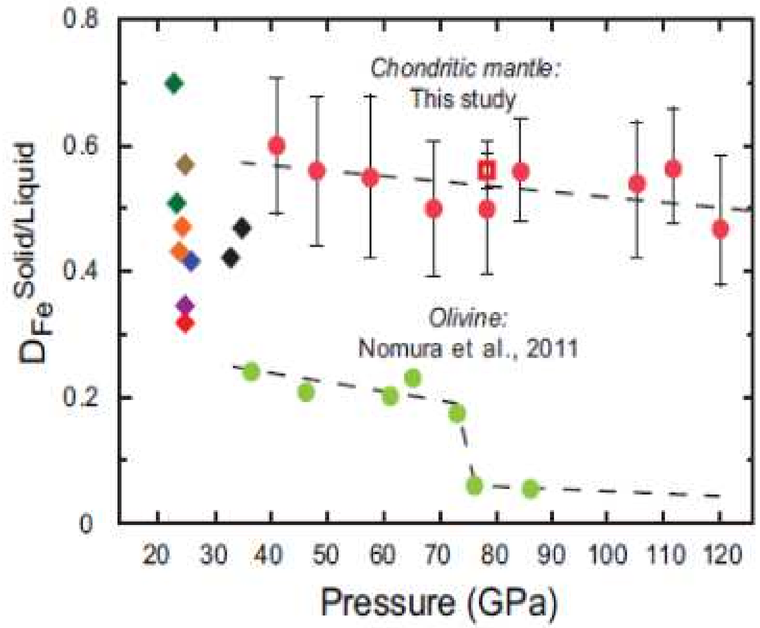
+

Distribution

XRD mapping



Phases

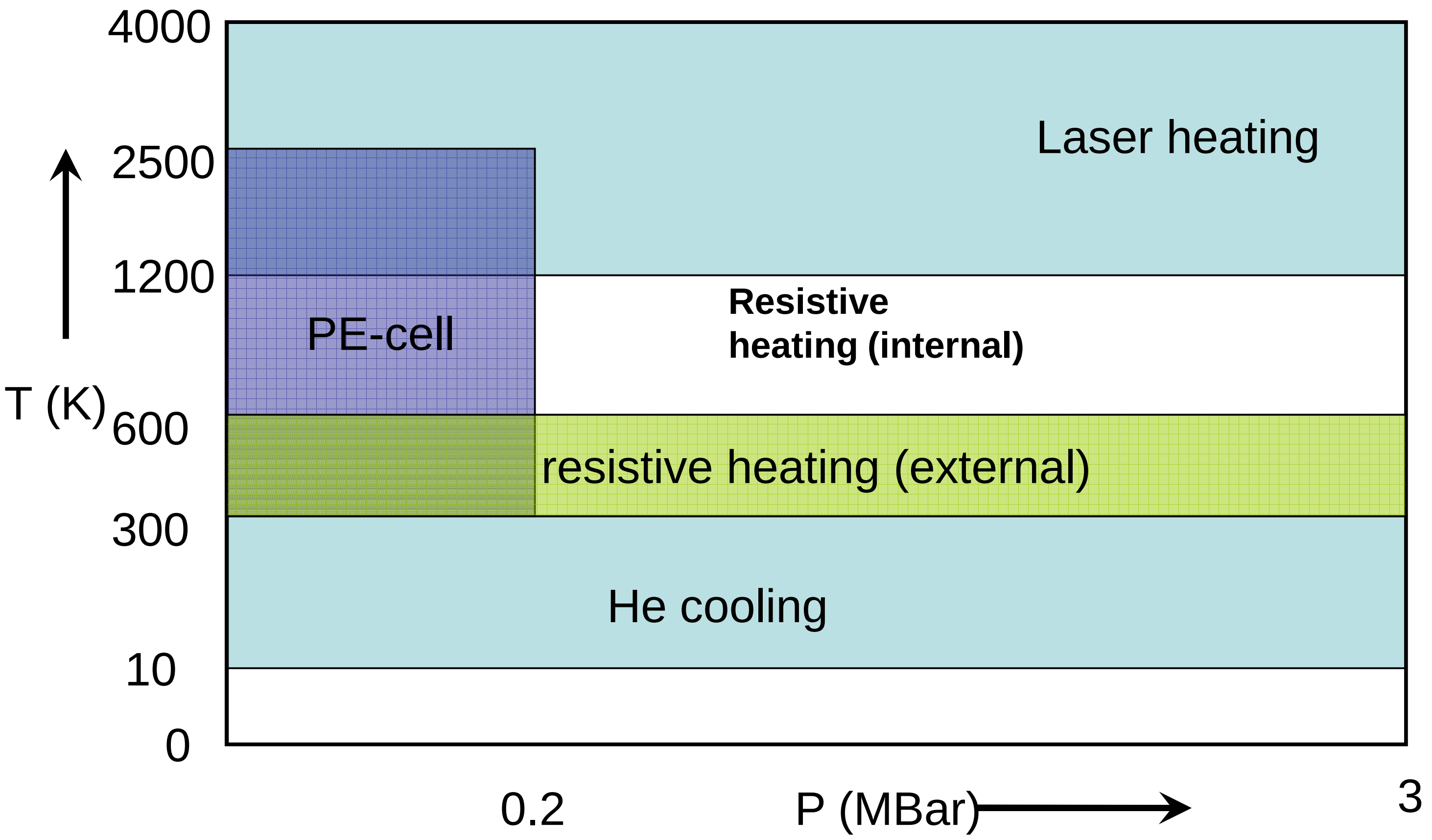


- ppm Molal
- spatial resolution 2μm

Andrault et al, Nature, 487, 354 (2012)

Summary

Temperature vs. Pressure diagram – HP/HT technologies using Synchrotron radiation



+ Time resolution

Thank you for your attention

For comments, suggestions, support request etc...

contacts:

Dr Gaston Garbarino

e-mail: gaston.garbarino@esrf.fr

address: European Synchrotron Radiation Facility (ESRF)

6 Rue Jules Horowitz, 38043, Grenoble, France

Melting at HP

New approach developed at beamline ID27 :

⇒ Fast *in situ* X-ray diffraction in the double-sided laser heated diamond anvil cell.

Advantages:
- It is sensitive to the bulk of the sample (#surface)
- The XRD measurements are performed at thermodynamic equilibrium
- It uses well established pyrometric methods

Also very important:

-X-ray diffraction in the laser heated DAC provides a clear signature of the melt: X-ray diffuse scattering

- and identifies chemical reactions if any

Large volume cell: P-E cell

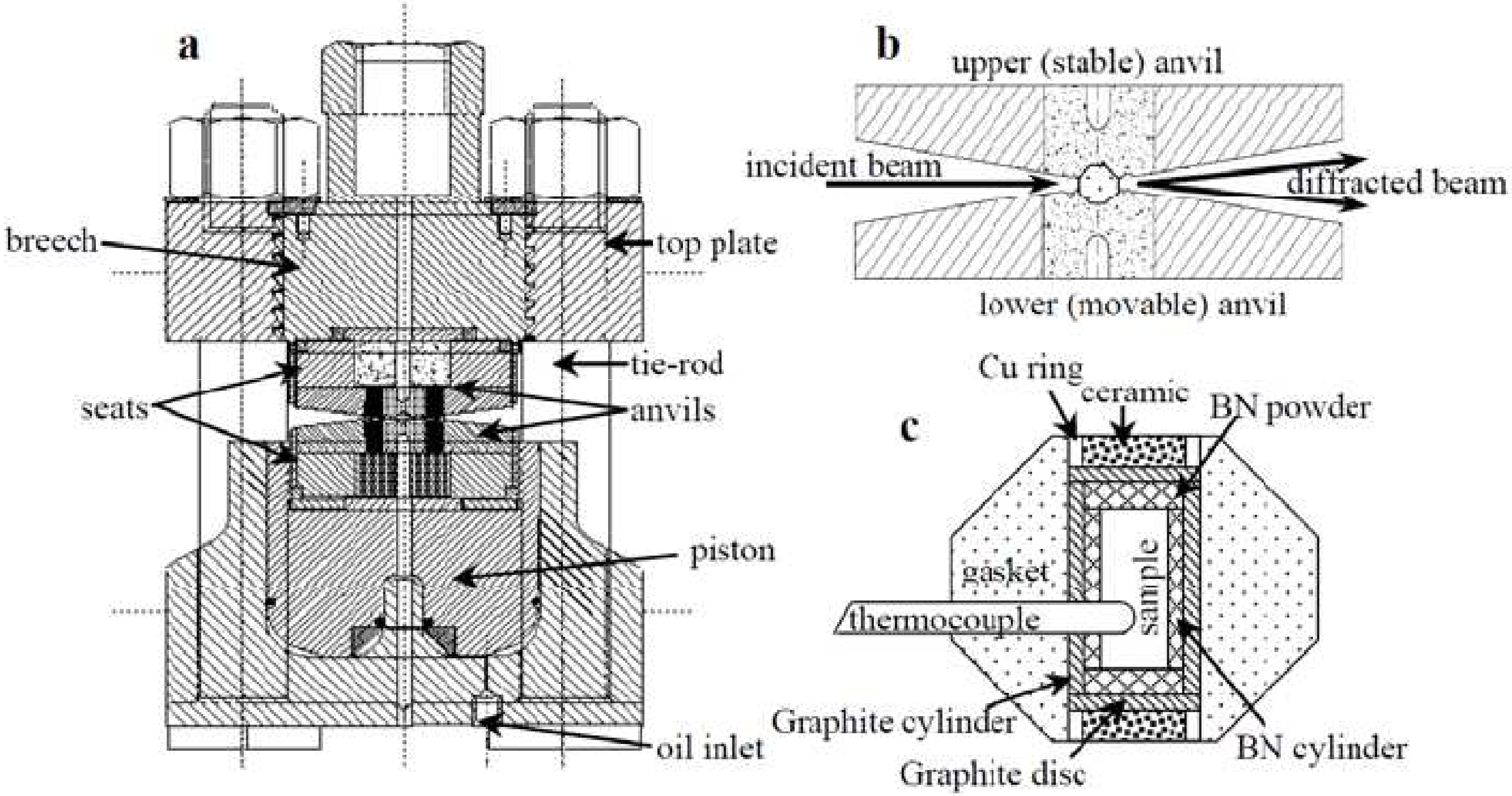


Figure 1: Cross-section of Paris-Edinburgh press (**a**), anvils/sample ensemble (**b**) and sample (**c**).