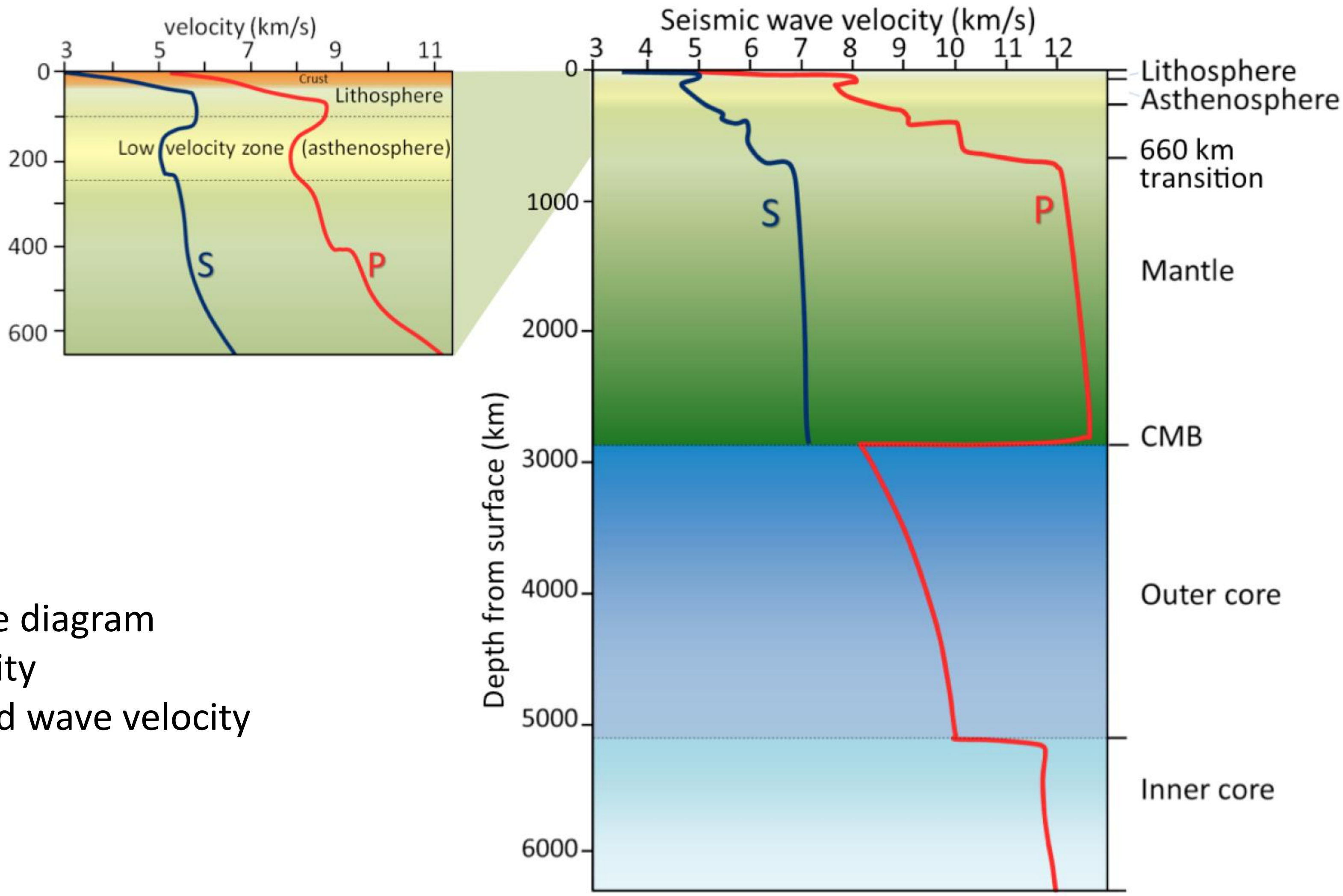
Diagnostics

What do you need to measure?

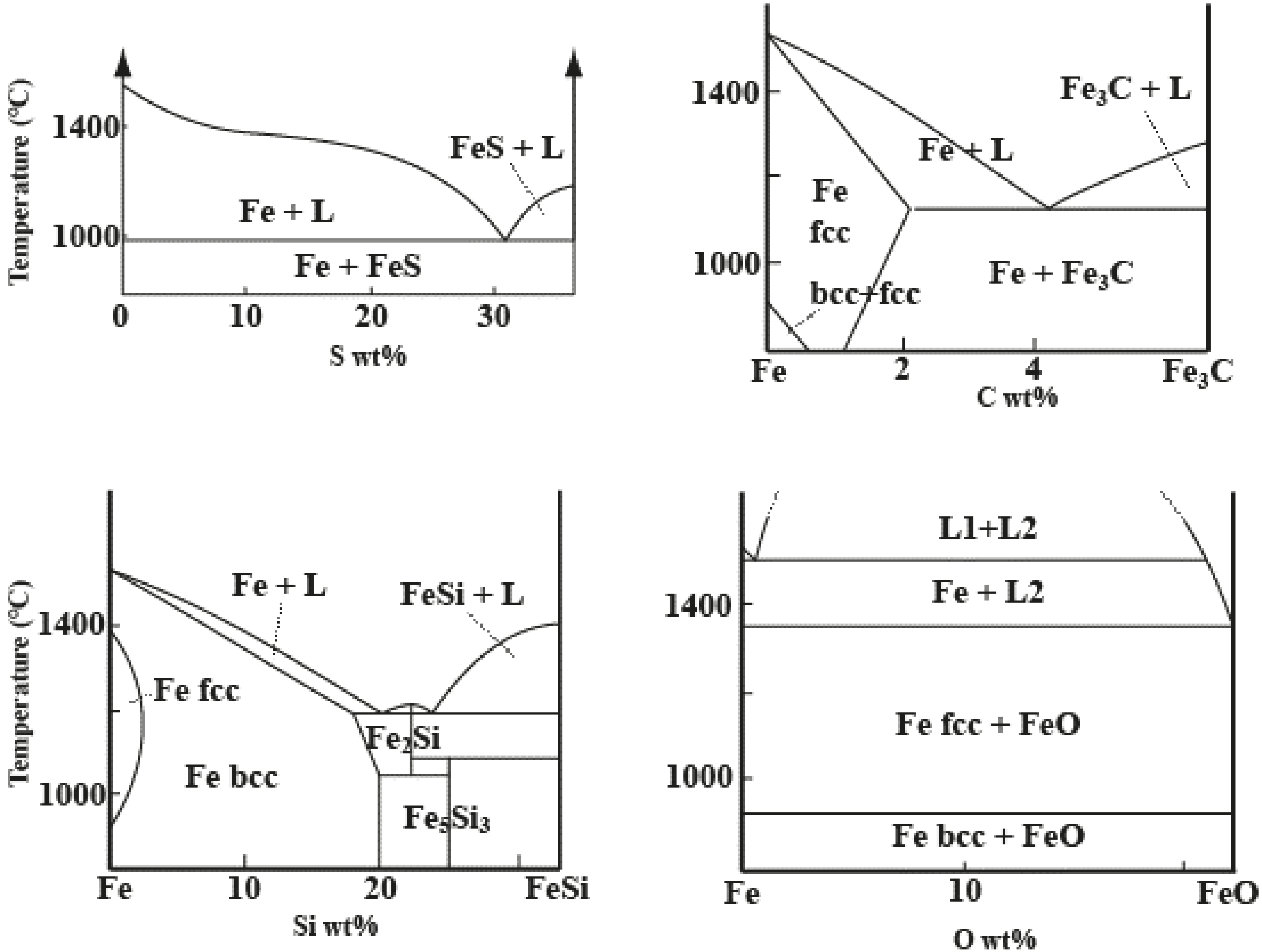


-Phase diagram
-Density
-Sound wave velocity

Now that we compressed materials under HP and HT, what shall we do?

1. Cook and look

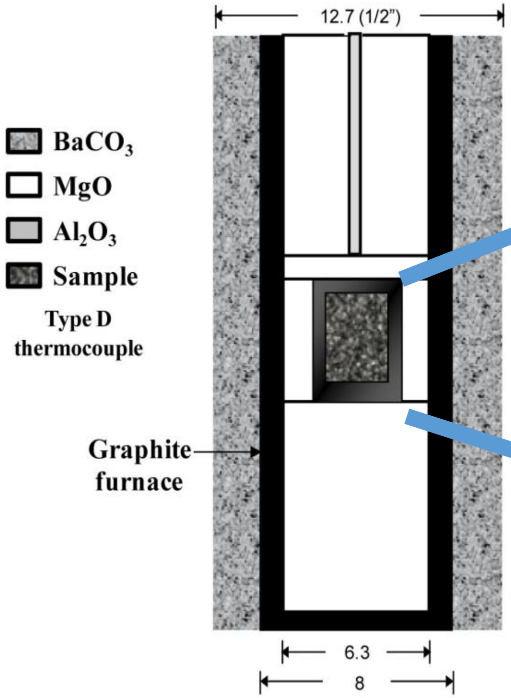
2. *In situ* diagnostics

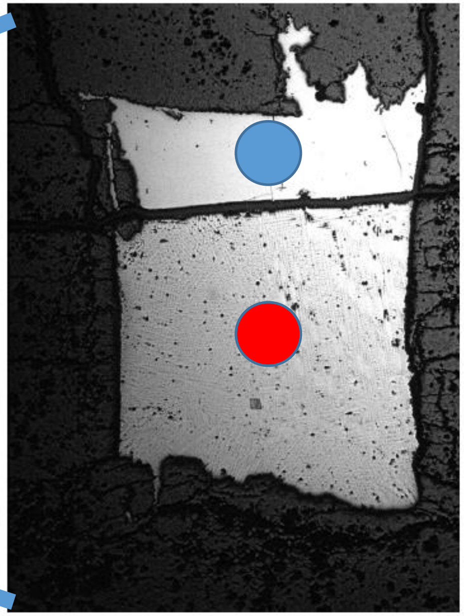


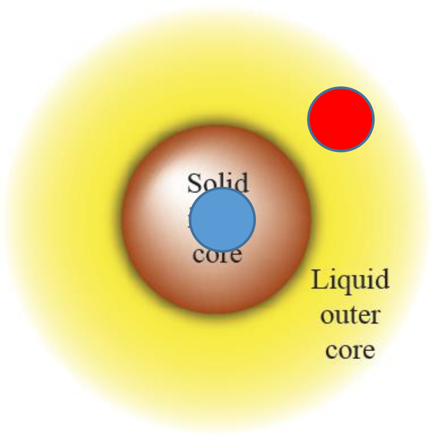
Fe binary alloys at ambient pressure

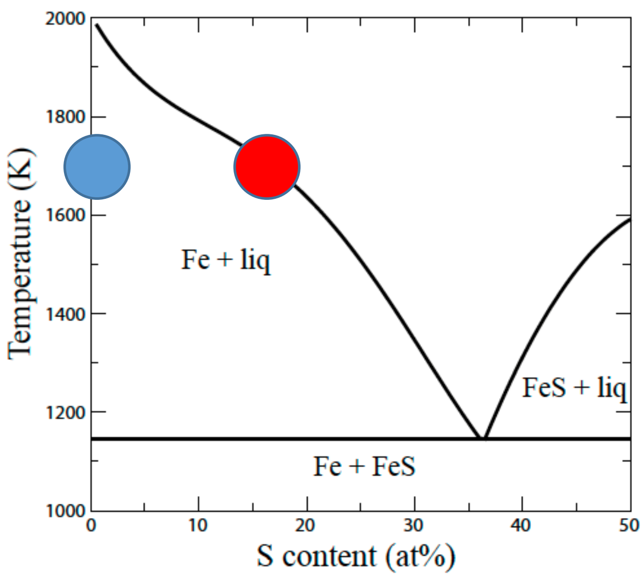
How to reconstruct experimentally a phase diagram?

I give you a machine to compress and heat up samples







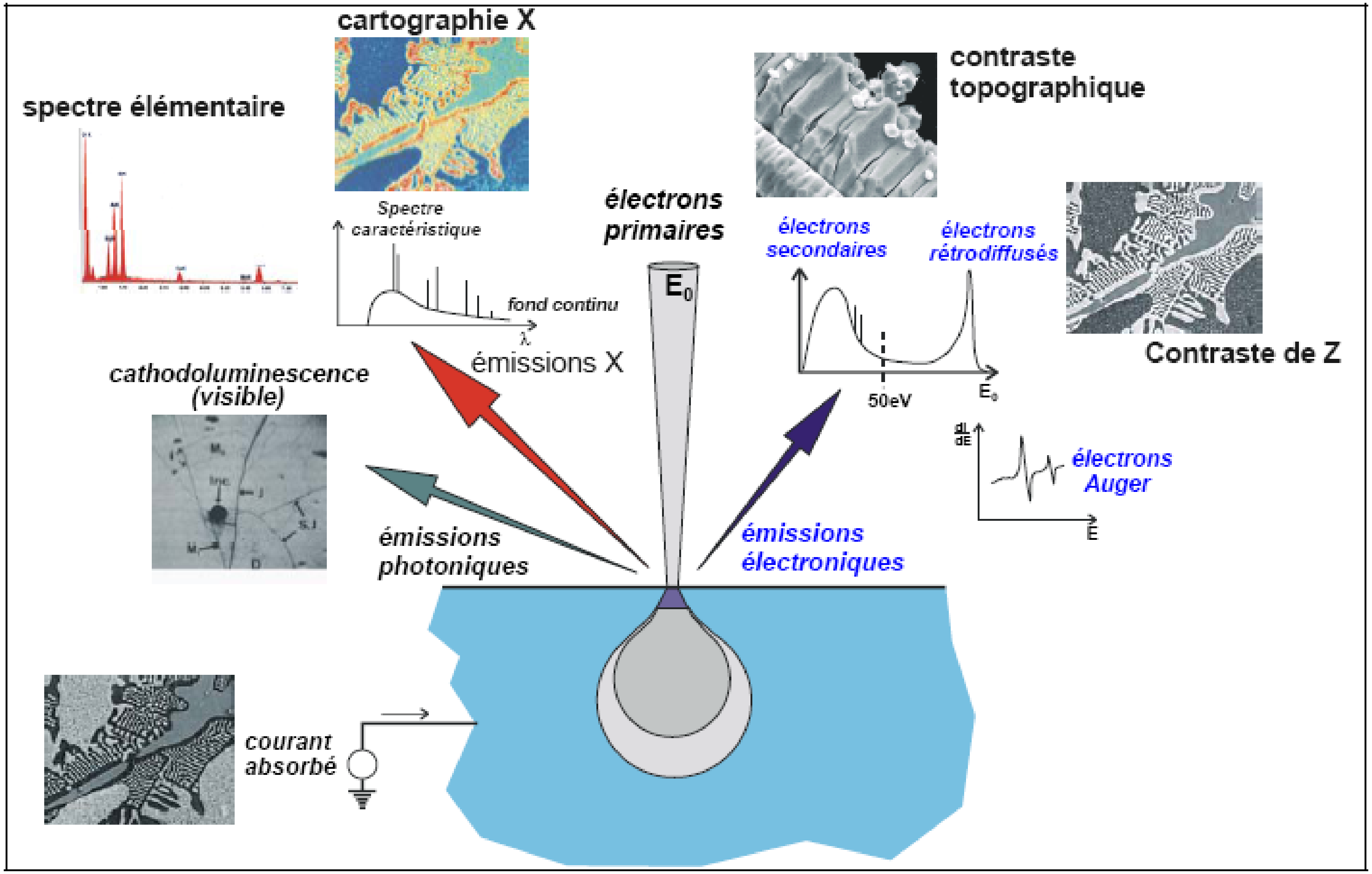


after Buono & Walker, GCA 2011

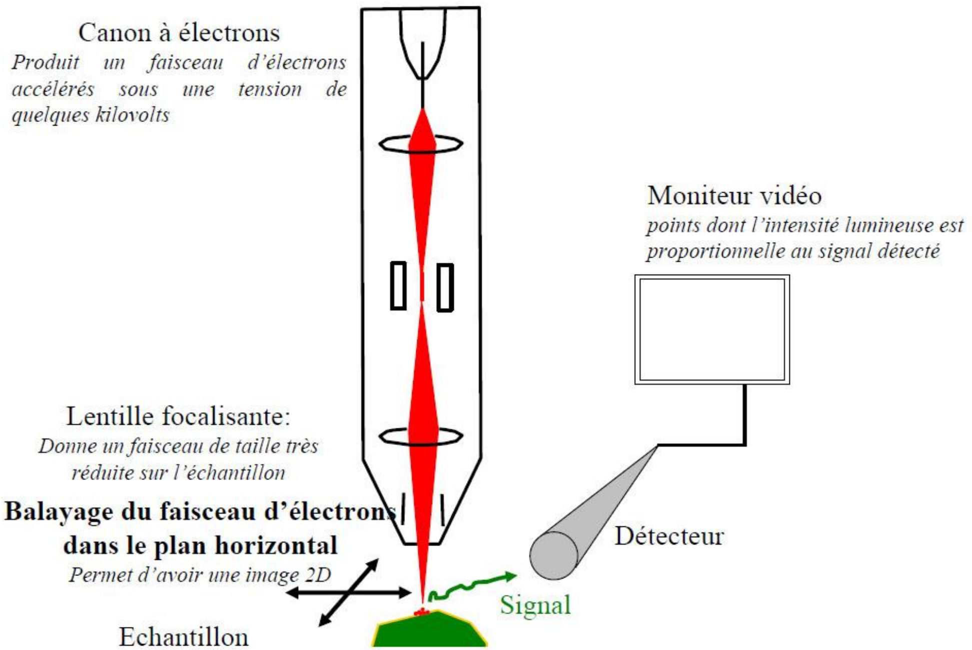
Cook and look

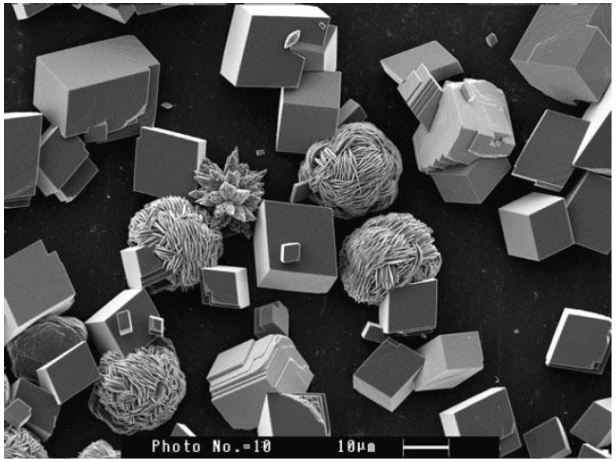
Méthodes analytiques

-Microscopie électronique

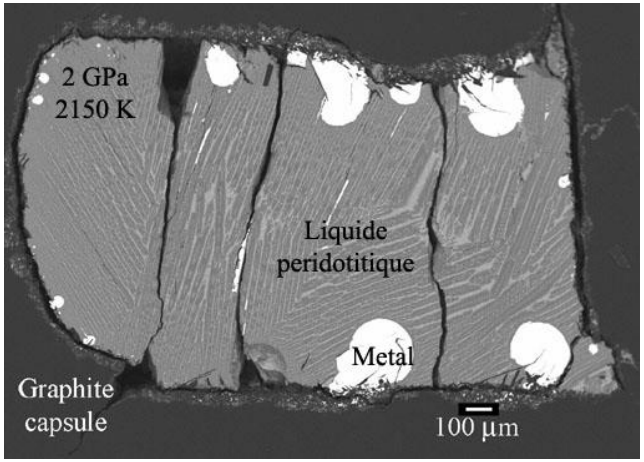


Scanning Electron Microprobe (SEM



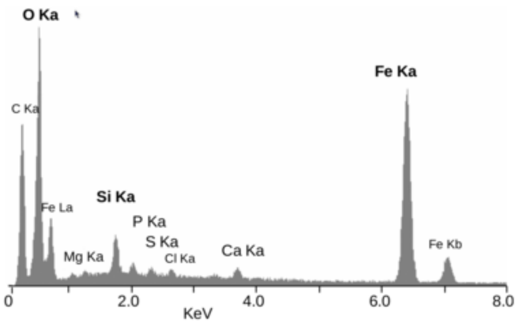


Electrons secondaires

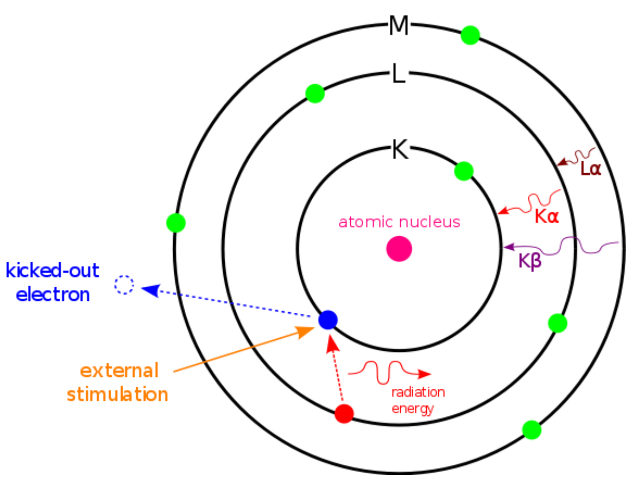


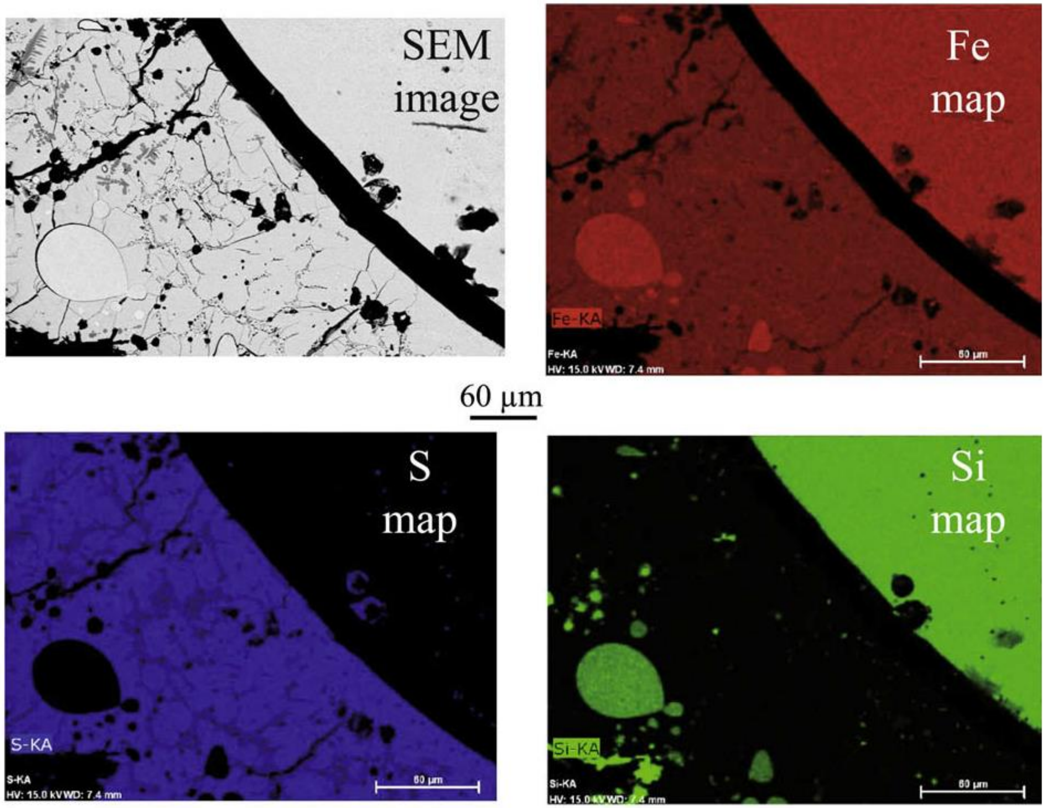
Electrons rétrodiffusés

Electron Microprobe (EPMA)



Spectre EDX (Energy Dispersive)



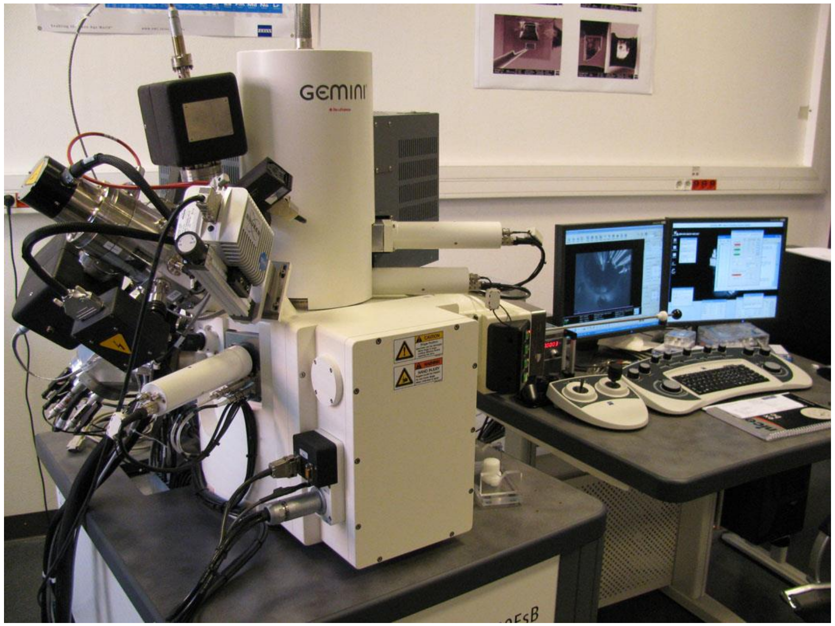


Example of immiscible texture

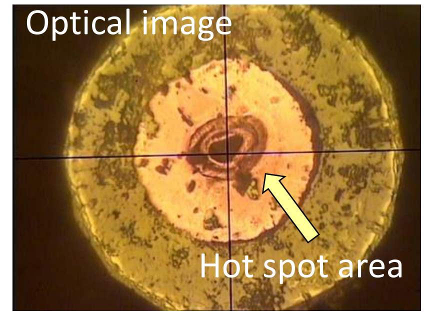
Qualitative and quantitative information

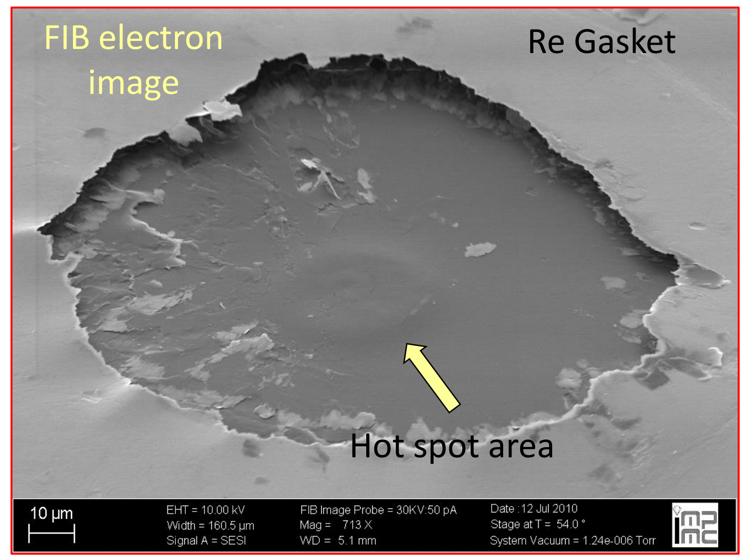
Sample recovery by FIB
(Focused ion Beam)

FIB crossbeam, IMPMC

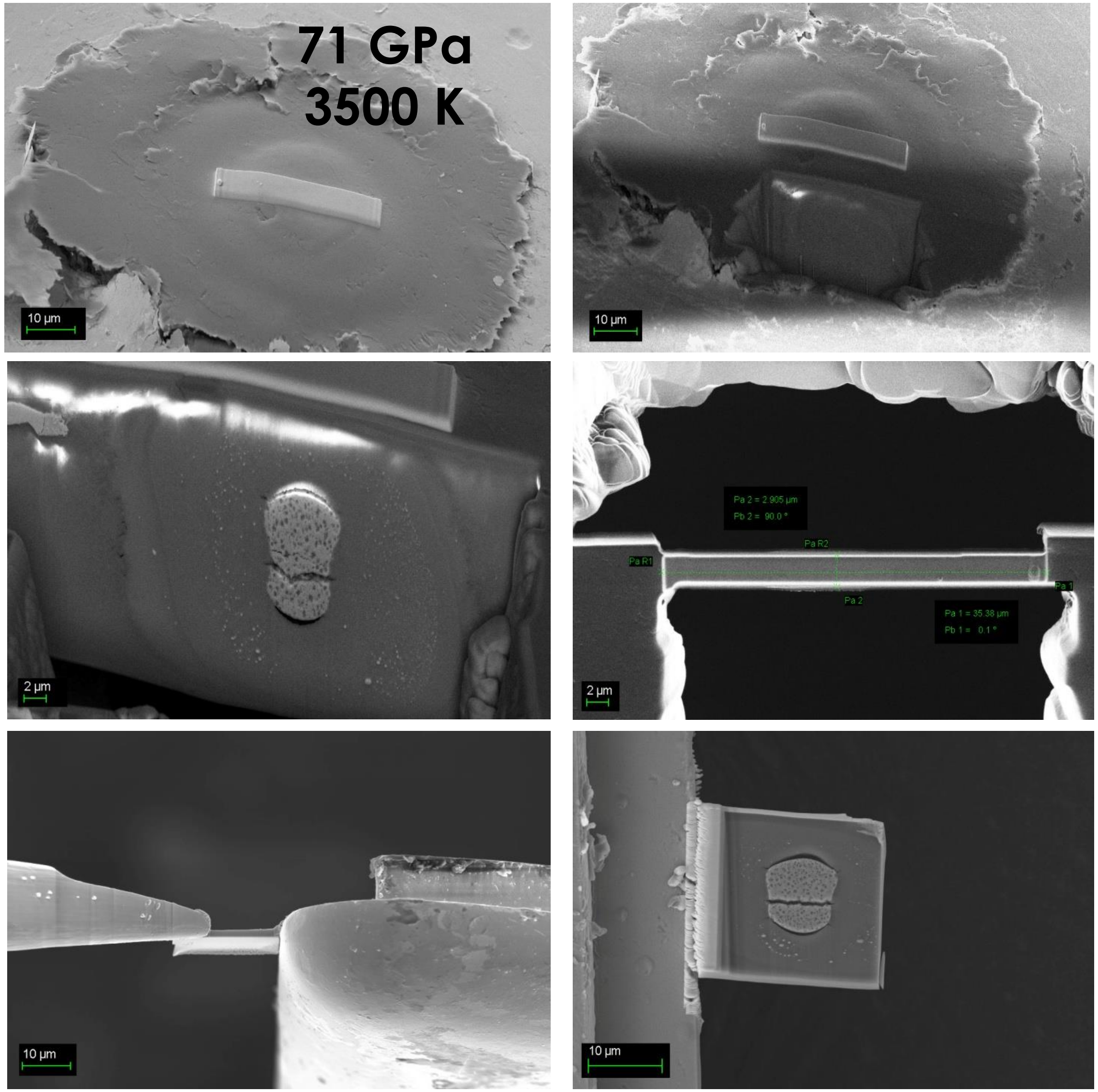


-Milling with Ga ion beam
-Electron image of the sample

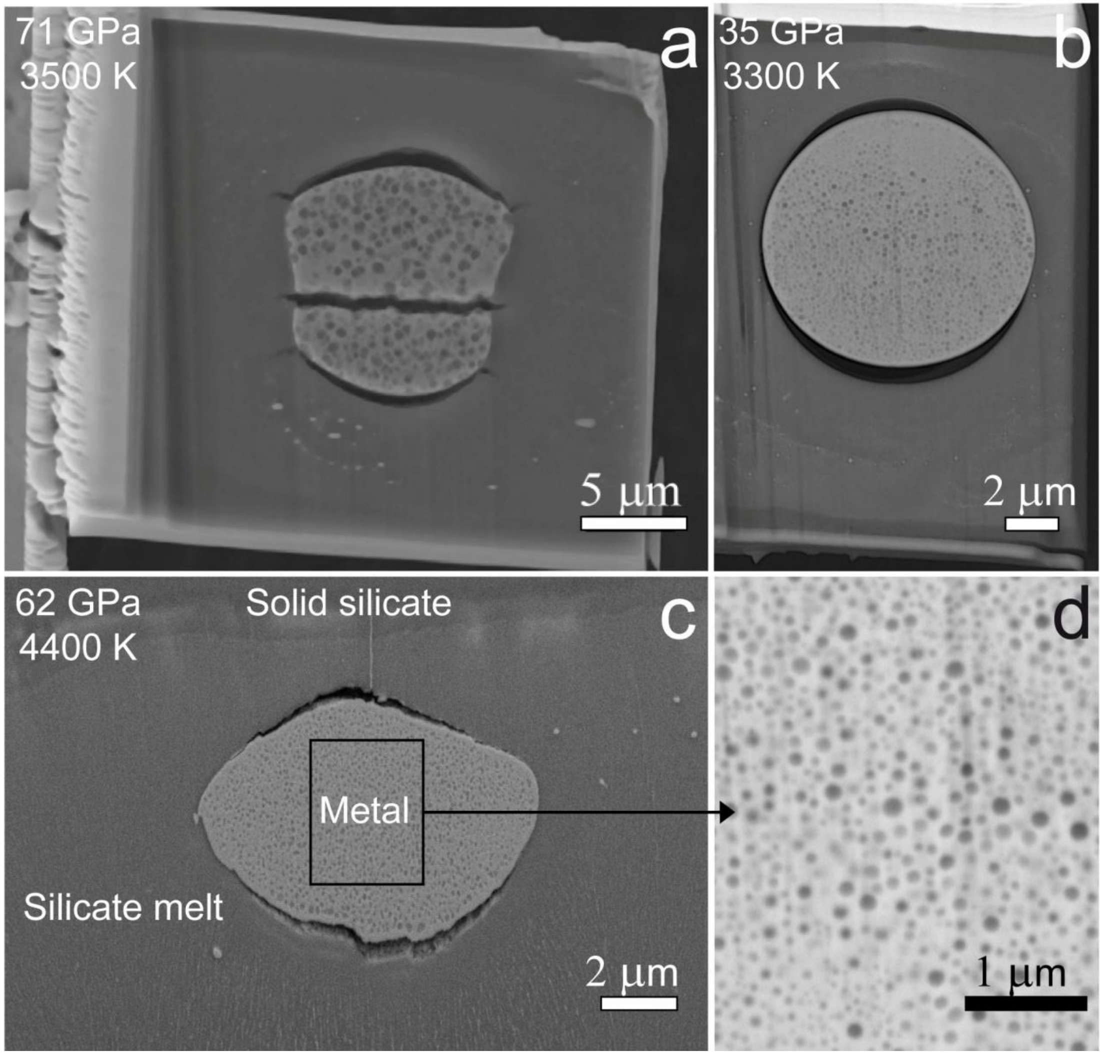




FIB at work


71 GPa
3500 K
10 µm
10 µm
2 µm
Pa 2 = 2.905 µm
Pb 2 = 90.0 °
Pa R2
Pa R1
Pa 1
Pa 2
Pa 1 = 35.38 µm
Pb 1 = 0.1 °
2 µm
10 µm
10 µm

Run products



Courtesy
Julien Siebert, IPGP

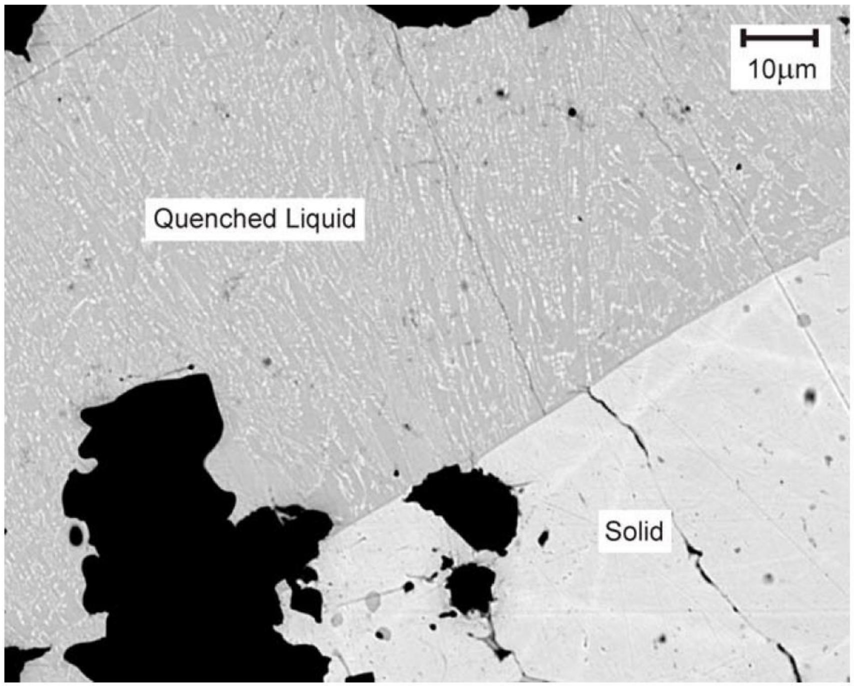
fO_2 -0.7 < IW < -1.5

Metal
- 3.5 wt% < O < 17 wt.%
- 0.3 wt.% < Si < 7 wt.%

EDX (SEM), WDX (EPMA), TEM (EDX, EELS work in progress) analysis

Liquid –solid equilibrium

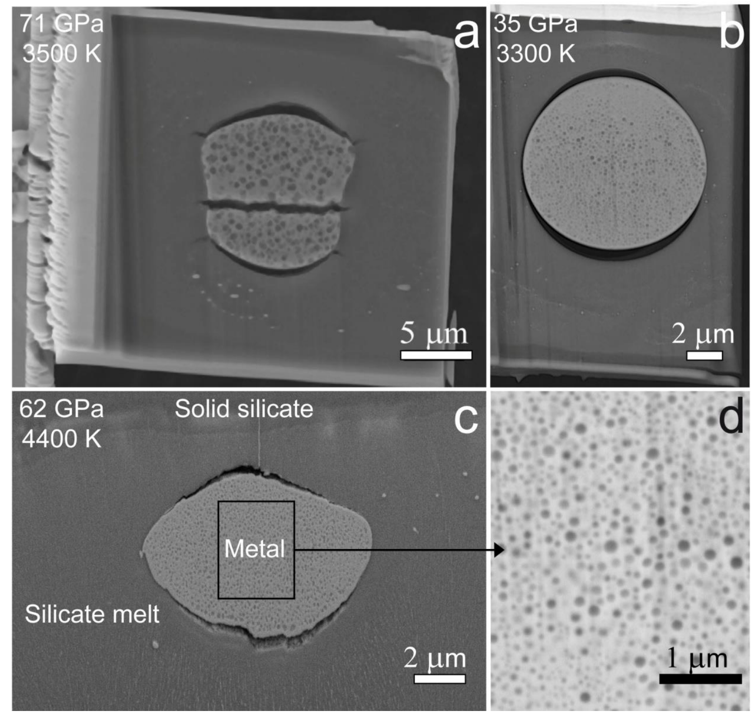
Solid metal-Liquid metal



Phase diagram under high pressure

Liquid-liquid equilibrium

Metal-silicate partitioning

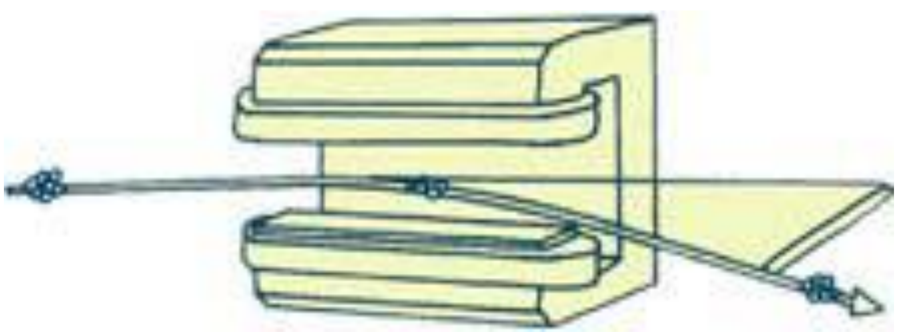


Planetary differentiation, core-mantle equilibrium, etc …

In situ diagnostics

- X-ray diffraction

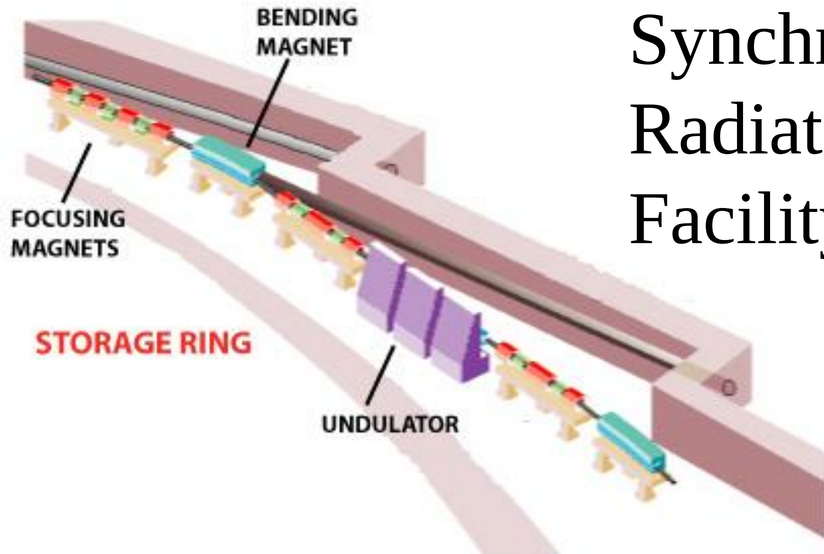
Bremsstrahlung is the electromagnetic radiation produced by the acceleration of a charged particle, such as an electron

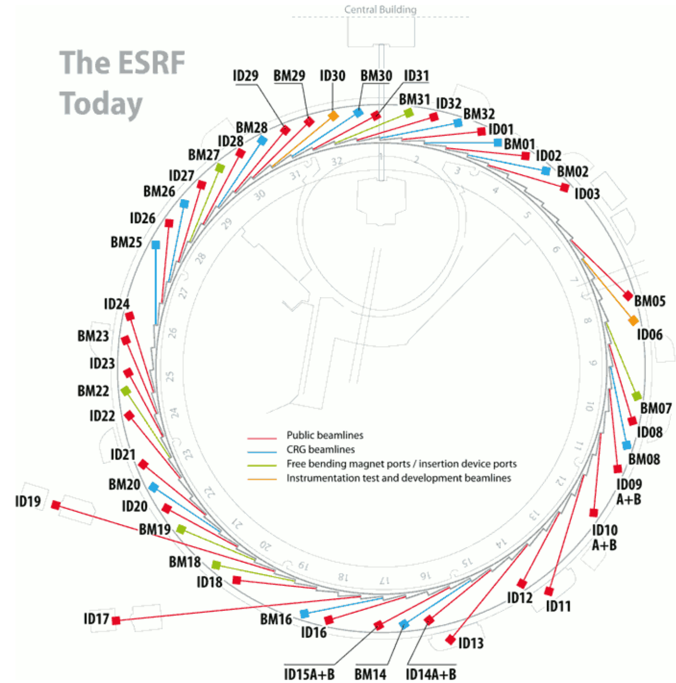


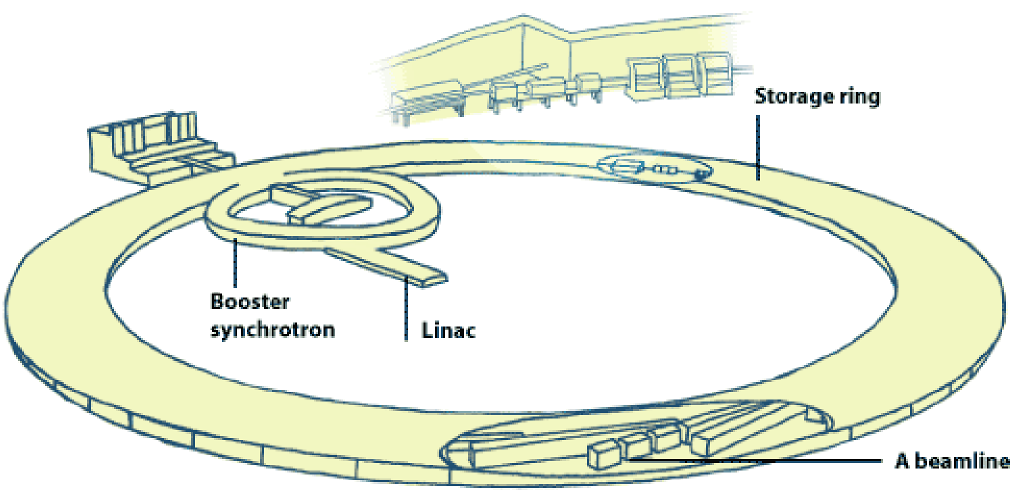
Synchrotron radiation

Hard X-ray

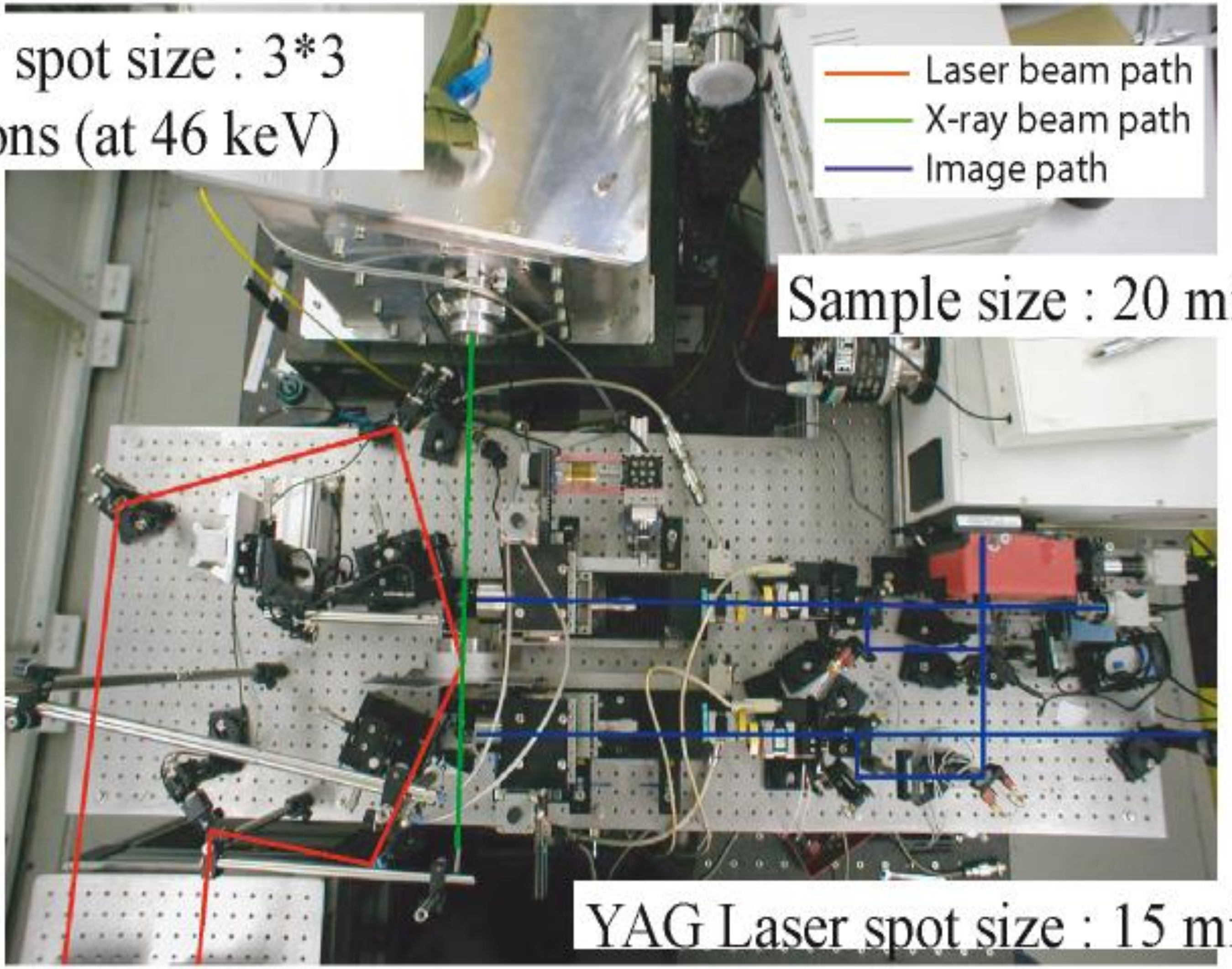
European Synchrotron Radiation Facility (ESRF)





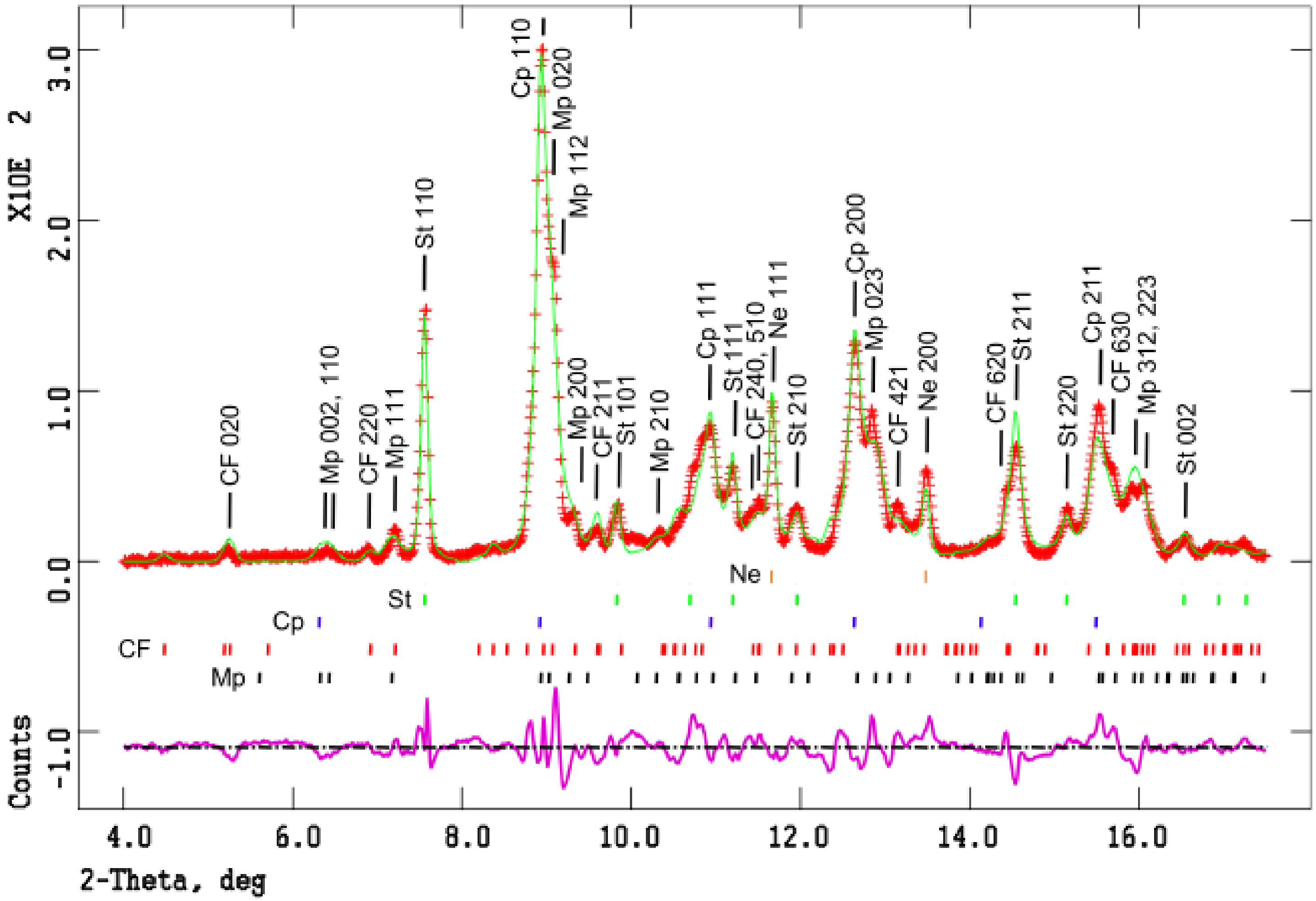


Double Sided Laser Heating DAC Experimental Set up



Exposure
time : 20 s

Diffraction pattern

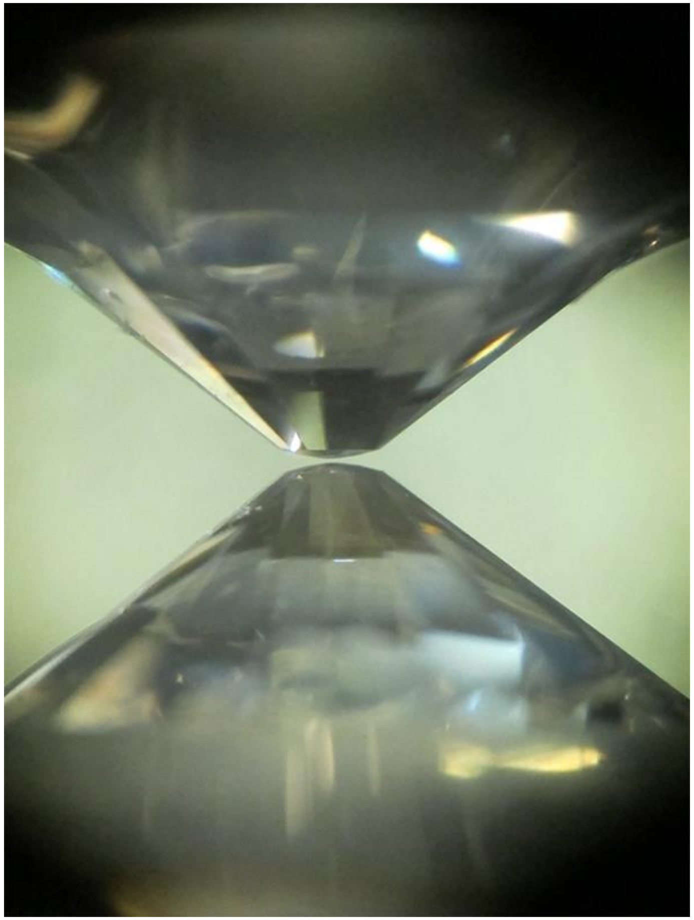


Equation of state

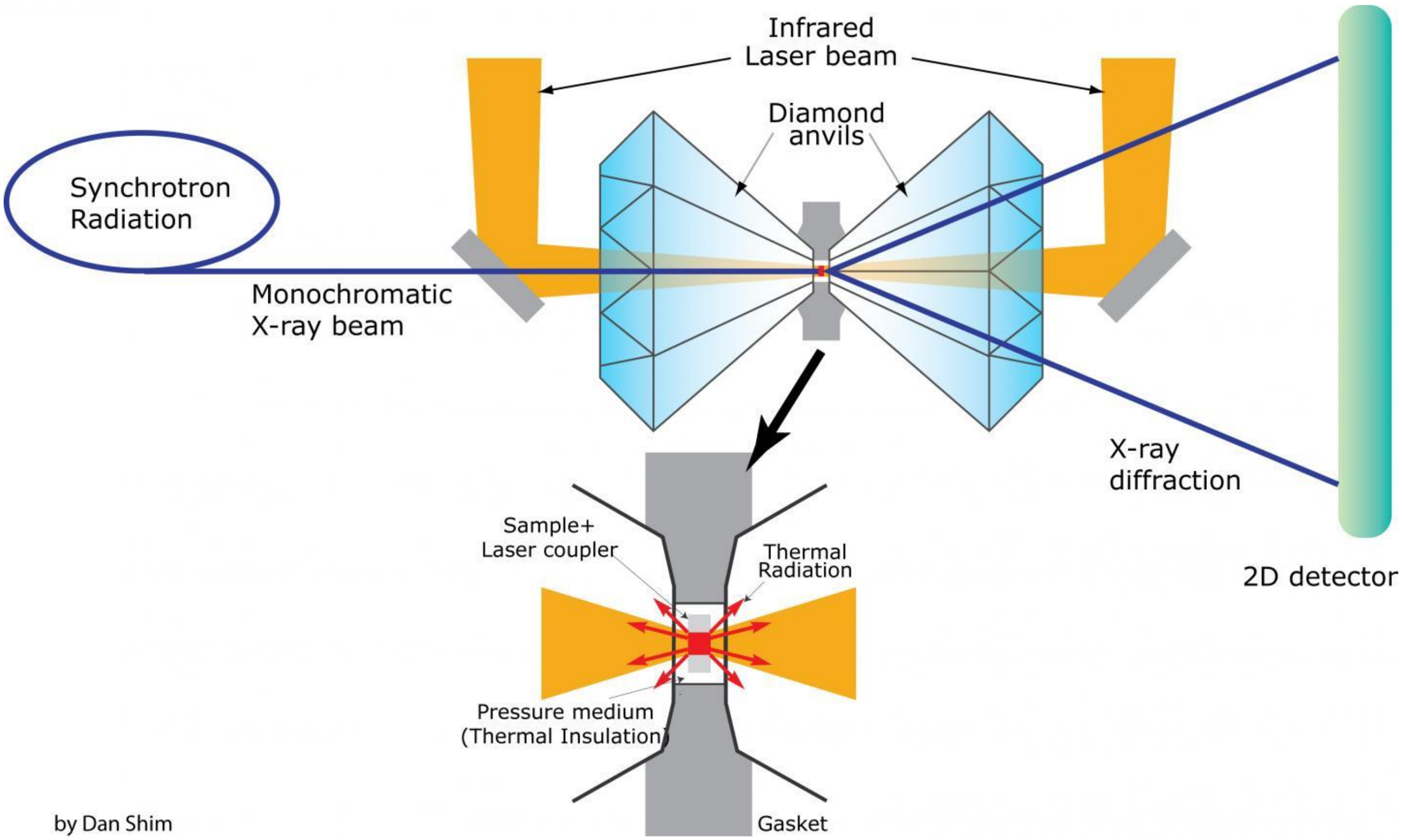
$$f(P, V, T) = 0$$

Formalism

- PV = nRT
- EoS 300 K
  - Birch Murnaghan
  - Vinet
- High Temperature
  - Mie Gruneisen

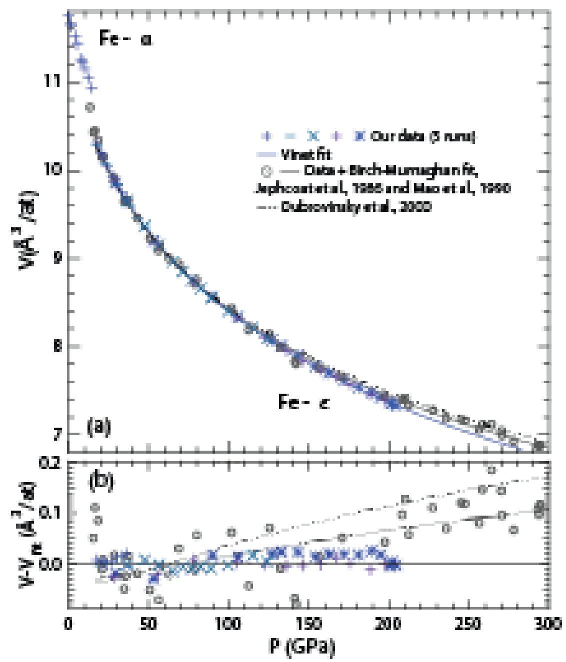


X-ray diffraction under high pressure and high temperature



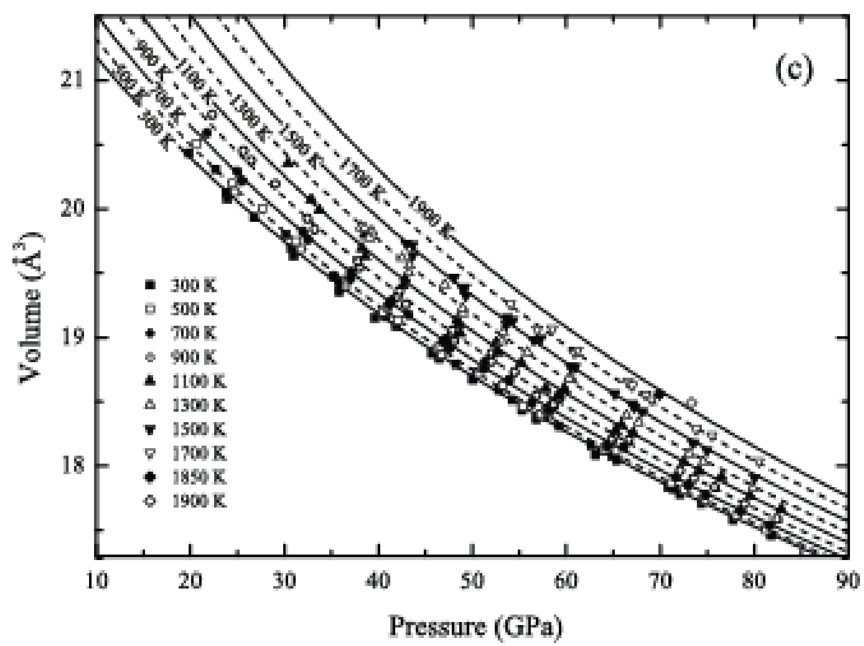
Equation of state

Compression at 300 K



After Dewaele et al, PRL, 2006

Increasing pressure AND temperature



From Yamazaki et al, GRL, 2012

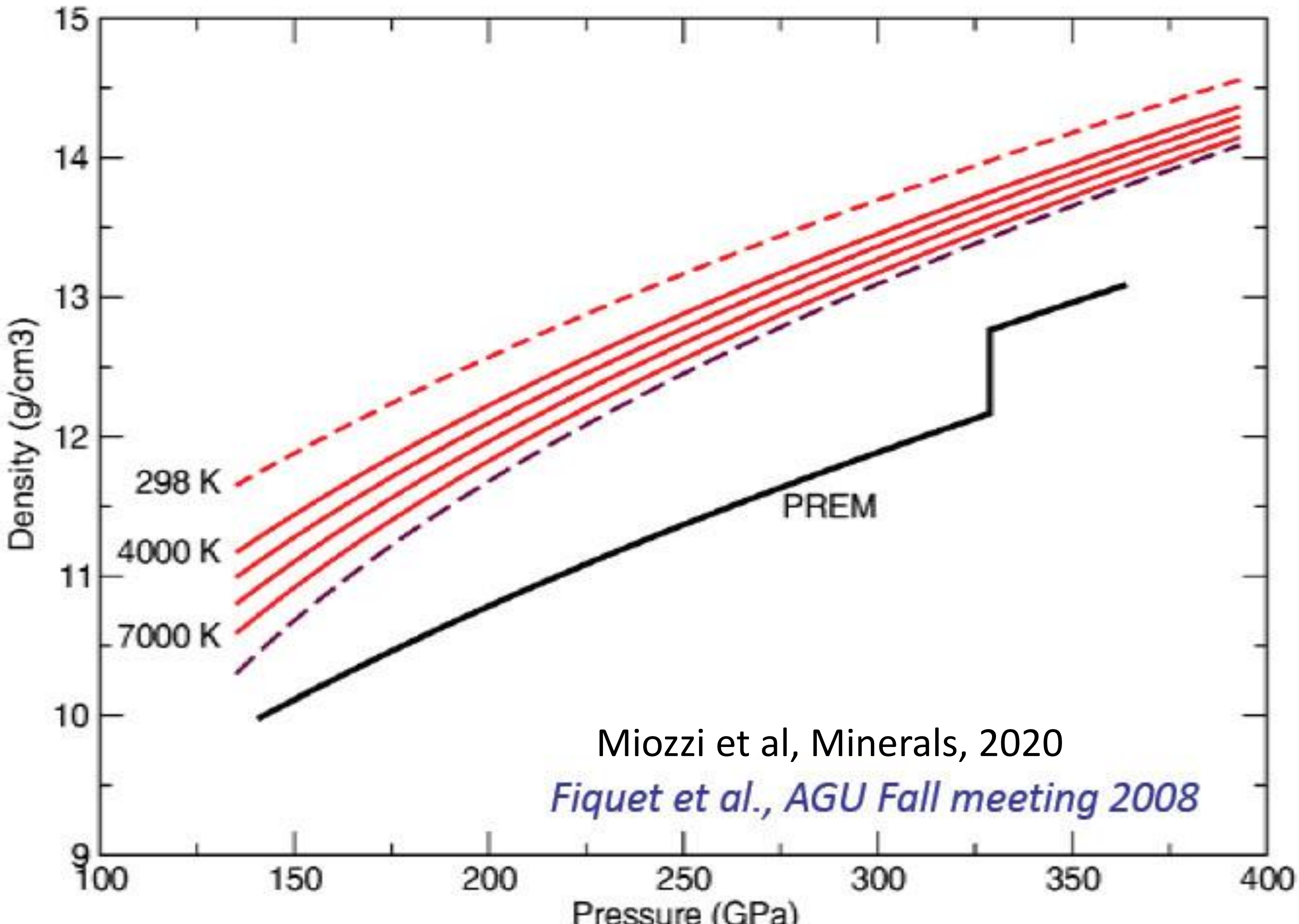
3rd order Birch-Murnaghan Equation of state

$$P_{T0} = \frac{3}{2}K_{T0}\left[\left(\frac{V}{V_0}\right)^{-7/3} - \left(\frac{V}{V_0}\right)^{-5/3}\right]\left\{1+\frac{3}{4}(K'_{T0}-4)\cdot\left[\left(\frac{V}{V_0}\right)^{-2/3}-1\right]\right\}$$

Thermal pressure

$$P_{th}(V,T) = \frac{9R\gamma}{V}\left[\frac{\theta}{8} + T\left(\frac{T}{\theta}\right)^3\int_0^{\theta/T}\frac{z^3}{e^z-1}dz\right] + \frac{3R}{2V}ma_0\left(\frac{V}{V_0}\right)^m T^2 + \frac{3R}{2V}ge_0\left(\frac{V}{V_0}\right)^g T^2 \quad (4)$$

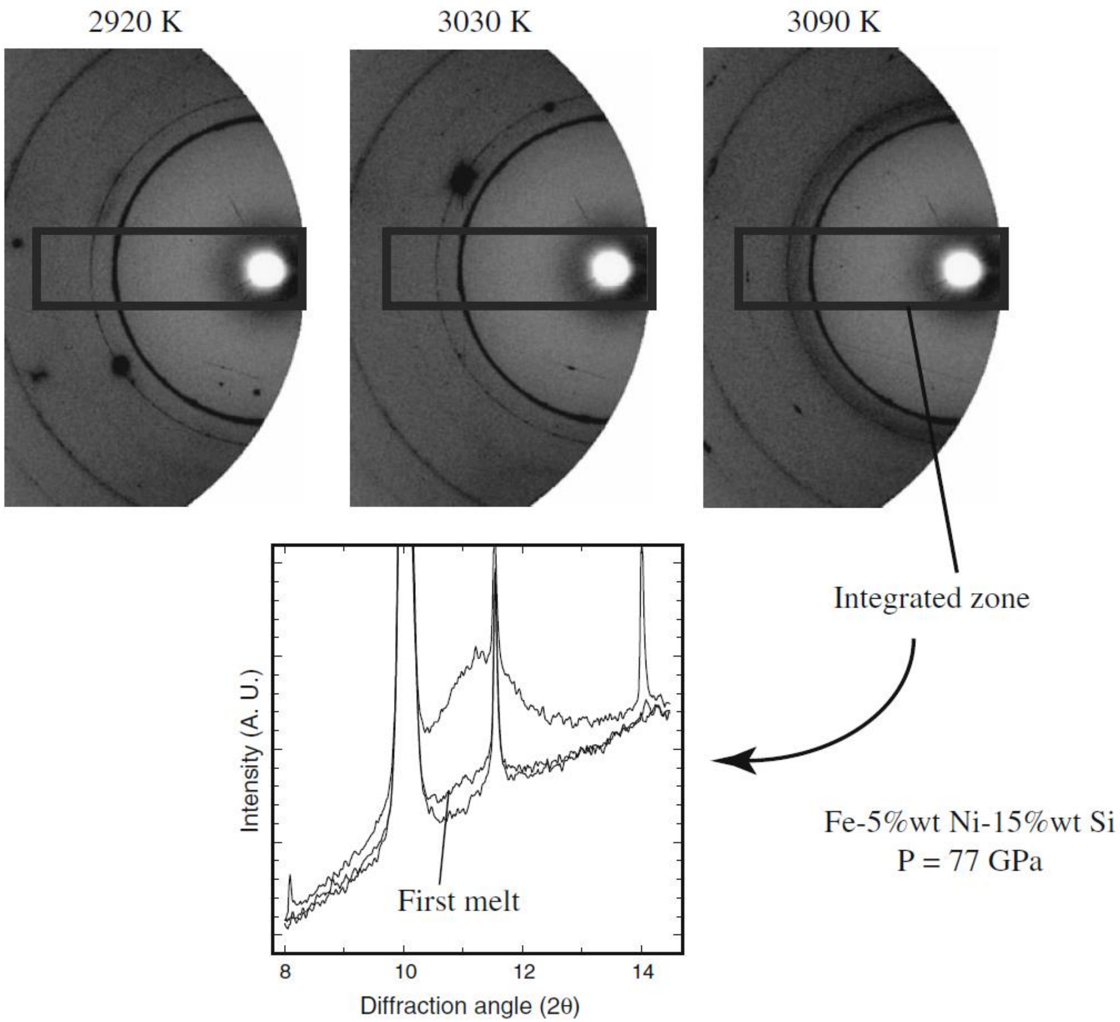
Model required to extrapolate P-T conditions



Miozzi et al, Minerals, 2020

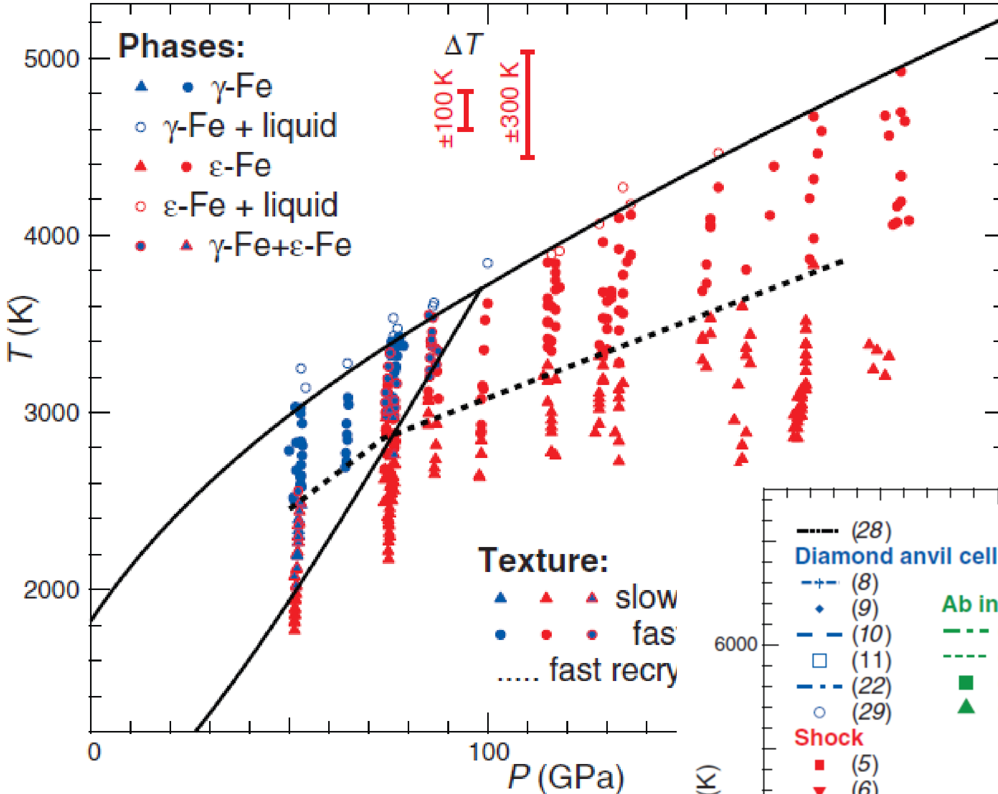
Fiquet et al., AGU Fall meeting 2008

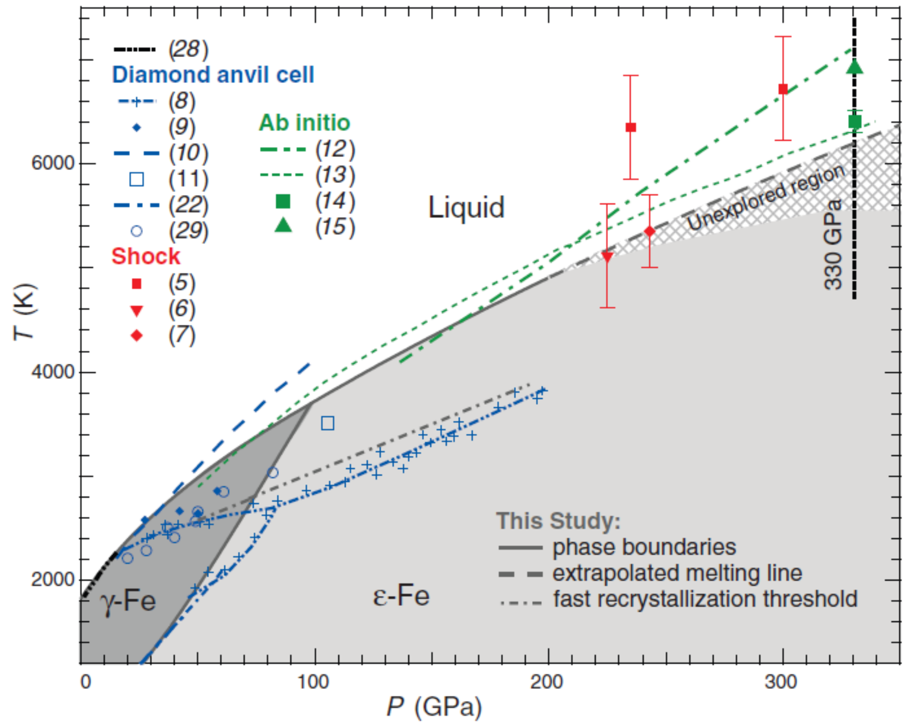
Melting temperature measurements



Contrary to solid, liquid have diffuse scattering rings

By integrating the image plate, we can have access to the melting temperature



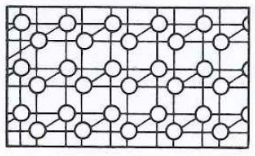
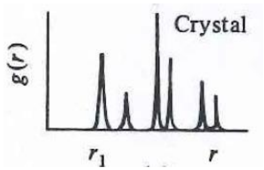


Iron phase diagram at the pressure of the Earth's core

Anzellini et al, Science, 2013

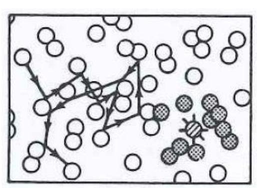
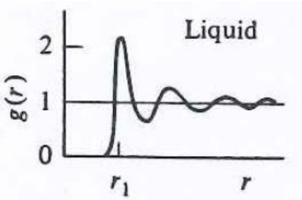
Liquids

- Crystalline solids:
- periodic structure

- Fixed atomic positions
- **Short range order**
- Long range order

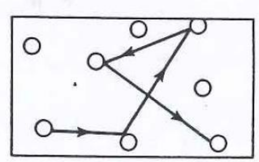
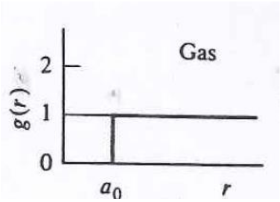
Liquids and amorphous solids:
continuous distribution of distances

- **Short range order**
- Fluctuations in the near-neighbor region: **absence of long range order**

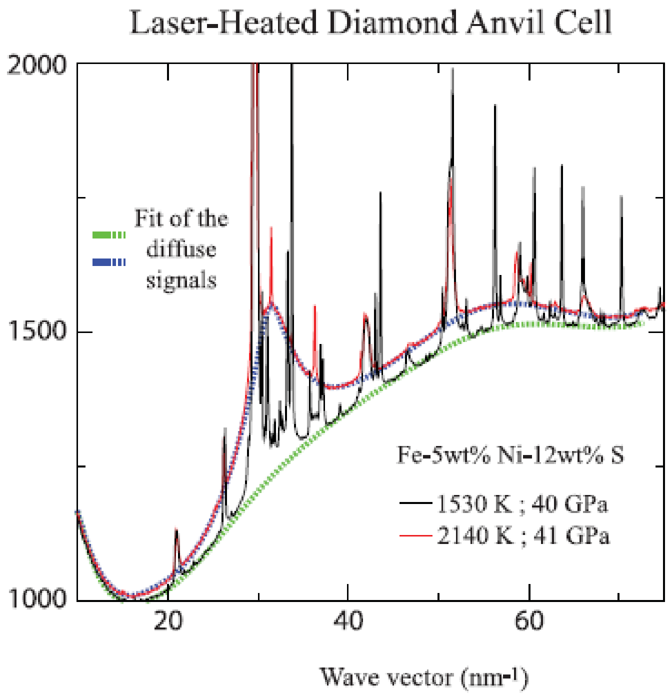
A liquid is described by a radial distribution function: g(r)

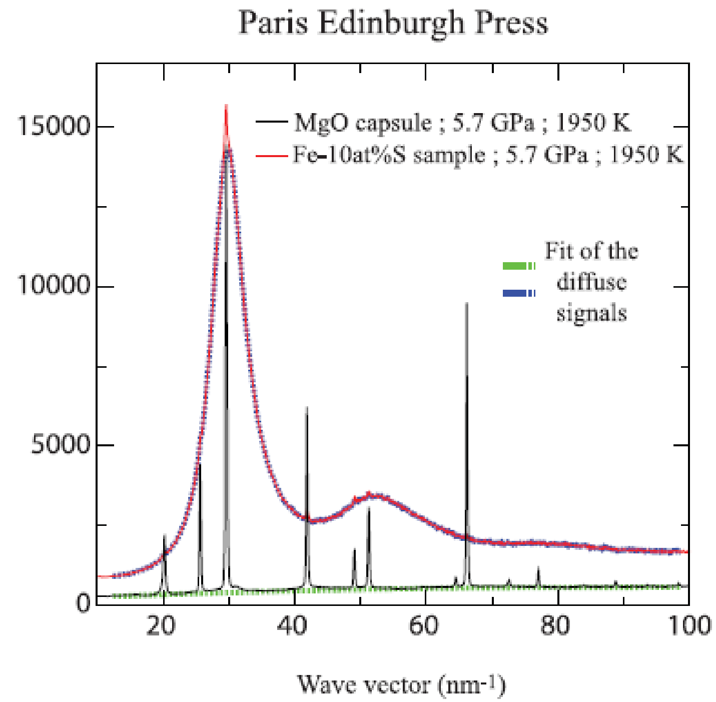
Gases: no structure

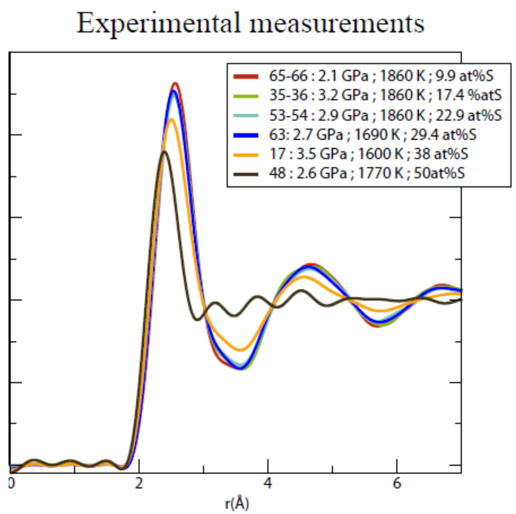
[Waseda 1980]

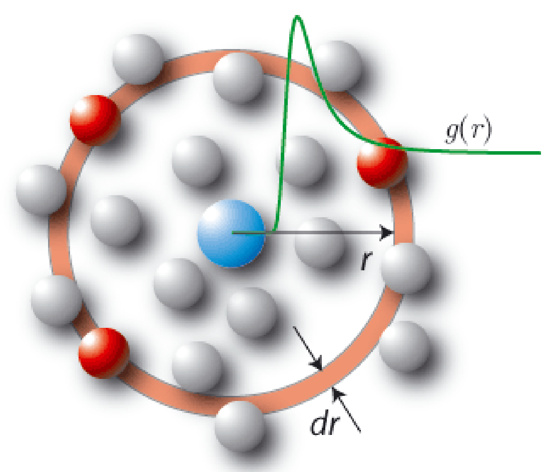
- Random atomic positions:
- absence of short range order
- **absence of long range order**





Density and structure of liquid phases as a function of pressure and temperature

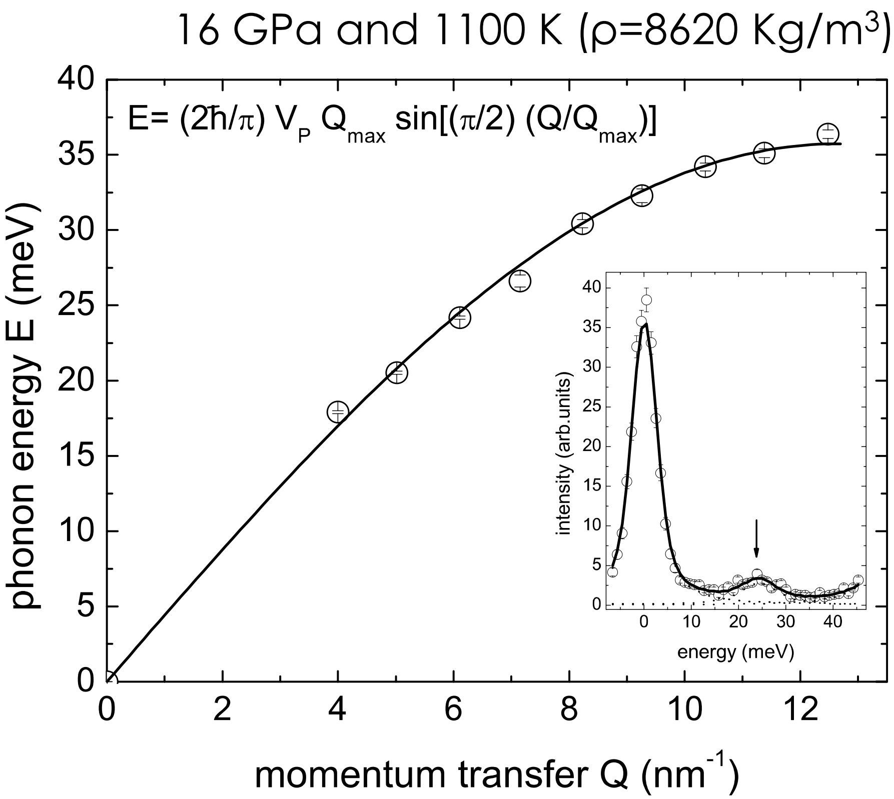


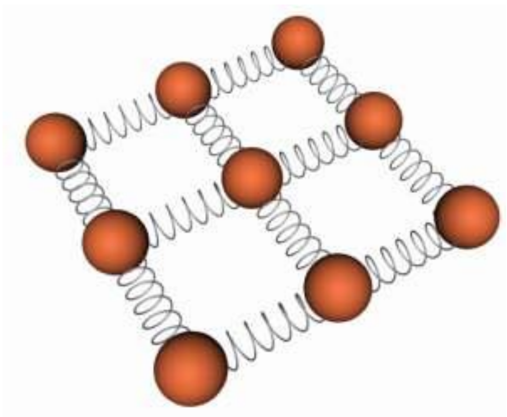


In situ diagnostics

- X-ray diffraction : density and phase diagram
- Sound velocity measurements

Phonon dispersions and sound velocities



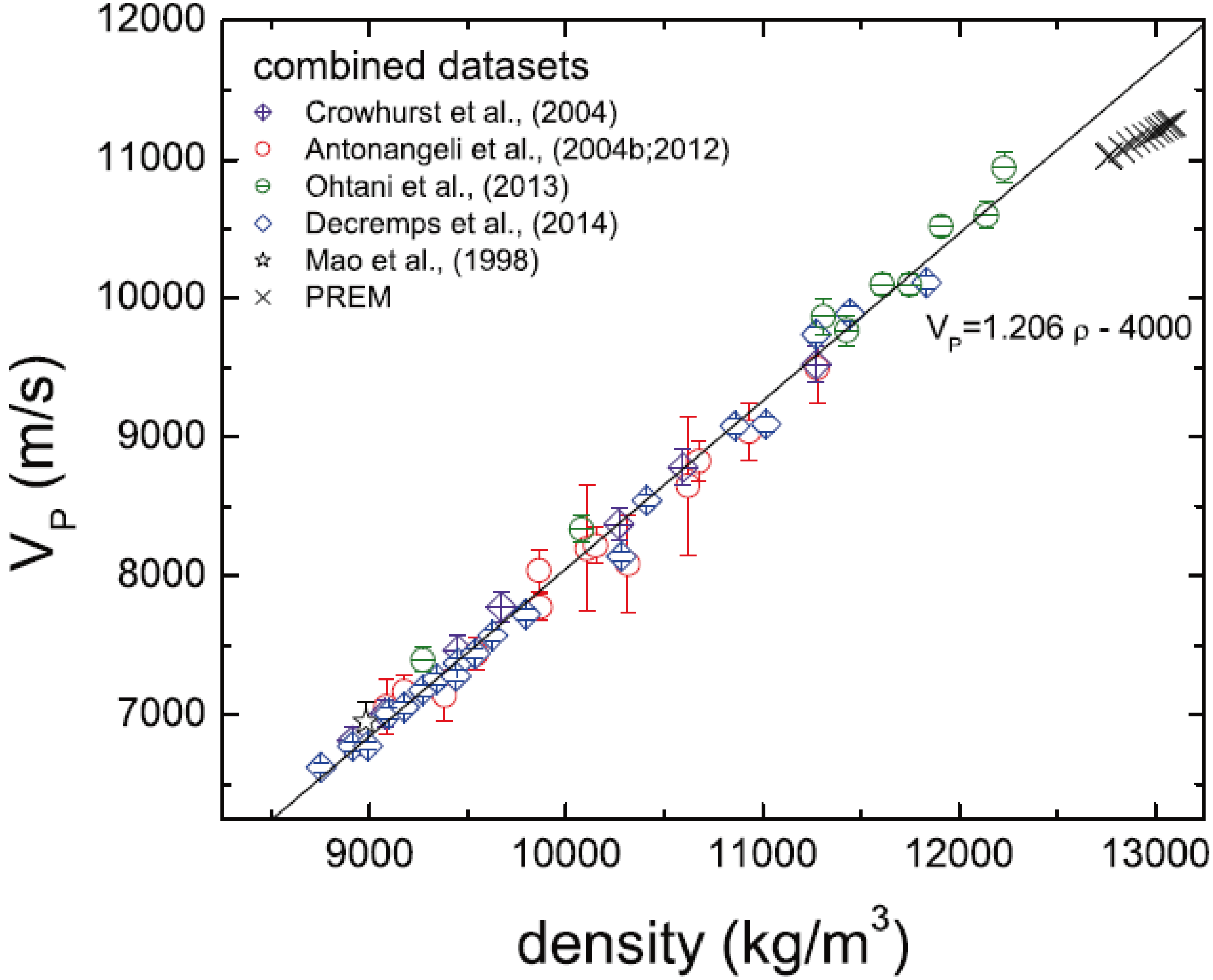


Elastic interaction :
X-ray diffraction (XRD)
→ compression curve
→ K

Inelastic interaction :
Inelastic X-ray scattering (IXS)
→ phonon dispersion (vibrations)
→ V_P

IXS+XRD → V_S

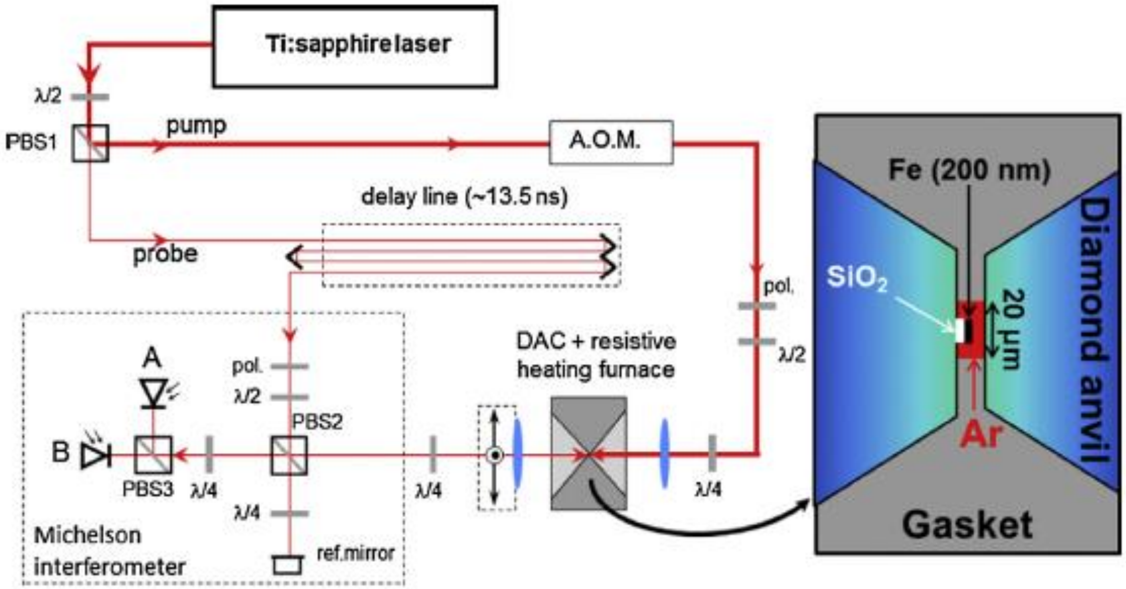
Close up on the inner core: very close to pure iron

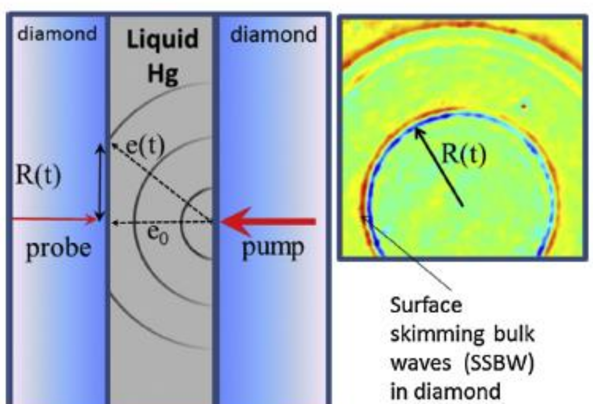


Antonangeli and Ohtani, 2015

Other methods

Picosecond acoustic: requires precise thickness of the sample





Decremps et al, Ultrasonics, 2015