Laser heating in the diamond anvil cell: The «basics»

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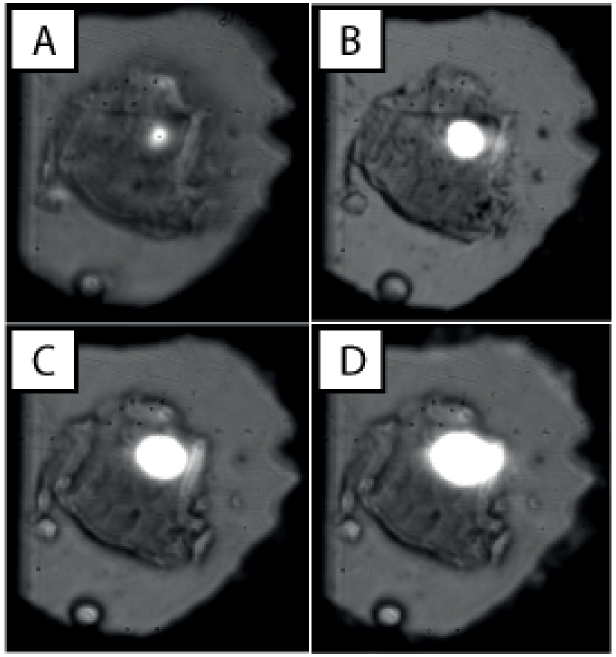


Photo: Pierru et al.

First scientific articles based on LH-DAC (WoS):

In canberra, Australia: Lin-Gun JIU (1974) discovery of the (Mg,Fe)SiO3 brigmanite, the major mineral on Earth. He then wrote ~10 other major articles based on LH-DAC.

In Washington-DC: By: T. YAGI, H.K. MAO, P.M. BELL (1978) Bridgmanite

In Hawaï (?), USA: L.C. MING and M.H. MANGHNANA (1979) Phase transition in MgF2

In Paris: A. LACAM, M. MADON, J.P. POIRIER (1980) Upper-Lower mantle discontinuity on Earth

First article using X-ray diffraction coupled with LH-DAC (WoS):

In canberra, Australia: Lin-Gun JIU (1974) discovery of the (Mg,Fe)SiO3 brigmanite.

First articles using synchrotron radiation coupled with LH-DAC (WoS):

In Washington-DC: Y. KUDOH, C.T. PREWITT, L.W. FINGER (1990). Bridgmanite EoS

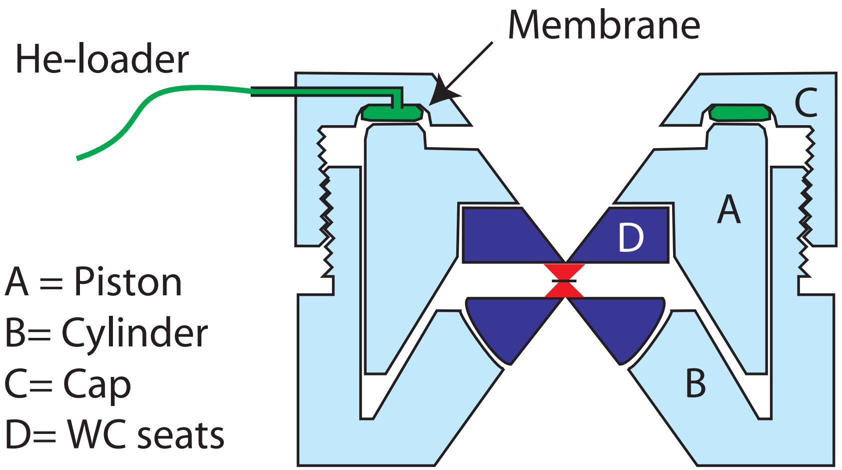
In Mainz, Germany: R. BOEHLER, N. VONBARGEN, A. CHOPELAS (1990). Melting of Fe

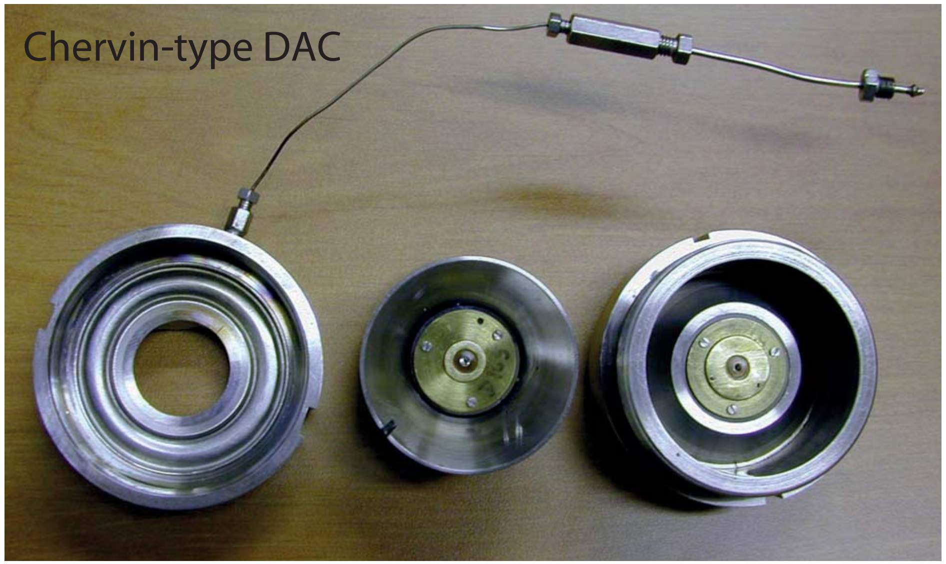
First article using ESRF and LH-DAC (WoS):

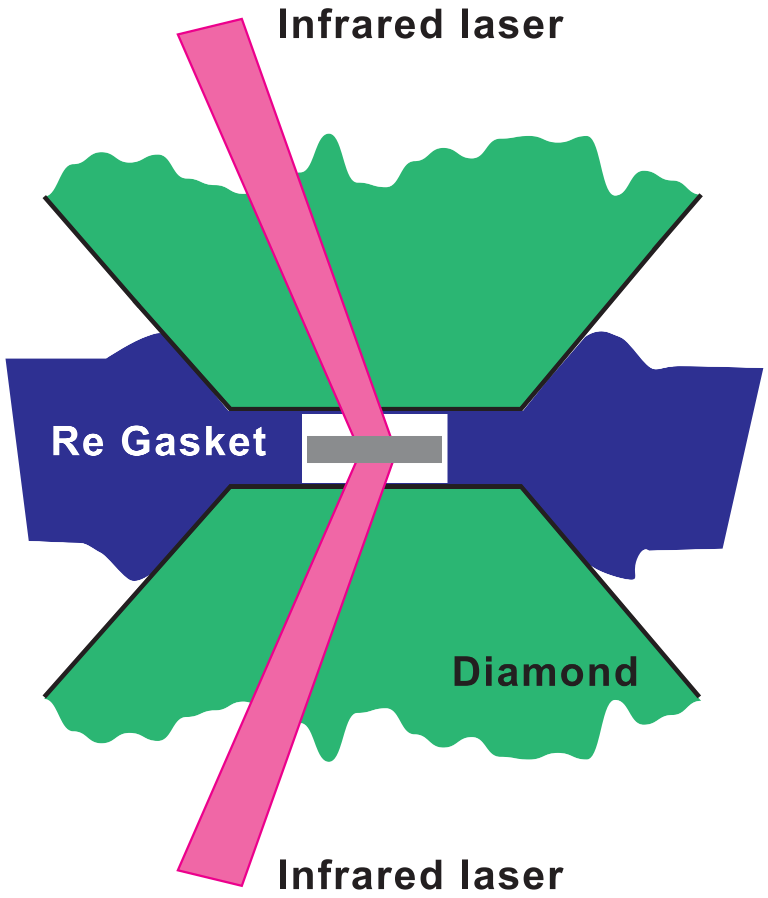
S.K. Saxena, L.S. Dubrovinsky, P. Lazor et al. (1996) Breakdown of perovskite (MgSiO3) in the Earth's mantle

G. Fiquet, D. Andrault, A. Dewaele et al. (1998) P-V-T equation of state of MgSiO3 perovskite

At the first glance, one could think that LH-DAC techniques did not evolved much in recent years



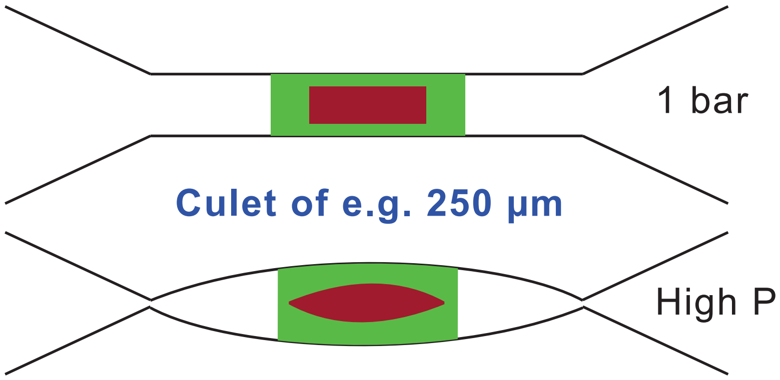




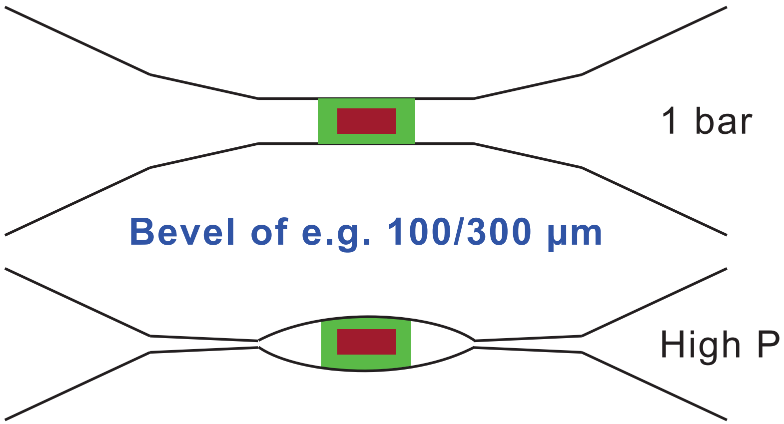
Calibrant => Pressure
Radiation => Temperature

In fact, there is a continual, and critical, evolution in the control of experimental conditions for a large part thanks to the use of synchrotron radiation

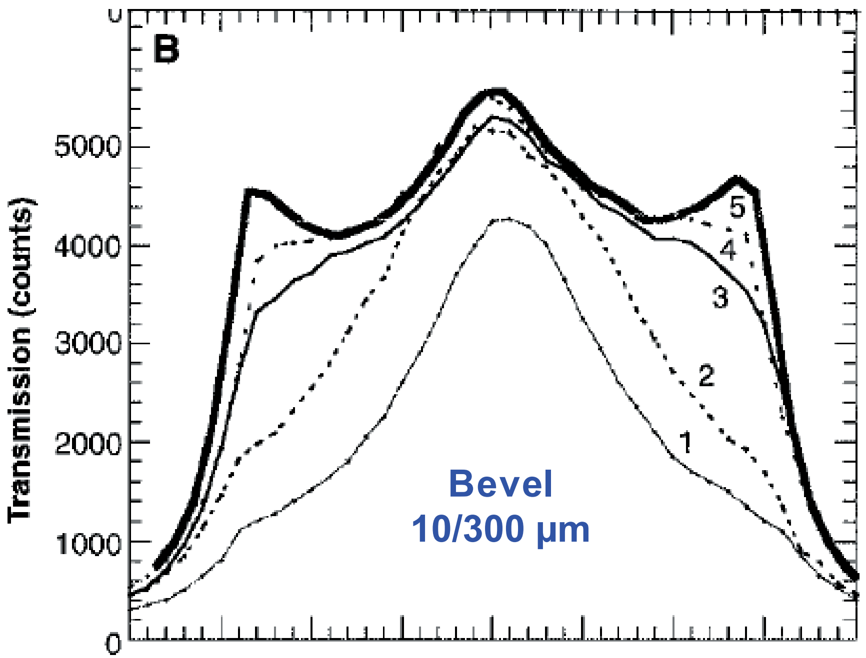
What is the limitation in pressure generation when using laser heating ?

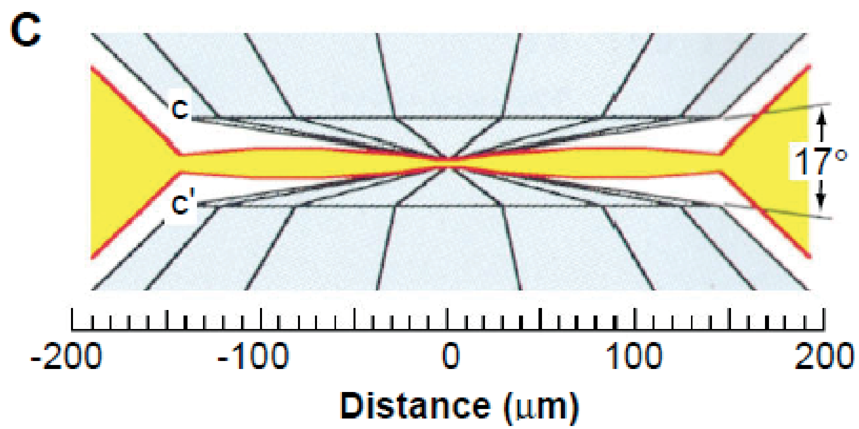


Ideal laser heating
up to ~60 GPa



Laser heating is still fine!
Max 200-300 GPa





No laser heating is possible without some space (microns) between the hot sample and the cold diamonds

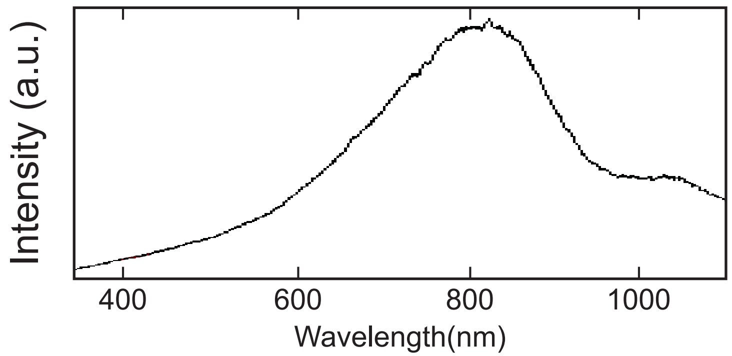
The maximum pressure is ~300 GPa today for temperatures of several 1000 K

What is the temperature limitation in the LH-DAC ?

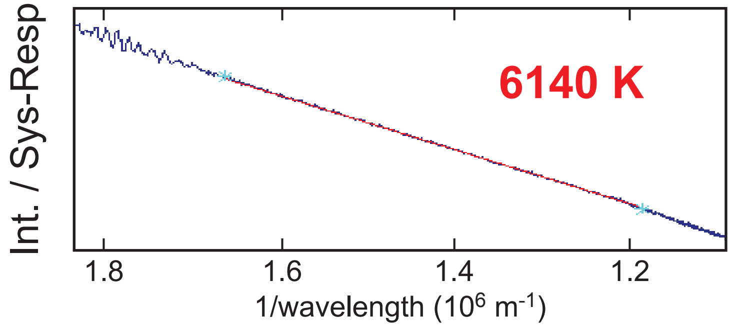
I do not known a limitation today

Temperature measurement at ID27

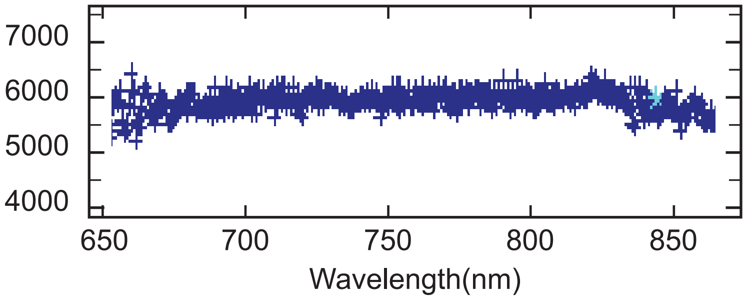
Raw pattern

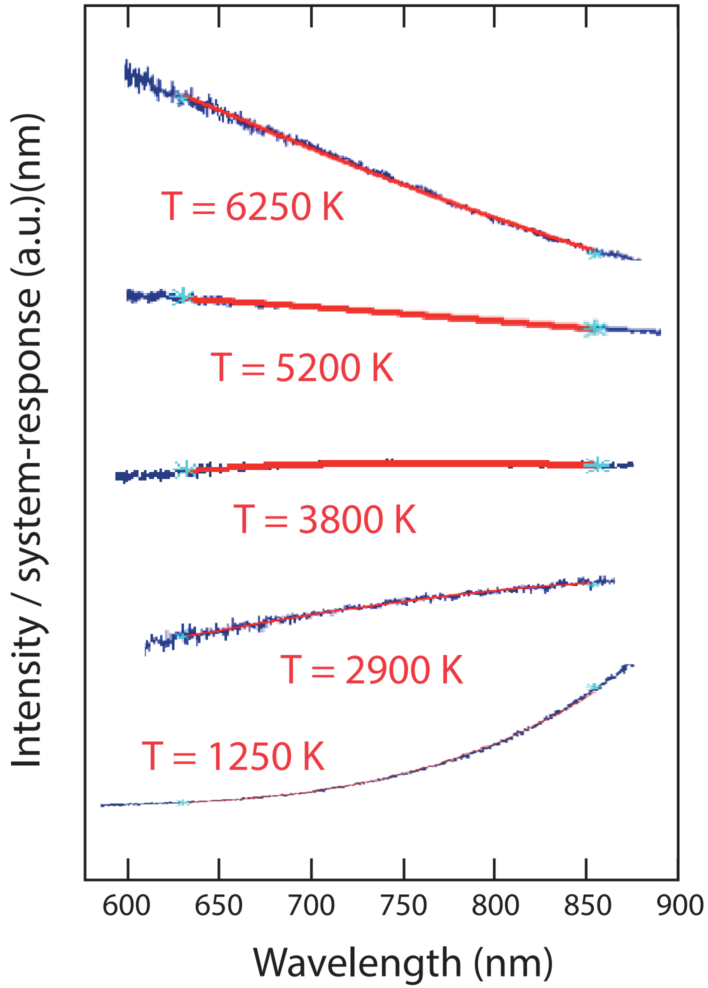


Corrected for system response and fitted



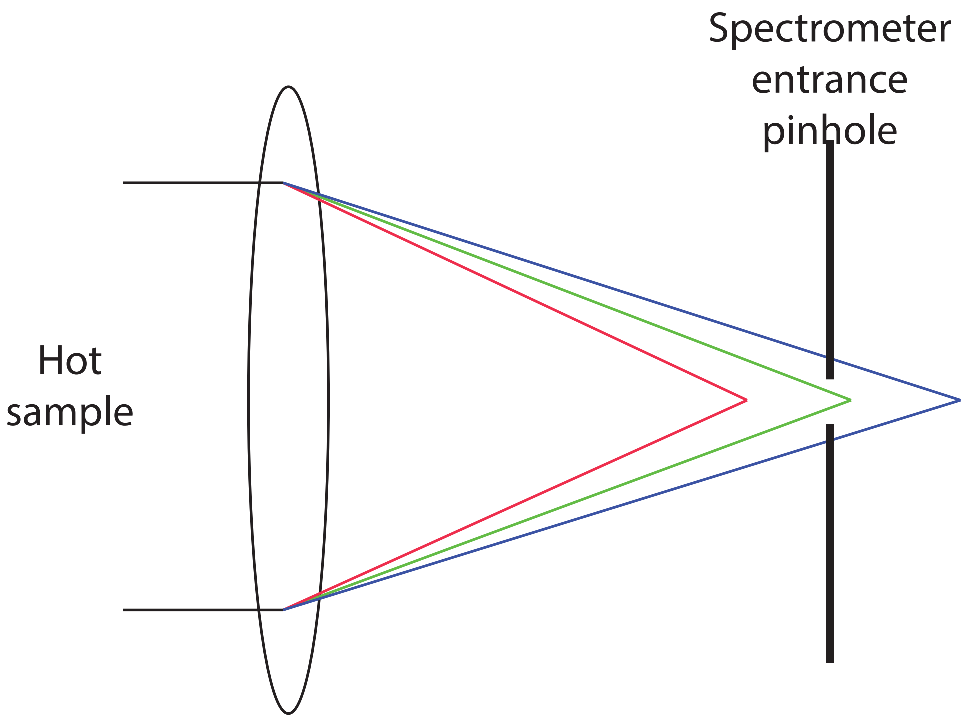
Two-color temperature

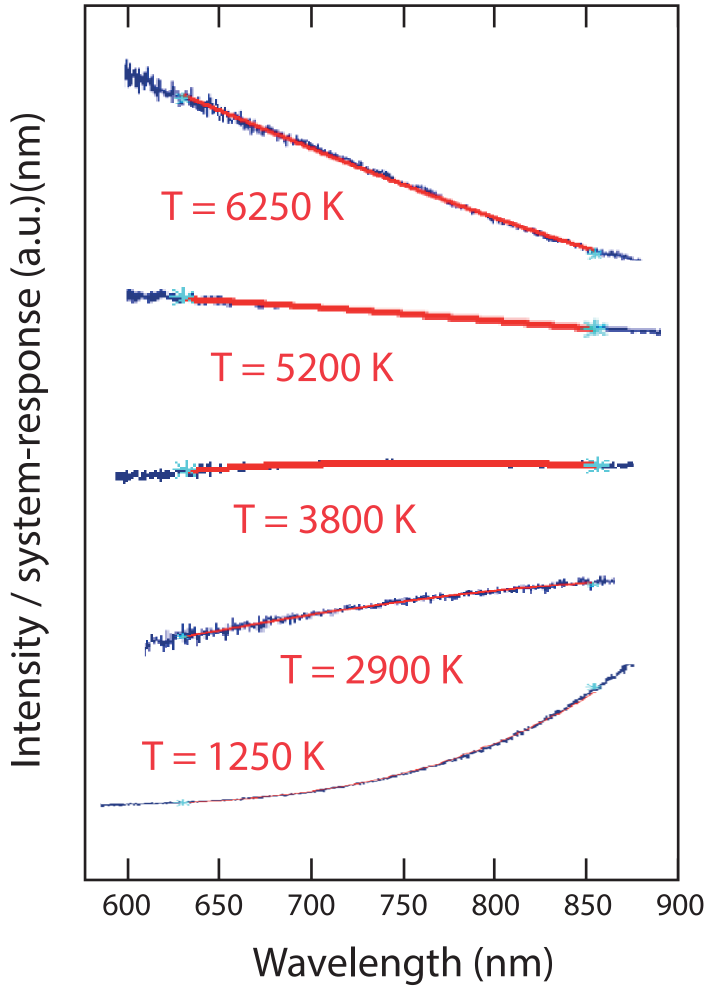




For measurement at high T, use reflective objectives !

Use of reflective objectives...

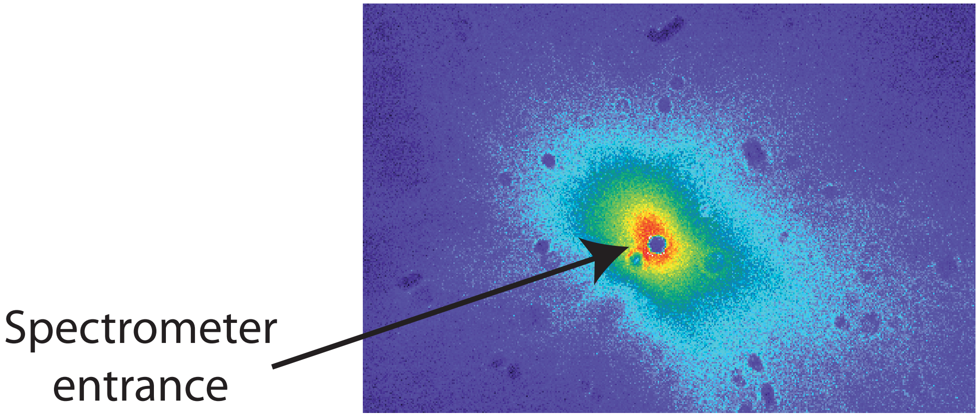




As we need the light intensity as a fonction of λ any chromatic abheration disables the temperature measurement

How to be sure that the sample properties are measured in the laser hot spot ?

Visualisation and alignement of the X-ray beam

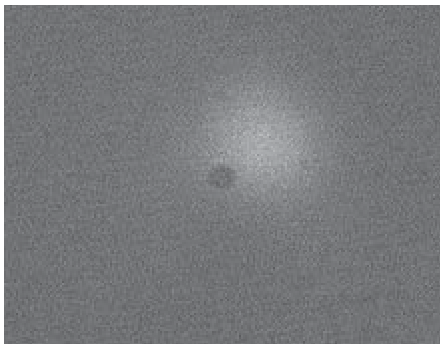


The use of pico or piezo motors
is very convenient for the
final optical alignements

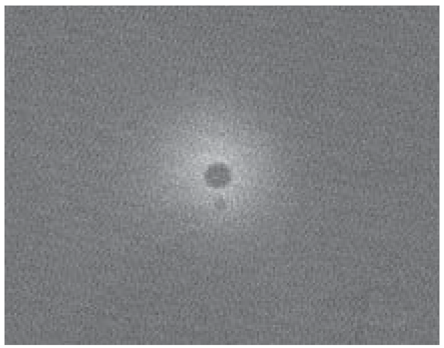
Warning: The good optical alignement
must be checked carrefully
at high laser power !

Alignement of the laser spot

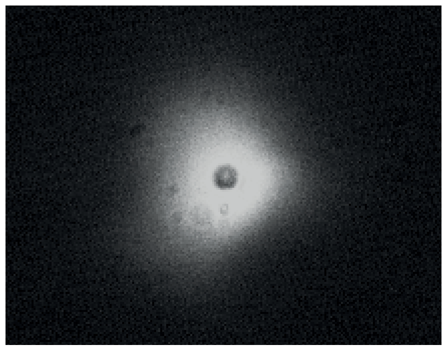
Misaligned : 1500 K



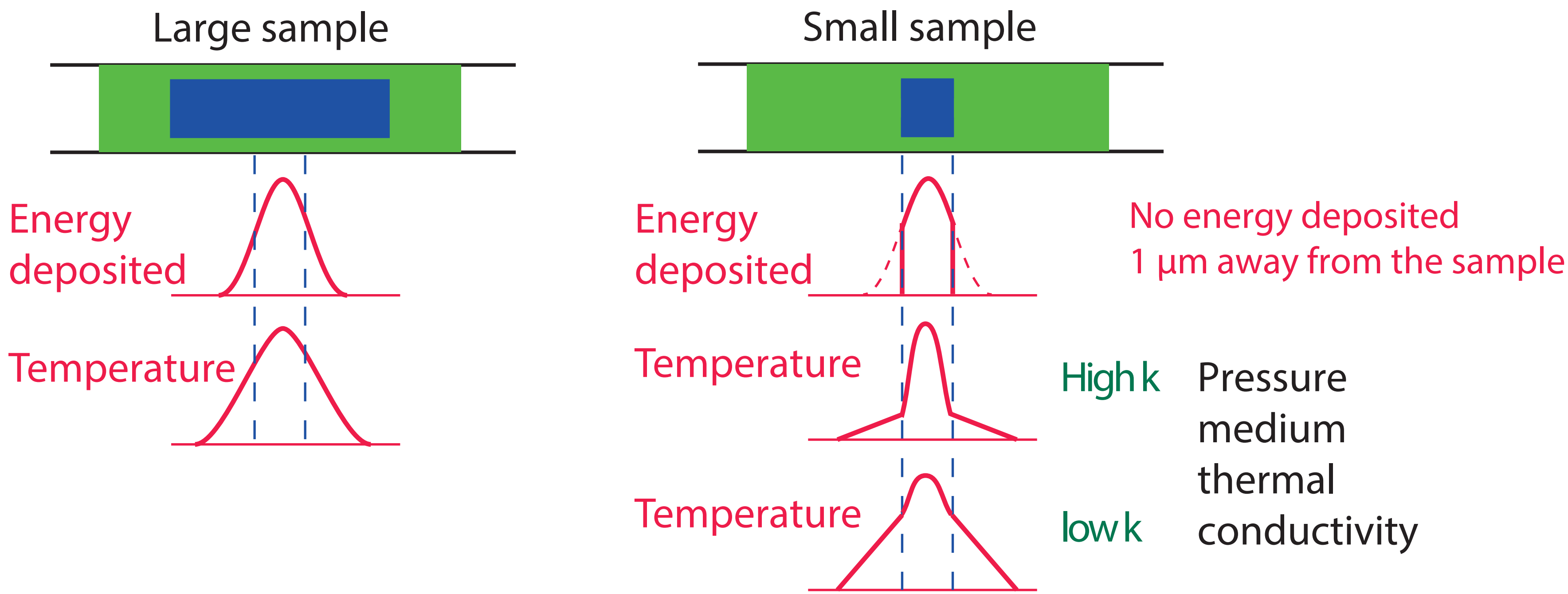
Aligned : 1500 K



2500 K

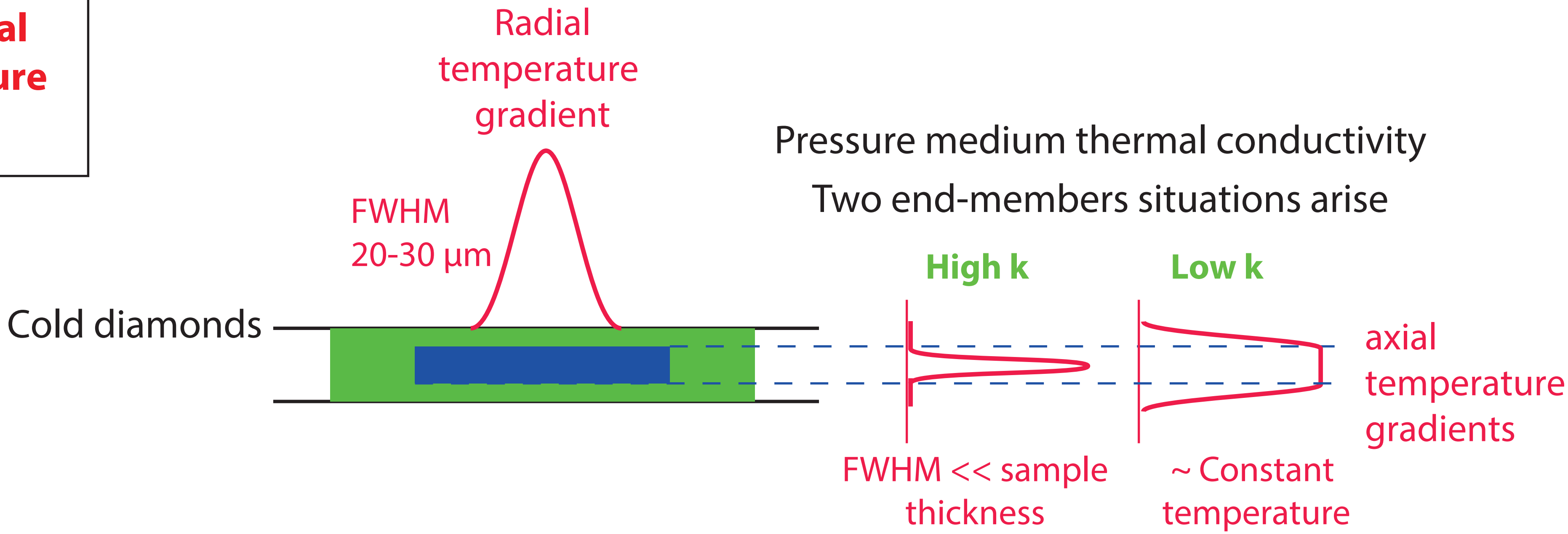


Can we resolve the radial temperature gradient using a very small sample ?



No, in most cases, it makes the radial gradient even worse !

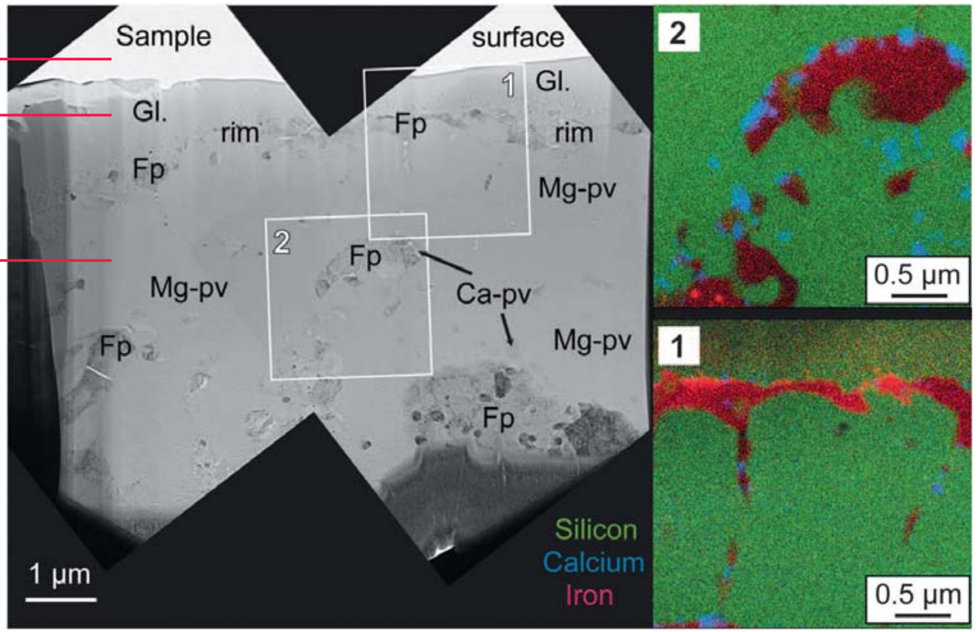
How critical is the radial temperature gradient ?



Diamond: ~500K

Glass < 1000K

Melt > 4000K

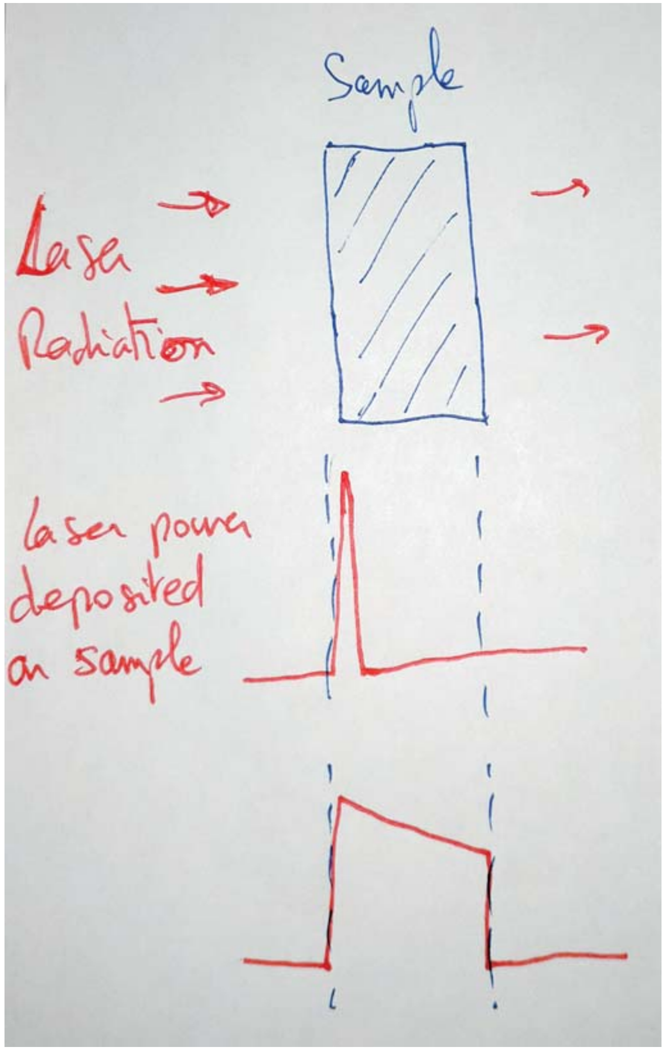


Melting of the Earth's mantle
Fiquet et al., 2010

Take away message: Minimizing the axial temperature gradient is often the major difficulty

Do I need double sided laser heating for my sample ?

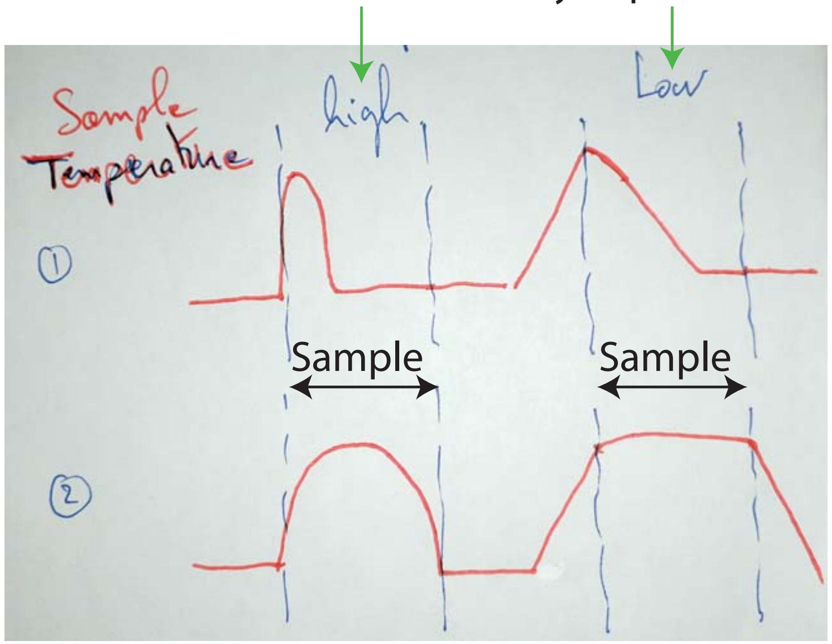
(I promised to good LH-DAC colleagues a «transparent-like» figure in this talk)



Laser absorption at the sample surface: Need 2 sides heating
- Metal heated by fiber laser (1µm)
- Oxide heated by CO2-laser

Laser absorption in the bulk of the sample (at λ=1µm):
- Oxide mixed with Pt-absorber power
- (Mg,Fe)-minerals

Thermal conductivity of pressure medium



What is the sample pressure in the laser spot ?

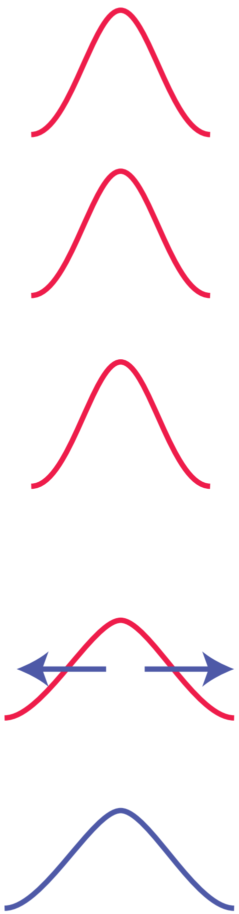
Energy deposited on sample

Temperature

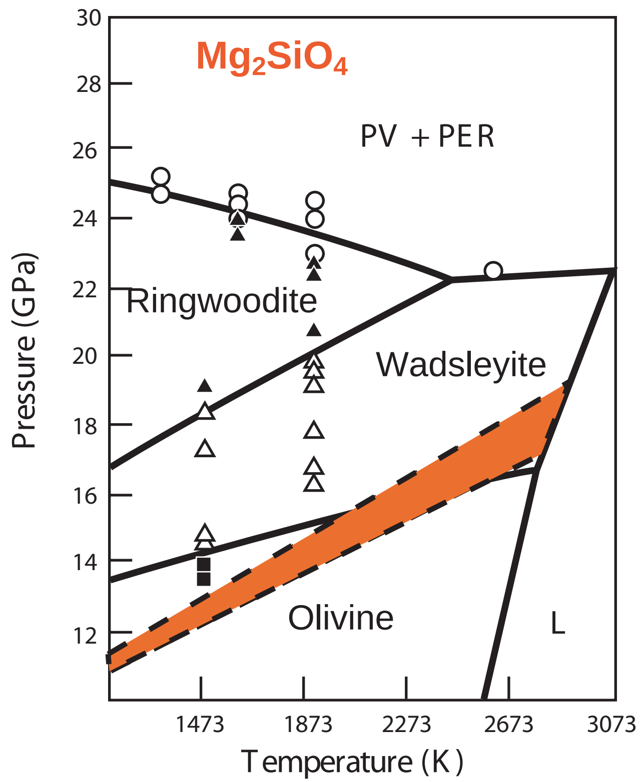
Theoretical thermal pressure $\Delta P_{th} = \alpha K \Delta T$

Relaxation of the pressure gradient

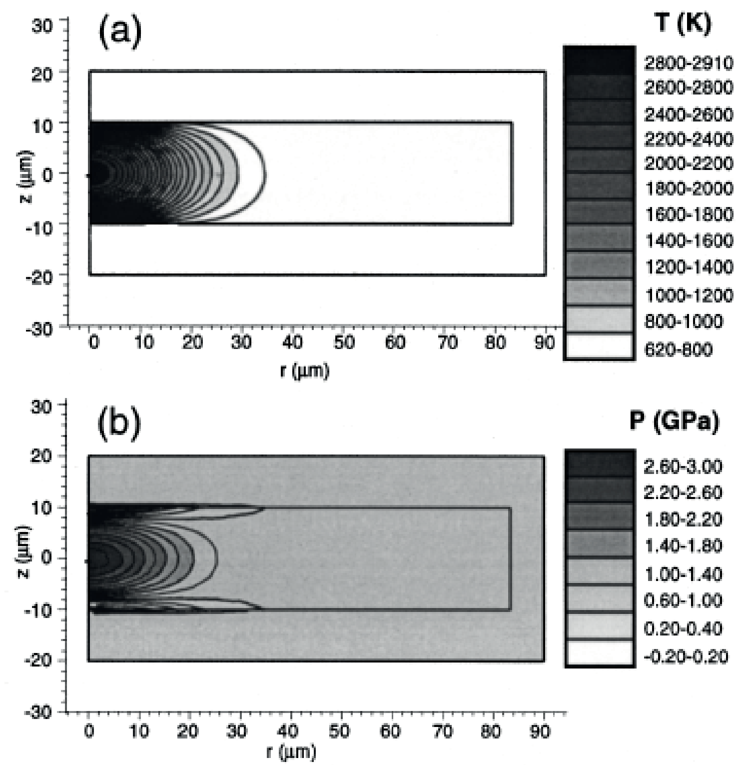
True pressure increase from 0.2 to 0.8 ΔP_{th}



We were successful to synthesize a HP polymorph based on thermal pressure



FInite element model
Dewaele et al., 1998

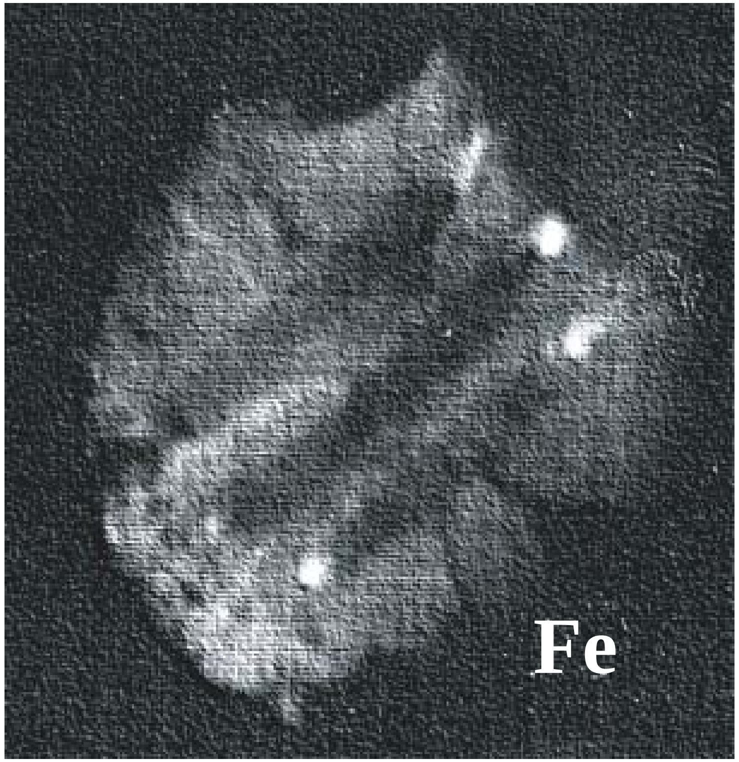


Pressure is significantly improved in the laser spot. It can be 2.5 GPa/1000K

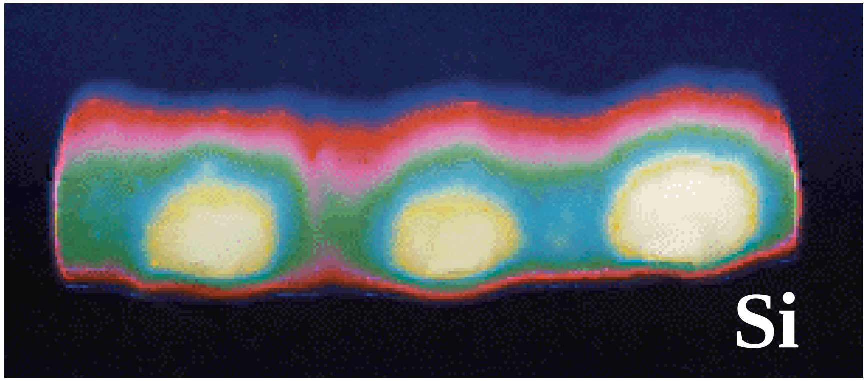
=> It is not possible to measure a PVT EoS without an internal pressure calibrant (e.g. Pt for MgSiO3)

Any potential problem with chemical migration in the laser spot ?

Yes, major problems !



Chemical analyses of olivine (Mg,Fe)2SiO4 recovered after laser scan of the laser over the entire sample surface

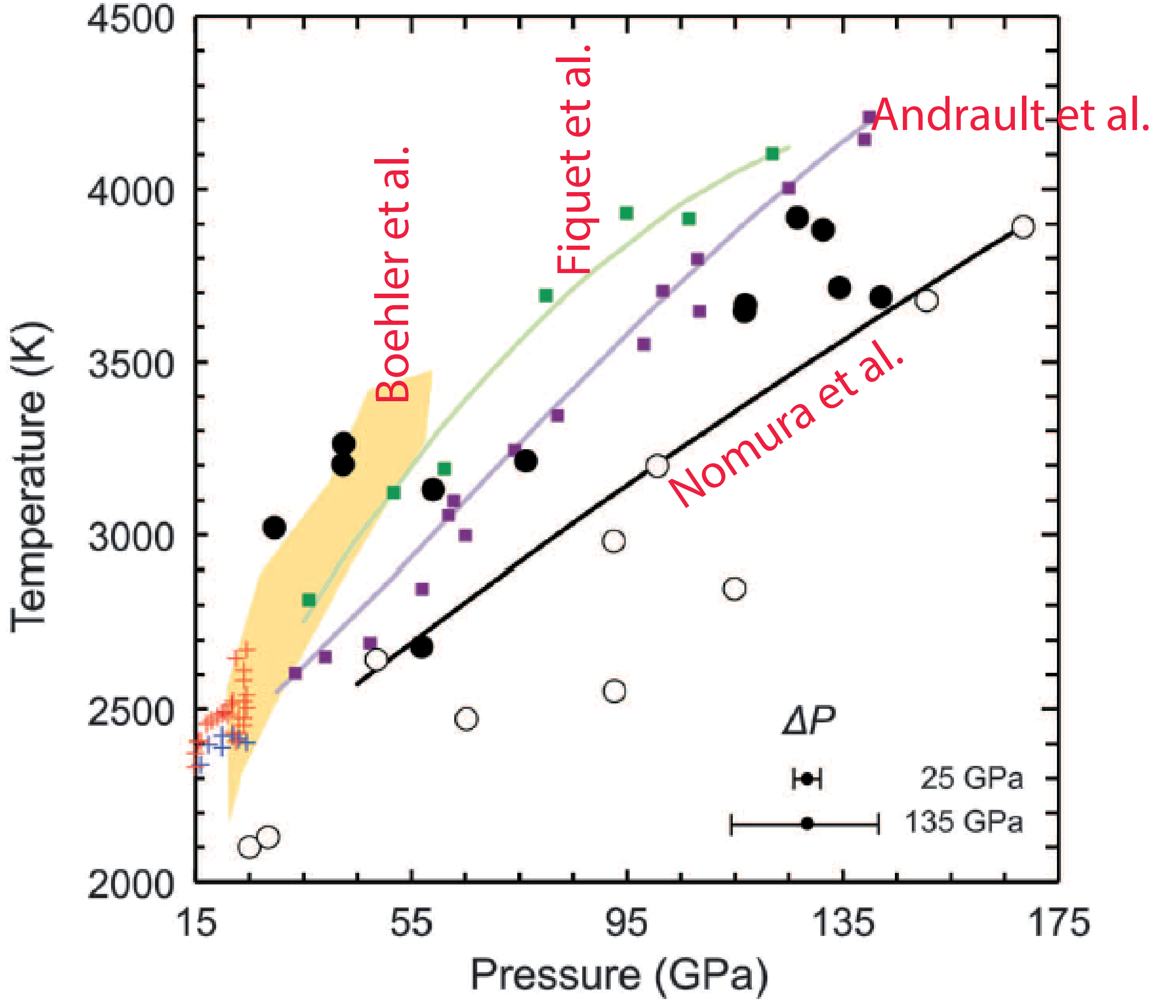


For this reason, the LH-DAC cannot provide constrains on the Equation of state of (Mg,Fe)-minerals; the interdiffusion is too easy.

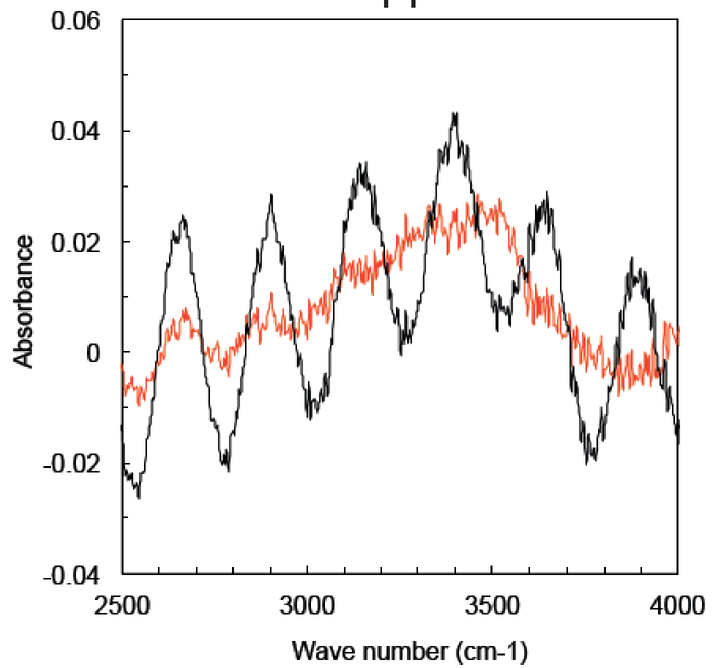
We need fast measurements => ESRF-EBS

Any potential problem of chemical pollution of the sample ?

Reported solidus for various mantle compositions



Infrared absorption of the recovered sample
=> 1510 ppm water



(Nomura et al., 2014) is a wet-solidus...

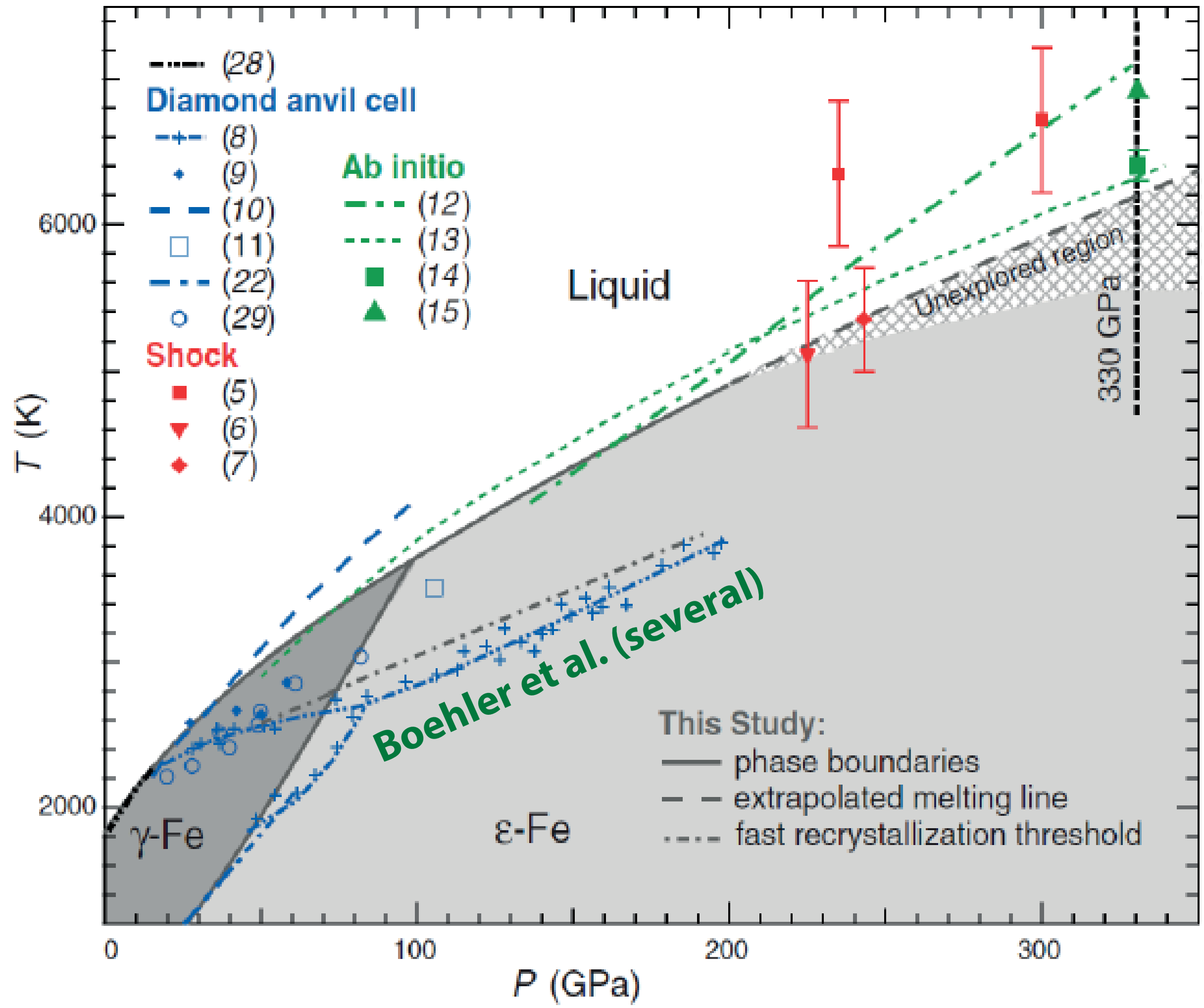
=> For high-T studies, always load your DAC in a glove bag under Ar/N2 flux

Any potential problem with chemical polution of the sample

Yes, major problems !

Carbon diffusion

Anzellini et al., 2013



At very high temperature, carbon diffuses from the diamond anvils to metallic sample.

The Fe-C melting curve is much lower than that of pure Fe

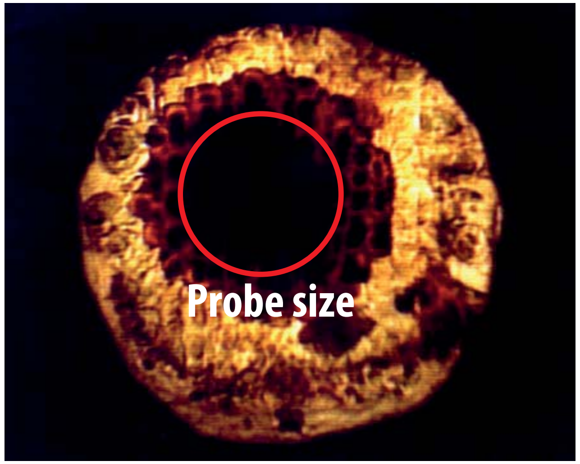
Only fast measurements can solve the melting curve of pure Fe

We need fast measurements => ESRF-EBS

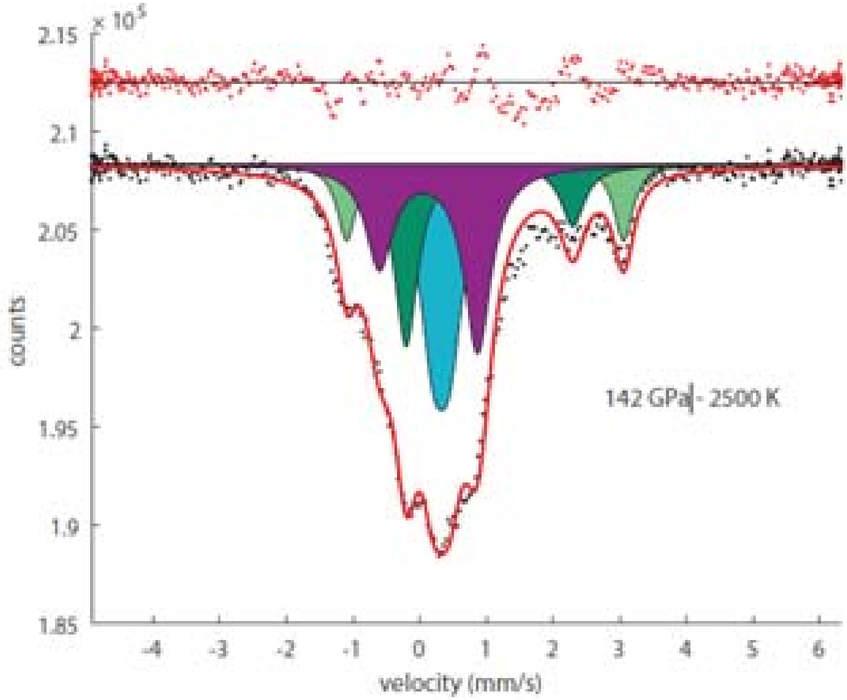
How critical is the size of the X-ray probe ?

Probe size >> hot spot

The laser can be scanned
several minutes on the sample
for partial homogeneization



Then, the data set can be deconvoluted:
Mossbauer spectrum registered at ID18
Deconvolution of a mixture of
Fe2+ (LS & HS) in ferropericlase
Fe2+ (LS and IS) and Fe3+ (LS) in bridgmanite



Probe size << hot spot => Good configuration: Small radial temperature gradient on sample

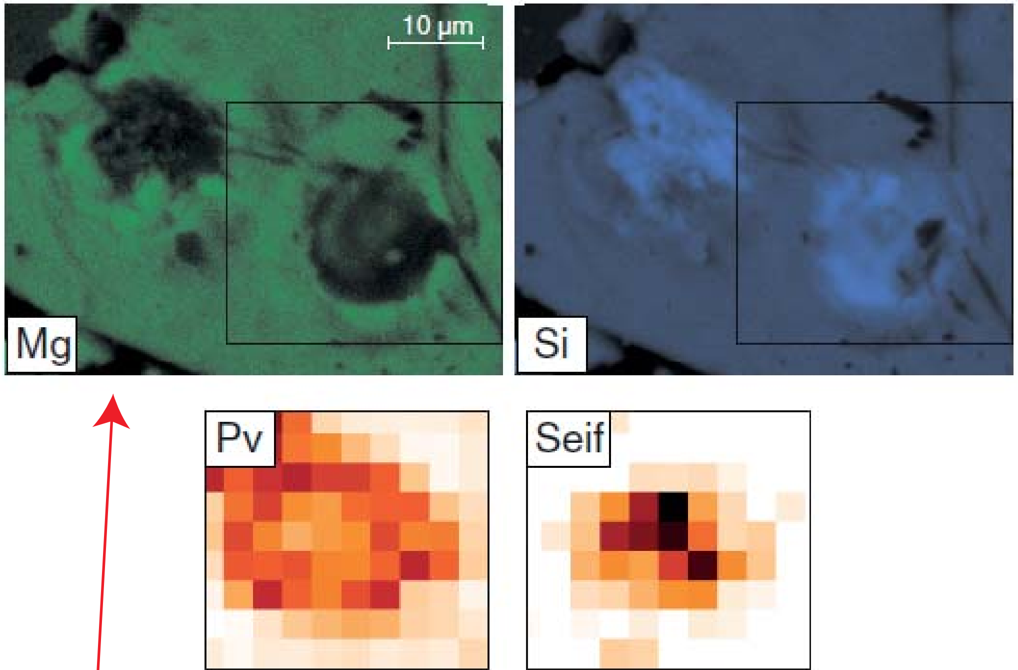
WE NEED
ESRF-EBS

How critical is the size of the X-ray probe ?

Probe size << hot spot + short acquisition time

=> This allows the mapping of the sample properties

Melting of a basalt at P=120 GPa



Electron microprobe
Chemical mapping

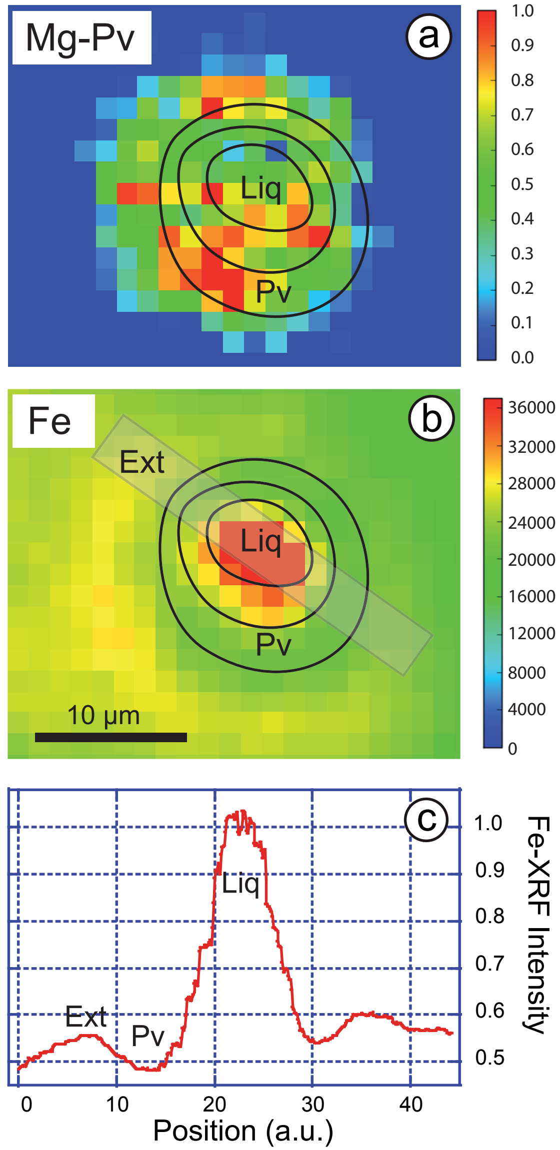
Mineralogical mapping
in situ X-ray diffraction

Melting of pyrolite at P=78 GPa

X-ray diffraction

X-ray fluorescence

Fe-content



Is any type of measurement available in the LH-DAC at ESRF ?

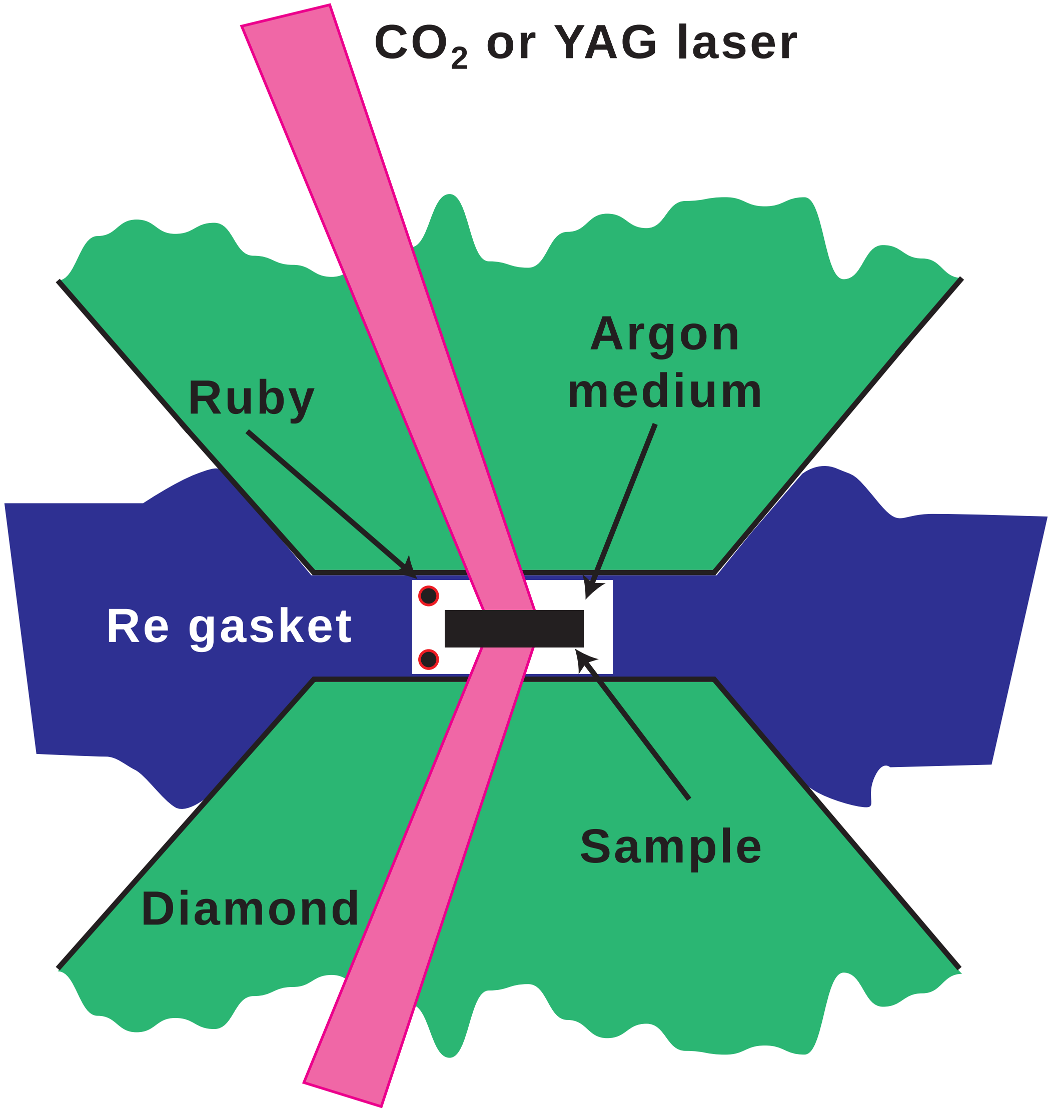
To date, these types of measurements were performed:

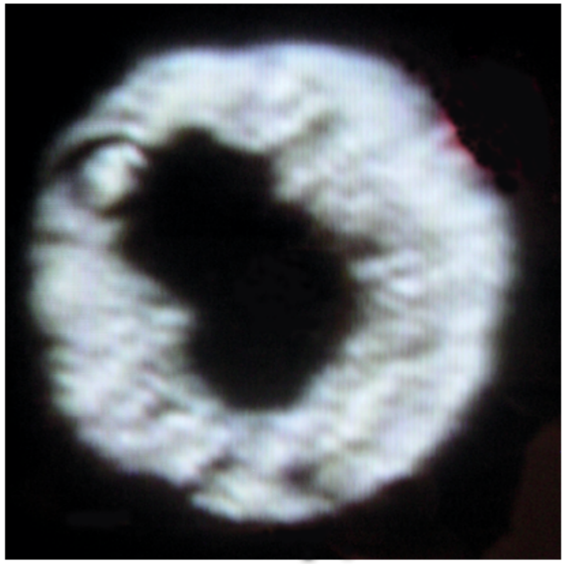
- X-ray diffraction
- X-ray absorption (XANES)
- Inelastic scattering (phonons)
- Mössbauer spectroscopy
- X-ray emission spectroscopy
- X-ray raman (?)
- and maybe others ?

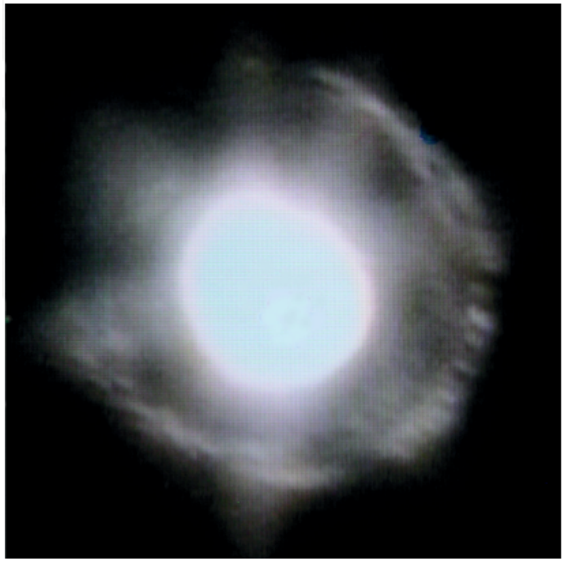
For some techniques, some limitations remain in :

- Acquisition time
- Size of the beam
- Absorption of diamond window

With the EBS-ESRF, many beamlines will offer a much smaller beam, which will make the LH-DAC system even more suited







Thanks for your attention!

Any comments on my «questions» ?
Any additional question that «we» would comment ?