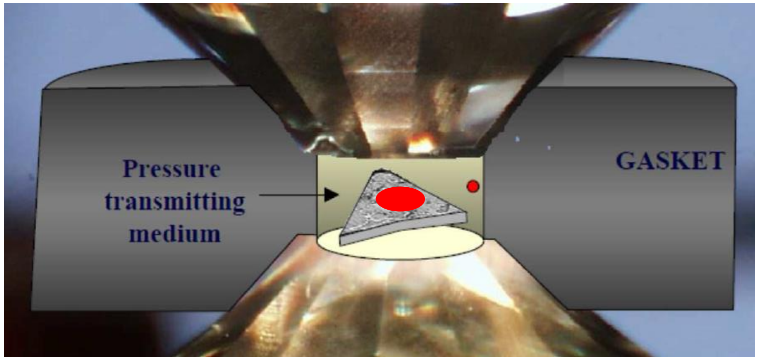
Guillaume MORARD

Institut des Sciences de la Terre

Phase transitions in laser heated diamond anvil cell: observations from in situ and ex situ analyses



Different type of phase transitions

-First order solid solid transition

-Second order solid solid transition

-Congruent melting

-Incongruent melting

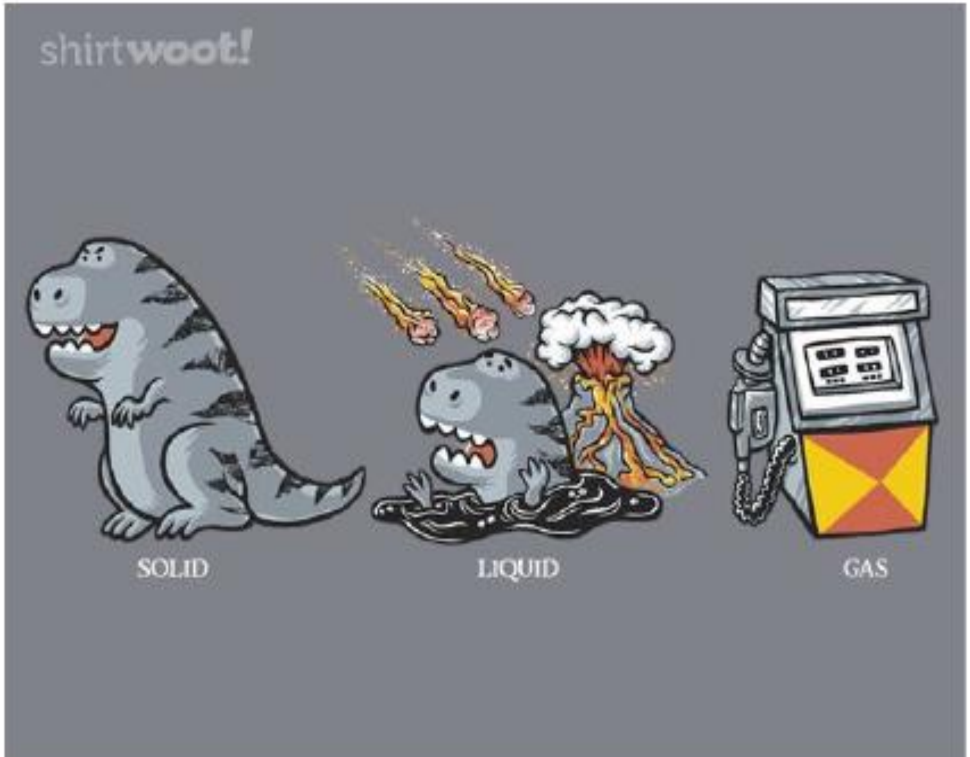
Different type of phase transitions

-First order solid solid transition

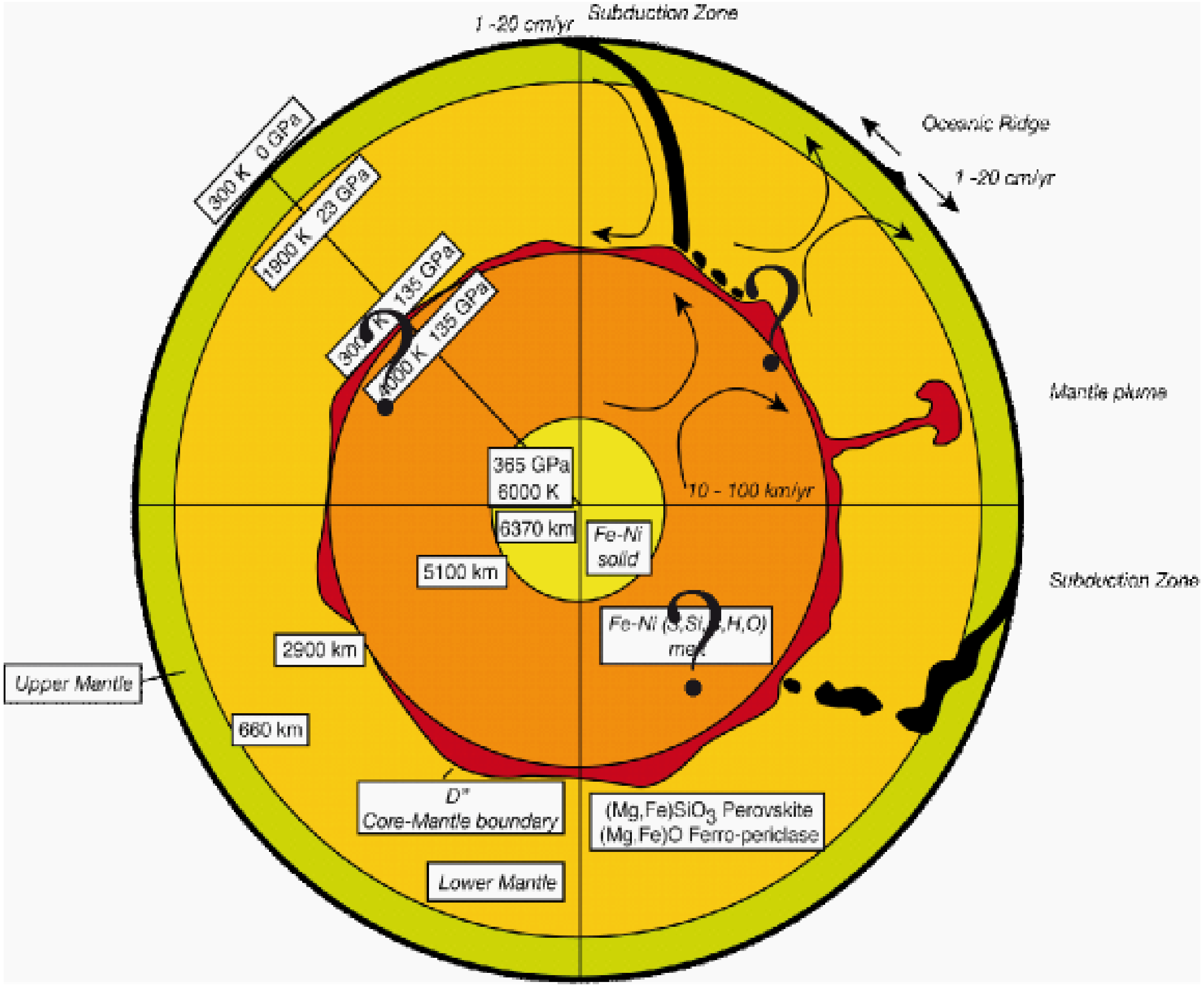
-Second order solid solid transition

-Congruent melting

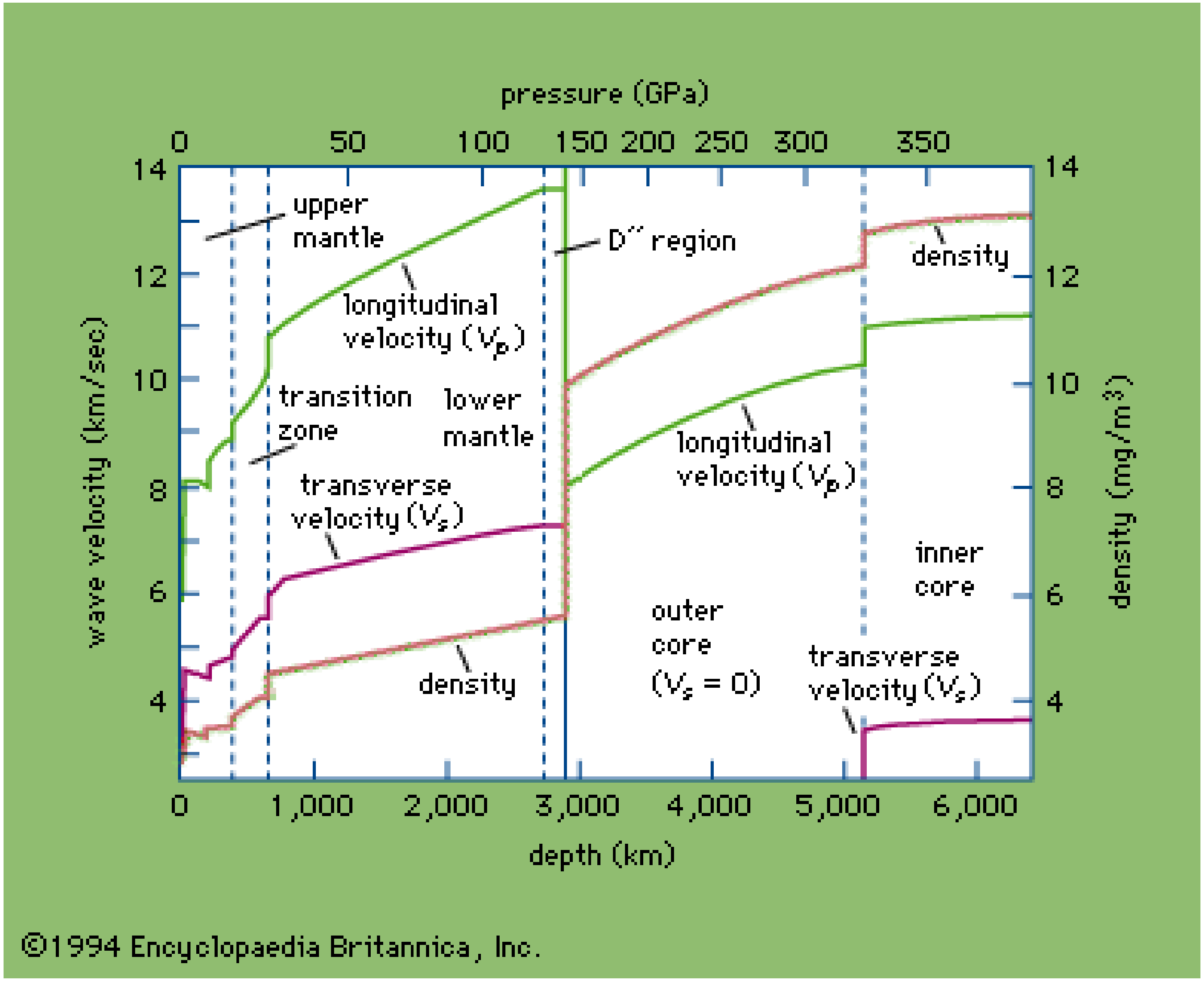
-Incongruent melting



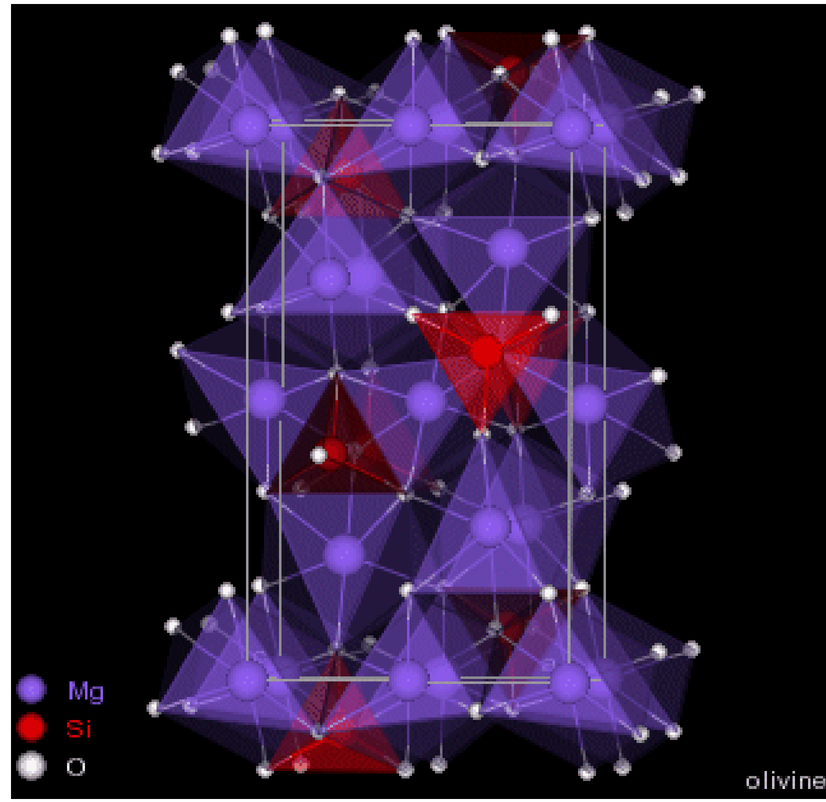
Internal structure of the Earth



Information on the Earth's interior

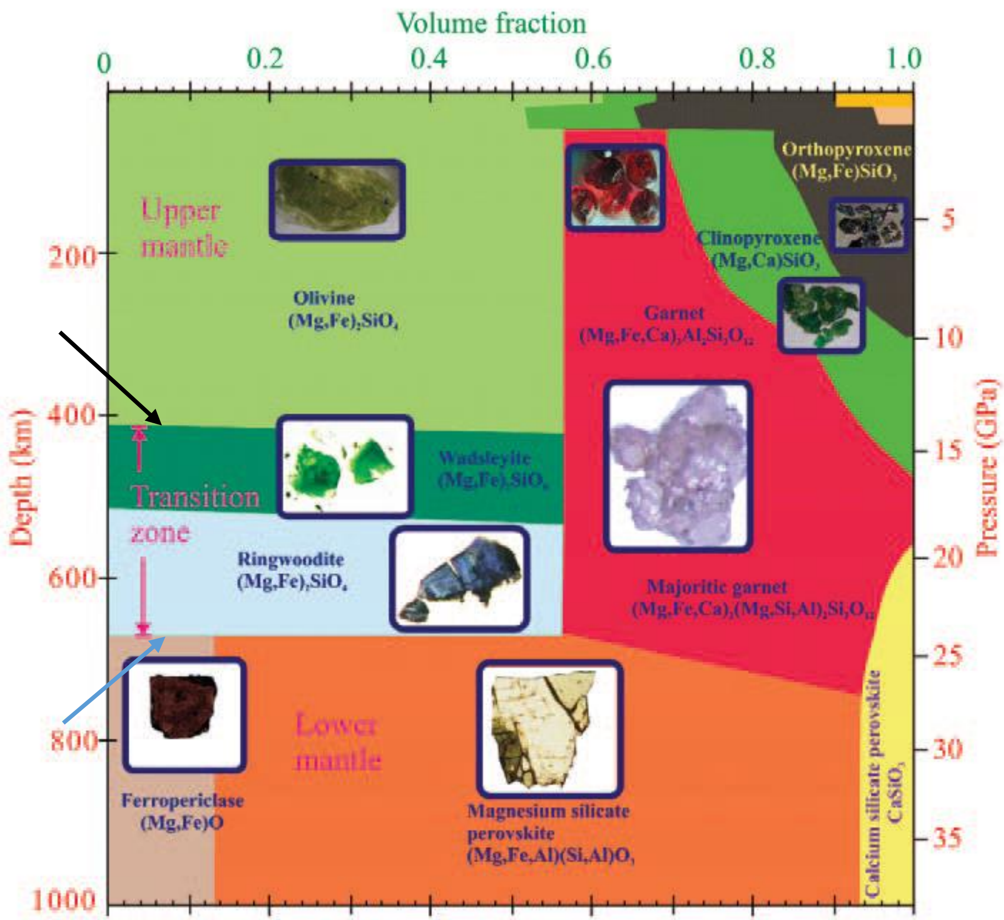


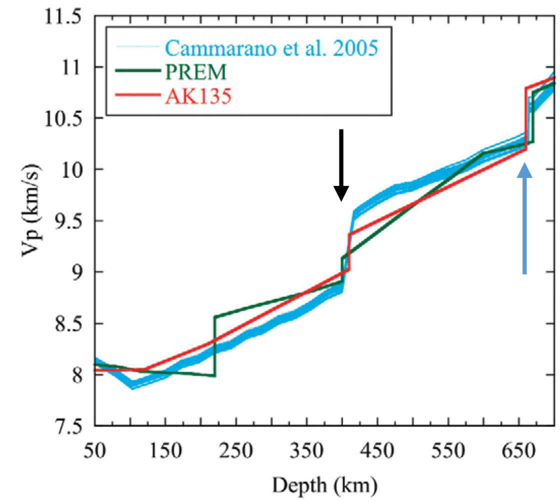
Olivine: 60% en volume des roches du manteau supérieur

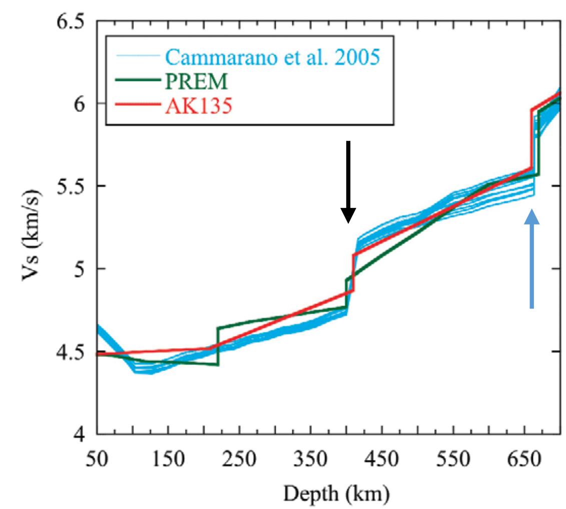


$(Mg,Fe)_2SiO_4$

Strong relation between seismological structure of the upper mantle and its mineralogy



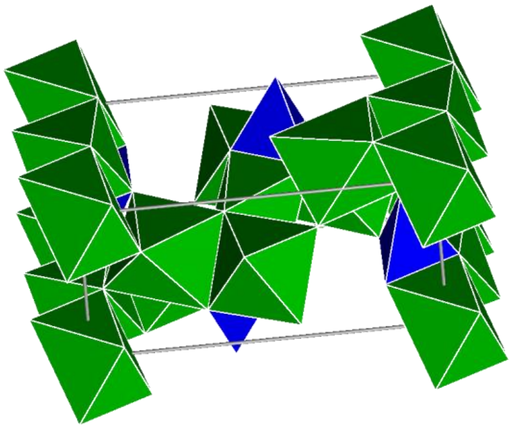




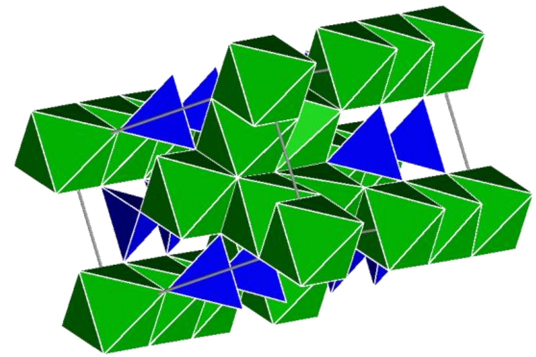
Frost, Elements, 2008

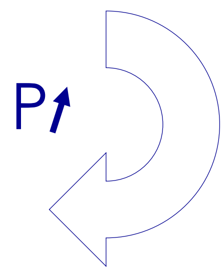
Transitions de phases induites par la pression

α-olivine $(Mg,Fe)_2SiO_4$

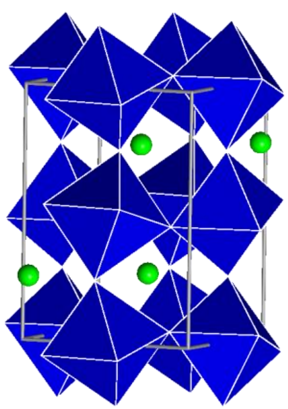


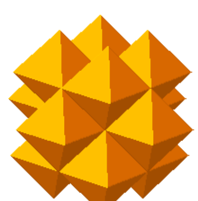
β-wadsleyite $(Mg,Fe)_2SiO_4$





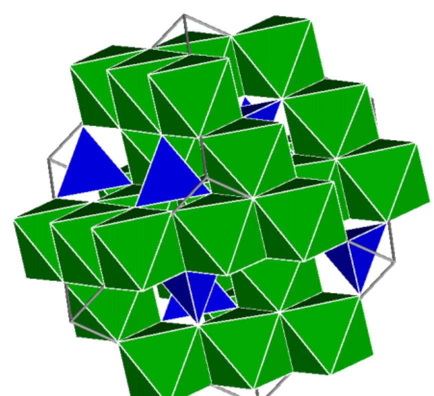
Perovskite $(Mg,Fe)SiO_3$



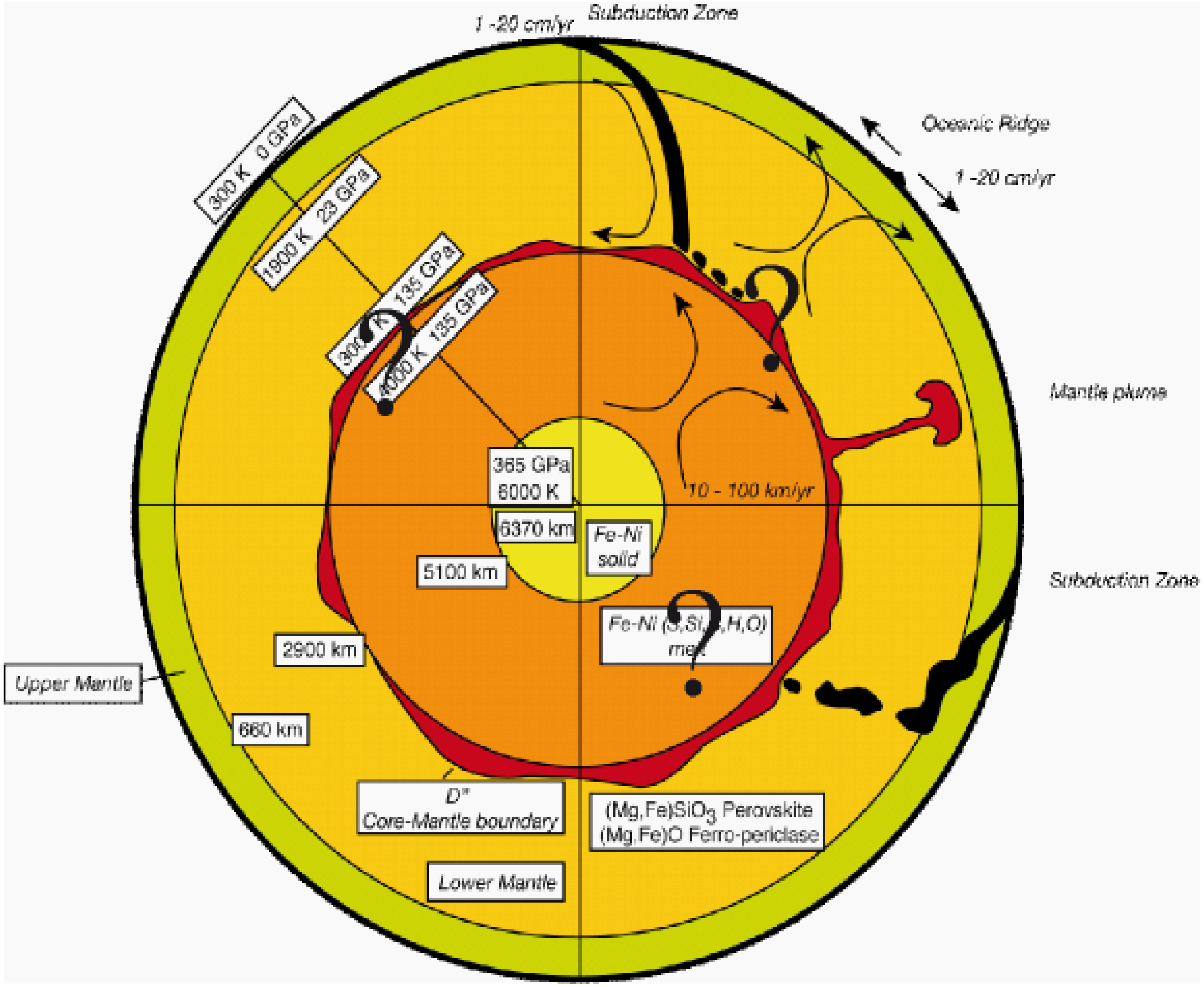


$+(Mg,Fe)O$

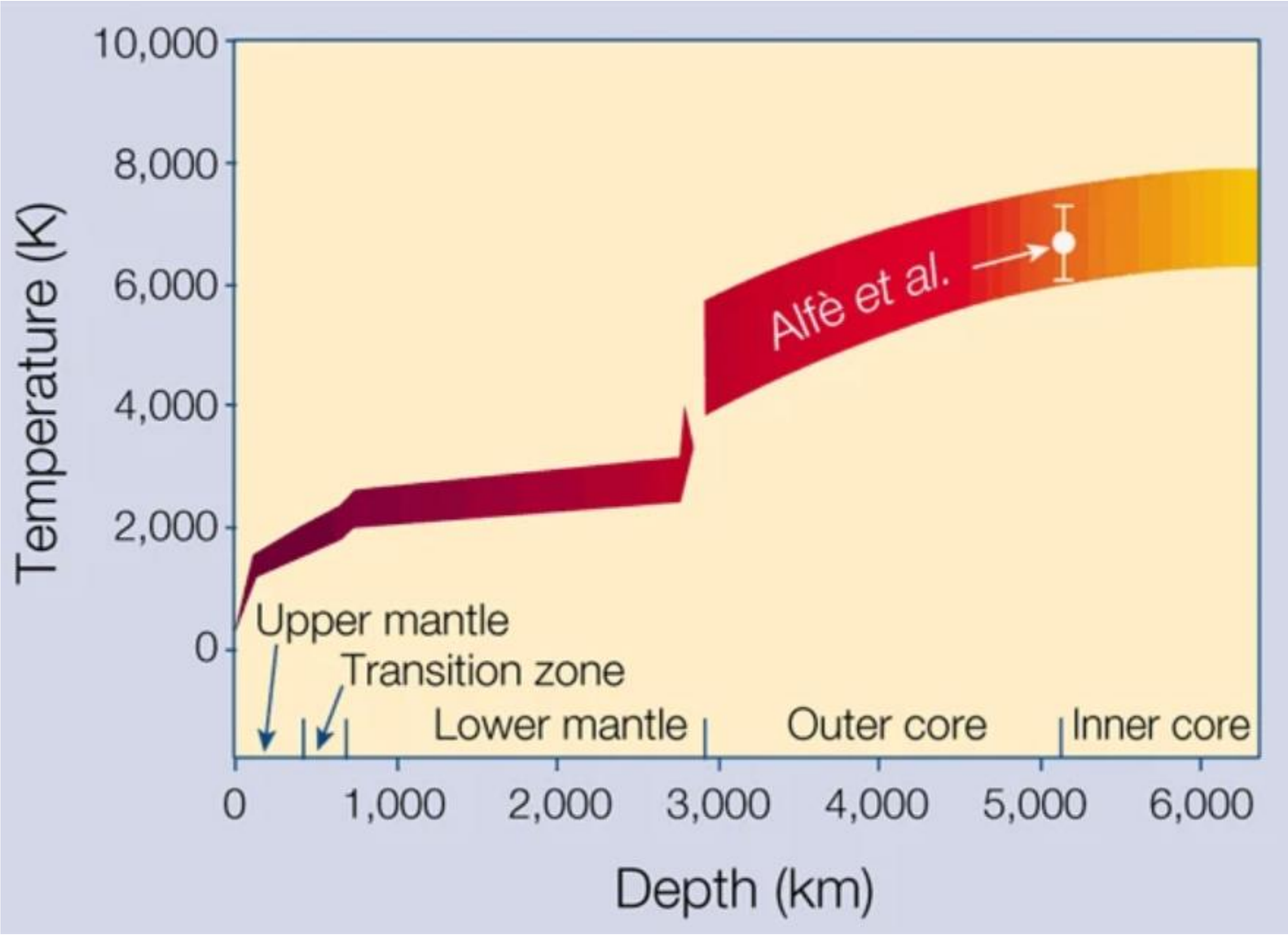
γ-ringwoodite $(Mg,Fe)_2SiO_4$

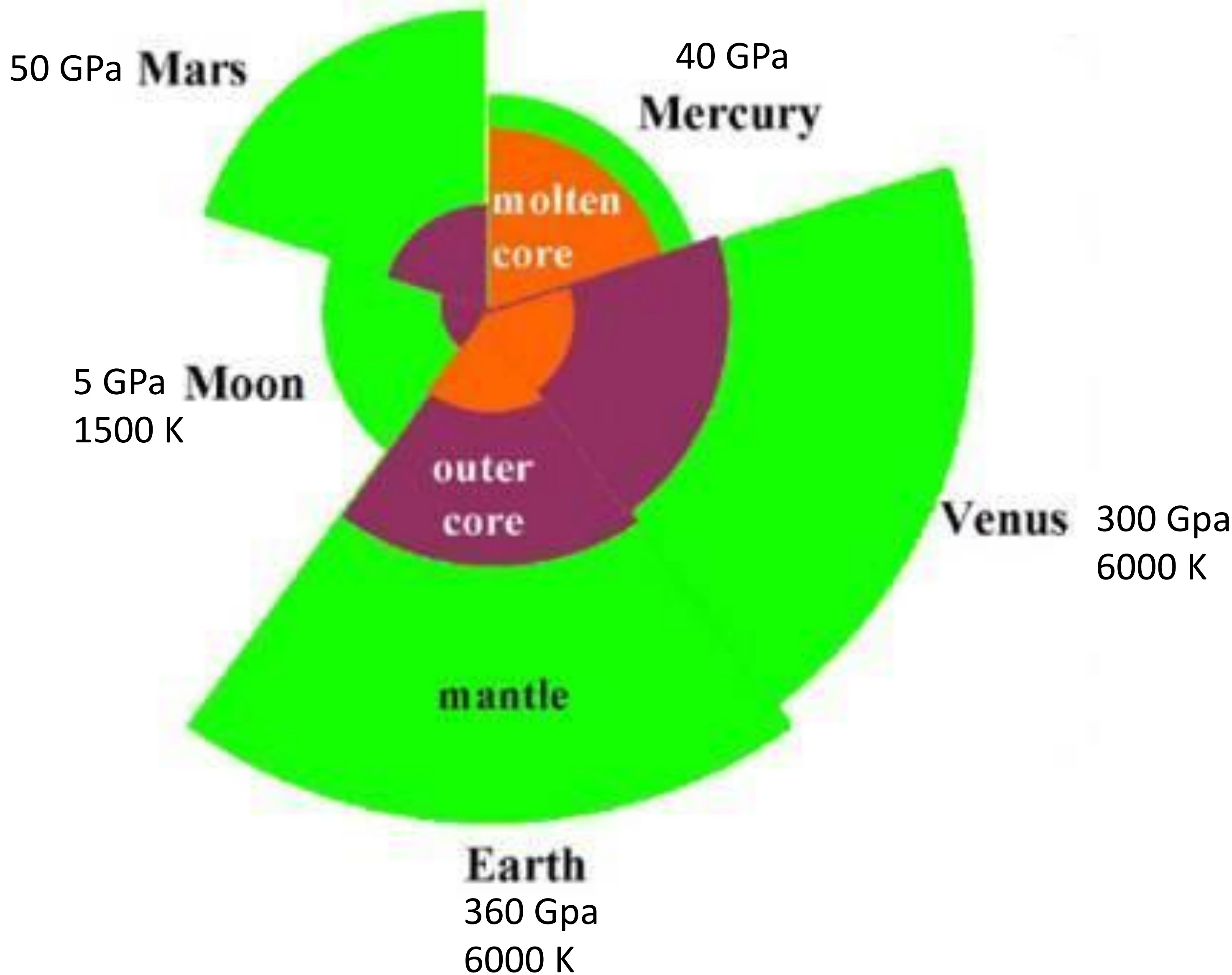


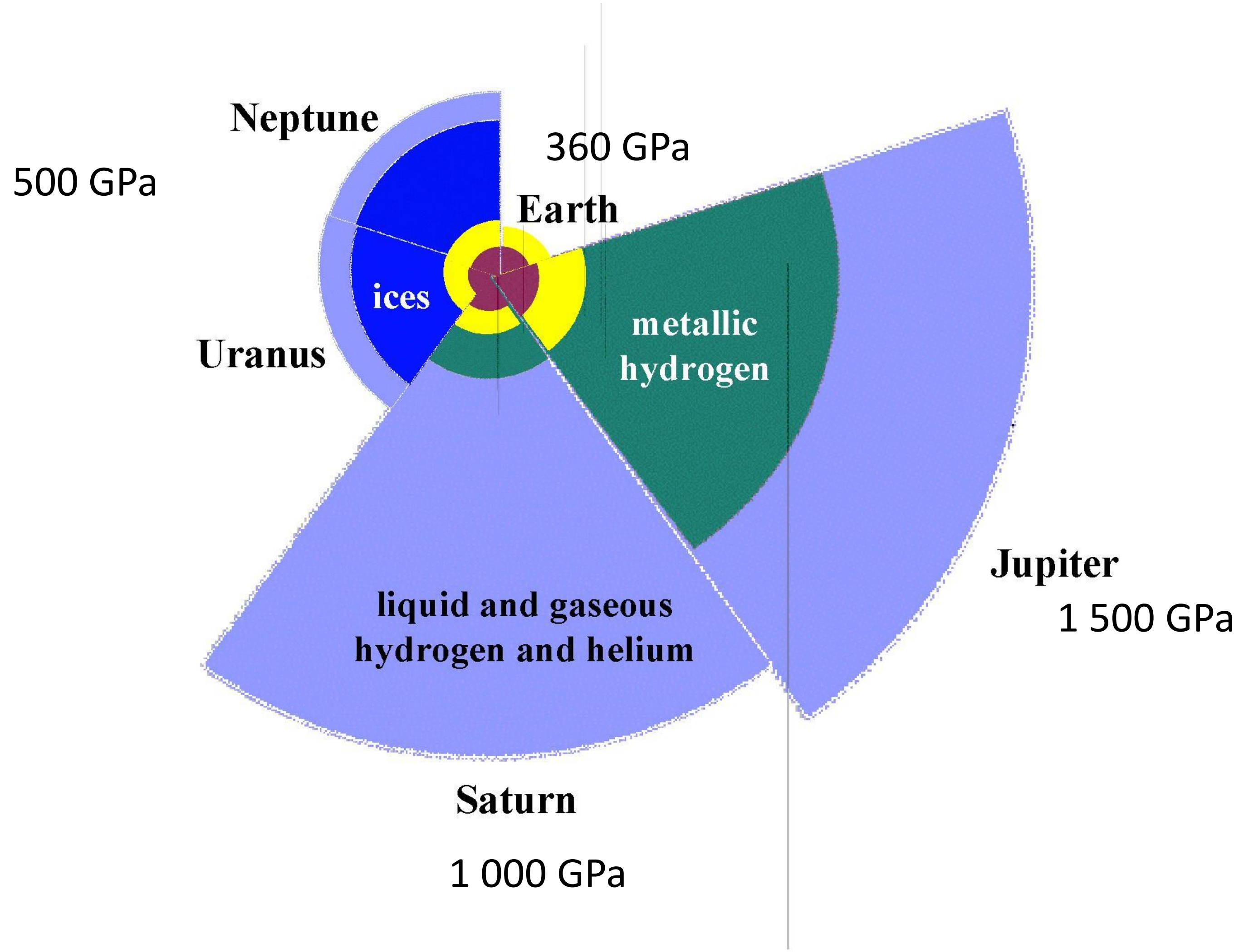
Internal structure of the Earth



Melting of core materials at ICB : anchoring point for the geotherm

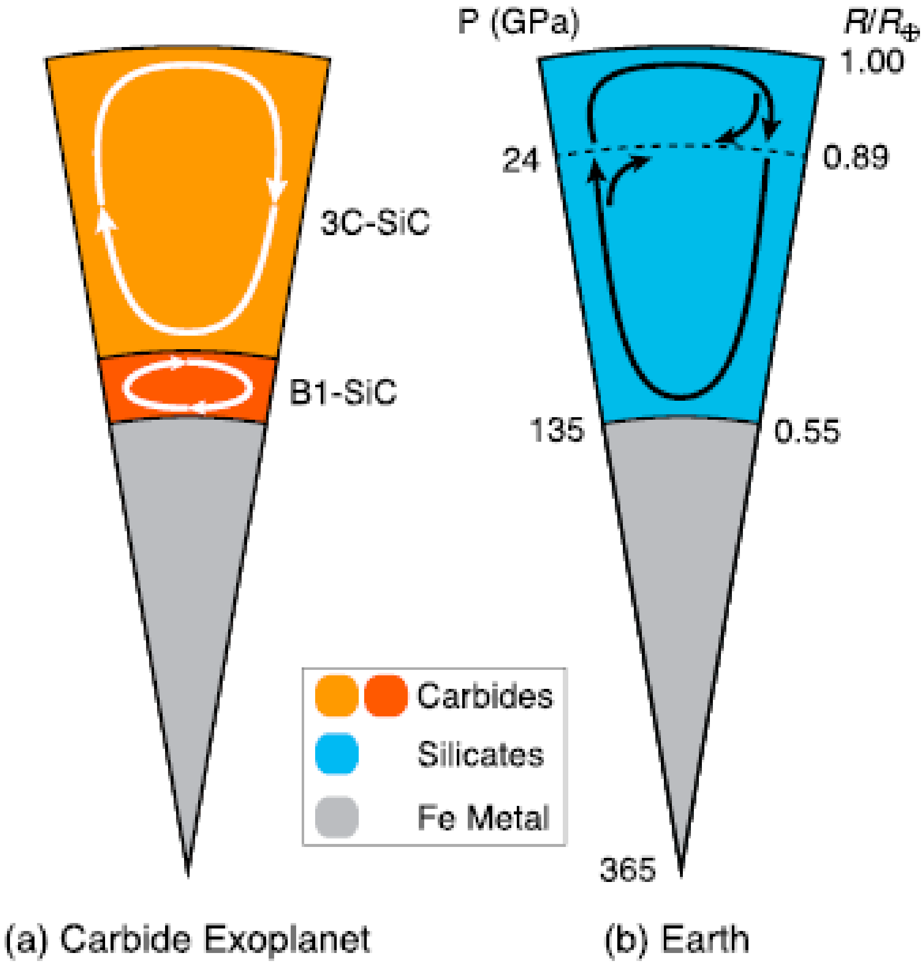



50 GPa Mars
40 GPa
Mercury
molten
core
5 GPa Moon
1500 K
outer
core
Venus 300 Gpa
6000 K
mantle
Earth
360 Gpa
6000 K

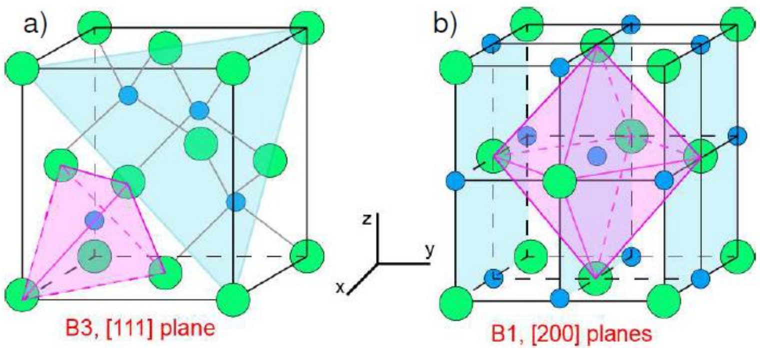


Neptune
500 GPa
360 GPa
Earth
ices
Uranus
metallic
hydrogen
liquid and gaseous
hydrogen and helium
Jupiter
1 500 GPa
Saturn
1 000 GPa

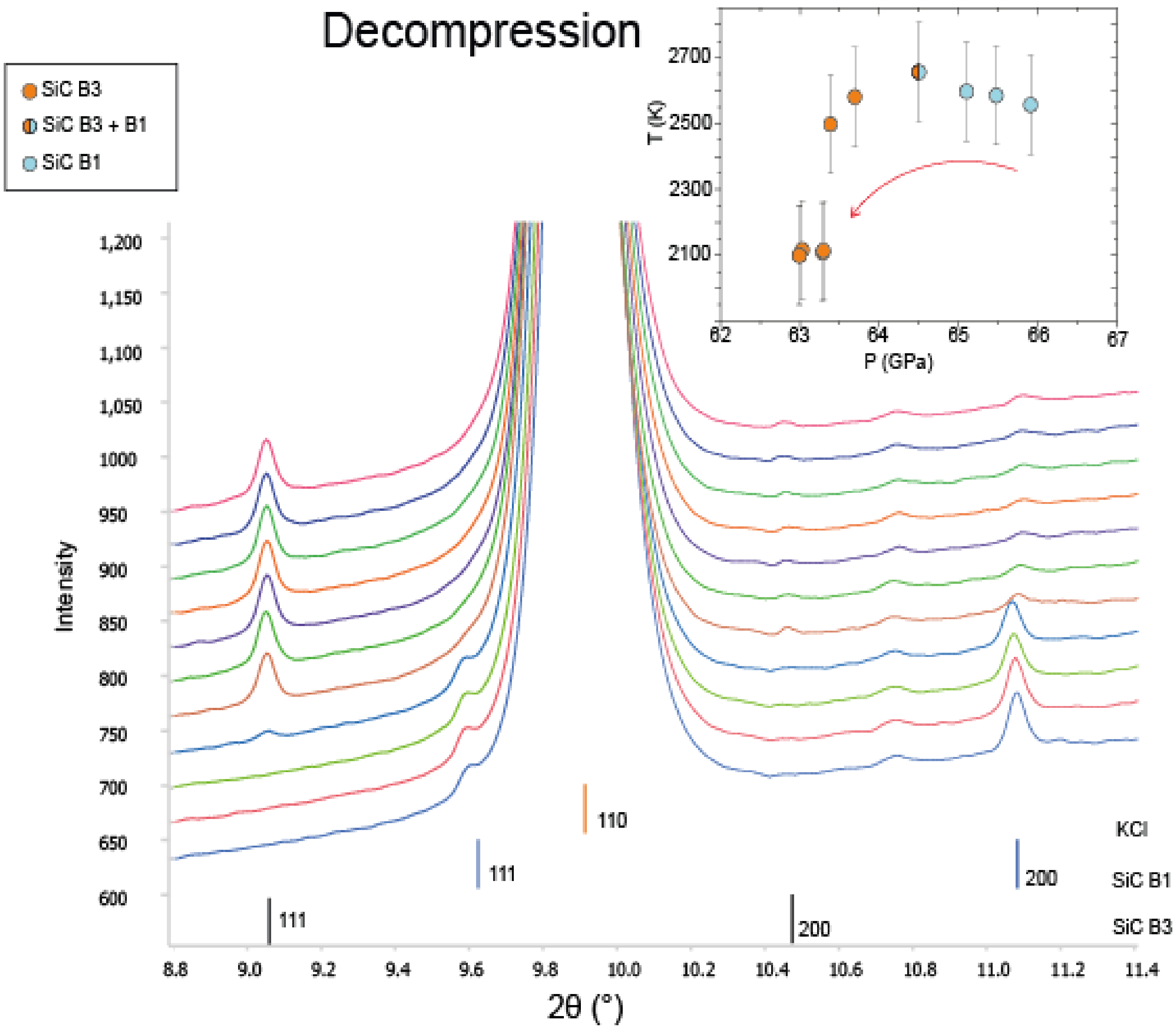
First order phase transition



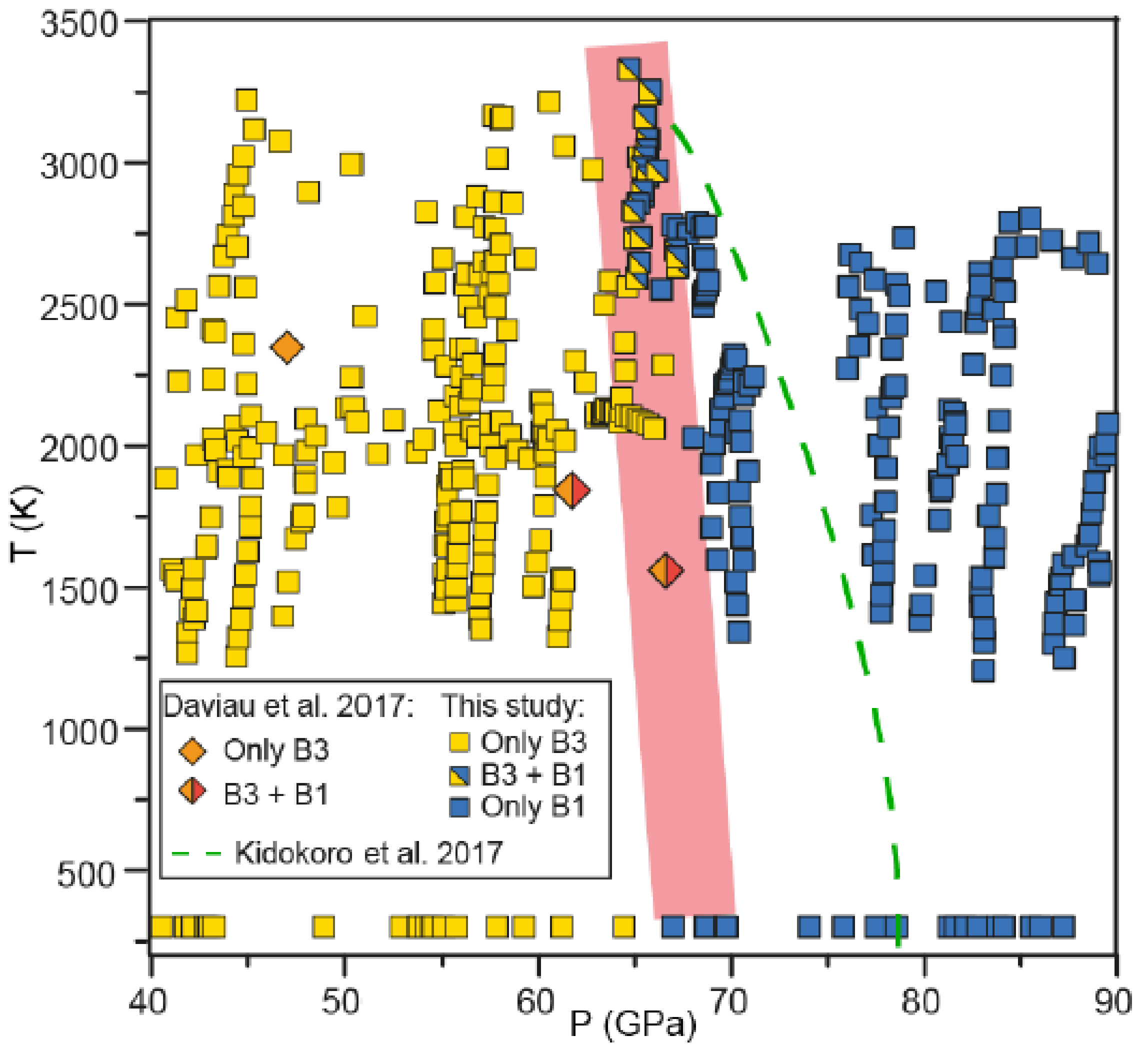
Potential existence of carbide exoplanets: Interest for SiC compound phase diagram under high pressure



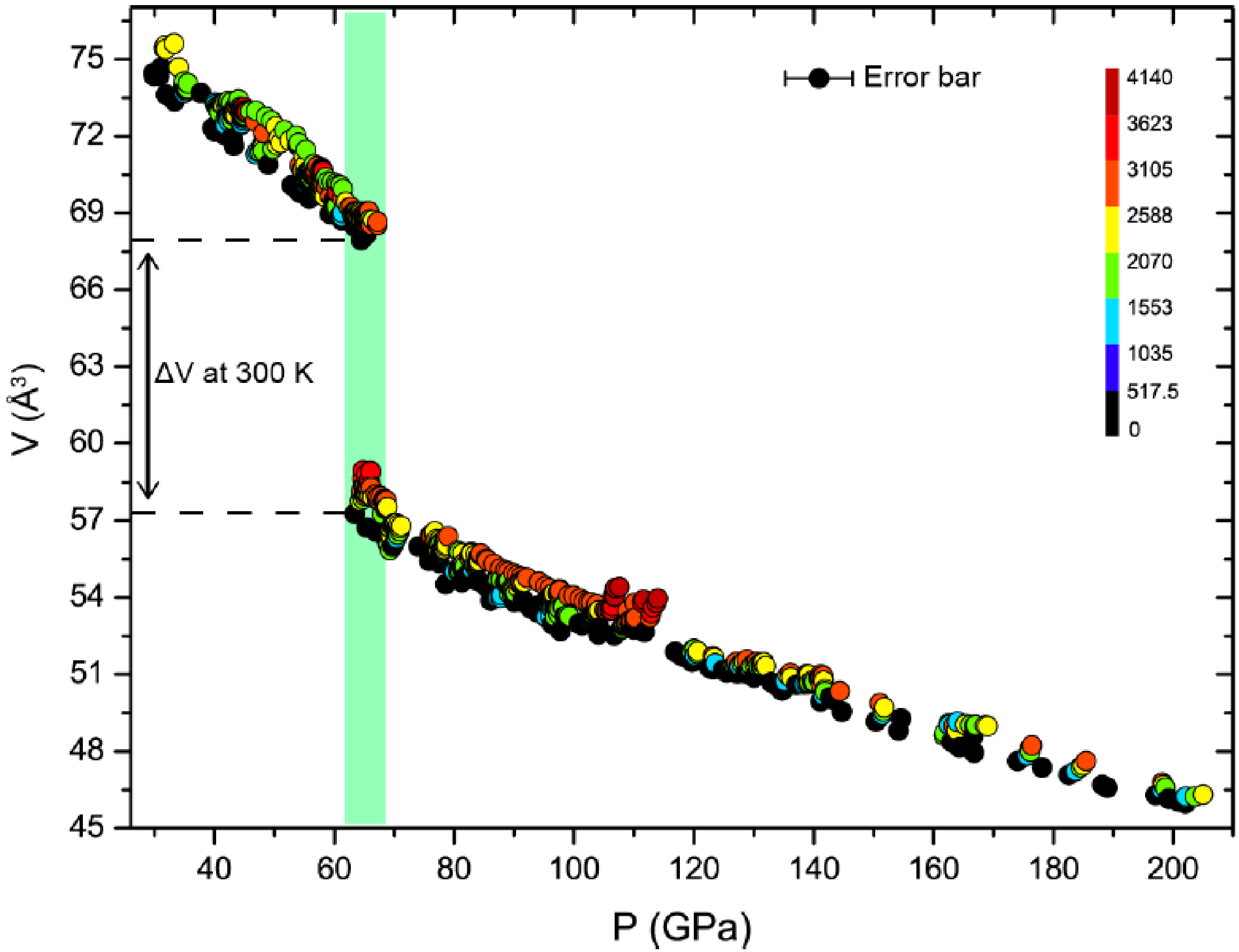
Phase transition by changing pressure at high temperature

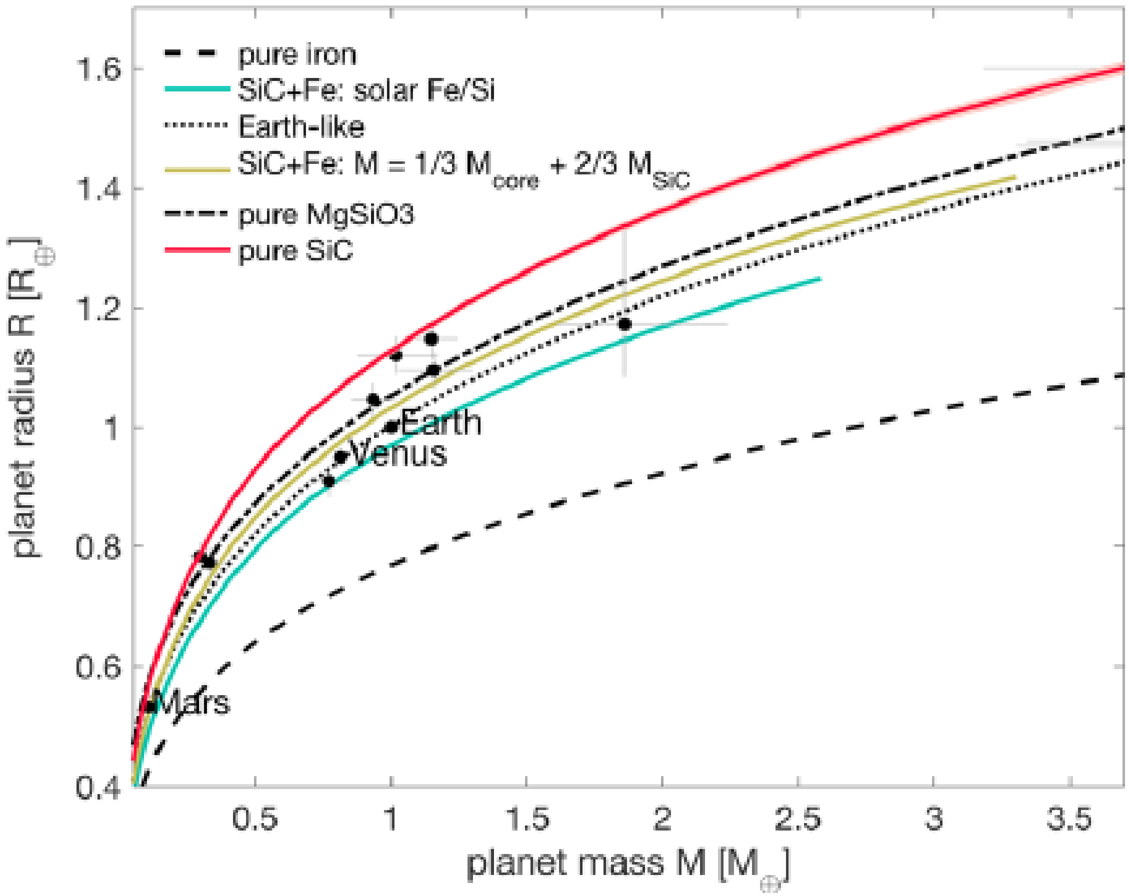


Accurate phase diagram could be then established



Change in structure is related with a large change in volume





Modeling a SiC+Fe planet

Journal of Geophysical Research: Planets

Equation of State of SiC at Extreme Conditions: New Insight Into the Interior of Carbon-Rich Exoplanets

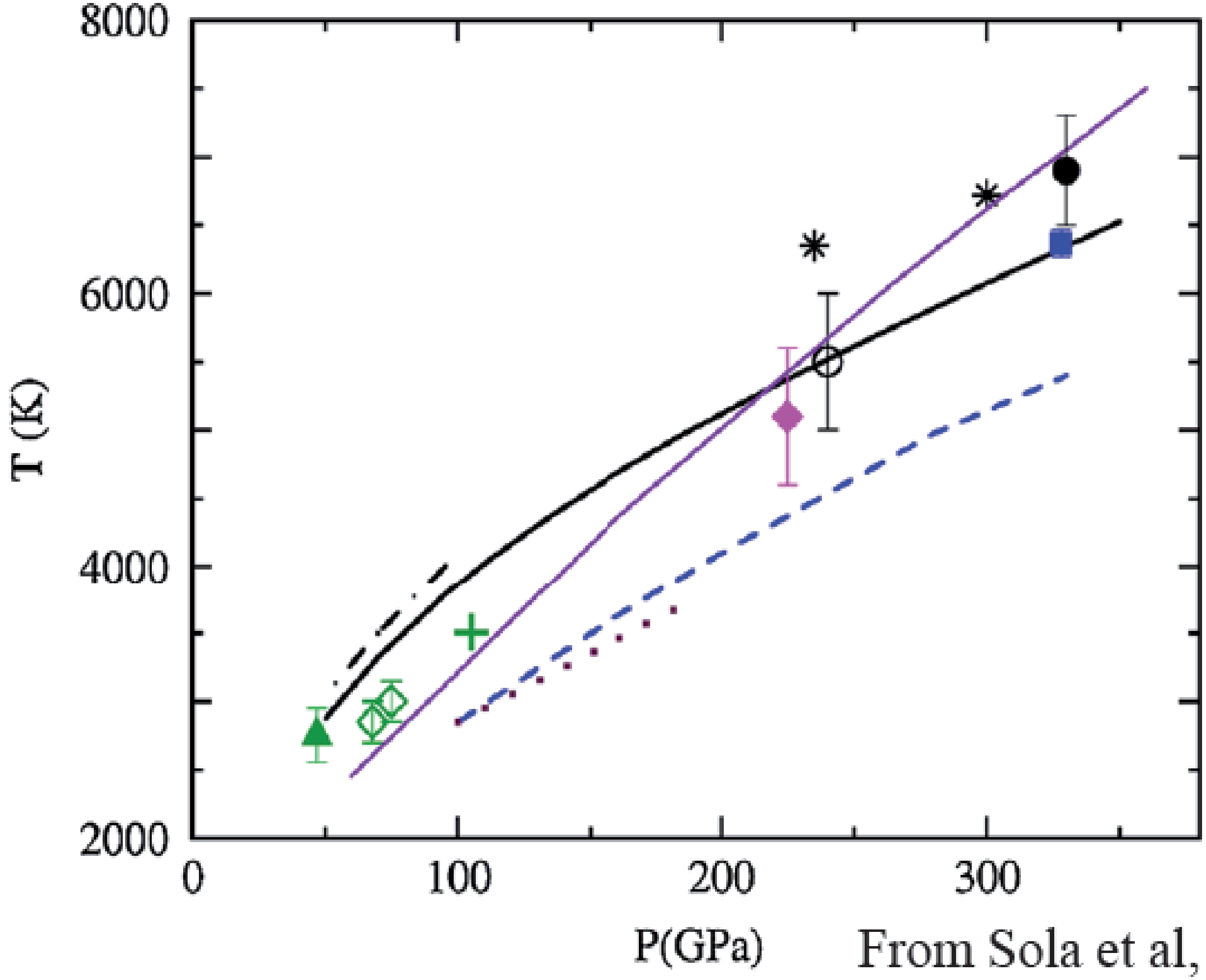
F. Miozzi[1], G. Morard[1], D. Antonangeli[1], A. N. Clark[2], M. Mezouar[3], C. Dorn[4], A. Rozel[5], and G. Fiquet[1]

2018

Melting : congruent

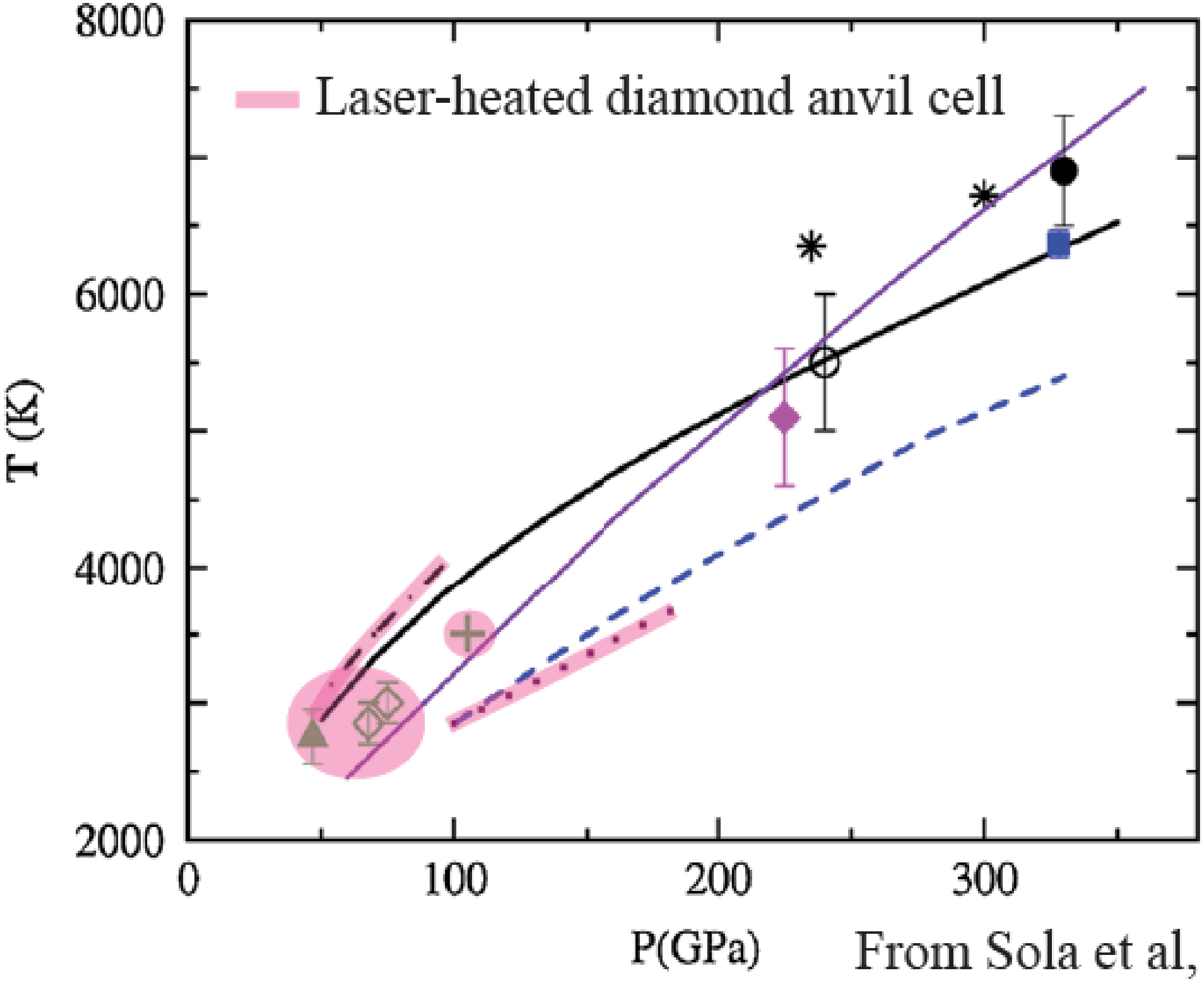
- Example of iron

Melting of pure iron: a controversial anchoring point for the geotherm



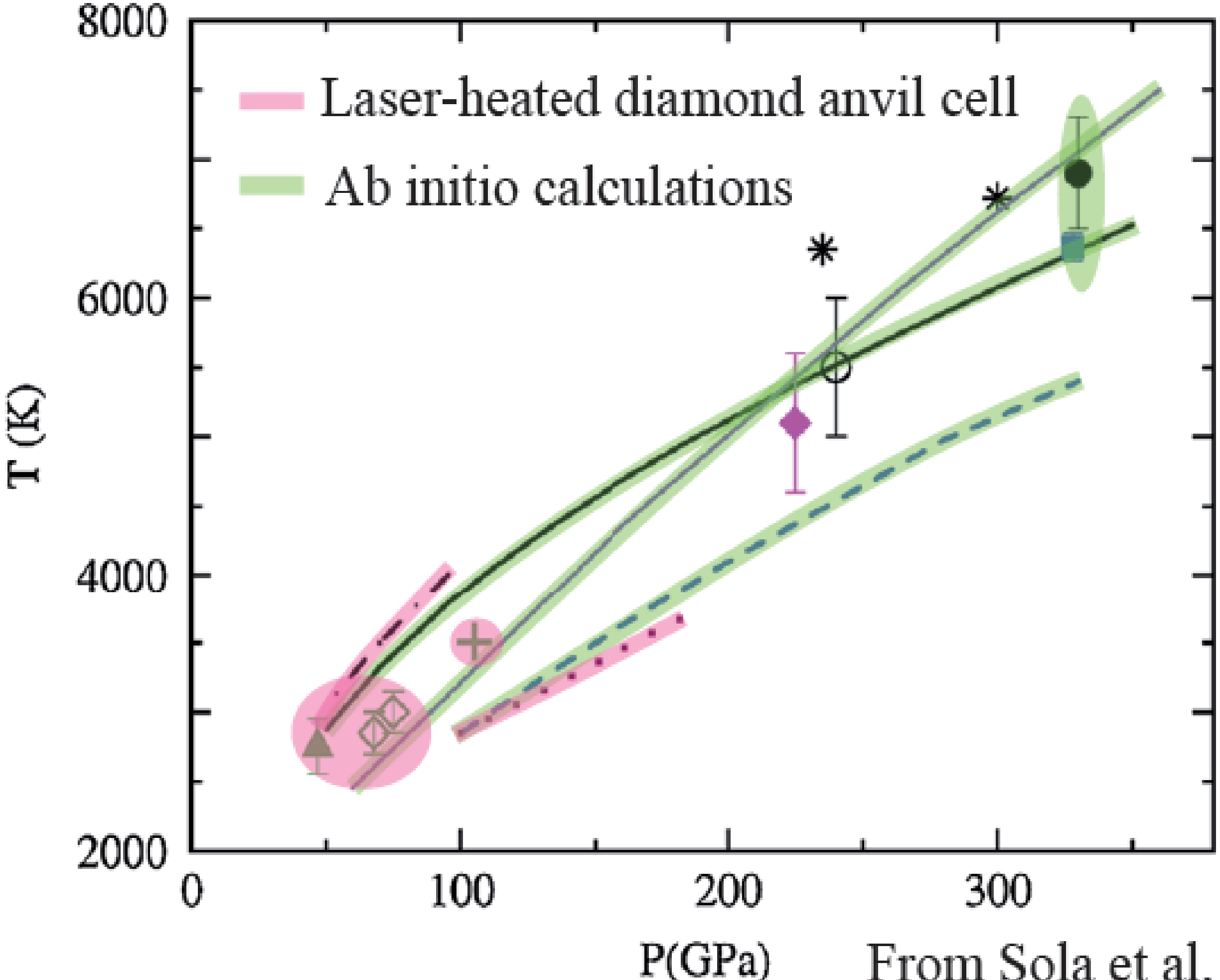
From Sola et al, 2009

Melting of pure iron: a controversial anchoring point for the geotherm



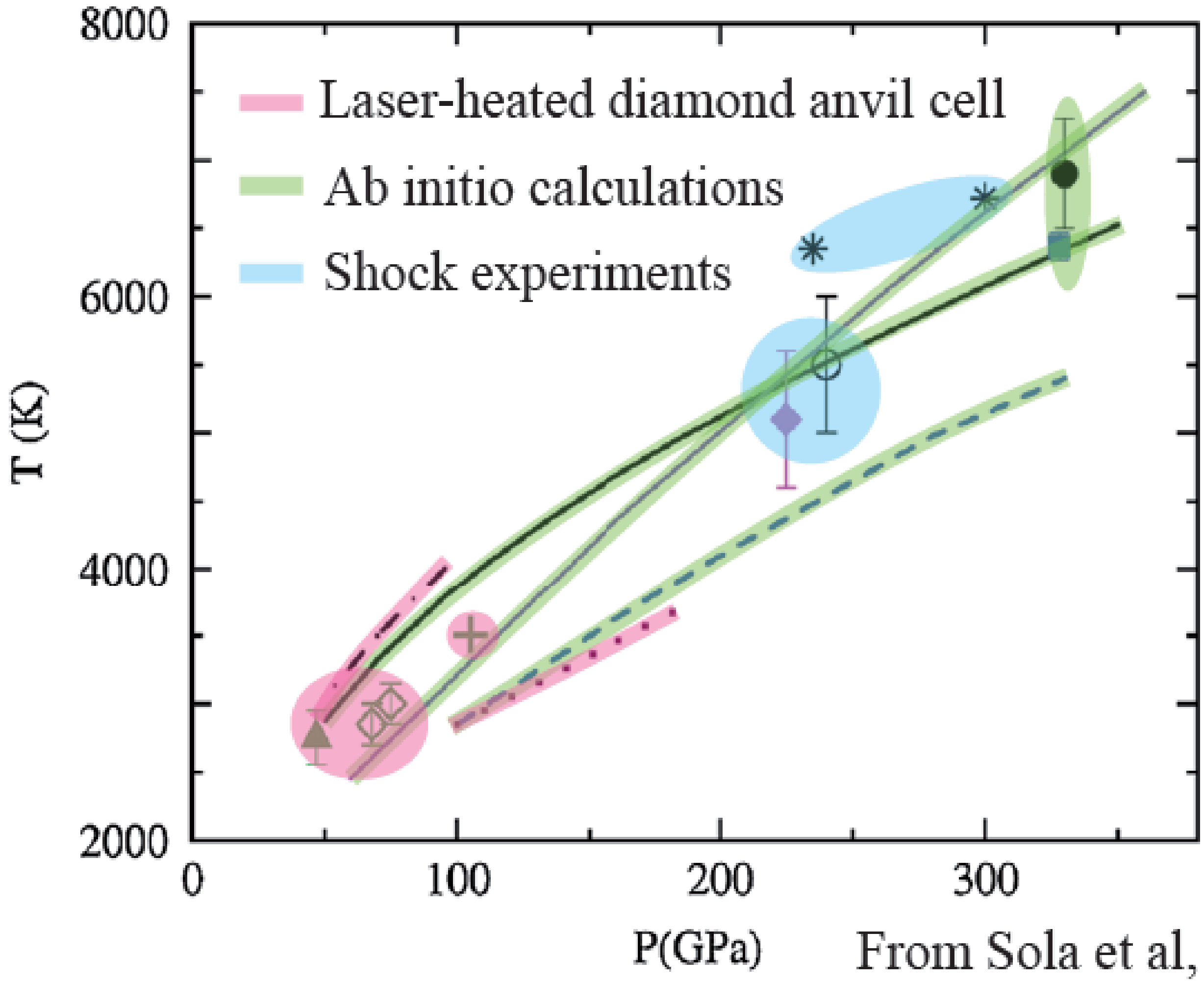
From Sola et al, 2009

Melting of pure iron: a controversial anchoring point for the geotherm



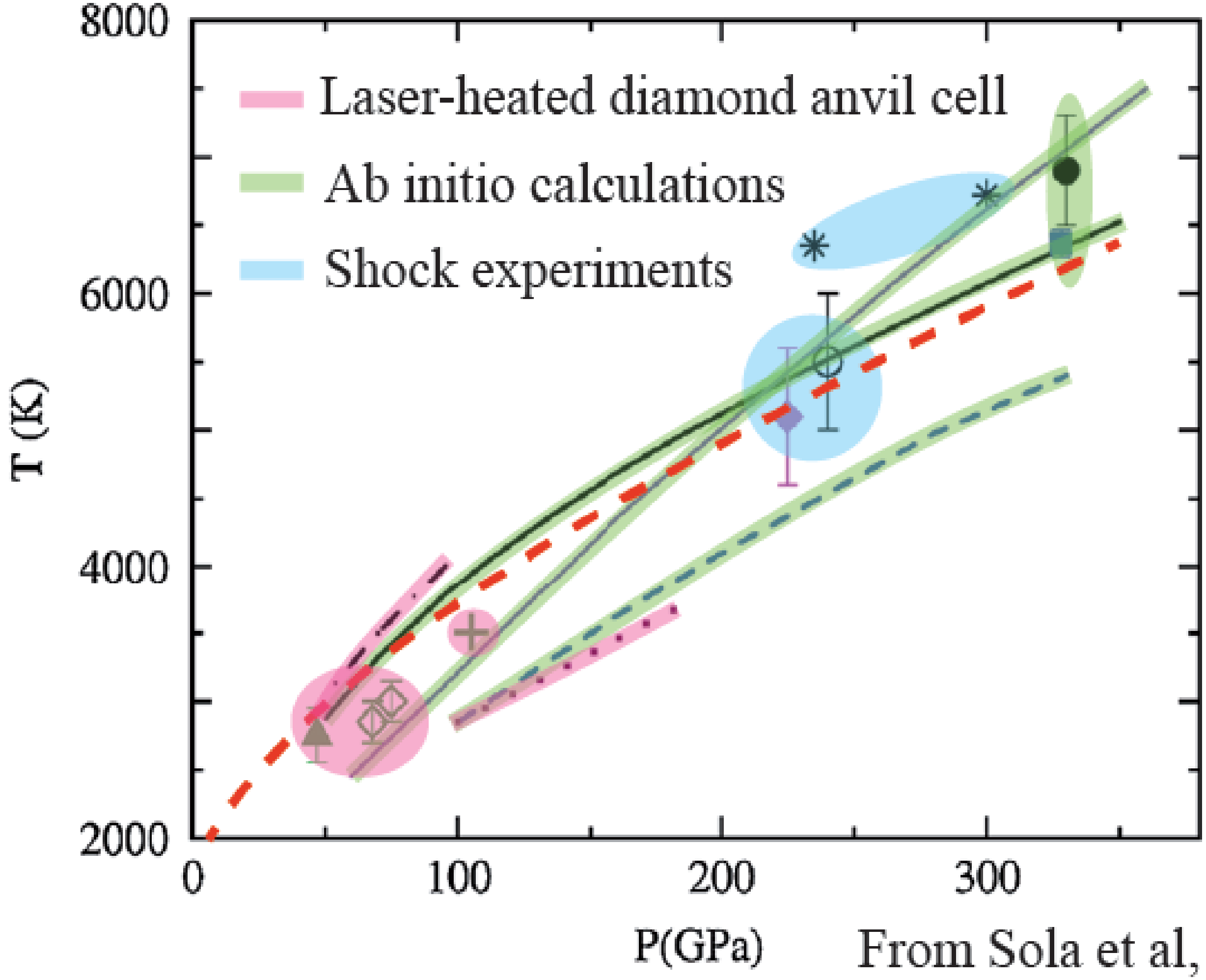
From Sola et al, 2009

Melting of pure iron: a controversial anchoring point for the geotherm



From Sola et al, 2009

Melting of pure iron: a controversial anchoring point for the geotherm



From Sola et al, 2009

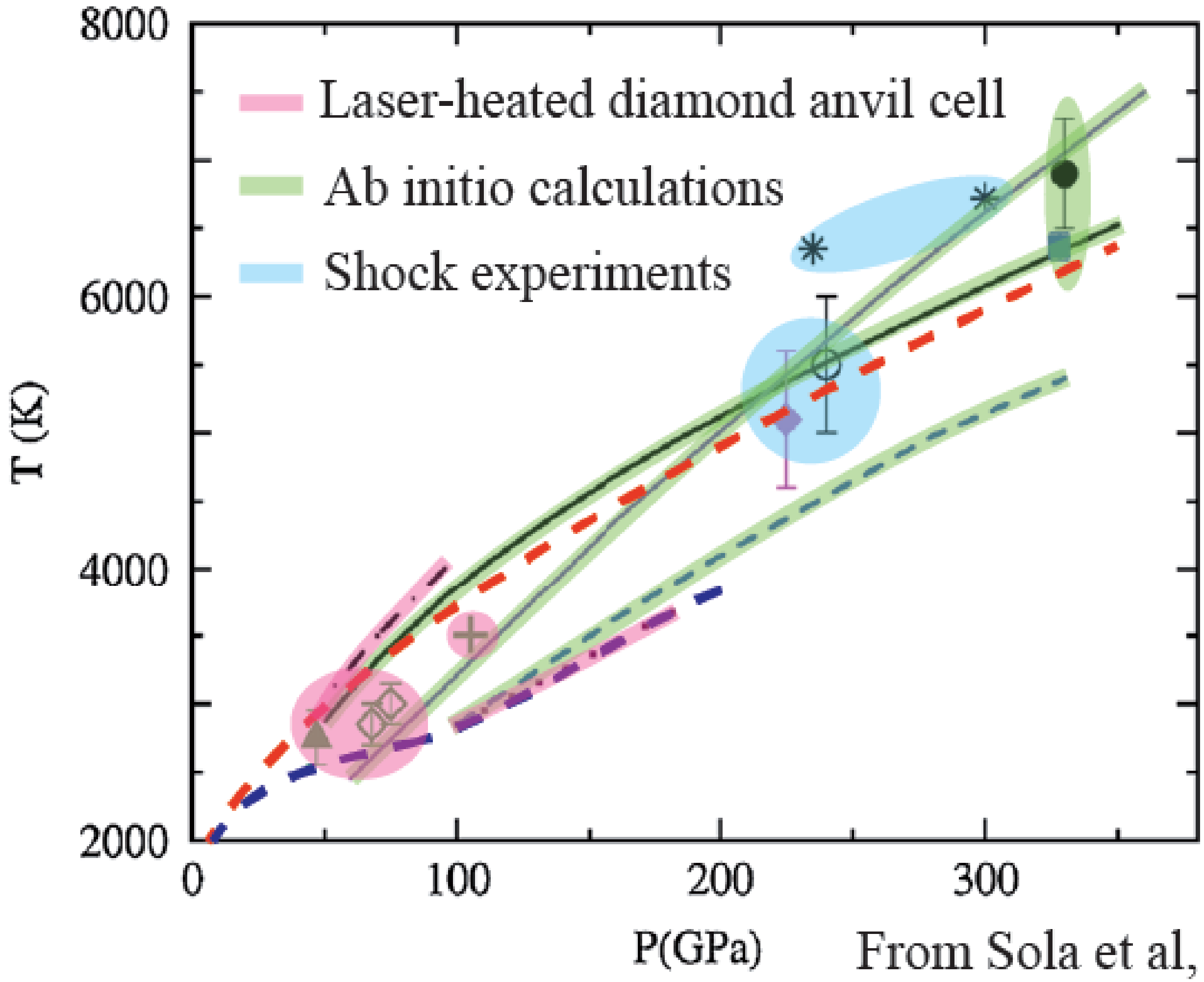
Anzellini et al, Science, 2013

In situ XRD study

Diffuse scattering

Reconciling ab initio calculations, static and dynamic compression

Melting of pure iron: a controversial anchoring point for the geotherm



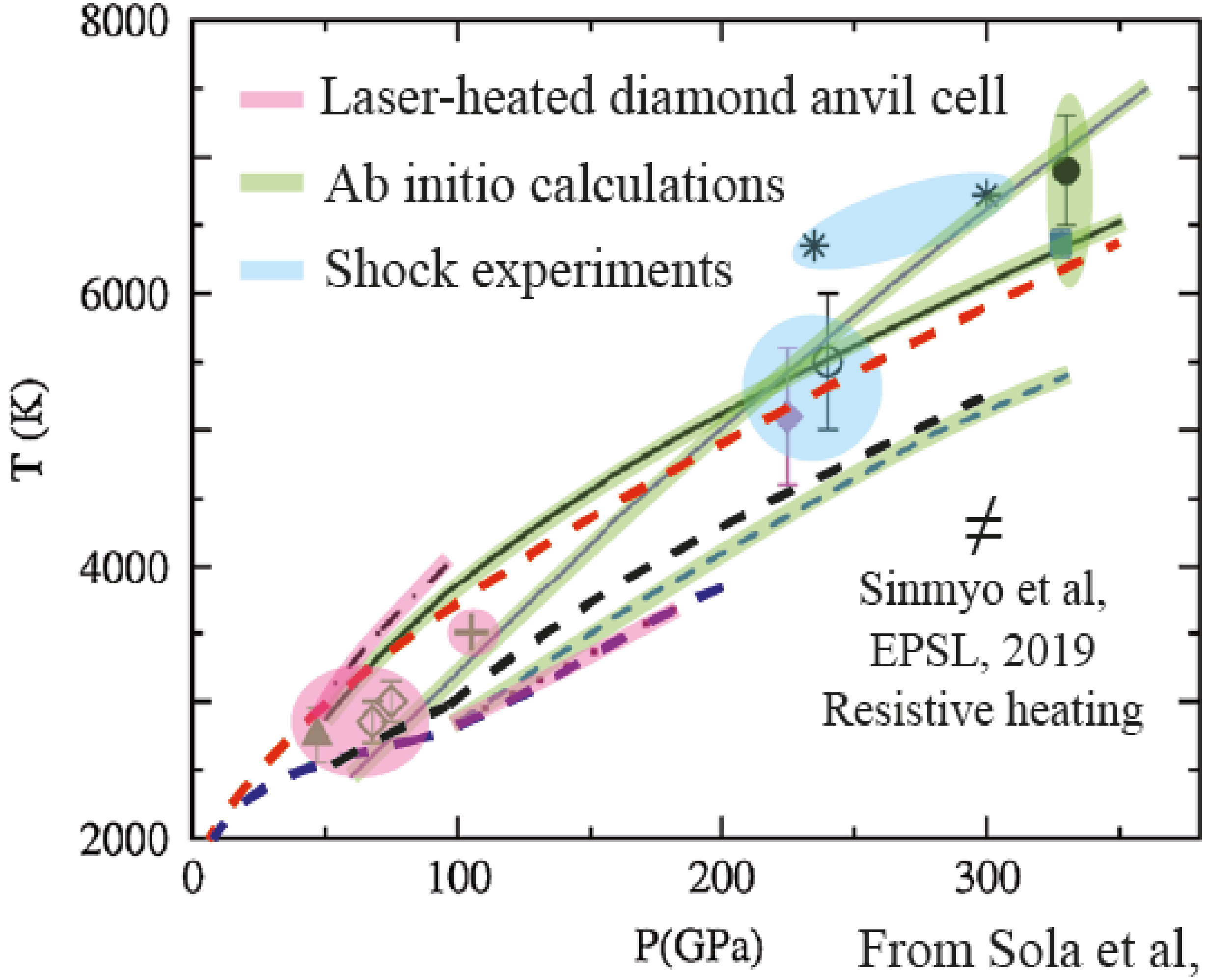
From Sola et al, 2009

Anzellini et al, Science, 2013
In situ XRD study
Diffuse scattering
Reconciling ab initio calculations, static and dynamic compression

≠

Aquilanti et al, PNAS, 2015
In situ XANES study
Back on Boehler , 1993

Melting of pure iron: a controversial anchoring point for the geotherm



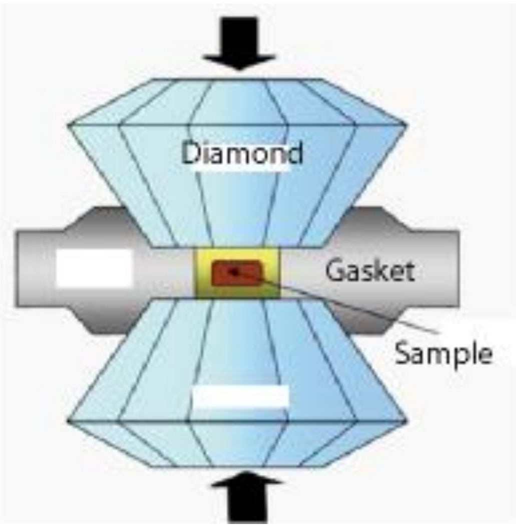
From Sola et al, 2009

Anzellini et al,
Science, 2013
In situ XRD study
Diffuse scattering
Reconciling ab initio calculations, static and dynamic compression

≠

Aquilanti et al,
PNAS, 2015
In situ XANES study
Back on Boehler , 1993

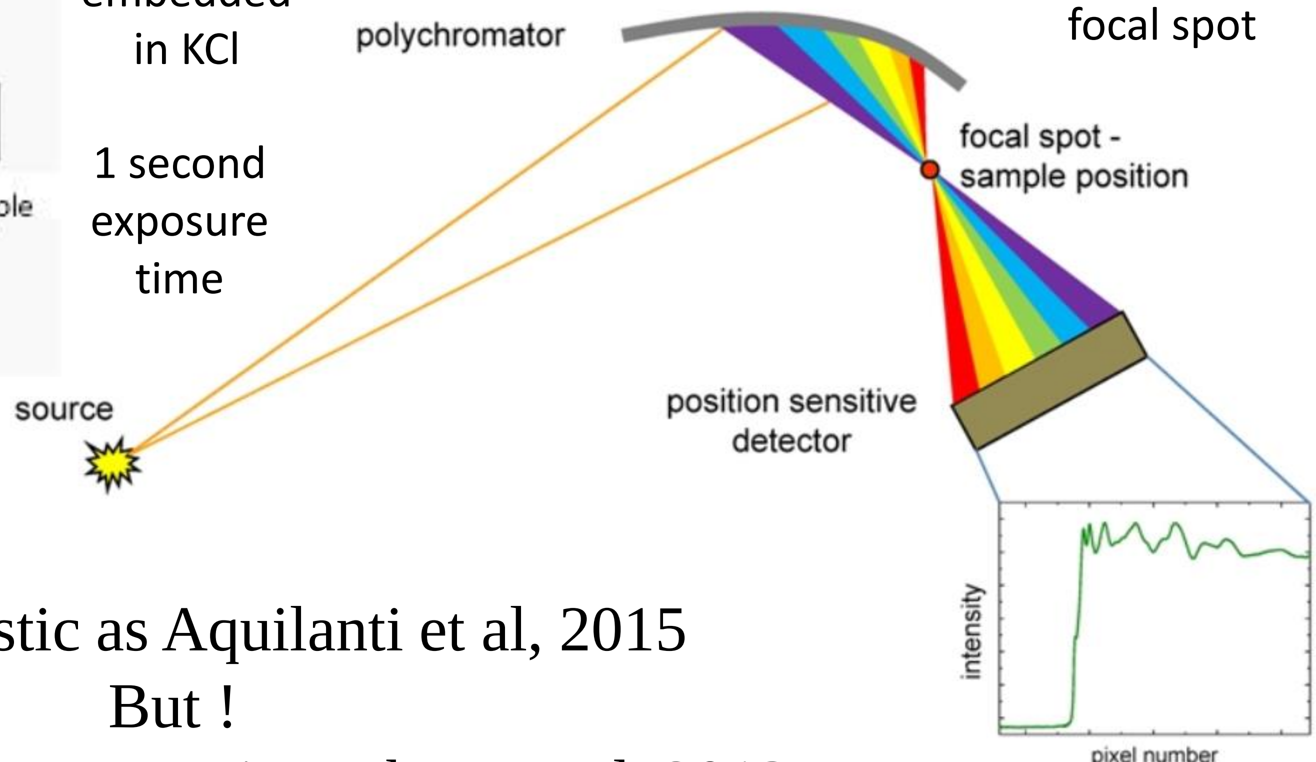
Energy dispersive EXAFS experimental set-up coupled with Laser-Heated Diamond Anvil Cell on ID24 beamline, ESRF



Fe foil embedded in KCl

1 second exposure time

6*4 µm focal spot



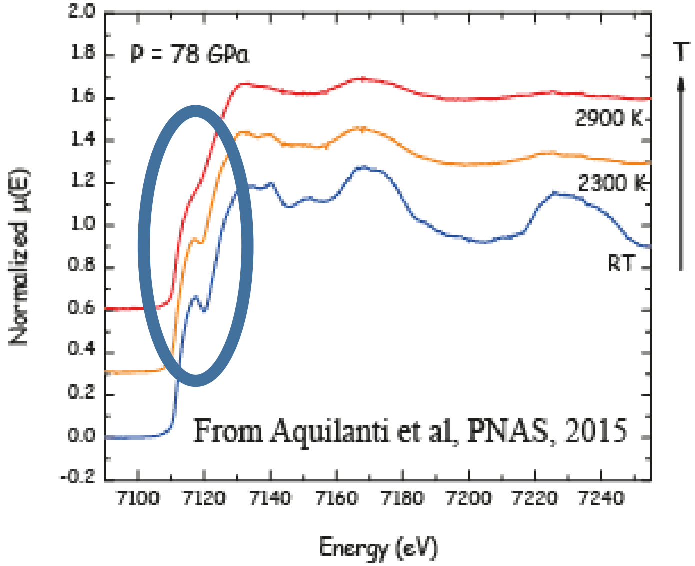
Same diagnostic as Aquilanti et al, 2015

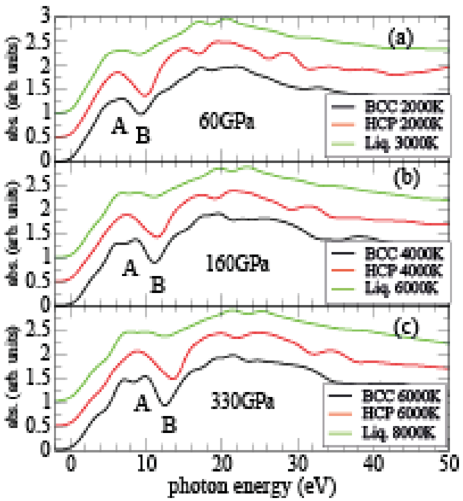
But !

Same sample geometry as in Jackson et al, 2013 and Anzellini et al, 2013

From DIAMOND synchrotron website

In situ criteria for XANES experiments

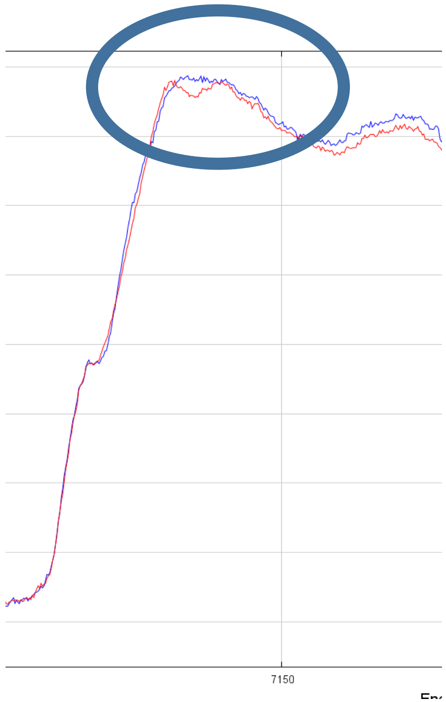




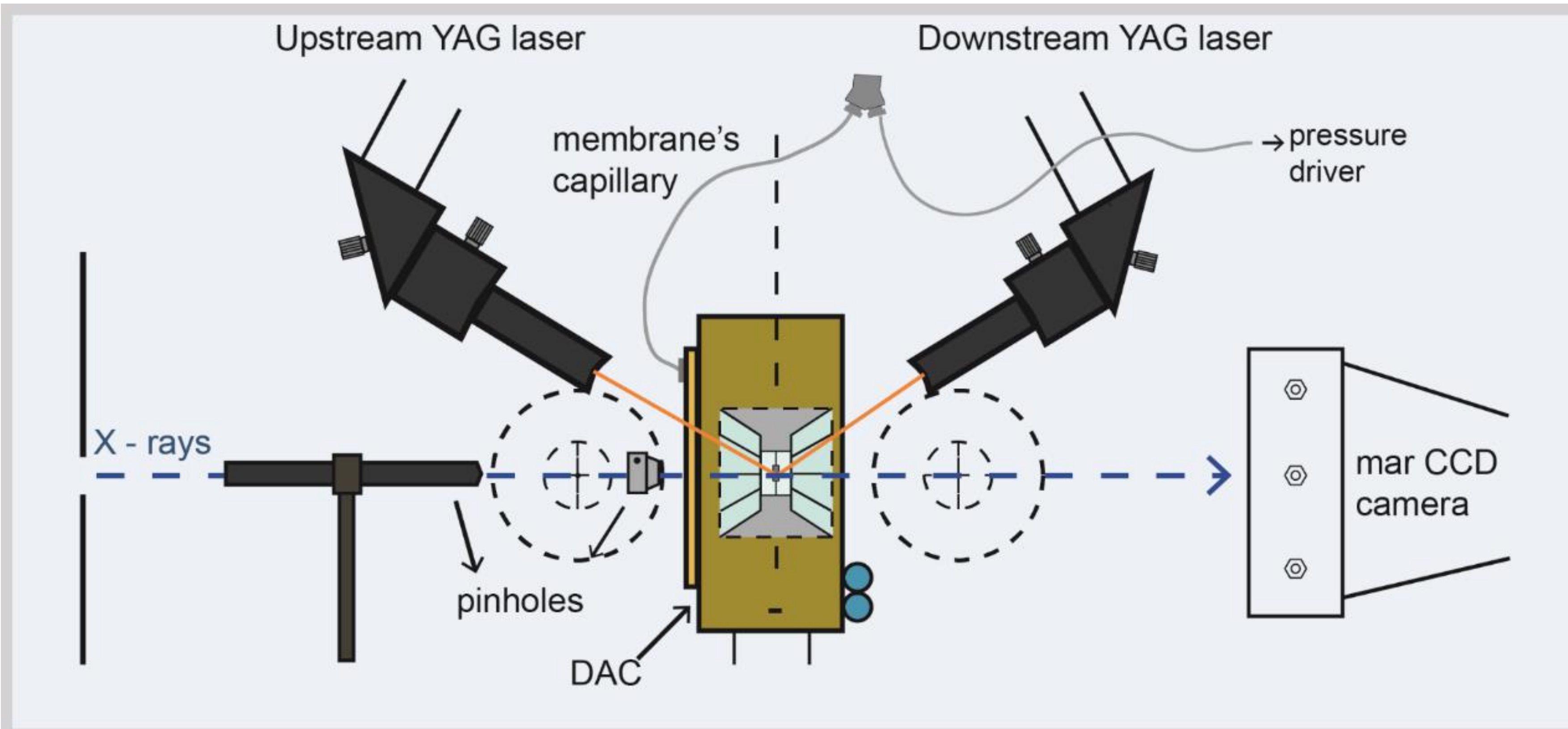
From Mazevet et al, Phys. Rev B, 2014

Change of the edge upon melting

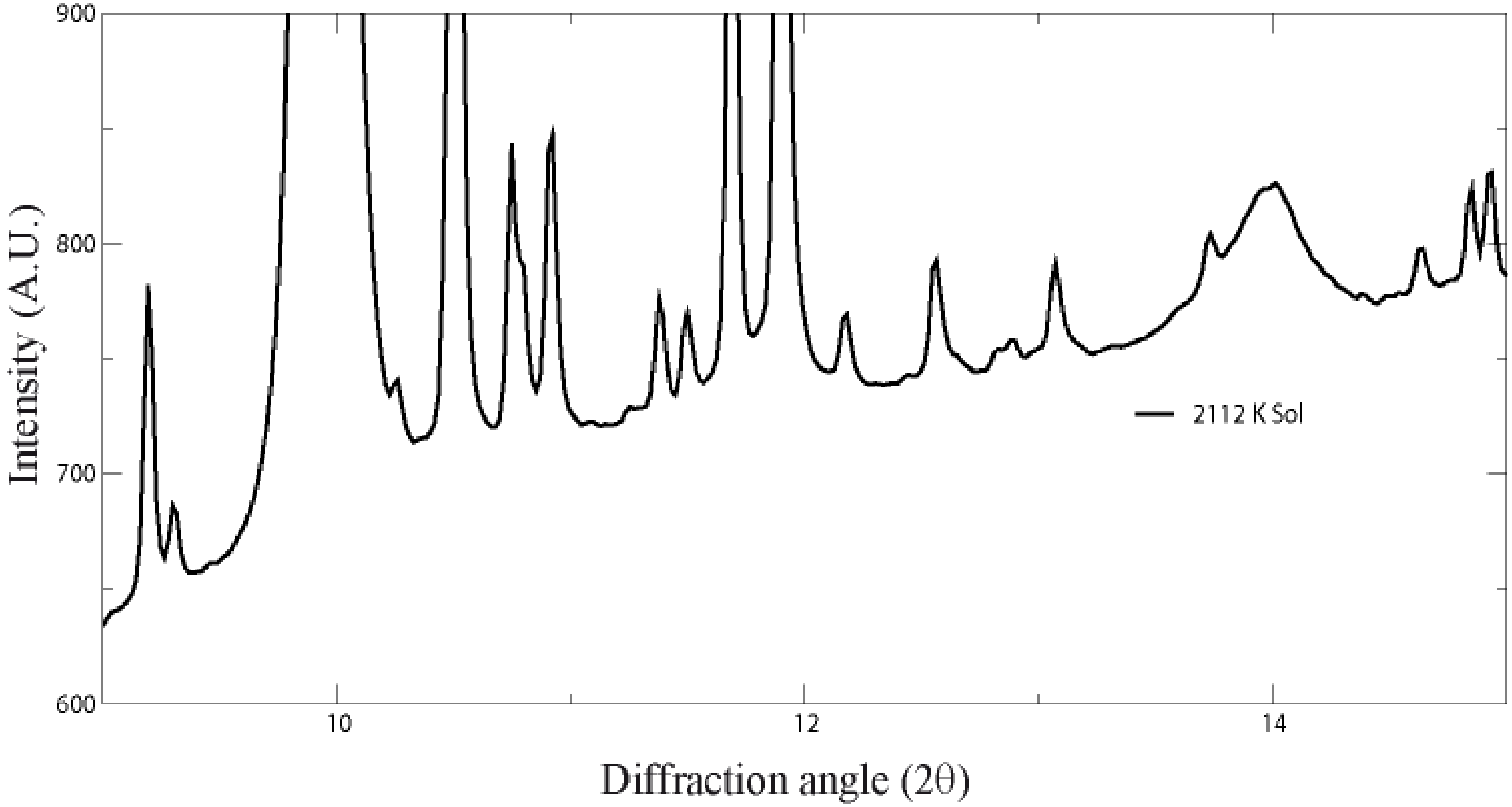
hcp-fcc transition



In situ X-ray diffraction on ID27

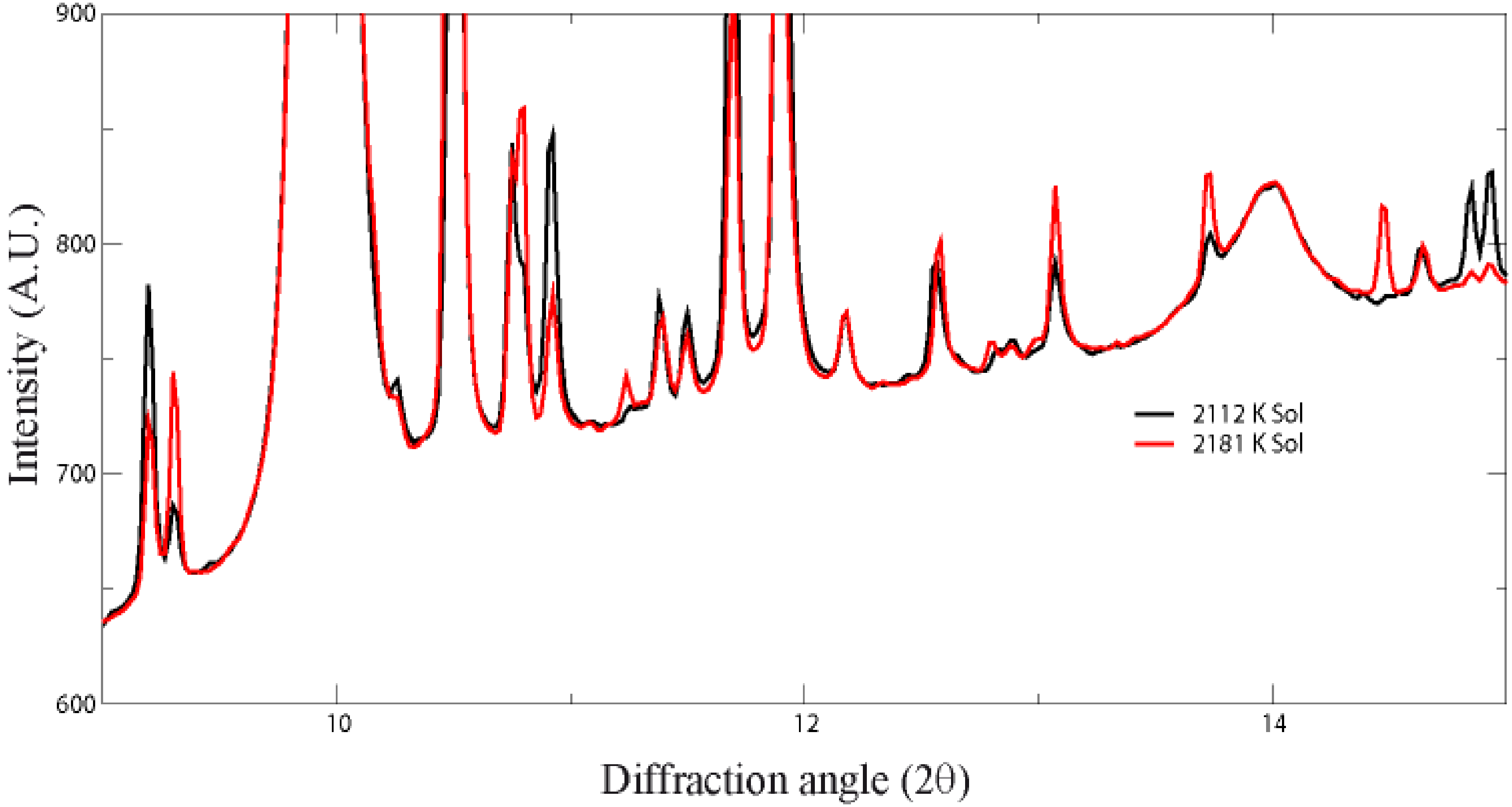


In situ detection of melting in LHDAC



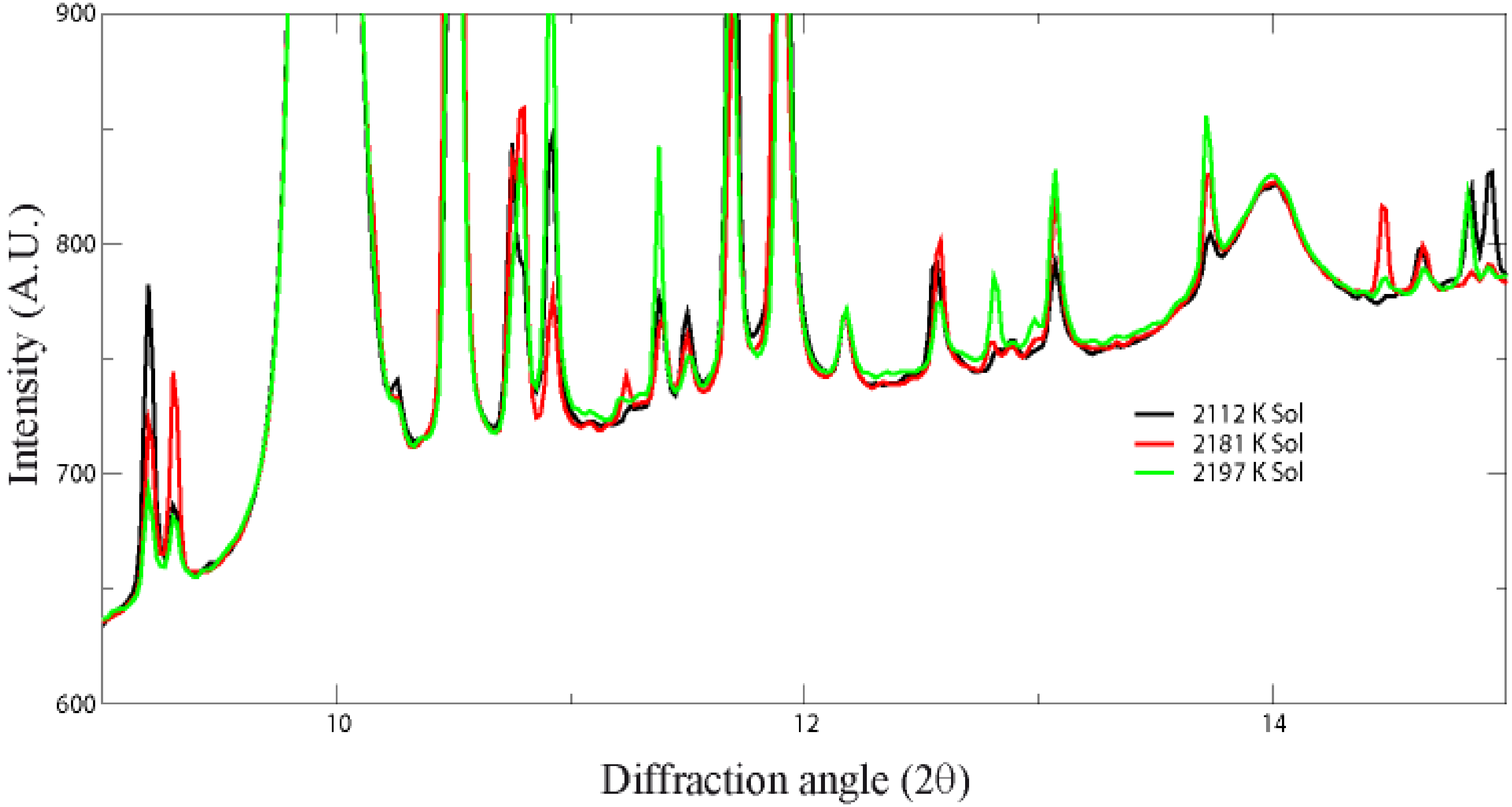
Fe-5wt% Ni-12wt% S ; P~67 GPa

In situ detection of melting in LHDAC



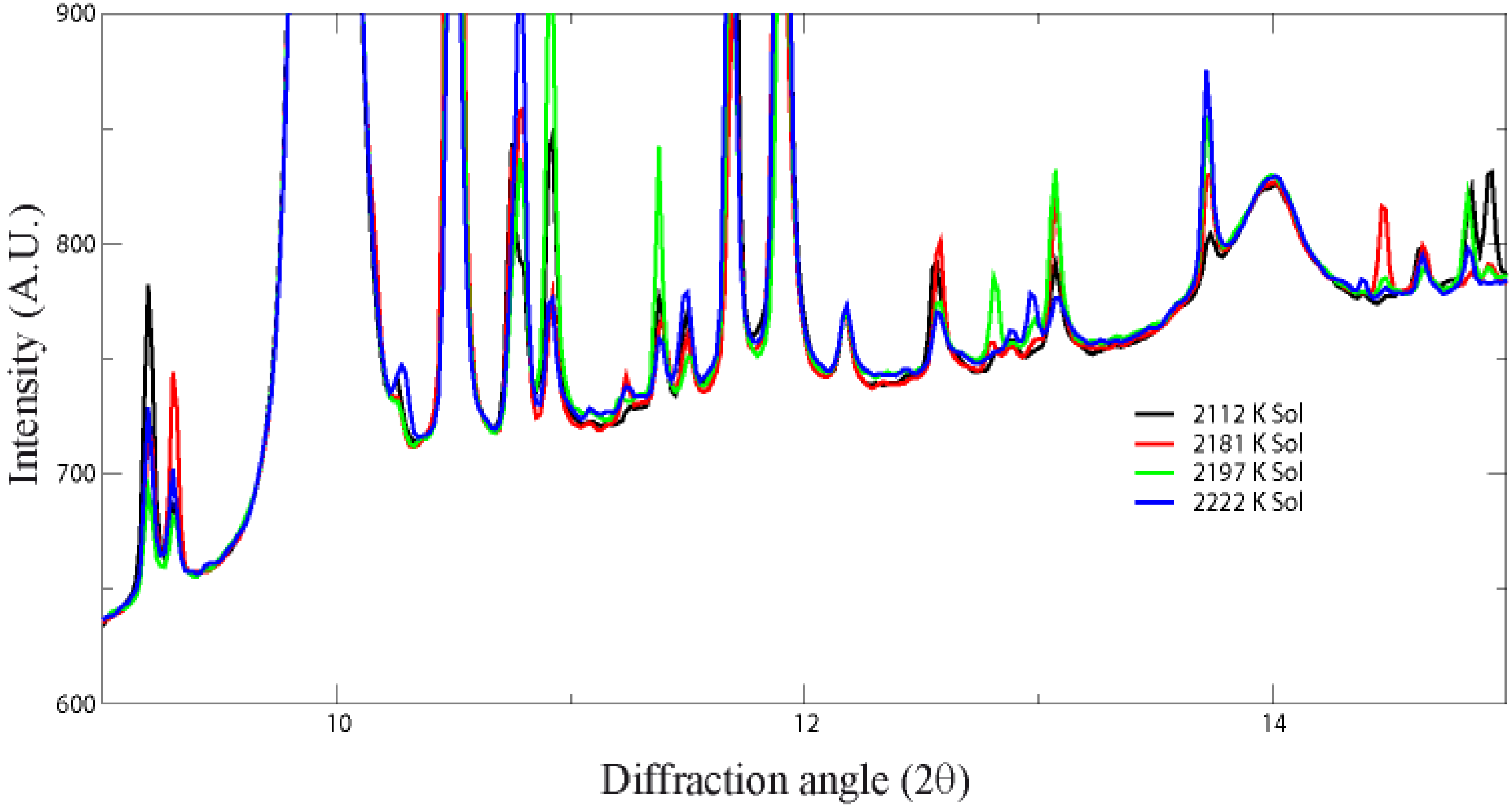
Fe-5wt% Ni-12wt% S ; P~67 GPa

In situ detection of melting in LHDAC



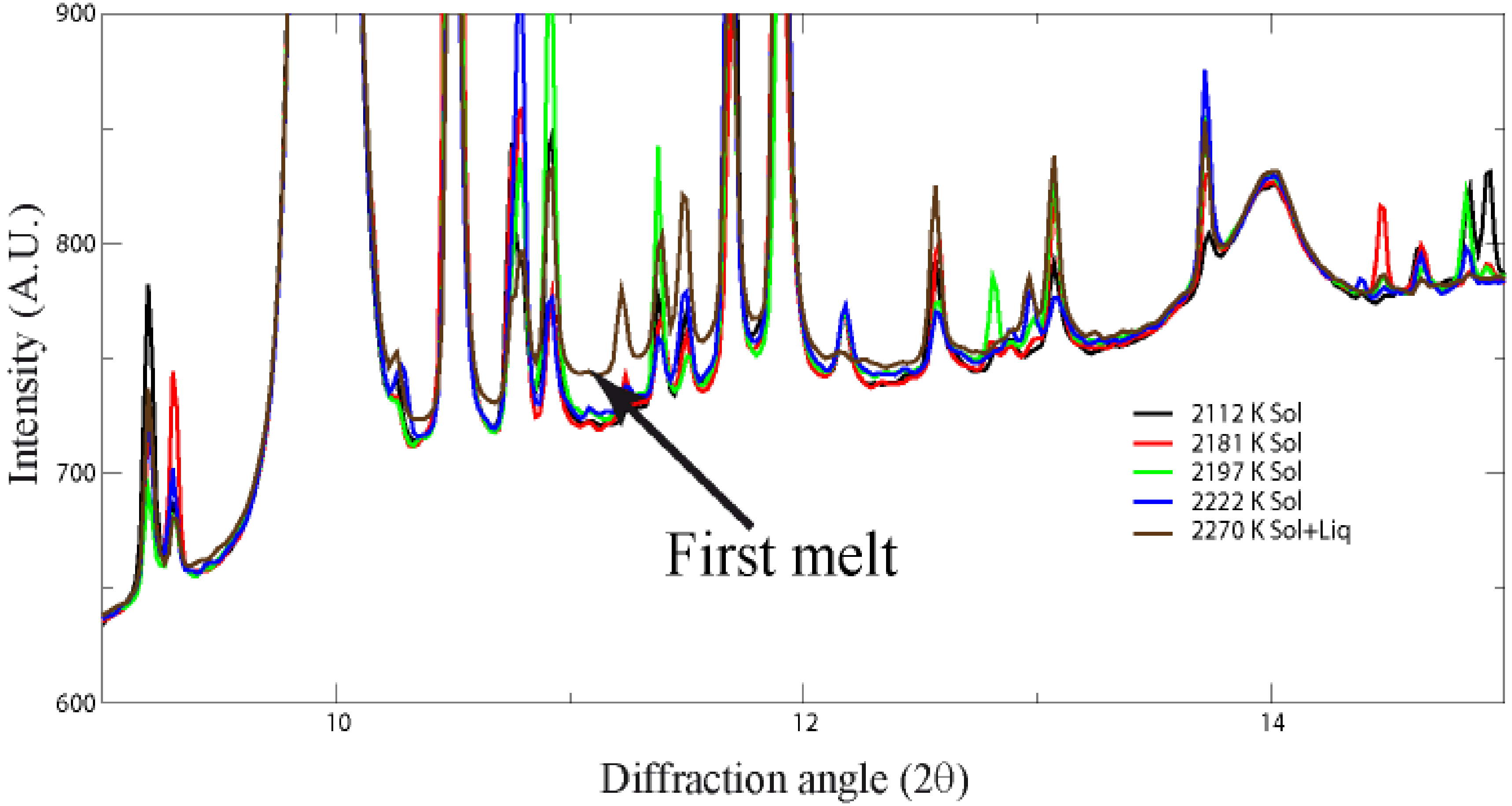
Fe-5wt% Ni-12wt% S ; P~67 GPa

In situ detection of melting in LHDAC



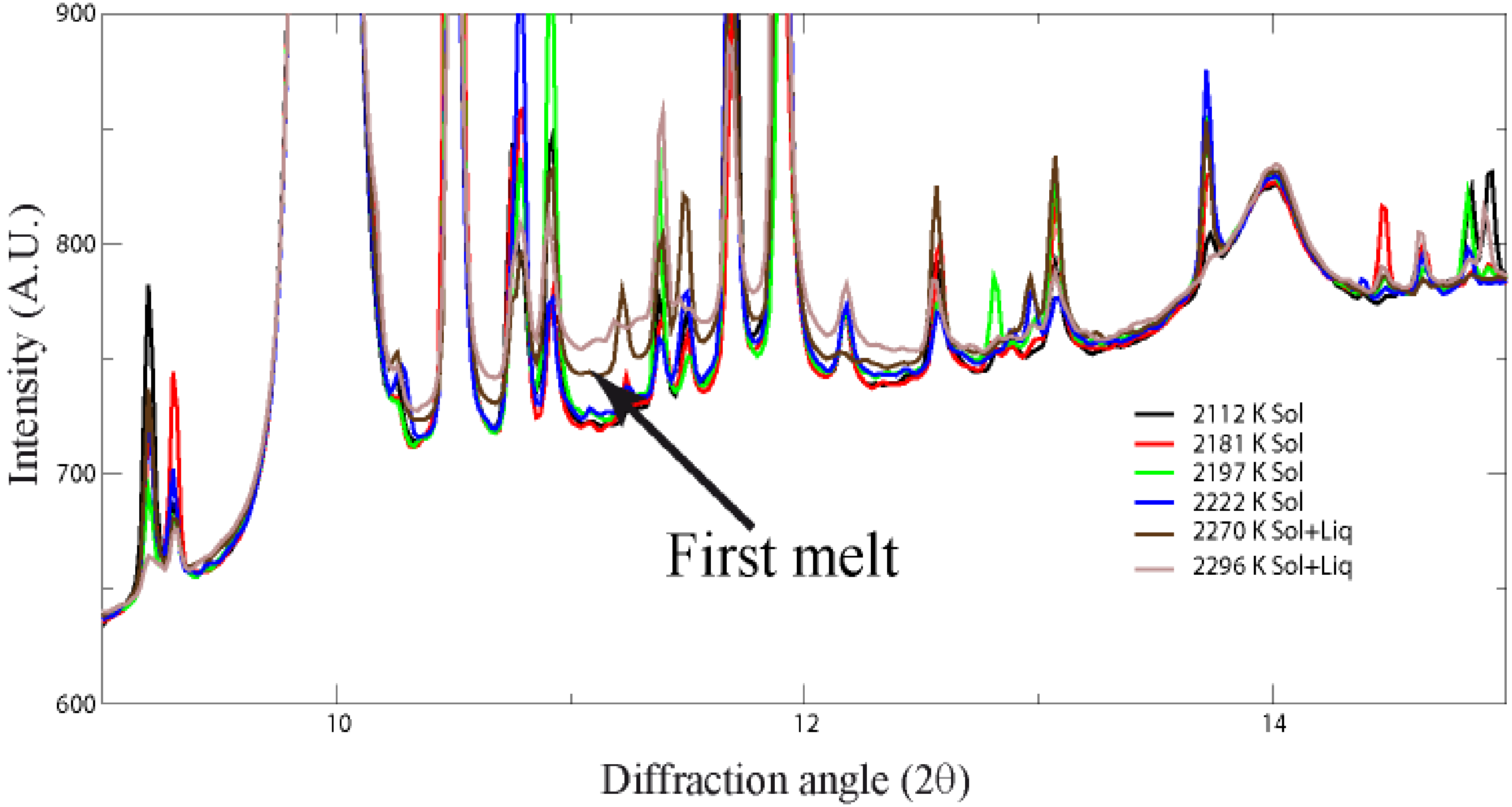
Fe-5wt% Ni-12wt% S ; P~67 GPa

In situ detection of melting in LHDAC



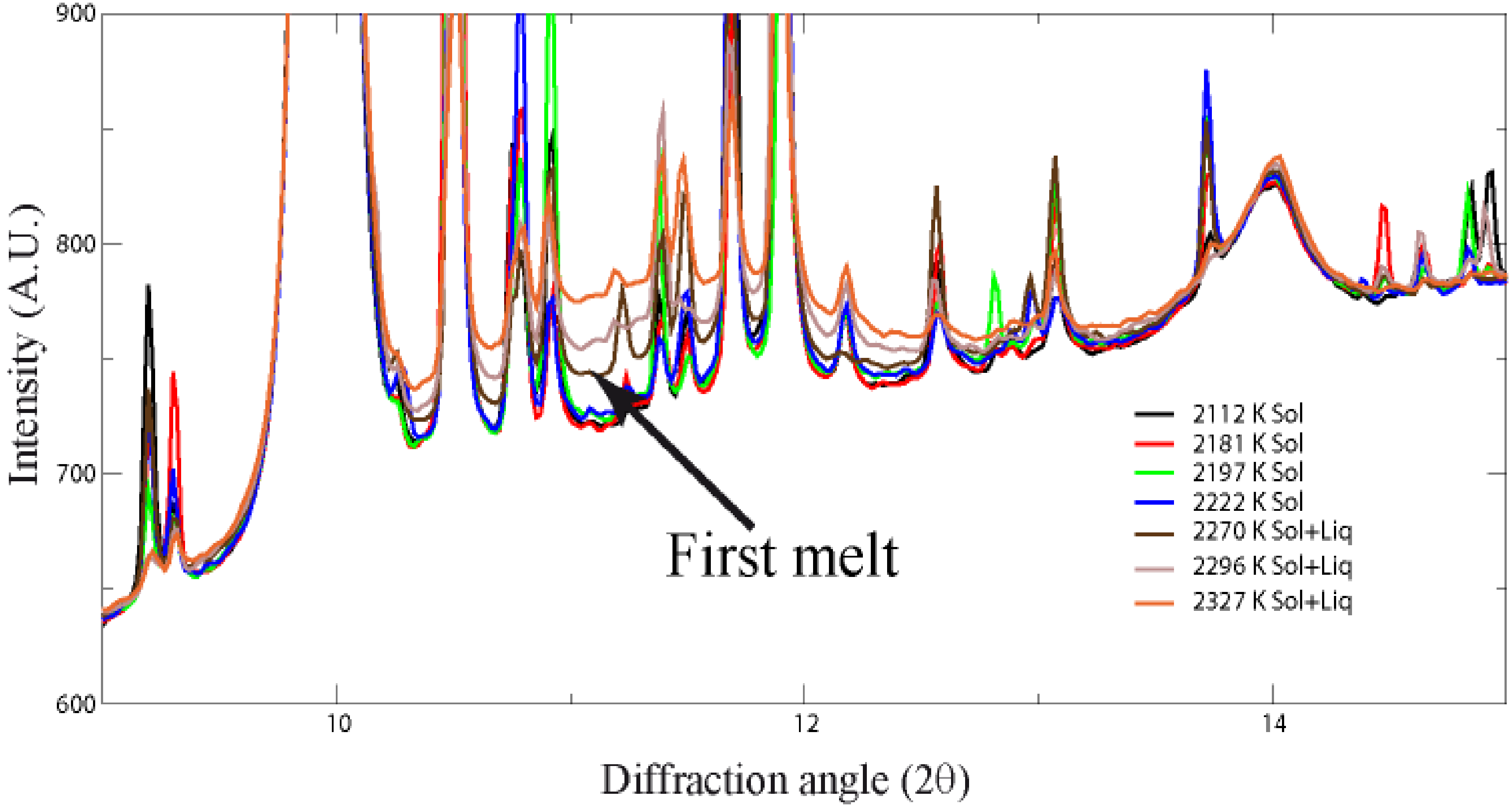
Fe-5wt% Ni-12wt% S ; P~67 GPa

In situ detection of melting in LHDAC



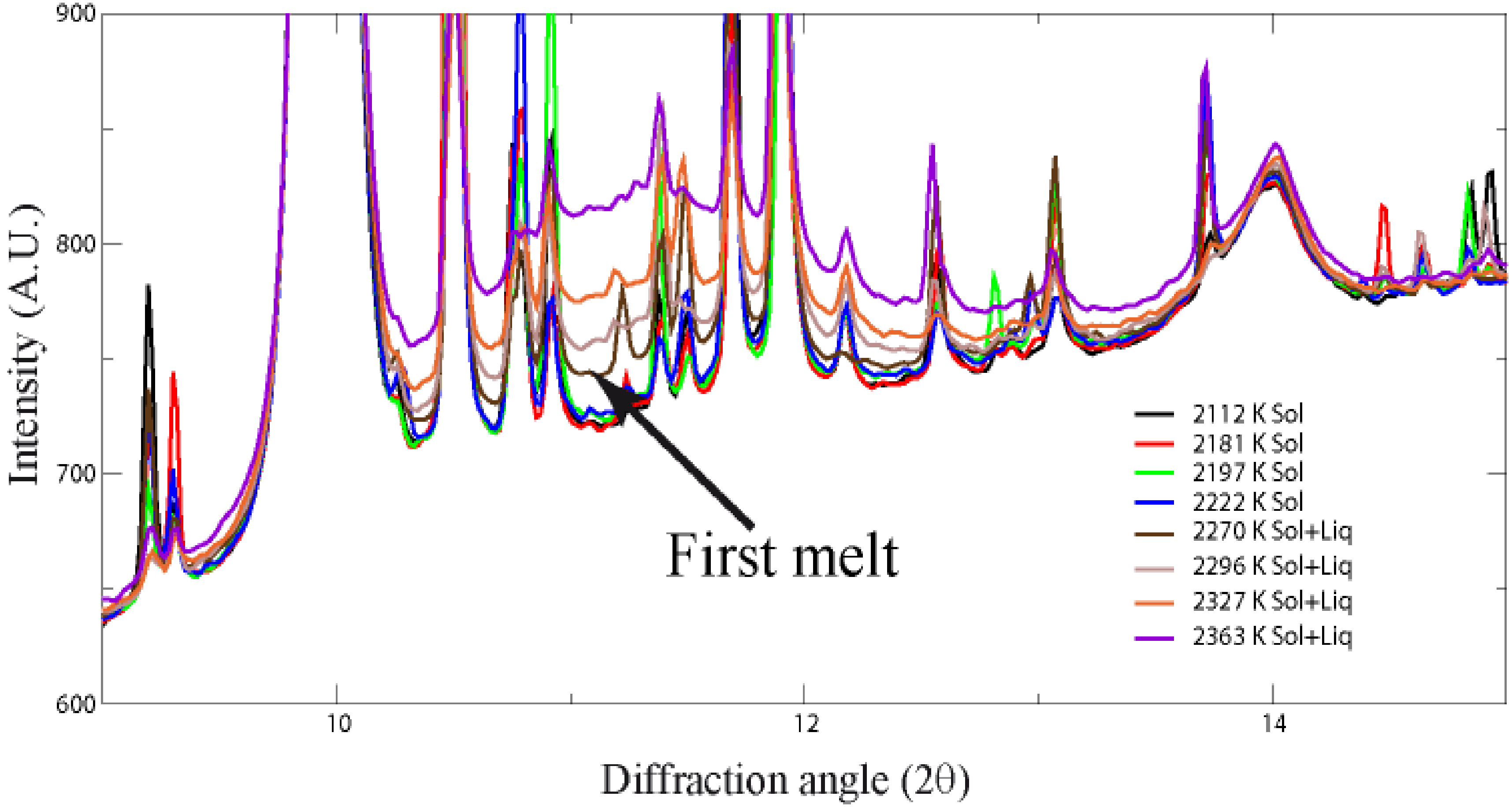
Fe-5wt% Ni-12wt% S ; P~67 GPa

In situ detection of melting in LHDAC



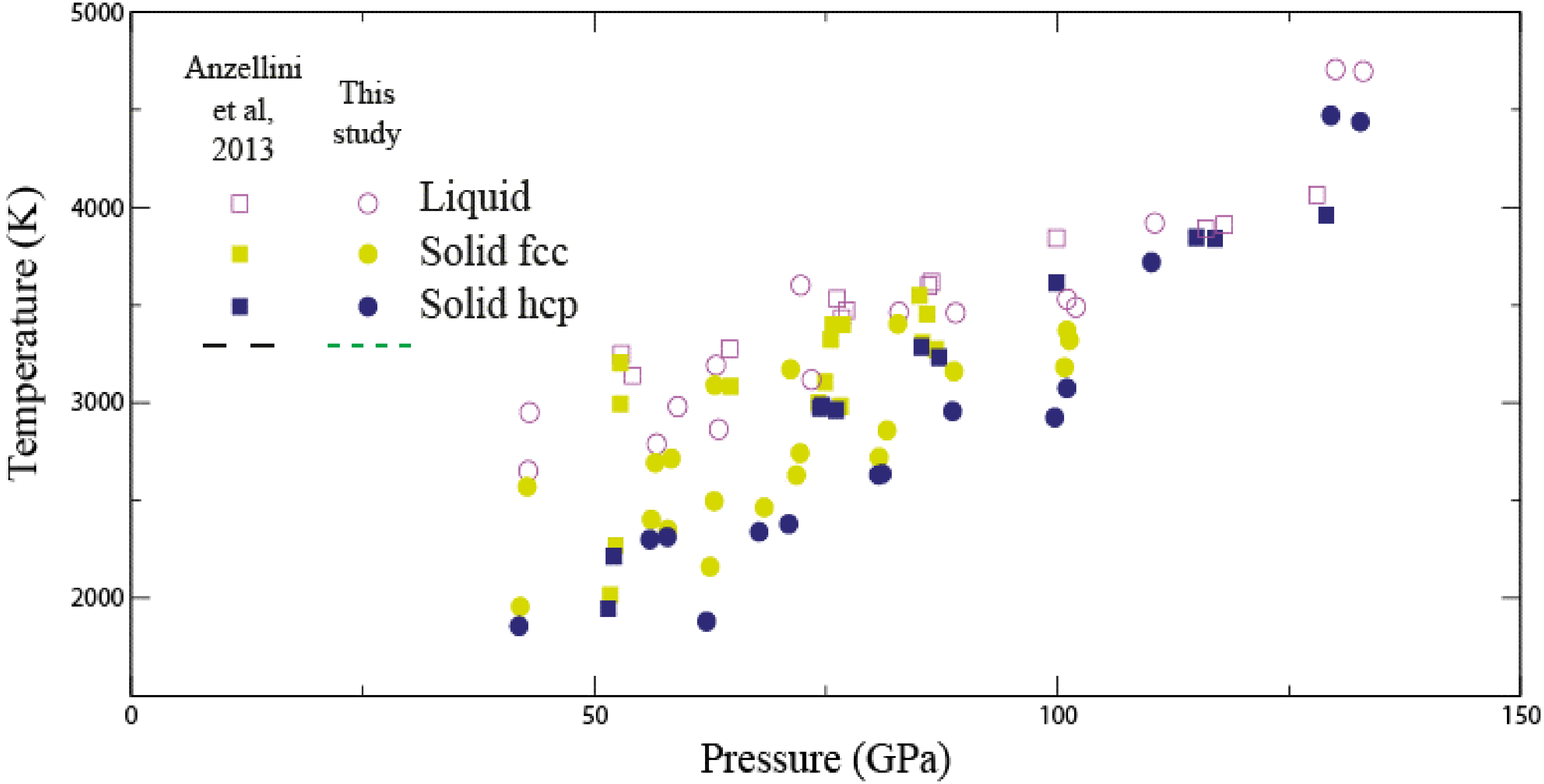
Fe-5wt% Ni-12wt% S ; P~67 GPa

In situ detection of melting in LHDAC

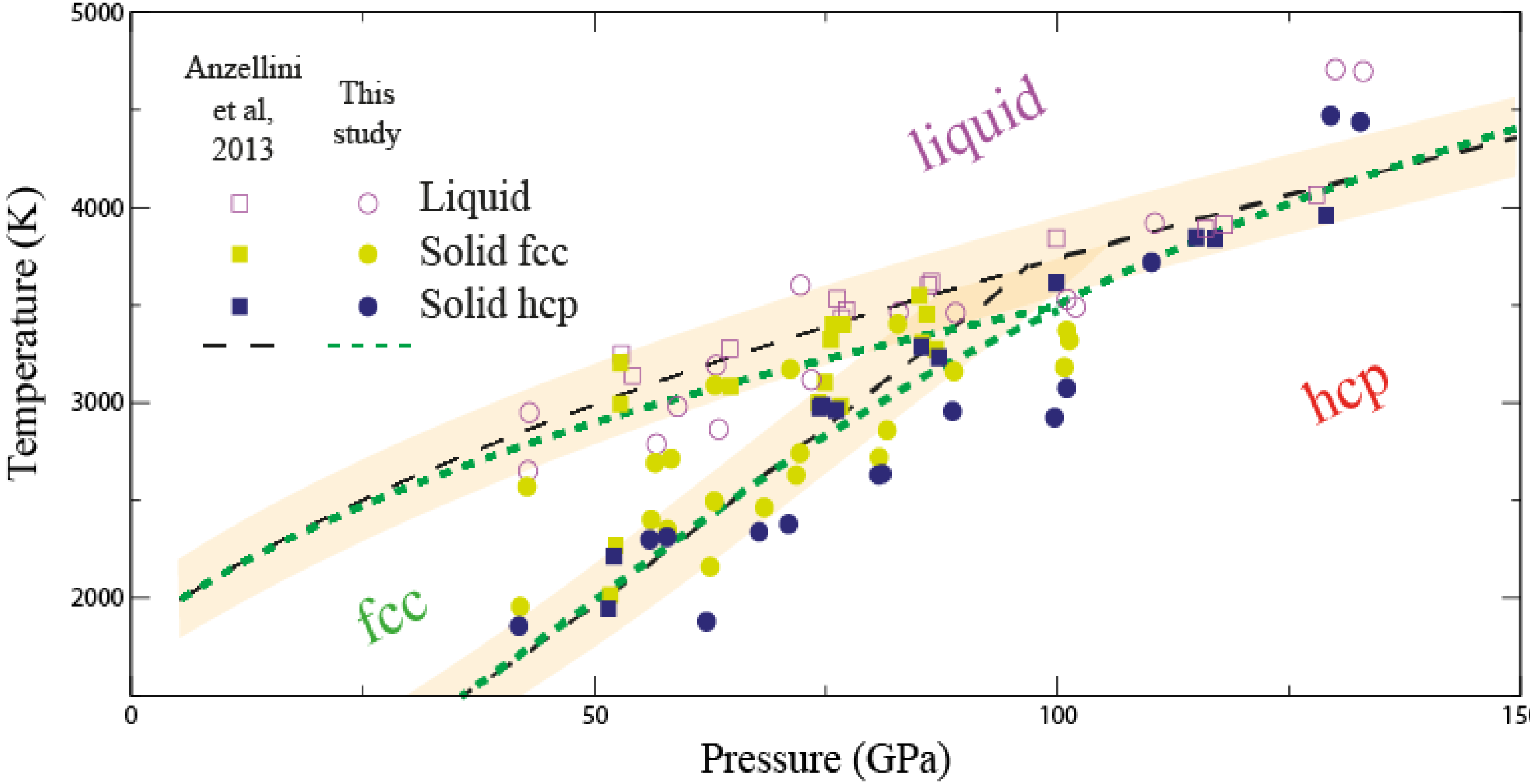


Fe-5wt% Ni-12wt% S ; P~67 GPa

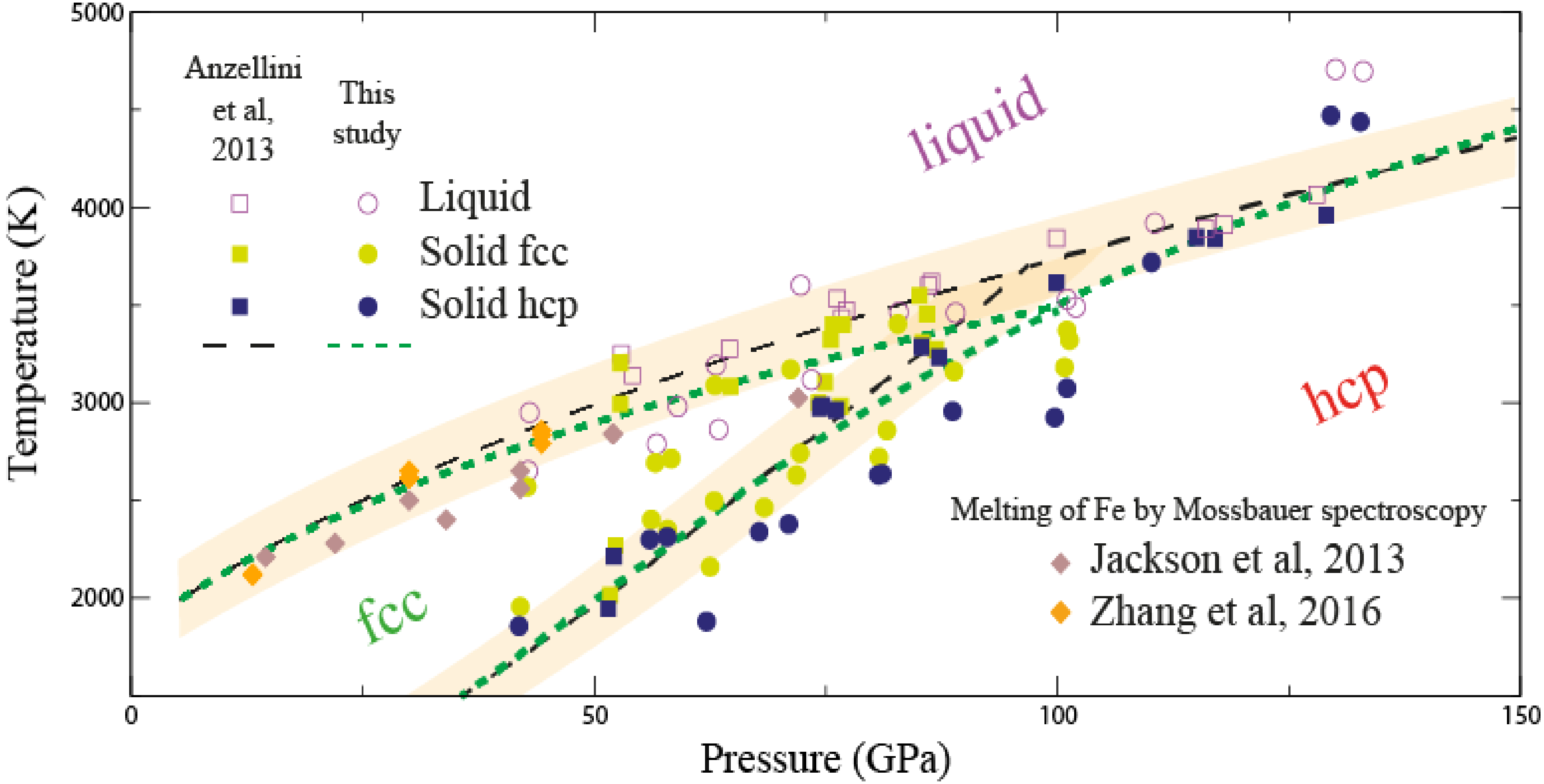
Comparison with previous studies



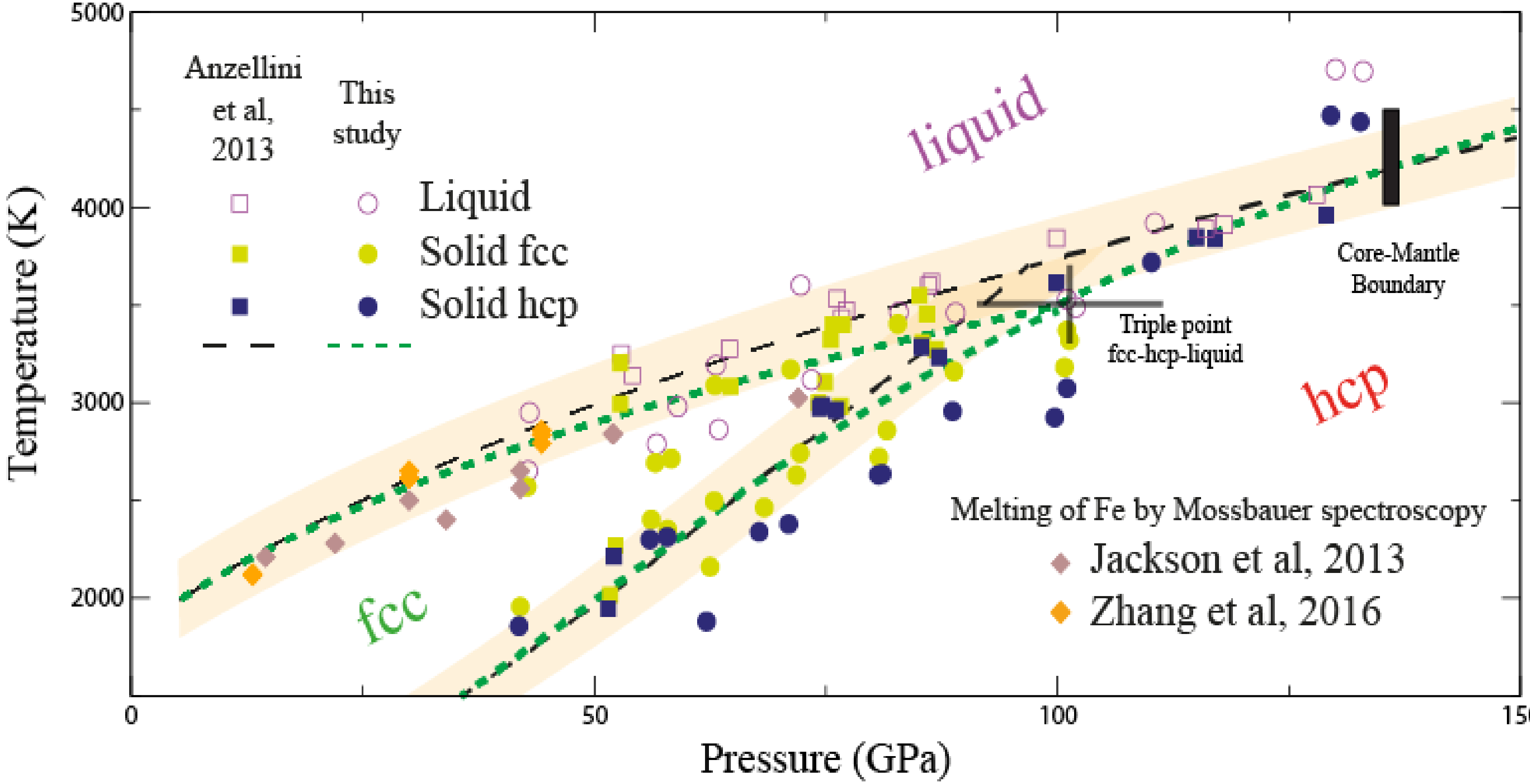
Comparison with previous studies



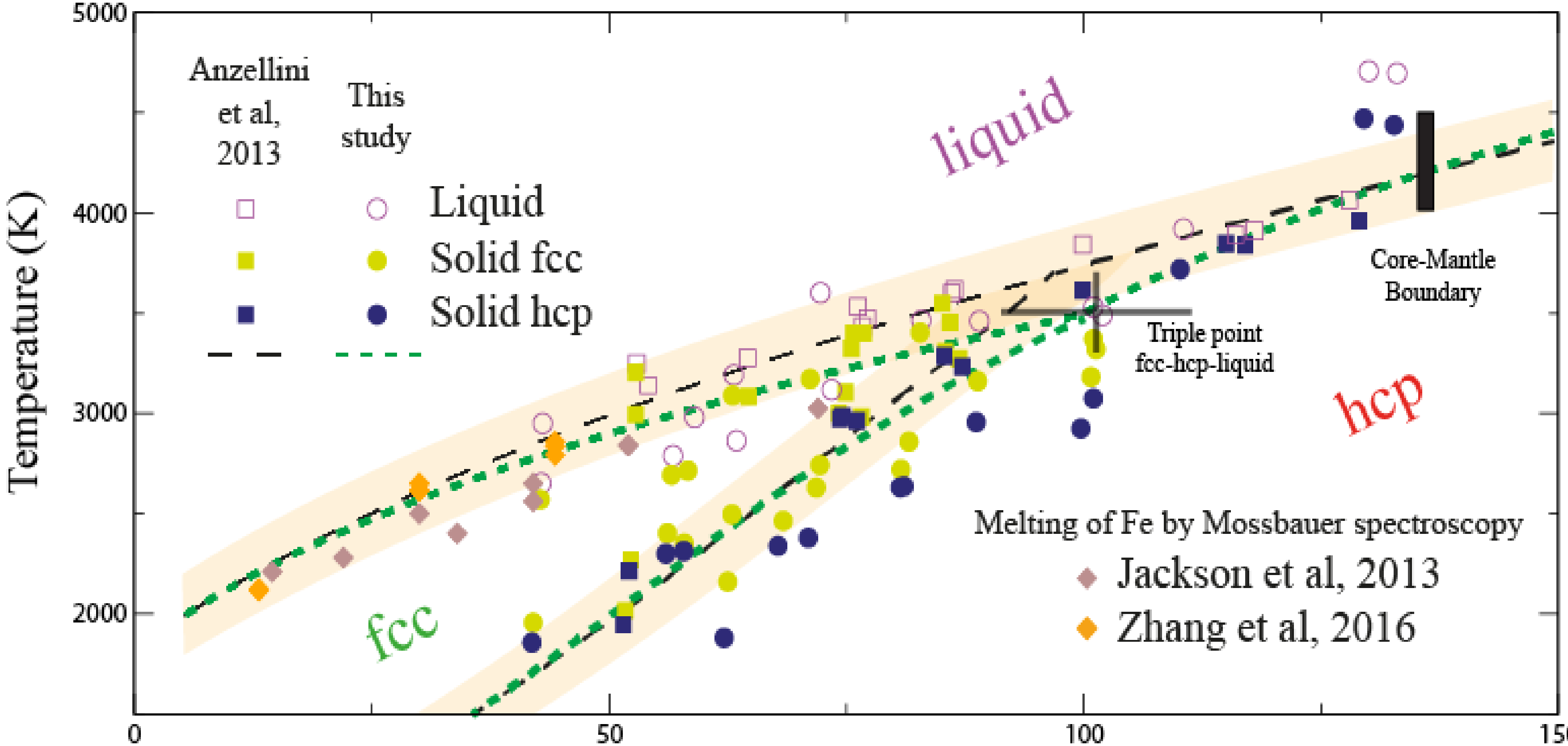
Comparison with previous studies



Comparison with previous studies



Comparison with previous studies

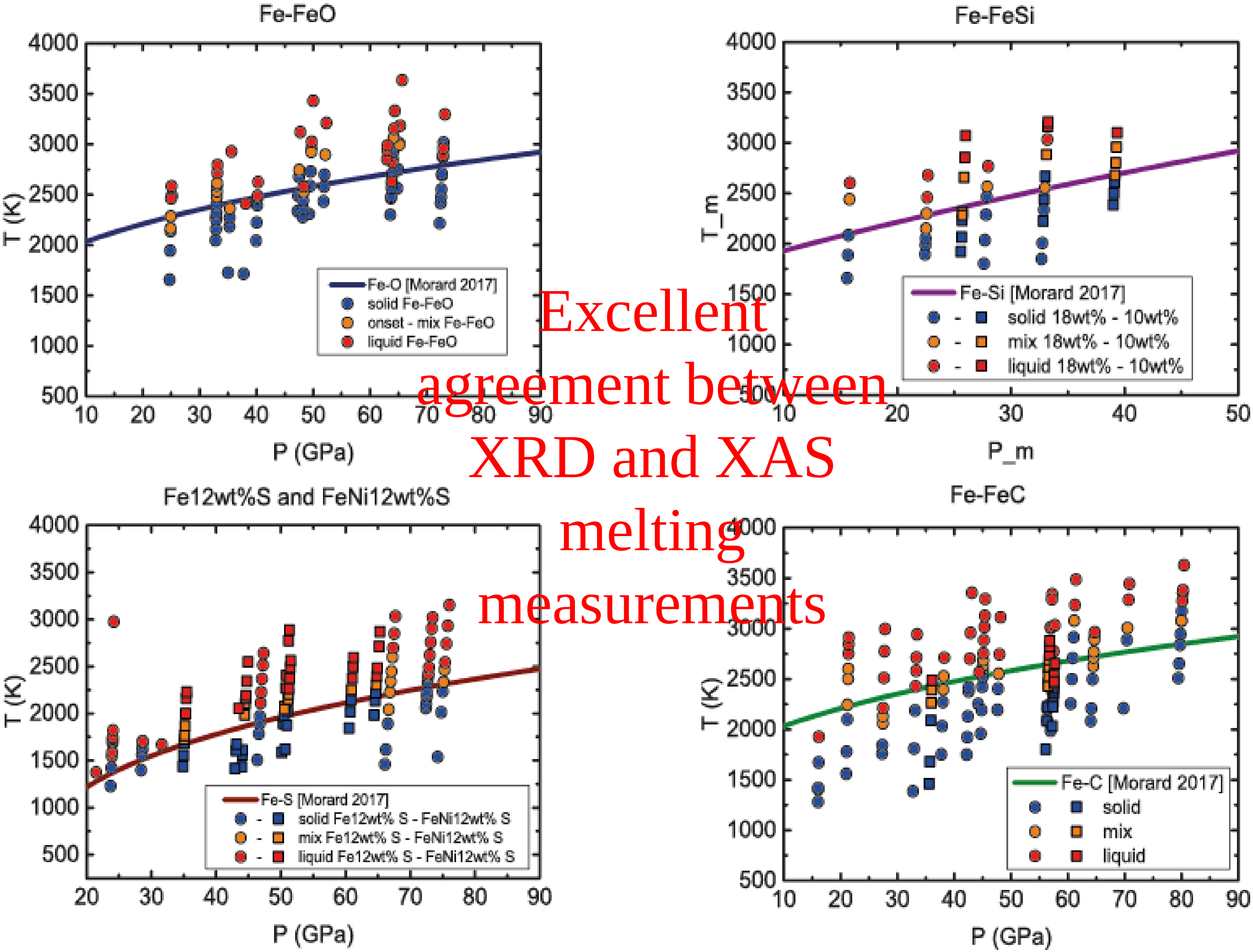


Geophysical Research Letters

Solving Controversies on the Iron Phase Diagram Under High Pressure

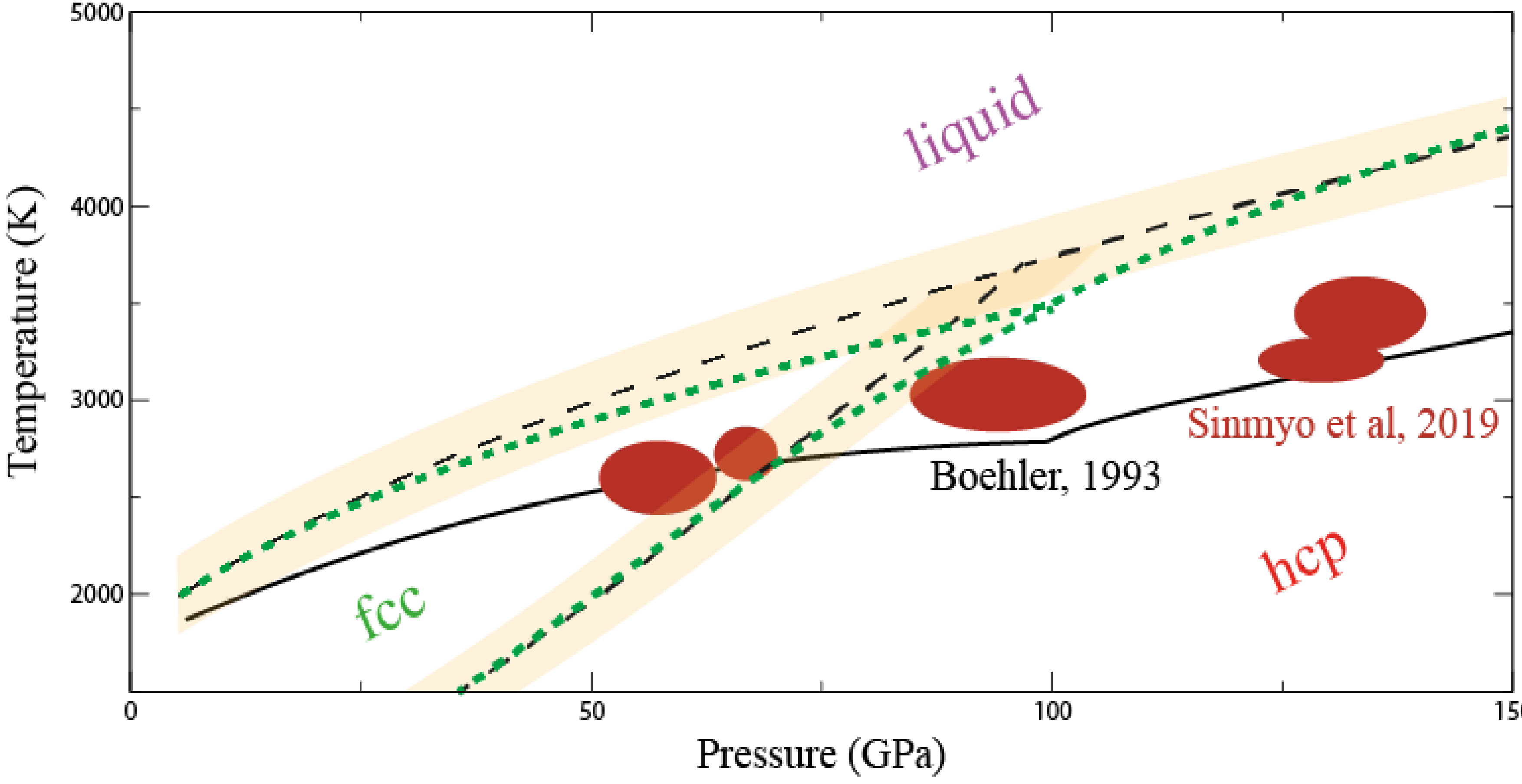
Guillaume Morard[1], Silvia Boccato[2], Angelika D. Rosa[2], Simone Anzellini[3], Francesca Miozzi[1], Laura Henry[2], Gaston Garbarino[2], Mohamed Mezouar[2], Marion Harmand[1], François Guyot[1], Eglantine Boulard[1], Innokenty Kantor[2,4], Tetsuo Irifune[5], and Raffaella Torchio[2]

2018



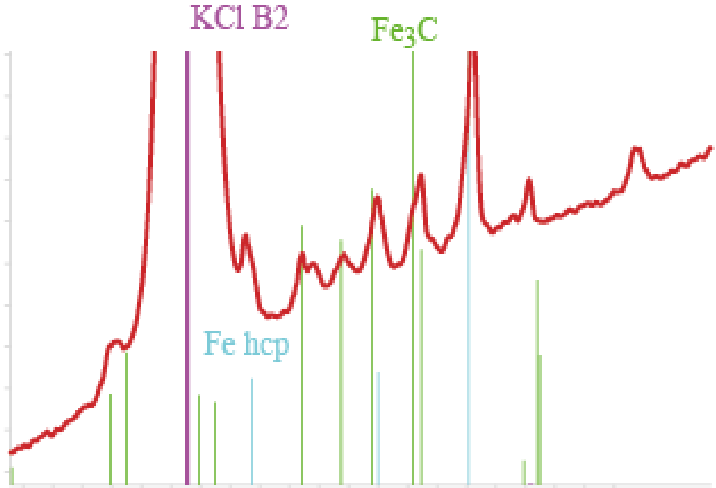
Excellent agreement between XRD and XAS melting measurements

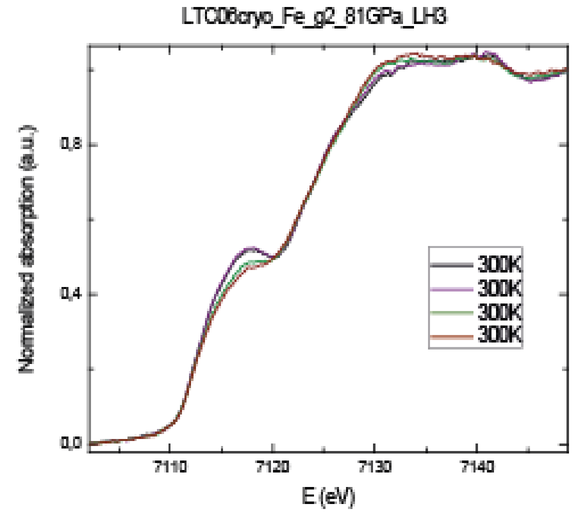
Comparison with previous studies

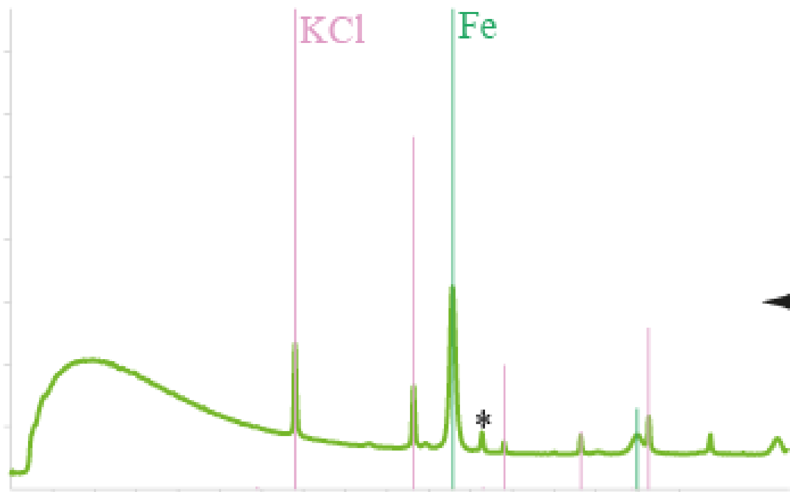


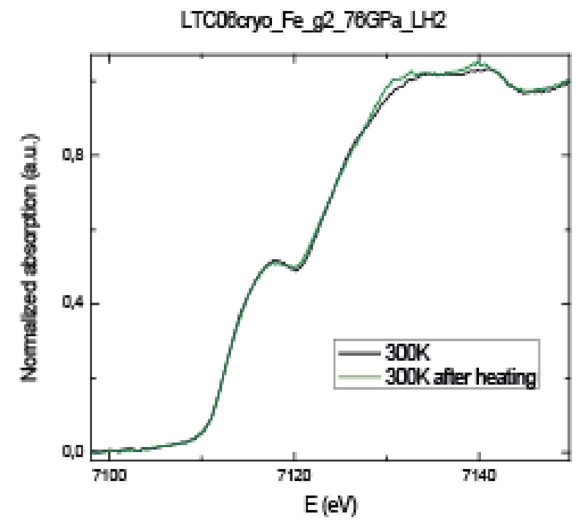
Diffraction on recovered samples

XANES spectra on quench sample between each temperature step



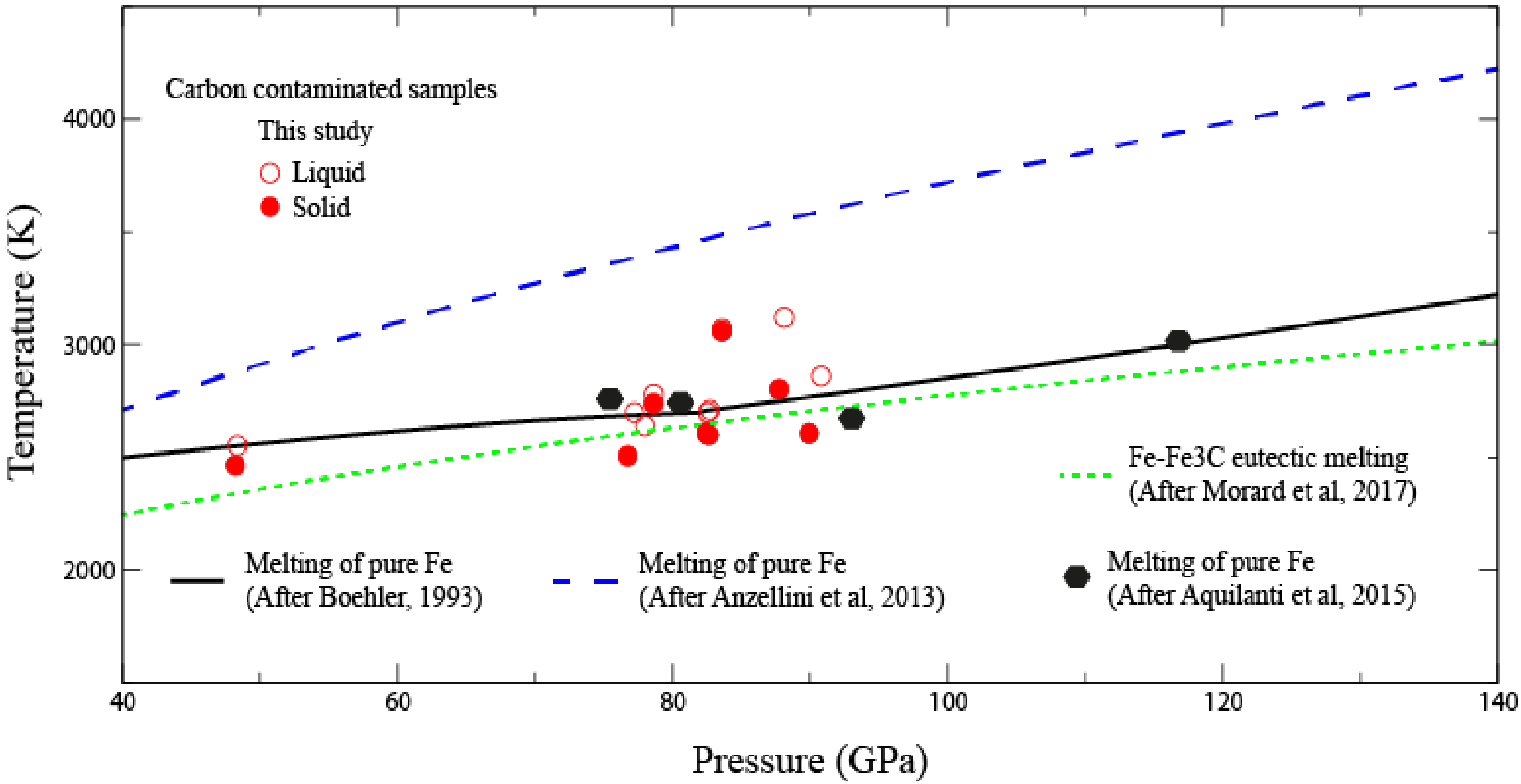




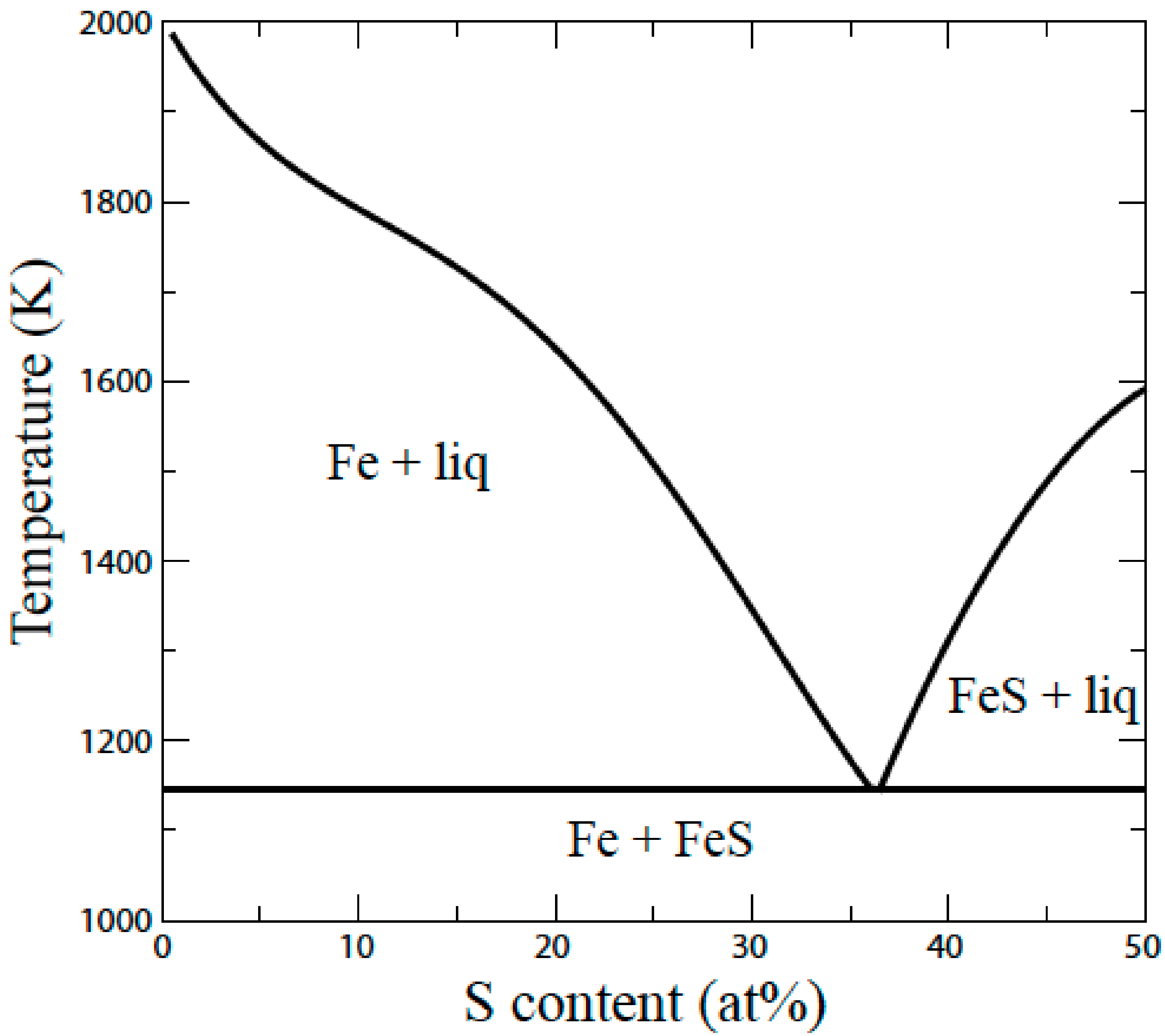


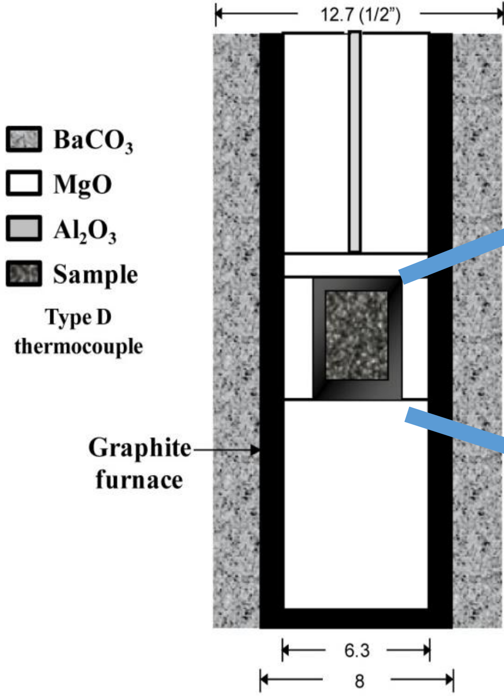
Reactions forming carbides could be clearly identified

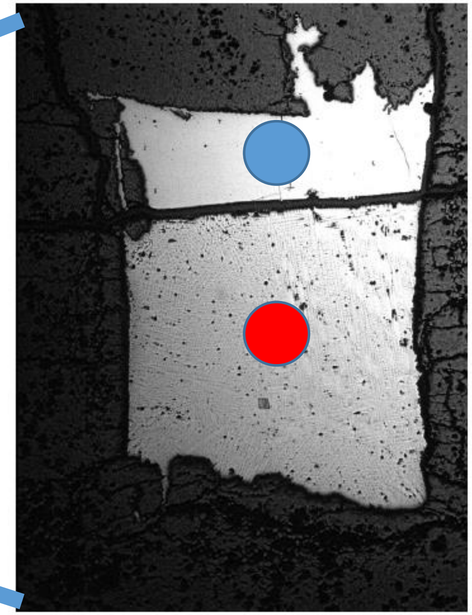
Melting of carbon contaminated iron samples

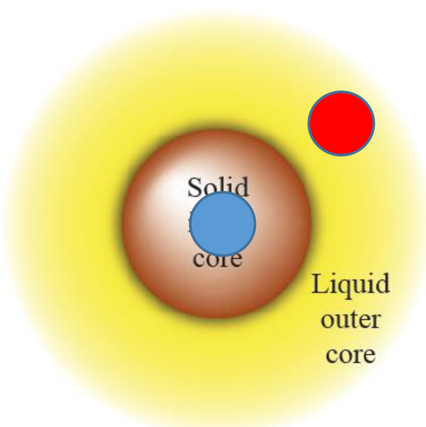


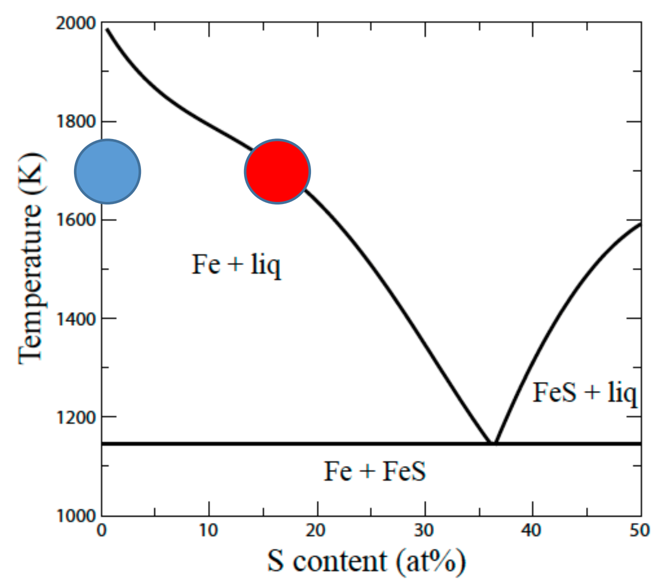
Incongruent melting



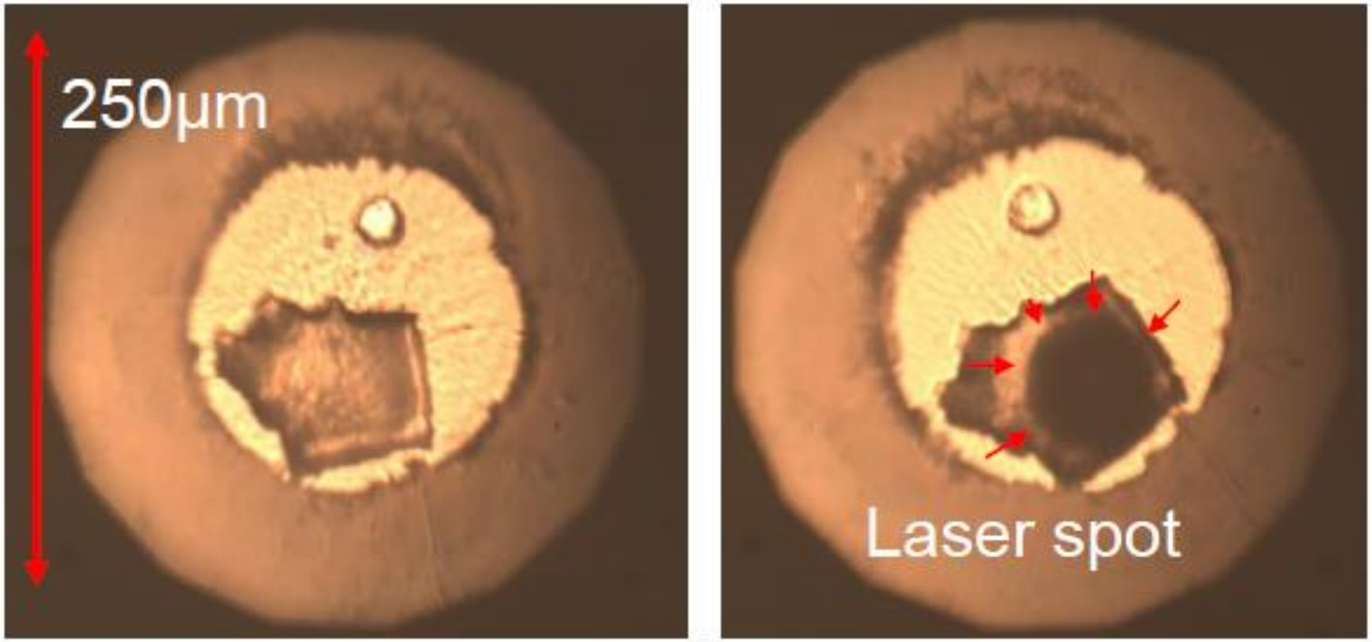




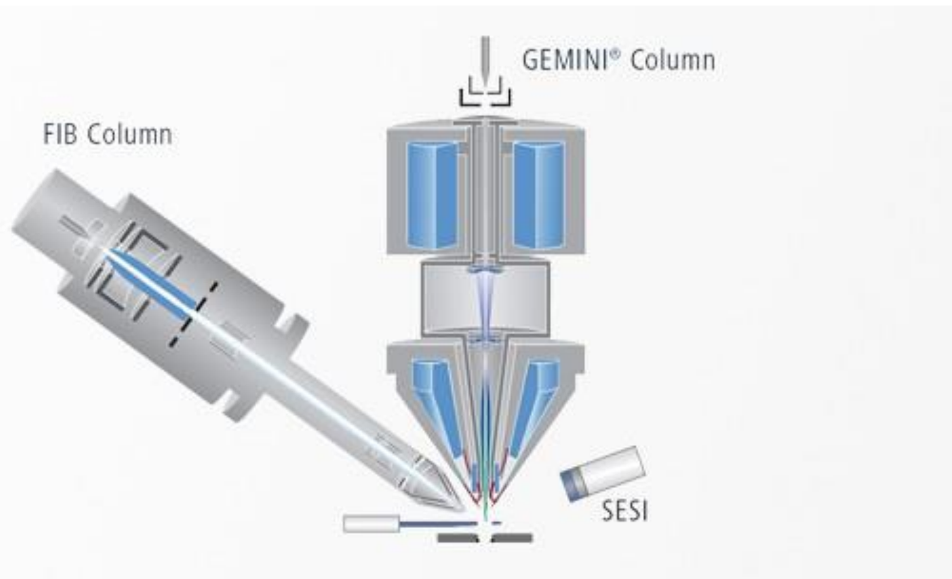


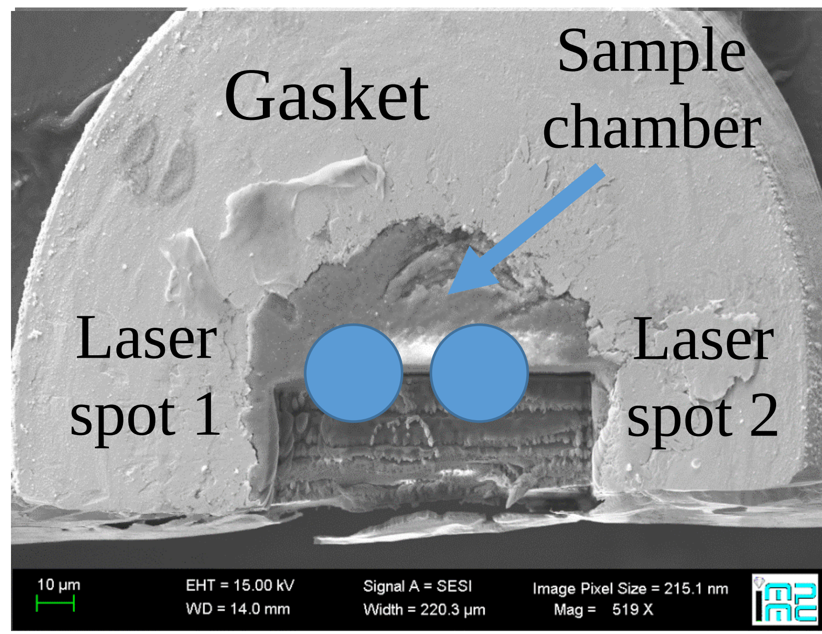


after Buono & Walker, GCA 2011


250µm
Laser spot

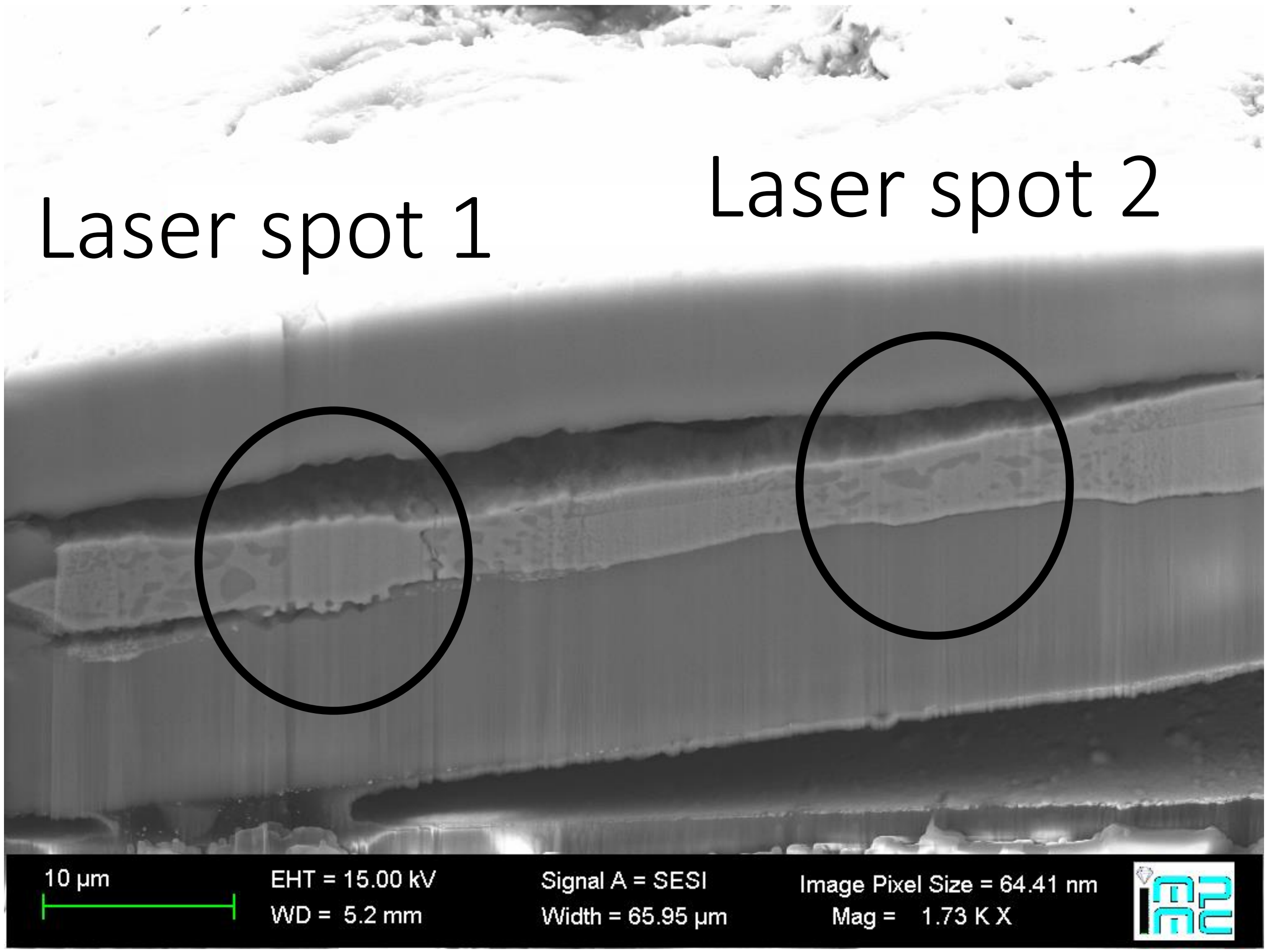
Analysis of post-experiment samples



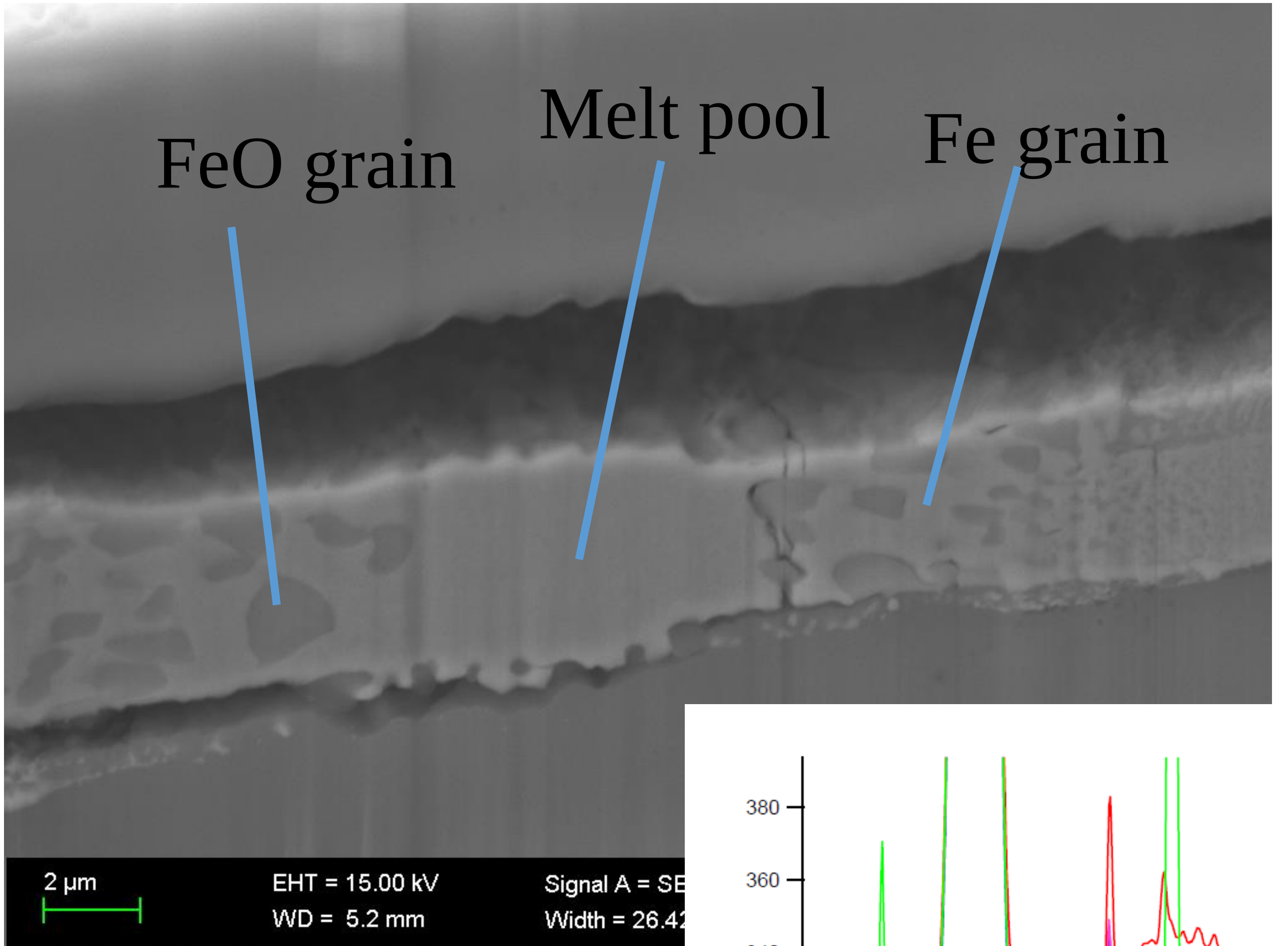


Sample recovered after laser heating experiment at 41 GPa

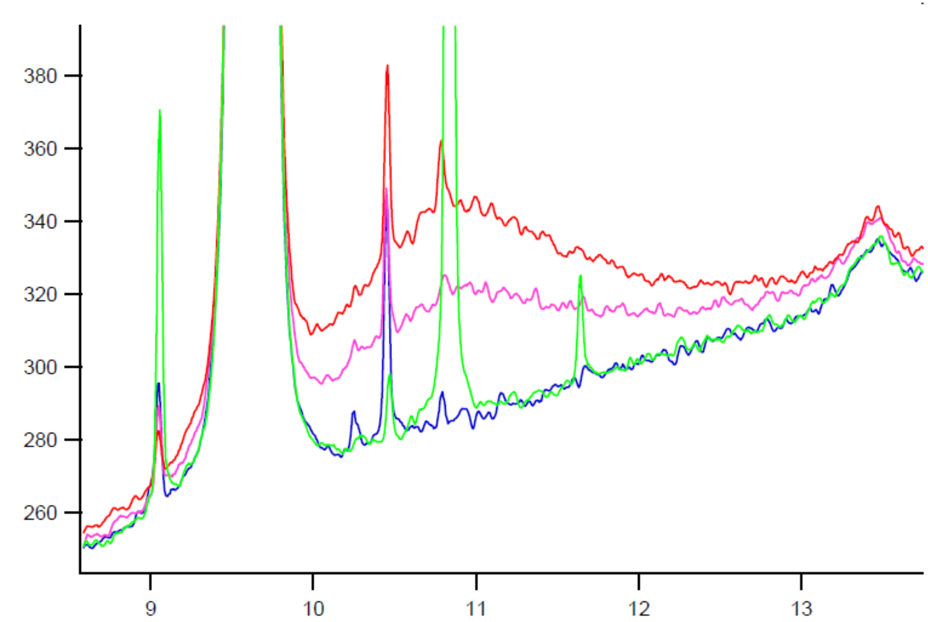
Confirmation from analysis of sample texture after laser heating



Laser spot 1

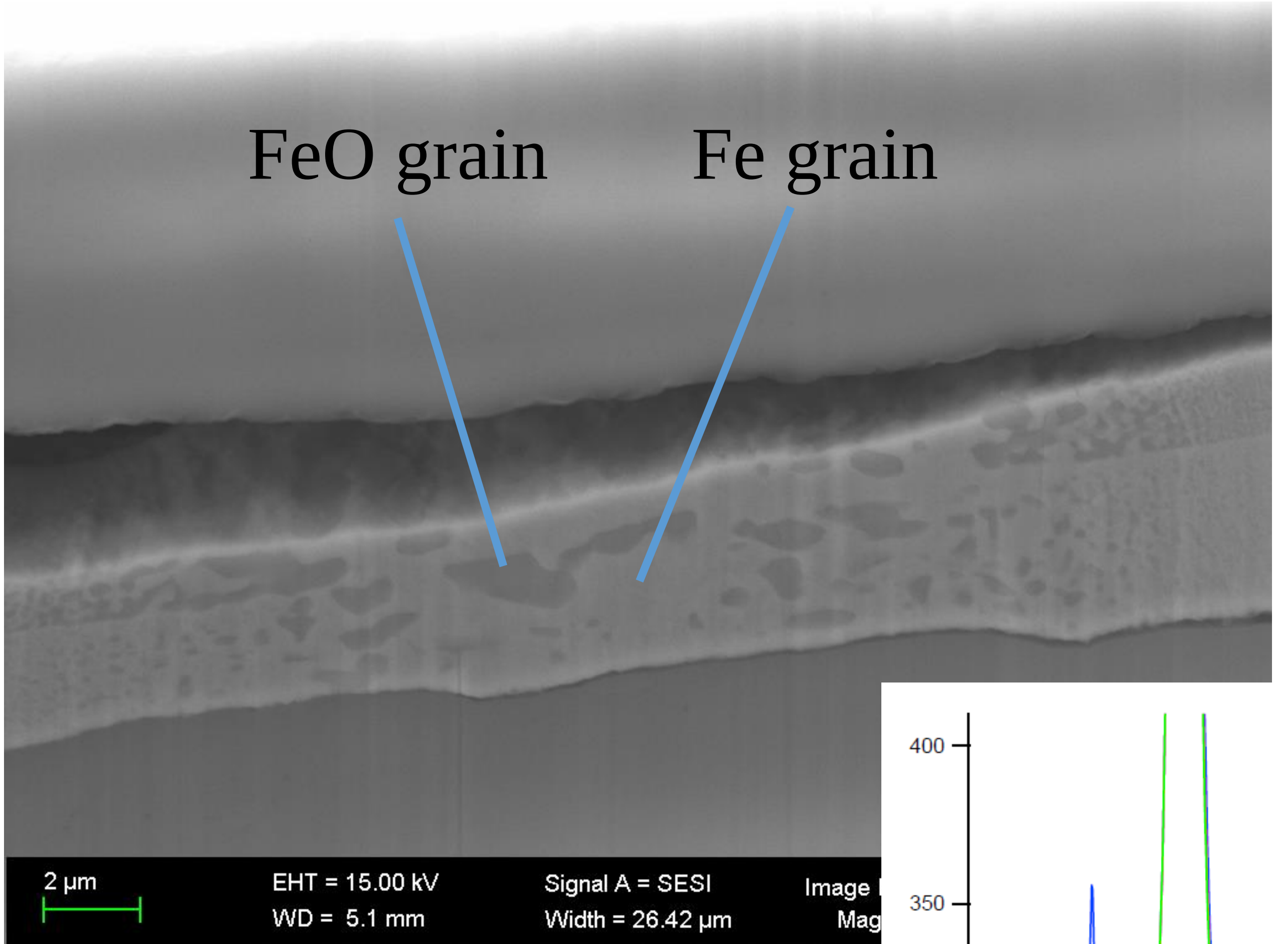


Above melting temperature

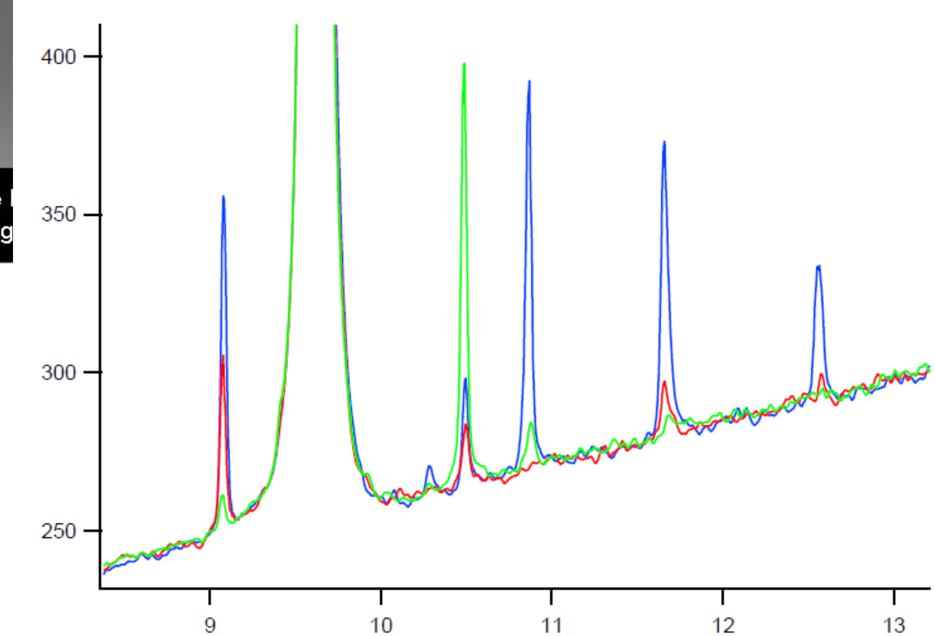


2110 K
2310 K
2390 K
2550 K

Laser spot 2



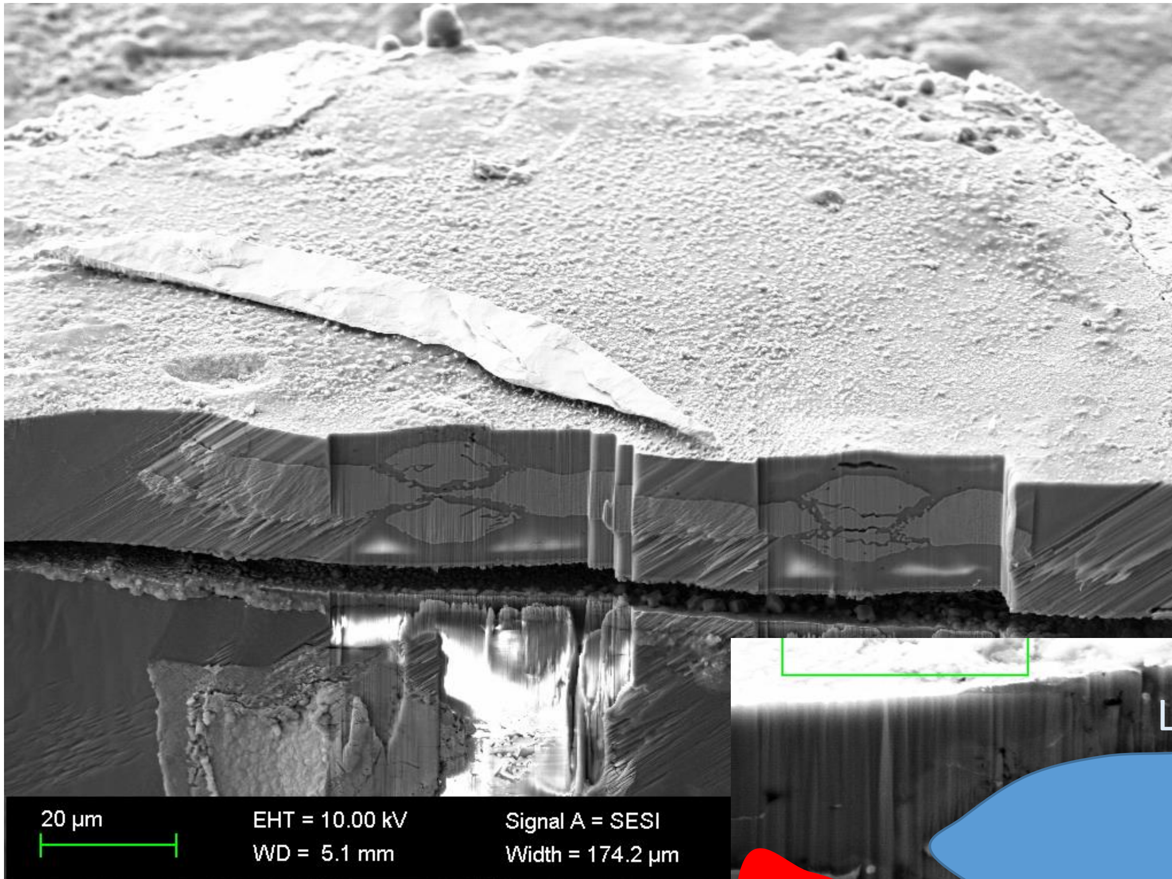
Below melting temperature

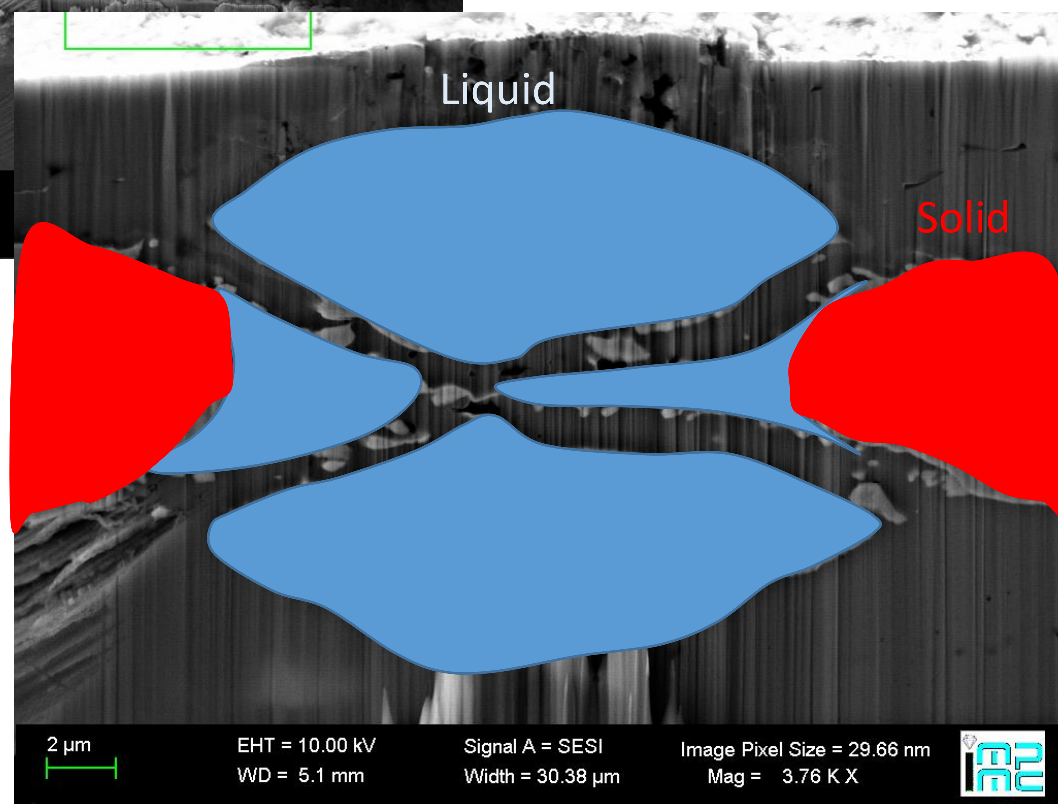


2070 K

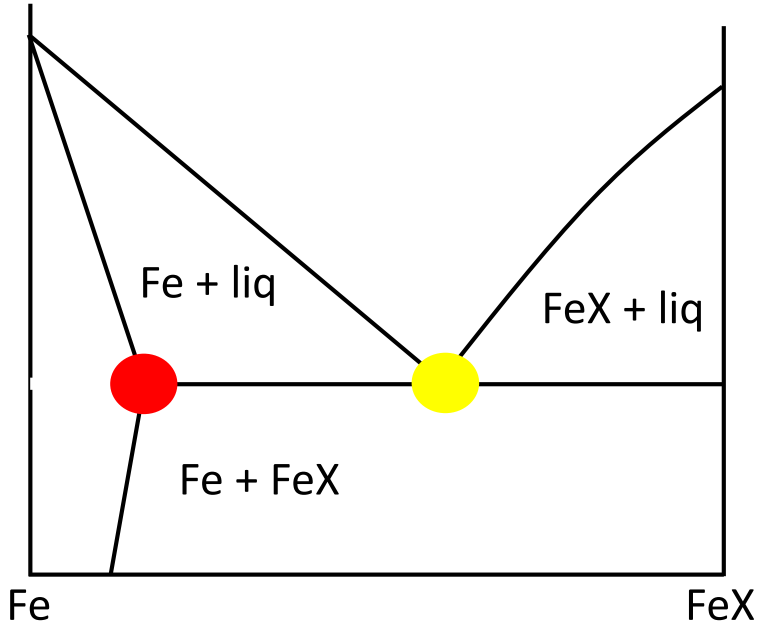
2150 K

2170 K

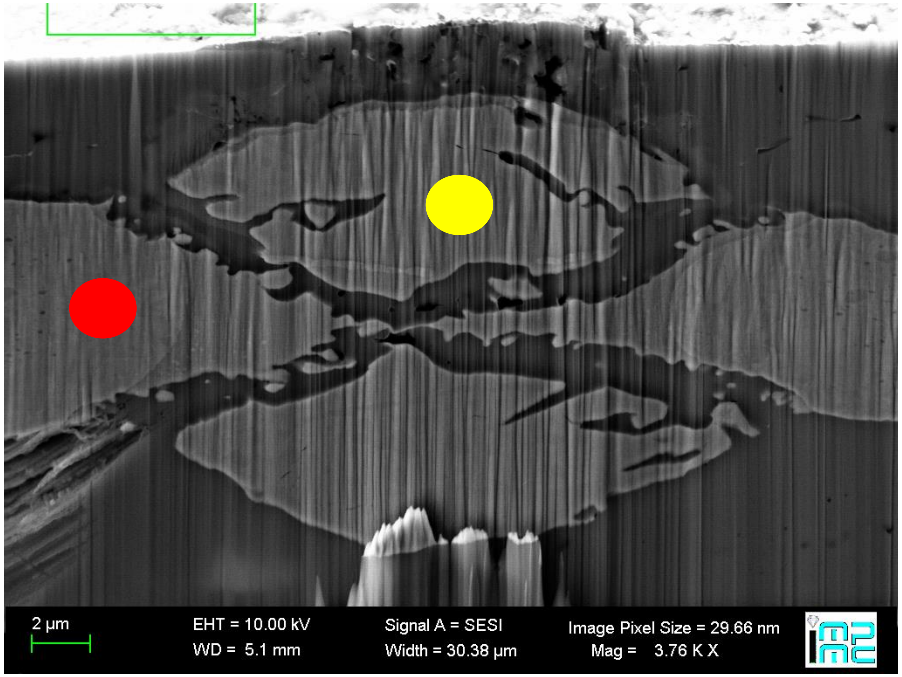


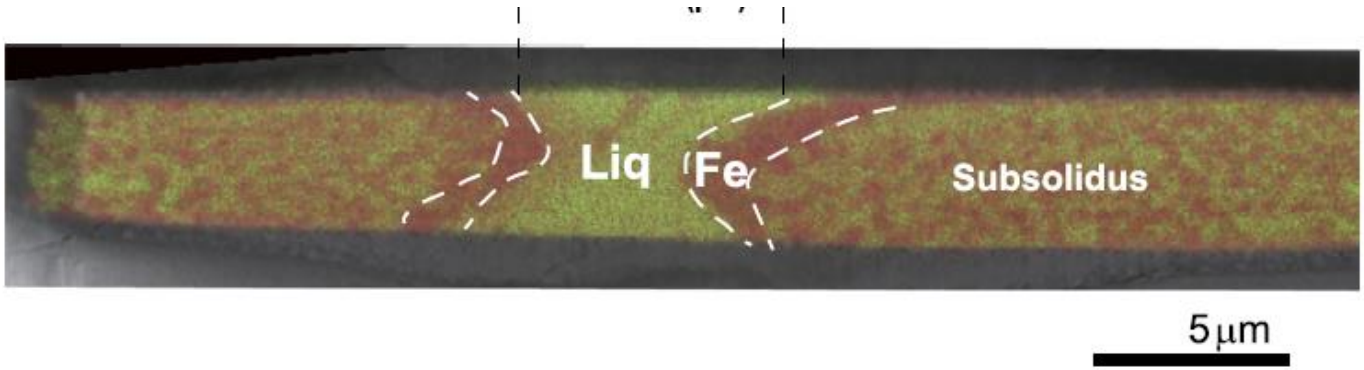


Creating a planetary core experimentaly

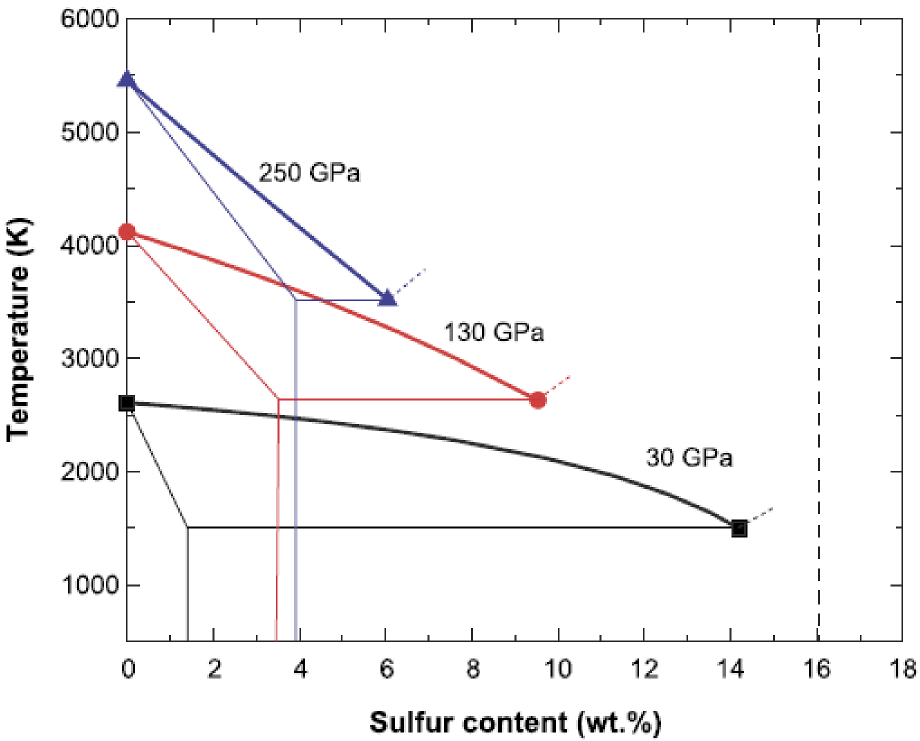


Determination of the phase diagram under high pressure





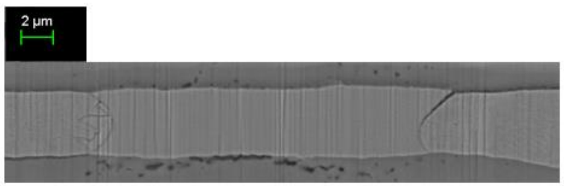
Reconstructing phase diagrams of iron alloys under Earth's core conditions

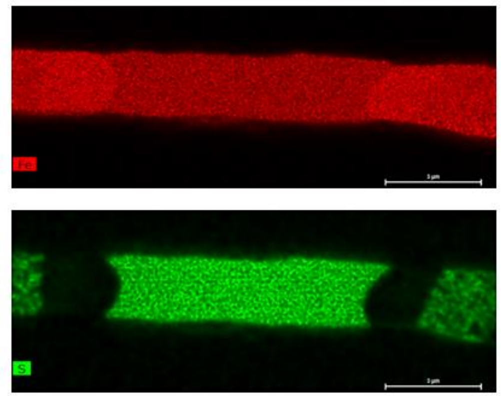


Mori et al, EPSL, 2017

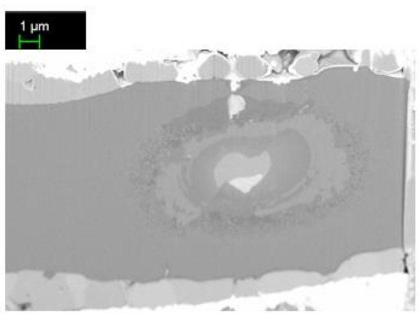
Problem of chemical segregation

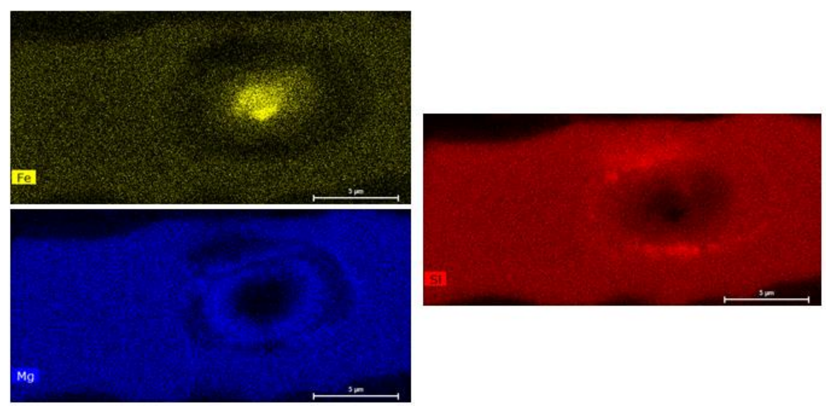
Fe-S alloy after melting at 30 GPa

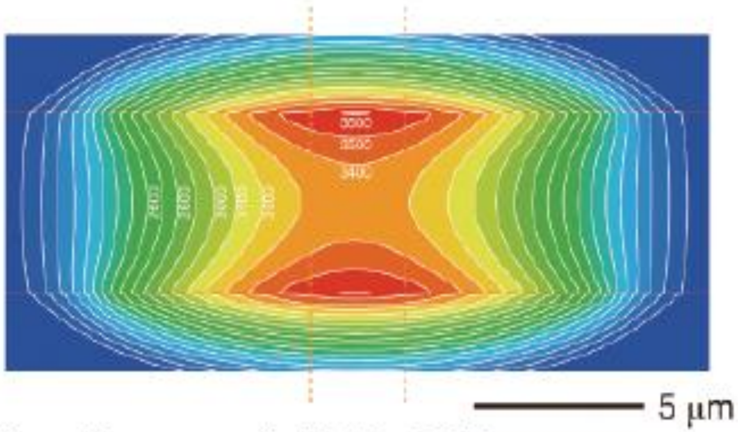




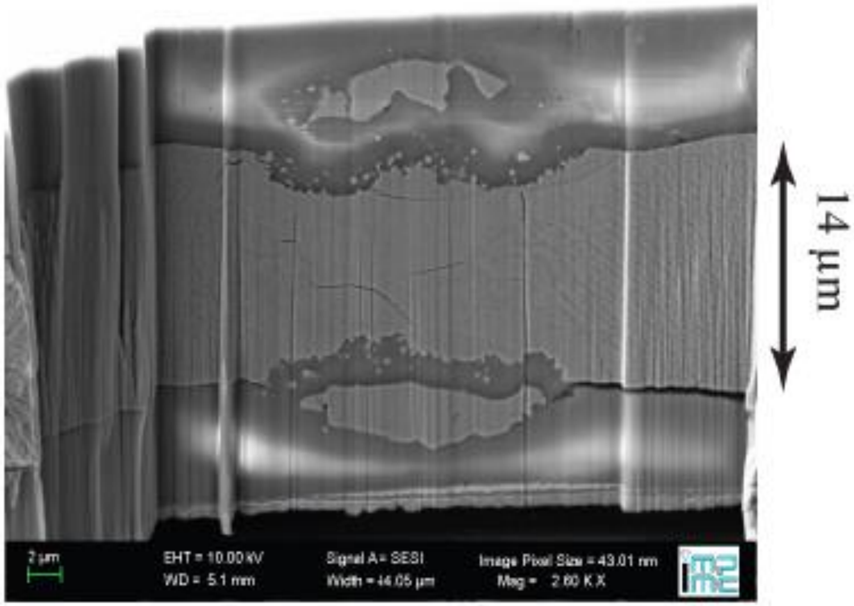
MORB silicate after melting at 65 GPa

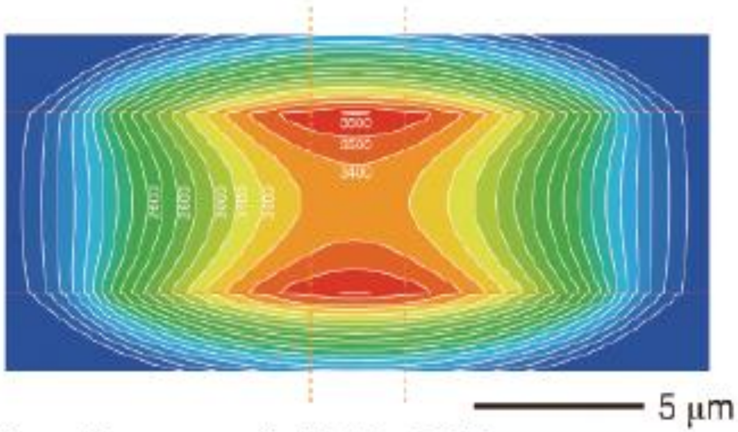




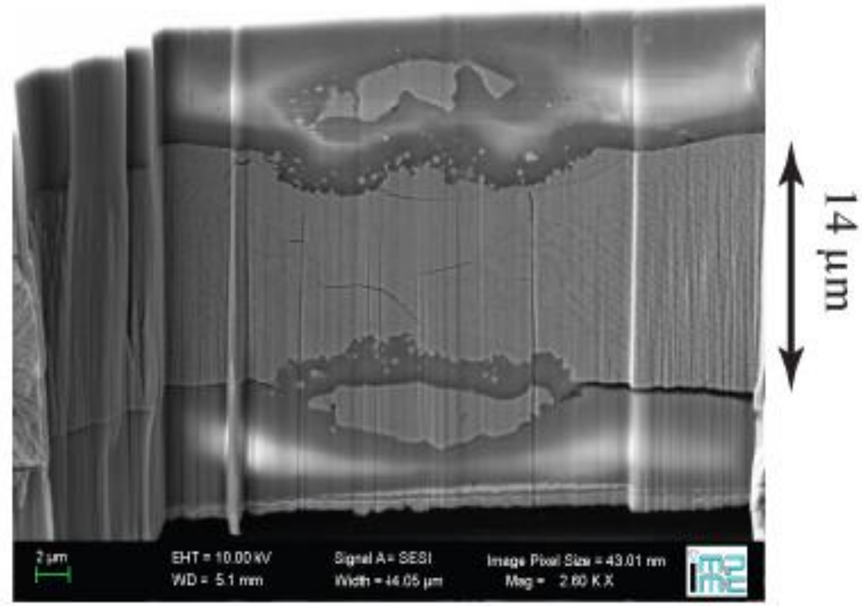


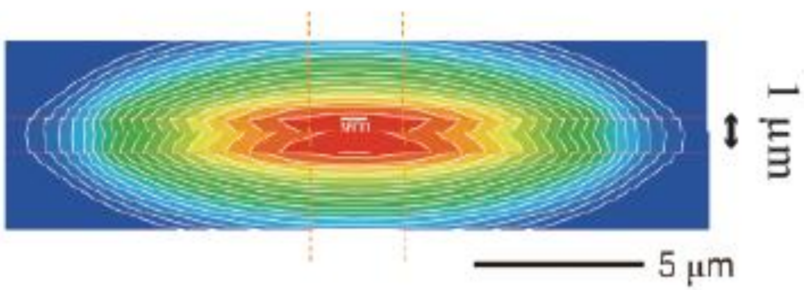
After Sinmyo et al, EPSL, 2019

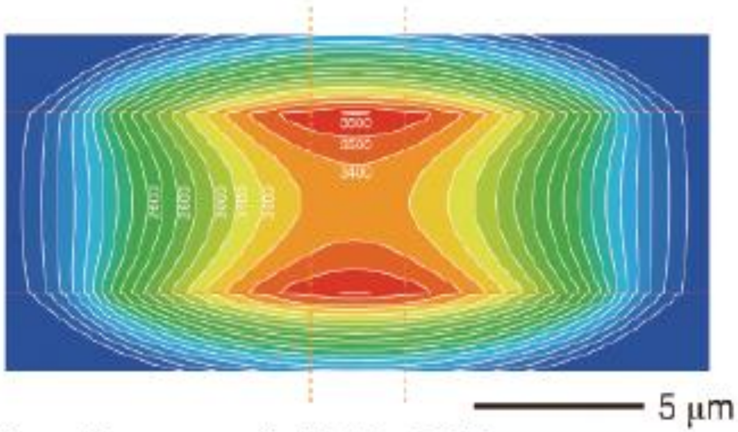




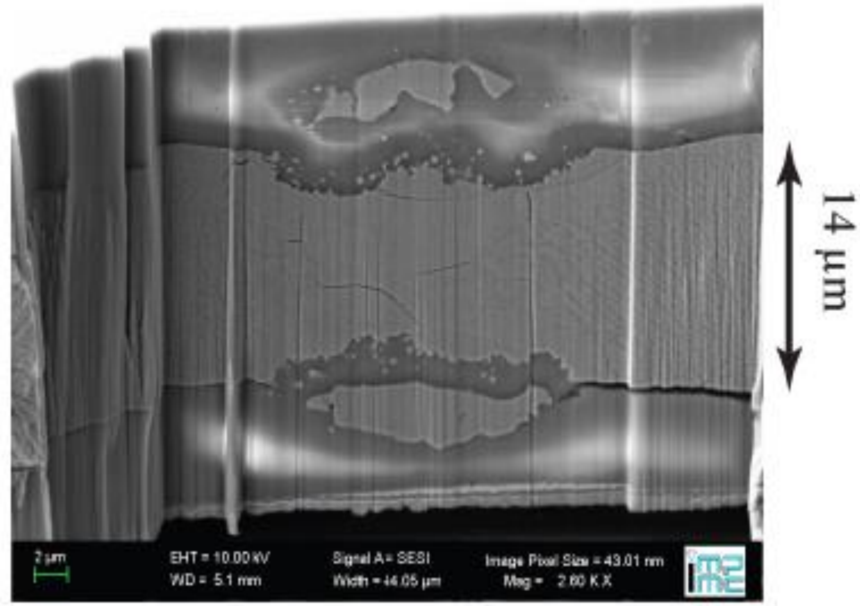
After Sinmyo et al, EPSL, 2019

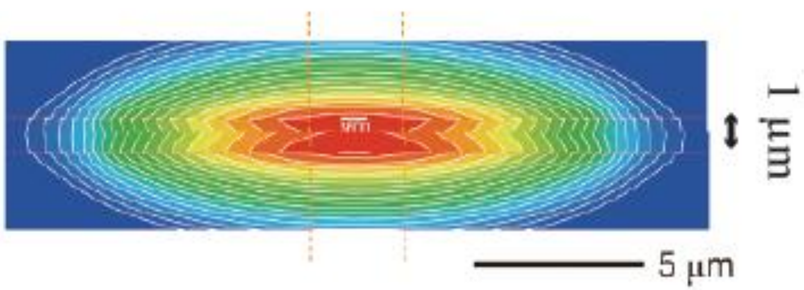


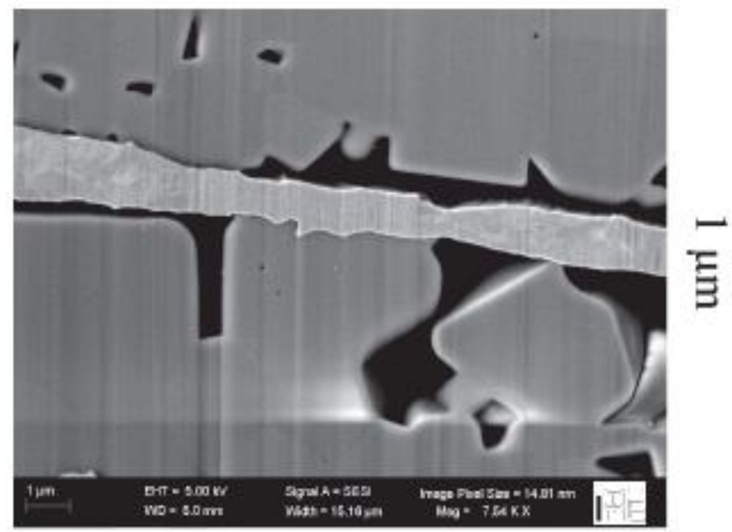


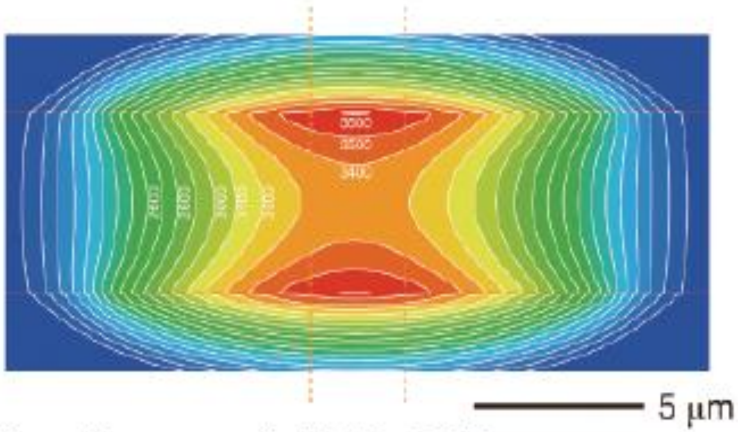


After Sinmyo et al, EPSL, 2019

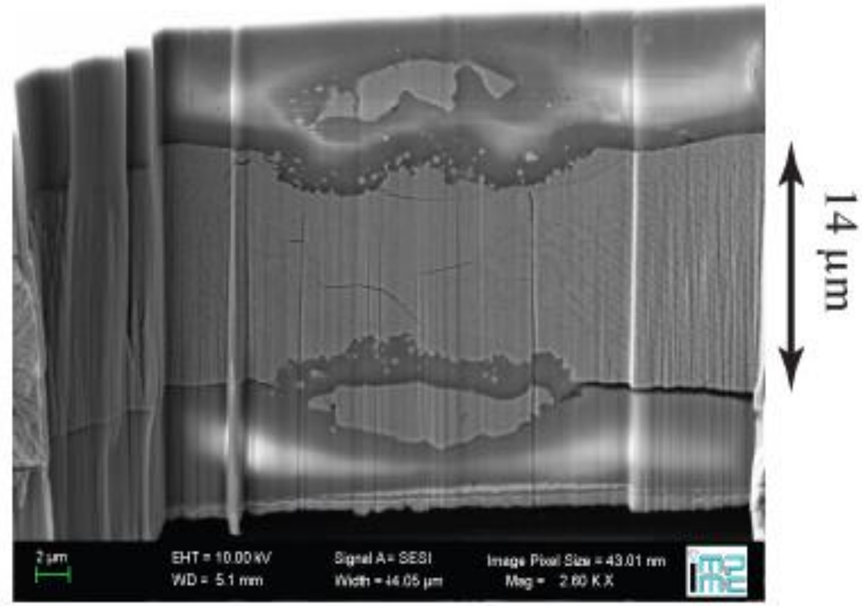


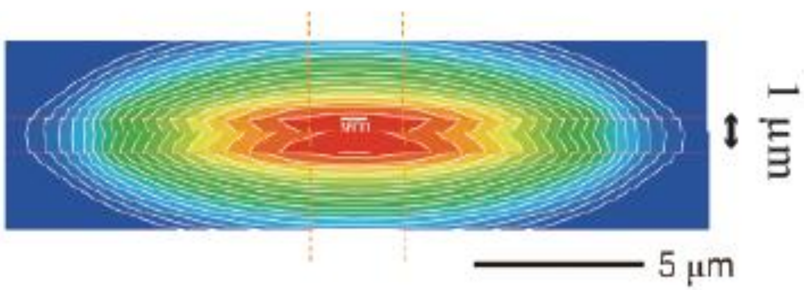


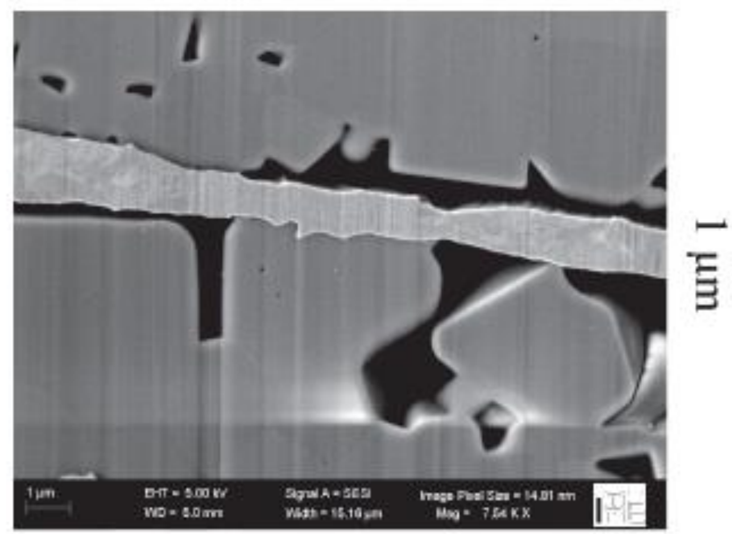




After Sinmyo et al, EPSL, 2019

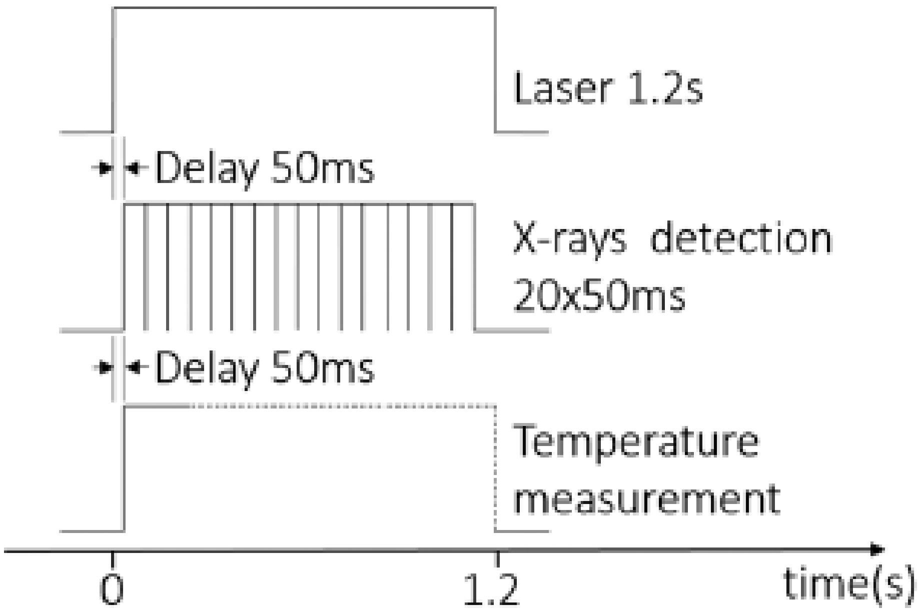






Temperature gradients minimal in laser heating experiments if sample under high pressure is thinner than ~2µm

Fast acquisition could be the key

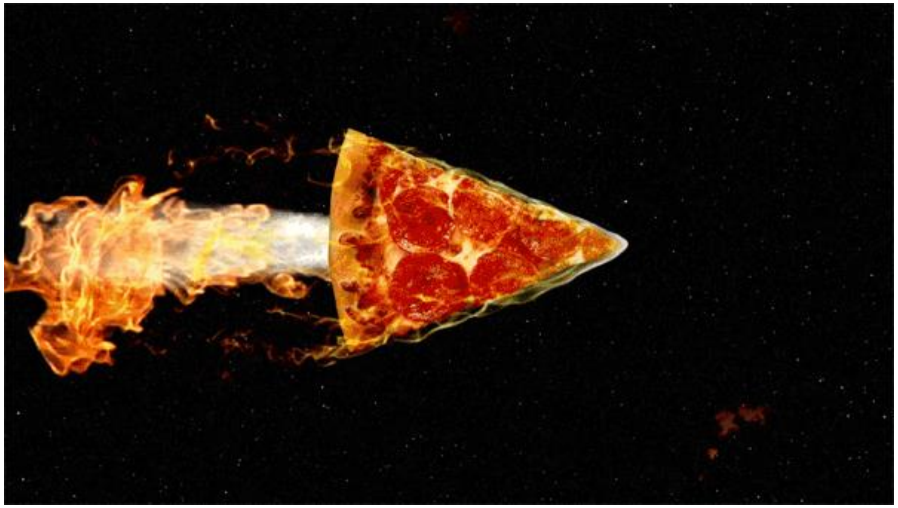


After Boccato et al, JGR, 2017

With the EBS,
flux * 100

Therefore
acquisition time
/100
(CdTe detector
250 Hz)

Data treatment of large amount of data ??



Thank you for your attention