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 types 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

![Graph showing wave velocity and density vs. depth and pressure](image-url)
Olivine: 60% en volume des roches du manteau supérieur

\[(\text{Mg},\text{Fe})_2\text{SiO}_4\]
Strong relation between seismological structure of the upper mantle and its mineralogy

Frost, Elements, 2008
Transitions de phases induites par la pression

\[ \alpha\text{-olivine} \ (\text{Mg,Fe})_2\text{SiO}_4 \quad \beta\text{-wadsleyite} \ (\text{Mg,Fe})_2\text{SiO}_4 \]

\[ \text{Perovskite} \ (\text{Mg,Fe})\text{SiO}_3 \quad \gamma\text{-ringwoodite} \ (\text{Mg,Fe})_2\text{SiO}_4 \]

\[ + (\text{Mg,Fe})\text{O} \]
Melting of core materials at ICB: anchoring point for the geotherm
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

Decompression

Graph showing intensity vs. 2θ (°) with peaks labeled 111 and 200. Another graph inset showing T (K) vs. P (GPa) with data points and error bars.
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. Miozzi1, G. Morard1, D. Antonangeli1, A. N. Clark2, M. Mezouar3, C. Dorn4, A. Rozel5, and G. Fiquet1
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

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

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

From Sola et al, 2009
Melting of pure iron: a controversial anchoring point for the geotherm

- Laser-heated diamond anvil cell
- Ab initio calculations
- Shock experiments

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

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Aquilanti et al, PNAS, 2015
In situ XANES study
Back on Boehler, 1993
Melting of pure iron: a controversial anchoring point for the geotherm

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

Sinmyo et al, EPSL, 2019
Resistive heating

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

From Sola et al, 2009

![Graph showing melting points of pure iron under various conditions.](image-url)
Energy dispersive EXAFS experimental set-up coupled with Laser-Heated Diamond Anvil Cell on ID24 beamline, ESRF

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

Change of the edge upon melting

From Aquilanti et al, PNAS, 2015


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
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In situ detection of melting in LHDAC

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

First melt
In situ detection of melting in LHDAC

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

First melt
Comparison with previous studies

![Graph showing comparison of temperature and pressure with previous studies. The graph compares the results of Anzellini et al. (2013) and the current study. Different markers represent different phases: Liquid, Solid fcc, and Solid hcp.]
Comparison with previous studies

Anzellini et al., 2013
This study

Temperature (K)

Solid fcc
Solid hcp
Liquid

Pressure (GPa)

fcc
hcp
liquid
Comparison with previous studies

Temperature (K) vs Pressure (GPa) graph showing:
- Liquid
- Solid fcc
- Solid hcp

Melting of Fe by Mossbauer spectroscopy:
- Jackson et al, 2013
- Zhang et al, 2016
Comparison with previous studies

Temperature (K) vs. Pressure (GPa) graph showing:
- Liquid
- Solid fcc
- Solid hcp

Anzellini et al., 2013
This study

Melting of Fe by Mossbauer spectroscopy
- Jackson et al., 2013
- Zhang et al., 2016

Core-Mantle Boundary
Triple point fcc-hcp-liquid
Comparison with previous studies

Solving Controversies on the Iron Phase Diagram Under High Pressure

Geophysical Research Letters

Guillaume Morard\textsuperscript{1}, Silvia Boccato\textsuperscript{2}, Angelika D. Rosa\textsuperscript{3}, Simone Anzellini\textsuperscript{3}, Francesca Miozzi\textsuperscript{1}, Laura Henry\textsuperscript{2}, Gaston Garbarino\textsuperscript{2}, Mohamed Mezouar\textsuperscript{2}, Marion Harmand\textsuperscript{1}, François Guyot\textsuperscript{1}, Eglantine Boulard\textsuperscript{1}, Innokenty Kantor\textsuperscript{2,4}, Tetsuo Irfune\textsuperscript{5}, and Raffaella Torchio\textsuperscript{2}
Excellent agreement between XRD and XAS melting measurements.
Comparison with previous studies

- **fcc**
- **hcp**
- **liquid**
- **Boehler, 1993**
- **Sinmyo et al, 2019**
Reactions forming carbides could be clearly identified.
Melting of carbon contaminated iron samples

Carbon contaminated samples
This study
- Liquid
- Solid

Fe-Fe3C eutectic melting
(After Morard et al, 2017)

Melting of pure Fe
(After Boehler, 1993)
Melting of pure Fe
(After Anzellini et al, 2013)
Melting of pure Fe
(After Aquilanti et al, 2015)
Incongruent melting

Diagram showing the phase diagram with axes labeled as follows:
- Vertical axis: Temperature (K)
- Horizontal axis: S content (at%)
after Buono & Walker, GCA 2011
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

Laser spot 2
Laser spot 1

Above melting temperature

FeO grain

Melt pool

Fe grain

2110 K

2310 K

2390 K

2550 K

EHT = 15.00 kV
WD = 5.2 mm
Width = 26.42
Below melting temperature
Creating a planetary core experimentally
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
Temperature gradients minimal in laser heating experiments if sample under high pressure is thinner than $\sim 2\mu m$.
Fast acquisition could be the key

With the EBS, flux \times 100

Therefore acquisition time /100
(CdTe detector 250 Hz)

Data treatment of large amount of data ??

After Boccato et al, JGR, 2017
Thank you for your attention