Phase diagrams of iron and iron oxides at Super-Earth interior conditions

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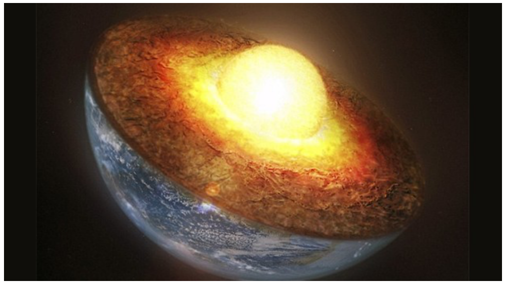
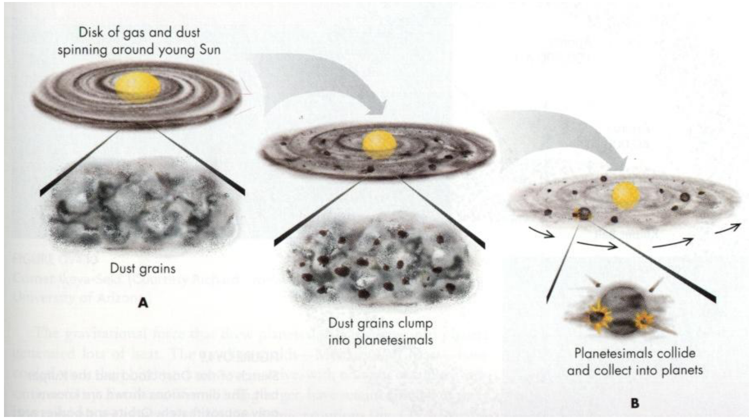
LMJ User Meeting– June 8th & 9th 2023

Matter under extreme conditions in geoscience

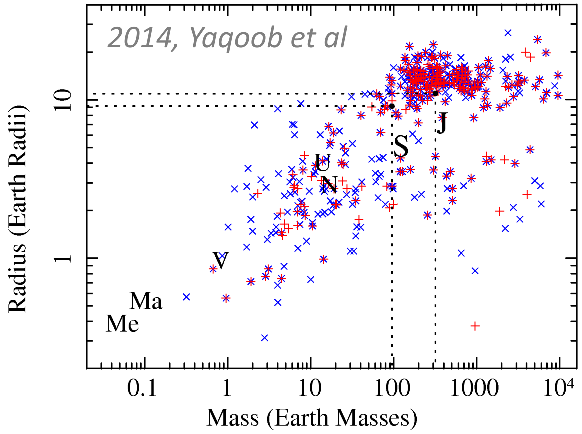
Planetary interiors



Accretion, high-velocity and giant impacts



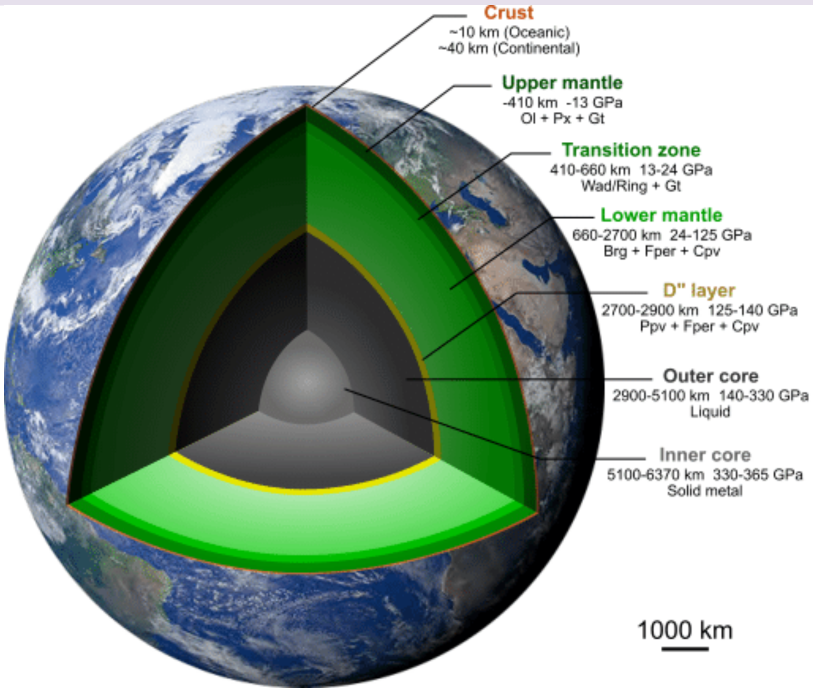
Exoplanets interiors and habitability



5338 confirmed exoplanets (9432 unconfirmed - 07.06.2023)
Discovery / 2 days
~63 habitable exoplanets (January 2023
https://phl.upr.edu/projects/habitable-exoplanets-catalog)

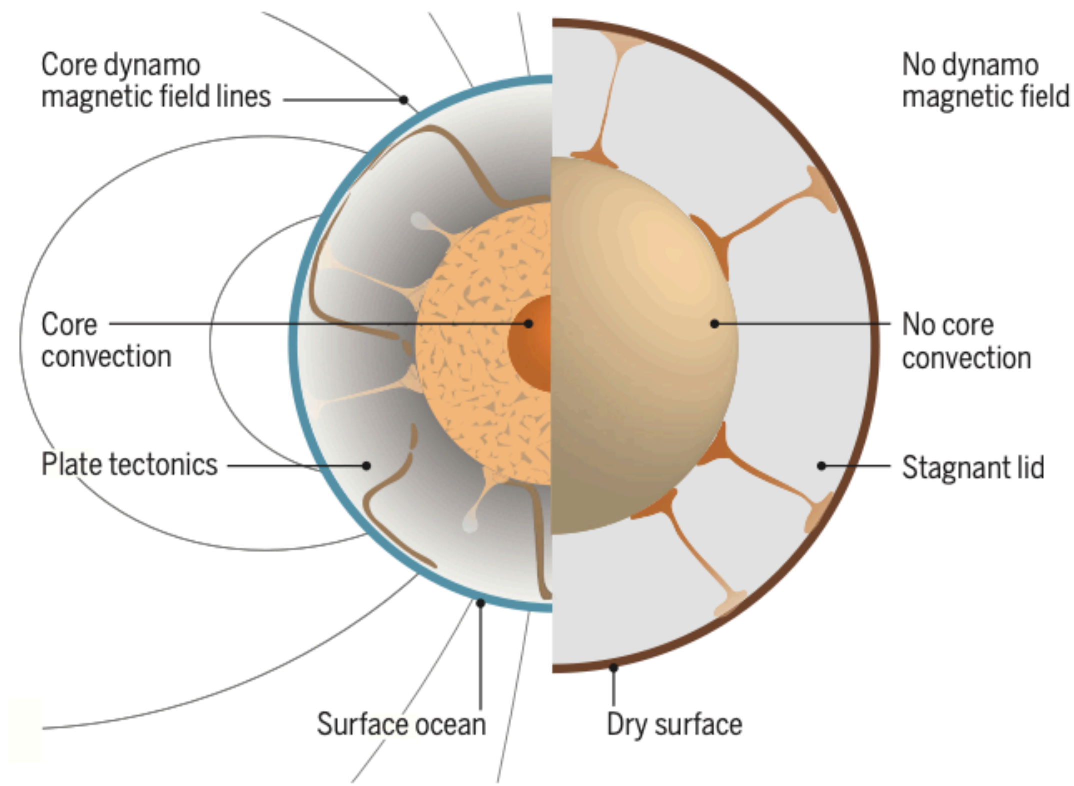
- Moon: Collision between a proto-Earth and a Mars size body, 4.5Gy ago
- Many debris from the collision and accretion to form the Moon ?
- How much is left from the external body in the Earth?

Exoplanets and Habitability



T. Yoshizaki Chemie der Erde 2021

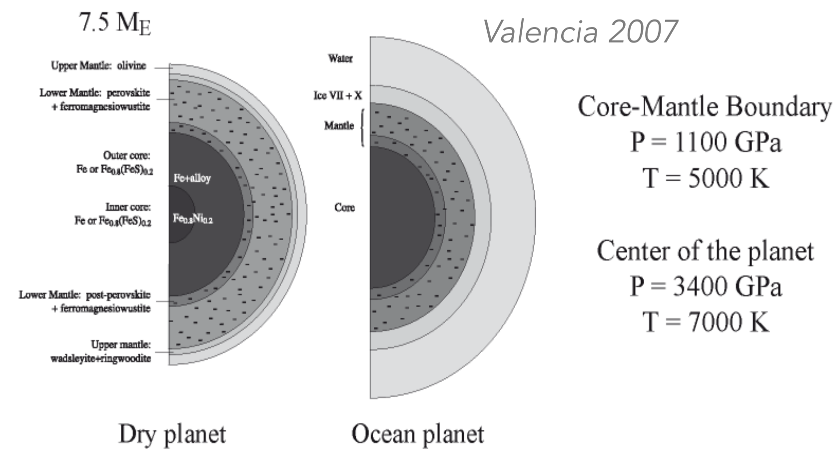
Shahar Science 2019



Big inner core, entirely solid?
Tectonic?
Geodynamo?
Liquid core?
…

Iron and Iron oxides in planetary interiors

Iron is the main component in the inner and outer core of the Earth
- Solid or liquid in Super Earth?
- Cristallographic phase and physical properties?

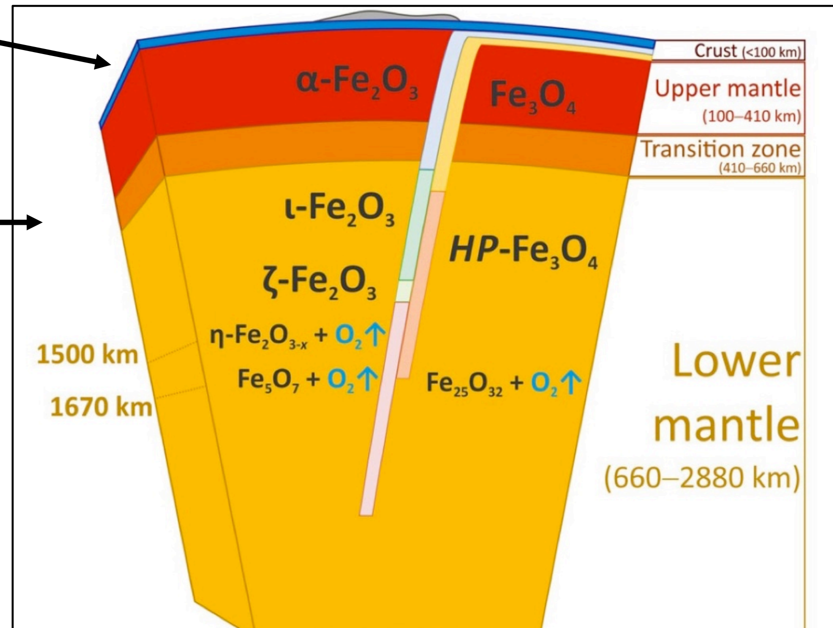


Valencia 2007

Iron oxides at ambient conditions:
FeO, Fe_2O_3, Fe_3O_4

Mantle component (135 GPa, 4000 K) :
- The oxidation state influences the redox state (Fe^{2+}/Fe^{3+} proportions) and iron partitioning in minerals [1]
- Spin state affect mineral properties [1]
- New stoichiometries (ex : FeO_2, Fe_5O_7, Fe_4O_5…) [2,3]
- O and H reservoirs?

[1] Badro, Annu. Rev. Earth Planet. Sci.,2014
[2] Hu et al., nature, 2016
[3] Lavina et al., PNAS, 2011 & Sci. Adv., 2015

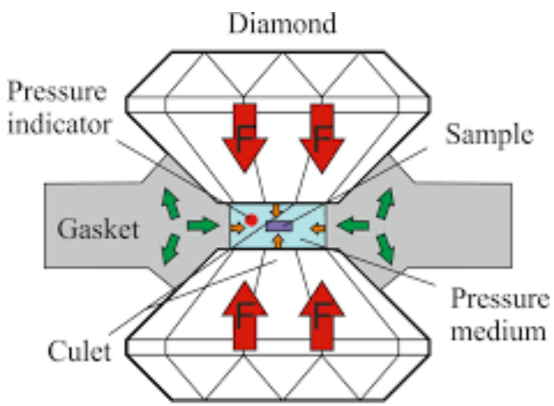


Bykova et al., Nat. Commun., 2016

Ferropericlase (Fe,Mg)O are major components in the Earth mantle

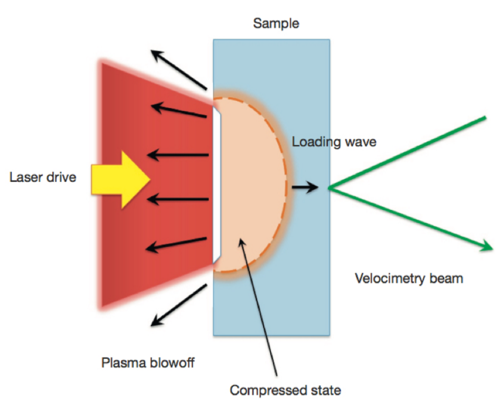
Studies in laboratories

Static compression heating
(LH- & RH-DAC)

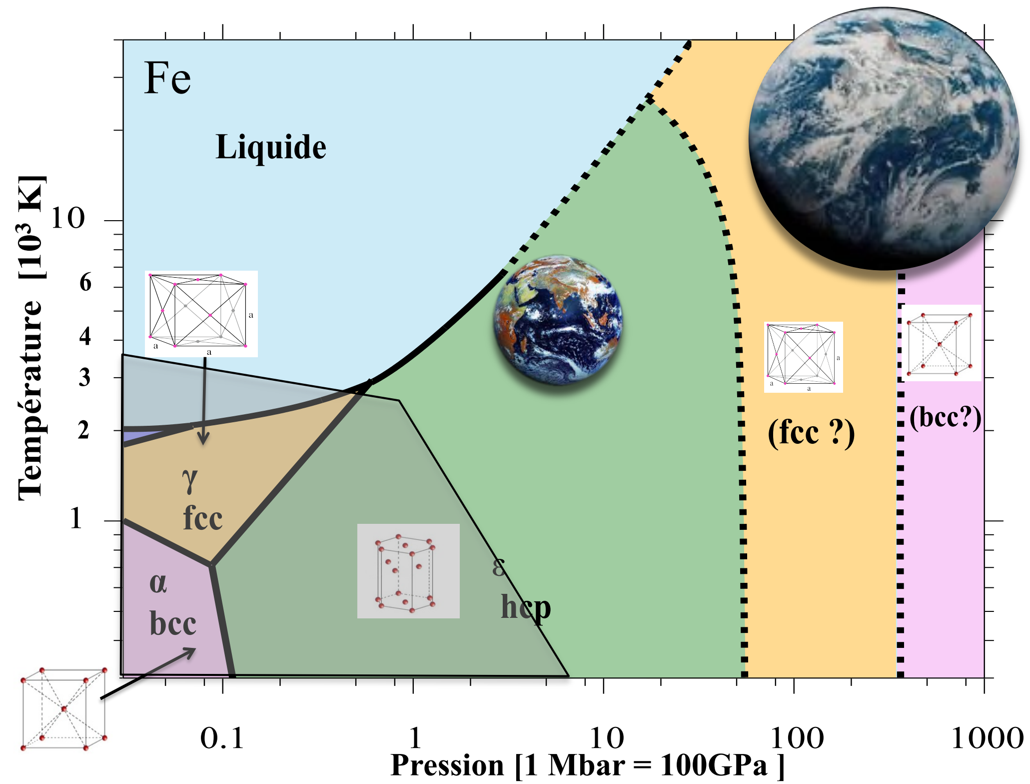


$$P = \frac{F}{S}$$

Dynamic compression and ultrafast heating with Lasers



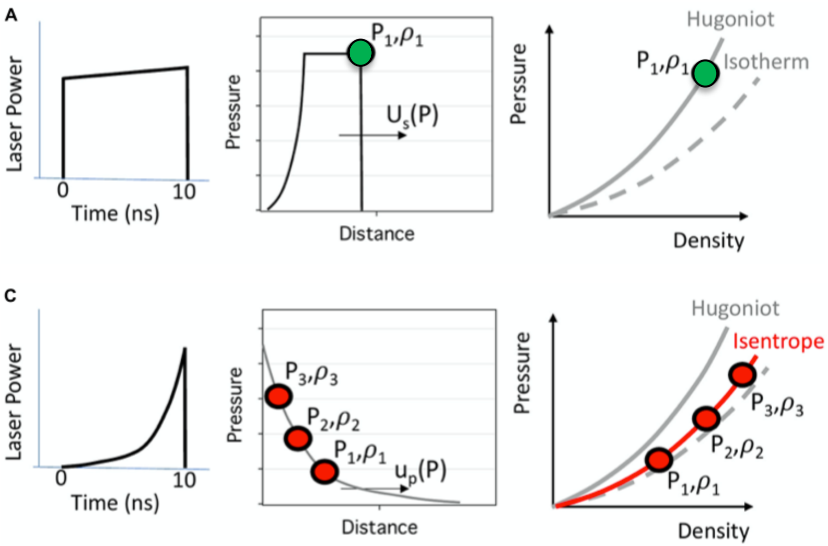
Modified from Stixrude 2014



Laser compression enables reaching extreme conditions of Pressure P, Temperature T (…and strain rates)

Explorer le diagramme de phase par compression laser

Modified from Duffy 2019



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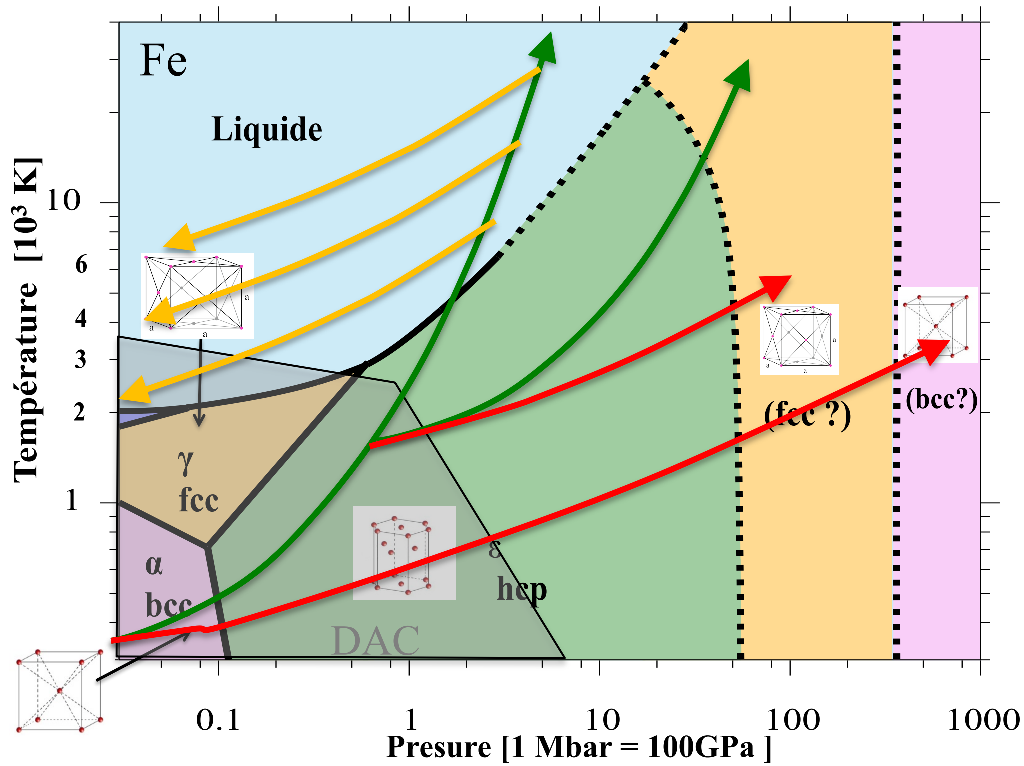
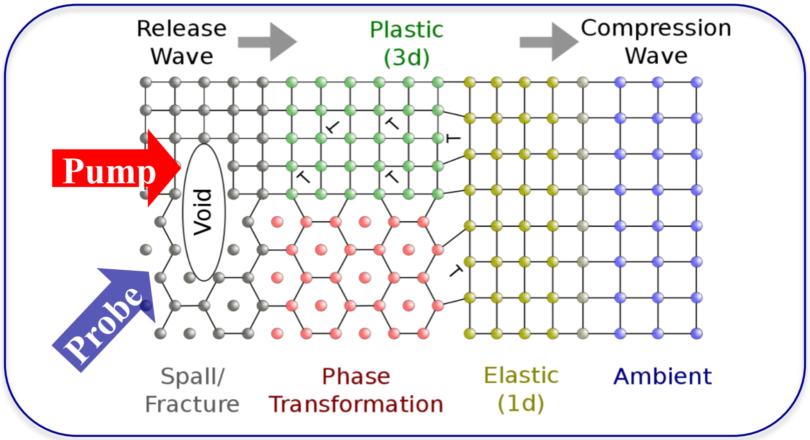


Ramp compression enables studying larger part of the phase diagram and more importantly at **low temperature**!
→ Fe at TPa by E. Brambrink's et al. (2023-2024)
→ Fe-O at TPa

Couple a structural probe with a high-energy laser

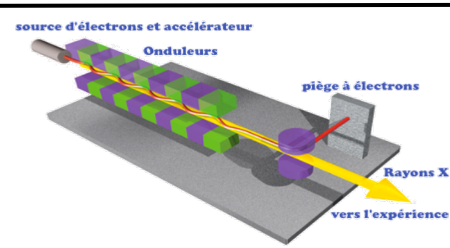
X-ray diffraction to probe in-situ phase transitons



Synchrotron

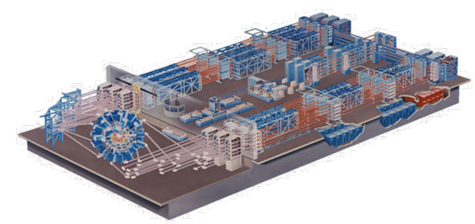
- Diagnostics developed since 40 years
- Synchrotron X-ray source > 10^7 photons / impulsion, Δτ ≈ 100 ps
- Recent implementation of ns lasers
- P-T domain is limited
- Mostly shock only (square pulse)
- Moderate repetition rate (100shots)

XFEL



- Recent facilities (≈ 10 years)
- XFEL X-ray source > 10^{12} photons / impulsion, Δτ ≈ 100 fs
- Recent implementation of ns lasers
- P-T domain is limited
- Mostly shock only (square pulse or very weak ramp)
- High repetition rates (> 1000shots)

Laser

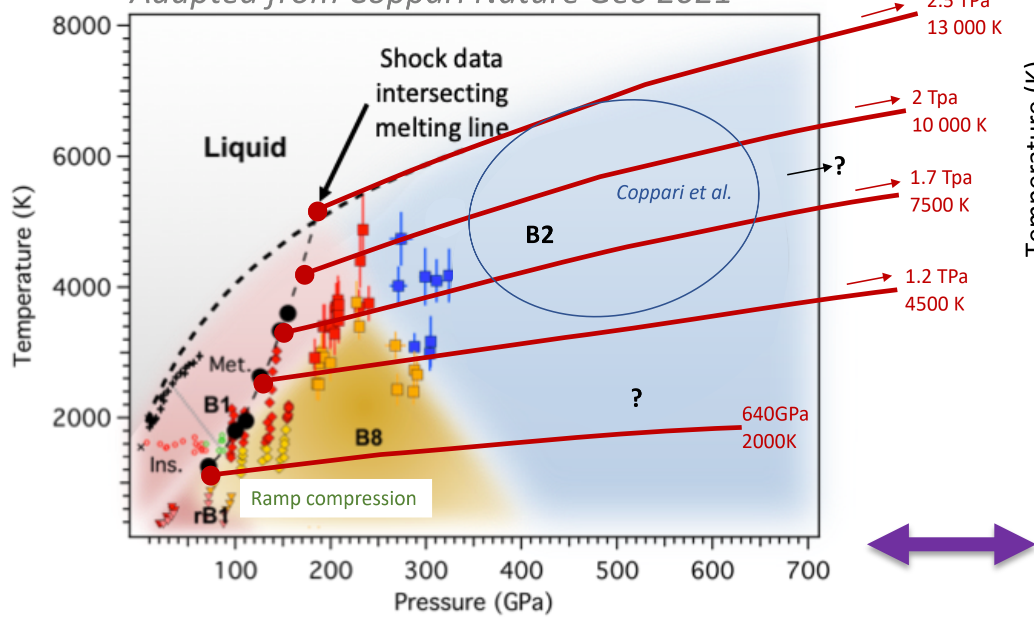


- Récent diagnostics, mostly XRD in transmission (OMEGA : 9 ans, NIF : 3 ans)
- Plasma X-)ray source: He_α emission (Δτ ≈ 1 ns), Kα emission to be improved (Δτ ≈ 1 ps)
- **Large P-T domain**
- High quality Shock and ramp
- Very little amount of shots

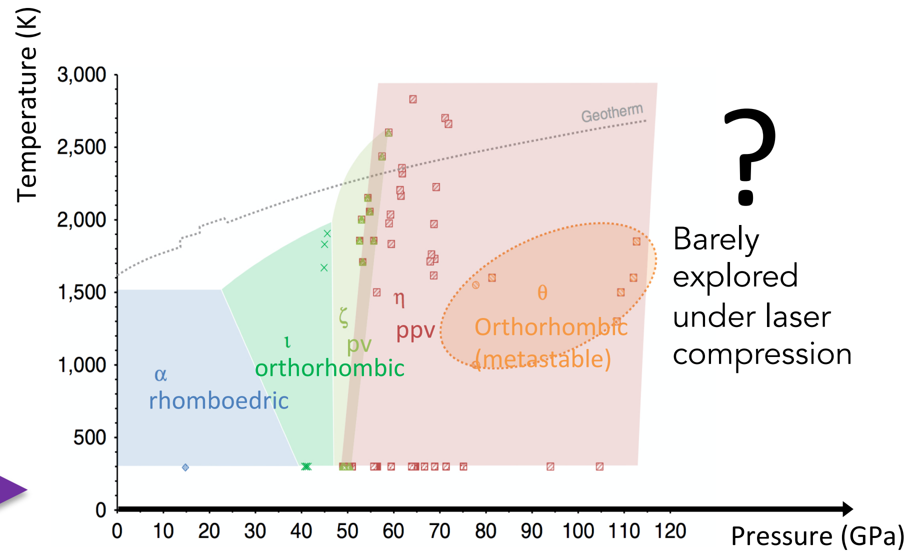
LMJ proposals on Fe-O

- Reach unexplored region of the Phase diagram (solid? Liquid? Cristallographic phases?)
- Study possible dissociation and/or dismutation of Fe-O (3FeO ←→ Fe_2O_3 + Fe)

Adapted from Coppari Nature Geo 2021



Adapted from Bykova Nature Com 2016



Preliminary steps

- Develop Target designs
- Explore Low pressure phase diagrams at XFELs and understand phase transitions processes using XRD
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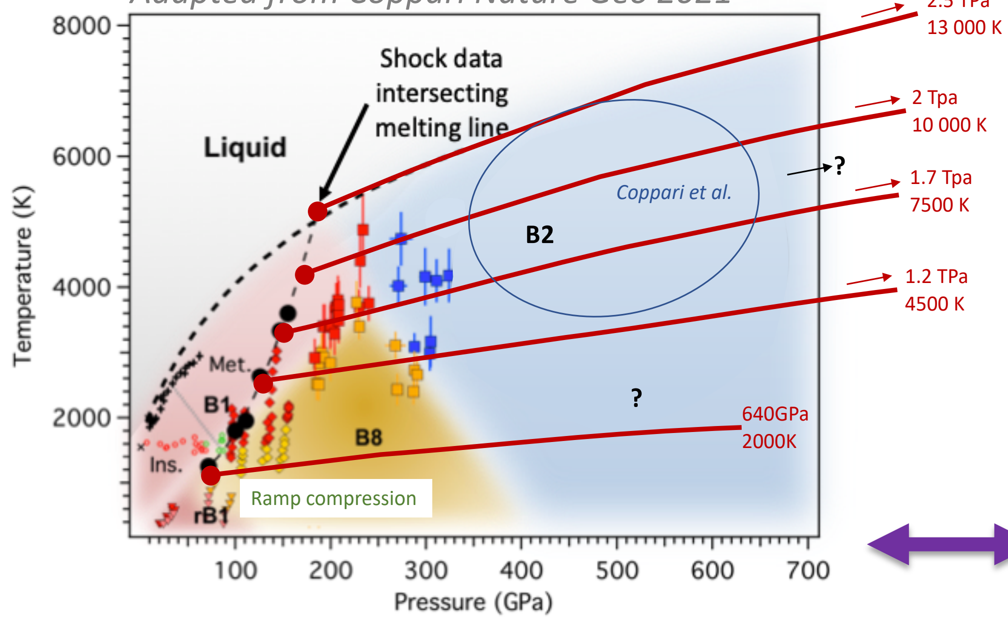

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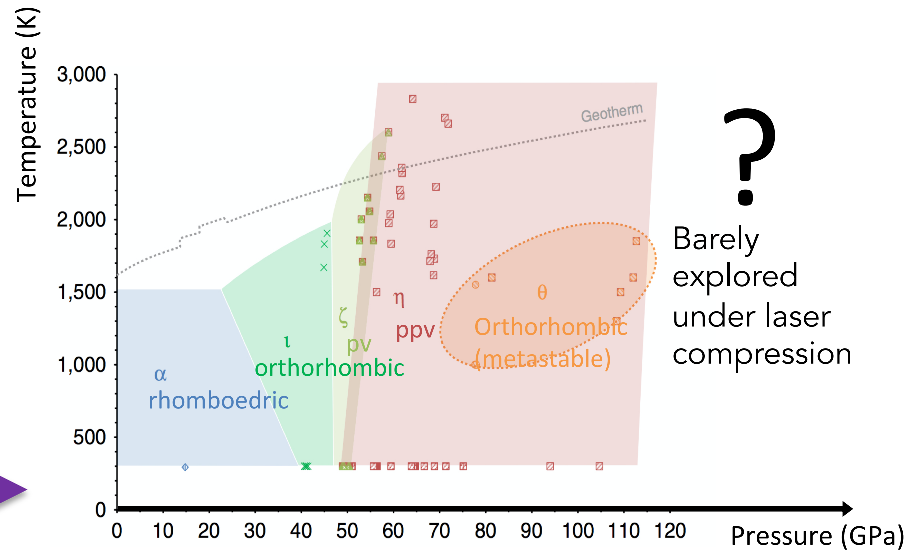

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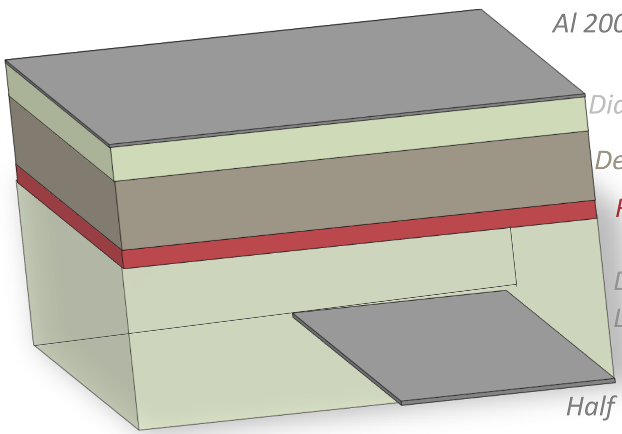
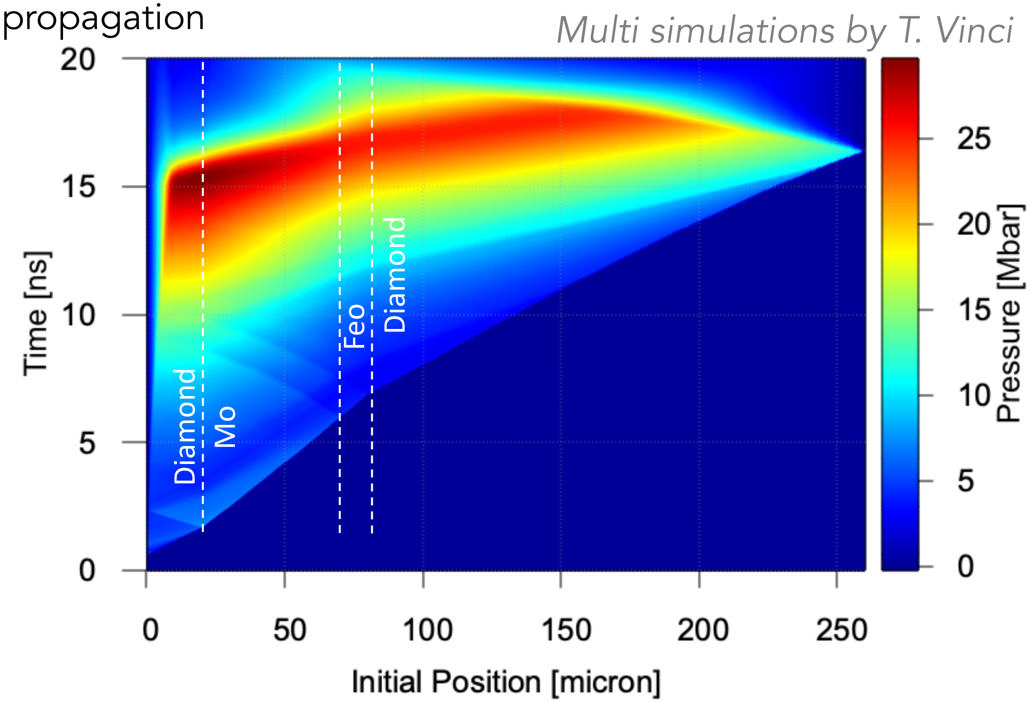
LMJ samples design and development

Multi layer samples

- → Ablator
- → Front plasma shileding
- → Half coated diamond optical Window for compression characterisation and maintaining P-T during the probe
- → Fe or Fe-O layer optimized for X-ray diffraction
- → Minimize glue layer to control the shock propagation



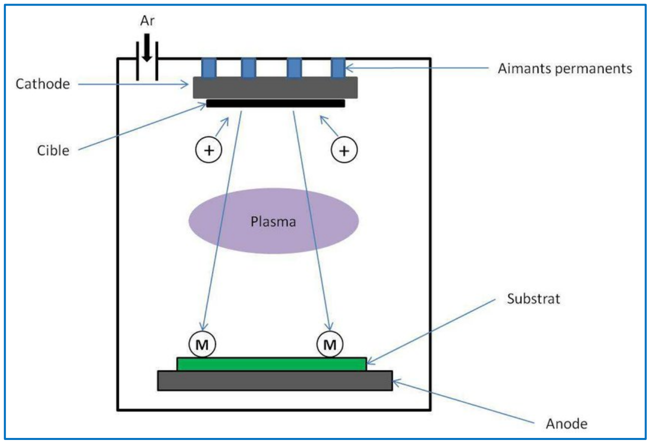


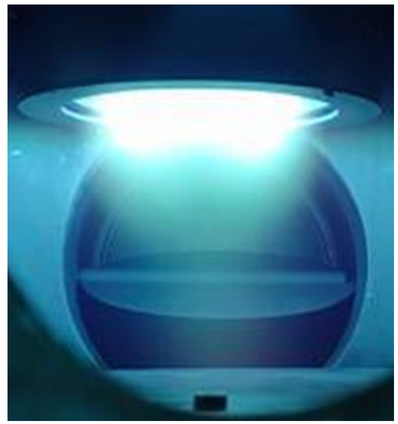
Understand (and possibly control) Phase transitions

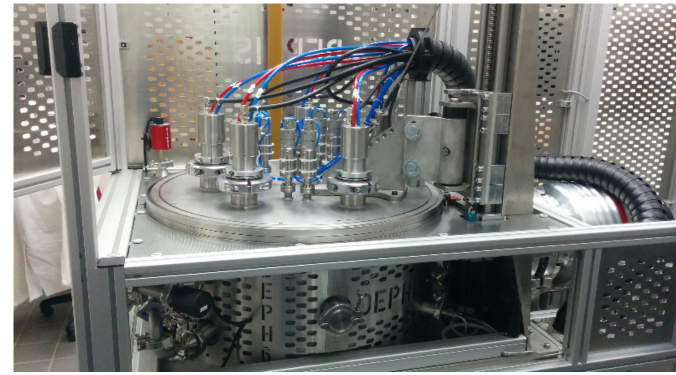
- → control the initial cristallographic phases
- → cristallographic orientations and textures
- → compositon and impurities

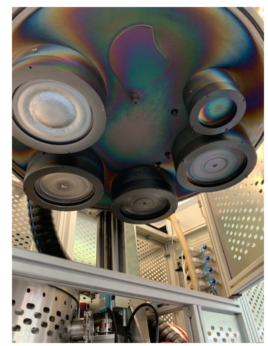
MIEL Platform: PVD deposition for target fabrication

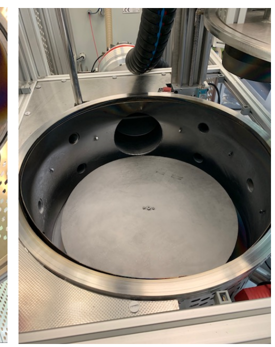
Synthesis of sample by Cathodique Magnetron Pulverisation








4 magnetrons


Rotating board


Large coated surfaces

- Thickness : few hundreds of nm to ~30µm
- Polycristallines samples or amorphous

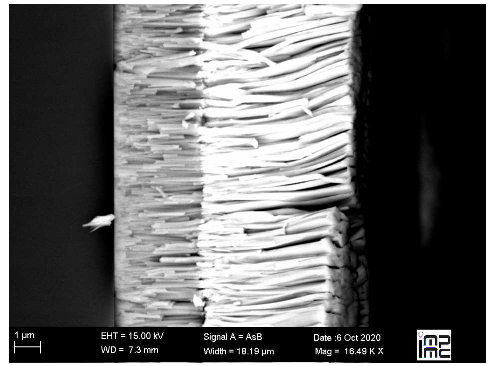
- Unique elements : Fe, Si, Al, Au, Ti, Mo, Ni, Ge…
- Fe alloys: Fe-Si, Fe-Si-C, Fe-Si-Mg, Fe-Si-O of variable composition
- Iron Oxides : Fe-O of variable composition(up 20 %), FeO, Fe2O3, Fe3O4 mixtures, (Fe,Mg)O of variable composition
- Other amorphous oxides : MgO, SiO, SiO2, MgSiO3, Mg-Si-O, Mg-Fe-Si-O of variable composition
- SiC

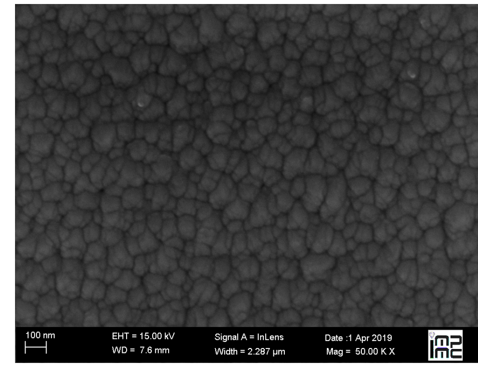
Planetary Diversity

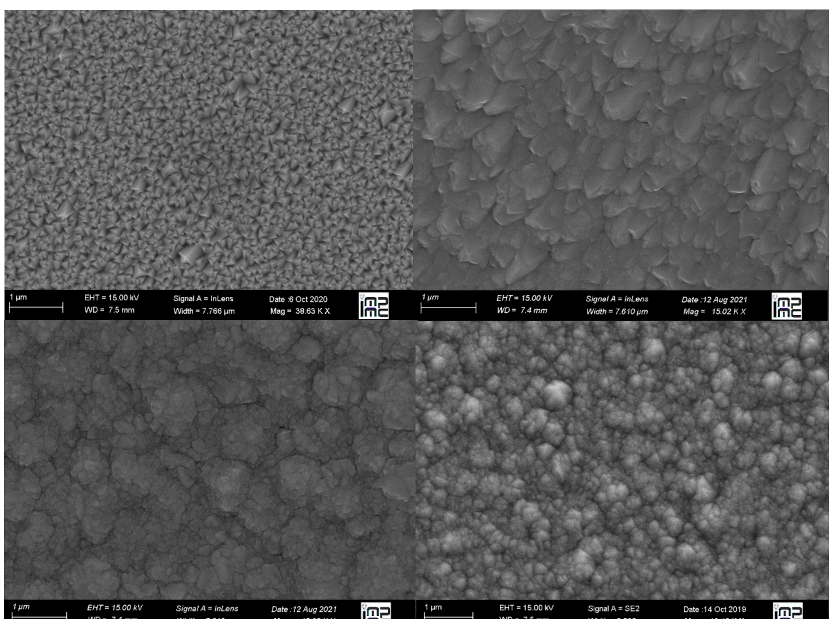
Characterisation of our starting materials

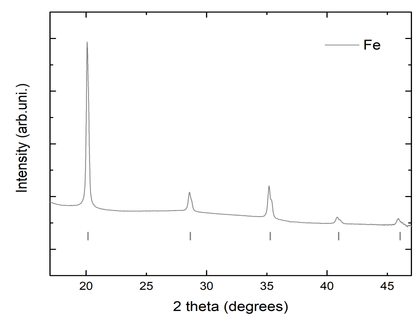
SEM – TEM
- Interface quality
- Preferential Orientation
- Composition
- Grain size

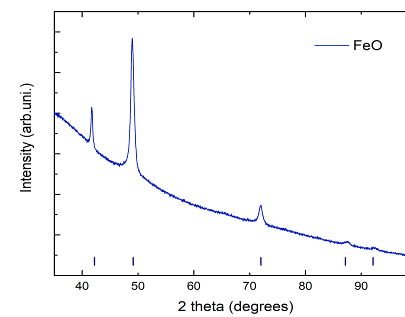
XRD
- Cristallographic phases
- Grain size
- Cristallinity
- Preferential Orientation
- XRD signal (2D and integrated)

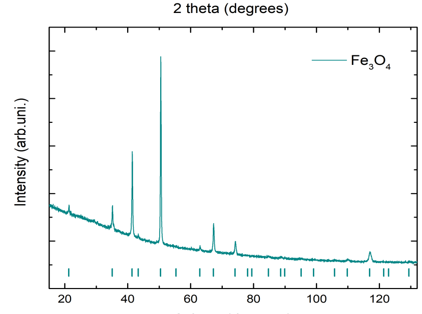


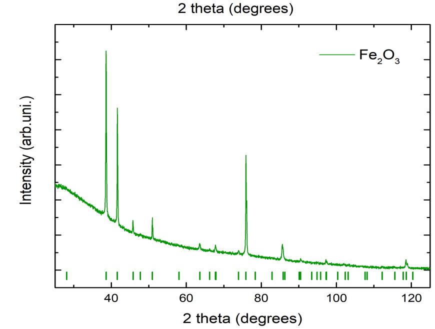












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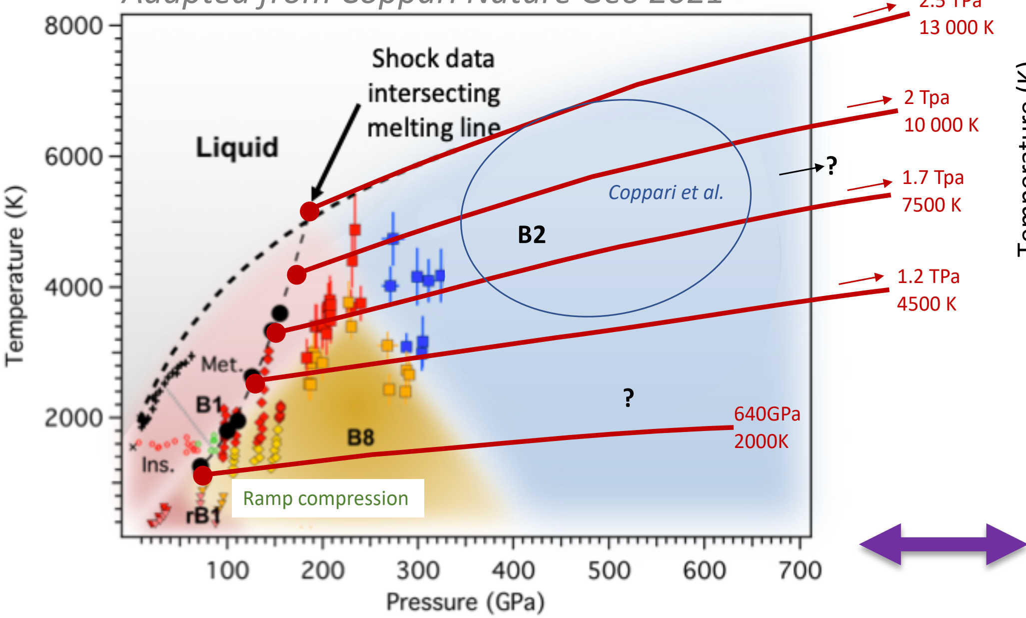
Adapted from Coppari Nature Geo 2021



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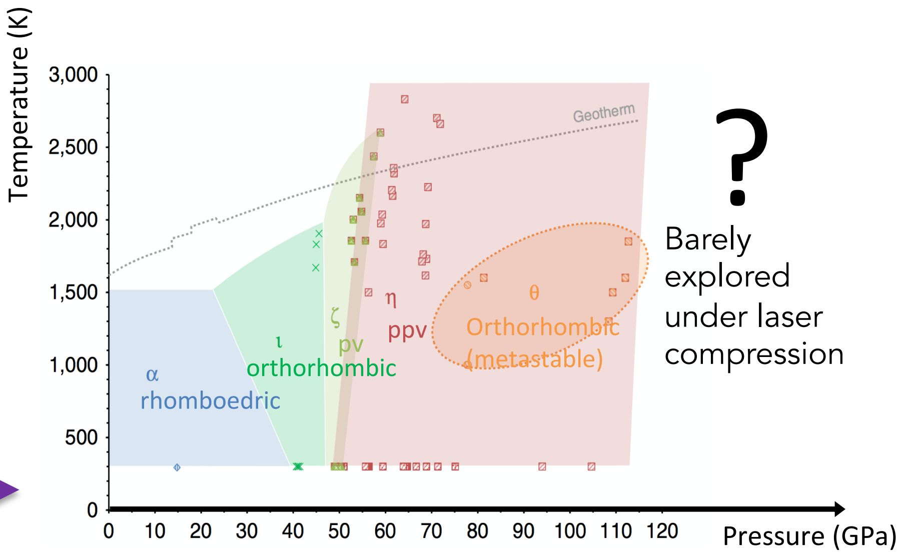

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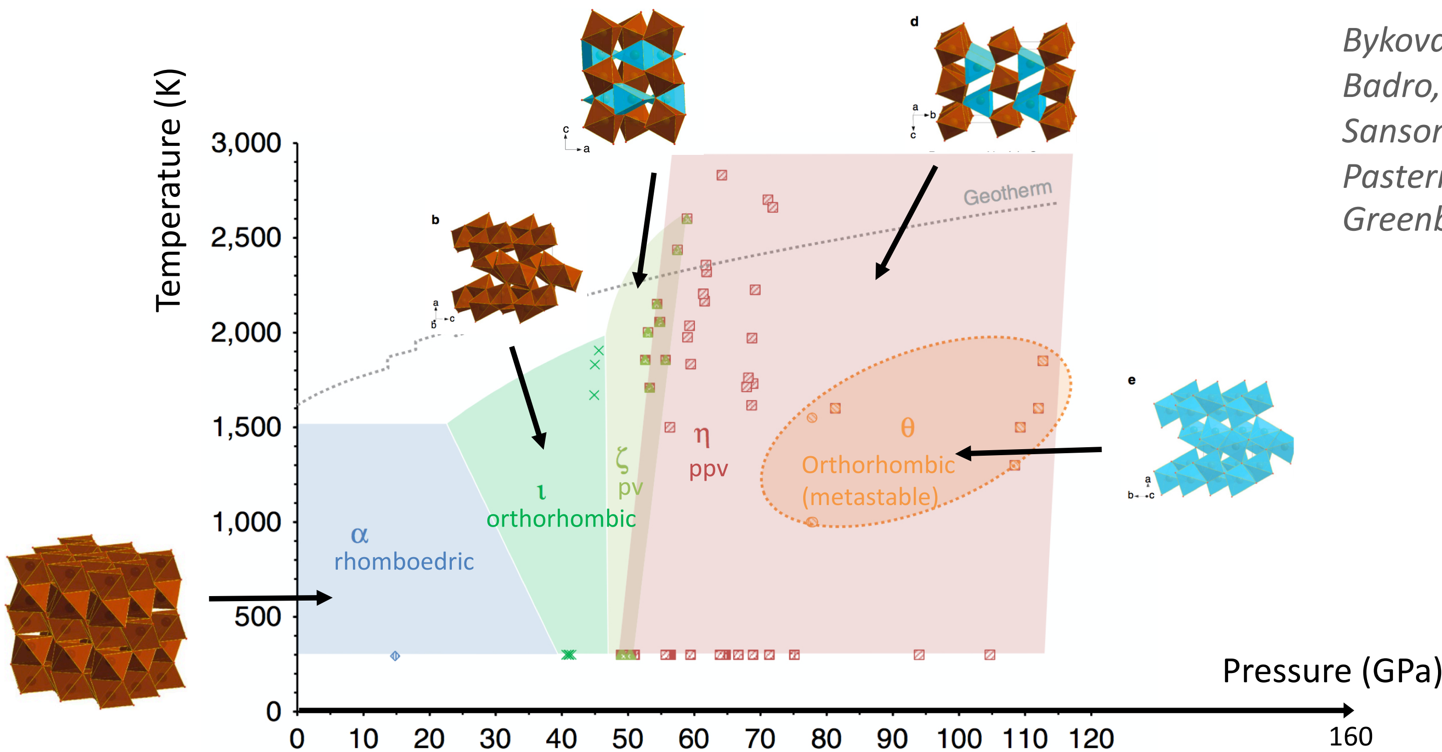
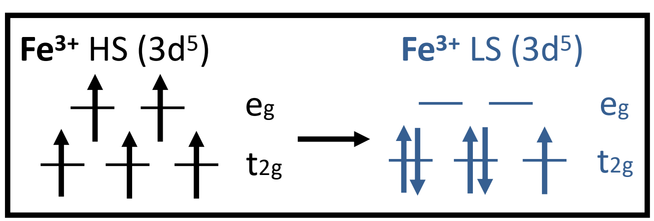
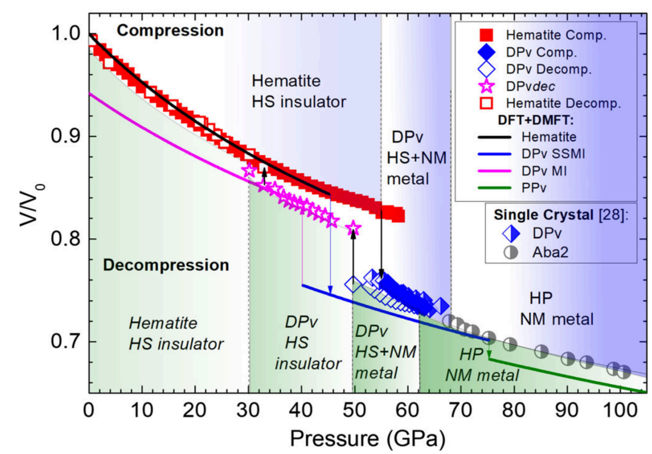
Fe_2O_3 phase diagram by LH-DAC

Bykova et al., Nat. Commun., 2016
Badro, Phys. Rev. Lett., 2002
Sanson, Phys. Rev. B, 2016
Pasternak, Phys. Rev. Lett.,1999
Greenberg et al. Phys Rev X 2018



Spin transition and Mott transition





10% volume collapse

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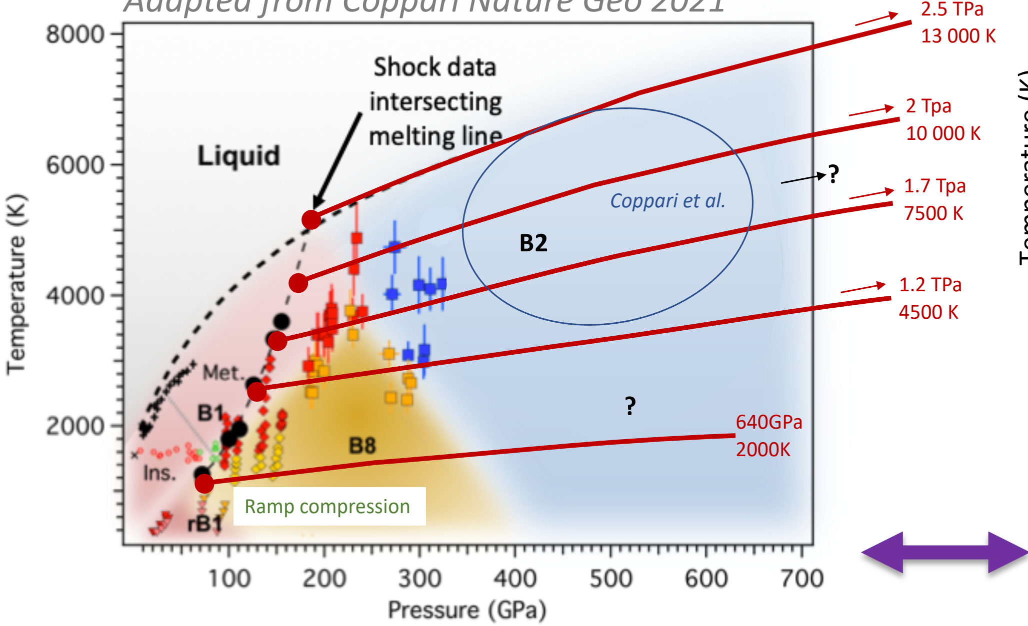

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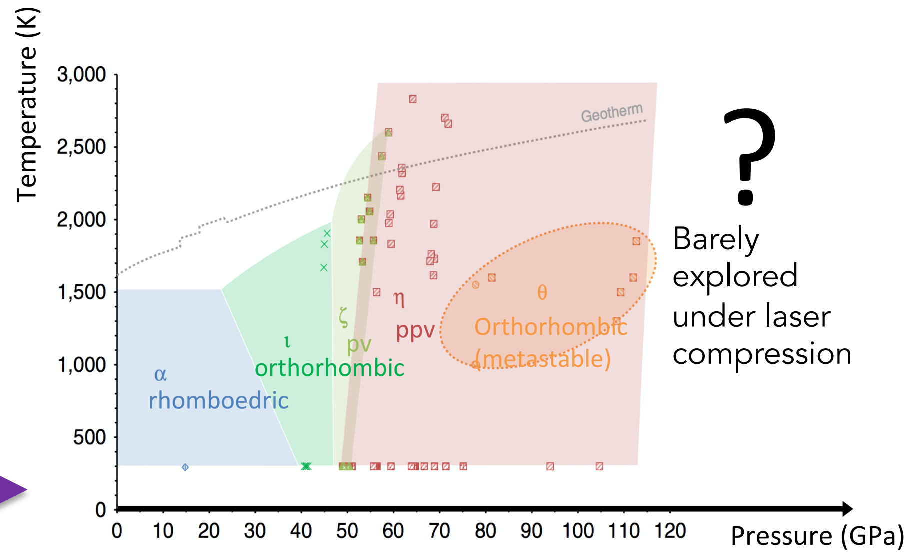

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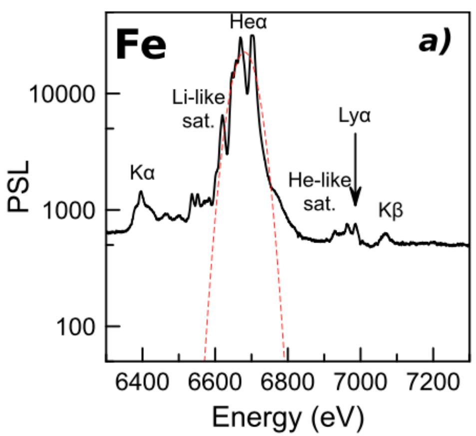
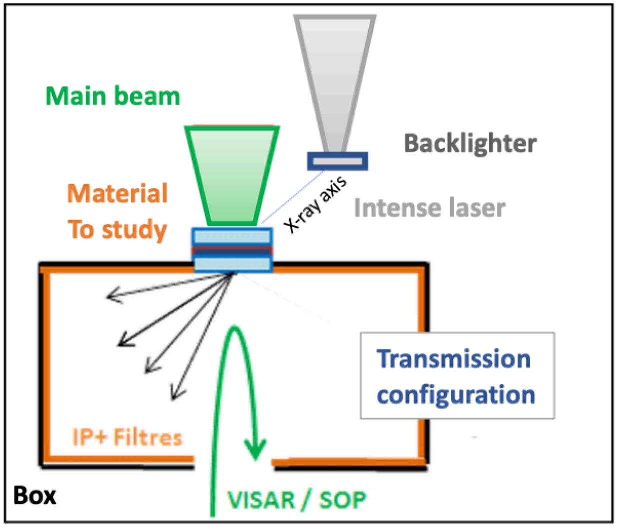
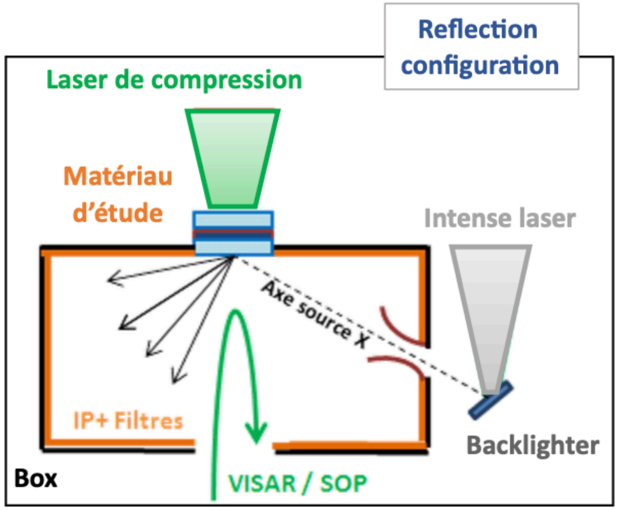
X-ray diffraction at LULI2000

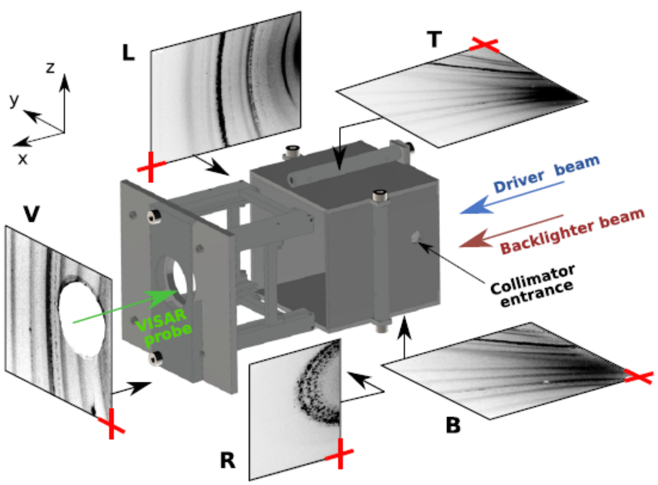
A. Denoeud et al.

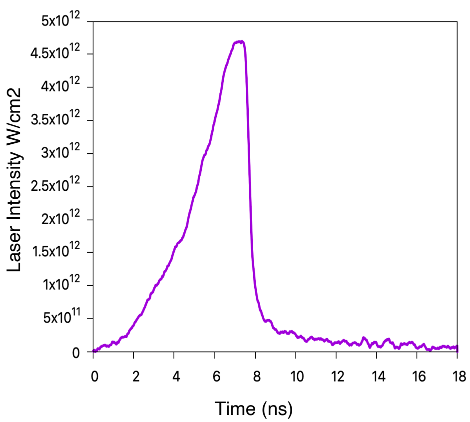
Courtesy A. Benuzzi-Mounaix

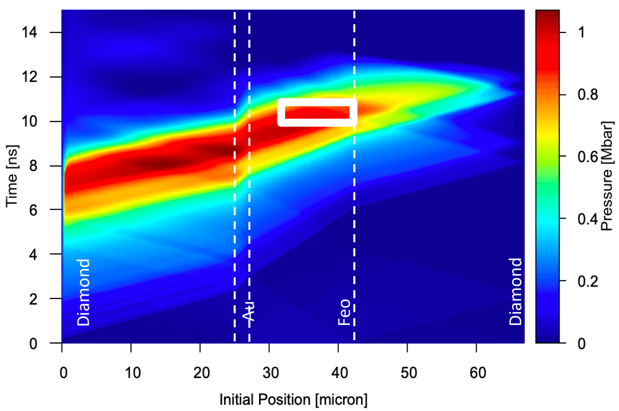








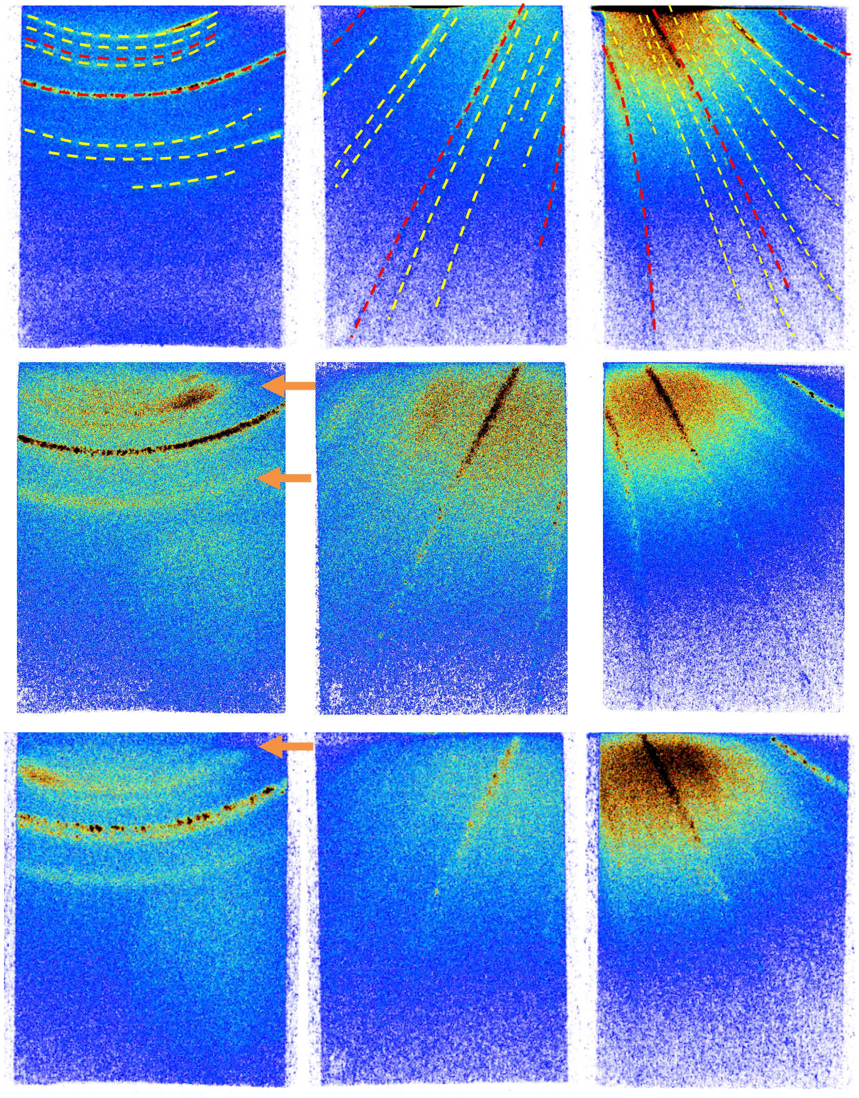




Possibility to couple XRD with laser ramp compression at LULI2000

A. Denoeud, J.A. Hernandez et al. RSI 2021
Multi Simulation by T. Vinci (LULI)

X-ray diffraction on FeO and Fe_2O_3 under ramp compression at ~60GPa



Shot 105 – Fe_2O_3 ambient

C* lines
Fe2O3 lines

Shot 114 – 184J
At breakout

Shot 113 – 197J
1ns after Breakout from Fe_2O_3

- Data under analysis
- The reflective XRD setup at LULI works very well

Collaboration with A. Denoeud et al.

X-ray diffraction at LMJ
Objectives: Fe at TPa pressures

E. Brambrink's campaign (XFEL)

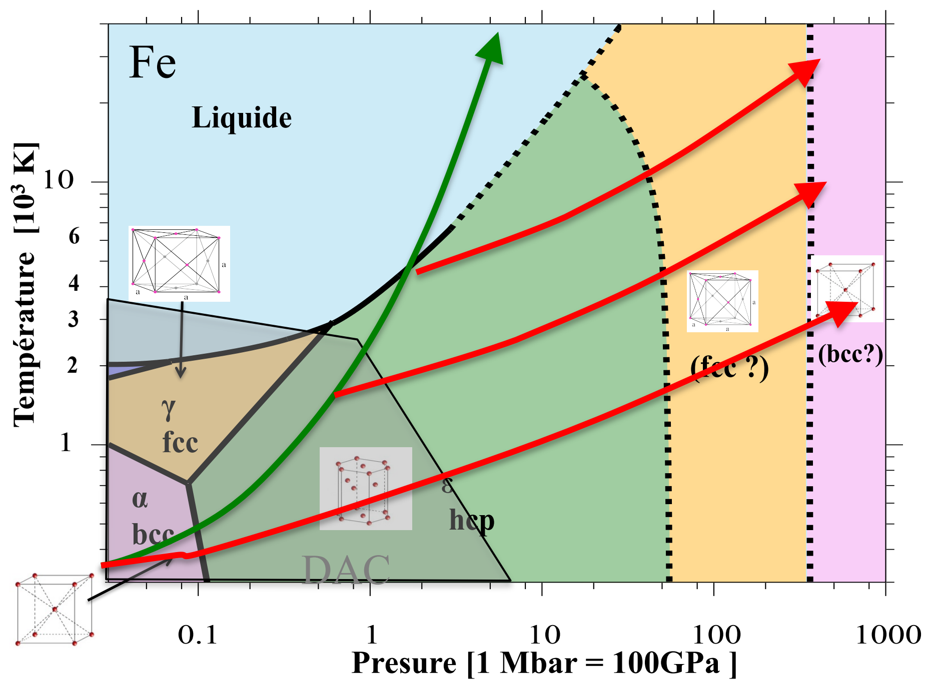
A. Denoeud, S. Brygoo, B. Fraisse, A. Sollier, L. Videau, C. Reverdin, B. Vauzour, A. Dizière, S. Debesset, P. Dupré, L. Le-Deroff, T. Caillaud, N. Blanchot, L. Jacquet, J.-P. Perlat, D. Hébert, P. Pradel, J. Martinez, J.-M. Chevalier, L. Bitaud (CEA)

J.-A. Hernandez (ESRF)
A. Benuzzi-Mounaix, T. Vinci, A. Ravasio, M. Koening, A. Berlioux, J. Houy (LULI),
M. Harmand, G.Fiquet, F. Guyot, G. Morard (IMPMC)

T. de Resseguier (Pprime)
G. Gregori, L. Chen (University of Oxford)
A. Higginbotham (University of York)
D. Riley (University of Belfast)
T. Boehly (LLE)
N. Ozaki, K. Miyanishi and R. Kodama (Osaka university)

G. Chabrier (ENS Lyon)
J. Vorberger (HZDR)
D. Gericke (University of Warwick)
R. Redmer, D.Cebulla (University of Rostock)

Modified from Stixrude 2014

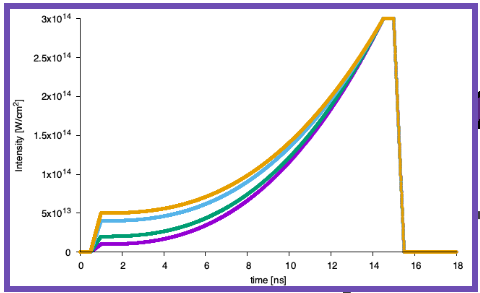


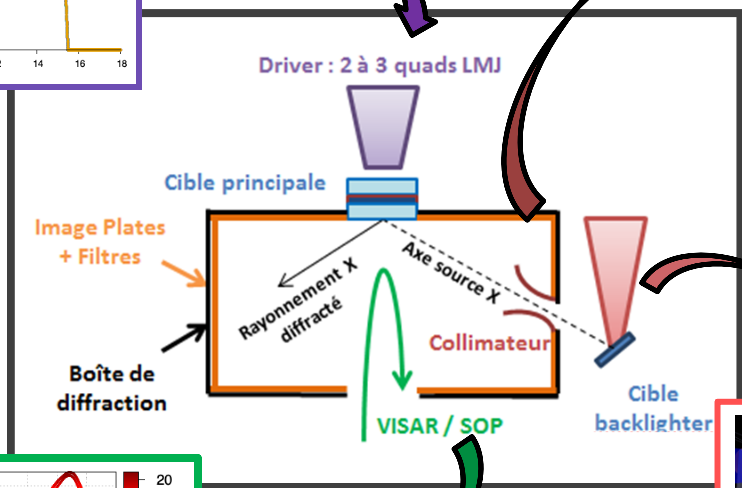
X-ray diffraction at LMJ

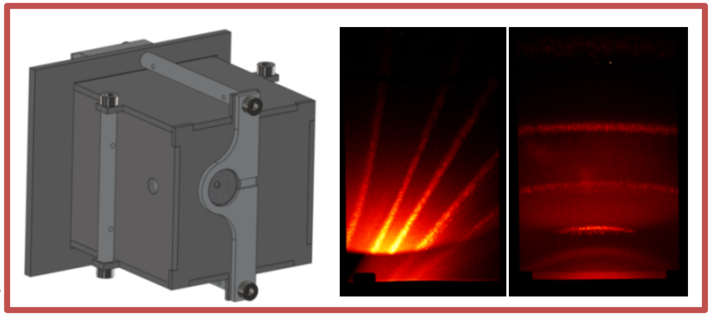
Experimental approach

Driver : Shock compression + laser ramp

- *Long duration temporal profile, « exotic » for LMJ*
- *Fully understand the shock + ramp thermodynamic path in relation with the laser temporal profile*

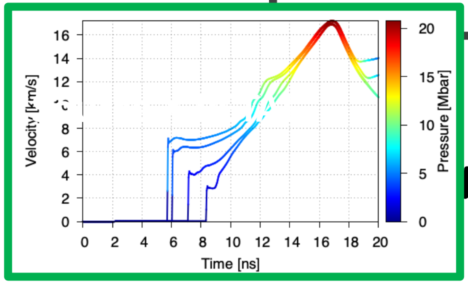






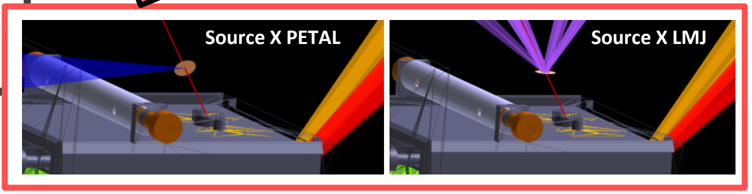
Principal Diagnostic 1 : XRD in reflexion

- *High quality Signal to noise ratio: external shielding in WC + internal shielding of the main target (Mo)*
- *Follow up of the LULI developpements*
- *Heavy and takes place in the LMJ chamber: real challenge to integrate it and align it at LMJ*



Principal Diagnostic 2 : VISAR (ω/2ω) + SOP

- *Measure the thermodynamic path (P-T) independantly from the X-ray diffraction*
- *Time of the probe and Compression time*



Probe : High flux X-ray source

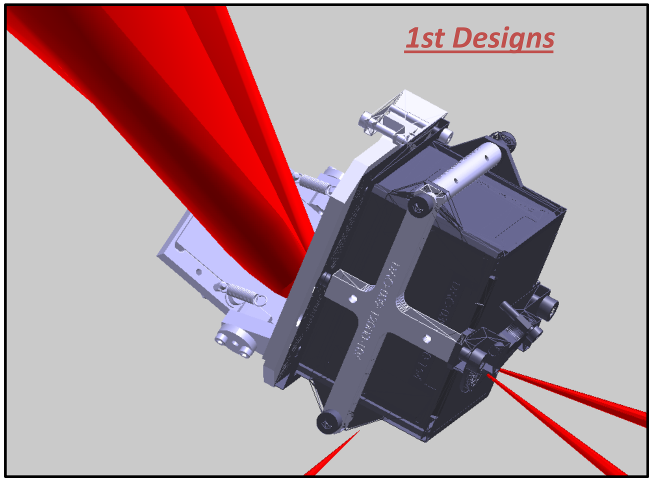
- *X-ray generation using PETAL beam (Kα) or 6-7 LMJ quads (Heα)*
- *1st part of the experiment: test the X-ray source*
- *X_ray spectra and flux characterized by SpectiX, DMX and Bremsstrahlung diagnostics*

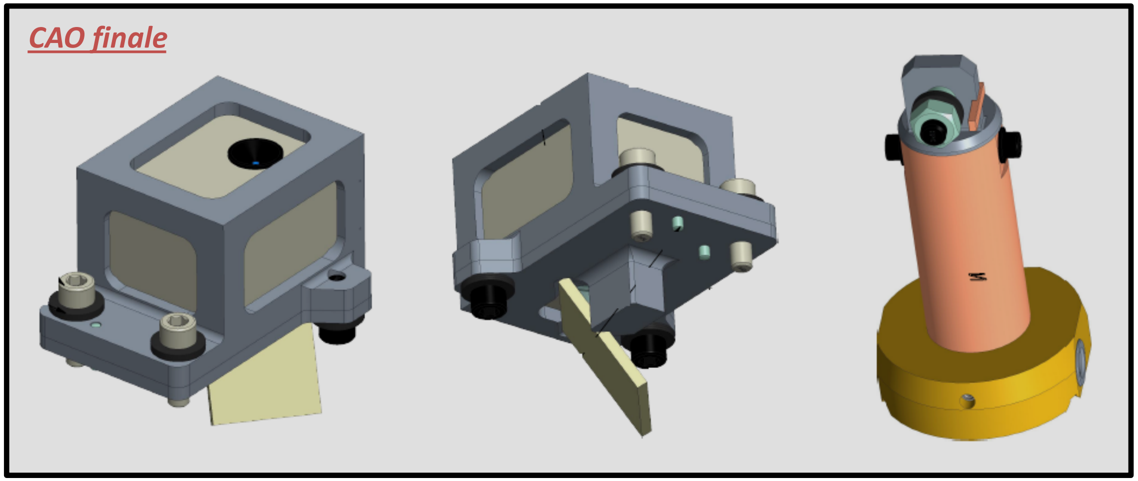
X-ray diffraction at LMJ
Improvements and adaptation of the XRD setup

Technical and physical challenges:

- Adaptation to the LMJ-PETAL environment
- Size and weight constrains
- Holding and alignement constrains
- Compatibility with Irradiation & simultaneoux pointing of the LMJ / PETAL / EOS-Pack beams
- Materials must be PETAL-compatible
- Debris Constrain

2 major modifications:
- Reduce the size of the box
- Remove all screws





X-ray diffraction at LMJ
1st shots results - E.Brambrink's campaign

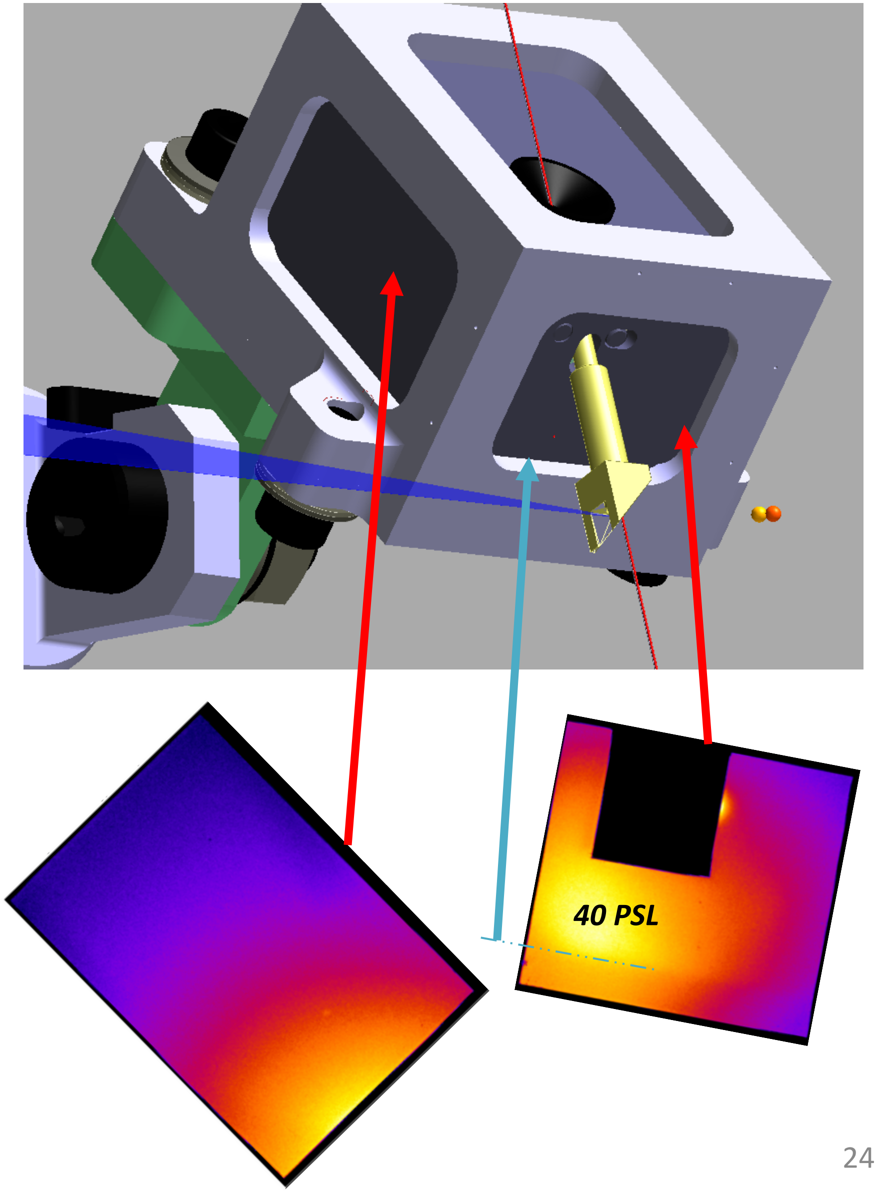
1st Part

→ 2 shots without compression : Study the X-ray source and the external shielding
- 1 shot with K_α (PETAL, 10 ps)
- 1 shot with He_α (LMJ, 700 ps, 6-8 quads)

→ 1 shot with compression : test the internal shielding of the main target (Mo)

2nd Part

→ 4 shots with different compression schemes (modifying the laser energy, the pulse shape and the initial shock)



1st shot with PETAL X-ray source
- Very high background
- coming from an area close to the backlighter, outside the XRD box
- Most probably due to hot electrons (no filtering possible at this disctance)
- Very different background than the LULI experiment

Conclusion and Perspectives

- Tremendous work and expertise by the CEA team to make this diagnostic work at LMJ!
- Follow up of a community work on XRD under laser compression at LULI2000

- Complementary with other X-ray sources to achieve different regime of pressure
- Need to better understand phase transformation under shock
  → X-ray time resolved experiments are opening new perspectives
  → simulation are required
  → specificity of the phase transitions under ramp compression

- Need of intermediate facilities between…

Specific conclusions for our future LMJ experiment

- Alignement method of the diffractometer is validated
- PETAL X-ray source

  Weak conversion for the Ge Kα @ 10keV : ~ 10^{-5} → 1000x less flux on target than at LULI2000

  Intense background from the outside of the XRD box → hot electrons ? Could be explained by a pre-pulse

  The background is 10x more intense than the LULI2000 → no possibility to shield (too heavy, not enough space)

  → The PETAL Ka X-ray source is not adapted

- Next test: LMJ X-ray source

  1 QUAD would be enough with type F phase plate to reproduce LULI flux

  3 and 6 QUADS → x7 to 45 flux

Collaborations for the presented results

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Thank you for your attention!

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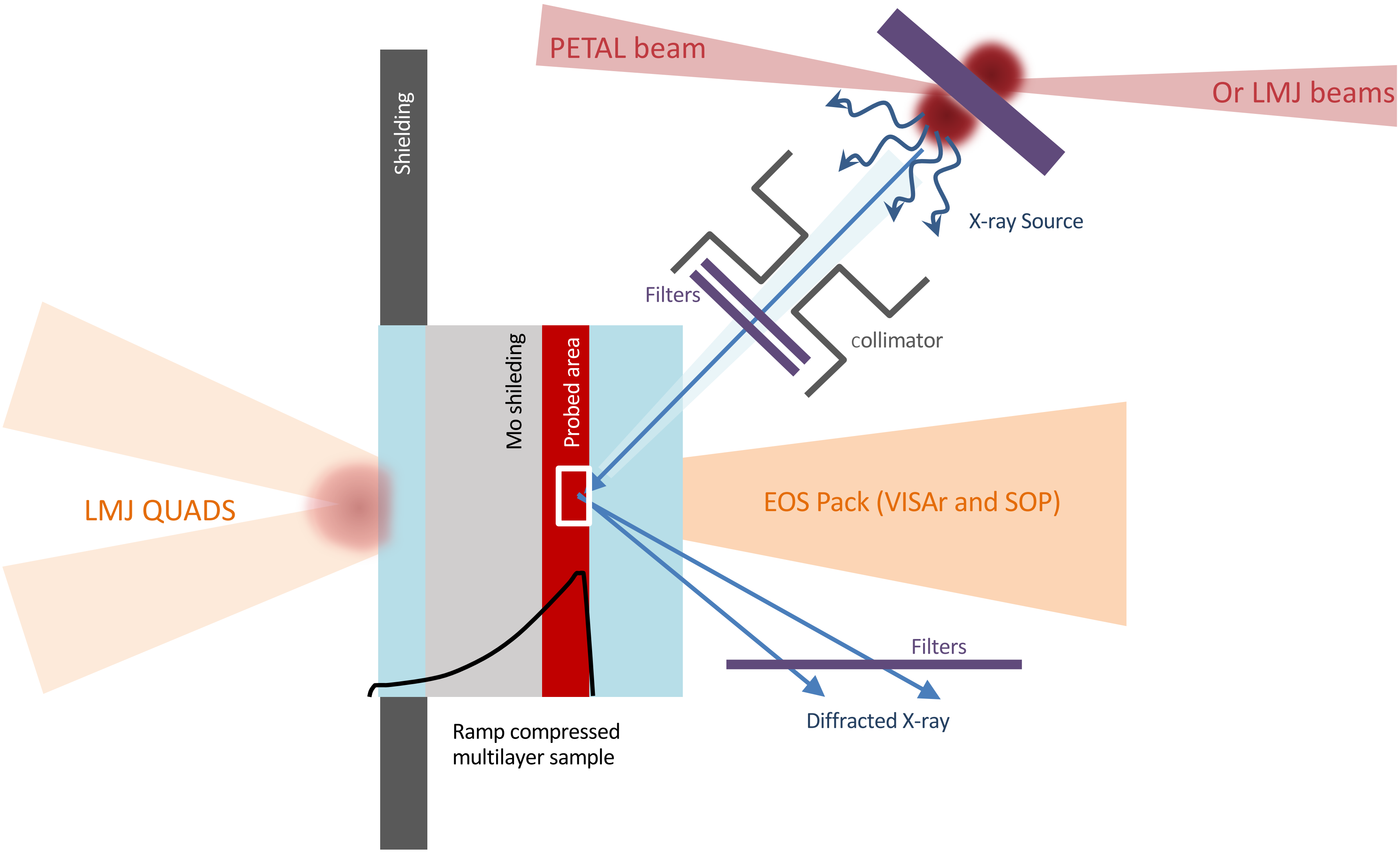

PETAL beam
Or LMJ beams
Shielding
X-ray Source
Filters
collimator
Mo shileding
Probed area
LMJ QUADS
EOS Pack (VISAr and SOP)
Filters
Diffracted X-ray
Ramp compressed
multilayer sample