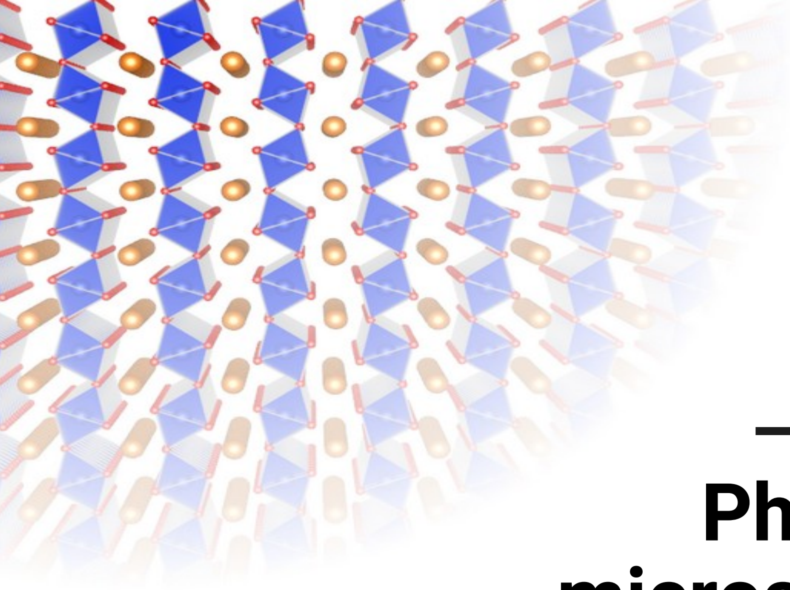


Université de Lille

Phase transformation and deformation microstructures in Earth's mantle minerals up to 110 GPa and 2000 K using multigrain X-ray crystallography

S. Merkel

Univ Lille, France, Lille, France

J. P. Gay, E. Ledoux, M. Krug, S. Speziale, C. Sanchez-Valle

Univ. Lille, Univ. Münster, GFZ Potsdam

TIMEleSS

ANR DFG

Phase TransformatIons, MicrostructurEs, and their Seismic Signals from the Earth's mantle


S. Merkel


C. Thomas


C. Sanchez Valle


S. Speziale

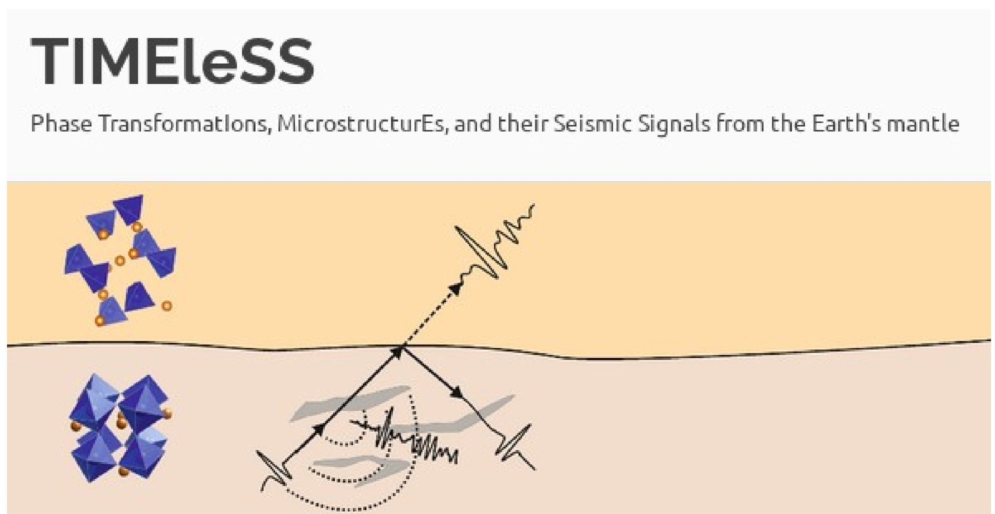

F. Rochira


J. K. Magali


M. Krug


E. Ledoux


J. Gay



Active research finished period finished Dec. 2022
Papers still being published now

Transport in the Earth mantle

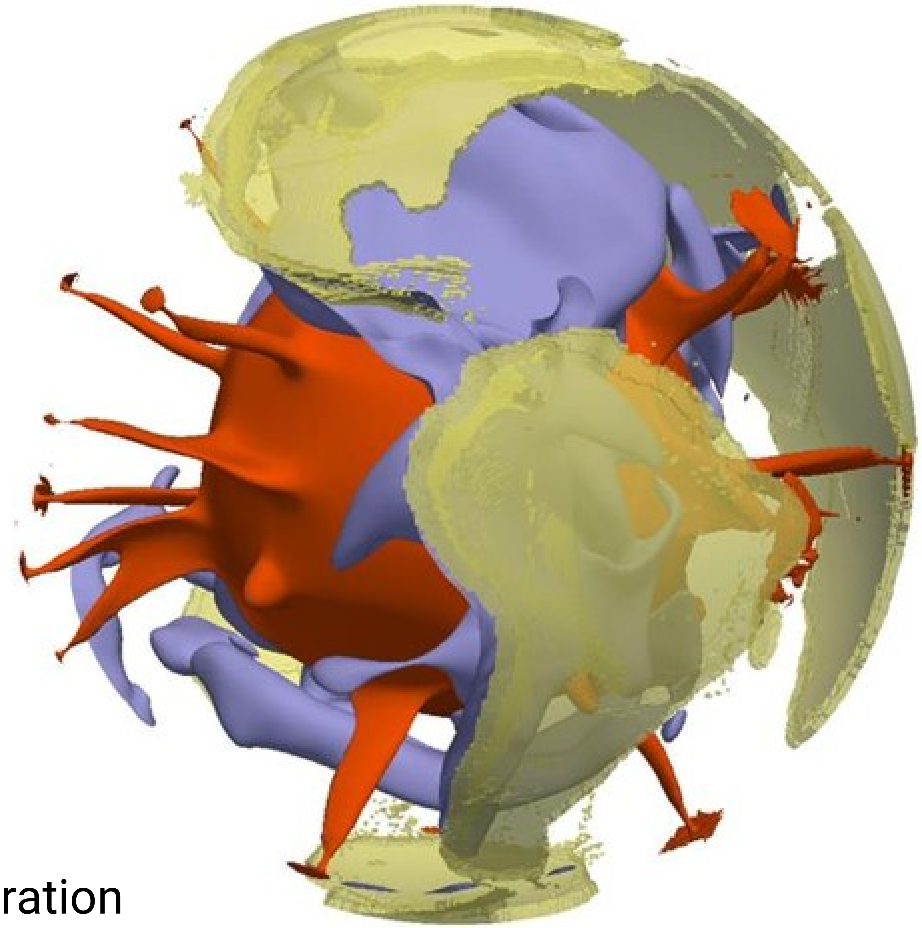


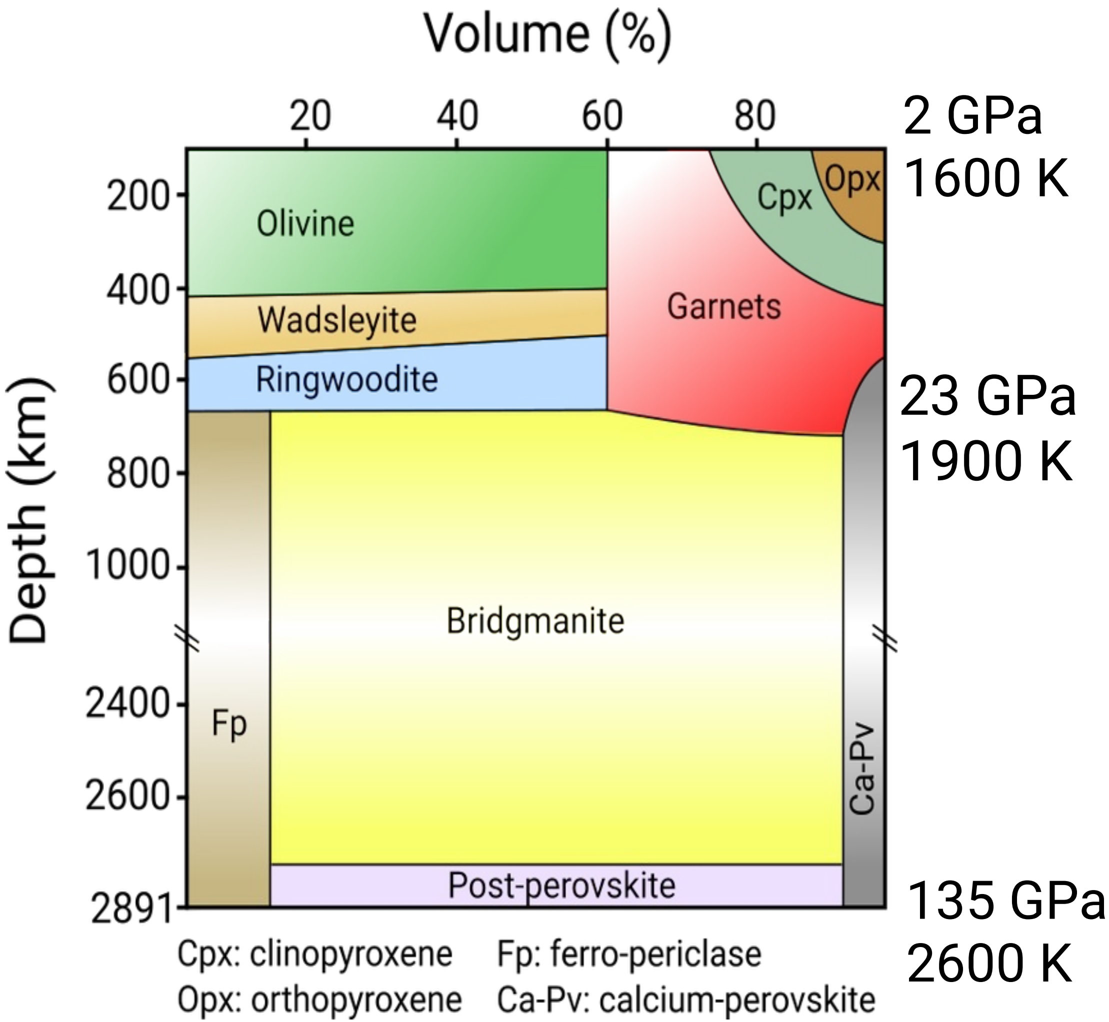
Illustration
N. Coltice, Univ. Nice

Heat transport in the Earth's mantle
- Conduction in upper and lower boundary layer (lithosphere and D")
- Solid-state convection

Velocities: cm/yr

Process: solid state deformation
- Dislocation glide
- Dislocation climb
- Diffusion
- Grain boundary sliding
- Etc

Phase transformations in the Earth's mantle



Main phase transitions in a pyrolitic composition

Sharp transitions at

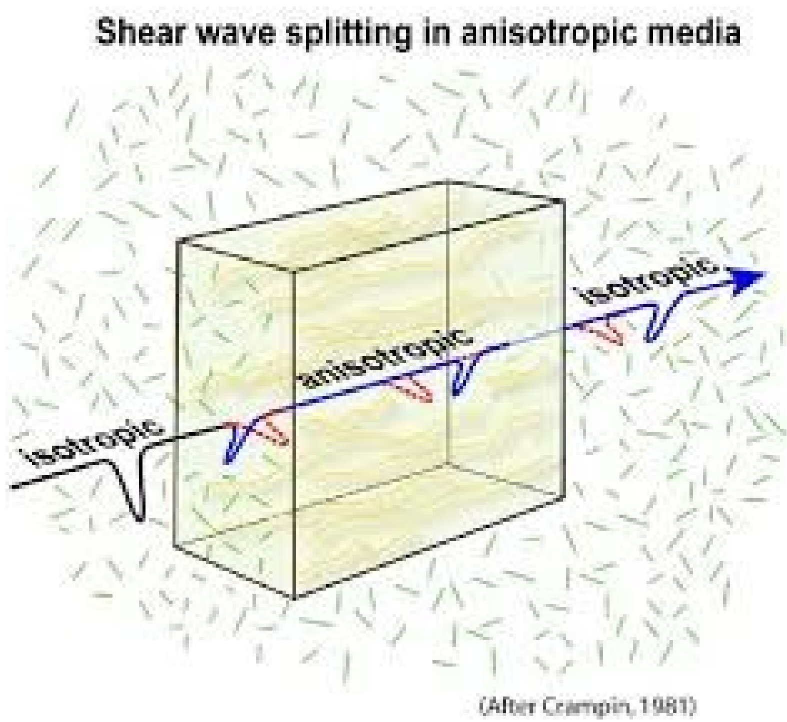
- 410 km (12 Gpa)
- 660 km (23 Gpa)

Regions of particular interest for geophysical cycles

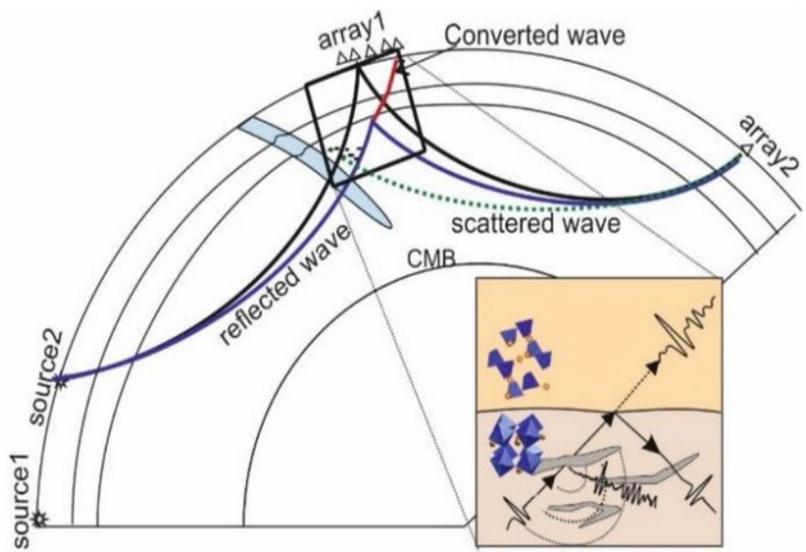
- Transition zone (410–660 km)
- D" (2800 – 3000 km)

Geophysical observations

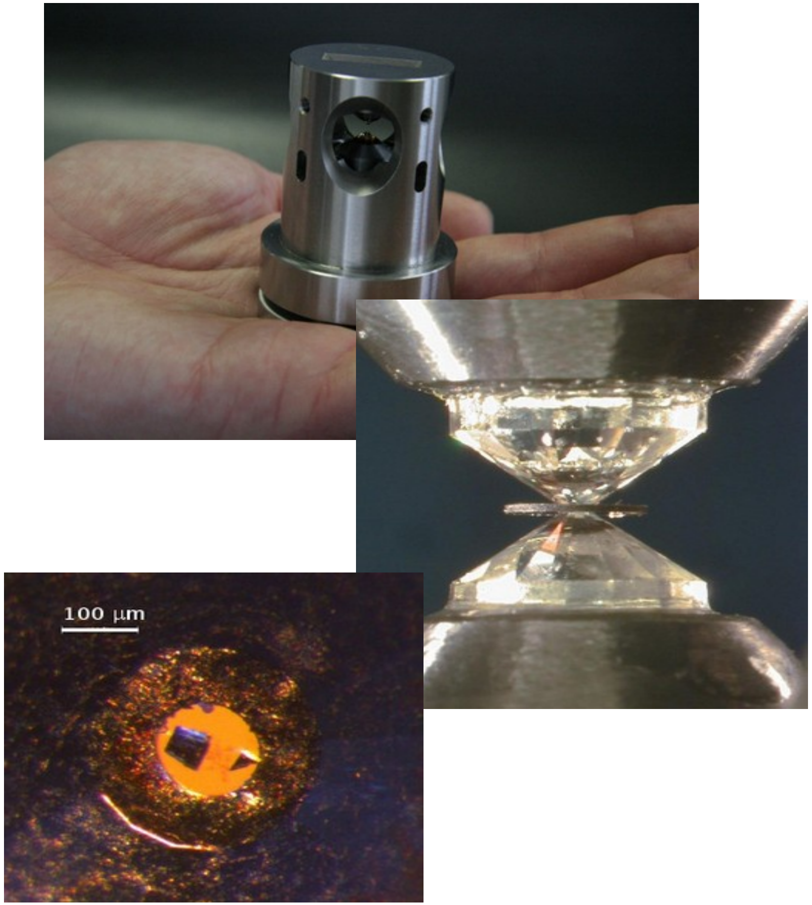
Sharp transition of physical properties
- Reflectors
- Wave conversions

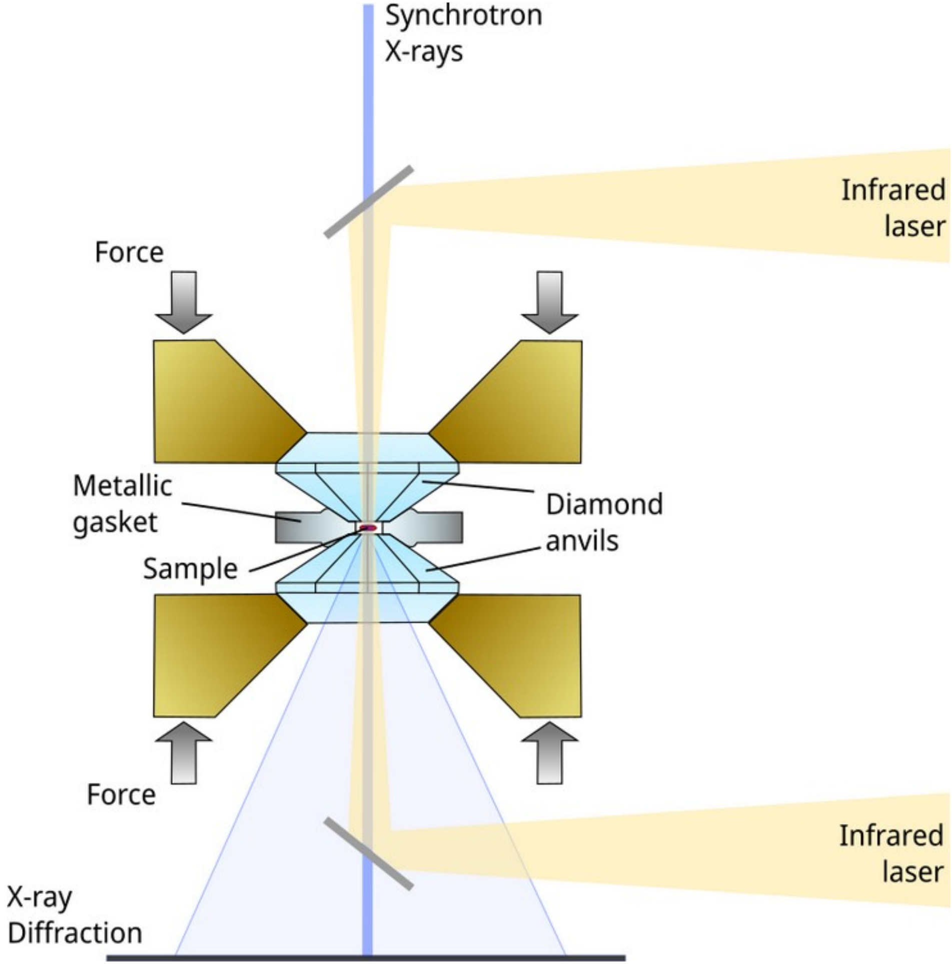


Microstructural imprint
- Texture
  - Anisotropic velocities
  - Splitting for shear wave polarities
- Waveform and polarity of reflected / converted waves



High P/T Experiments on Polycrystals
Diamond anvil cells

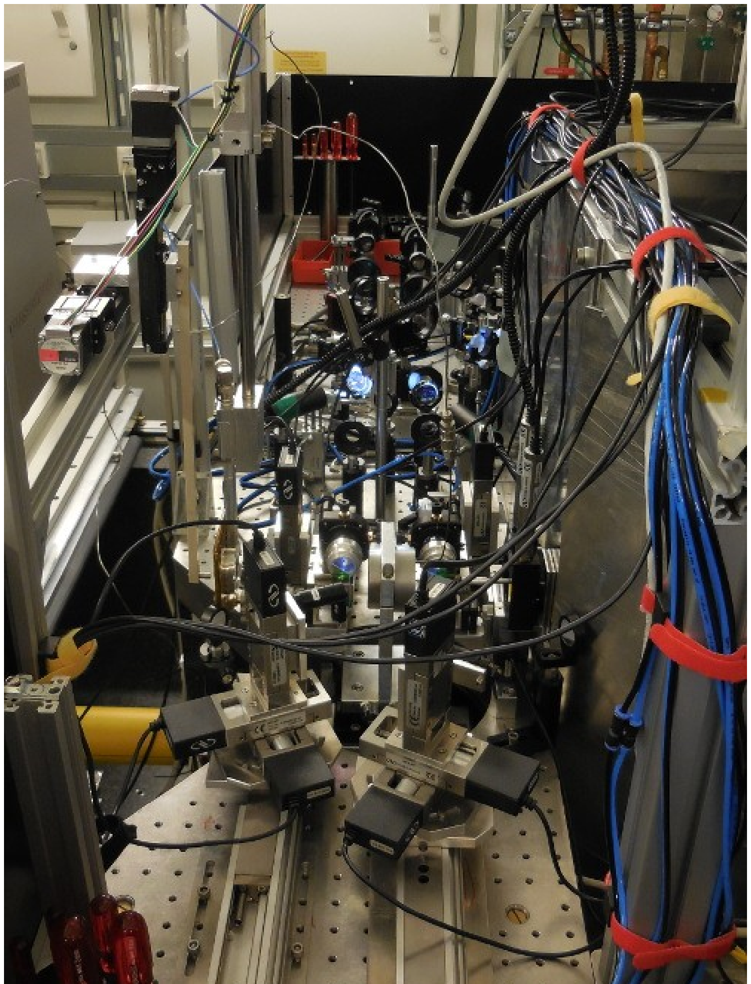


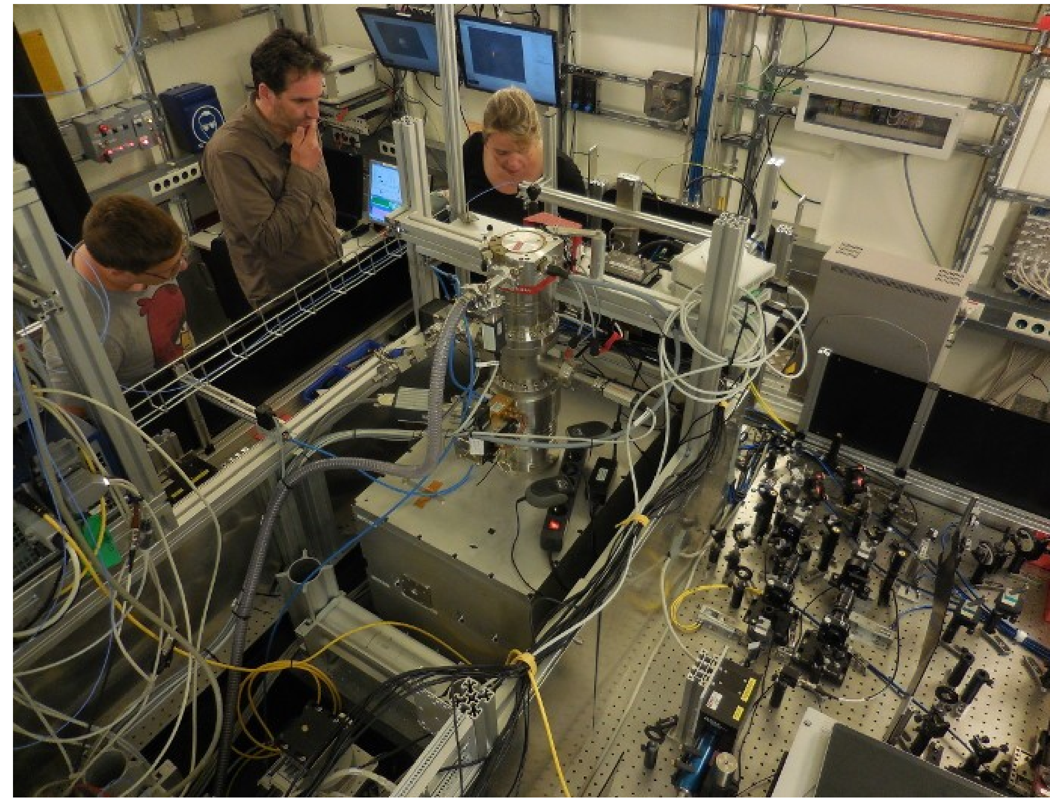


Diamond anvil cells @ Univ Lille

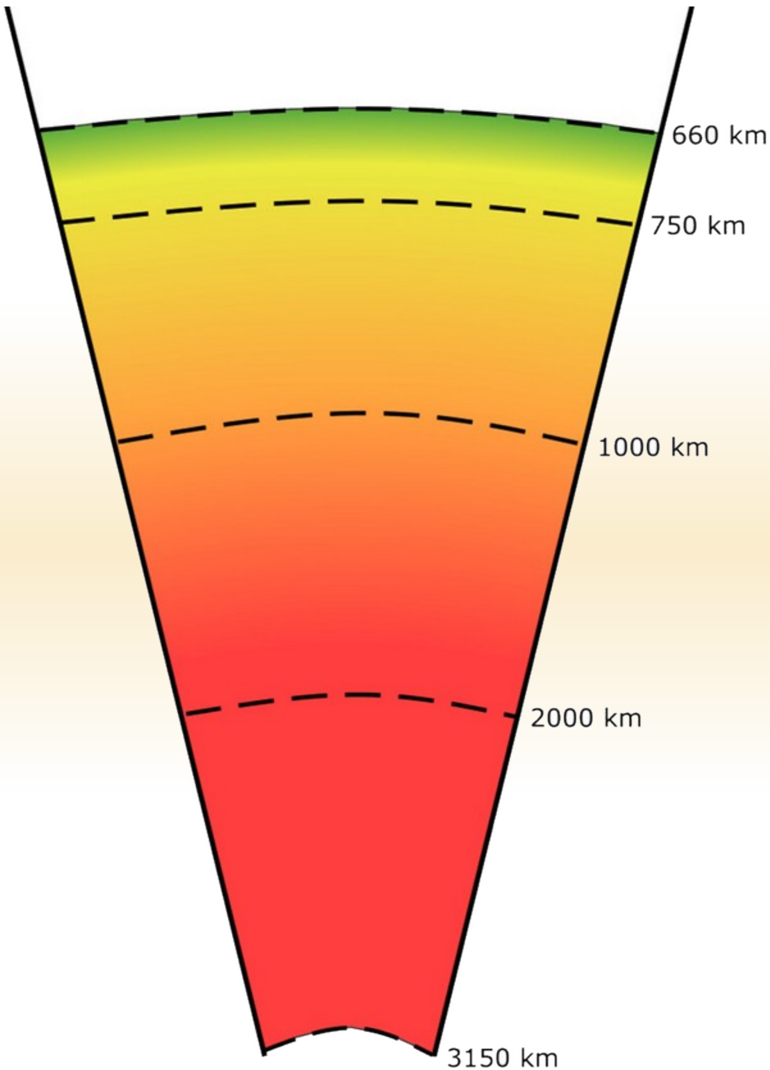
Université de Lille

Experimental layout





X-ray diffraction + laser heating + diamond anvil cell
P02.2, PETRA III, Hambourg



Pyrolite in the lower mantle

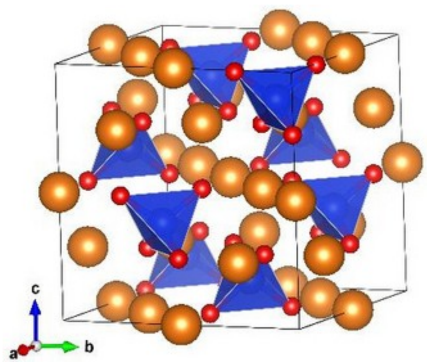
Gay et al, EPSL, 2023
Gay et al, GRL, 2024
Magali et al, in writing

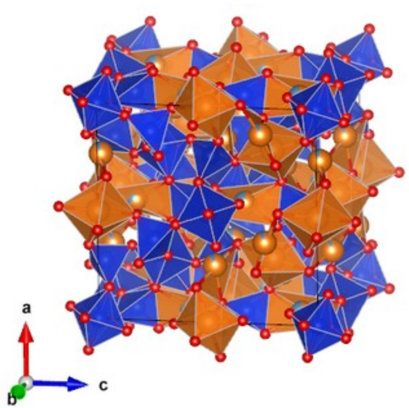
Université de Lille

660 km discontinuity

Lower transition zone

60% ringwoodite
$(Mg,Fe)_2SiO_4$

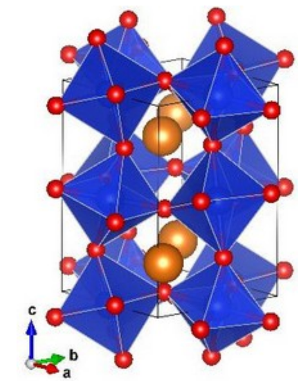




40% garnet
$(Mg,Fe,Al,Ca)_3(Al,Fe)_2(SiO_4)_3$

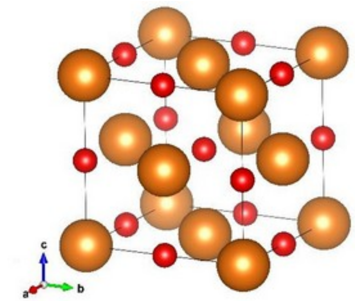
660
km
24 GPa
1900 K

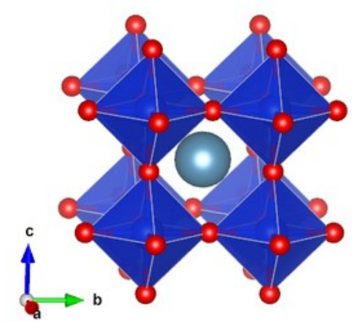
Lower mantle



80% bridgmanite
$(Mg,Fe)SiO_3$

15% ferropericlase
$(Mg,Fe)O$





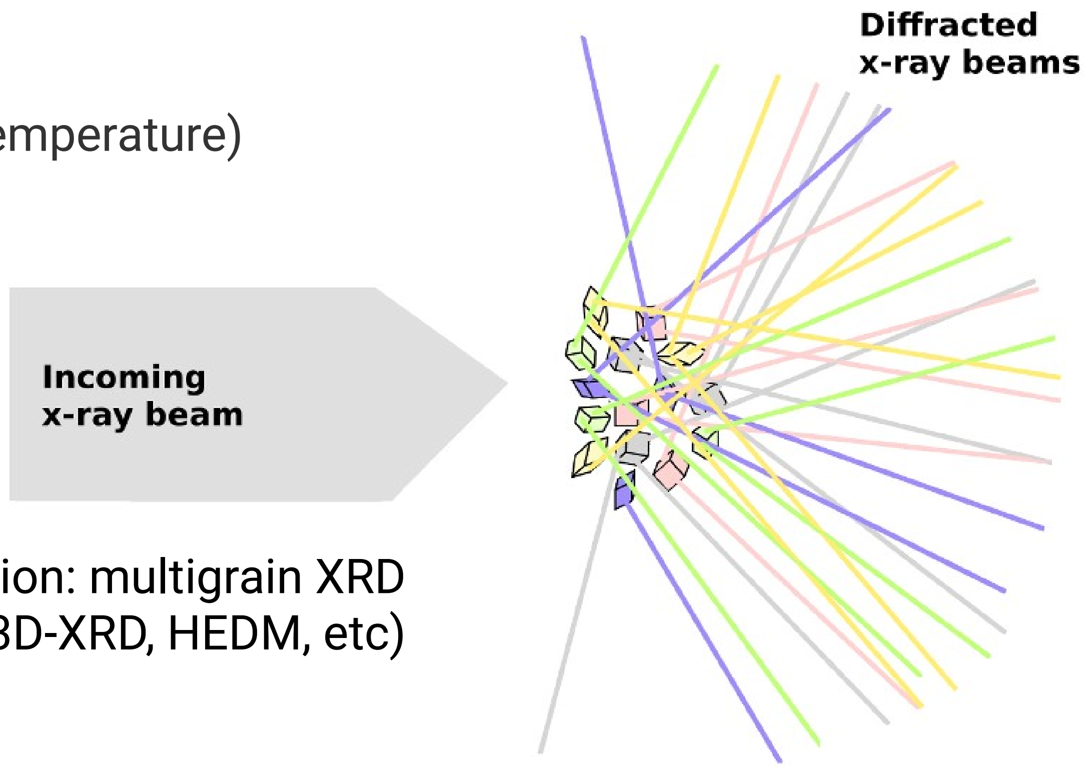
5% davemaoite
$CaSiO_3$

Université de Lille

Follow grain rotations under pressure?

Goal

- Use
  - A diamond anvil cell
  - Synchrotron x-ray diffraction
- Increase pressure (at Earth's mantle temperature)
  - Requires laser heating
- Follow grain rotations between pressure increases
- All in-situ, without opening the DAC….

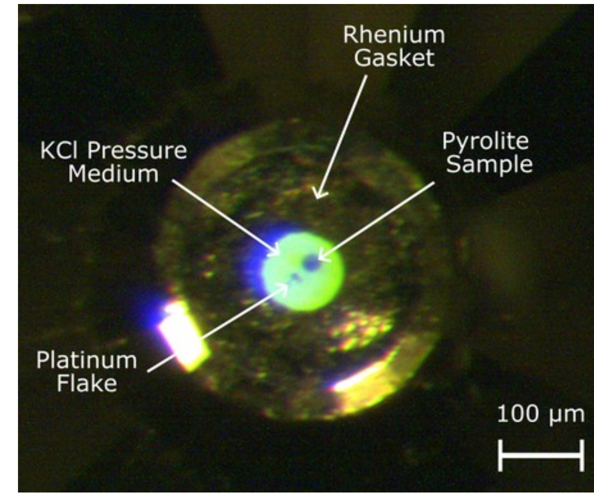


Solution: multigrain XRD
(3D-XRD, HEDM, etc)

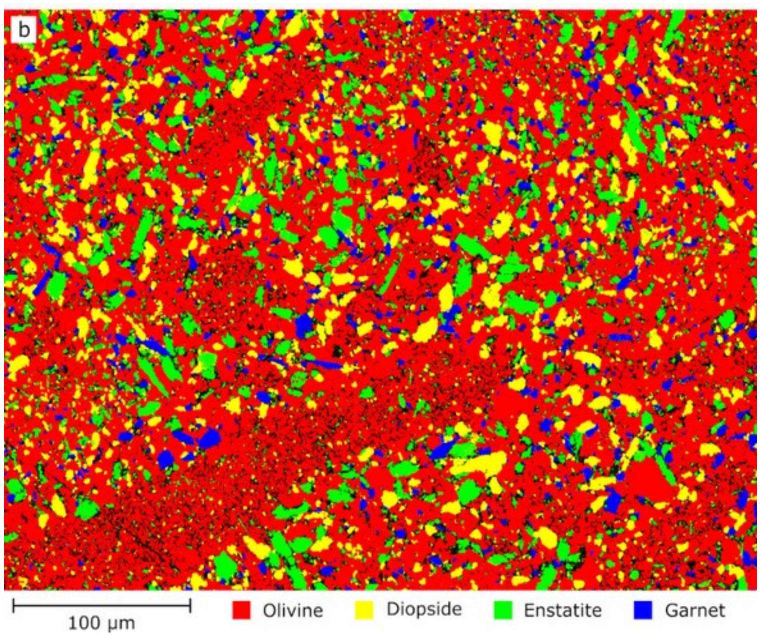
Sample preparation

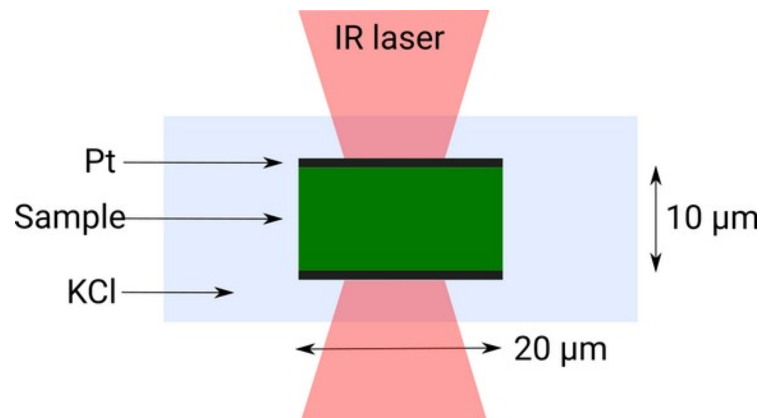
Process

- Pyrolitic composition sintered in a piston-cylinder
- Cut and polished to 20 μm diameter / 10 μm thickness
- Coated with 200 nm Pt
- Load in DAC with KCl pressure medium

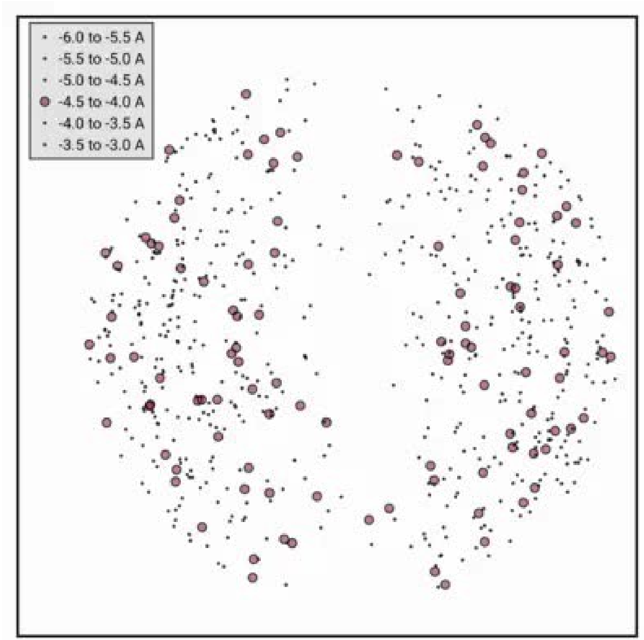


Starting material EBSD phase map

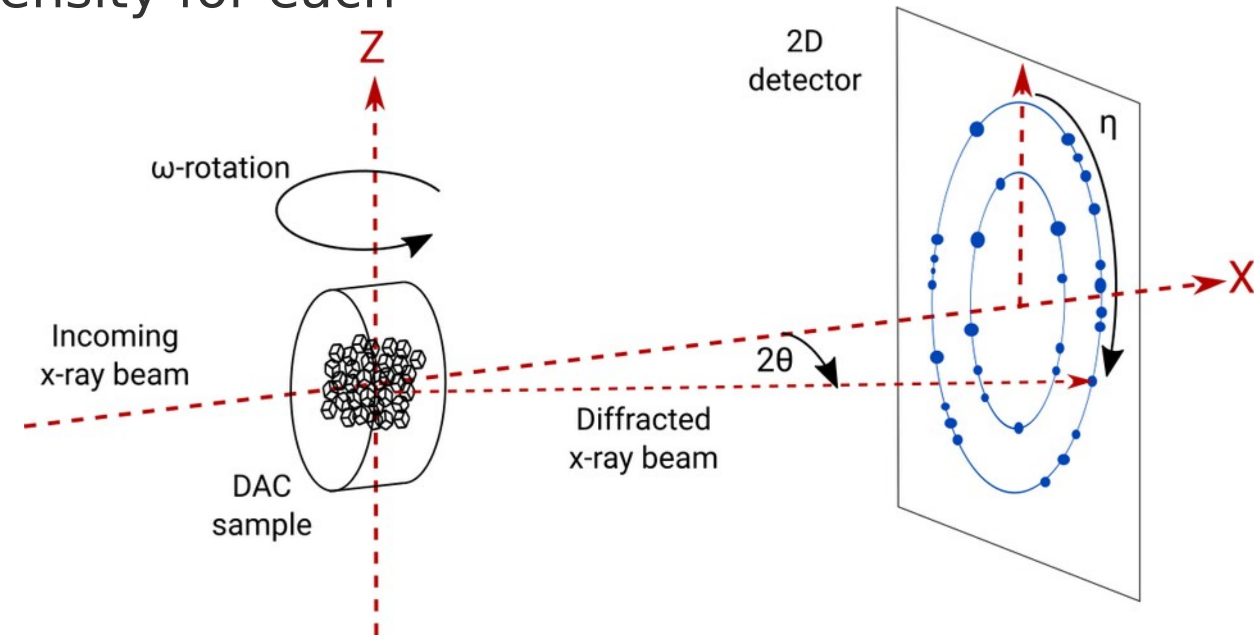




Multigrain X-ray diffraction in the DAC



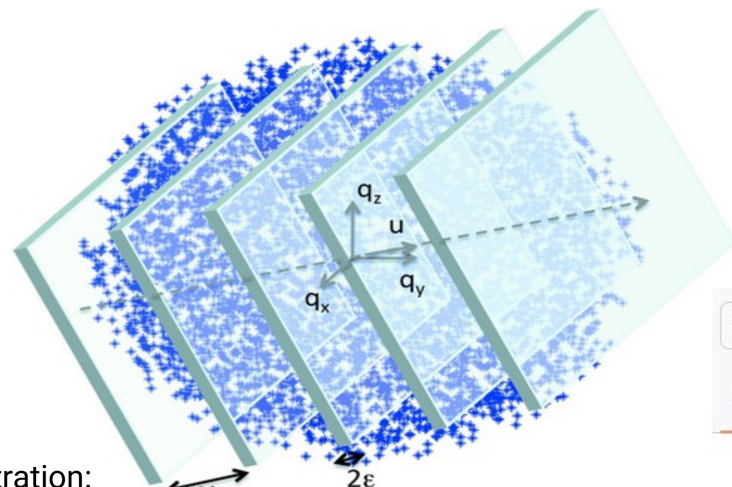
- DAC sitting on ω rotation stage
- Collect data every δω step (0.5°) over Δω range (-28° to +28°)
- Extract diffraction spot database with 2θ, η, ω, intensity for each



Indexing with Fable-3DXRD

Typical numbers

- ~10^4 spots per P/T point
- Random walk through orientation space to identify grains with convergence criteria
- ~ 10^6 iterations
- ~ 5.10^2 to 1.10^3 indexed grains per P/T point



Ilustration:
Wejdemanna and Poulsen,
J. Appl. Cryst. 2016

Results

Average sample

- Fine matrix vs. grains volume ratio
- Phase proportions
- Average cell parameters

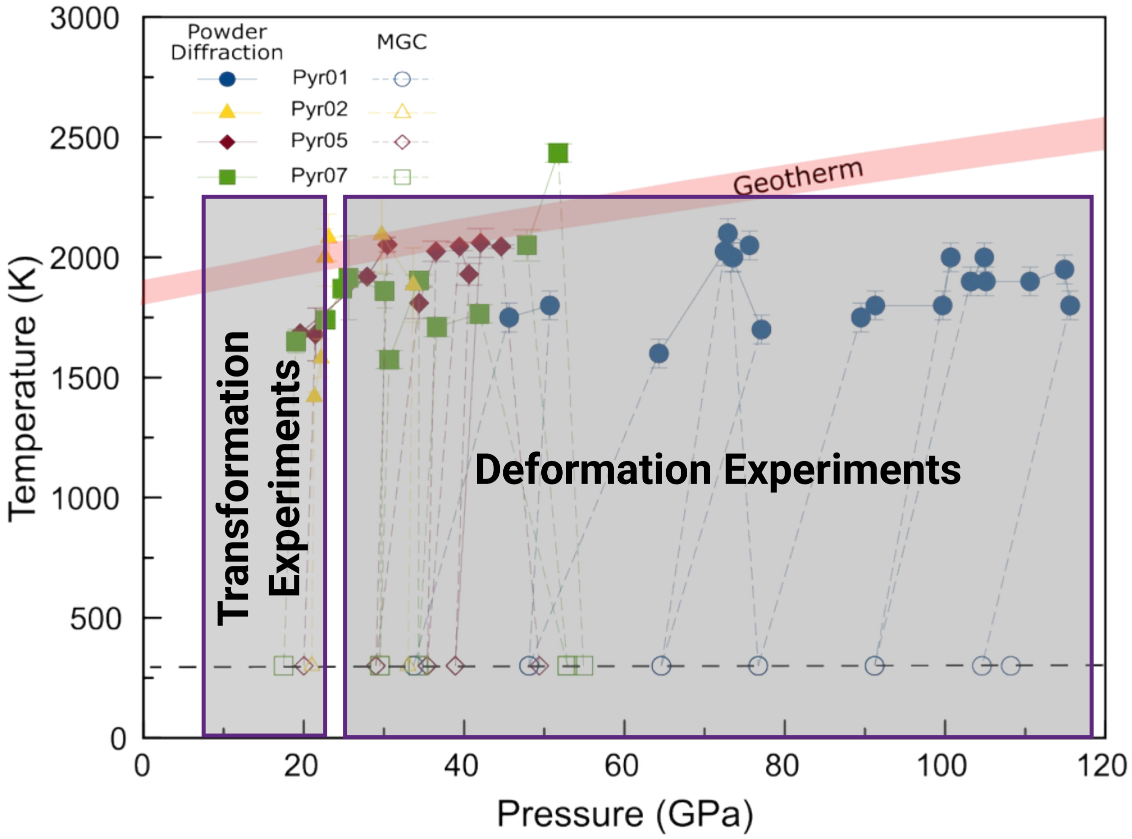
Grain scale, for each indexed grain

- Orientation
- Cell parameters
- Relative volume

Nisr *et al*, 2012, 2014
Rosa *et al*, 2015, 2016
Langrand *et al*, 2017
Krug *et al*, 2022
Ledoux *et al*, 2023a, 2023b
Gay *et al*, 2023, 2024

Université de Lille

Experimental P/T path

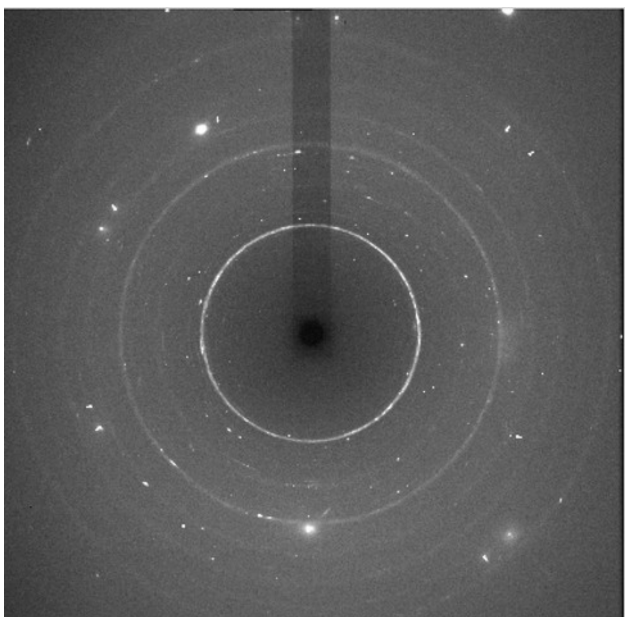


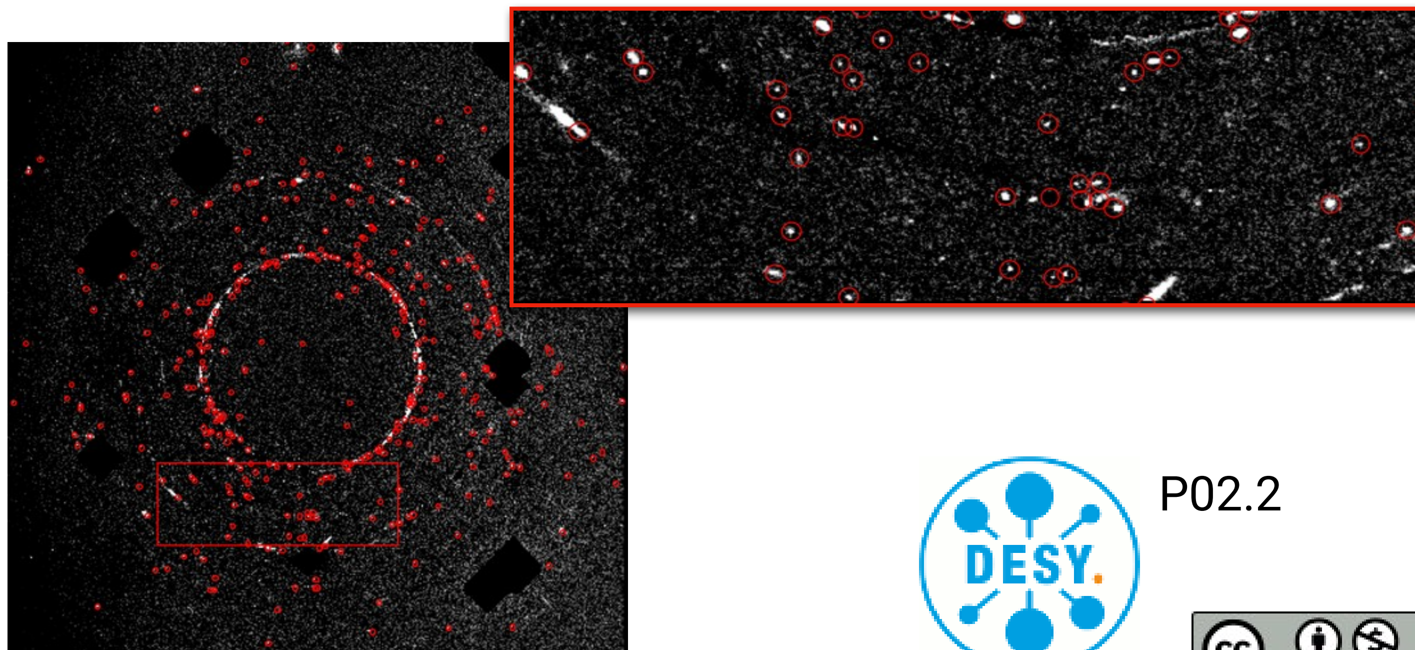
- 4 experiments on pyrolitic composition
- Trying to follow geotherm temperatures
- Powder diffraction images taken to monitor phase occurrence
- Pressure increases and phase transformations at high P and T
- Multigrain to characterize sample microstructures collected after quenching as close to transformation conditions as possible

Sample data

Raw diffraction image

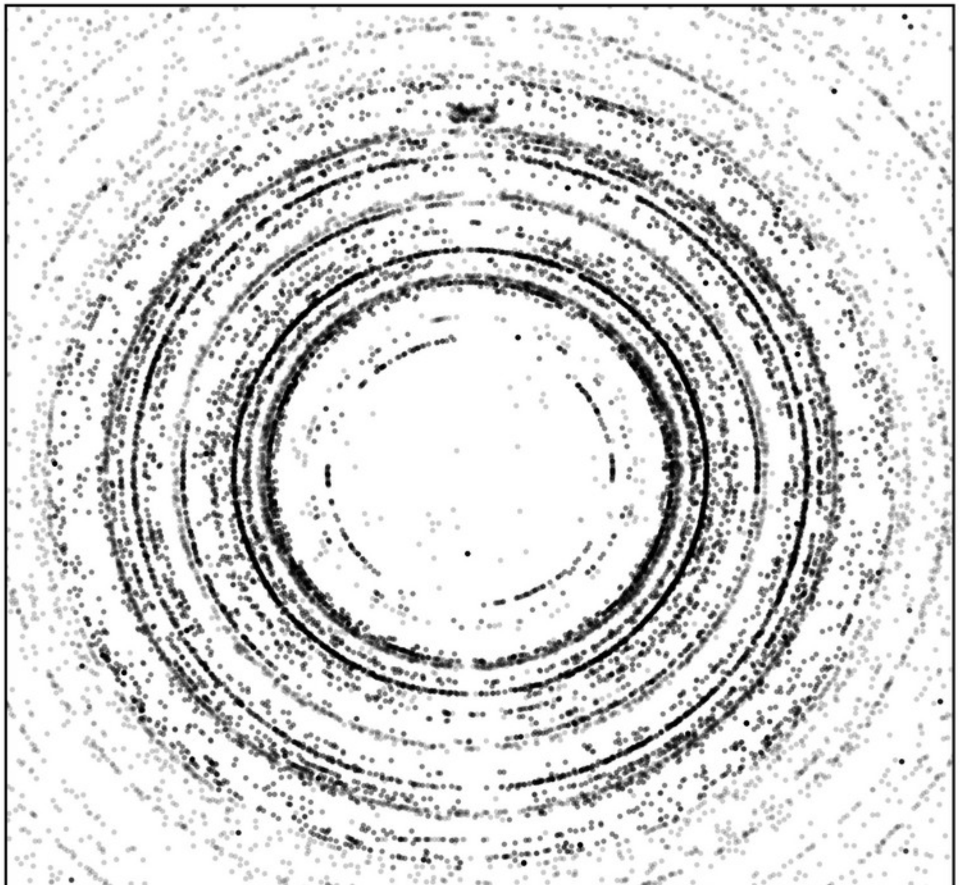
- Powder from pressure medium (KCl), spots from sample grains, diamond spots
- Median background filter
- Diamond spot easy to detect and mask (large intensity)
- High pass intensity filter → sample diffraction spots





P02.2

Phase identification – Step 1
Peaks extraction



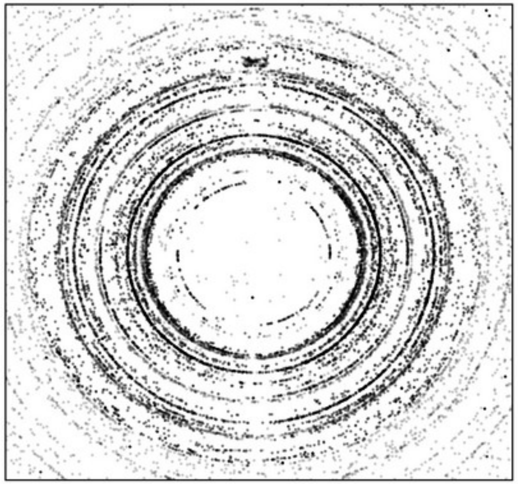
Pyrolite + KCl, 34 GPa

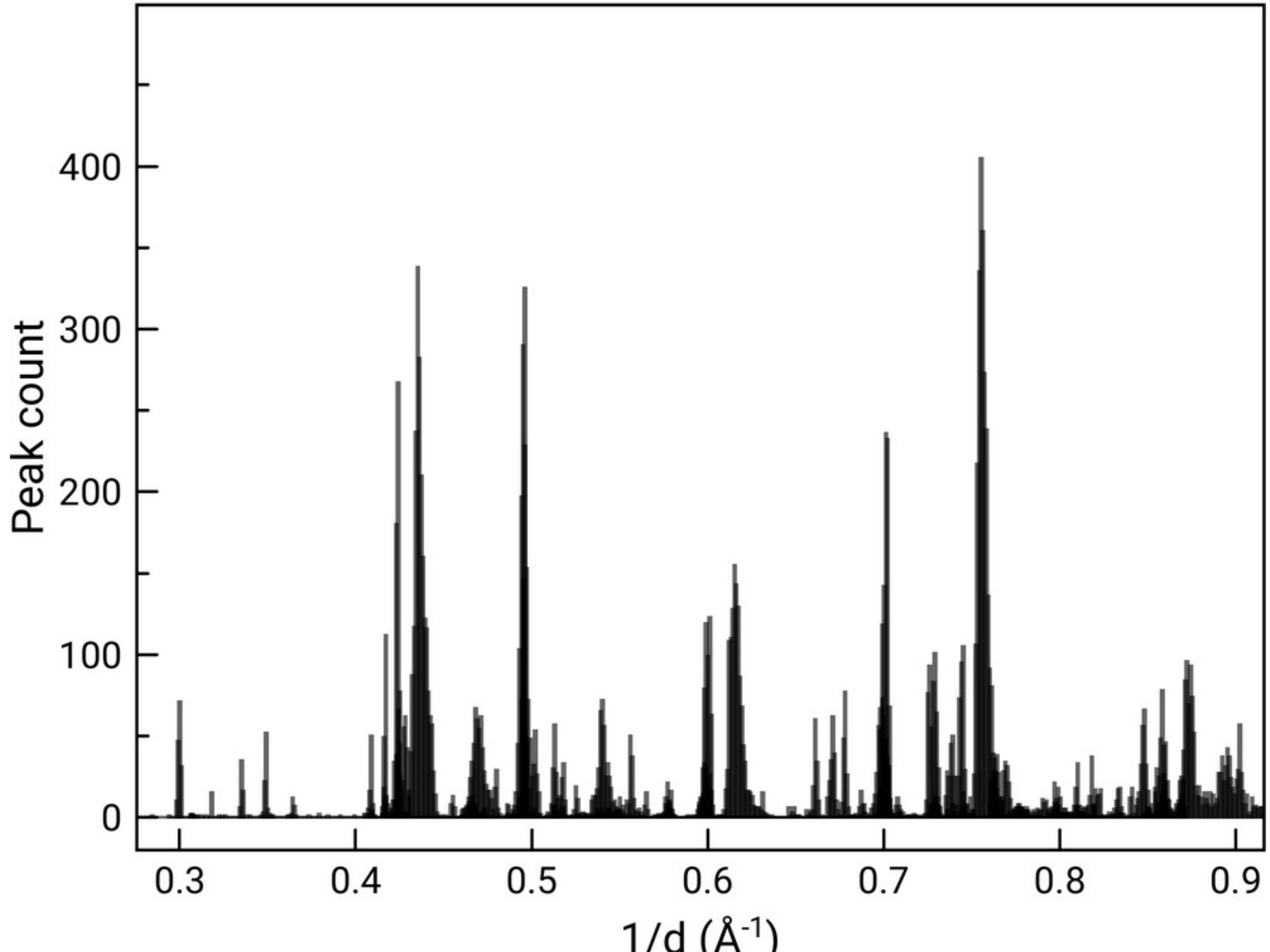
Potential phases:

- Bridgmanite $(Mg,Fe)SiO_3$
- Ferropericlase (Mg,Fe)O
- Davemaoite $CaSiO_3$
- Left-over garnets

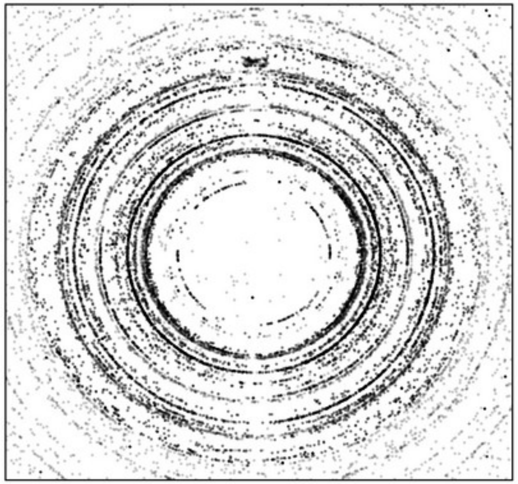
24000 extracted peaks, with know mean 2θ, η, ω, intensity for each

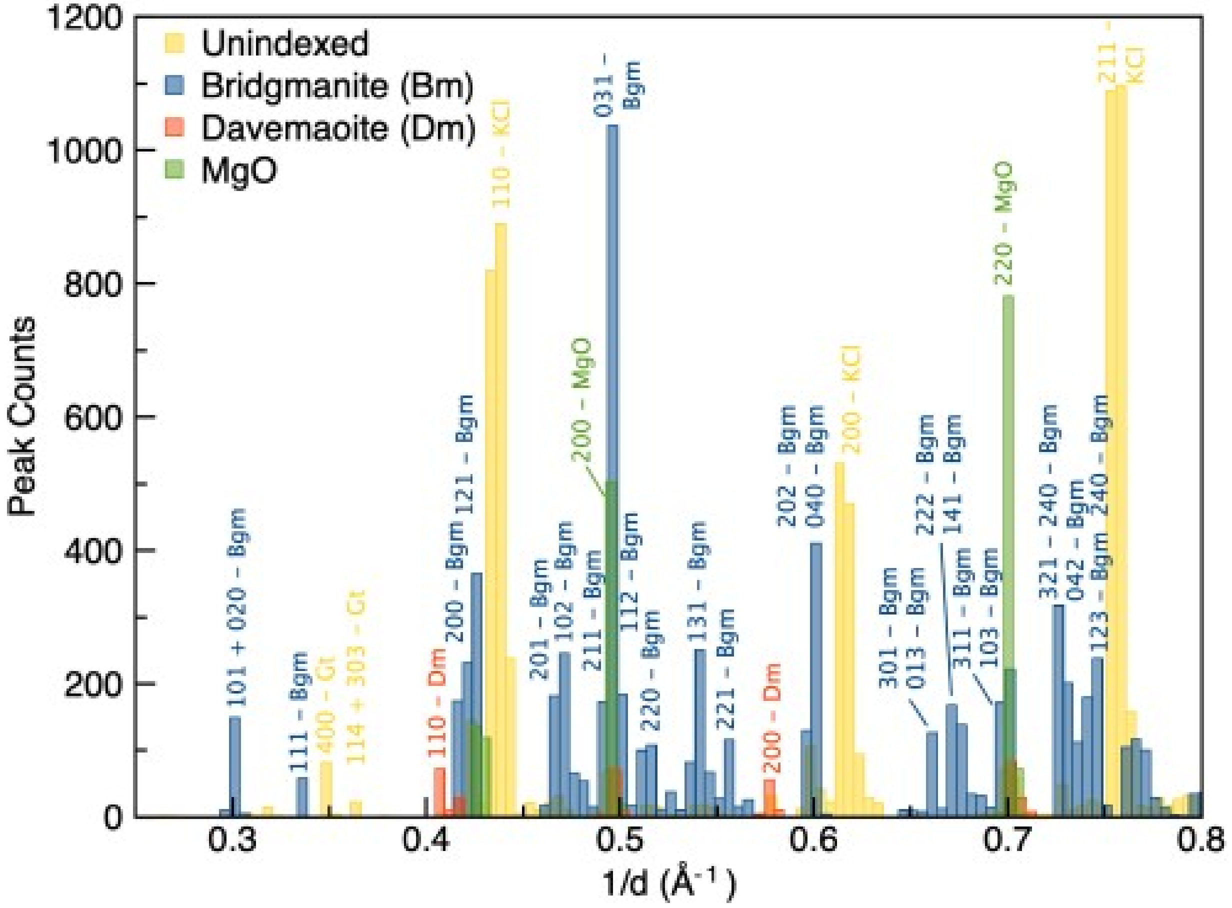
Phase identification – Step 2
Peak histogram vs. 1/d (or 2θ)





Phase identification – Step 3
Le Bail analysis





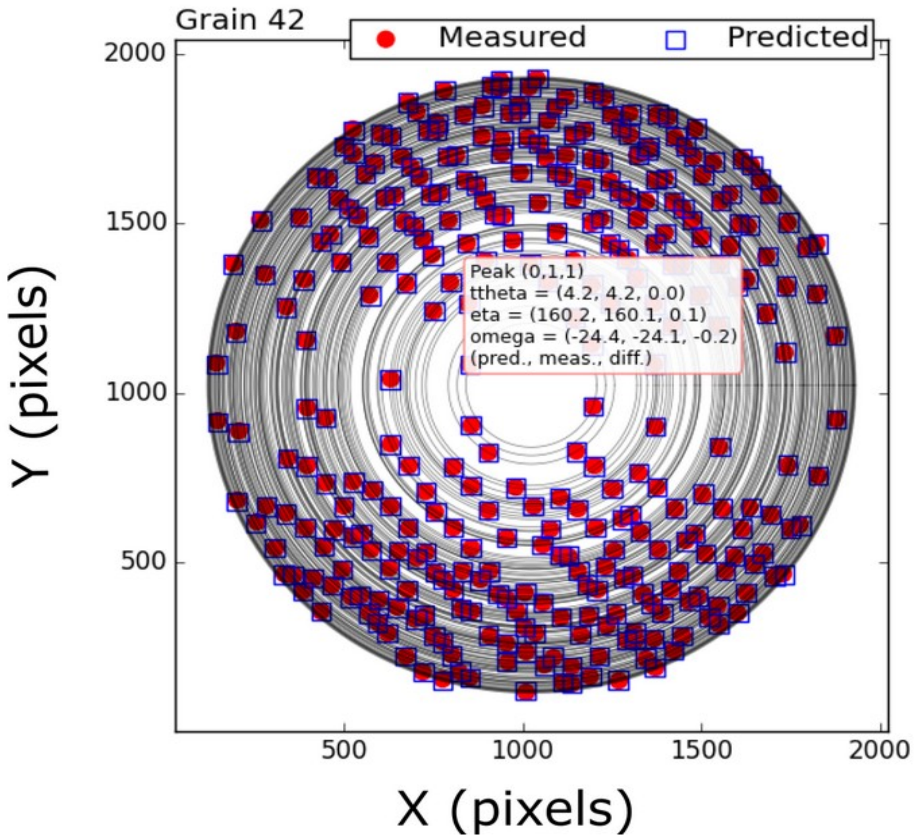
Phase identification

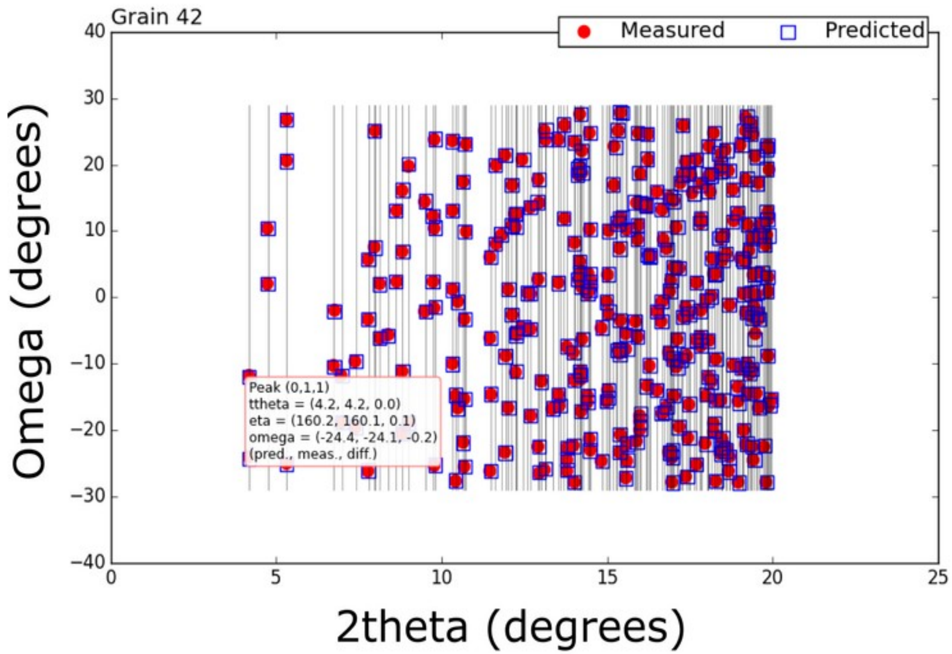
Unit-cell parameters for each

Cut-out

- 2θ regions with Kcl
- Regions with large 2θ (too much overlap)

Sample indexing result





Sample bridgmanite grain inside a polycrystal

Peaks:

- Squares: predicted
- Circles: measured

Indexing, Pyrolite + KCl, 34 GPa

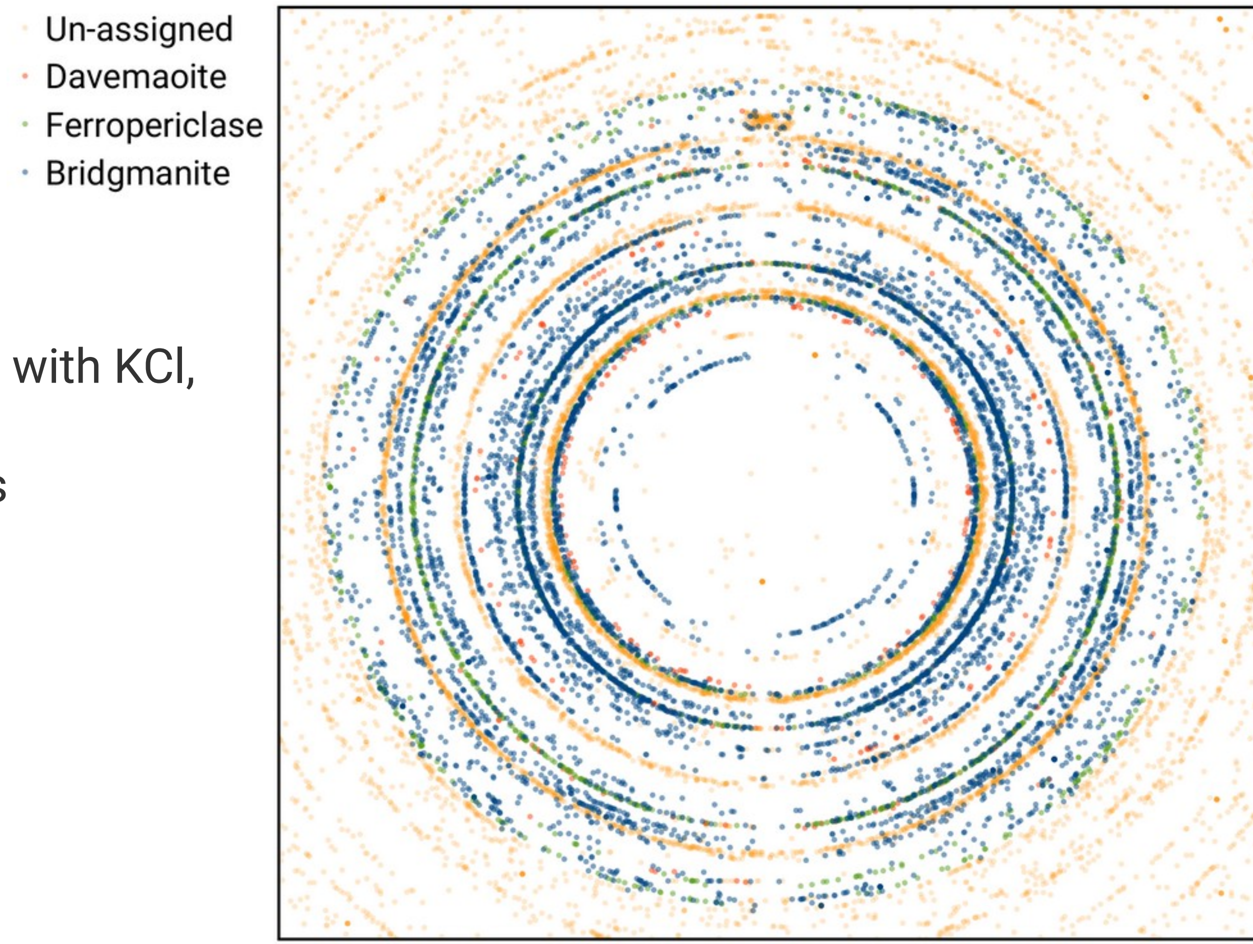
GrainSpotter Indexing

Peaks

- 14000 un-assigned (overlap with KCl, or out of 2θ range)
- ~2000 ferro-periclase peaks
- ~400 davemaoite peaks
- ~7600 bridmanite peaks

Indexed grains

- Brigmanite: 241
- Davemaoite: 36
- Ferropericlase: 144



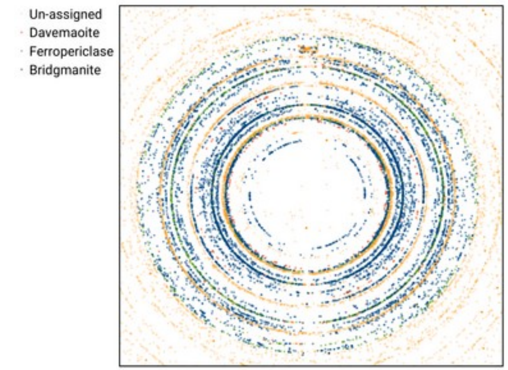
Université de Lille

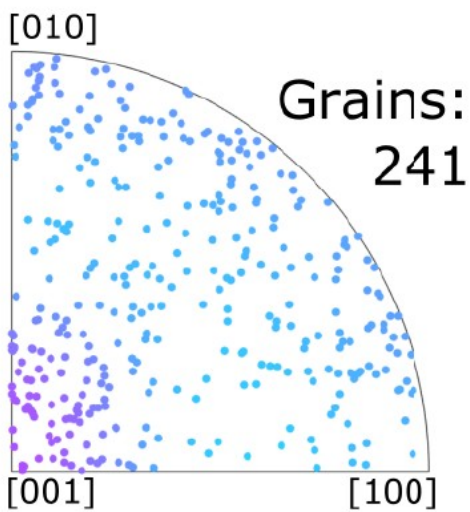
Indexing, Pyrolite + KCl, 34 GPa

GrainSpotter Indexing

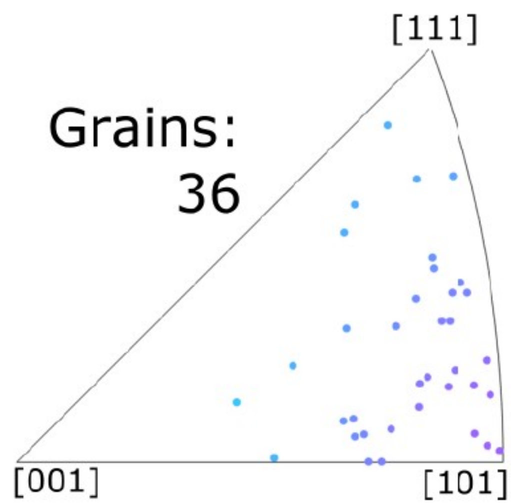
Indexed grains

- Brigmanite: 241
- Davemaoite: 36
- Ferropericlase: 144

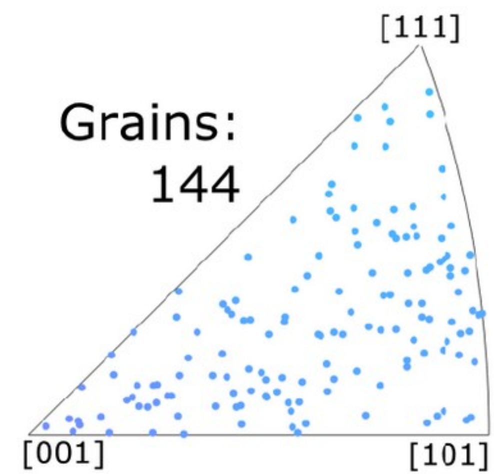




Bridgmanite grain orientations

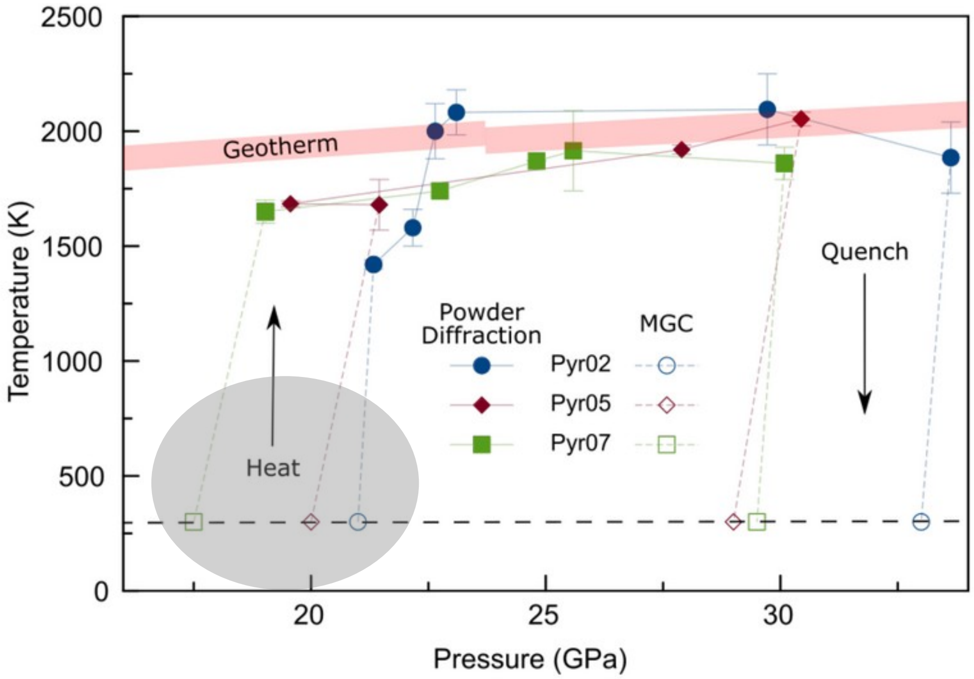


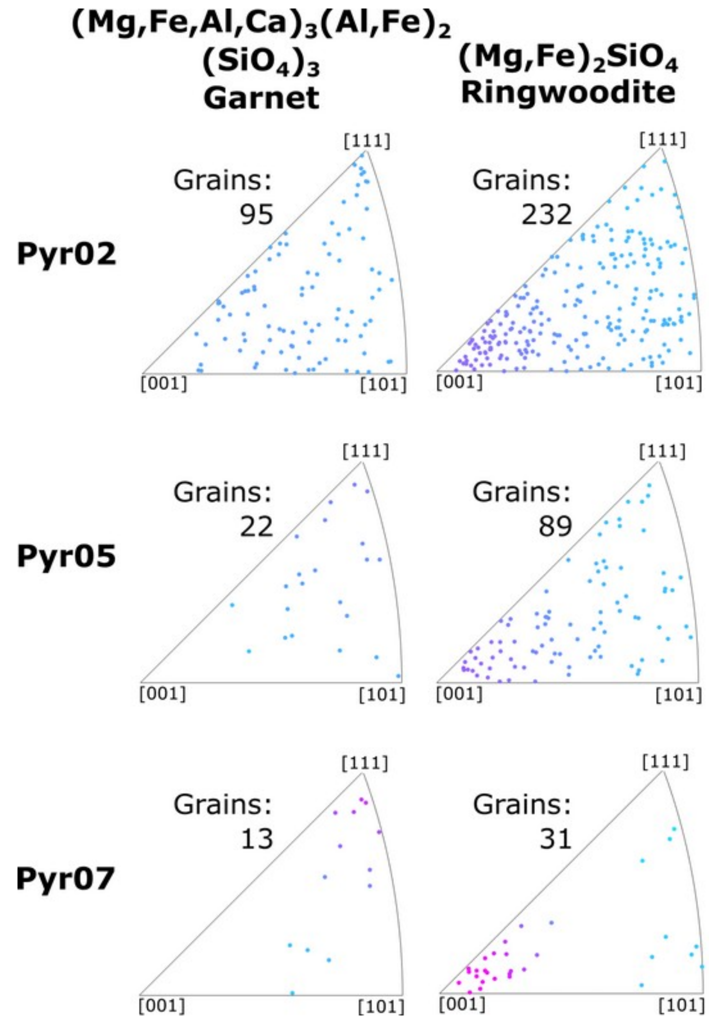
Davemaoite grain orientations



Ferropericlase grain orientations

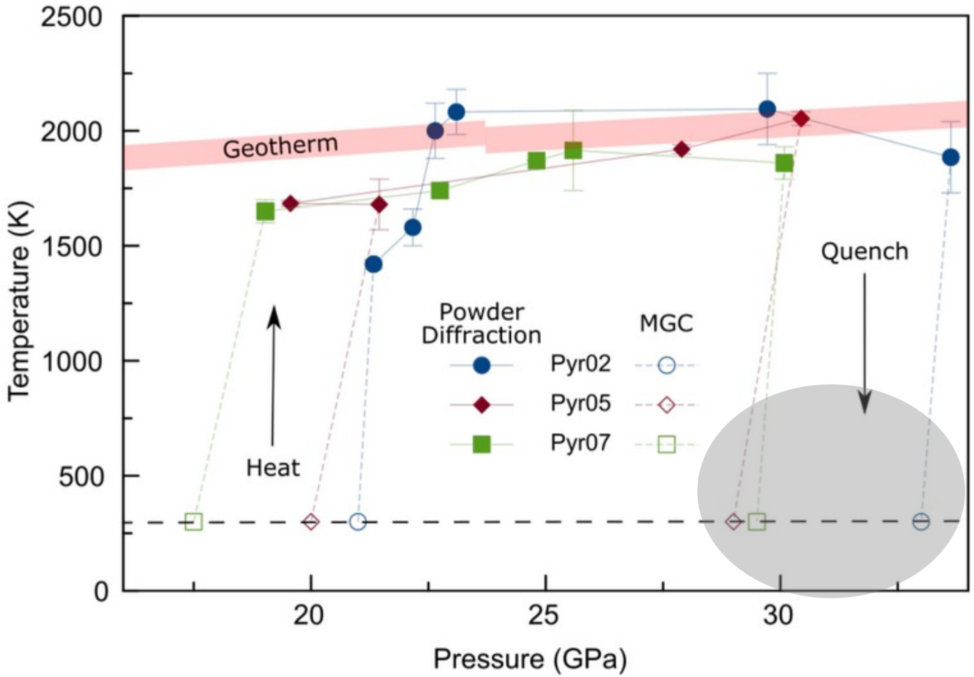
660 km discontinuity experiment

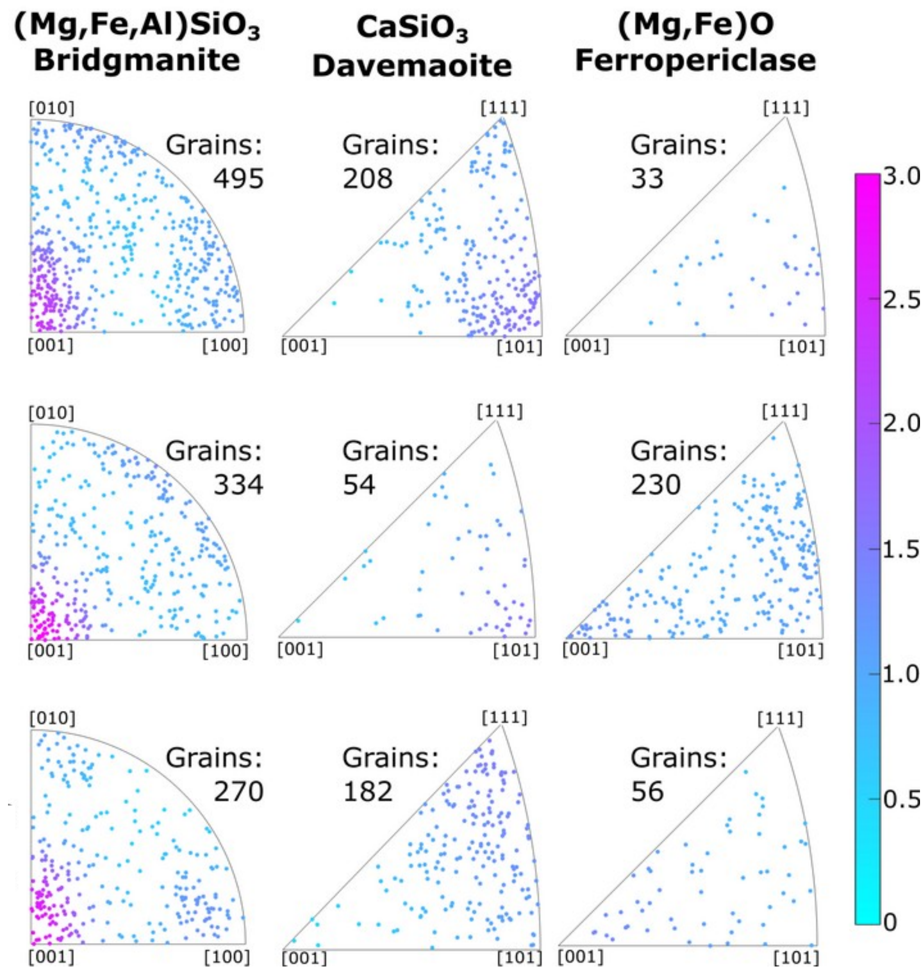




Gay et al, EPSL, 2023

660 km discontinuity experiment

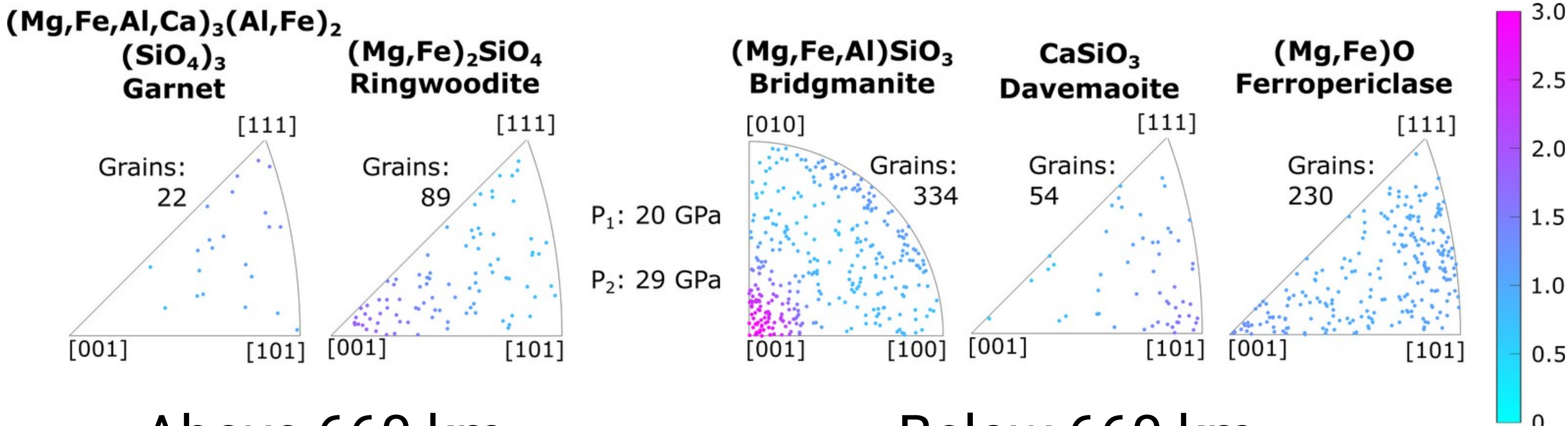




Gay et al, EPSL, 2023

Université de Lille

660 km discontinuity experiment



Above 660 km

Below 660 km

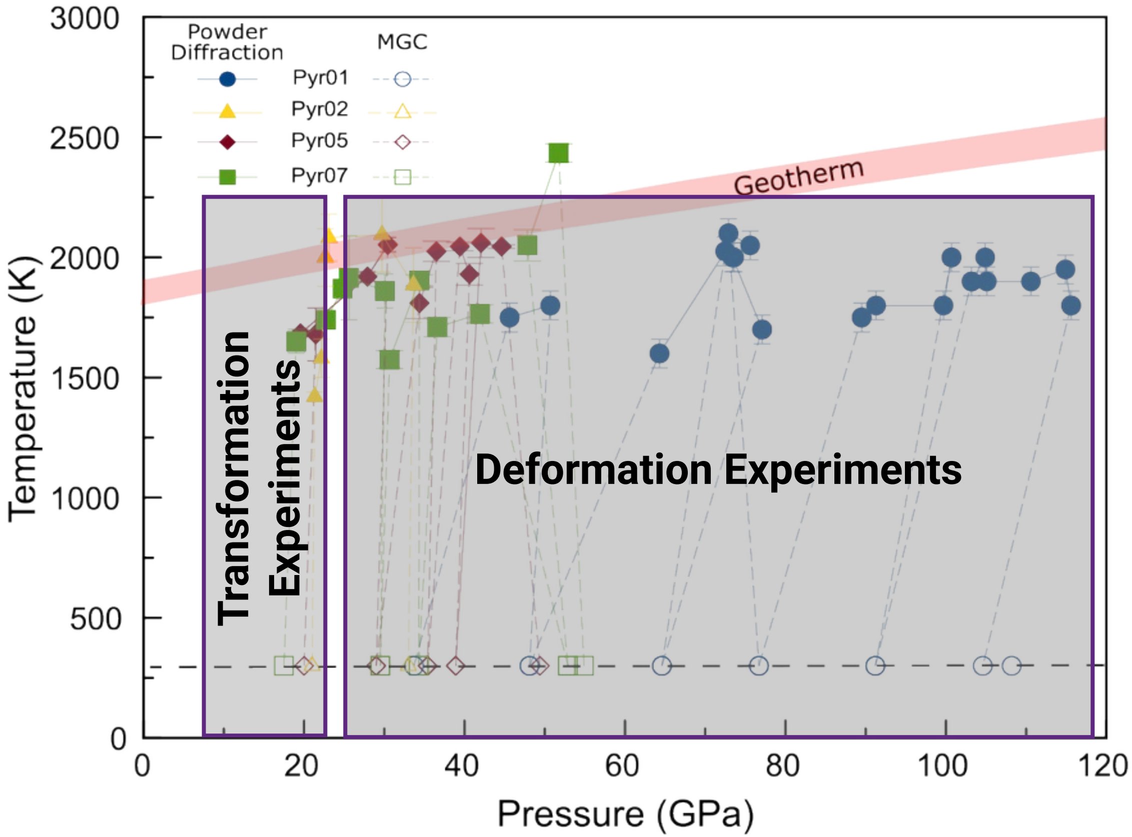
Systematic 001 transformation texture in bridgmanite

011 / 111 transformation texture in CaPv / davemaoite

Origin: nucleation / growth under compressive stress

Gay et al, EPSL, 2023

Deep mantle deformation

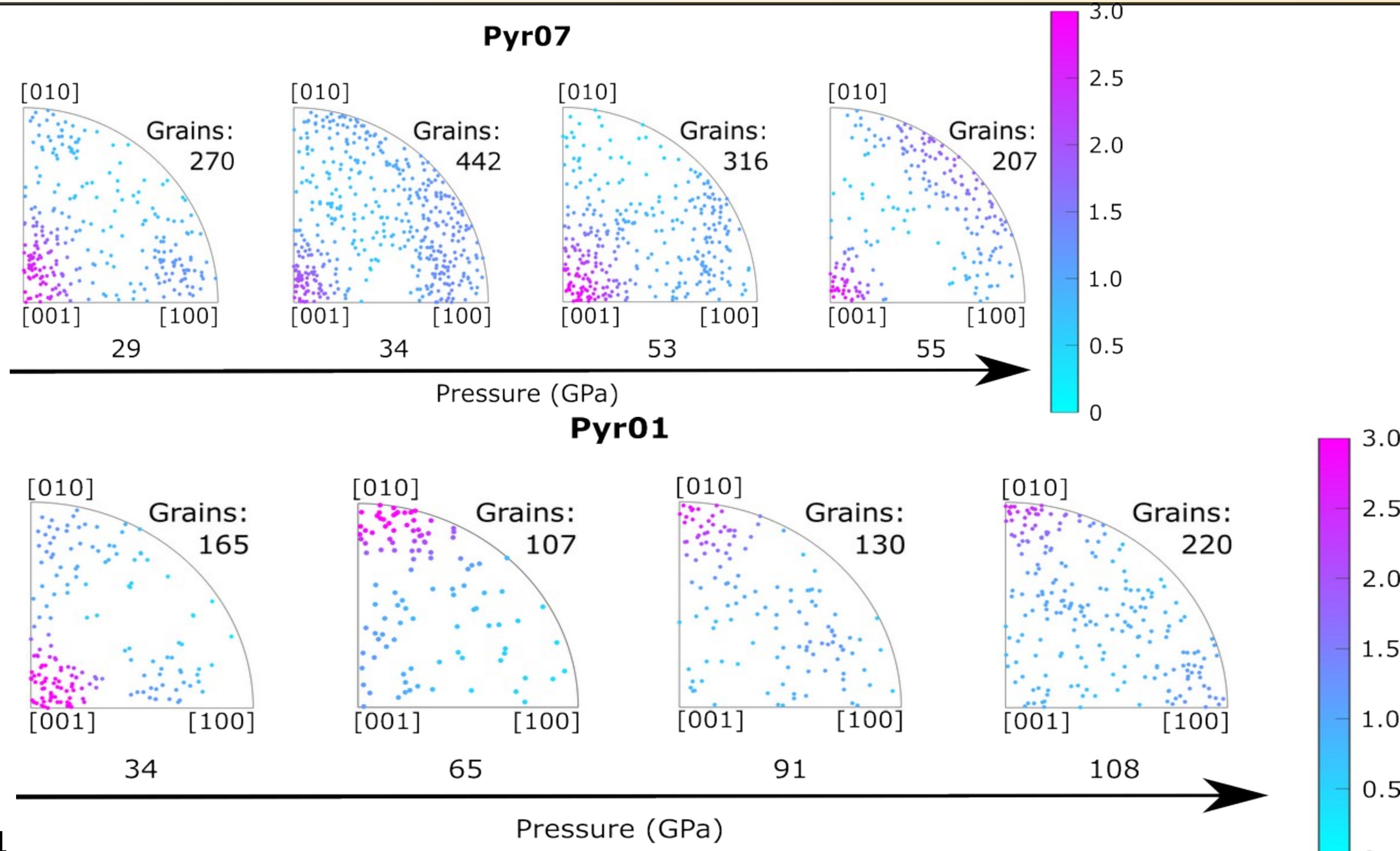


- 4 experiments on pyrolitic composition
- Trying to follow geotherm temperatures
- Powder diffraction images taken to monitor phase occurrence
- Pressure increases and phase transformations at high P and T
- Multigrain to characterize sample microstructures collected after quenching as close to transformation conditions as possible

Deep mantle deformation

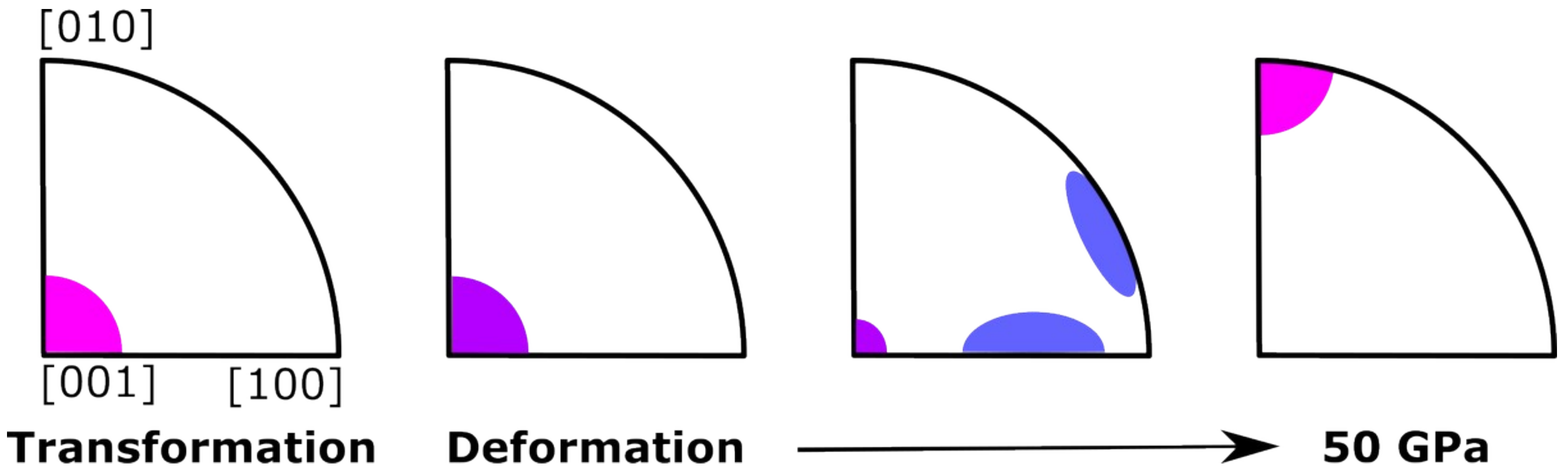
Up to 55 GPa

Up to 108 GPa



Gay et al, GRL, 2024

Deep mantle deformation



001 transformation textures

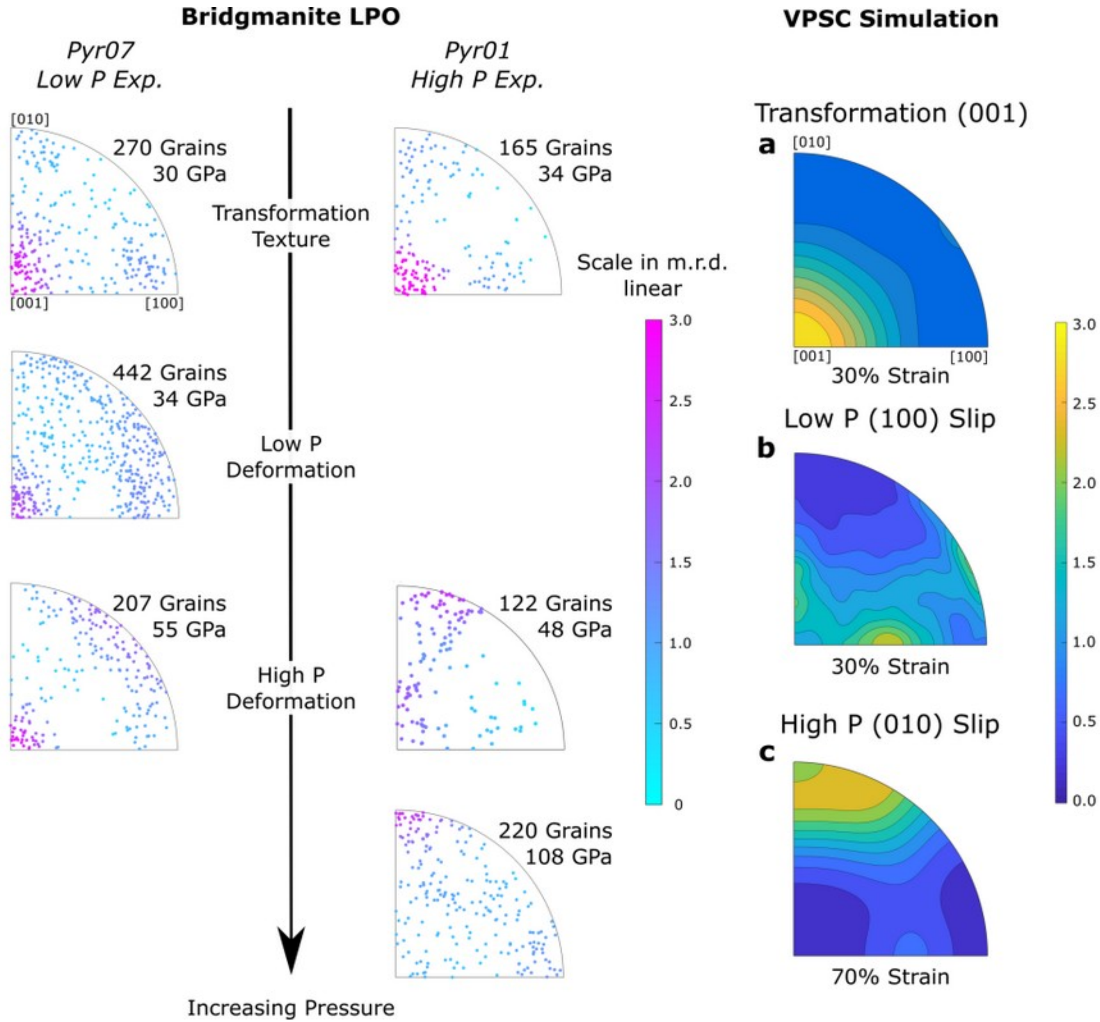
Upon further compression grains reorient to weak intermediate 100 orientations

At and above 50 GPa 010 orientations dominate in bridgmanite

→ transformation texture + deformation textures with change of dominant slip system below 50 GPa

Gay et al, GRL, 2024

Analysis based on VPSC simulations



Identification of 3 regimes of grain orientations

- Transformation microstructures, with (001) orthogonal to maximum stress direction
- Deformation below ~50 GPa, dominated by slip on [001](100)
- Deformation at P >~ 50 GPa, dominated by slip on (010), either [100] or [001]

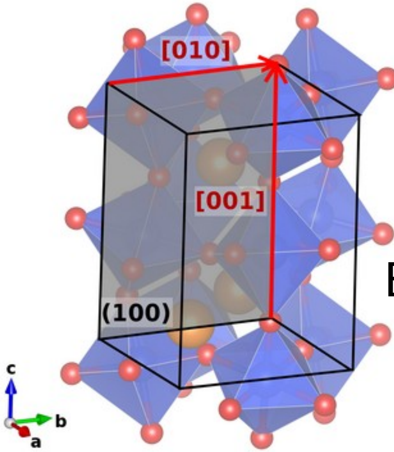
Gay et al, GRL, 2024

Deep mantle deformation

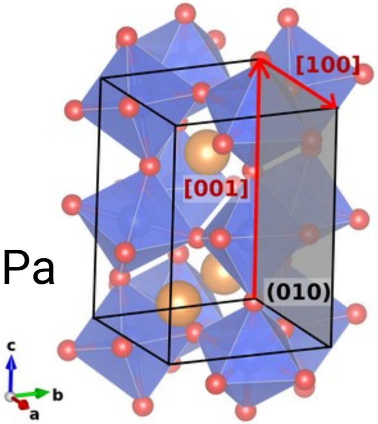
Table 1. Relative CRSS and Slip System Activity in Bridgmanite Modeled Using VPSC

Slip system	Low P		High P	
	CRSS	Activity (%)	CRSS	Activity (%)
(100)[010]	2	18.0	4	0.6
(100)[001]	1	34.7	4	9.0
(100)〈011〉	3	6.0	5	5.9
(010)[100]	4	2.2	1	36.9
(010)[001]	2	11.3	1	34.4
(010)〈101〉	3	9.4	4	1.4
(001)[100]	2	4.3	5	4.6
(001)[010]	2	11.3	3	1.3
(001)〈110〉	5	1.2	5	4.3
{111}〈110〉	30	1.6	30	1.6

Gay et al, GRL, 2024



Below <~ 50 GPa



Above >~ 50 GPa

Conclusions and outlook

Multigrain XRD at high pressure

- Procedure for diamond anvil cell experiments
- Can follow phase transformation / crystal rotations
- Several 100's of Gpa
- Laser heating up to 1000's of degrees
- Uses
  - Identification of new crystal phases
  - Transformation / deformation microstructures

Pyrolite in the Earth's lower mantle

- 001 transformation texture at 660 km
- Dominant slip on (100) below 50 GPa
- Dominant slip on (010) above 50 Gpa
- Predictions for velocities provided to seismologists