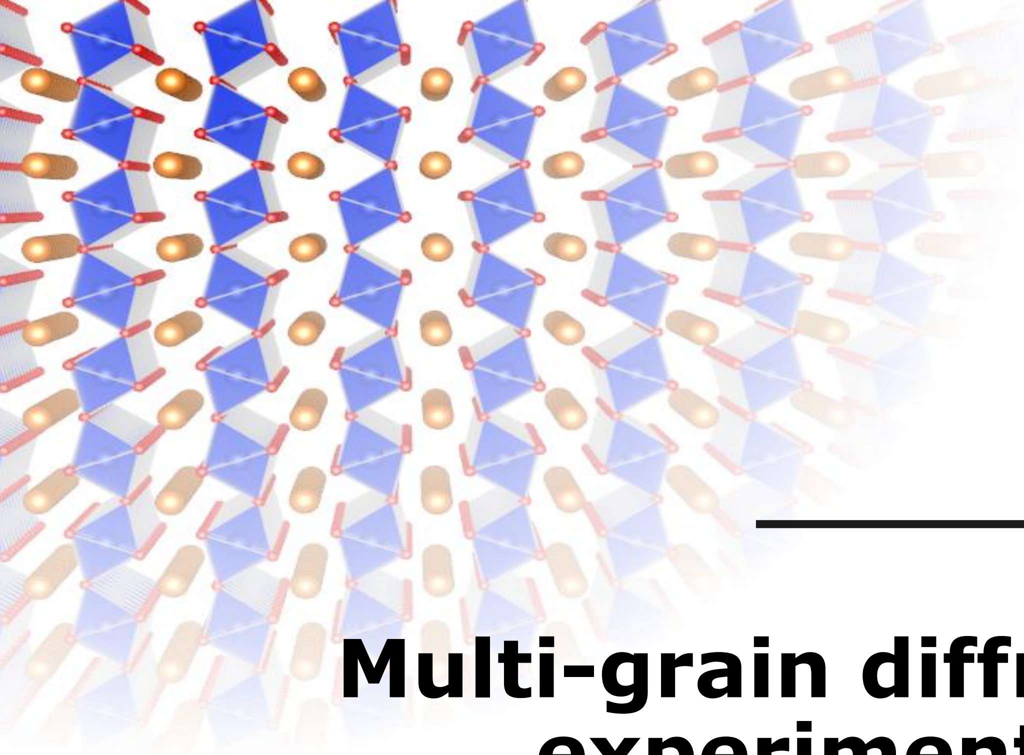

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Multi-grain diffraction and deformation experiments in diamond anvil cells

TIMEleSS

ANR DFG

S. Merkel

TIMEleSS project 2018-2022


S. Merkel


C. Thomas


C. Sanchez Valle


S. Speziale


F. Rochira


J. K. Magali


M. Krug


E. Ledoux


J. Gay

WWU MÜNSTER

GFZ Helmholtz-Zentrum Potsdam

cnrs

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AGENCE NATIONALE DE LA RECHERCHE ANR

DFG Deutsche Forschungsgemeinschaft

Transport in the Earth mantle

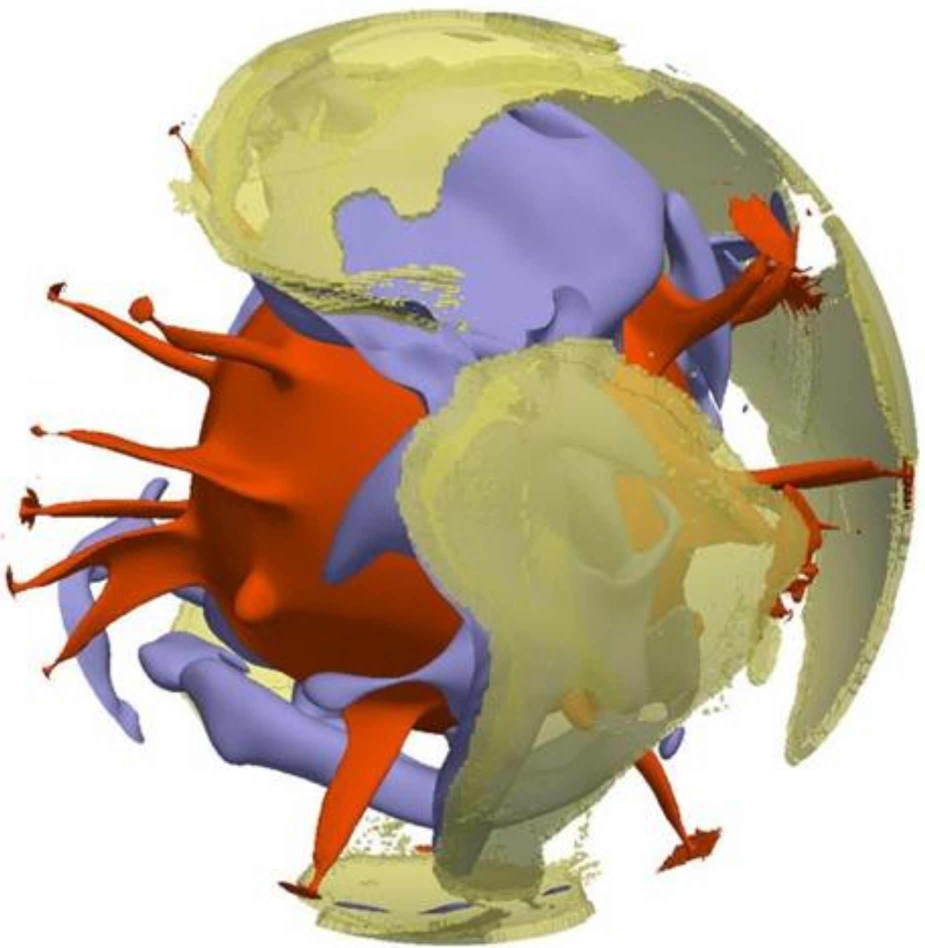


Illustration N. Coltice, Univ. Nice

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

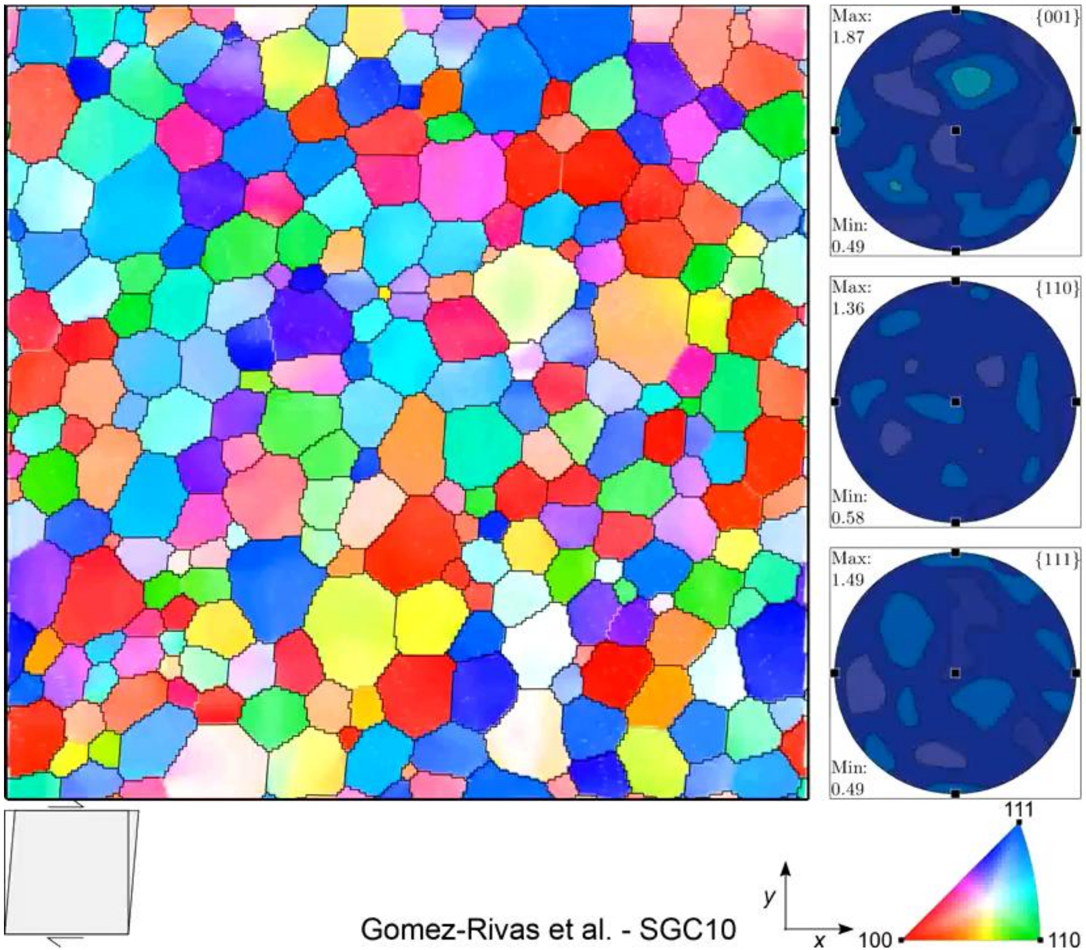
Velocities: cm/yr

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

Changes in P and T

Phase transformations

Solid-state deformation



Gomez-Rivas et al. - SGC10

Sample ELLE simulation: halite during shearing

Colors ↔ grain orientations

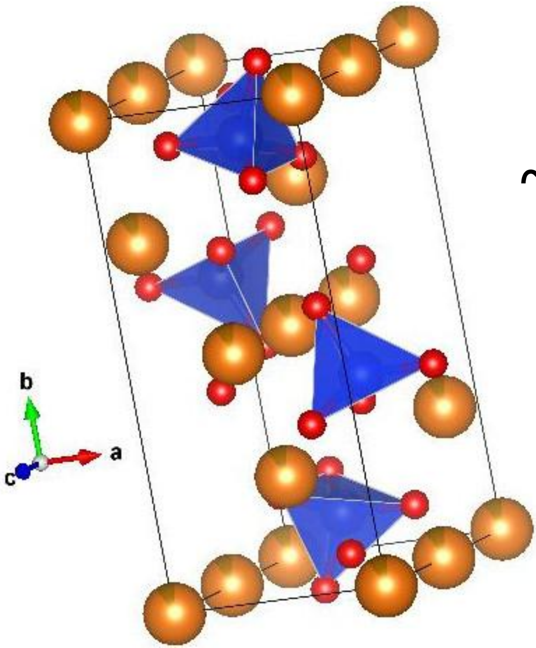
Grain shapes and orientations change with deformation → changes in "*microstructures*"

Impact on

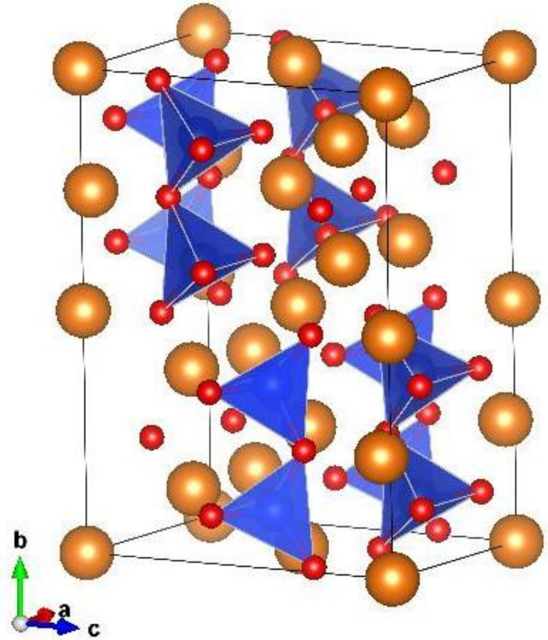
- Microstructures
- Mechanical properties
- Wave propagation features (i.e. *observations with seismology*)

Video at: https://www.youtube.com/watch?v=mfqprkOH6fE

Phase transformations



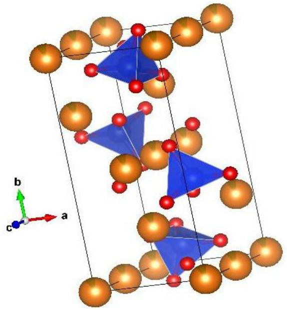
~ 13 GPa



Olivine
α- phase

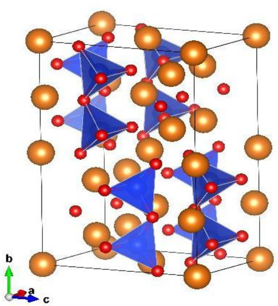
Wadsleyite
β-phase

Phase transformation microstructures

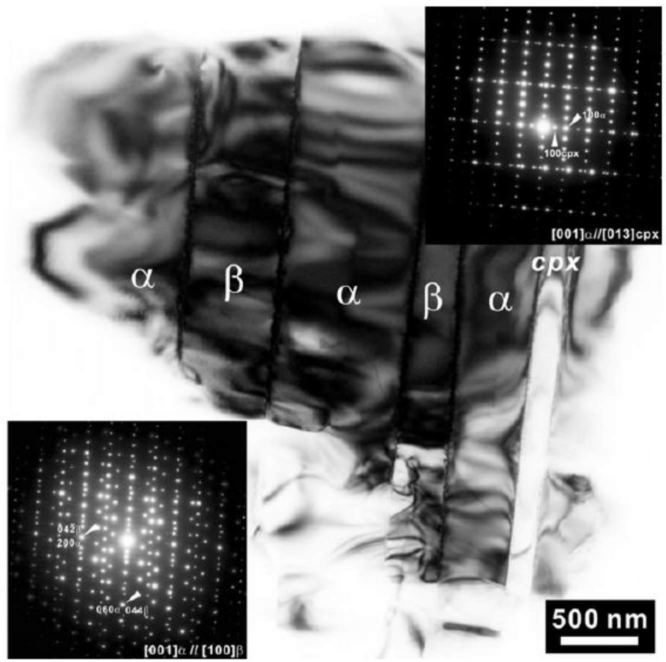


Olivine
α- phase

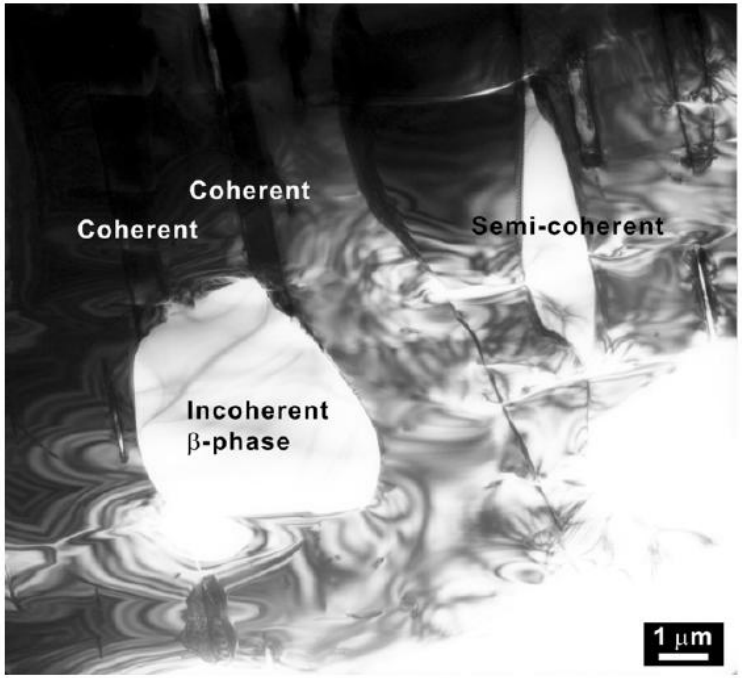
~ 13 GPa



Wadsleyite
β-phase



Coherent transformation
$(200)_{ol}$ // $(042)_{wd}$,
$(060)_{ol}$ // $(0\ 4\ 4)_{wd}$
$[001]_{ol}$ // $[1\ 0\ 0]_{wd}$



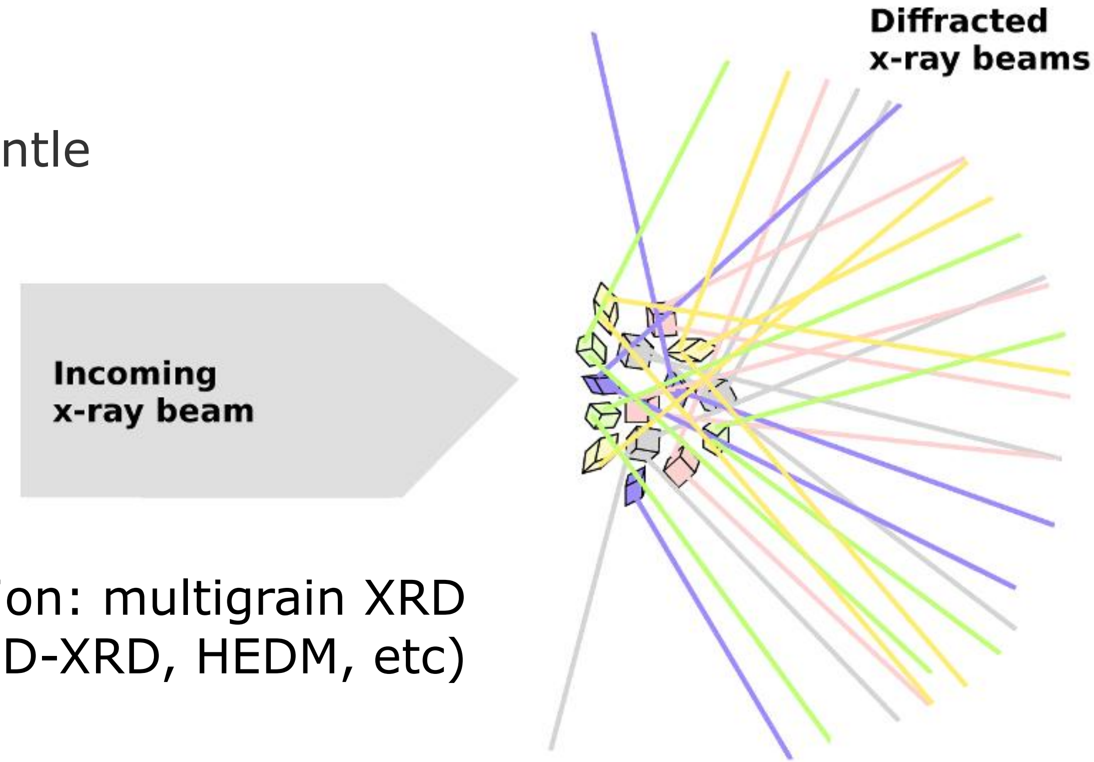
Incoherent
transformation

Smyth et al, PEPI, 2012

Follow grain rotations under pressure?

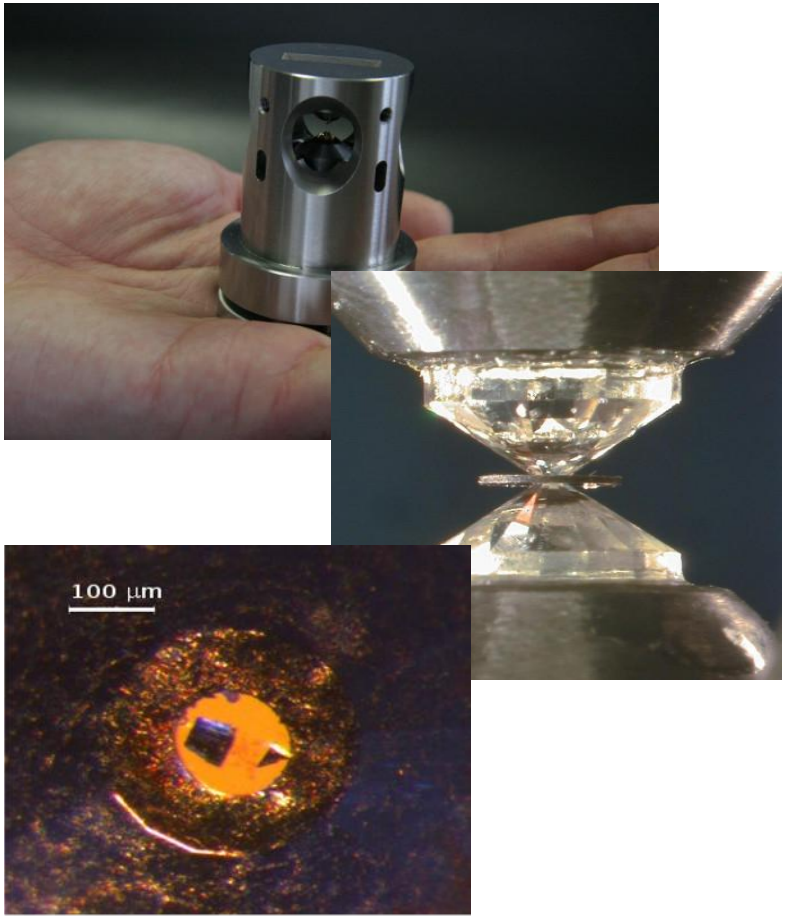
Goal

- 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….

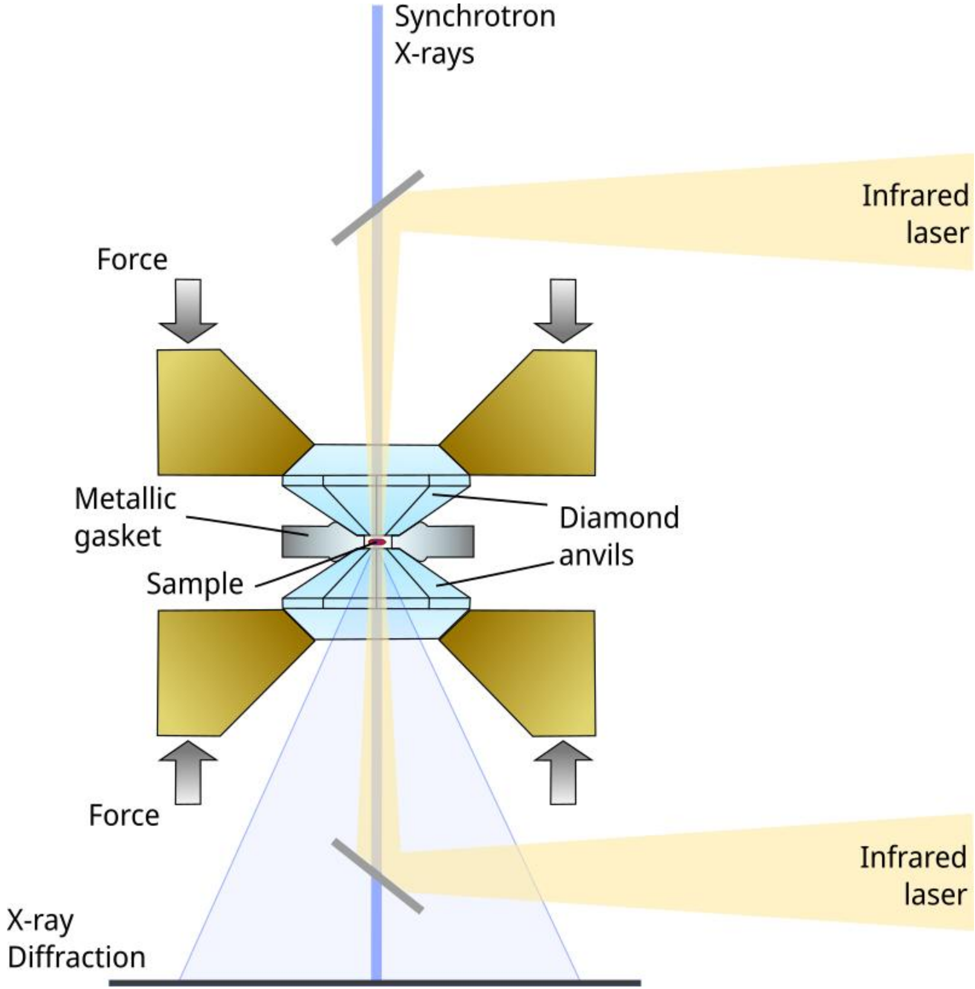


Solution: multigrain XRD
(3D-XRD, HEDM, etc)

Diamond anvil cell



Diamond anvil cells @ Univ Lille



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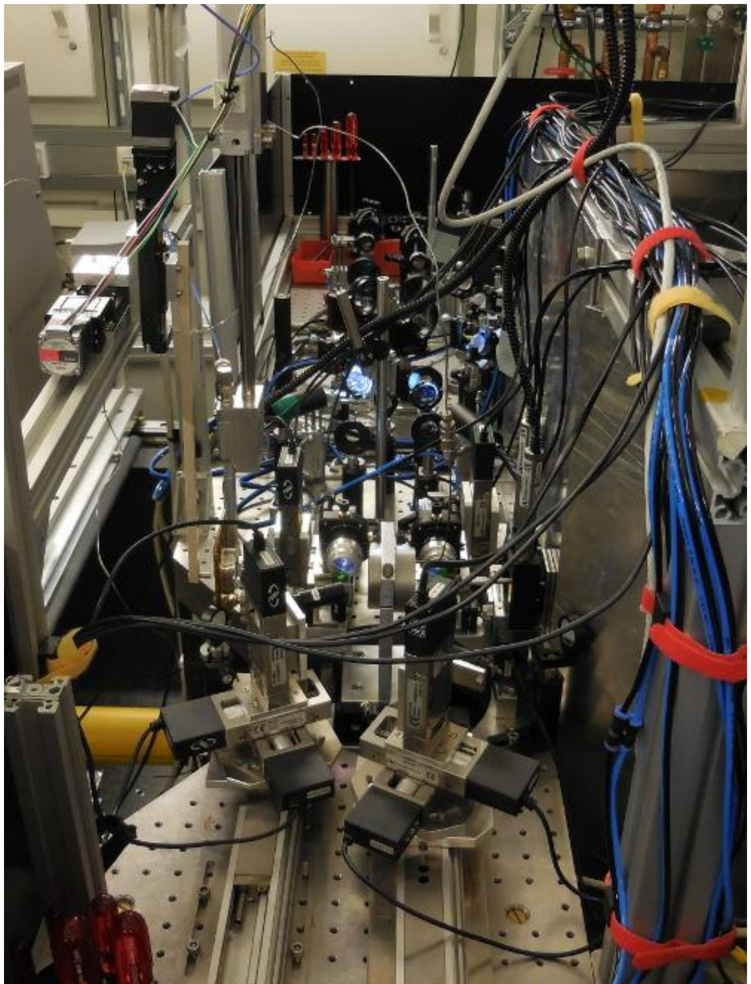
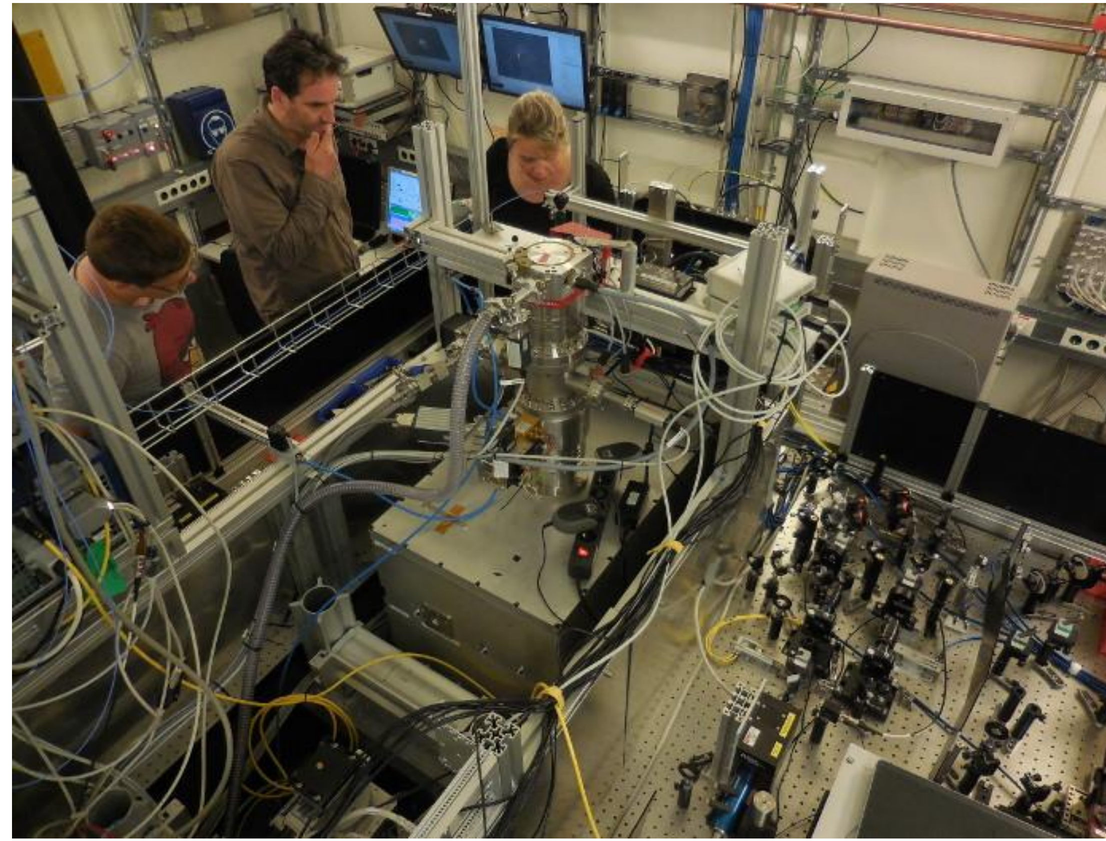
Experimental layout

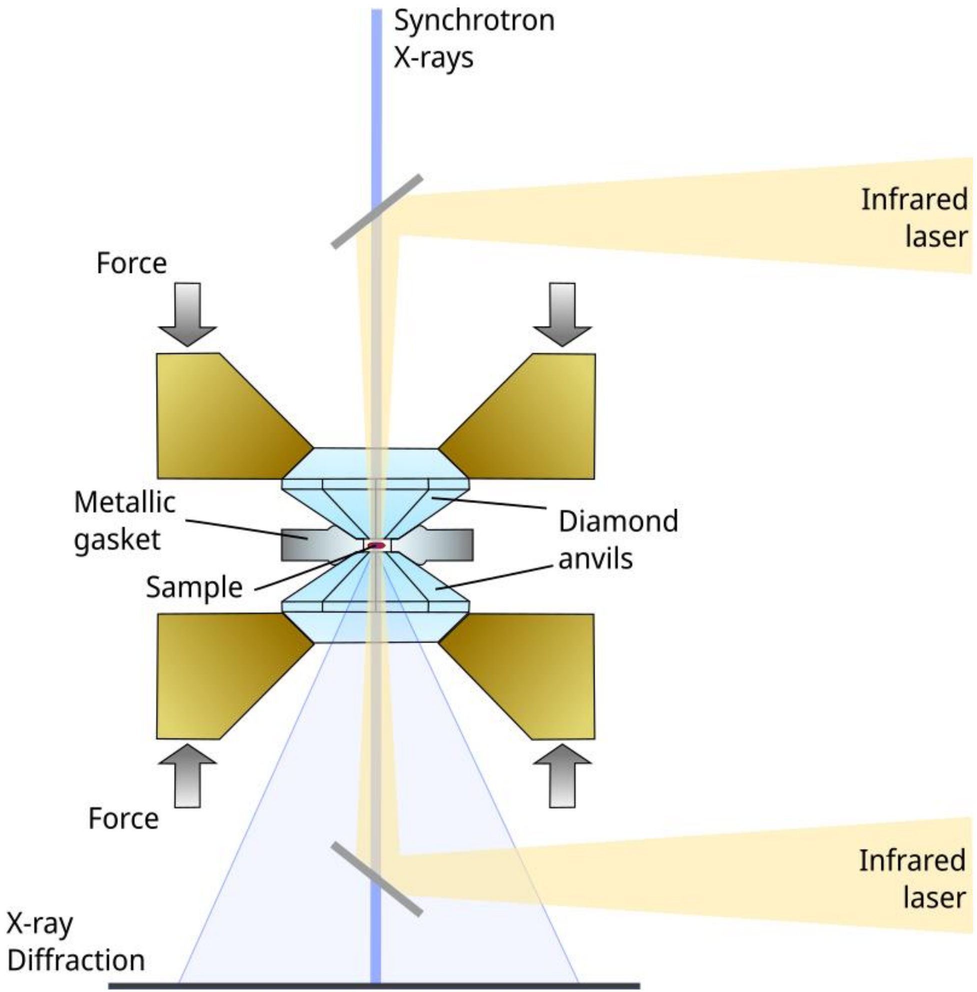




X-ray diffraction + laser heating + diamond anvil cell
P02.2, PETRA III, Hambourg

High quality equivalent beamline at GSECARS, APS

Specifics of LH-DAC experiments



Sample is small

- ~20 to 50 μm diameter
- ~10 μm thickness
- Grain sizes: ~ 1 μm or less

Laser heating

- Shape of the laser → typically gaussian
- Homogeneous temperatures over ~5 μm diameters

X-ray

- Beam focused to less than 5 μm
- Available 2θ range: up to ~25°

Follow grain rotations under pressure?

Goal

- Use
  - A diamond anvil cell
  - Synchrotron x-ray diffraction
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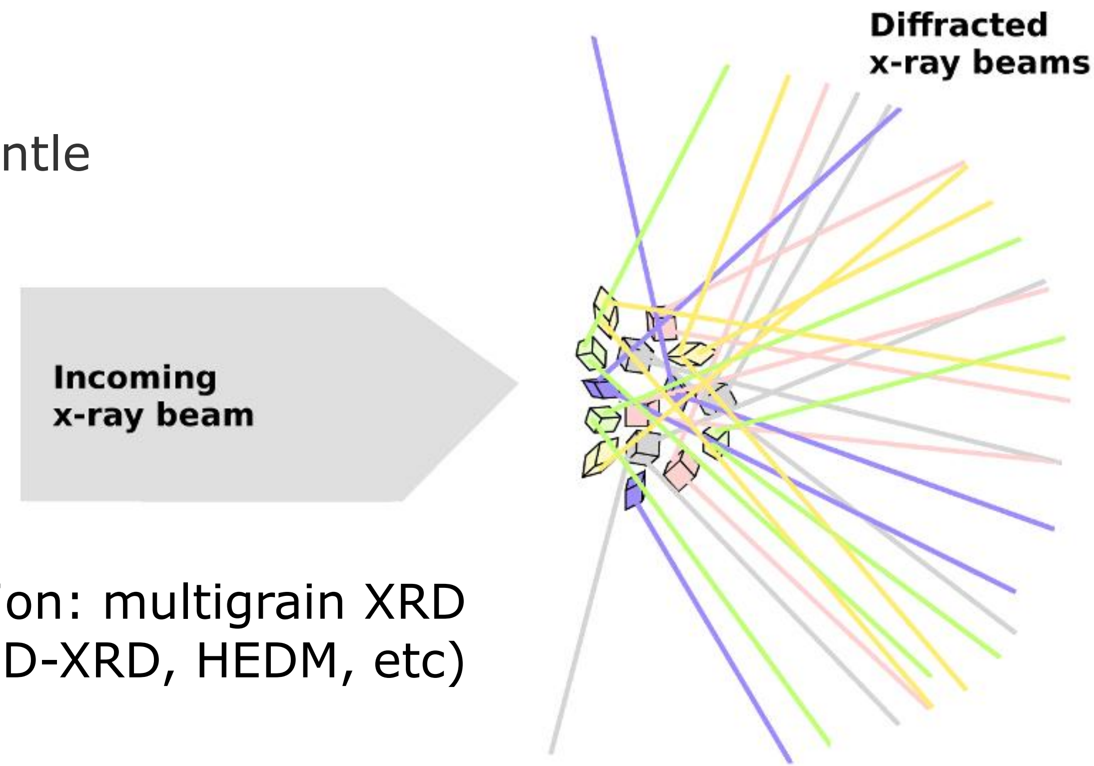

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(3D-XRD, HEDM, etc)

Follow grain rotations under pressure?

Goal

- Use
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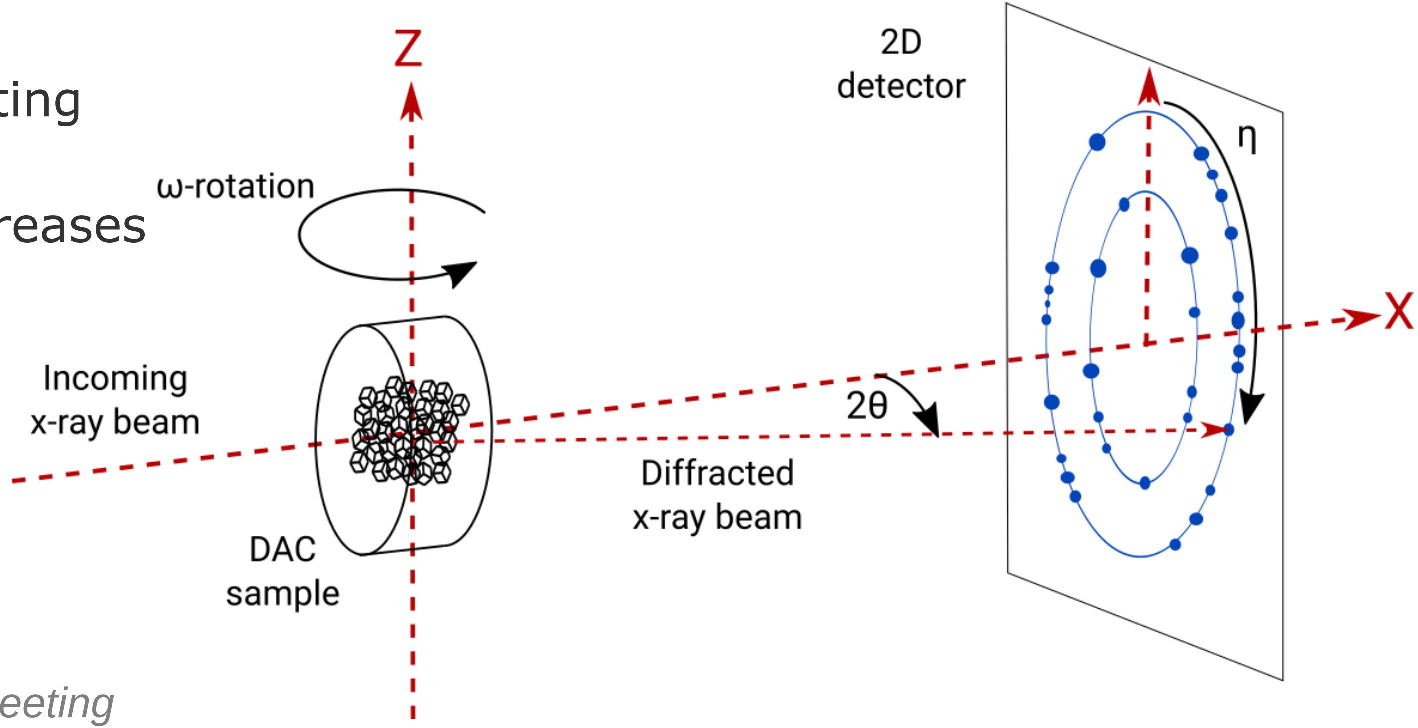
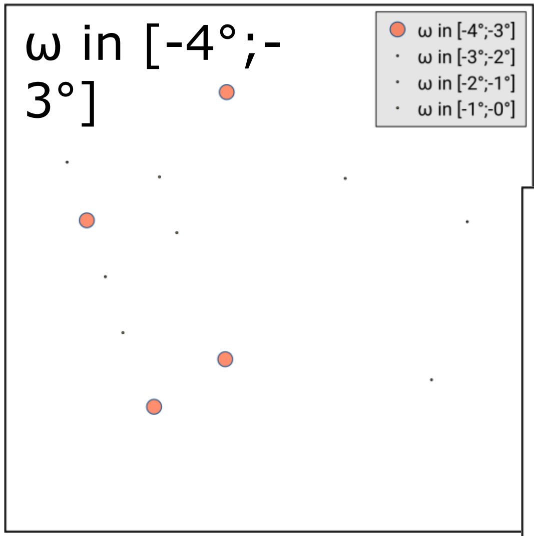
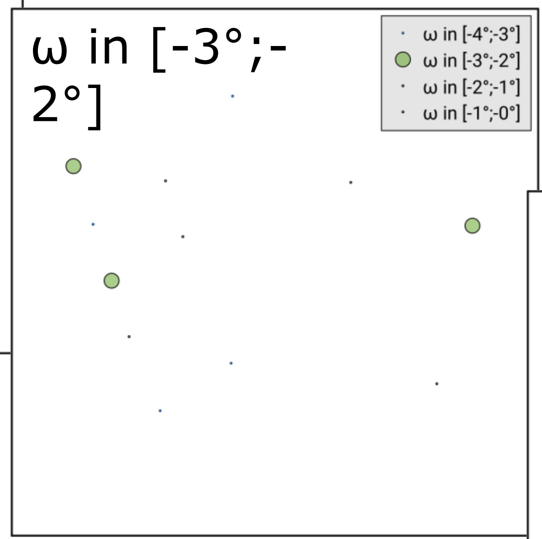
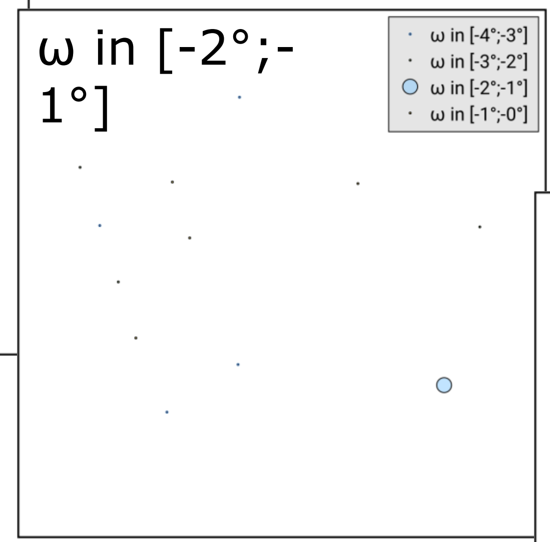
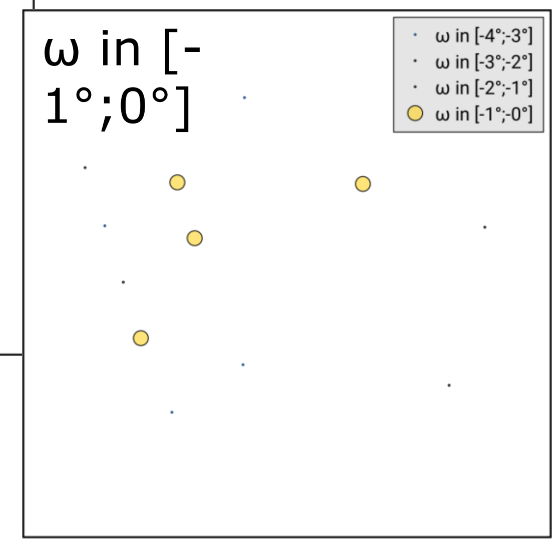


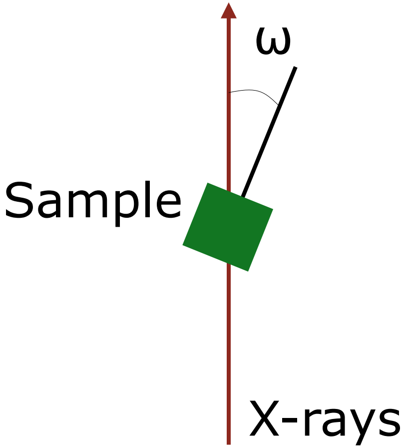
X-ray diffraction: single crystal





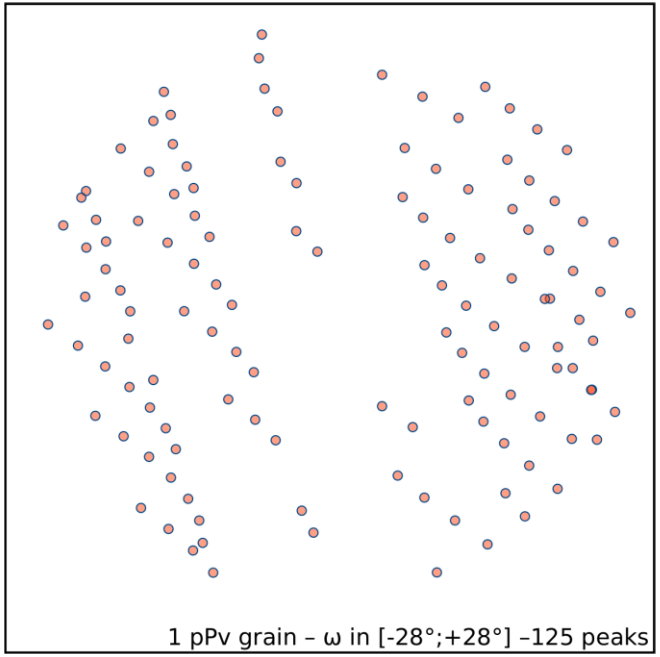




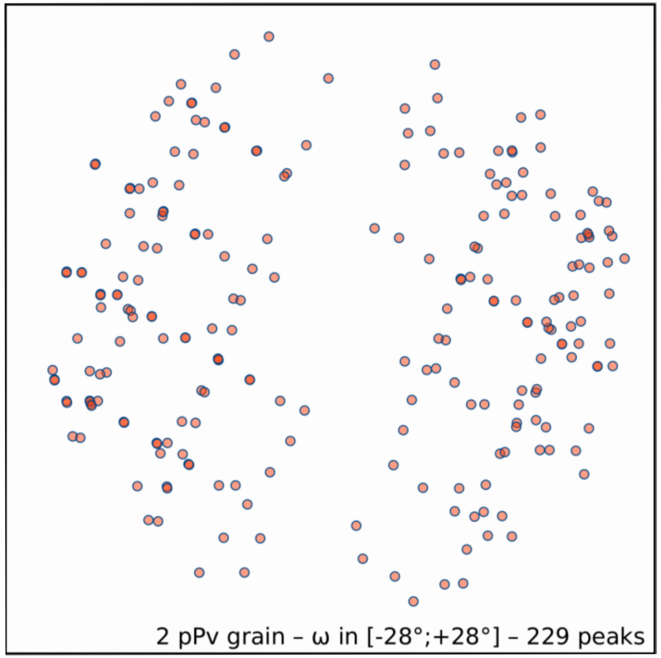


Polyxsim simulations for post-perovskite (orthorhombic *Cmcm* space group)
Random crystal orientation
Ideal peak positions

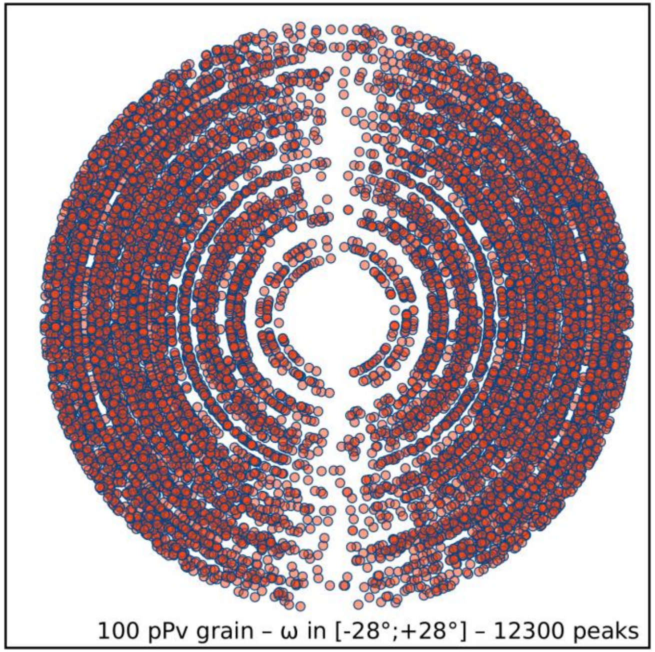
X-ray diffraction stackings



ω in [-28°;+28°]
1 grain
125 peaks in 2θ range



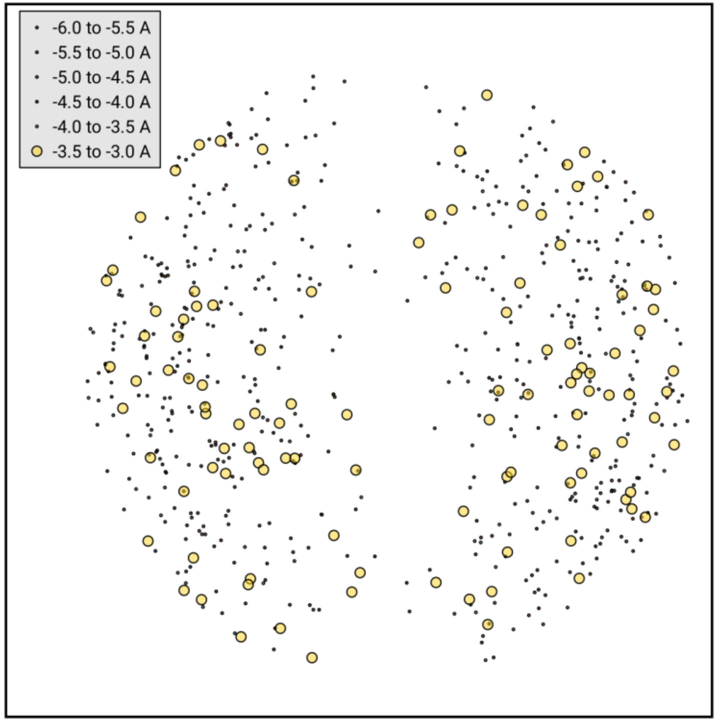
ω in [-28°;+28°]
2 grains
229 peaks in 2θ range



ω in [-28°;+28°]
100 grains
12300 peaks in 2θ range

Polyxsim simulations for post-perovskite (orthorhombic Cmcm space group)

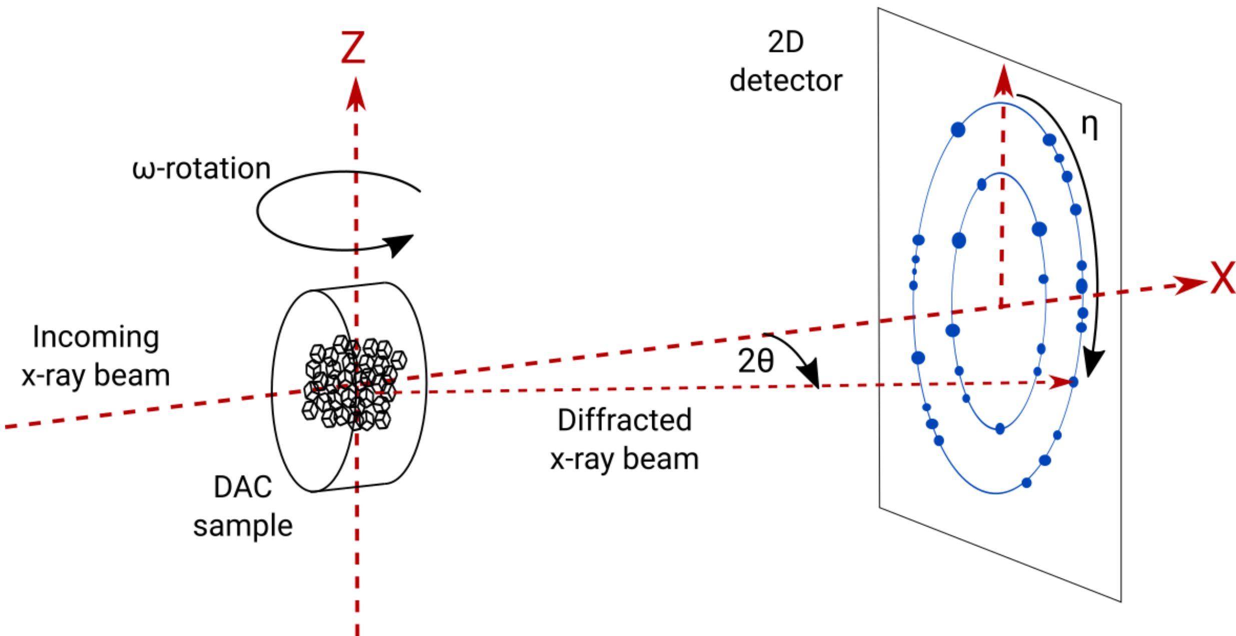
Multigrain X-ray diffraction in the DAC



DAC sitting on ω rotation stage

Collect data every δω step (0.5°) over Δω range (-28° to +28°)

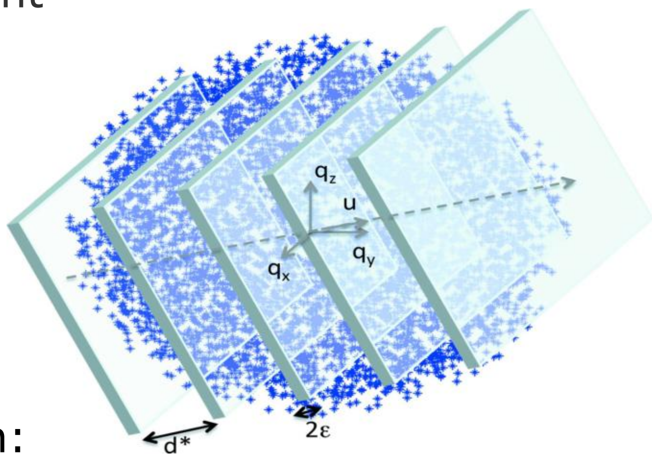
Extract diffraction spot database with 2θ, η, ω, intensity for each



Indexing procedure

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

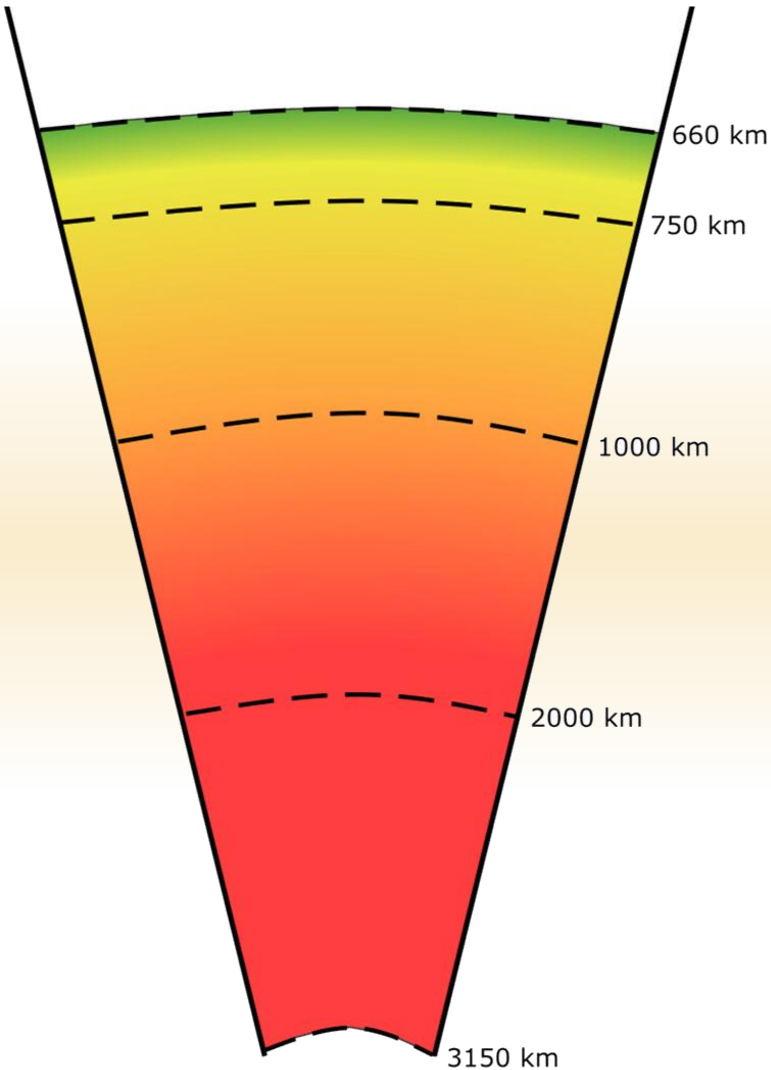
Average sample

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

Grain scale, for each indexed grain

- Orientation
- Cell parameters
- Relative volume

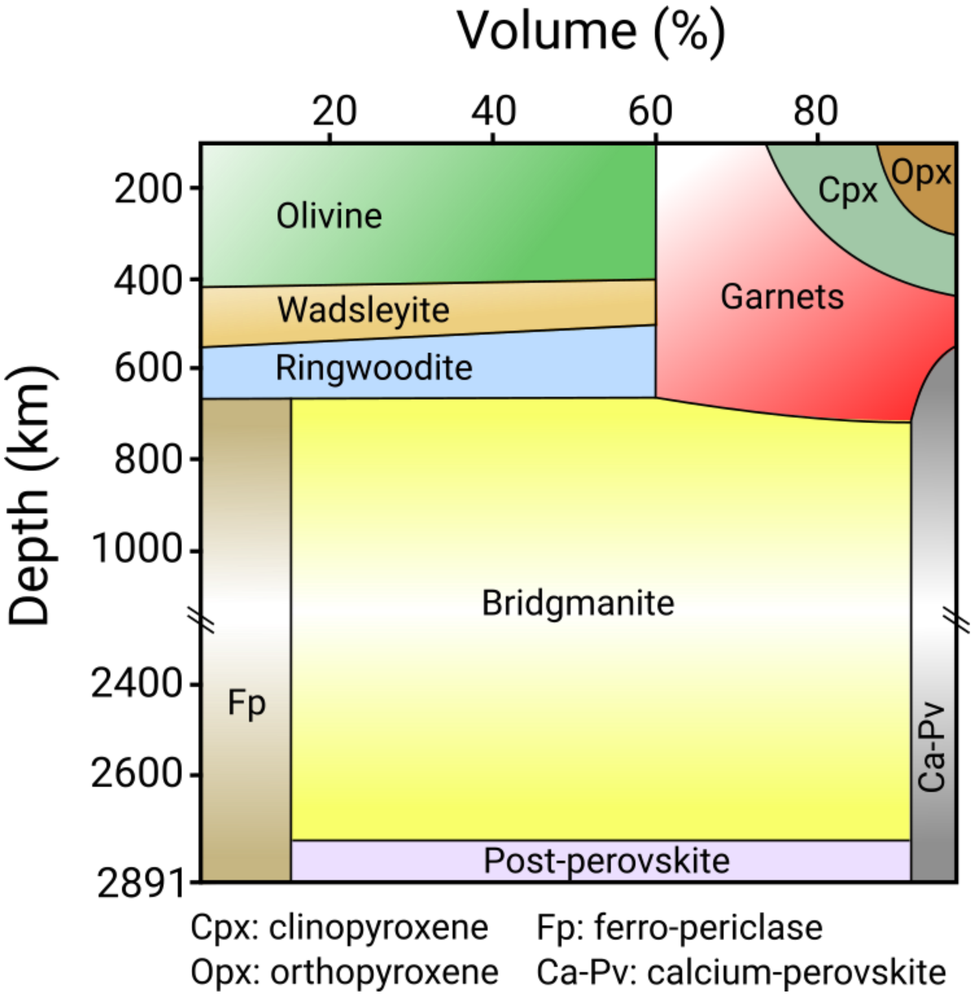
Nisr *et al*, *J. Geoph Res.* 2012
Nisr *et al, High Pres. Res.* 2014
Rosa *et al*, *J. Appl. Cryst.* 2015
Rosa *et al*, *J. Geoph Res.* 2016
Langrand *et al*, *J. Appl. Cryst.* 2017
Gay et al, *EPSL*, 2023
Ledoux et al, *Am. Min.* 2023
Ledoux et al, G^3, 2023
Gay et al, *Geophys. Res. Lett.*, 2024



Application example : Pyrolite microstructures in the lower mantle

Gay et al, *EPSL*, 2023
Gay et al, *GRL*, 2024
Magali et al, *Nature Geoscience*, in review

Phase transformations and deformation in pyrolite



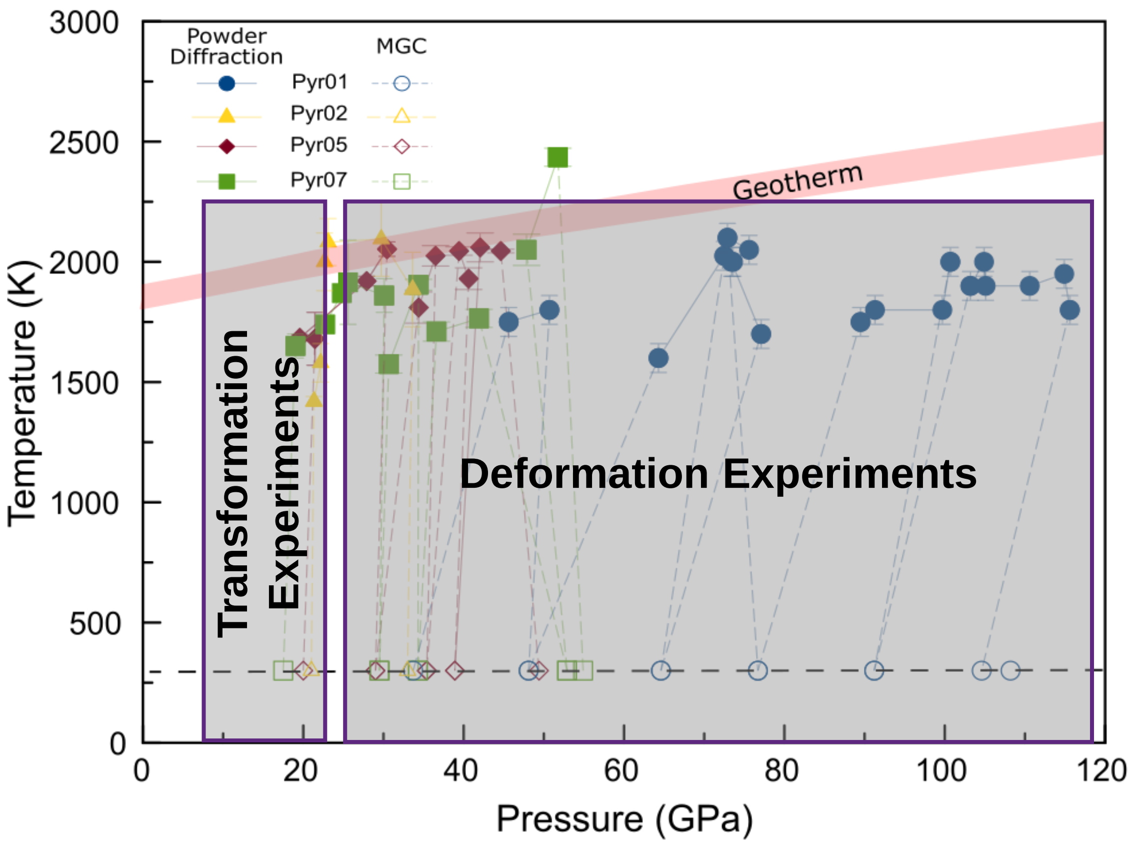
Pyrolite = model composition for the Earth's mantle

- In the upper mantle: 60% olivine, garnet, pyroxenes
- 600 km: 60% ringwoodite, garnet
- 1000 km: 15% ferropericlase, 75% garnet, 10% davemaoite

Effect of phase transformations on grain orientations?

Effect of deformation on grain orientations?

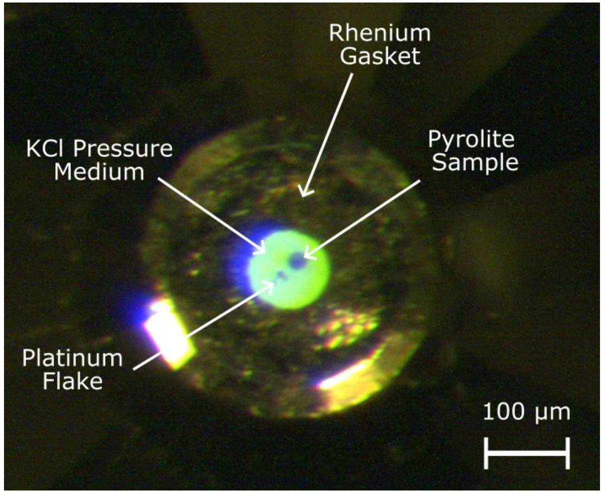
Experimental P/T path



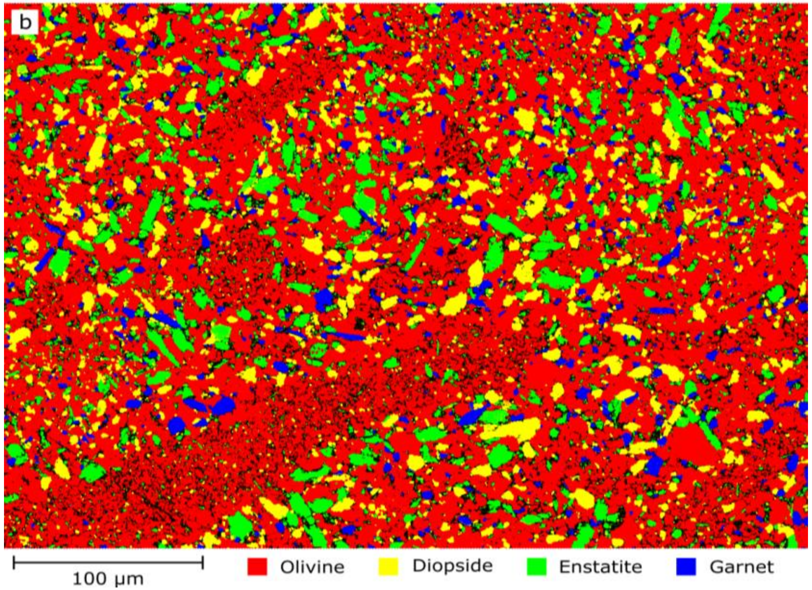
- 4 experiments on pyrolitic composition
- Trying to follow geotherm temperatures
- Powder diffraction images taken to monitor phase occurrence
- Pressure increases and phase transformations at high P and T
- Multigrain to characterize sample microstructures collected after quenching as close to transformation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

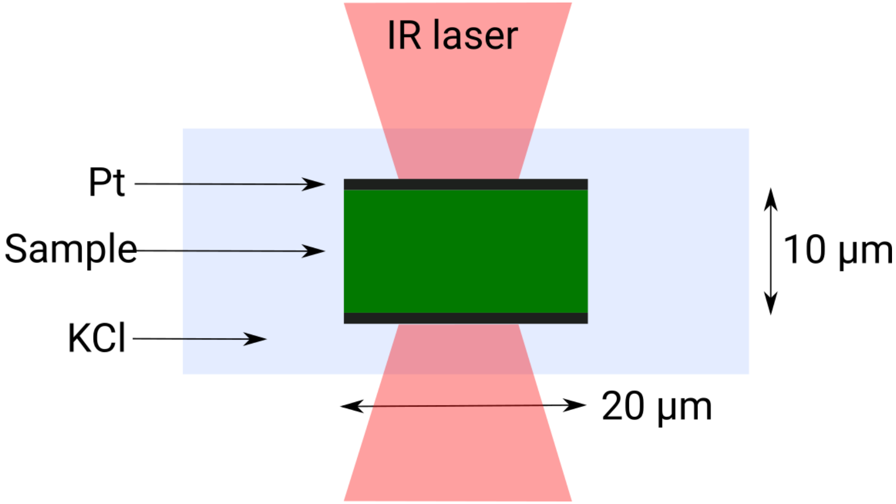
Sample preparation

Process
1) Pyrolitic composition sintered in a piston-cylinder
2) Cut and polished to 20 μm diameter / 10 μm thickness
3) Coated with 200 nm Pt
4) Loaded in DAC with KCl pressure medium
5) Laser heating + XRD



Starting material EBSD phase map

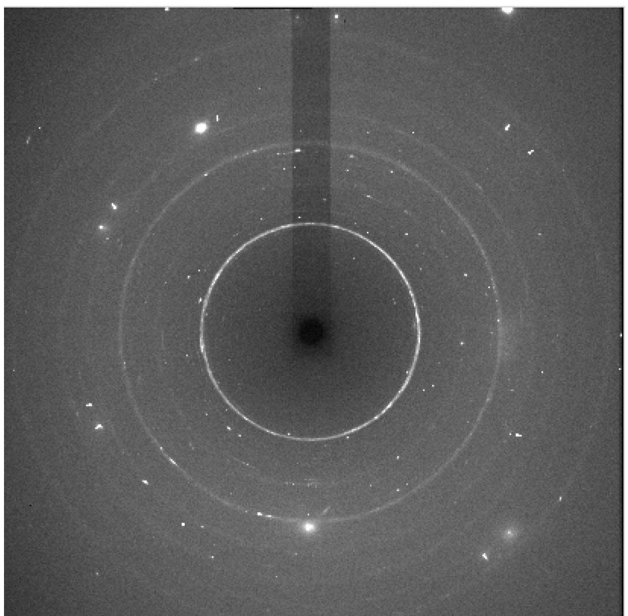


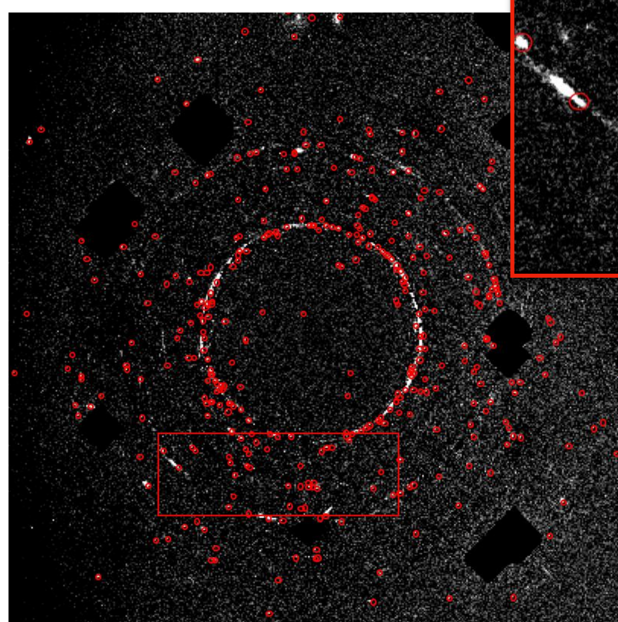


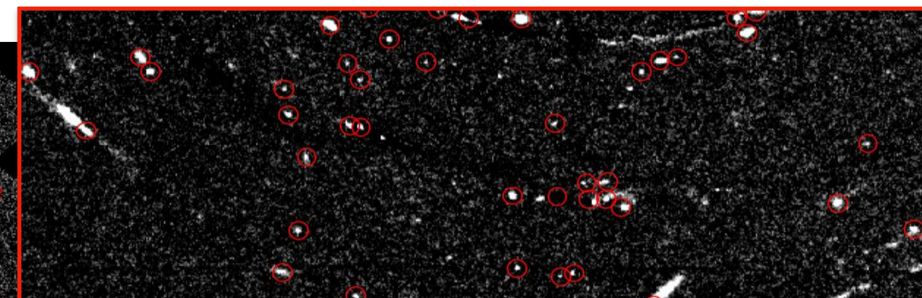
Sample data

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

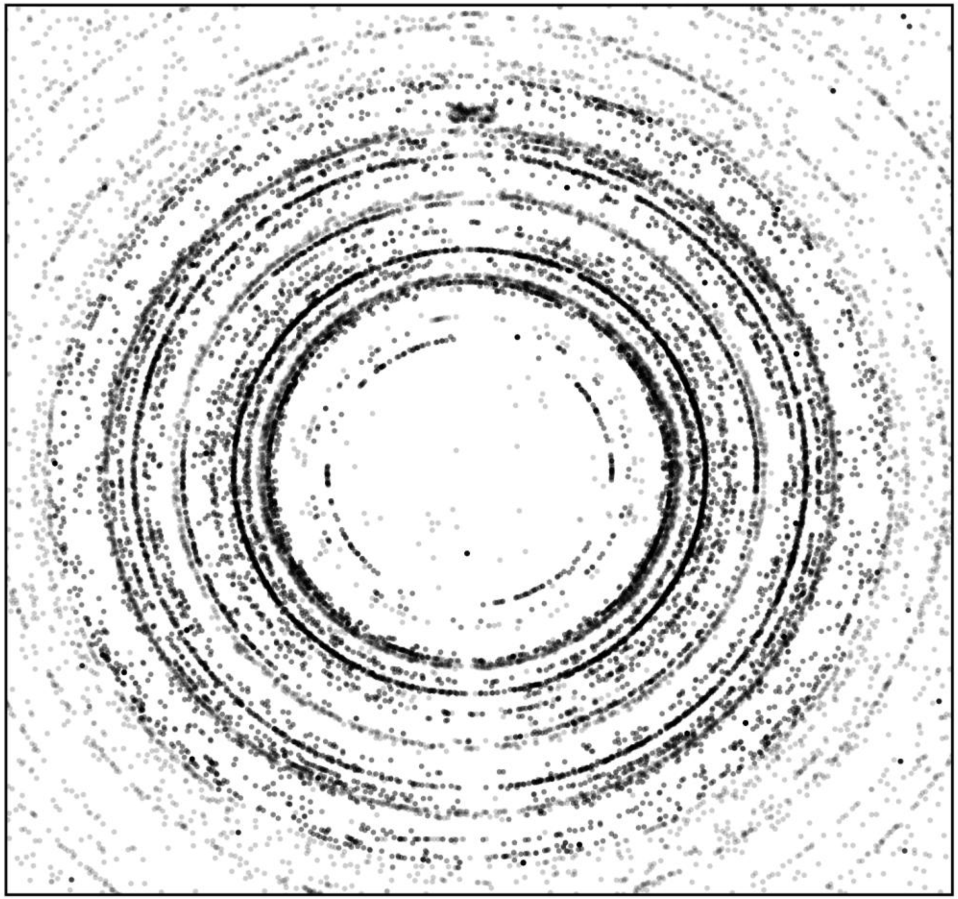






P02.2

Phase identification – Step 1
Peaks extraction



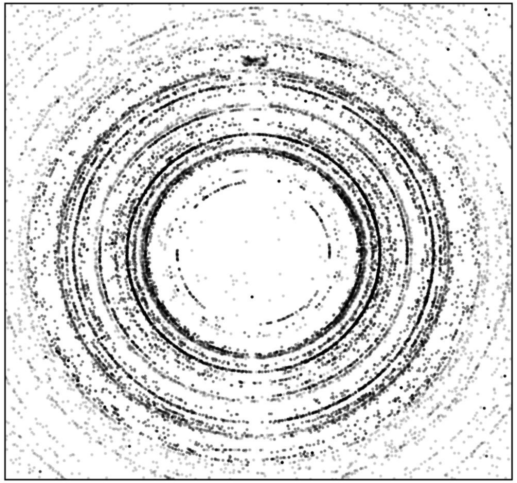
Pyrolite + KCl, 34 GPa

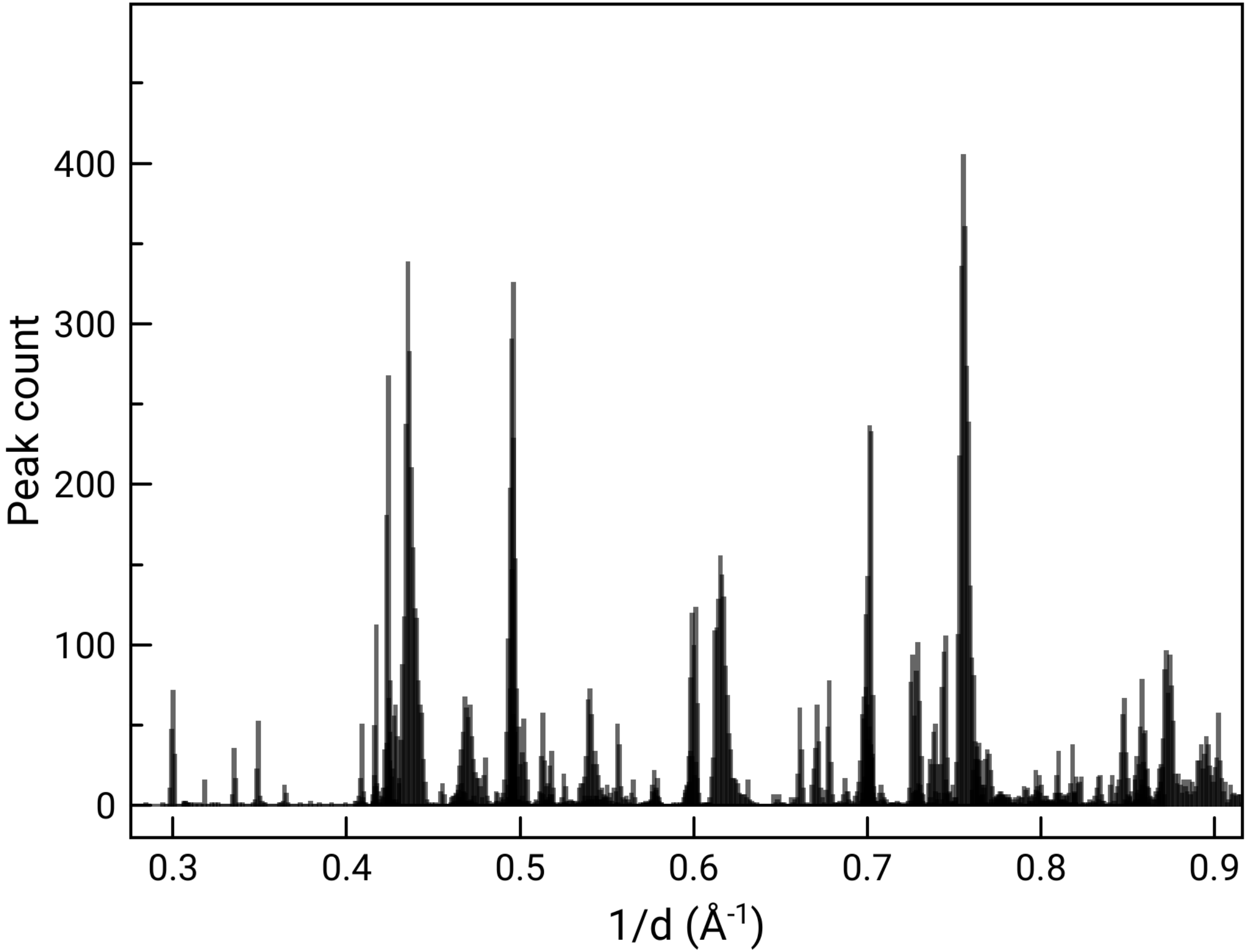
Potential phases:

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

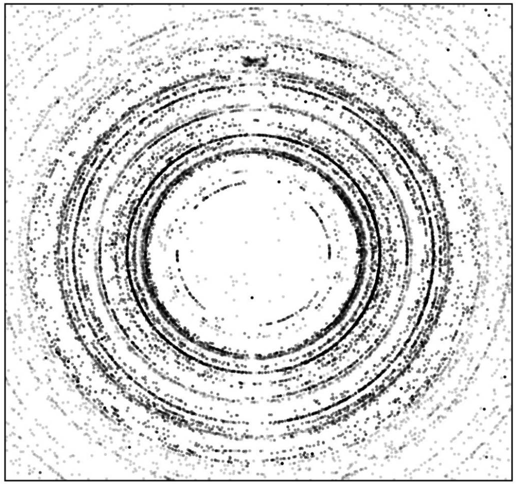
24000 extracted peaks, with known mean 2θ, η, ω, intensity for each

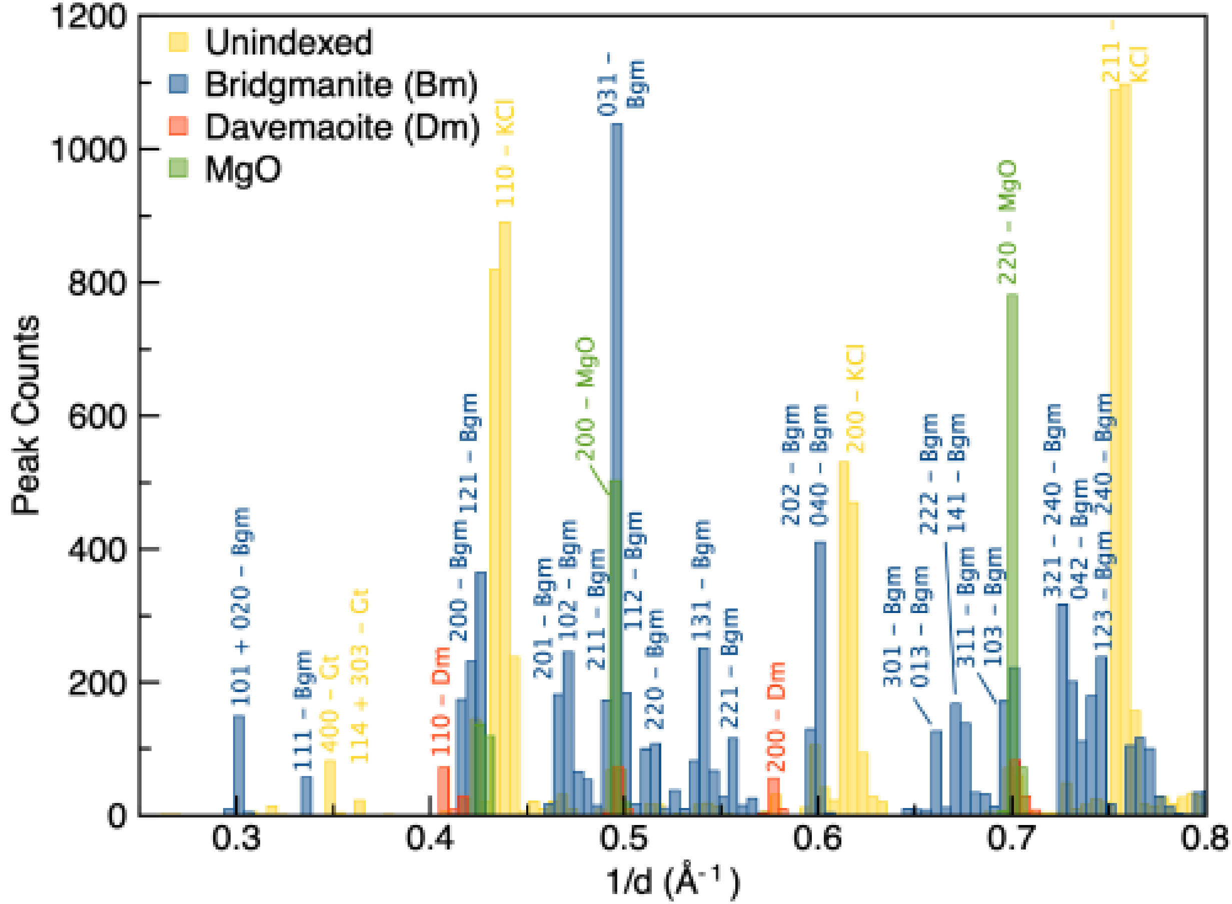
Phase identification – Step 2
Peak histogram vs. 1/d (or 2θ)





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)

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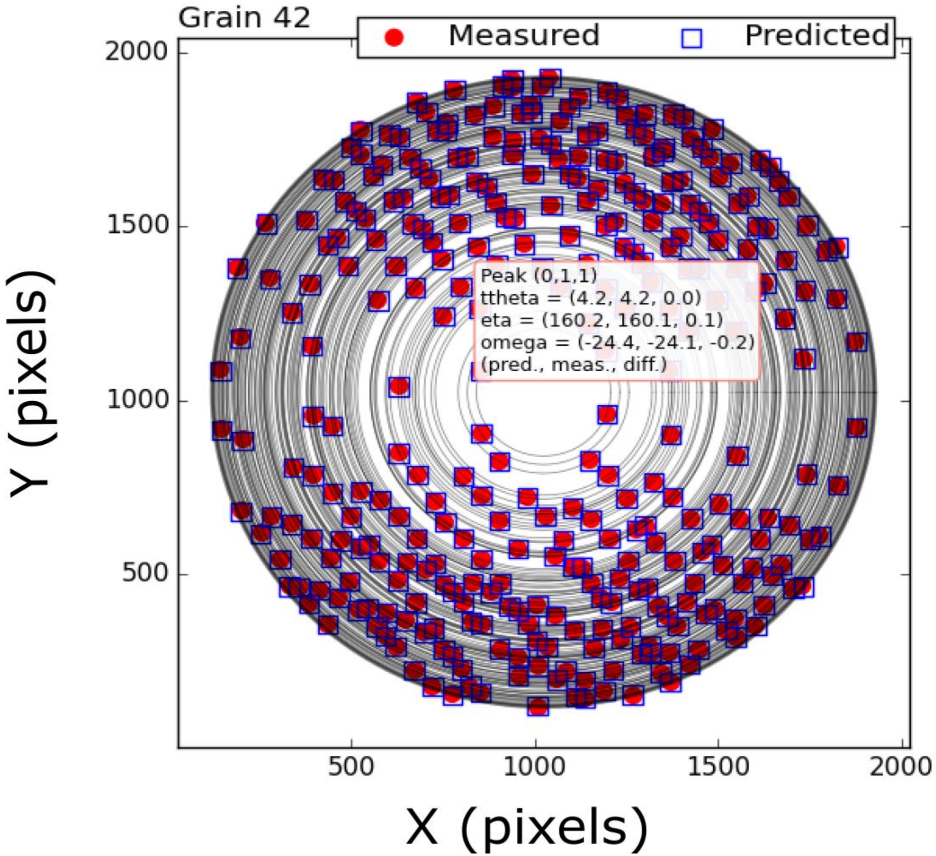
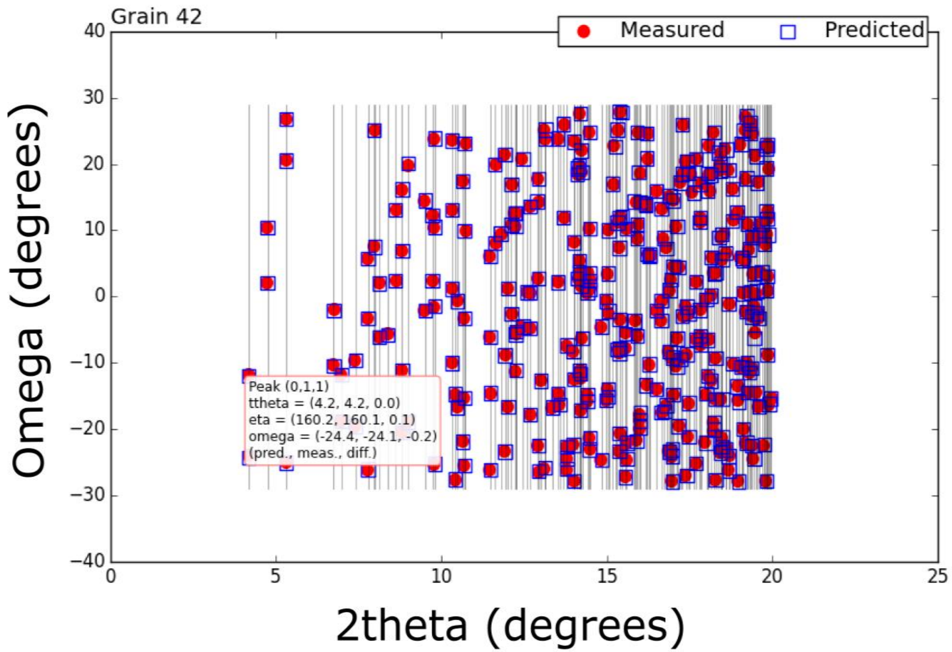
Sample indexing result





Sample bridgmanite grain inside a polycrystal

Peaks:

- Squares: predicted
- Circles: measured

Indexing, Pyrolite + KCl, 34 GPa

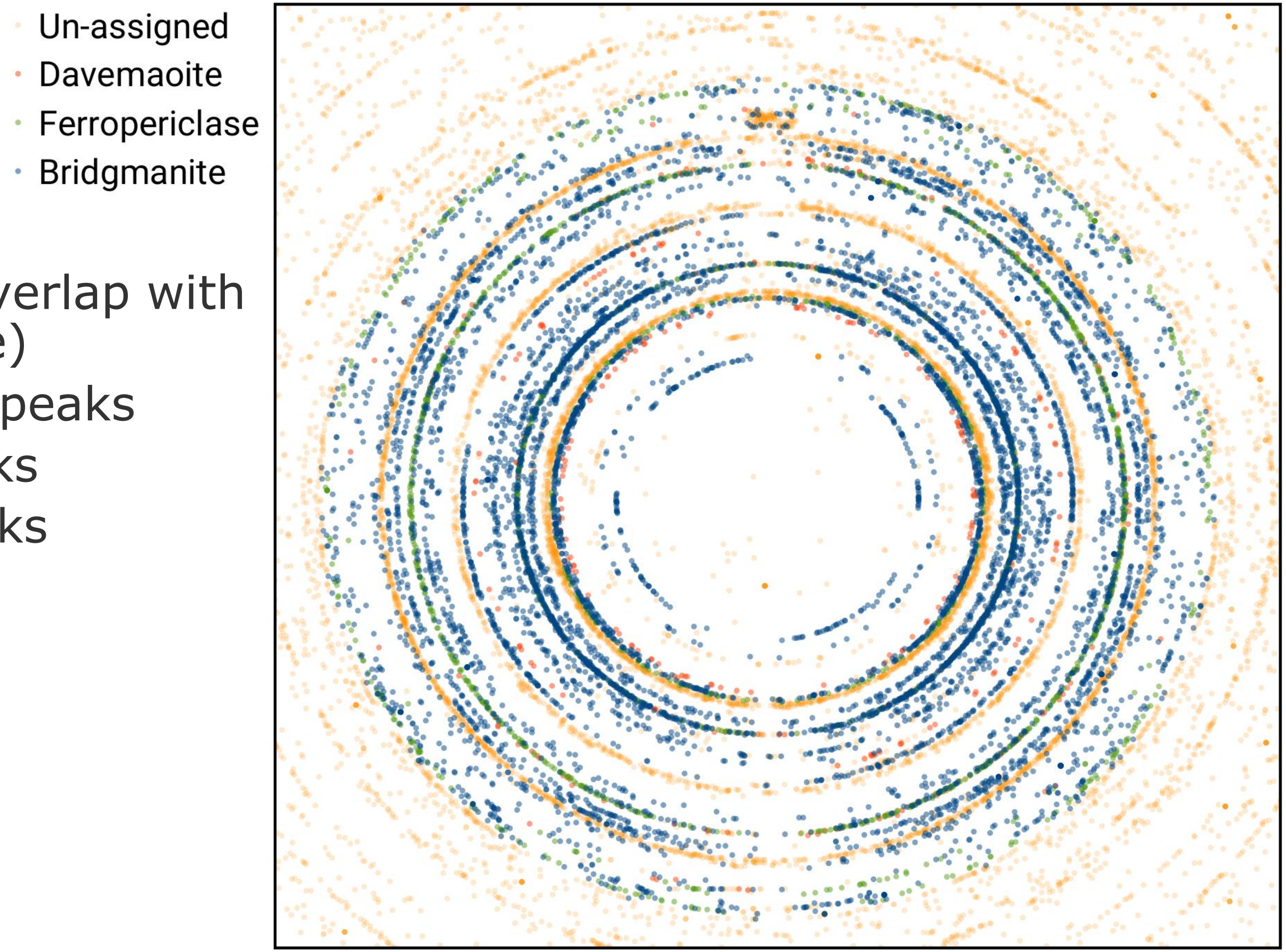
GrainSpotter Indexing

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



Indexing, Pyrolite + KCl, 34 GPa

GrainSpotter Indexing

Indexed grains

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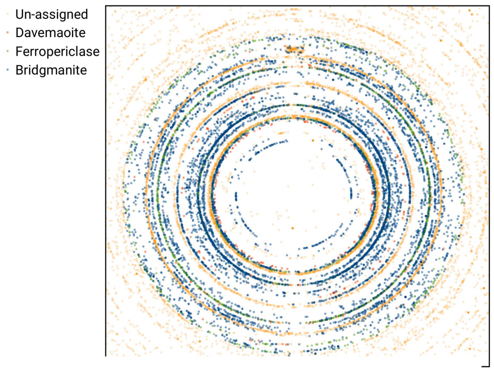
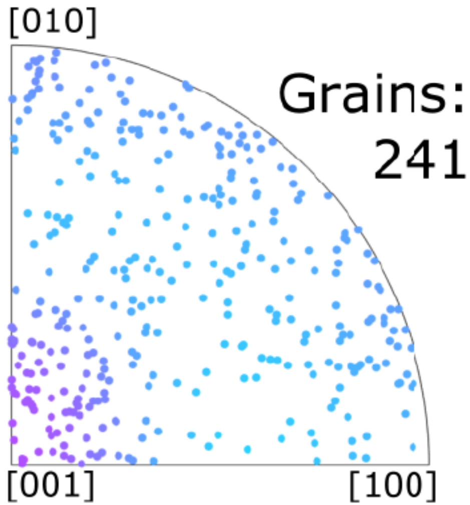
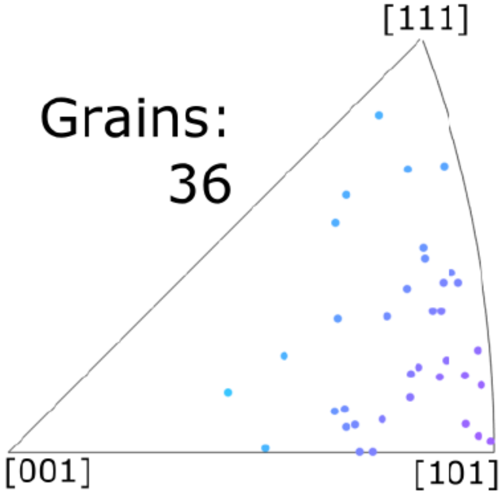
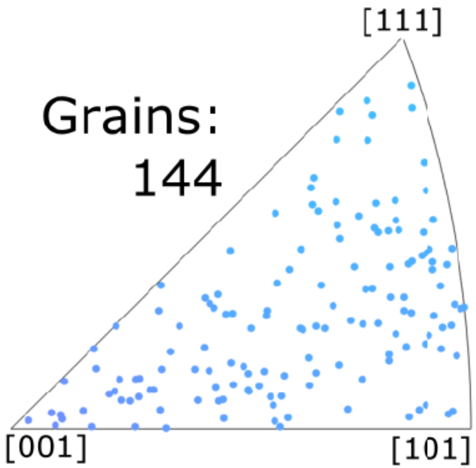




Bridgmanite grain orientations



Davemaoite grain orientations

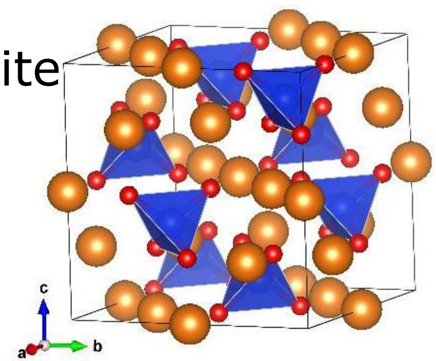


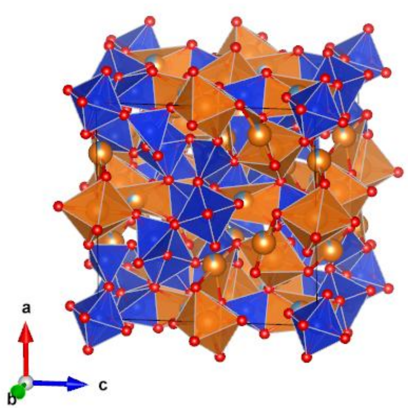
Ferropericlase grain orientations

660 km discontinuity

Lower transition zone

60% ringwoodite
$(Mg,Fe)_2SiO_4$

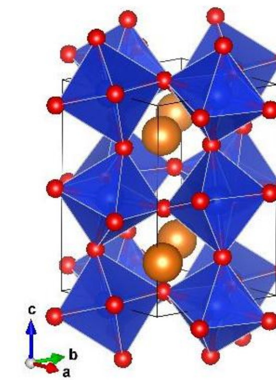




40% garnet
$(Mg,Fe,Al,Ca)_3(Al,Fe)_2(SiO_4)_3$

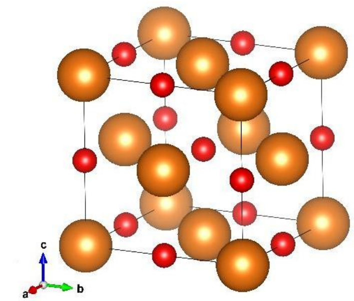
660 km
24 GPa
1900 K

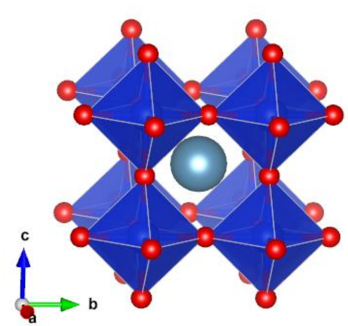
Lower mantle



80%
bridgmanite
$(Mg,Fe)SiO_3$

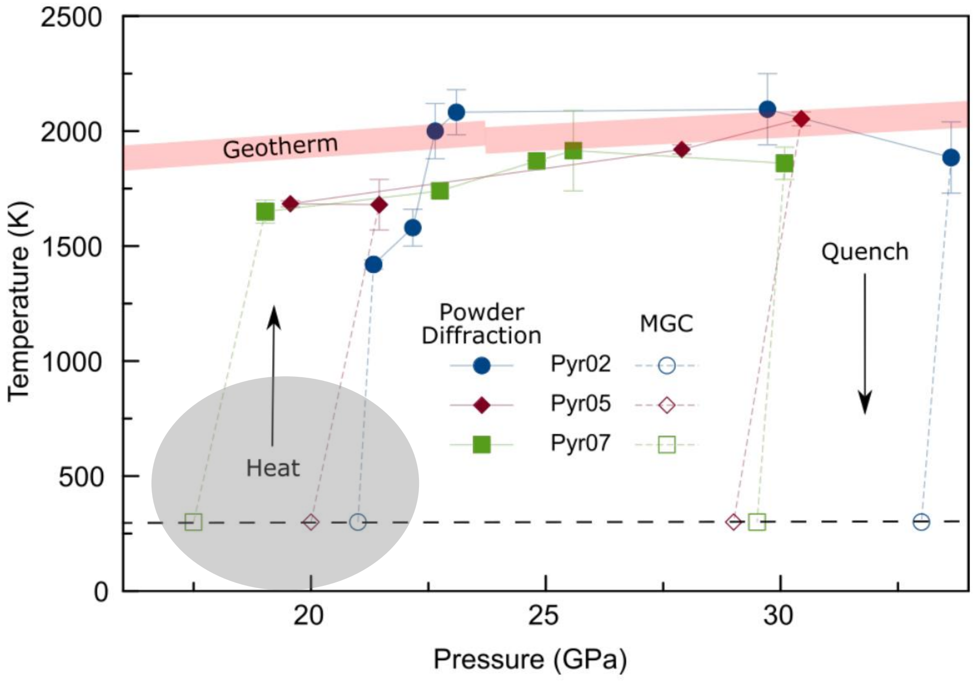
15%
ferropericlase
$(Mg,Fe)O$

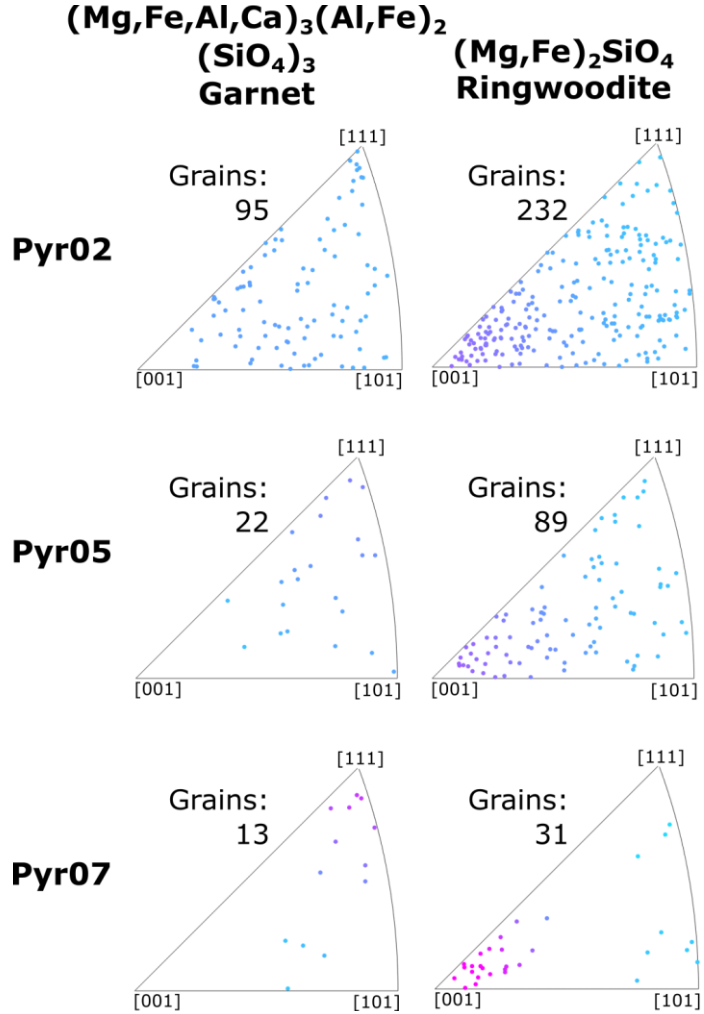




5%
davemaoite
$CaSiO_3$

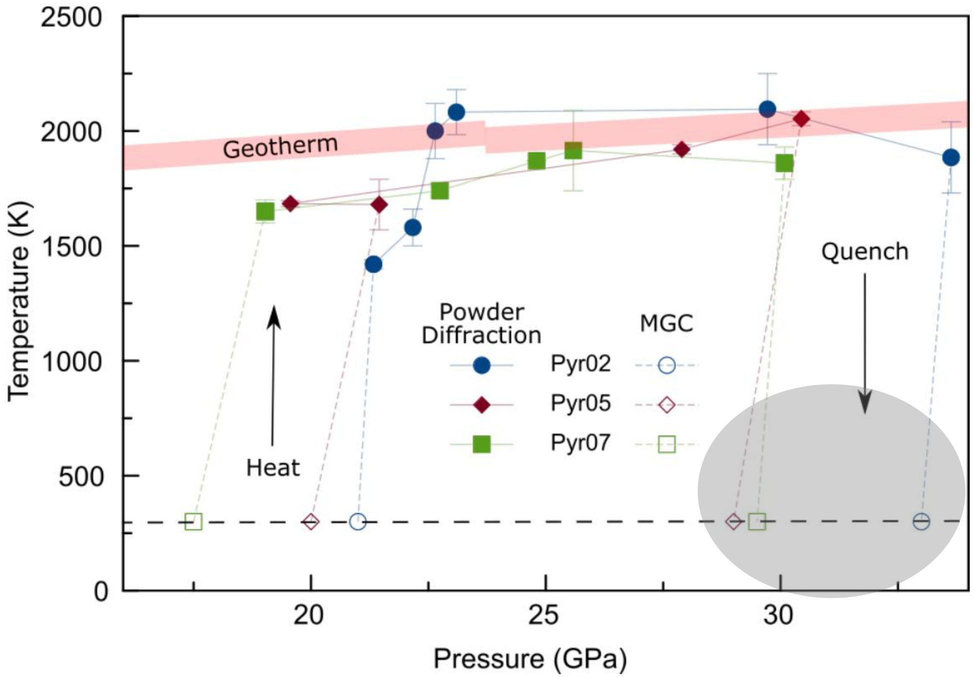
660 km discontinuity experiment

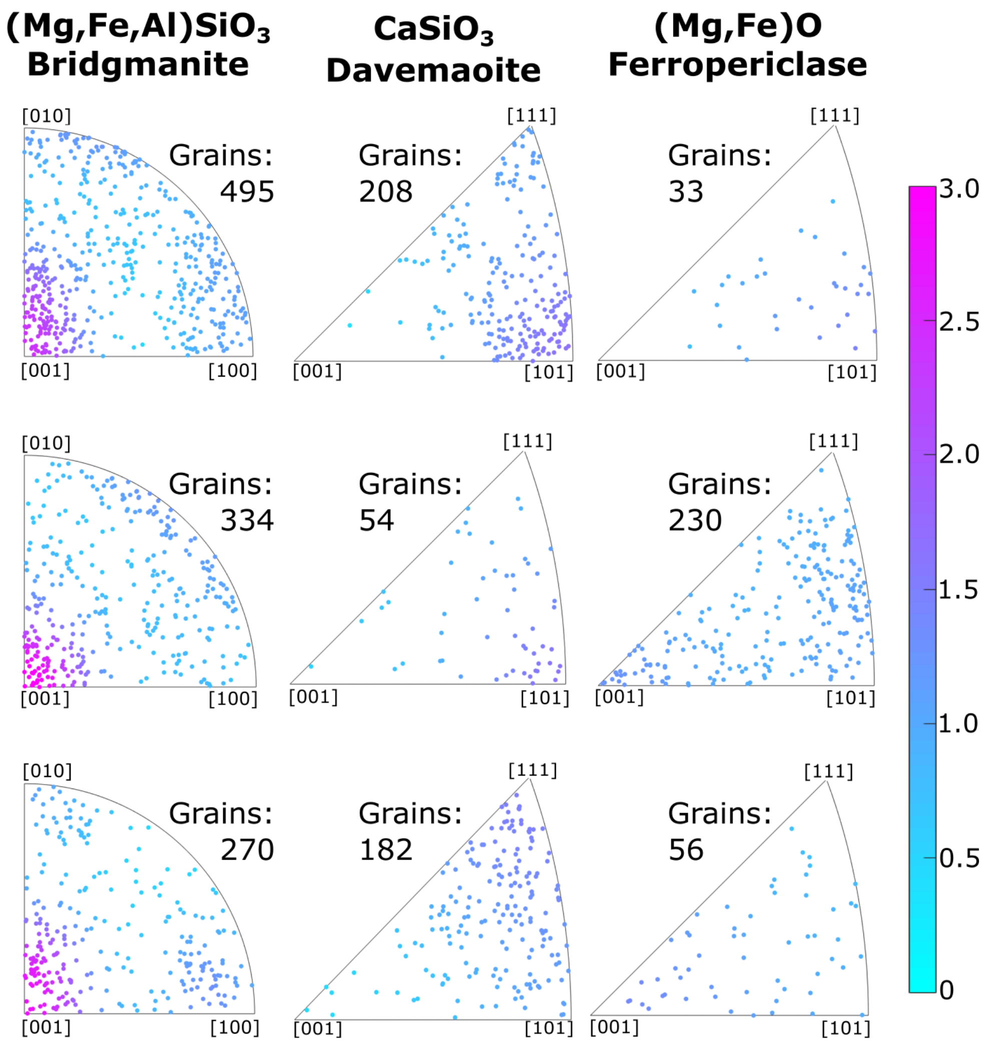




Gay et al, EPSL, 2023

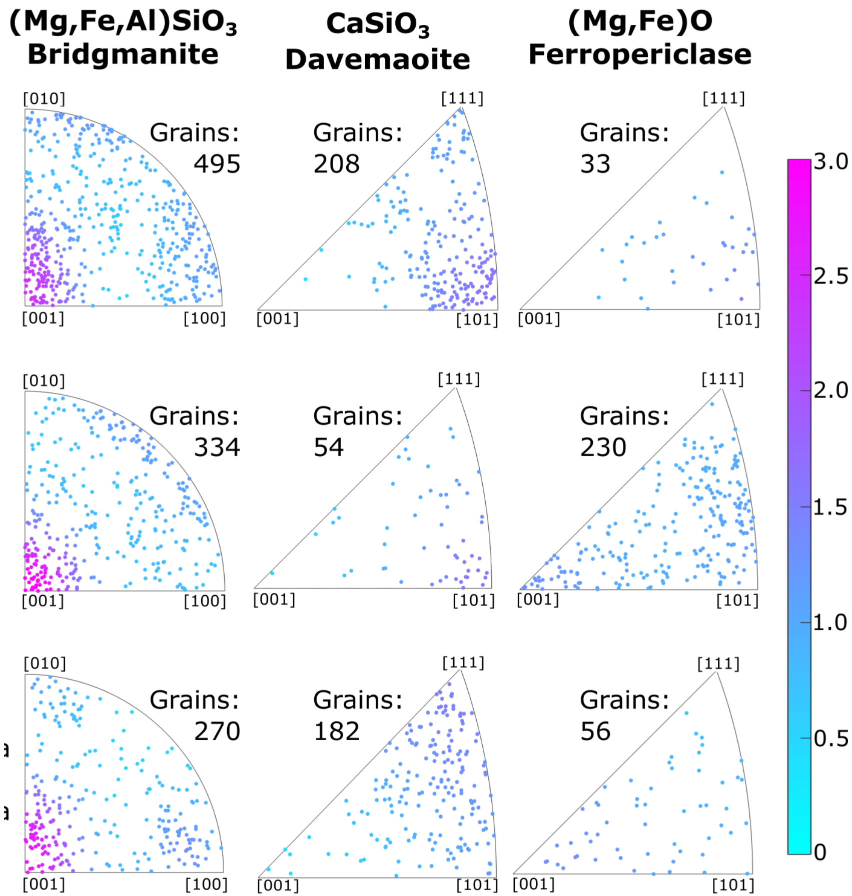
660 km discontinuity experiment





Gay et al, EPSL, 2023

660 km discontinuity experiment



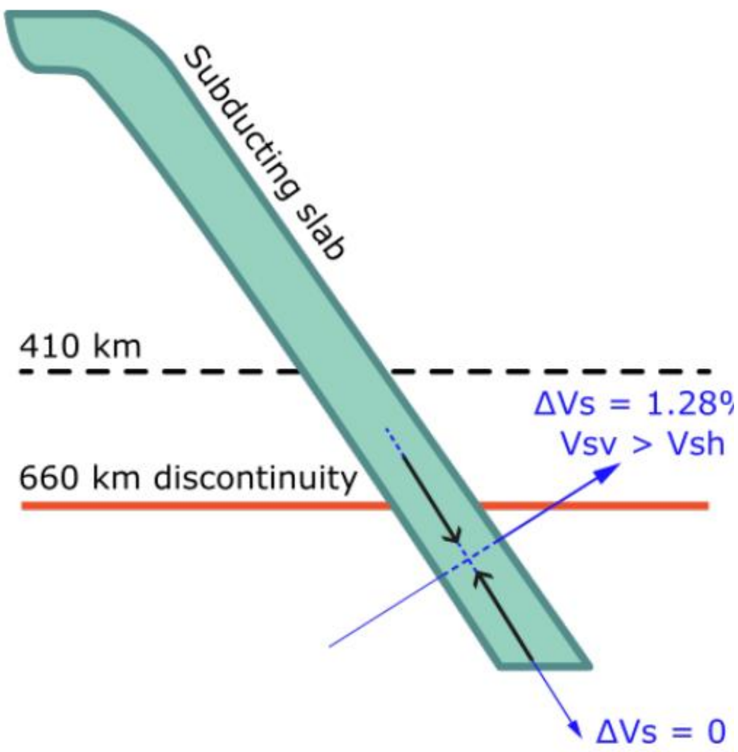
Systematic 001 transformation texture in bridgmanite

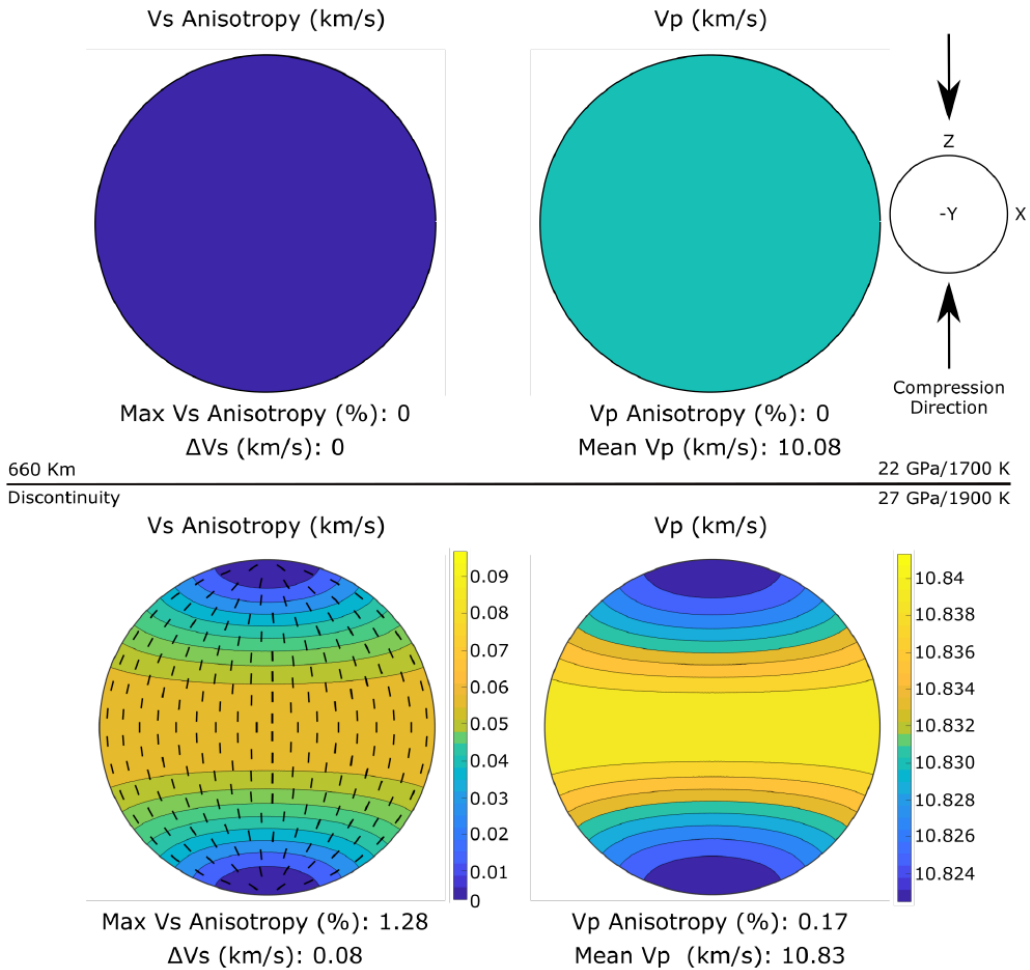
011 / 111 transformation texture in CaPv / davemaoite

Origin: nucleation / growth under compressive stress

Gay et al, EPSL, 2023

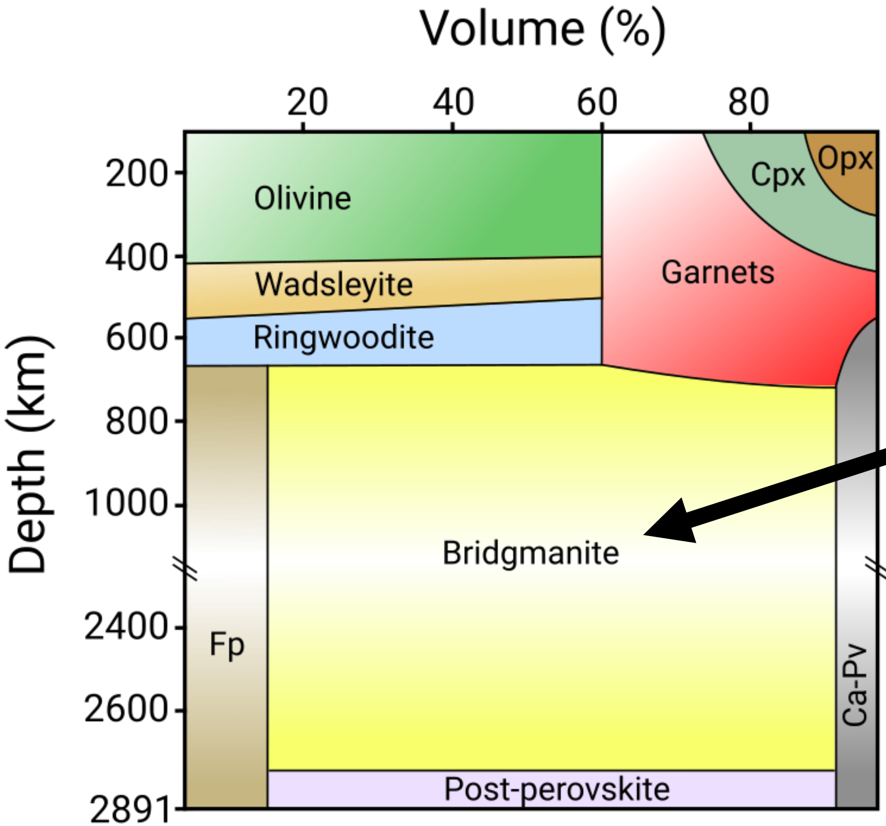
Seismic anisotropy in subducting slab



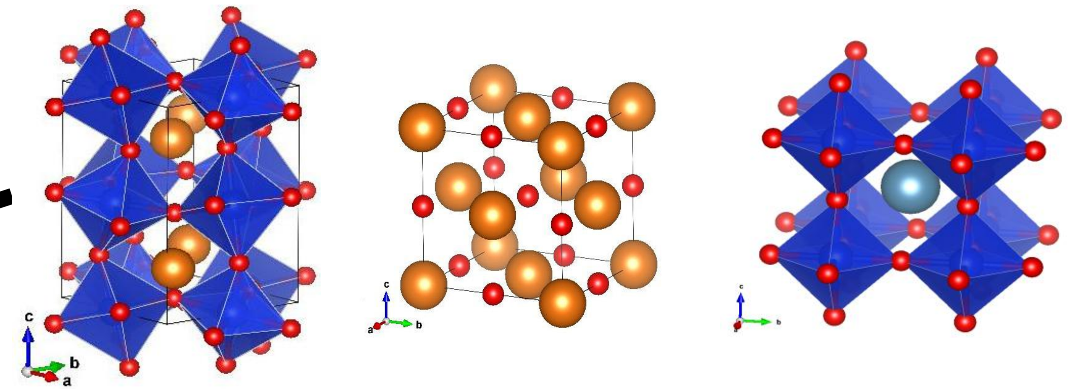


Gay et al, EPSL, 2023

Deep mantle deformation



Cpx: clinopyroxene
Opx: orthopyroxene
Fp: ferro-periclase
Ca-Pv: calcium-perovskite



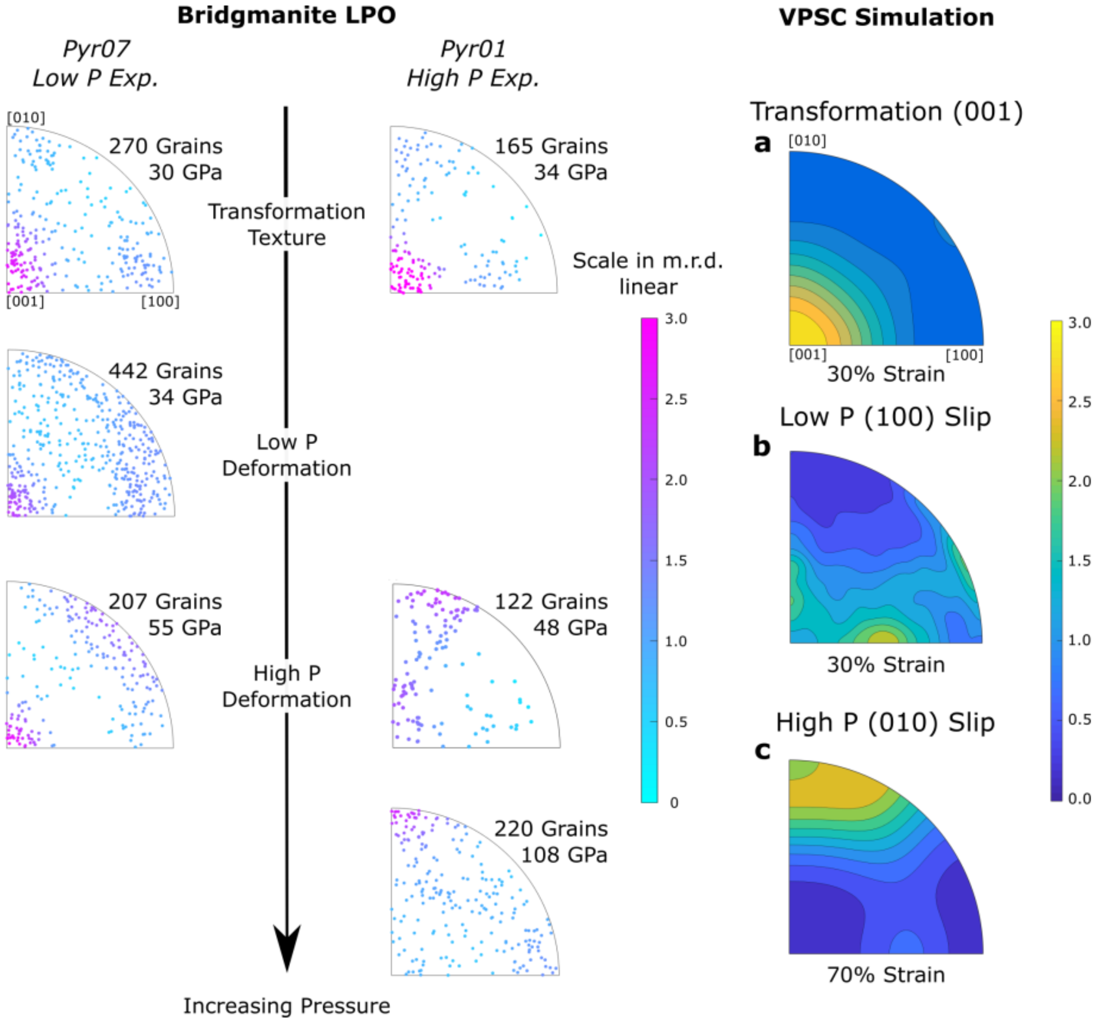
Bridgmanite
$(Mg,Fe)SiO_3$

Ferropericlase
$(Mg,Fe)O$

Davemaoite
$CaSiO_3$

Gay et al, GRL 2024

Deep mantle deformation



Identification of 3 regimes of grain orientations

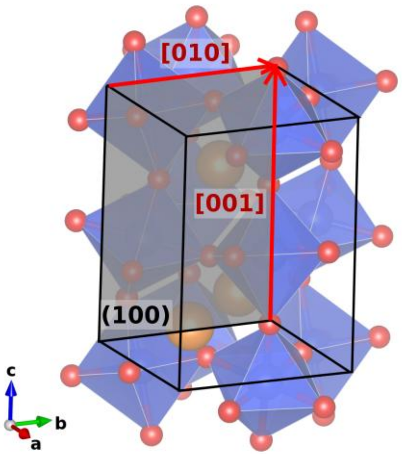
- Transformation microstructures, with (001) orthogonal to maximum stress direction
- Deformation below ~50 GPa, dominated by slip on [001](100)
- Deformation at P >~ 50 GPa, dominated by slip on (010), either [100] or [001]

Gay et al, GRL 2024

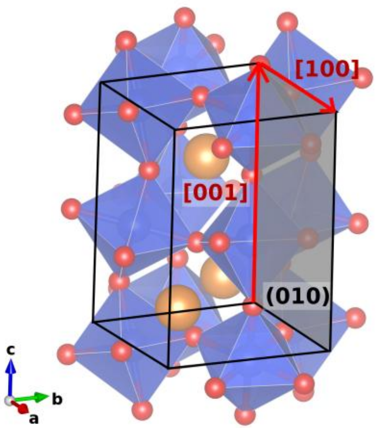
Deep mantle deformation

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

	Low P		High P	
Slip system	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



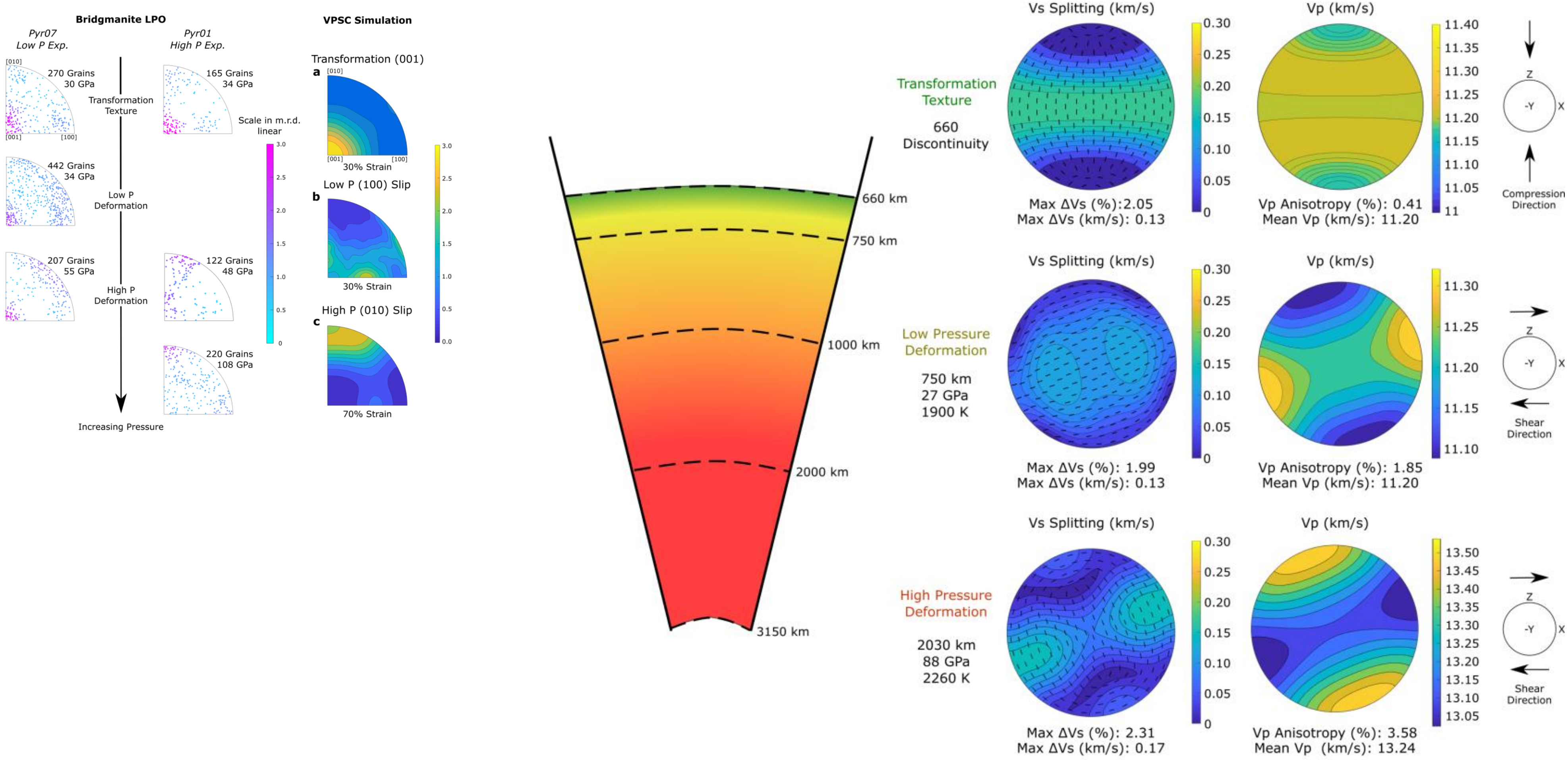
Below
<~ 50 GPa



Above
>~ 50 GPa

Gay et al, GRL 2024

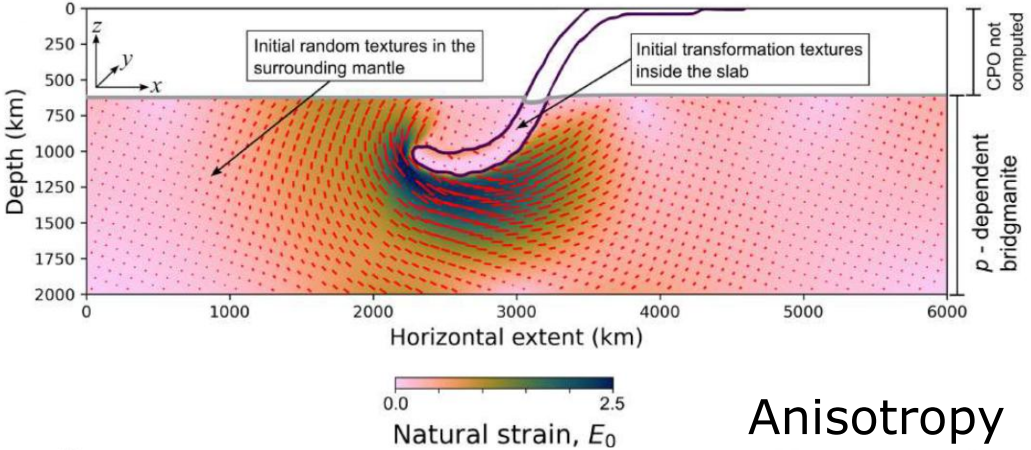
Anisotropy in the Earth's deep mantle



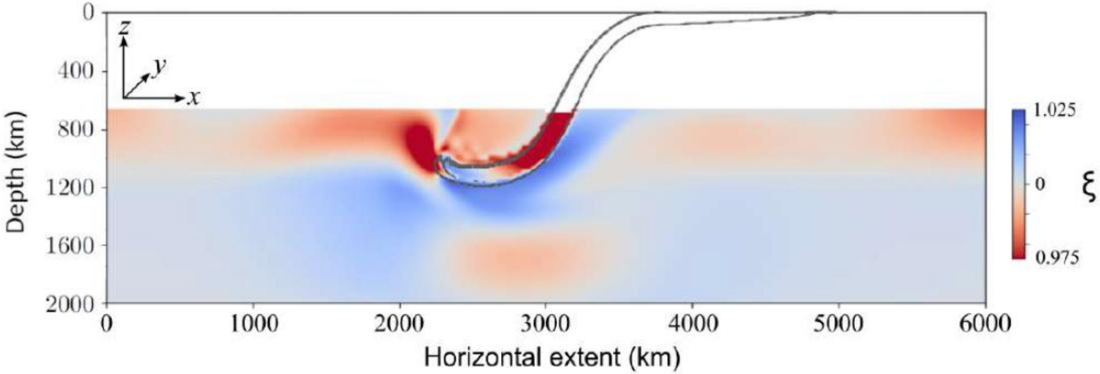
Gay et al, GRL 2024

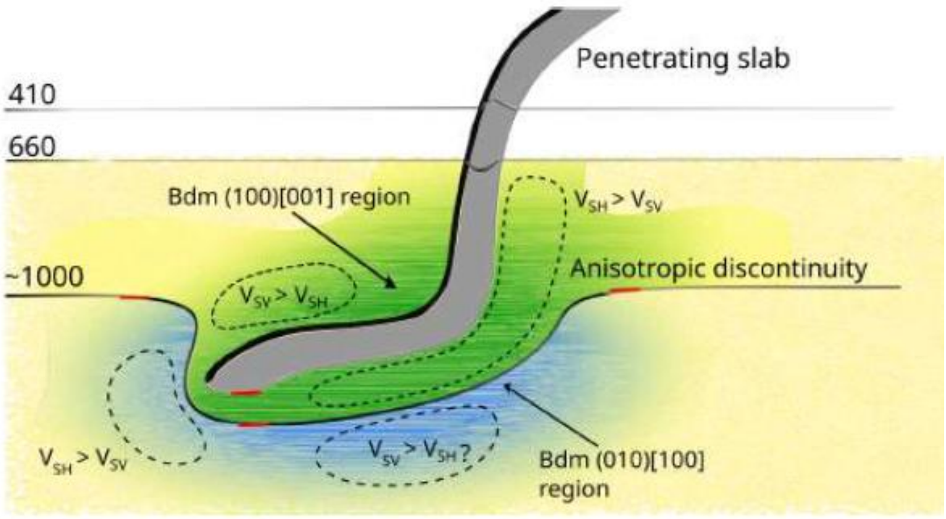
Integration into mantle evolution model

Deformation model



Anisotropy

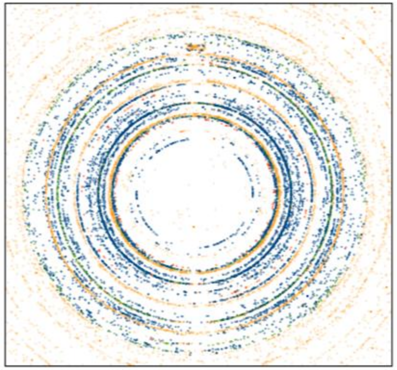


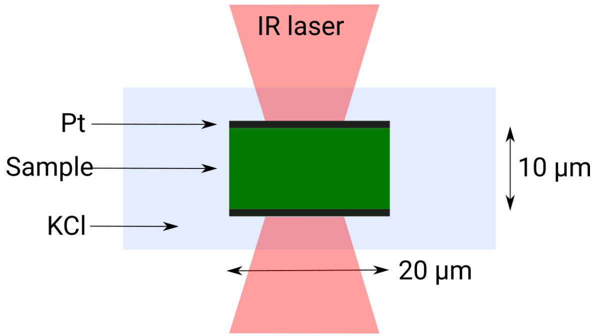


Depth-dependent deformation in the Earth's lower mantle reveals anisotropic discontinuity at 1000 km

Magali et al, *Nat. Geoscience*, in review

On Multigrain Crystallography in the DAC





Novel diffraction technique in high pressure science

- Study bulk samples using X-ray diffraction
- Map individual grain

In the diamond anvil cell

- Map grain orientations / rotations
- Phase transformation / deformation microstructures
- Several 100's of grains, multiple phases
- Grain positions ?
  - We never succeeded, grains and samples are small
- Strains ?
  - I never succeeded, but others did (B. Chandler in Berkeley)
  - Need high resolution detector (i.e. data on Perkin-Elmer not suitable)
- Requires further investigations

Publications on MGC in the DAC

Gay *et al* Depth Dependent Deformation and Anisotropy of Pyrolite in the Earth's Lower Mantle (2024) *Geophysical Research Letters* [doi: 10.1029/2024gl109433]

Ledoux *et al*, Deformation mechanisms, microstructures, and seismic anisotropy of wadsleyite in the Earth's transition zone (2023) *Geochemistry, Geophysics, Geosystems* [doi: 10.1029/2023gc011026]

Ledoux *et al*, In-situ study of microstructures induced by the olivine to wadsleyite transformation at conditions of the 410 km depth discontinuity (2023) *American Mineralogist* [doi: 10.2138/am-2022-8731]

Gay *et al*, Transformation microstructures in pyrolite under stress: Implications for anisotropy in subducting slabs below the 660 km discontinuity (2023) *Earth and Planetary Science Letters* [doi: 10.1016/j.epsl.2023.118015]

Krug *et al*. Textures induced by the coesite-stishovite transition and implications for the visibility of the X-discontinuity (2022) *Geochemistry, Geophysics, Geosystems* [doi: 10.1029/2022GC010544]

Langrand *et al*. Reliability of multigrain indexing for orthorhombic polycrystals above 1 Mbar: application to $MgSiO_3$ post-perovskite (2017) *J. Appl. Crystallogr.* [doi: 10.1107/S1600576716018057]

Rosa *et al*. Evolution of grain sizes and orientations during phase transitions in hydrous (2016) Mg_2SiO_4 *J. Geophys. Res.* [doi: 10.1002/2016JB013360]

Rosa *et al*. In situ monitoring of phase transformation microstructures at Earth's mantle pressure and temperature using multi-grain XRD *J. Appl. Cryst.* (2015) [doi: 10.1107/S1600576715012765]

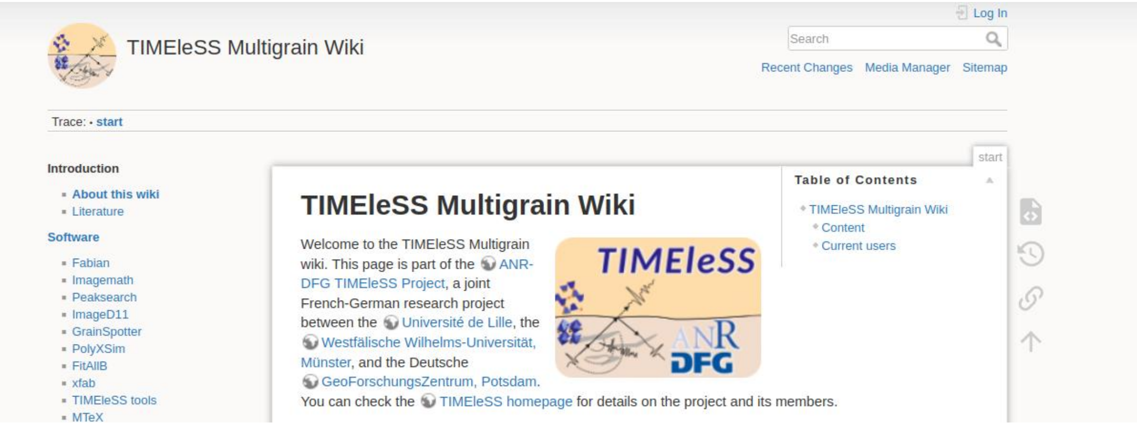
Nisr *et al.* Three-dimensional X-ray diffraction in the diamond anvil cell: application to stishovite (2014) *High Pres. Res*. [doi: 10.1080/08957959.2014.885021]

Nisr *et al*. High resolution three-dimensional X-ray diffraction study of dislocations in grains of $MgGeO_3$ post-perovskite at 90 GPa (2012) *J. Geophys. Res*. [doi: 10.1029/2011JB008401]

Details and manuals

iUCR commission on Diffraction Microstructure Imaging

- Established 2021
- Goals standards, community, training, and discussions
- Please get in touch!



TIMEleSS Multigrain Wiki

- Manual written by the Lille and Münster team
- Dedicated instructions and software tools for DAC experiments
- Free and online, anytime!