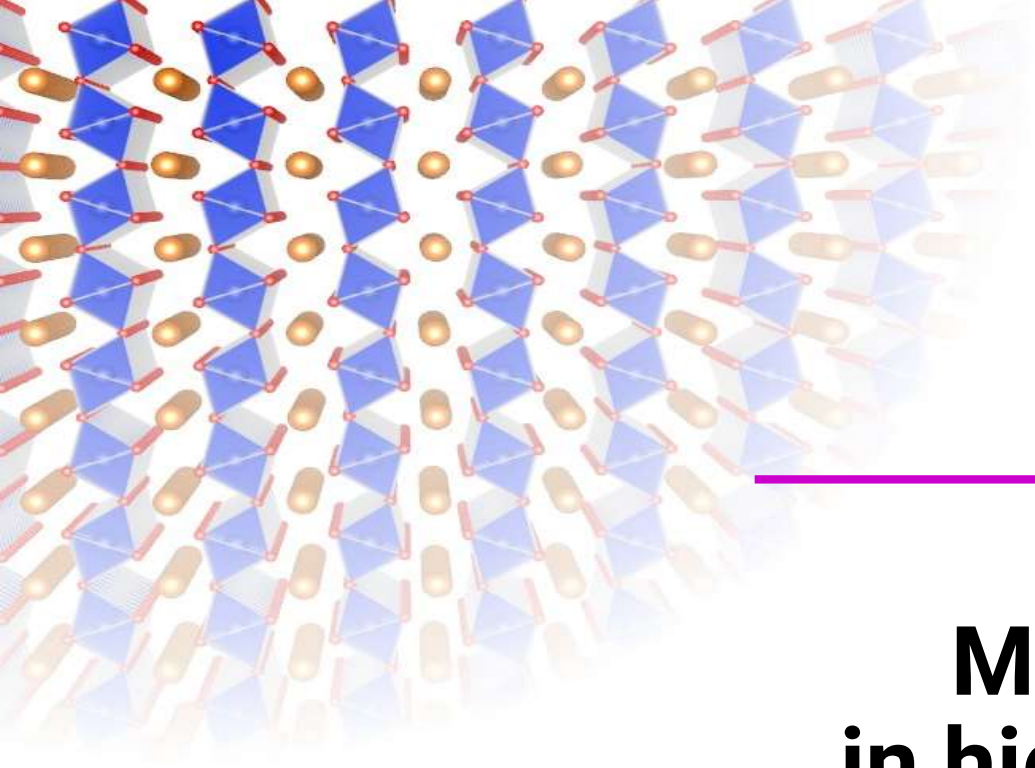


S. Merkel

Université de Lille, France

Materials microstructures in high pressure experiments

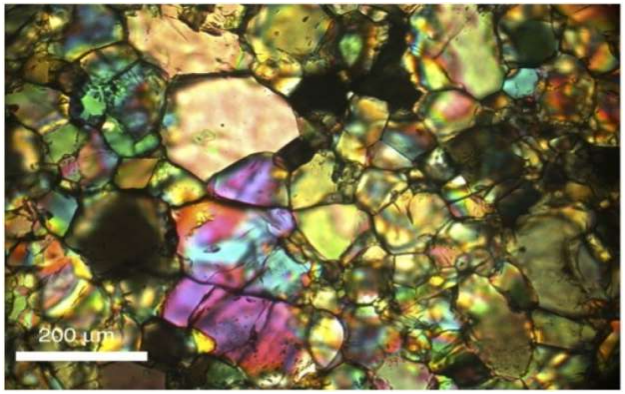
Université de Lille

cnrs

Materials microstructures

Microstructures = arrangement of elements in a material

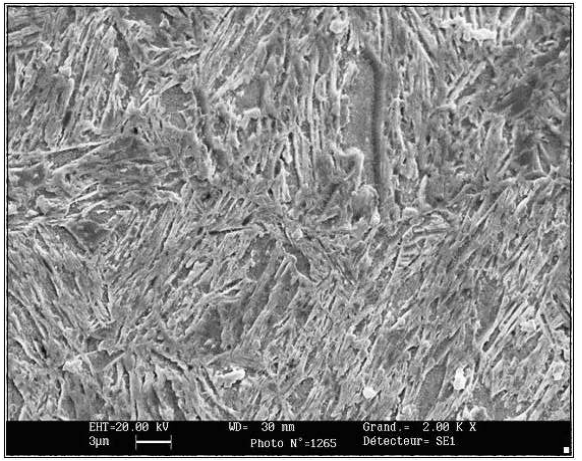
- Distribution of phases
- Grain sizes
- Grain orientations
- Grain boundaries
- Etc



Olivine polycrystal
Image S. Demouchy, U. Montpellier

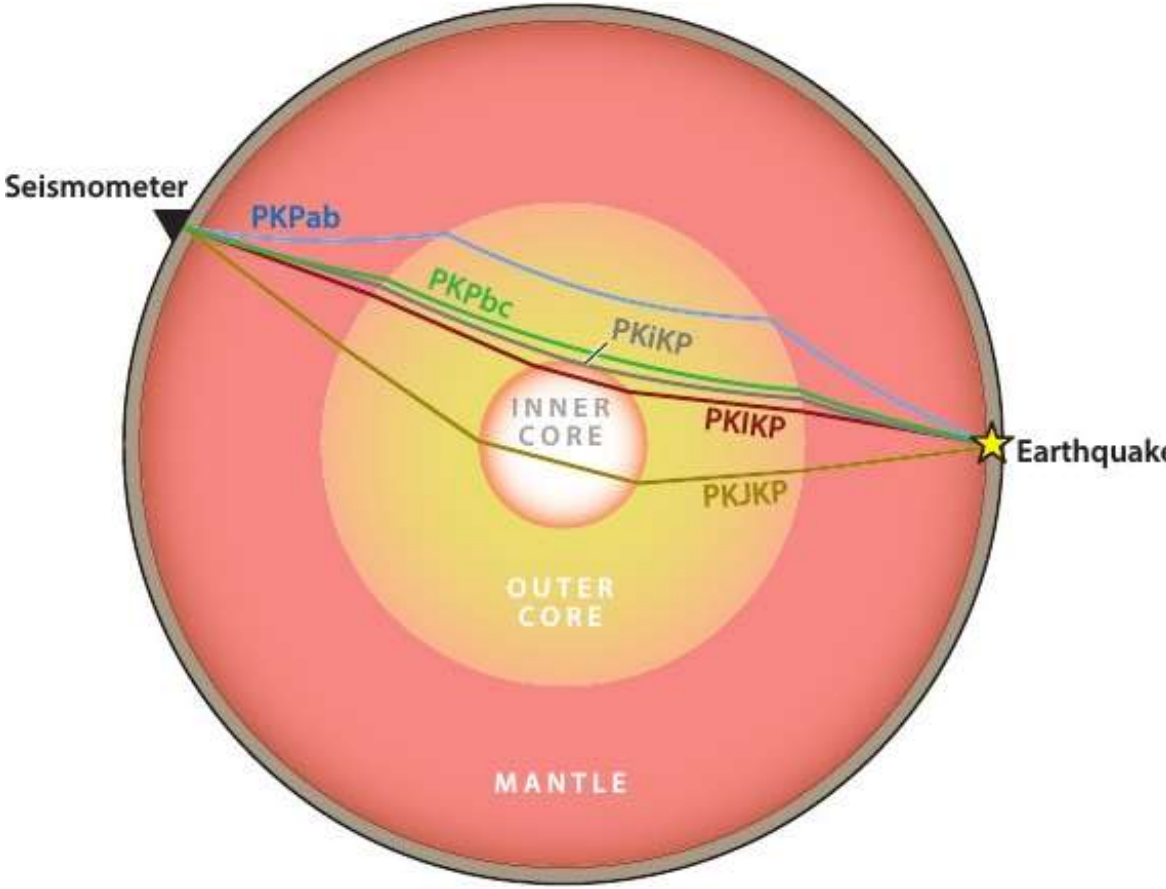
Relevance for material properties

- Rheology
- Mechanical properties
- Strength
- Wave propagation
- Elastic anisotropy



Bainitic steel
Image S. Demouchy, Arcelor Mittal

Sample Application in Geosciences
Inner-Core Anisotropy



Observation in Earth's inner-core

North-South waves faster than those in the equatorial plane

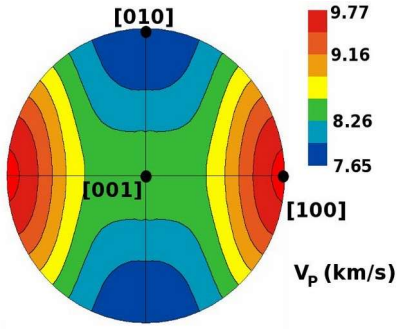
Origin?

Information about inner core processes?
Information about inner core history?

Illustration : Tkalčić, *Rev Geophys*. 2015
Deuss, *Annu. Rev. Earth Planet. Sci*. 2015

At the Microscopic Scale

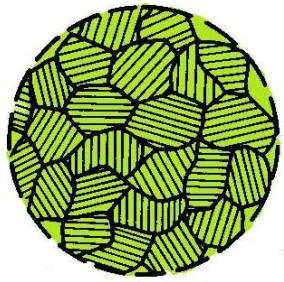
Single crystal

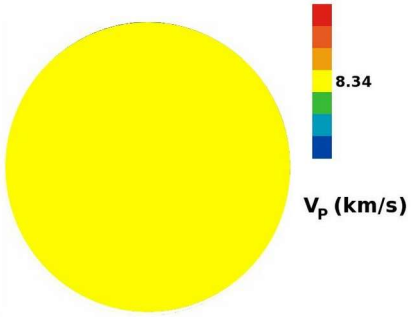


Anisotropy in wave propagation

Depends on elasticity

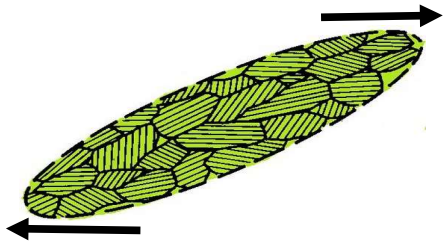
Random polycrystal

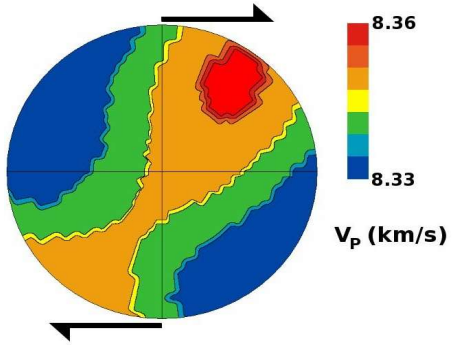




No anisotropy

Textured polycrystal





Anisotropy

Depends on single-crystal **AND** on microstructures

Sample Application in Geosciences
Inner-Core Anisotropy

AGU PUBLICATIONS

Geophysical Research Letters

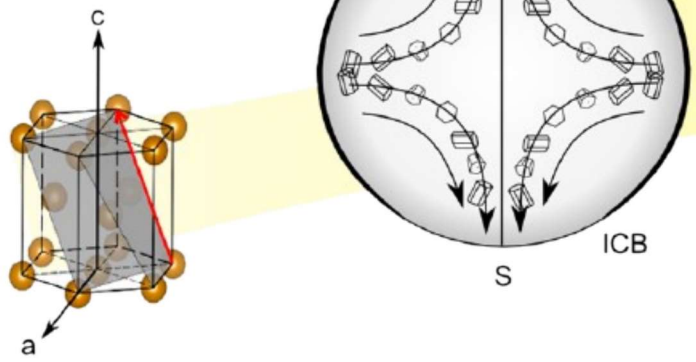
RESEARCH LETTER
10.1002/2015GL067019

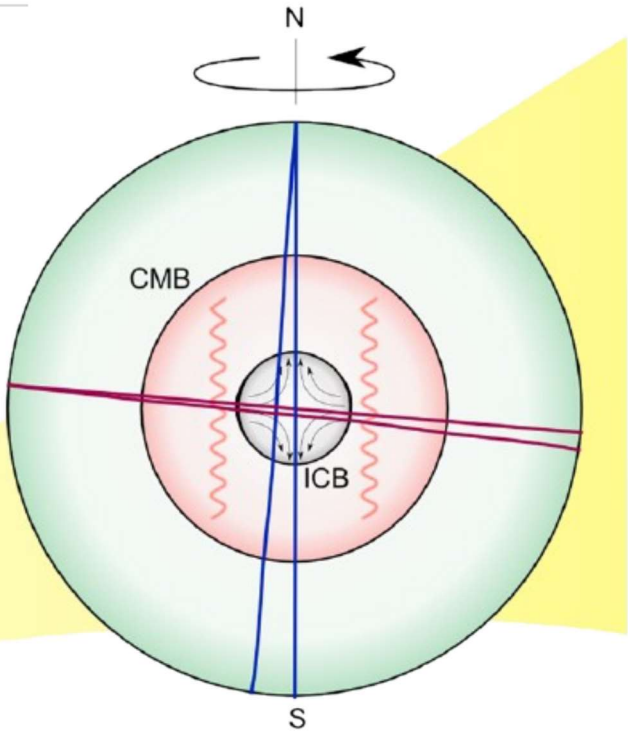
Multiscale model of global inner-core anisotropy induced by hcp alloy plasticity

A. Lincot[1,2], Ph. Cardin[1], R. Deguen[3], and S. Merkel[2,4]

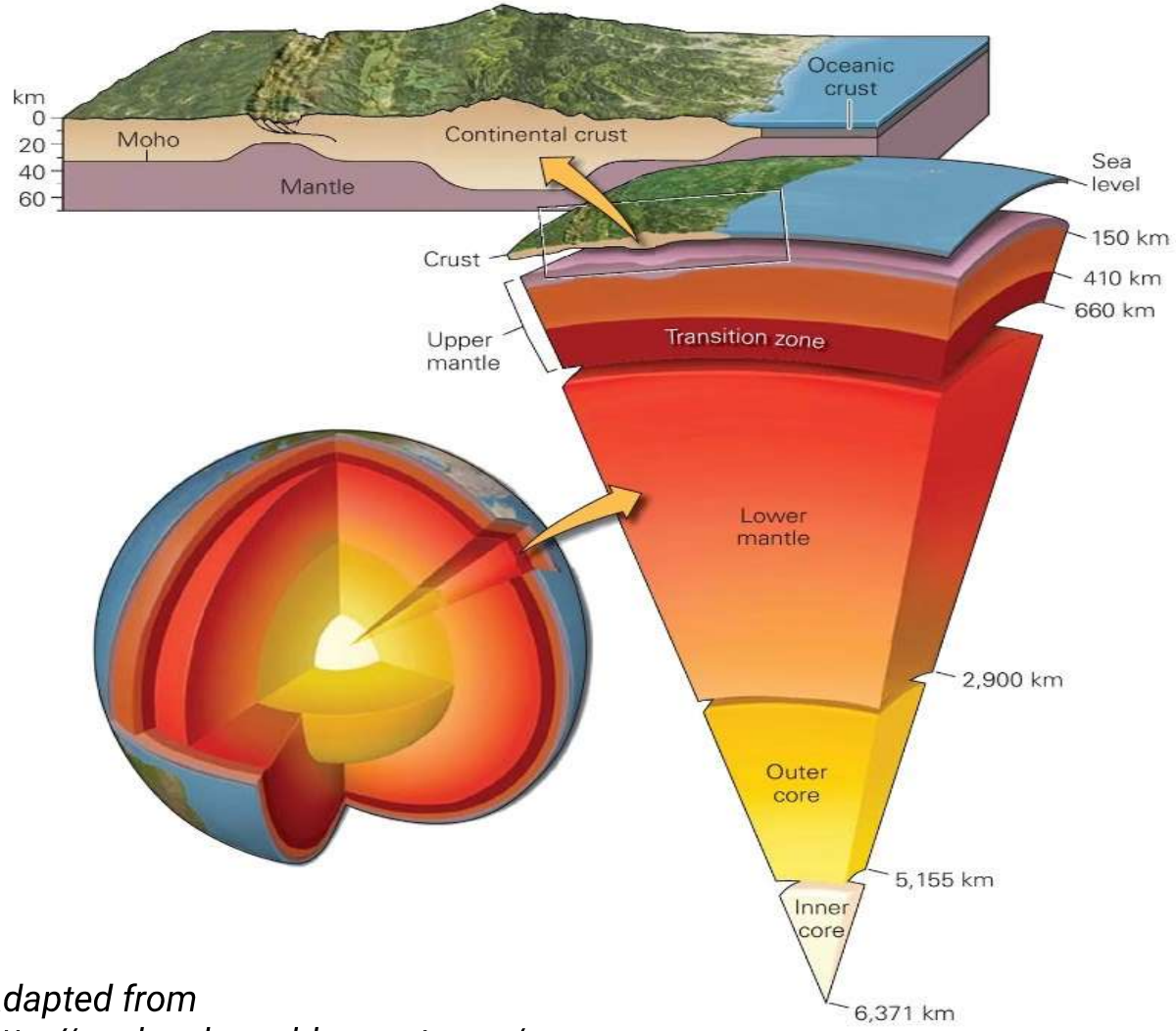
Key questions

- *What is driving microstructures ?*
- *Effect of plastic flow ?*
- *Effect of phase transformations ?*





Earth's Interior



Adapted from
http://geologylearn.blogspot.com/

Bottom of the crust
~ 70 km, 2 GPa, 1600 K

Mantle

Core-mantle boundary
2900 km, 135 GPa, 3000 K

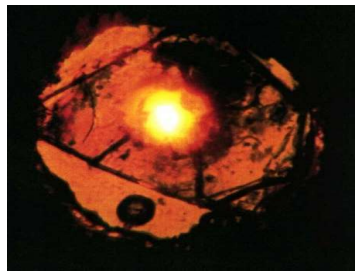
Core

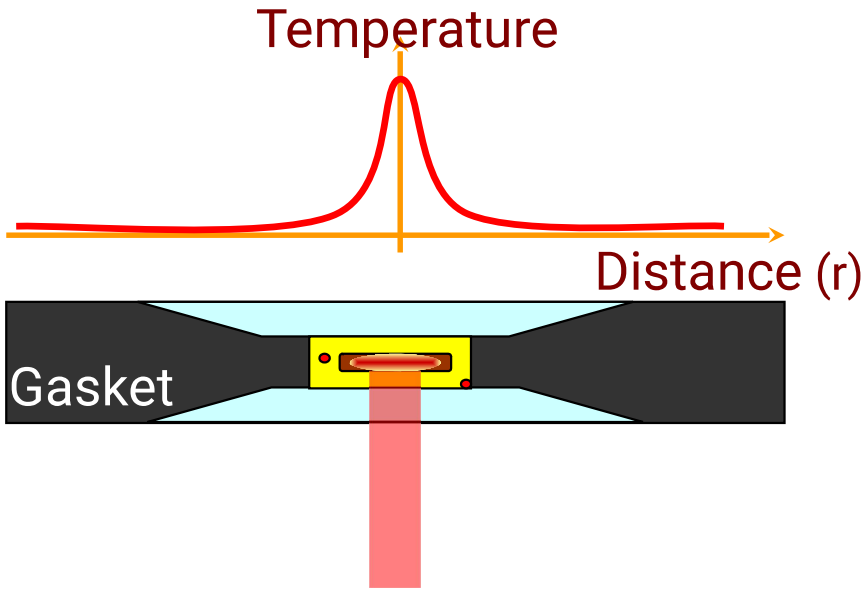
Center of the Earth
6371 km, 365 GPa, 6000 K

High P/T Experiments on Polycrystals
Laser-Heated Diamond Anvil Cells

Diamond anvil cells
S. Merkel, Univ. de Lille

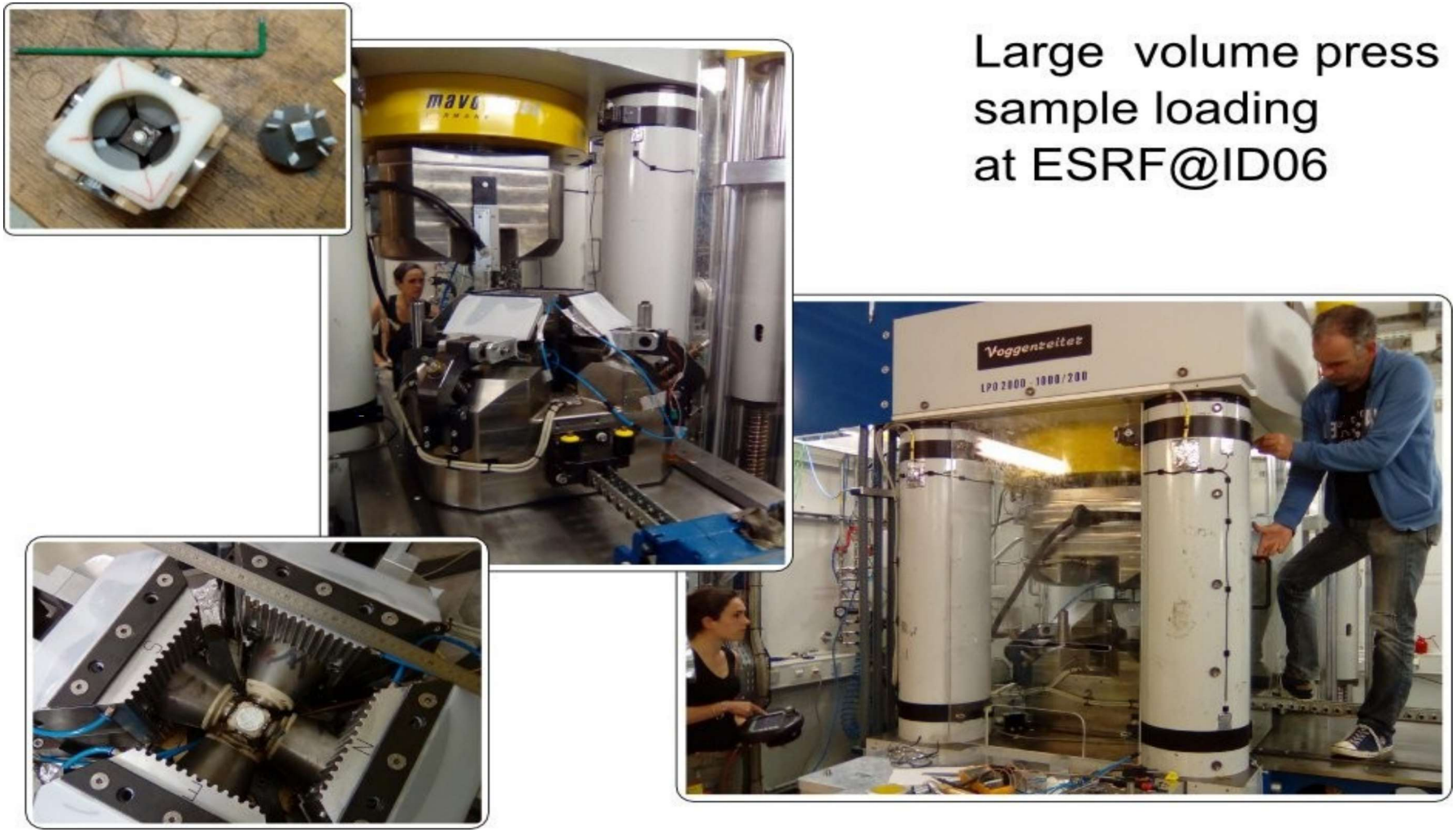
Laser heating in the diamond anvil cell





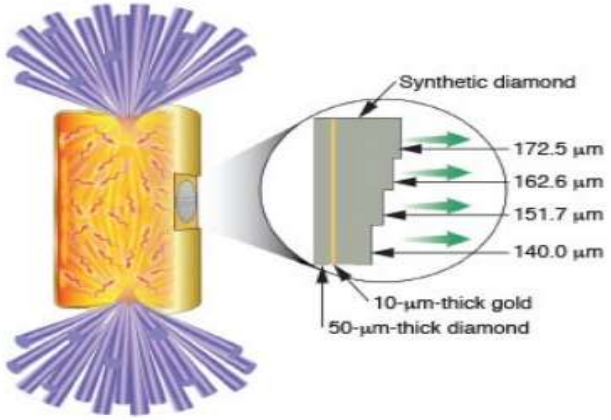
Images: *H. Cardon, J. Pargamin*
ENS Lyon

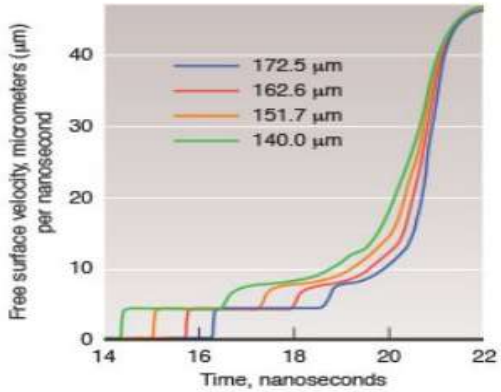
Large volume presses

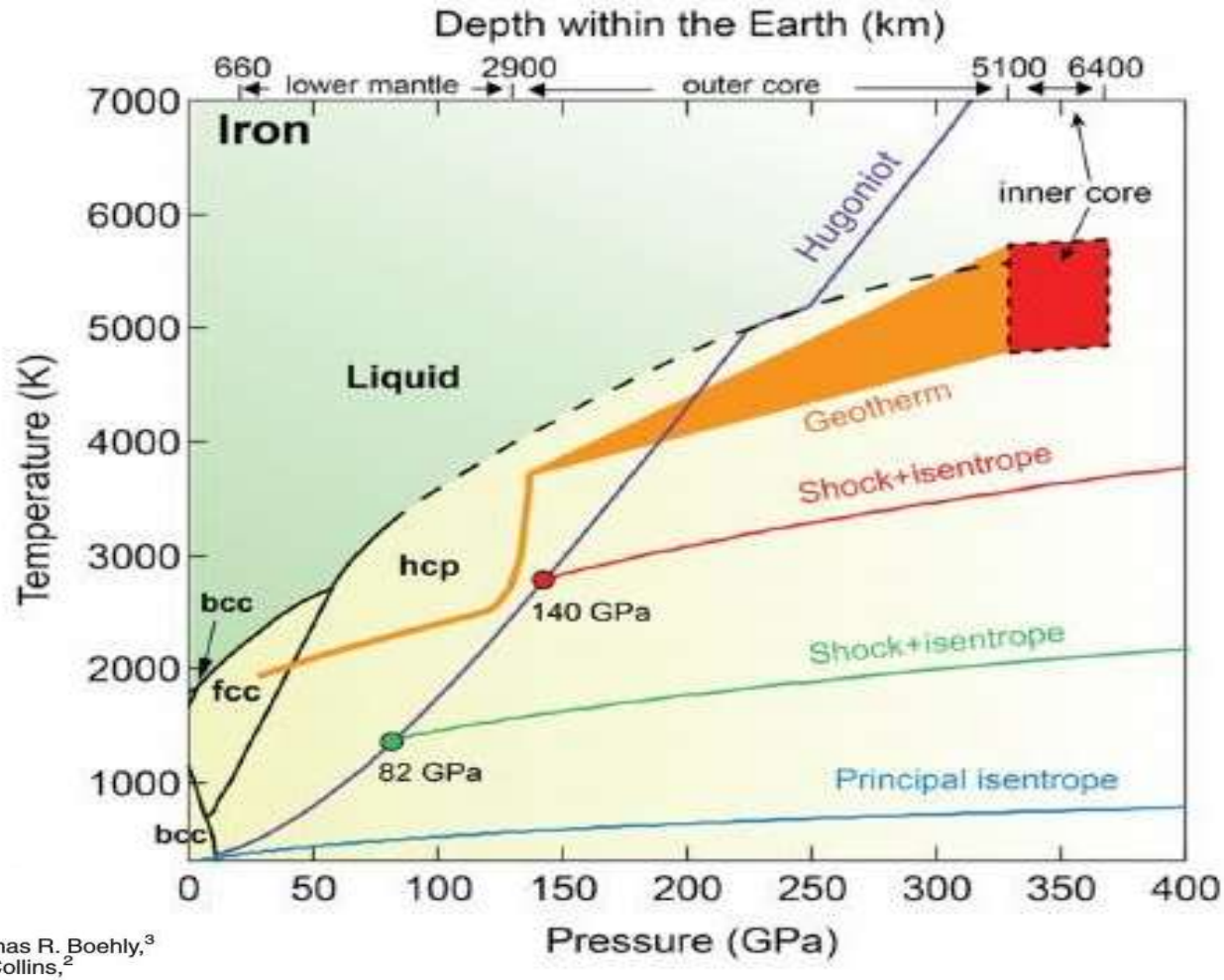


Illustrations: S. Merkel

Laser Driven Compression







JOURNAL OF APPLIED PHYSICS **114**, 023513 (2013)

Ramp compression of iron to 273 GPa

Jue Wang,[1] Raymond F. Smith,[2] Jon H. Eggert,[2] Dave G. Braun,[2] Thomas R. Boehly,[3] J. Reed Patterson,[2] Peter M. Celliers,[2] Raymond Jeanloz,[4] Gilbert W. Collins,[2] and Thomas S. Duffy[1]

Measurements on large scale facilities

ESRF Synchrotron, Grenoble

Soleil synchrotron, outside Paris

Dedicated beamlines on large scale international facilities

Intense, focalized x-ray sources, perfectly suited for in-situ high P/T experiments

Dedicated setups, in place and aligned

Mesurements :

- X-ray diffraction ;
- Radiography, tomography ;
- EXAFS ;
- Etc.

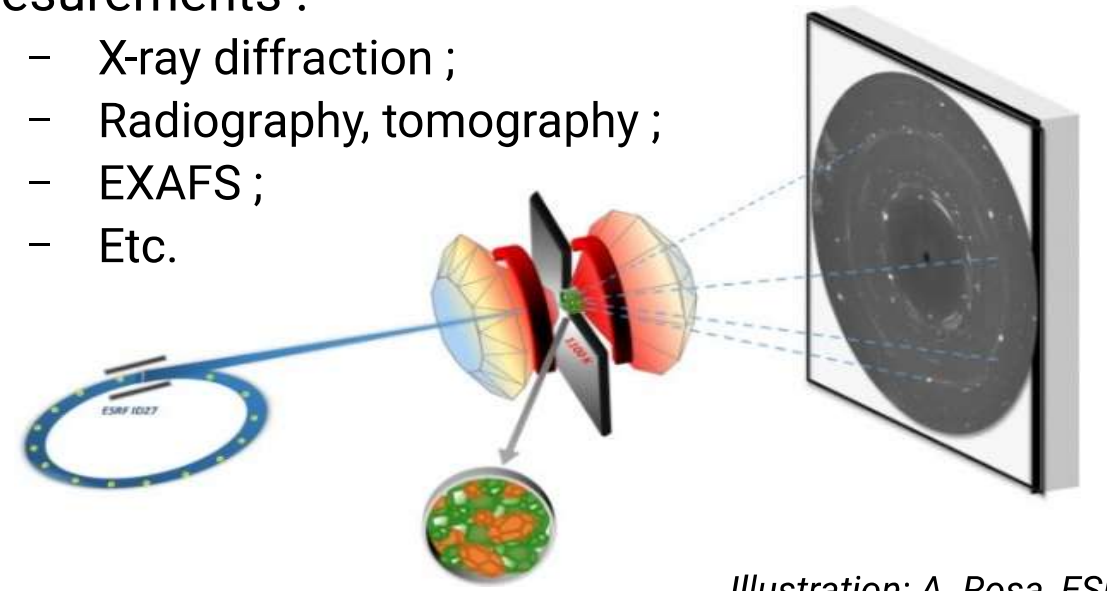
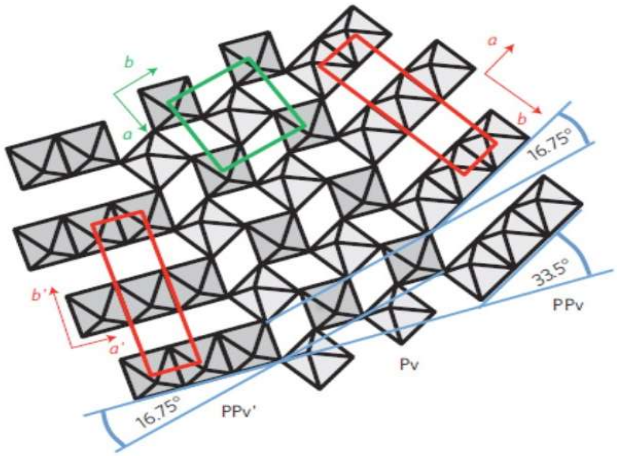


Illustration: A. Rosa, ESRF

Microstructures induced by phase transformation



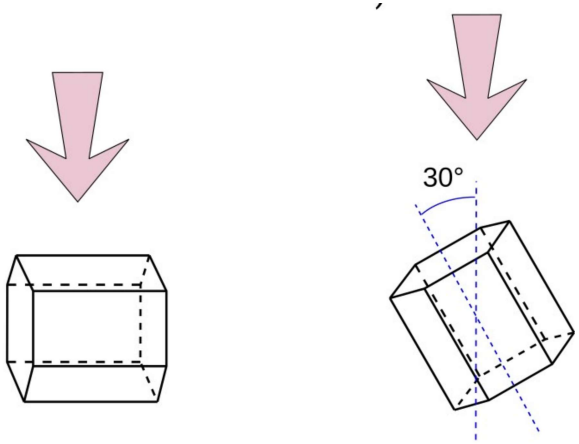
Phase transition in deep Earth oxides

Effects on grain sizes

Effects on grain orientations

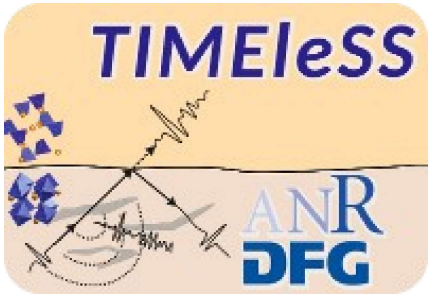
Geophysical implications

Stress and texture in shock compressed Fe



Deformation mechanism in hcp metals (Zn, Ti, Zr, Be, Cd)

Stress and strength in high strain rate deformation experiments



TIMEleSS

French-German grant on transformation microstructures
in Earth mantle minerals

Transformation microstructures

Work of many PhD students at ULille and WWU Münster

Christopher Langrand
Jeff Gay
Estelle Ledoux
Matthias Krug

Perovskite / post-perovskite transition

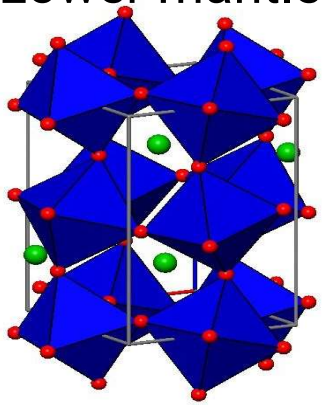
Post-Perovskite Phase Transition in $MgSiO_3$

Murakami et al,
Science, 05/2004

Theoretical and experimental evidence for a post-perovskite phase of $MgSiO_3$ in Earth's D'' layer

Oganov and Ono
Nature, 07/2004

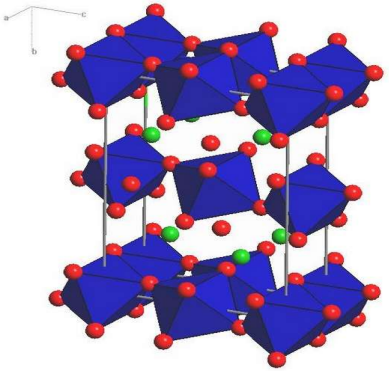
Dominant mineral
Lower mantle



Perovskite structure
$(Mg,Fe)SiO_3$

120-140 GPa

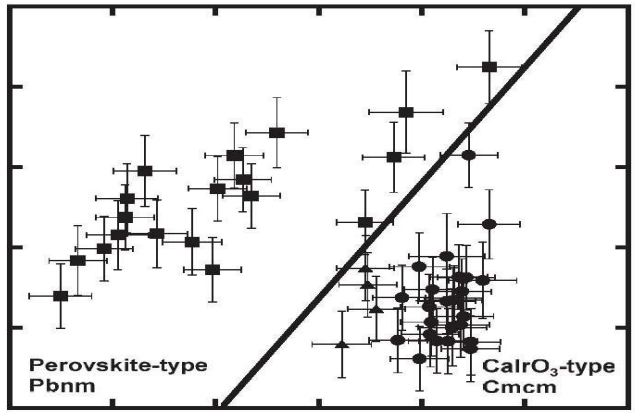
Dominant phase
D'' layer ??



Post-perovskite structure
$(Mg,Fe)SiO_3$

3500 K

1000 K



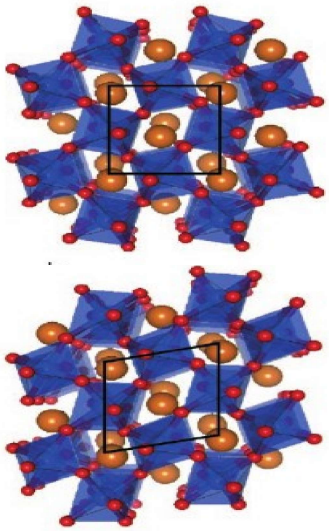
120 GPa

140 GPa

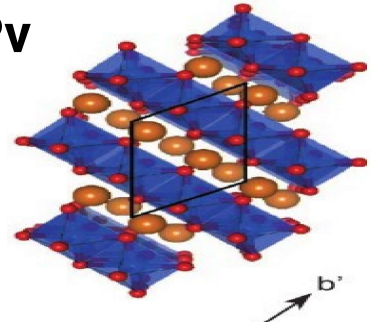
Pv / pPv Coherent Mechanisms

Tsuchiya et al, 2004
simulations $MgSiO_3$

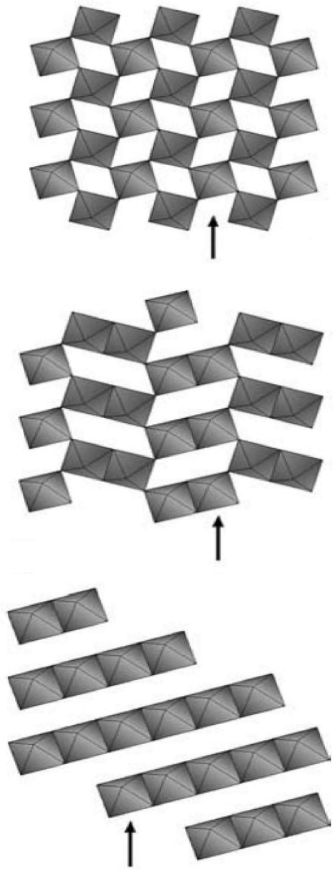
Pv



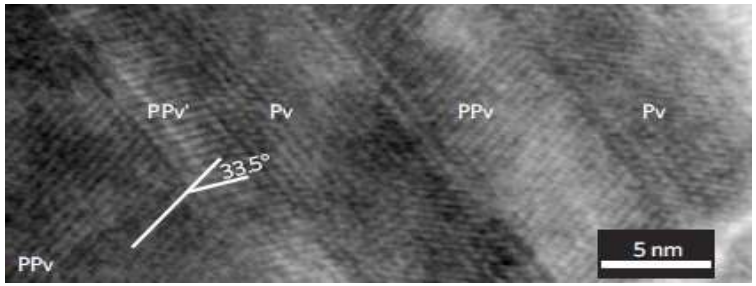
pPv



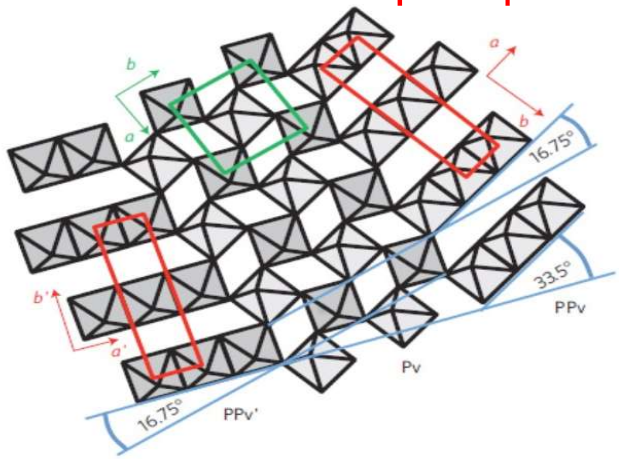
Oganov et al, 2005
simulations $MgSiO_3$



Dobson et al, 2013
TEM + experiments $NaNiF_3$



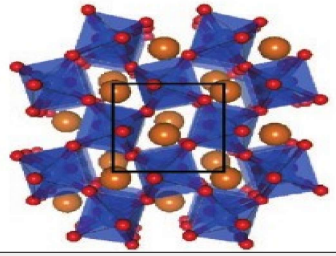
Green: perovskite
Red: post-perovskite



Pv / pPv Coherent Mechanisms

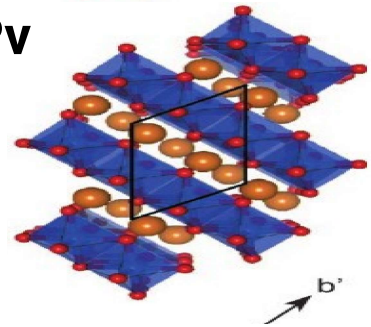
Tsuchiya et al, 2004
simulations $MgSiO_3$

Pv

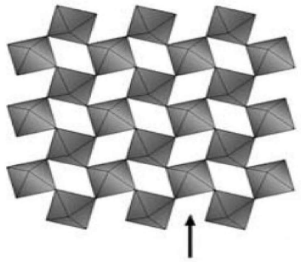


~45° rotation around c-axis

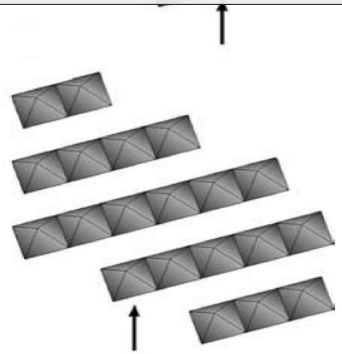
pPv



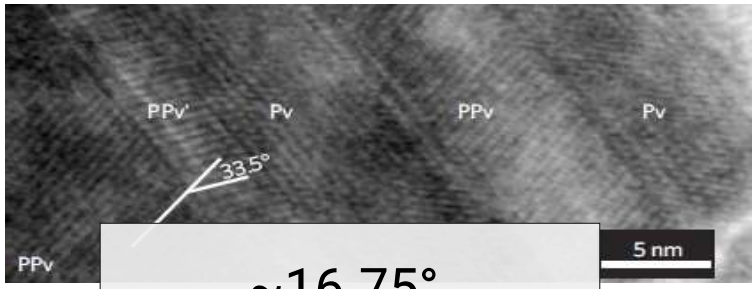
Oganov et al, 2005
simulations $MgSiO_3$



random rotation around c-axis

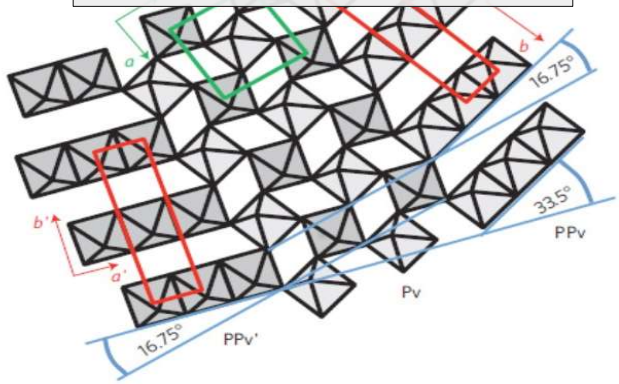


Dobson et al, 2013
TEM + experiments $NaNiF_3$



~16.75° rotation around c-axis

Green: perovskite
Red: post-perovskite



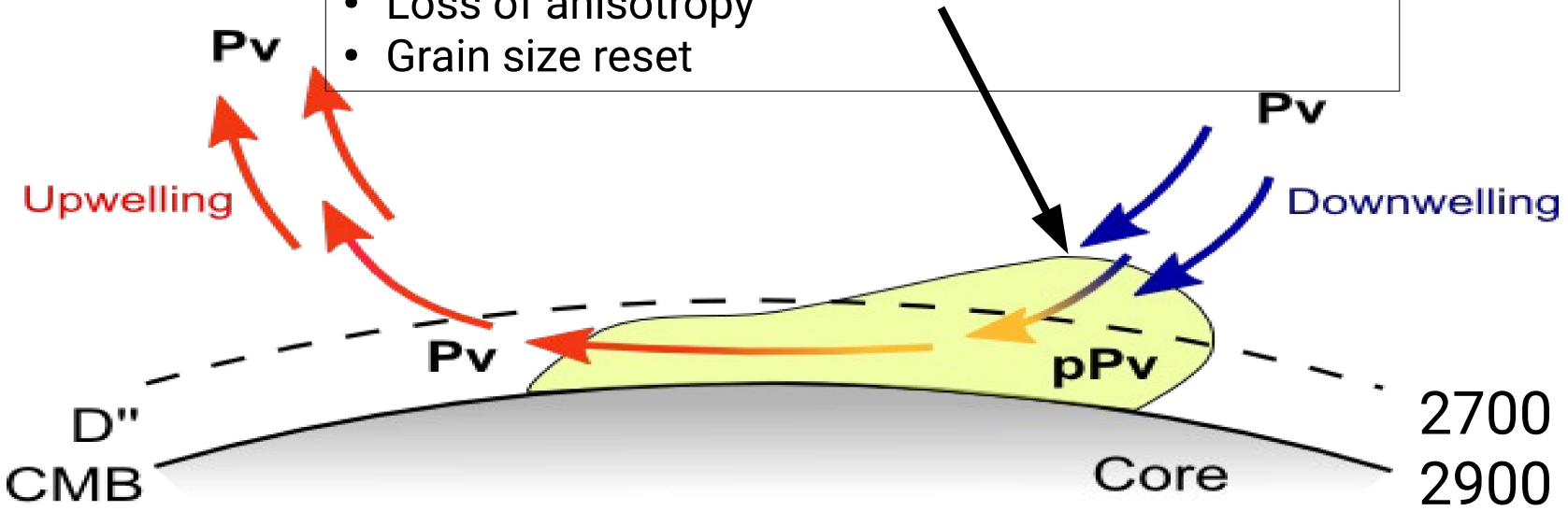
Why do we care?

Perovskite to post-perovskite transformation

If martensitic (with orientation relationships):
- Texture memory, deformation history preserved
- Anisotropy
- Grain size memory

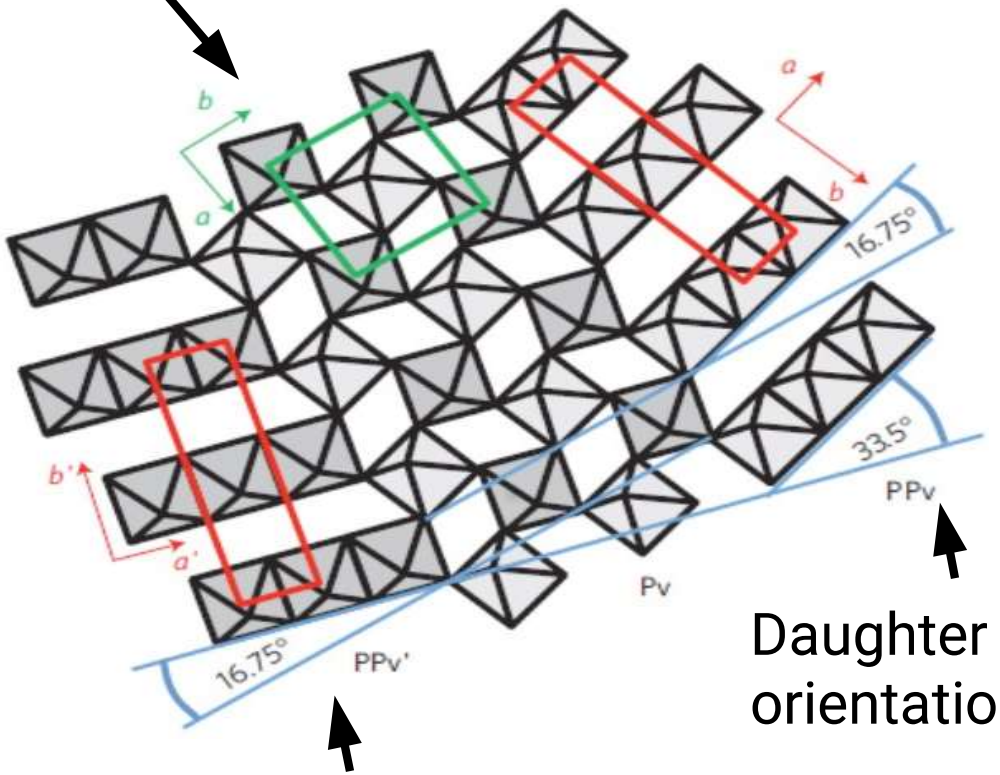
If no orientation relationships
- Loss of memory, Random texture
- Loss of anisotropy
- Grain size reset

Earth's mantle



Example of mechanism

Parent Pv orientation



Daughter pPv orientation n.2

Daughter pPv orientation n.1

Pv to pPv transformation according to Dobson et al, 2013

Parent Pv orientation

→ 2 daughter pPv orientations

Effect of grain size

- Equal distribution between children?
- Farmer's style inheritance, one child takes it all?
- External factors?

Multigrain Crystallography

Diamond anvil cell sitting on a rotation stage

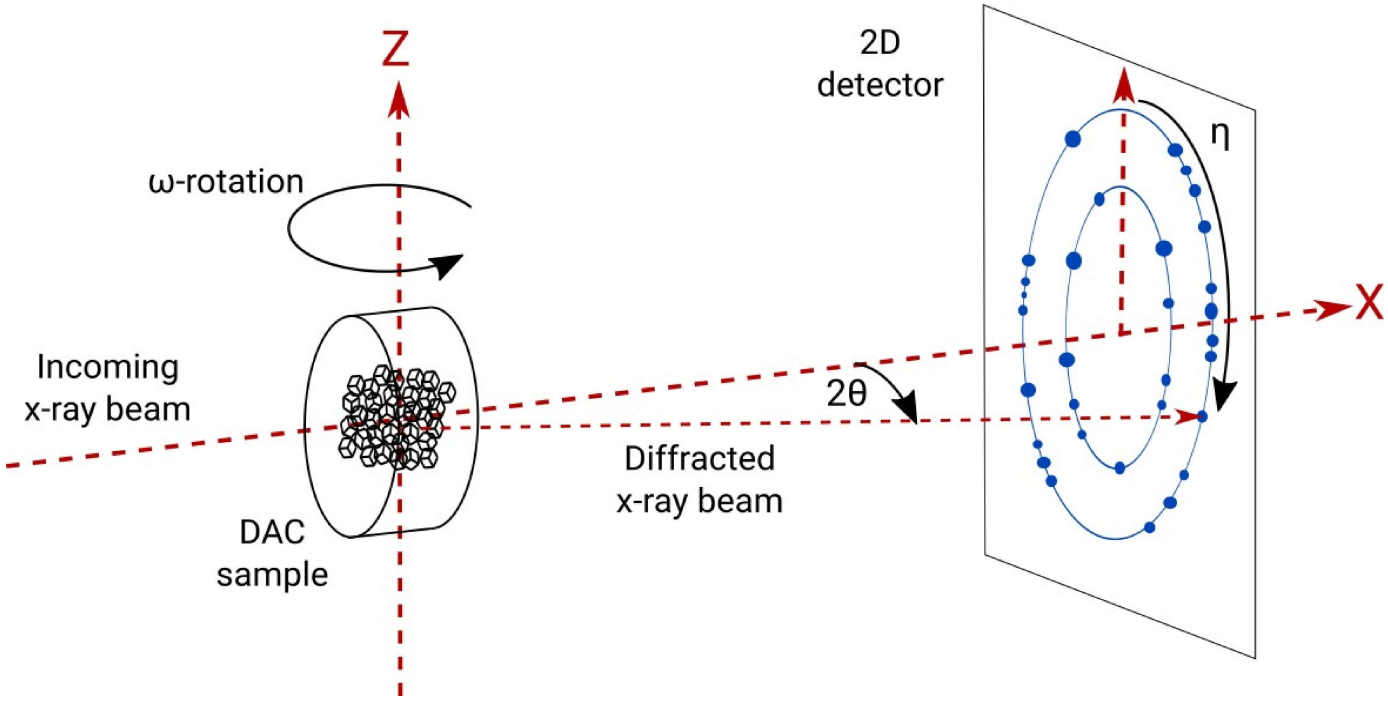
Collect images at every rotation step

Typical numbers

- Rotation range: $\Delta\omega = 45°$
- Rotation step: $\delta\omega = 0.5°$
- 90 diffraction images per P/T point

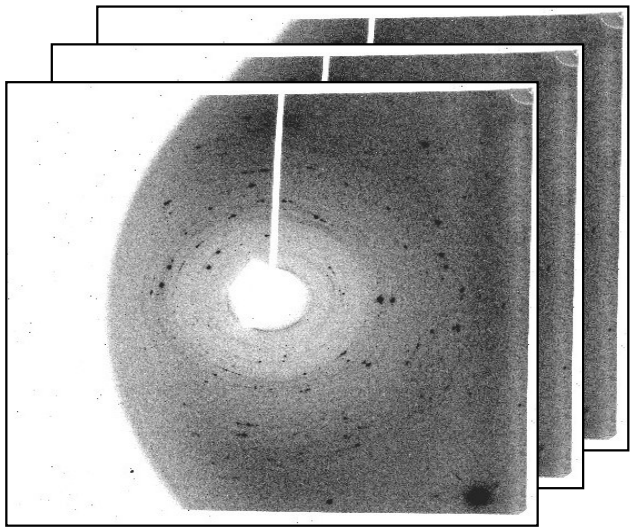
At each P/T point

- Extract a database of diffraction spots vs. η, 2θ, and ω

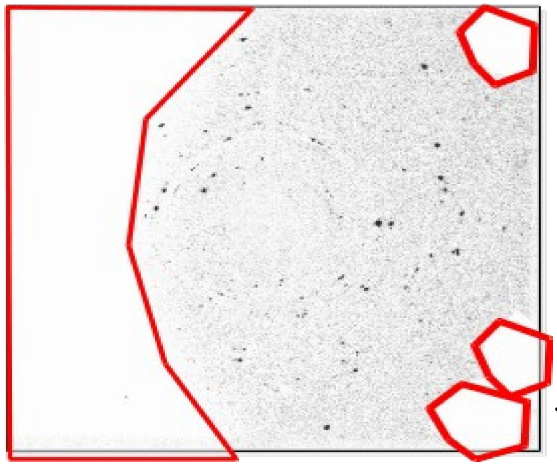


Nisr *et al*, *High Pres. Res*. 2014

Image Processing

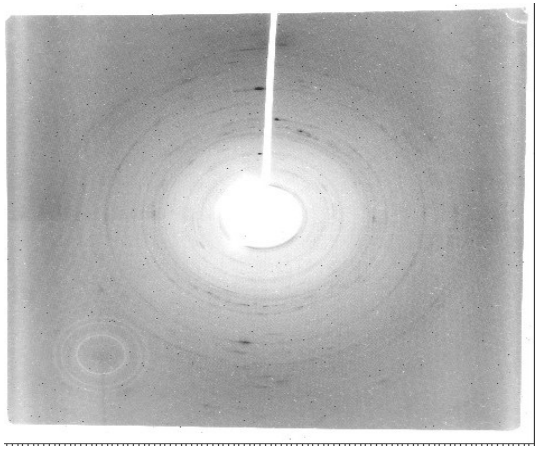


ω-images

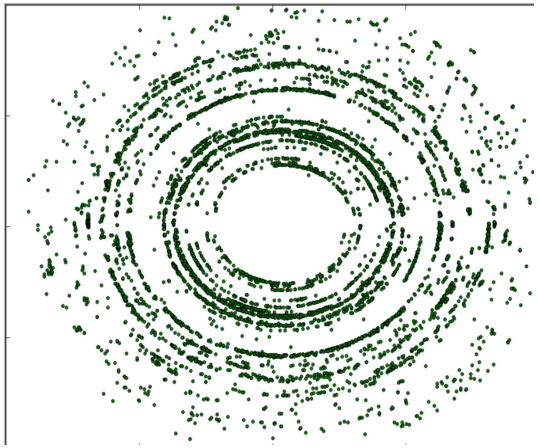


Median subtraction and data cleaning for each image

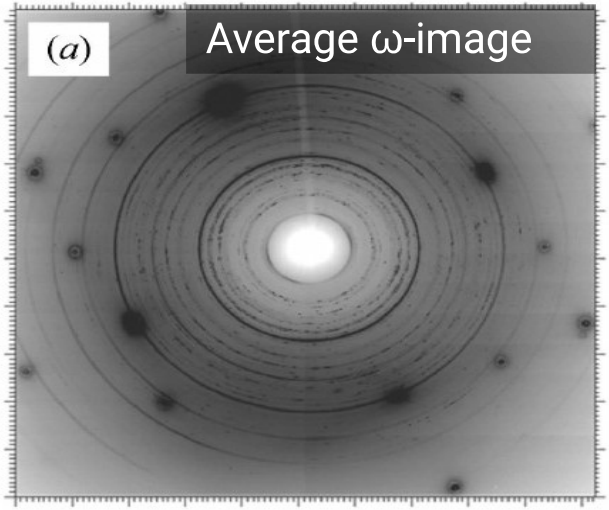
Calculation of median and average images

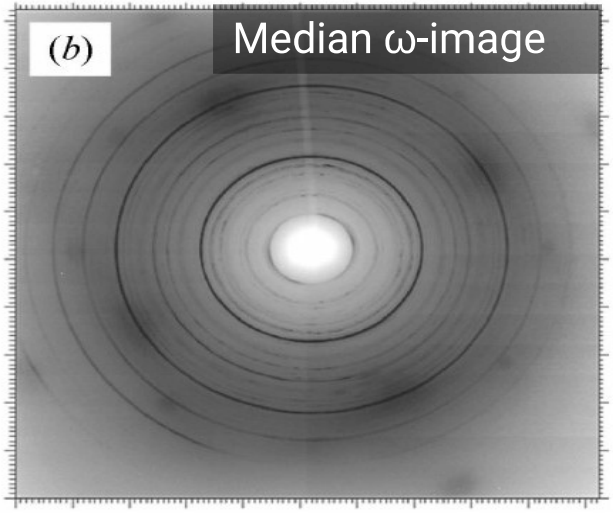


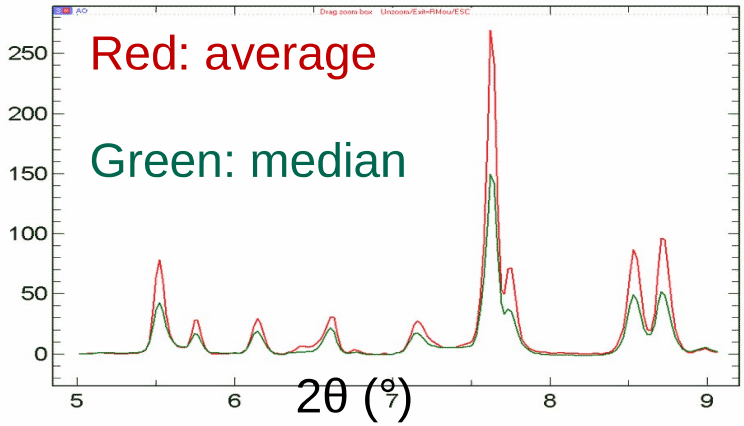
Peak searching



Average Sample Properties







Rosa *et al*, *J. Appl. Cryst.* 2015

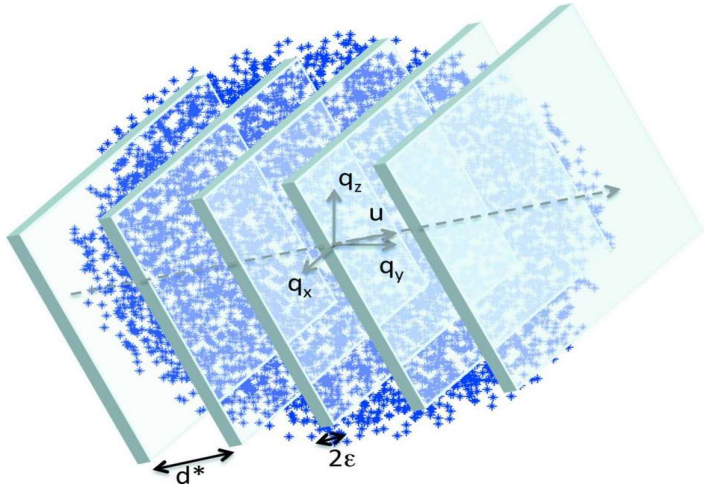
Average image
- Background + fine sample matrix + larger sample grains

Median image
- Same background
- Fine sample matrix only
- No grains

Scaling factor
- fine matrix vs. large grains for each phase

Typical numbers

- ~10^4 spots per P/T point
- Random walk through orientation space to identify grains
- ~ 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

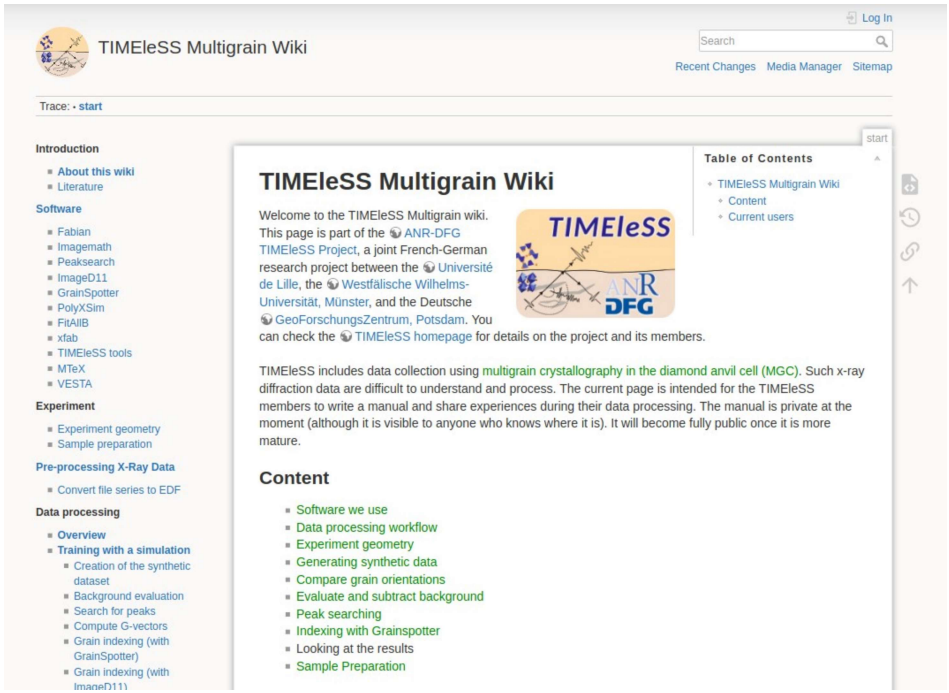
Average sample

- 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



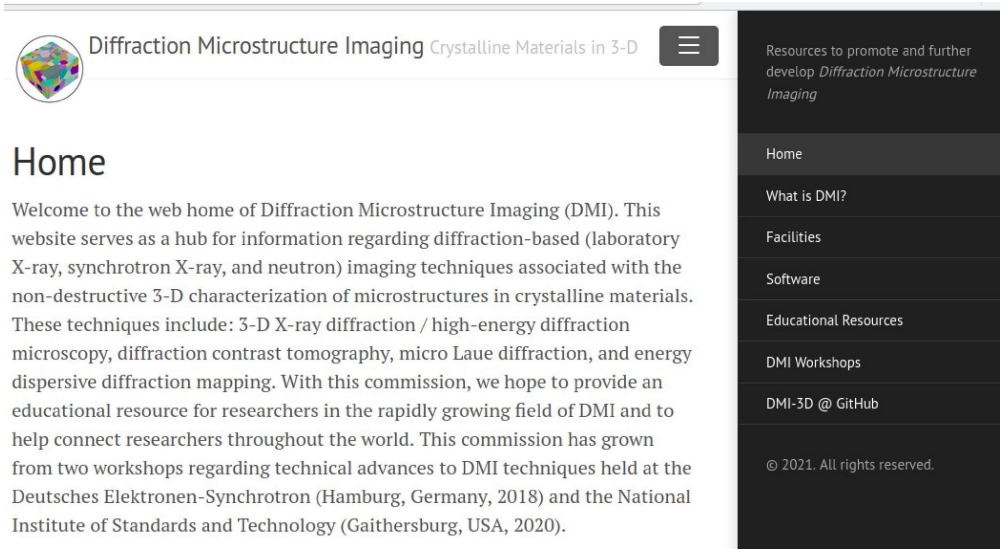
TIMEleSS Multigrain Wiki

Run by our group @Ulille and collaborators in Münster

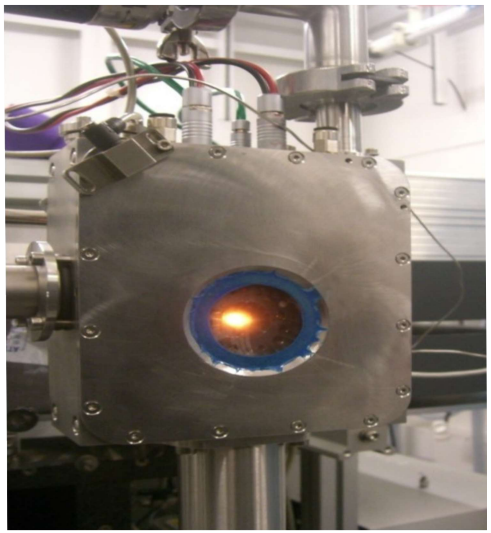
http://multigrain.texture.rocks

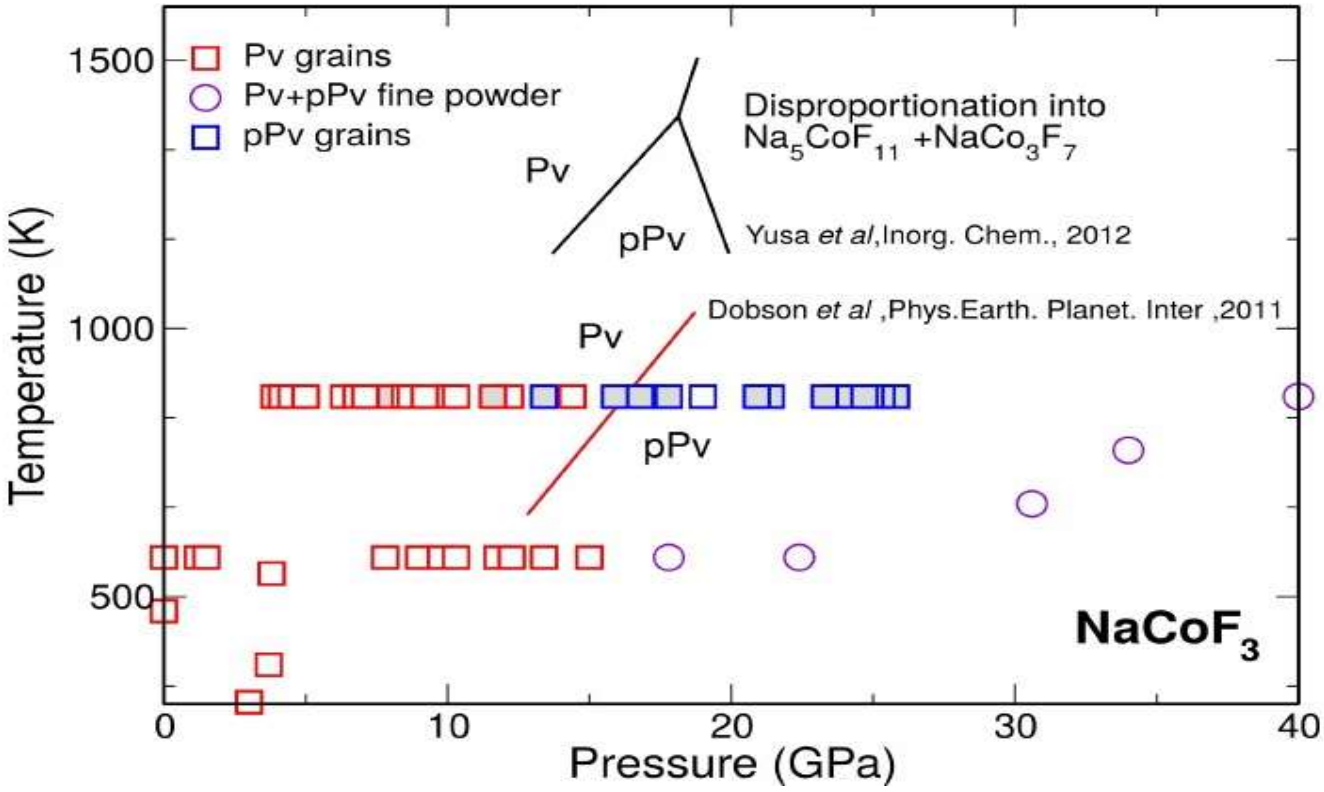
Diffraction Microstructure Imaging Project for iUCR working group

http://dmi-3d.github.io



Pv – pPv transformation in $NaCoF_3$





Sample

- $NaCoF_3$
- Pv to pPv transition at ~15 GPa

Confinement

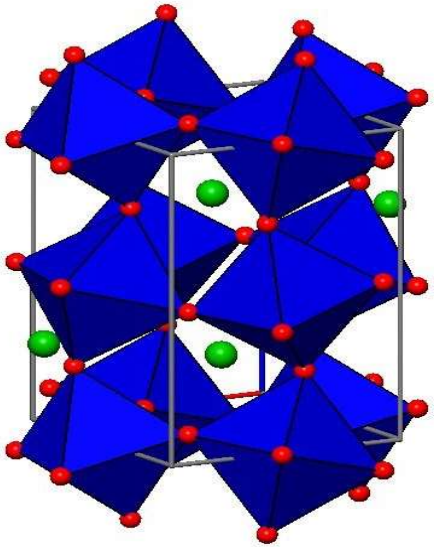
- Resistive heating DAC
- Rhenium gasket
- Run duration: ~10s of hours for each T

ID 27

$NaCoF_3$ Perovskite

Before Transformation

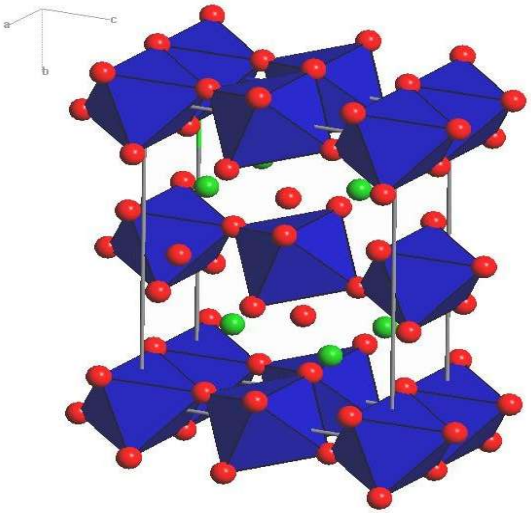
P = 14.4 GPa – T = 900 K



$NaCoF_3$ post-Perovskite

After Transformation

P = 17.5 GPa – T = 900 K

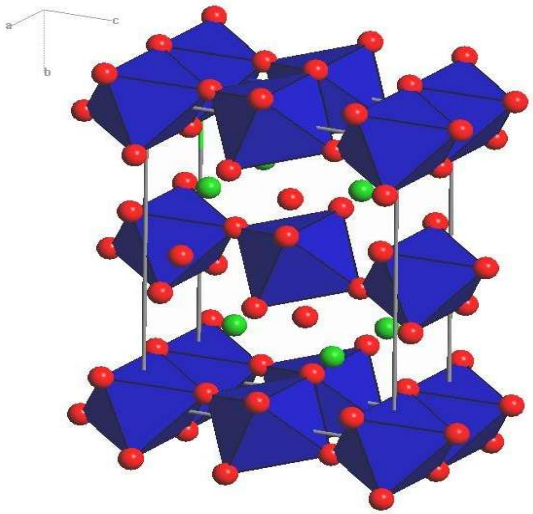


Langrand *et al*, in prep.

$NaCoF_3$ post-perovskite

Before Transformation

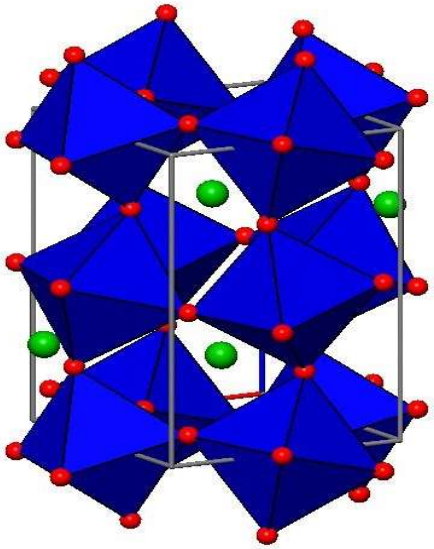
P = 17.8 GPa – T = 900 K



$NaCoF_3$ Perovskite

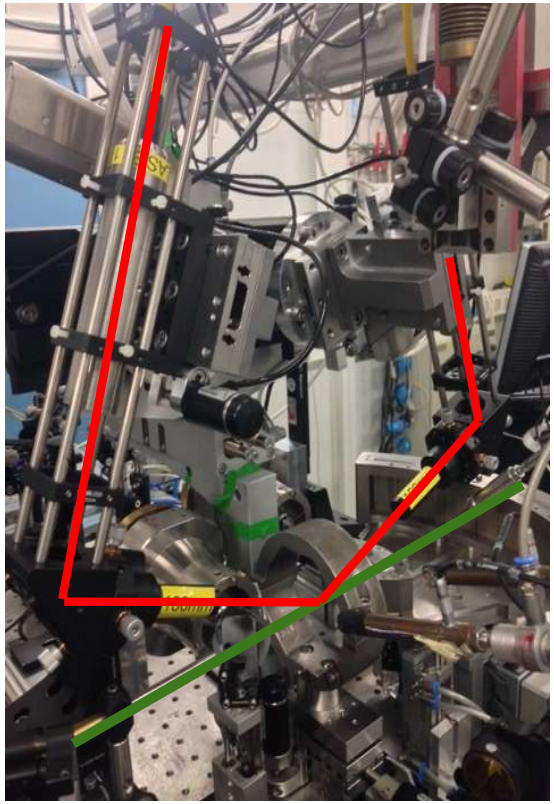
After Transformation

P = 13.4 GPa – T = 900 K

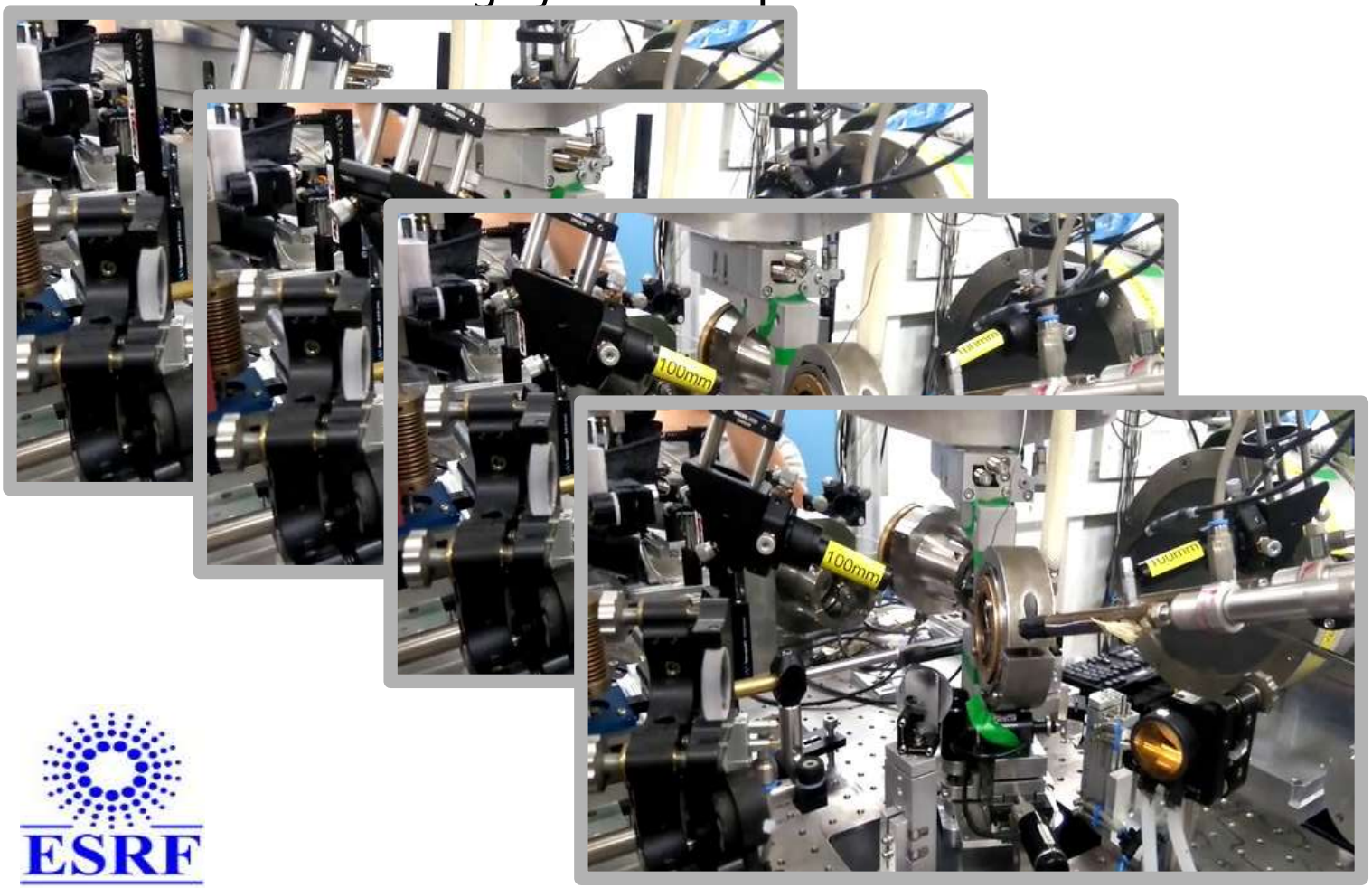


Langrand *et al*, in prep.

Outlook: Laser-Heated Diamond Anvil Cell



Laser-heating system coupled with ω-rotation



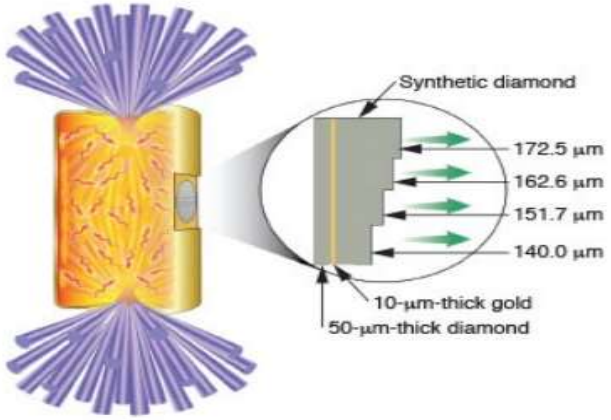
ID 27

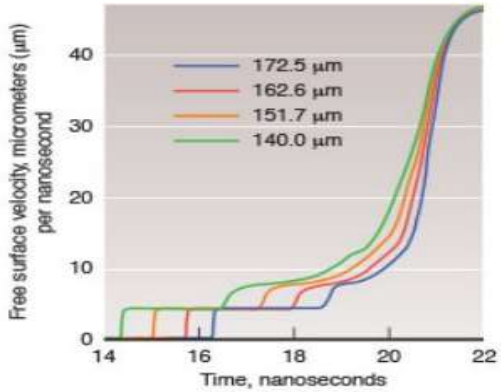
ESRF

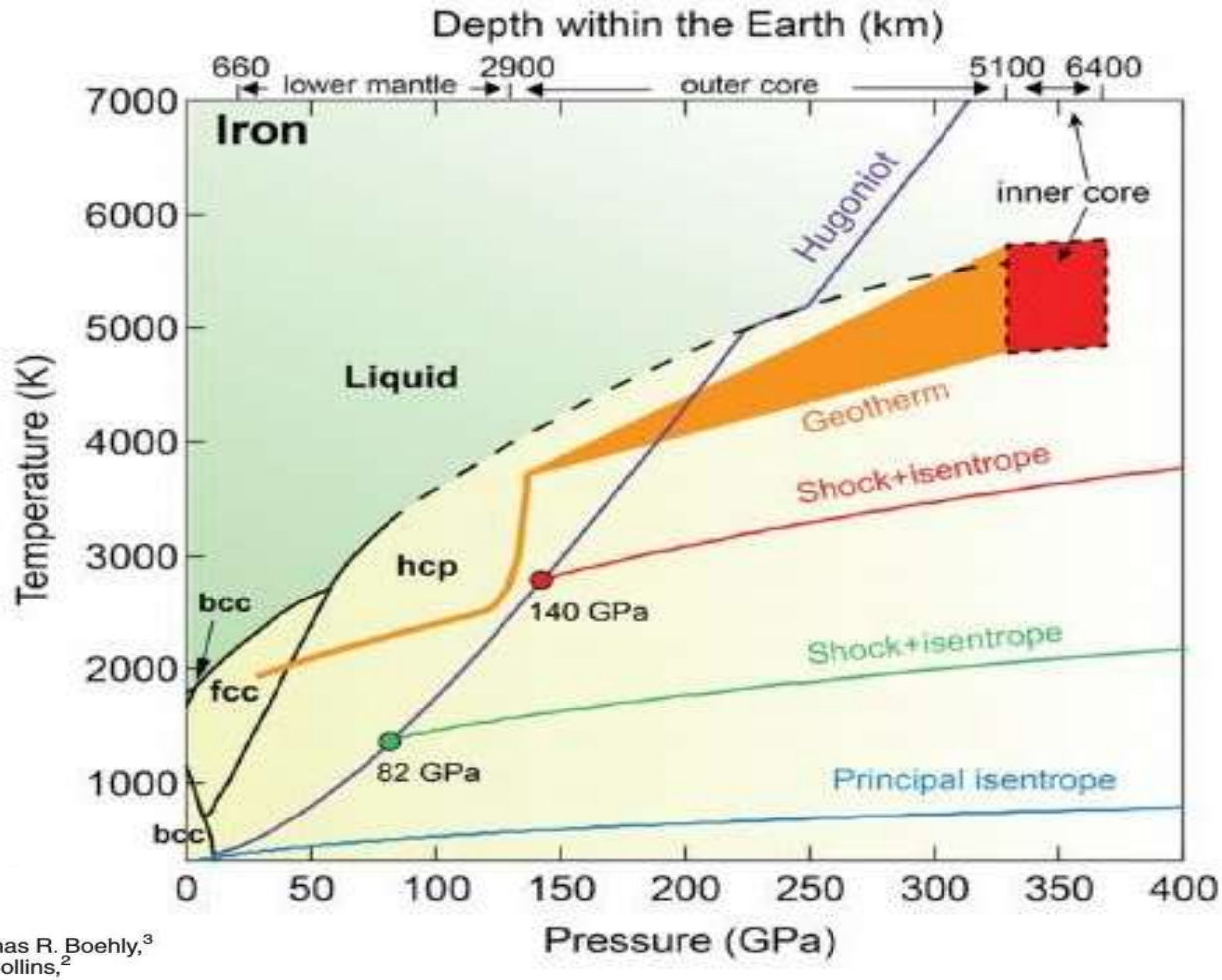
Stress and texture in shock-compressed hcp-Fe

Collaboration
A. Gleason, Stanford / SLAC
S. Hok, W. Mao, D. Rittaman, Stanford
C. Bolme, K. Ramos, B. Morrow, Los Alamos
E. Galtier, B. Nagler, E. Granados, H.J. Lee, SLAC
A. Hashim, UC Berkeley

Laser Compression







JOURNAL OF APPLIED PHYSICS **114**, 023513 (2013)

Ramp compression of iron to 273 GPa

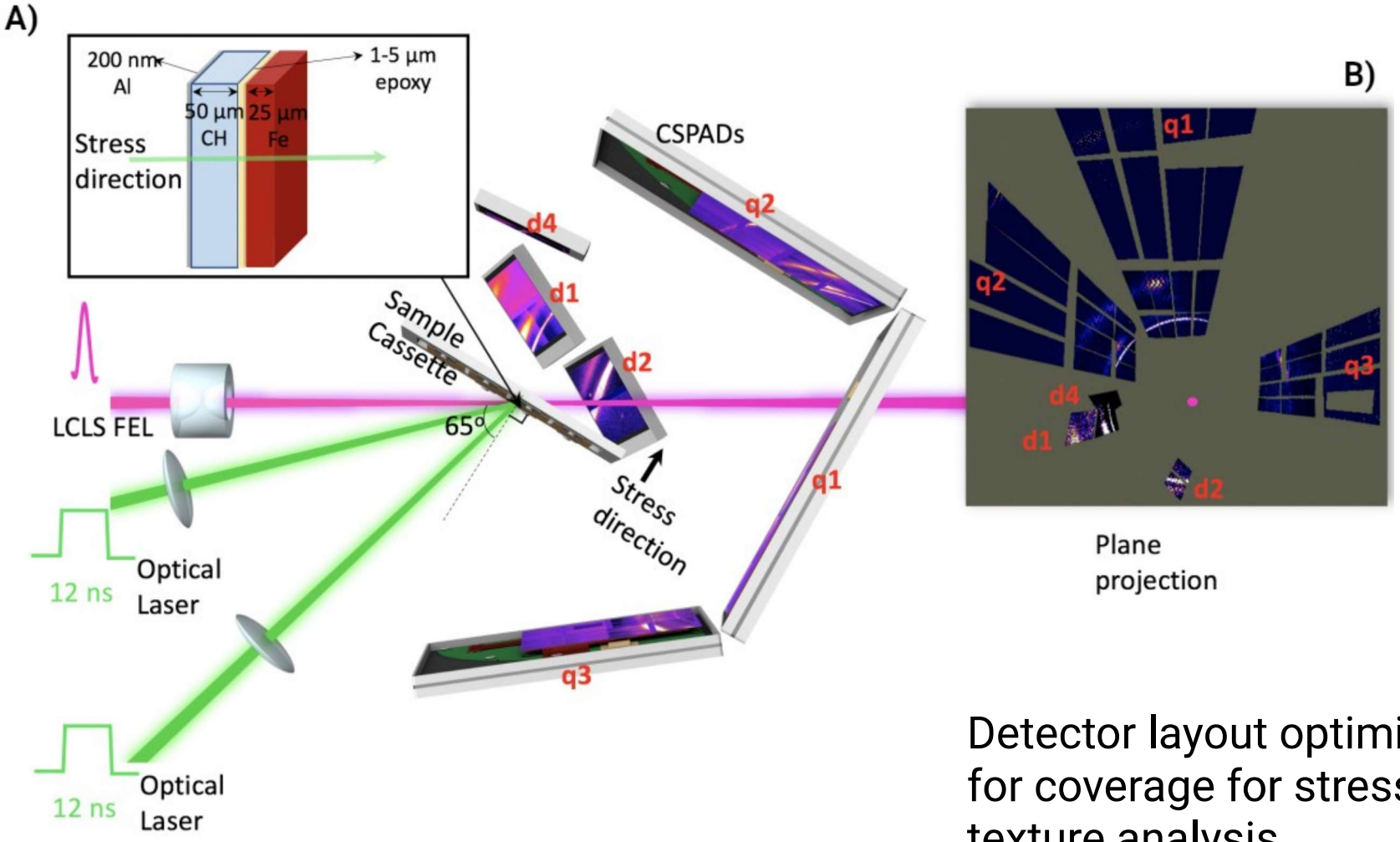
Jue Wang,[1] Raymond F. Smith,[2] Jon H. Eggert,[2] Dave G. Braun,[2] Thomas R. Boehly,[3] J. Reed Patterson,[2] Peter M. Celliers,[2] Raymond Jeanloz,[4] Gilbert W. Collins,[2] and Thomas S. Duffy[1]

Laser compression at SLAC

MEC beamline at
LCLS/SLAC, Stanford

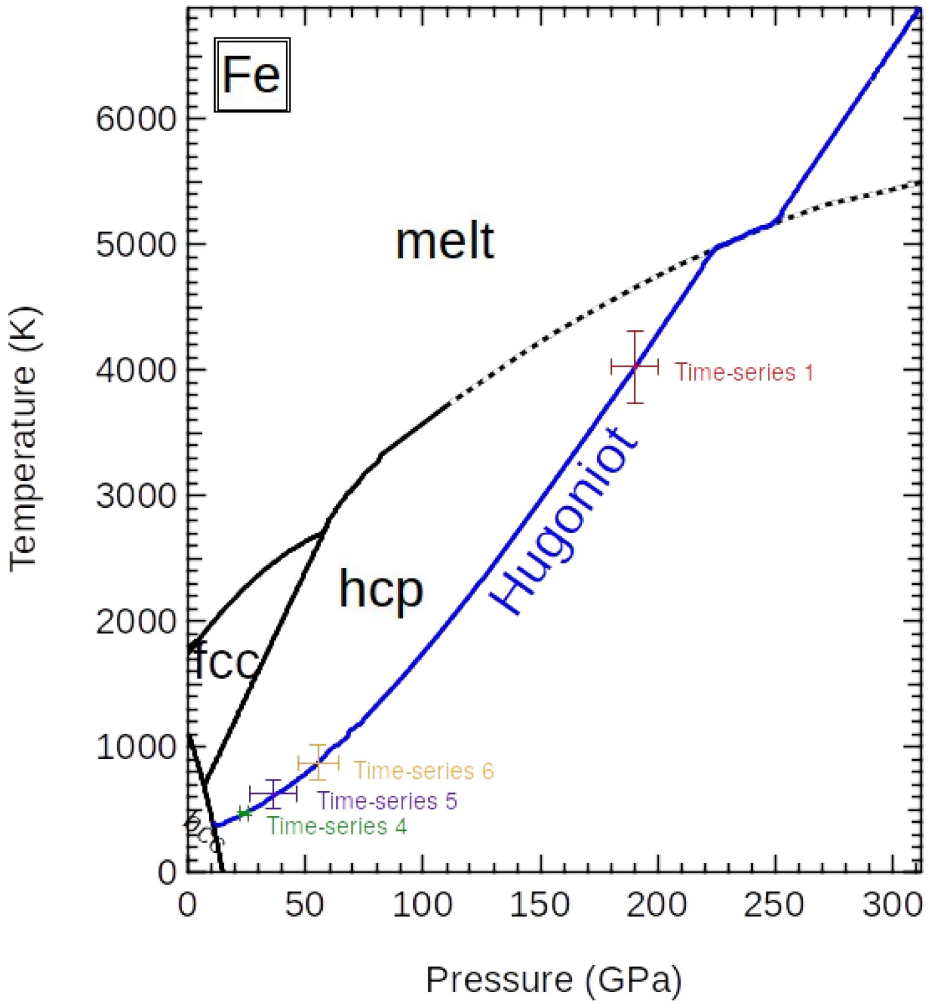
Université de Lille

Experimental Layout



Detector layout optimized for coverage for stress and texture analysis

Pressure and Temperature Range



4 times series

- 23(2) GPa and 494(14) K
- 36(10) GPa and 630(115) K
- 55(9) GPa and 875(140) K
- 187(10) GPa and 4070(285) K

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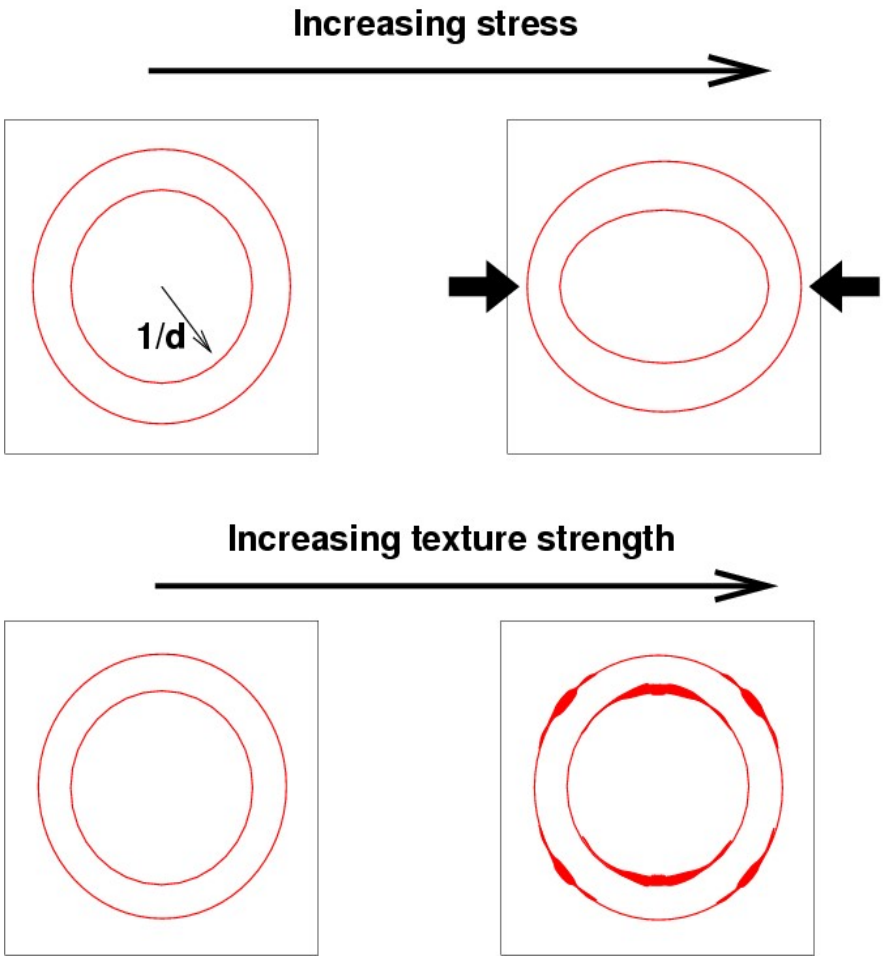
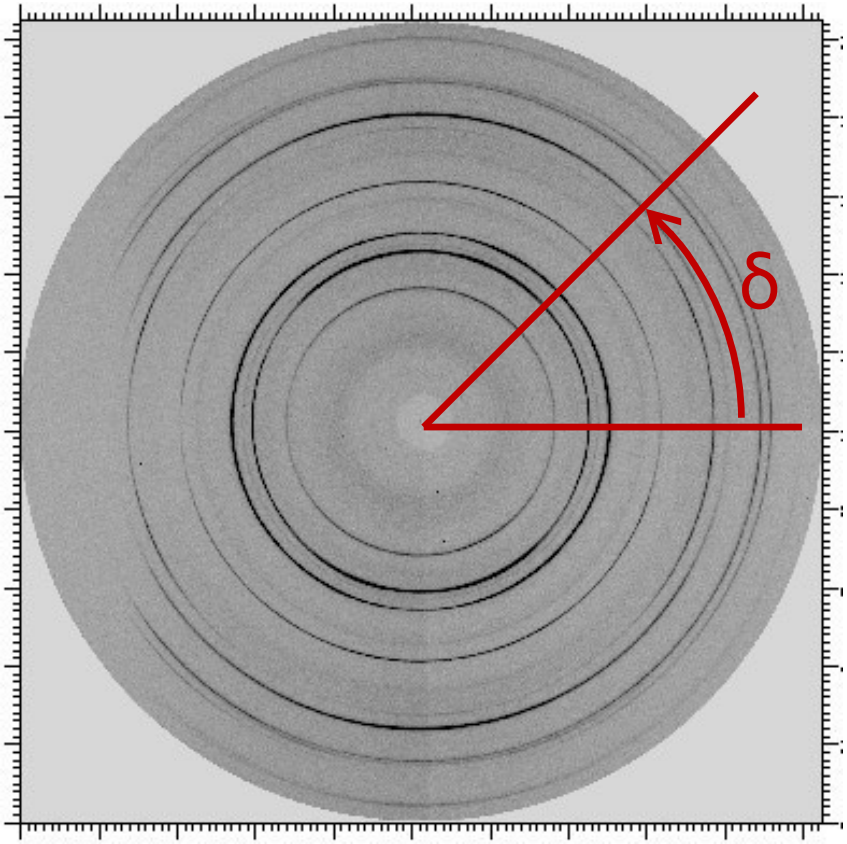
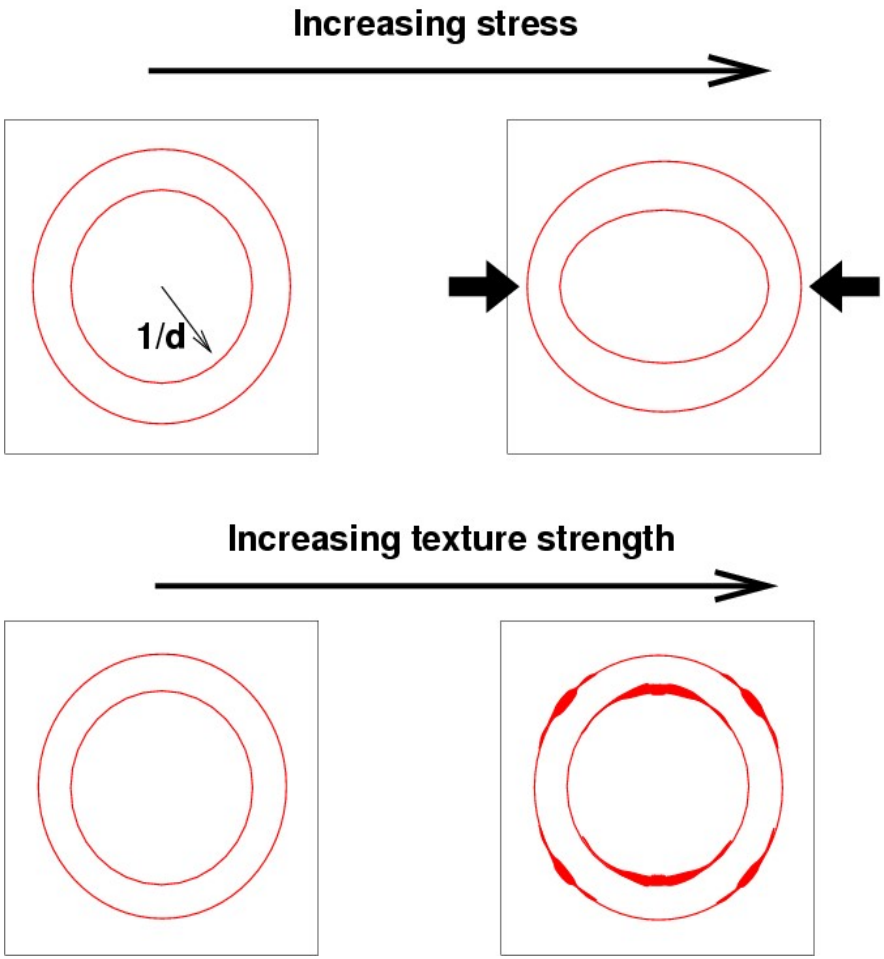
Stress and Texture from X-ray Diffraction

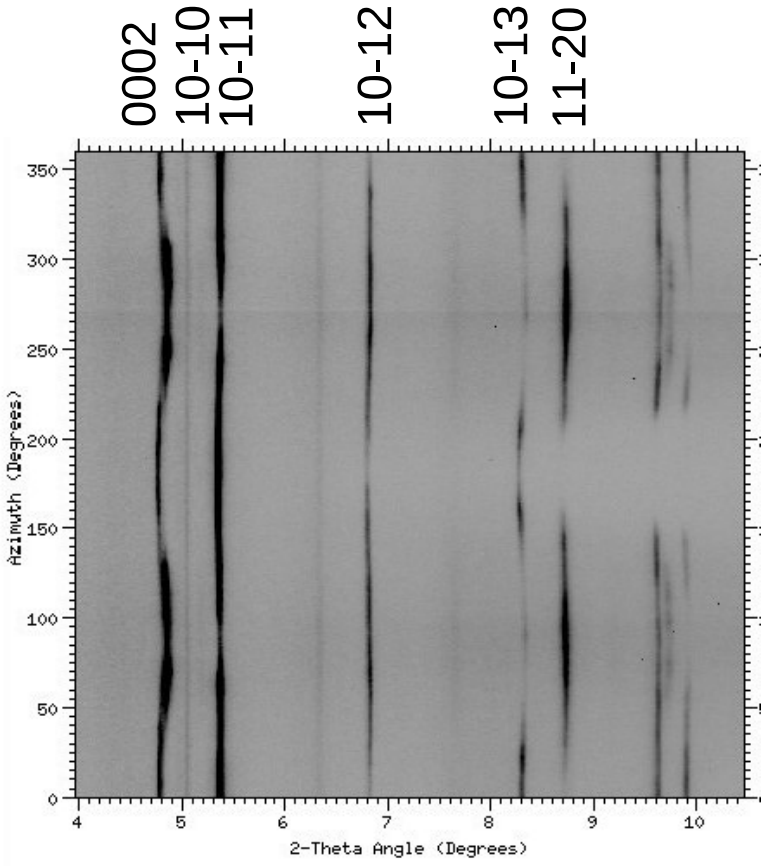




In situ x-ray diffraction
Zn wire sample, 2 mm high
P ~ 10.5 GPa in a static experiment

Stress and Texture from X-ray Diffraction





In situ x-ray diffraction
Zn wire sample, 2 mm high
P ~ 10.5 GPa in a static experiment

In-Situ X-Ray Diffraction on Shocked hcp-Fe

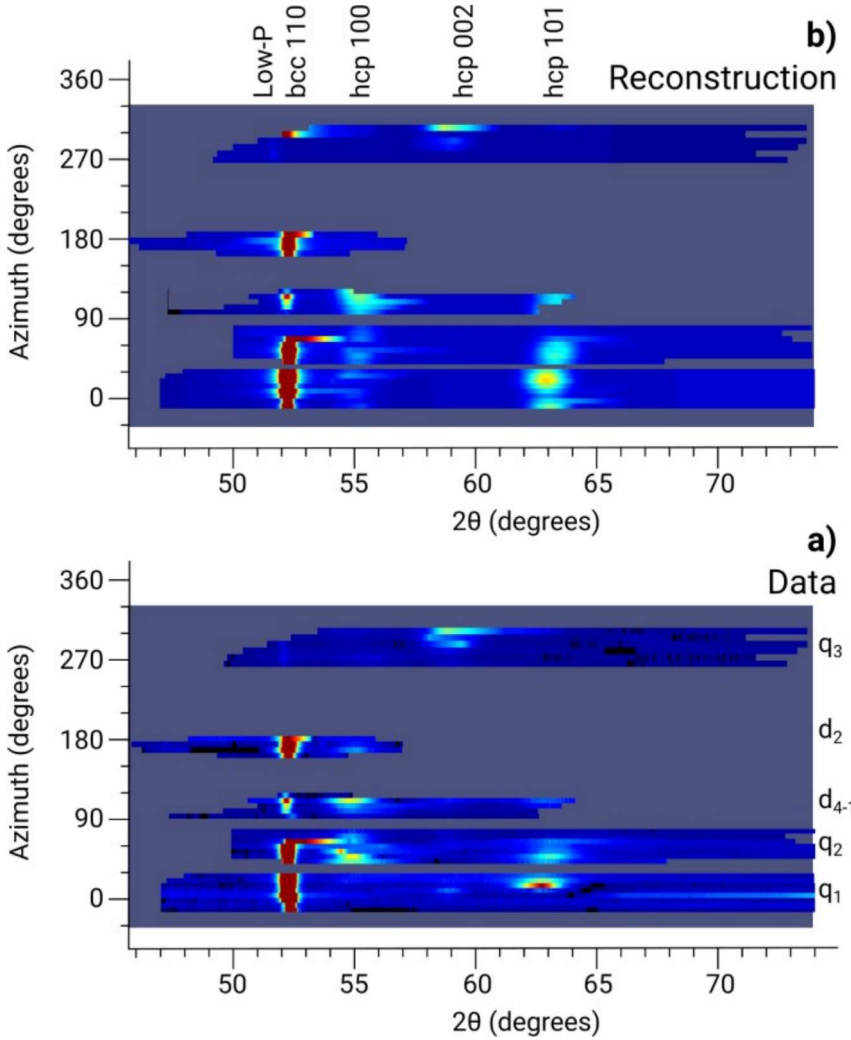
In-shock X-ray diffraction and MAUD reconstruction

Hcp-Fe + ambient P bcc-Fe

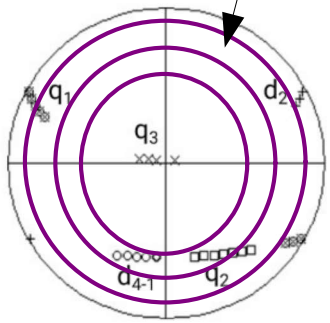
- 193 GPa
- 4230 K

Fit of

- Unit cell parameters
- Lattice strains (effect of stress)
- Lattice preferred orientations (assuming axial symmetry around compression)

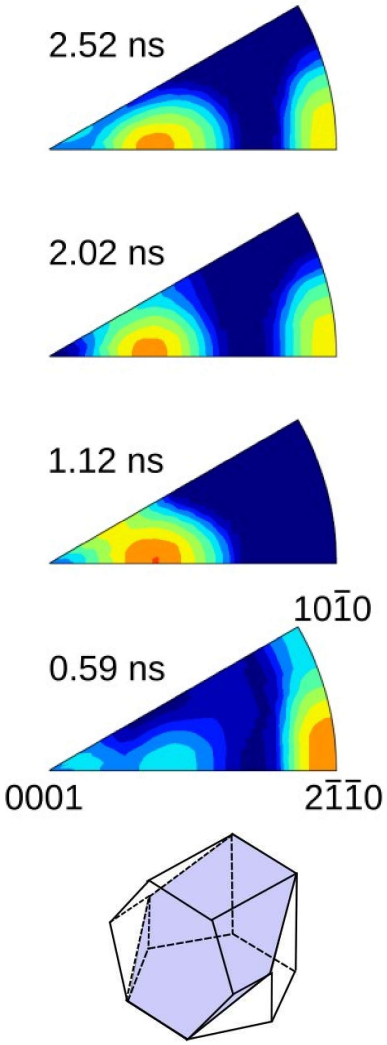


Effect of axial symmetry



c)
Pole figure coverage

Plasticity of Shock Compressed hcp-Fe



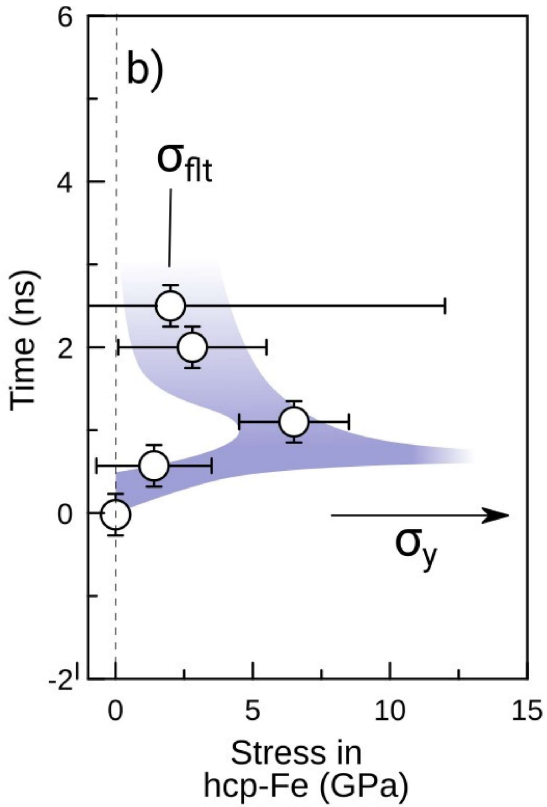
In-situ X-ray diffraction measurement of

- Stress
- Lattice preferred orientation

During laser-driven shock compression

Observations

- Elastic overshoot prior to elastic-to-plastic transition
- Stress relaxation with plastic flow
- Polycrystal grain reorientation in less than 1 ns



Conclusions

Université de Lille

One year ago...

Good Afternoon,

We have you scheduled for a seminar on March 12th, 2020. I was hoping you could send Paul or Myself your abstract for your talk by Thursday, February 27th?

We Look forward to your talk, Please let me know if you have any questions.

Thank you,
Jessica Letteer

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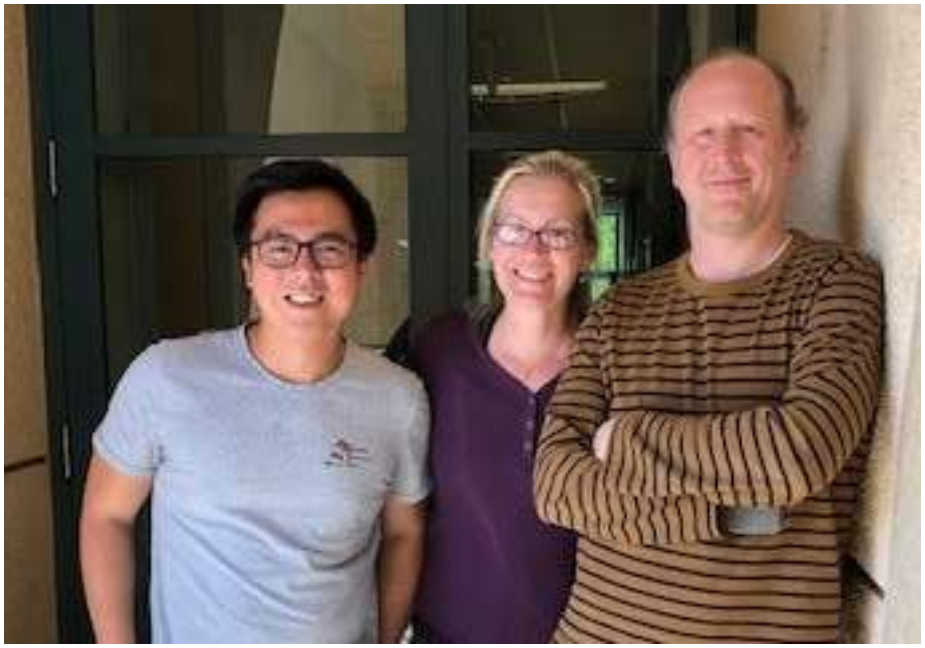
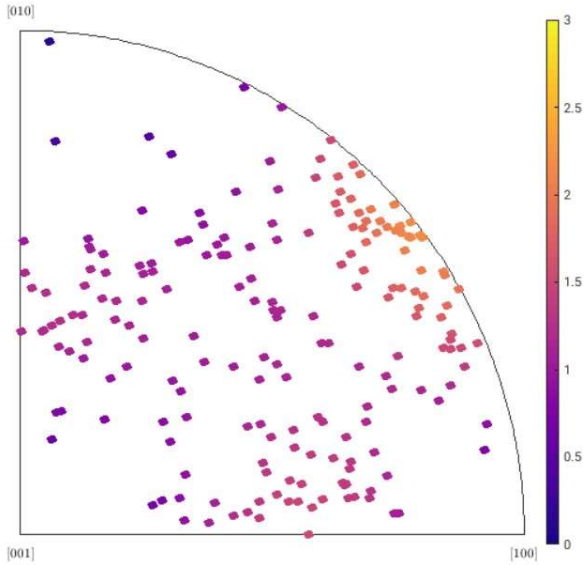


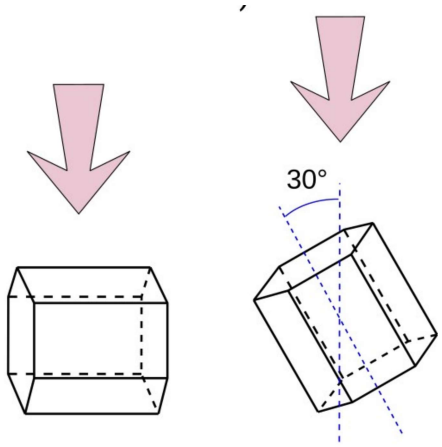
Escaping from Stanford on March 12, 2020!

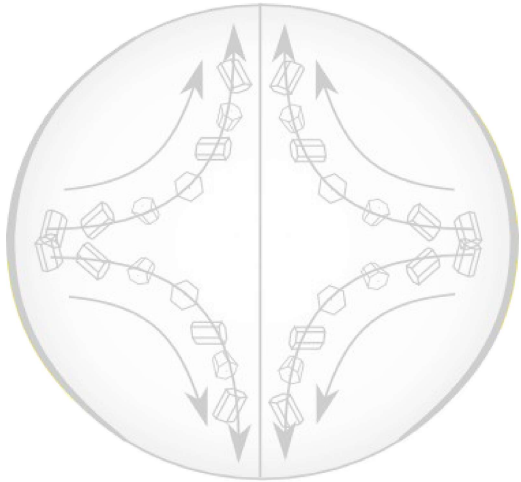
My Work in Recent Years



Monitoring single silicate grains rotate and undergo phase transformations inside a polycrystal in a DAC

Watching hcp metals polycrystals build stress and texture in deformation experiments, static or dynamic.





Incorporate materials plasticity into Earth's dynamical models and predict its properties and evolution