

# An Experimental View of the Earth's Upper Mantle: Densification, Deformation and Recovery of Olivine-rich Rocks

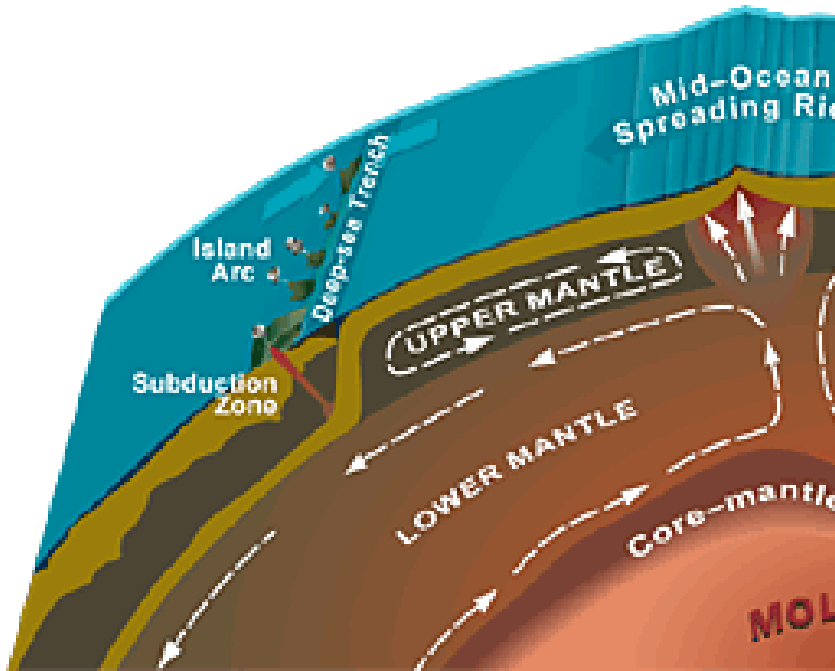
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Mark Zimmerman<sup>1</sup>, Seth Kruckenberg<sup>2</sup>

<sup>1</sup>University of Minnesota, Department of Earth Sciences

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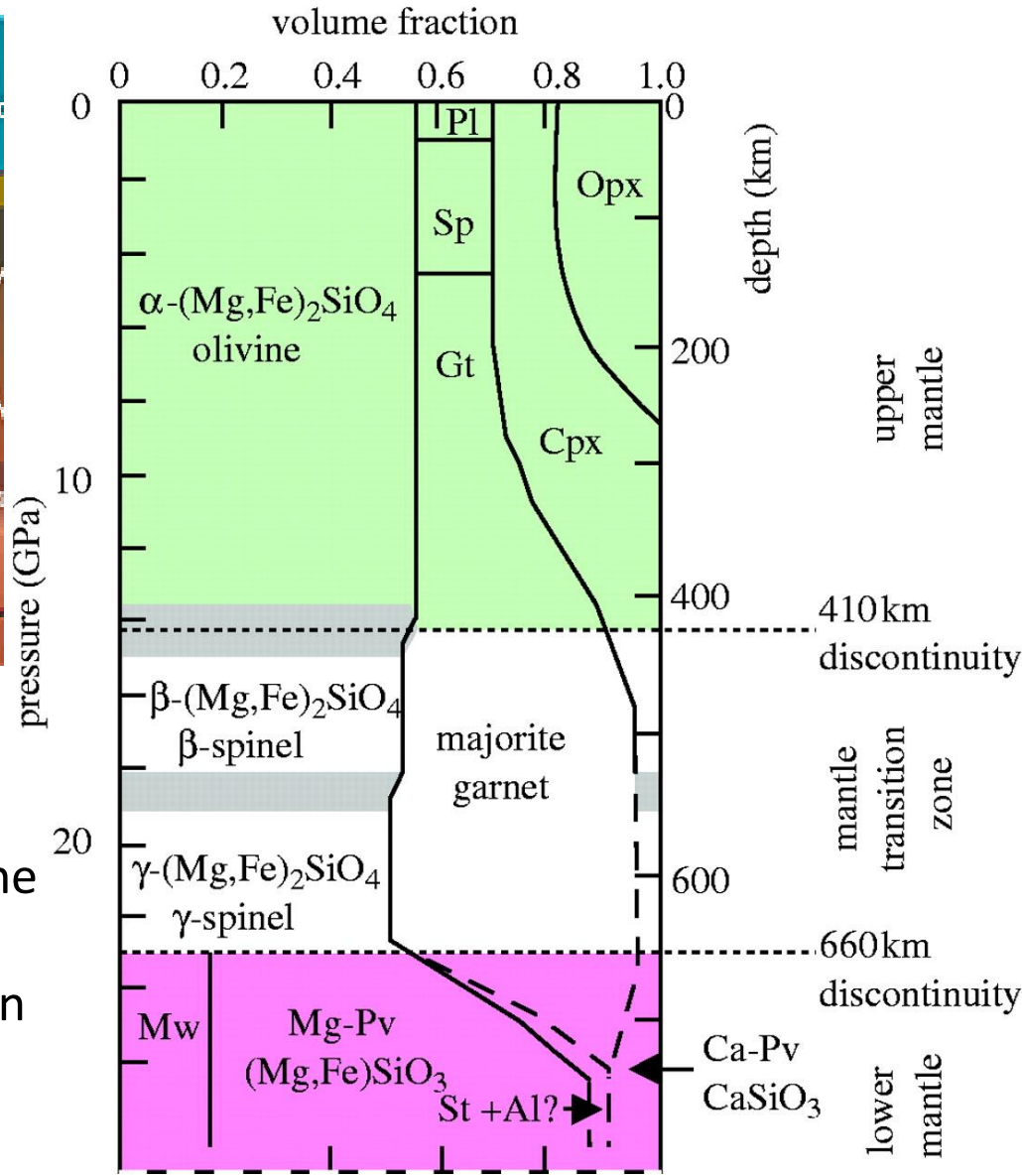
DOE NNSA SSGF Annual Program Review 2018

# Introduction to the Earth's mantle



Detrick (2004)

**Olivine** makes up nearly 60% of the Earth's upper mantle and is considered to control deformation in this region of the Earth



Bovolo (2005)

# Deformation Mechanisms and Power-law Creep

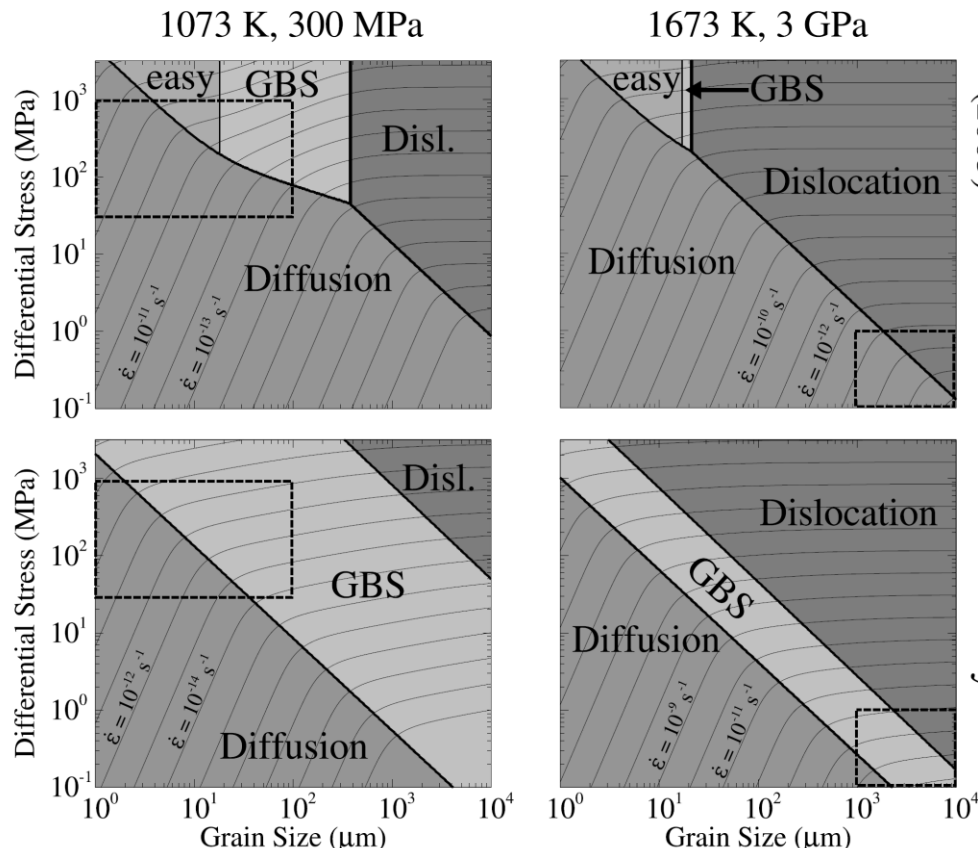
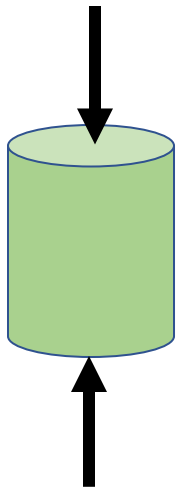
$$\dot{\epsilon} = A(T, P, \mu_i) \sigma^n d^{-m}$$

Strain rate is related to stress and grain-size by power-law;  
exponents  $n$  and  $m$  indicate deformation mechanism

Diffusion Creep:  $n = 1; m = 3$

Dislocation Creep:  $n = 3 - 5; m = 0$

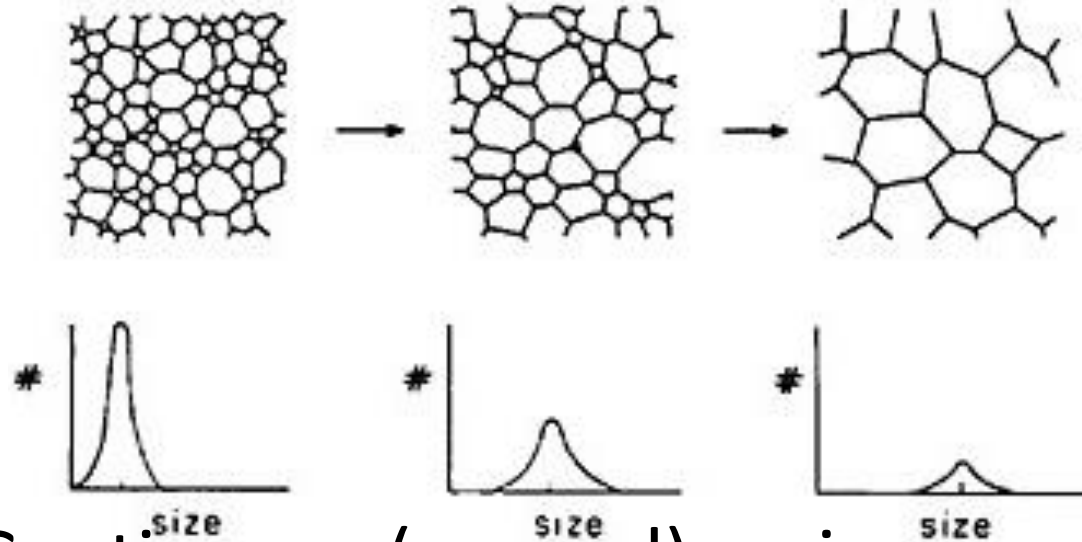
Dislocation Accommodated GBS:  $n = 2 - 5; m = 1$



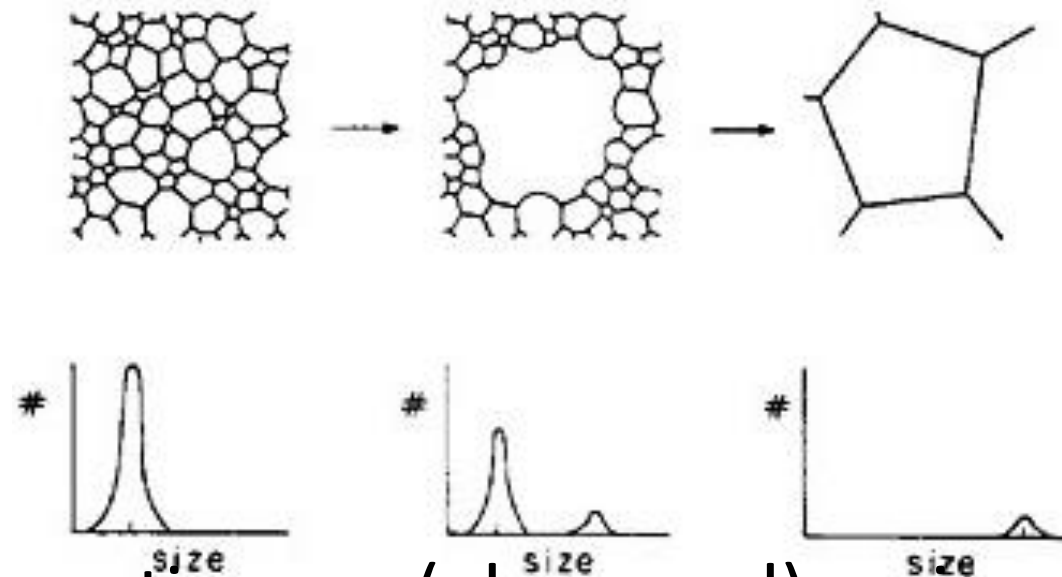
Hirth and Kohlstedt  
(2003)

This Study

# Mechanisms of grain growth

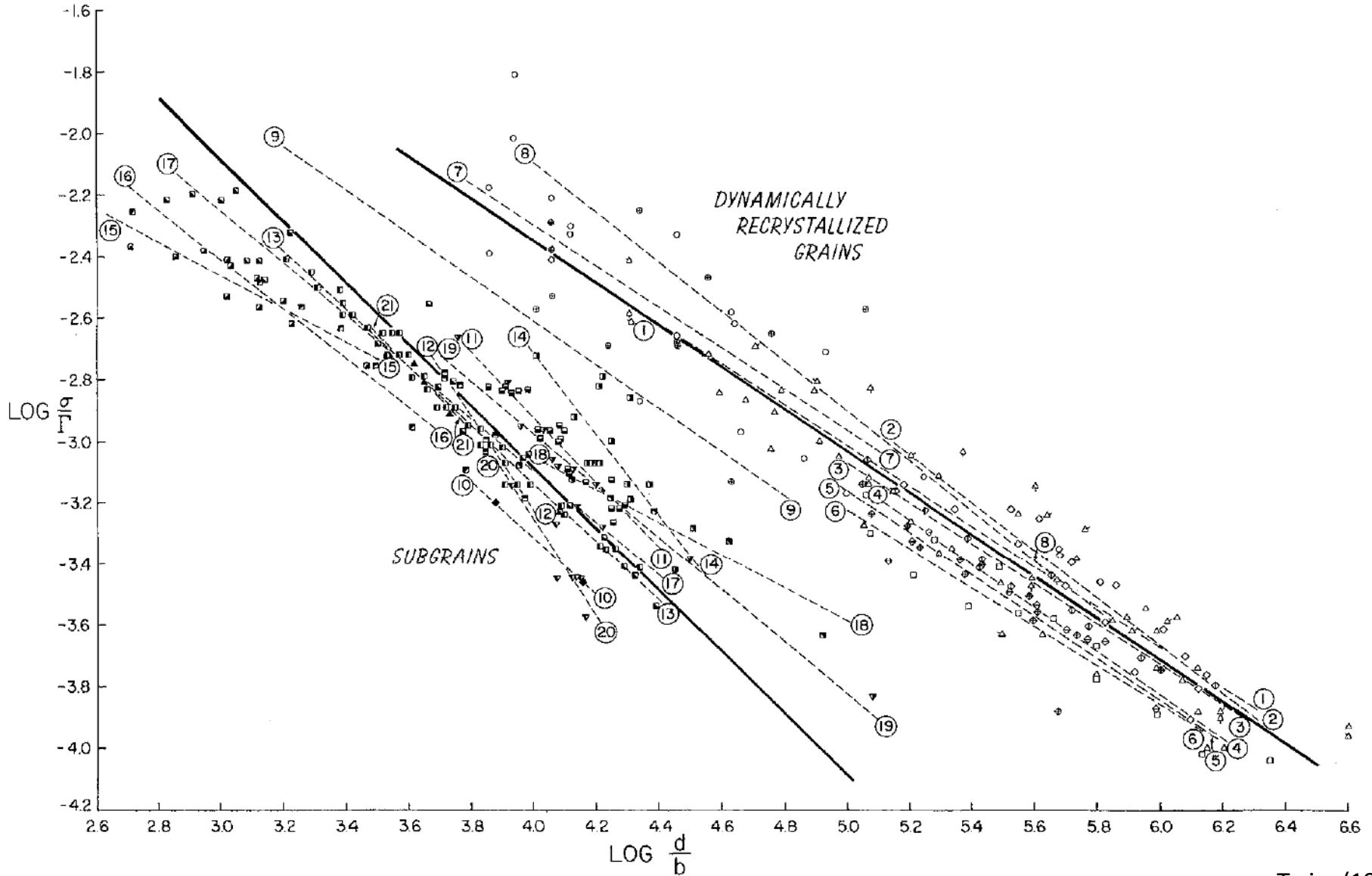


Continuous (normal) grain growth

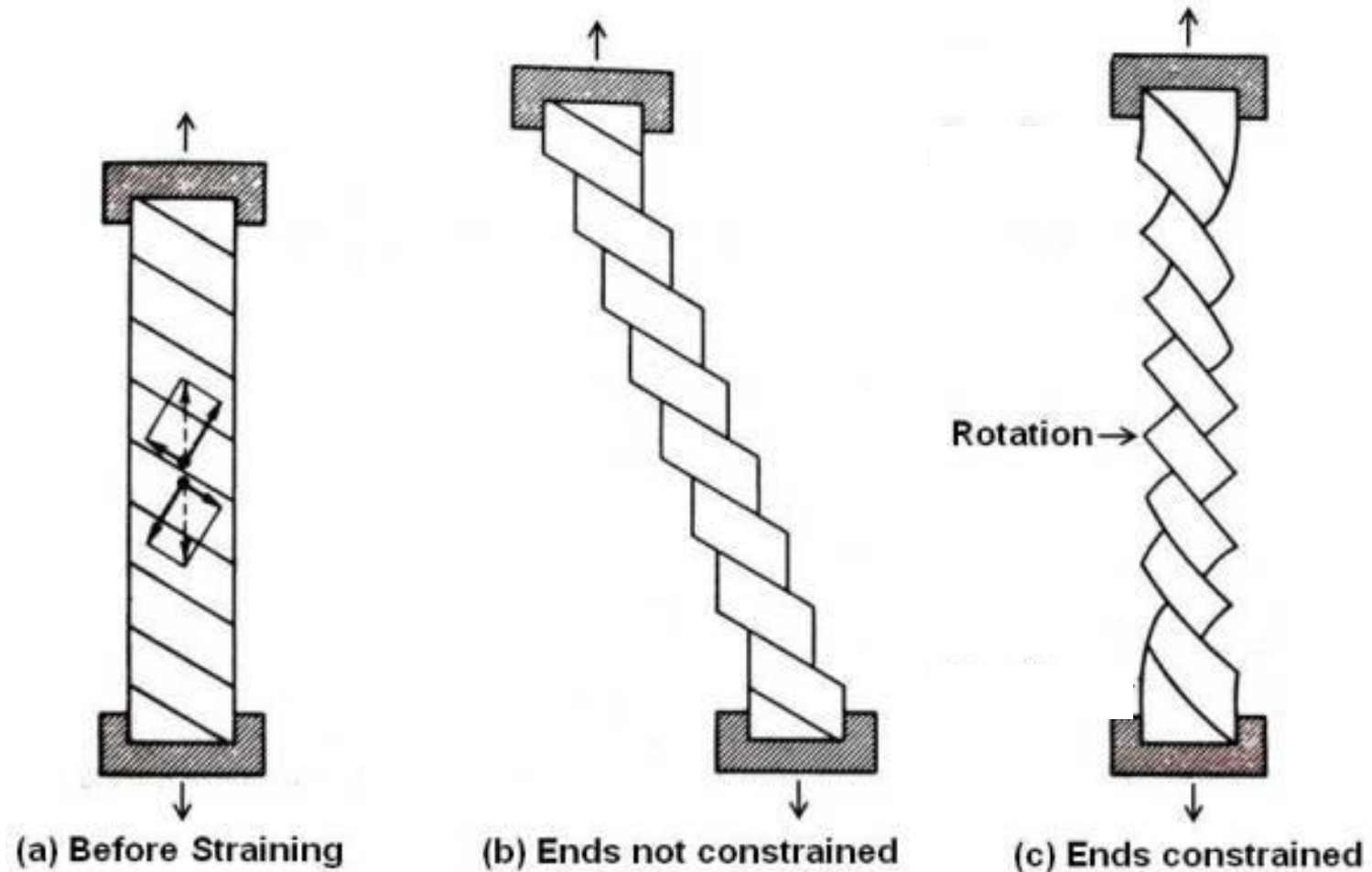


Discontinuous (abnormal) grain growth

# Dynamic Recrystallization: Grain-size reduction induced by deformation

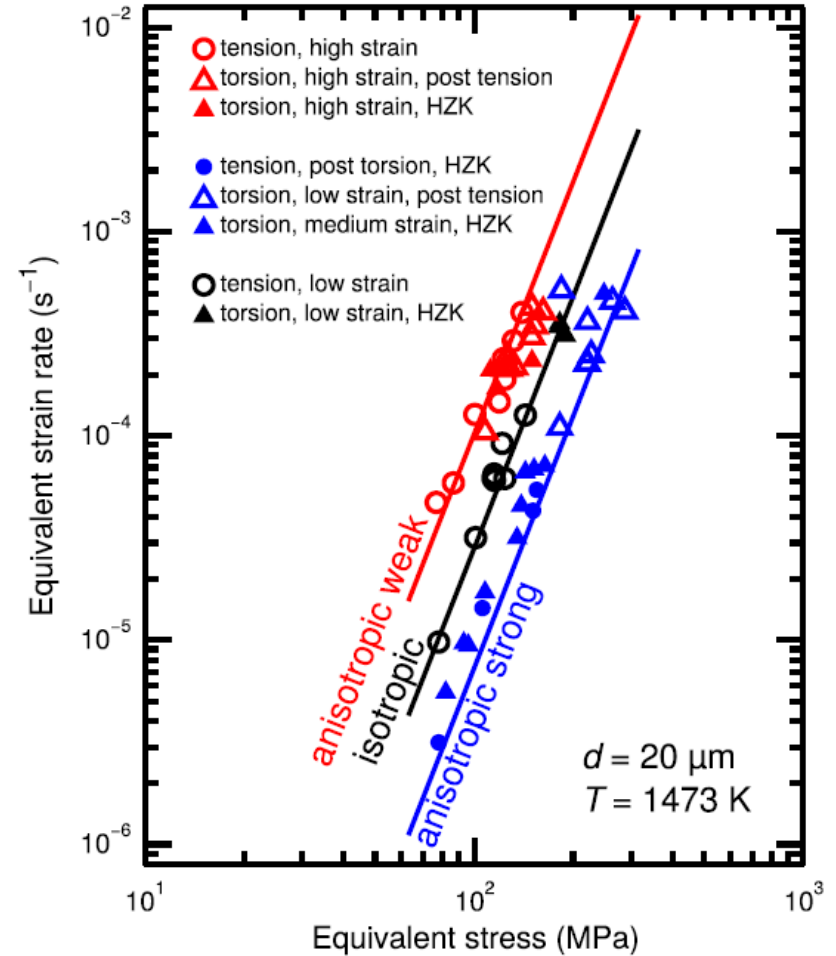
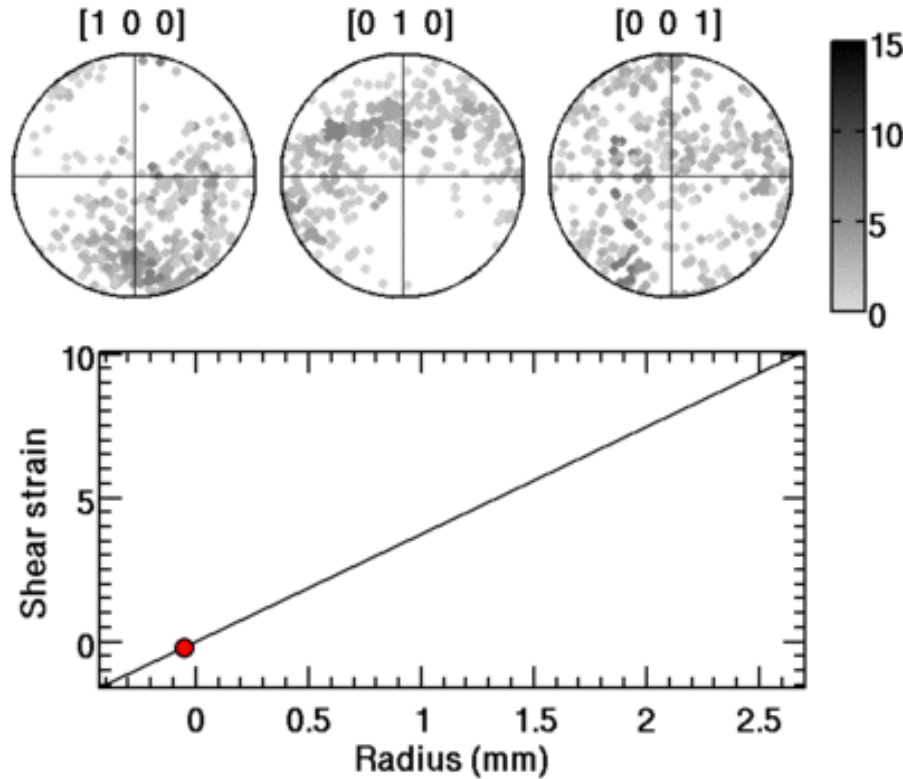


# Crystallographic Preferred Orientation (CPO): rotation and crystallographic alignment of grains due to slip of dislocations

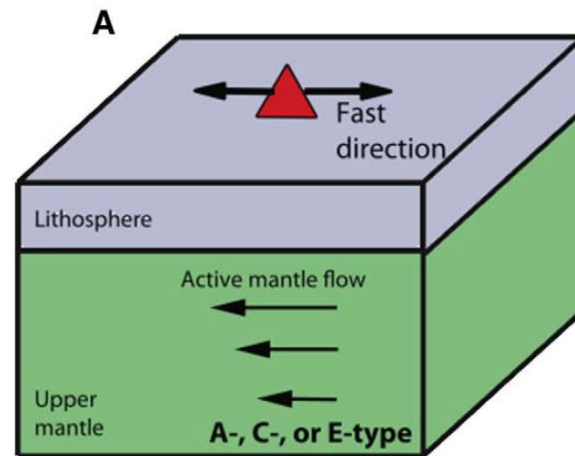
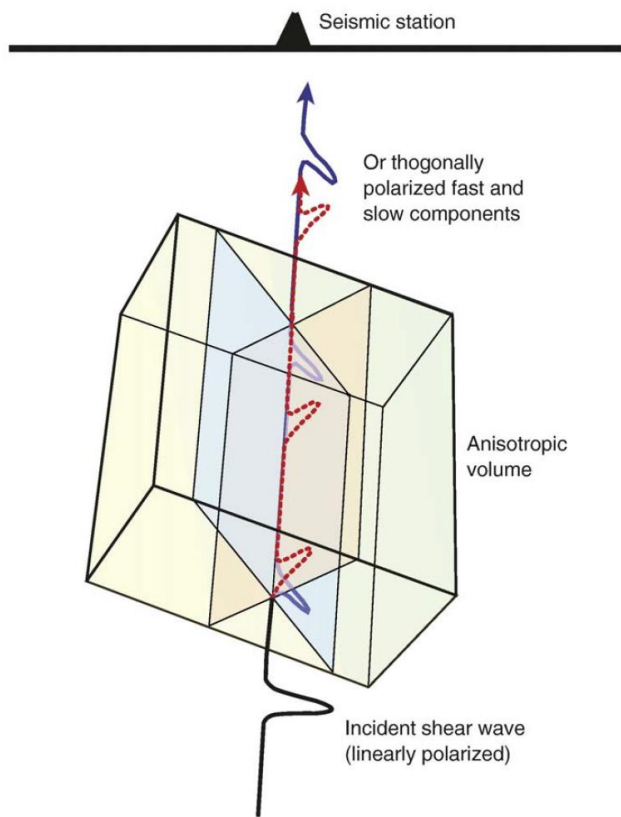


**Schematic Representation of Slip in Tension**

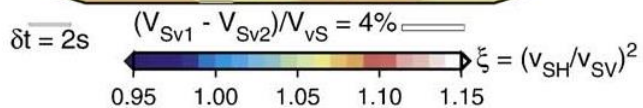
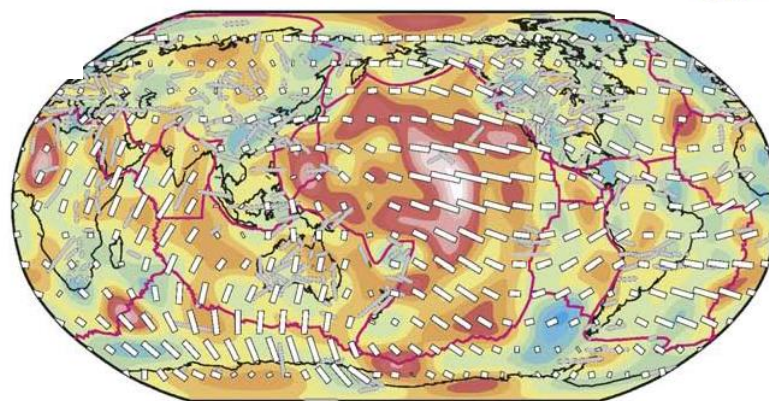
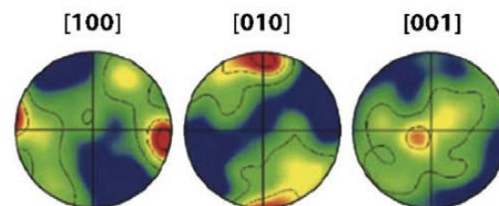
# Crystallographic Preferred Orientation (CPO) and Anisotropic Viscosity



# Seismic Anisotropy

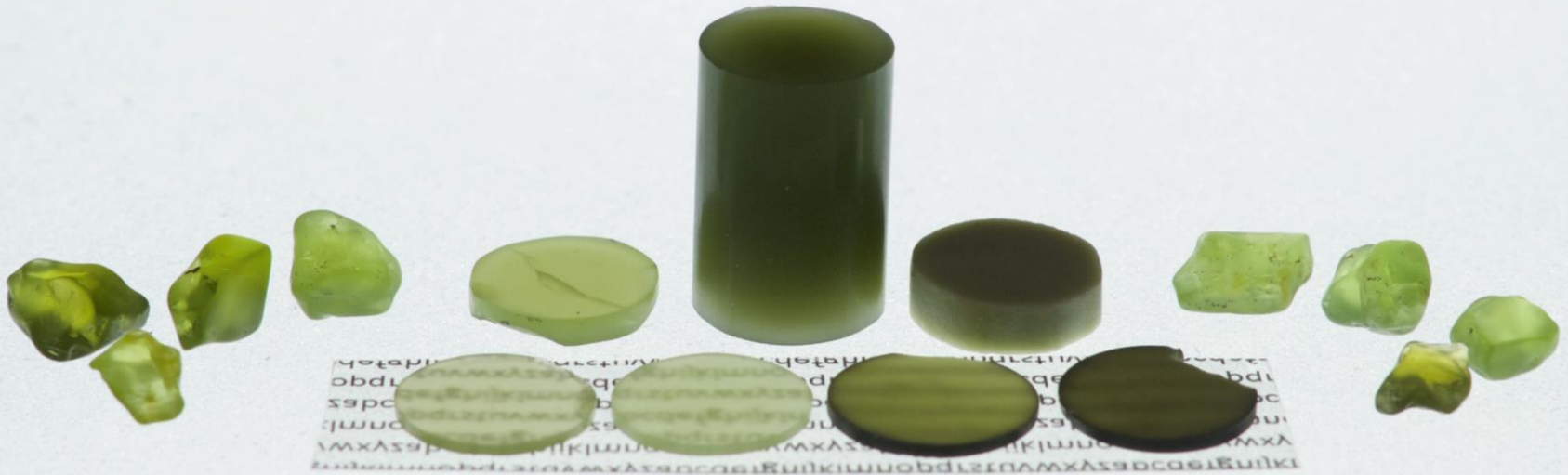


Olivine A-type

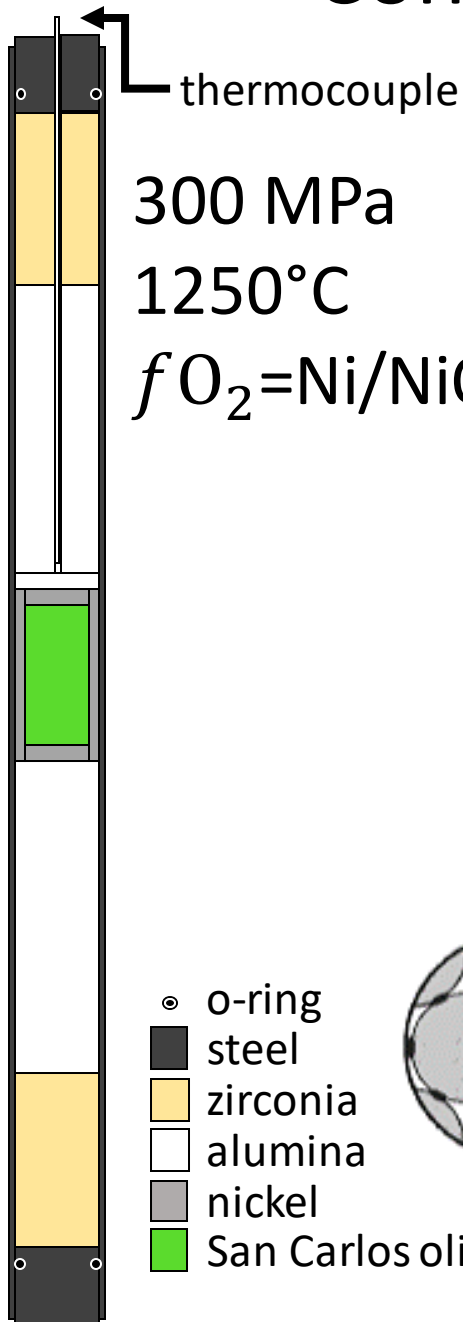




# Development of improved starting material for experimental studies on olivine-rich rocks: Evacuated Hot-Pressing



# Conventional hot-pressing



Gem-quality crystals of San Carlos olivine (hand picked)



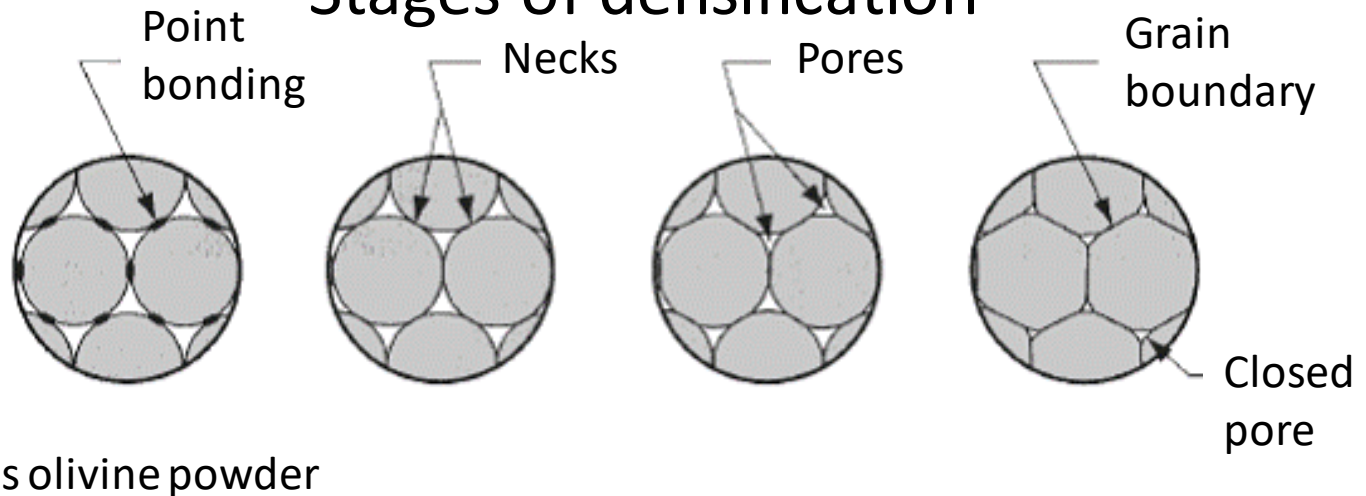
Pulverized by fluid-energy mill ( $6 \pm 2 \mu m$ )  
Heated in mixed gas to  $T \approx 1000^\circ C$



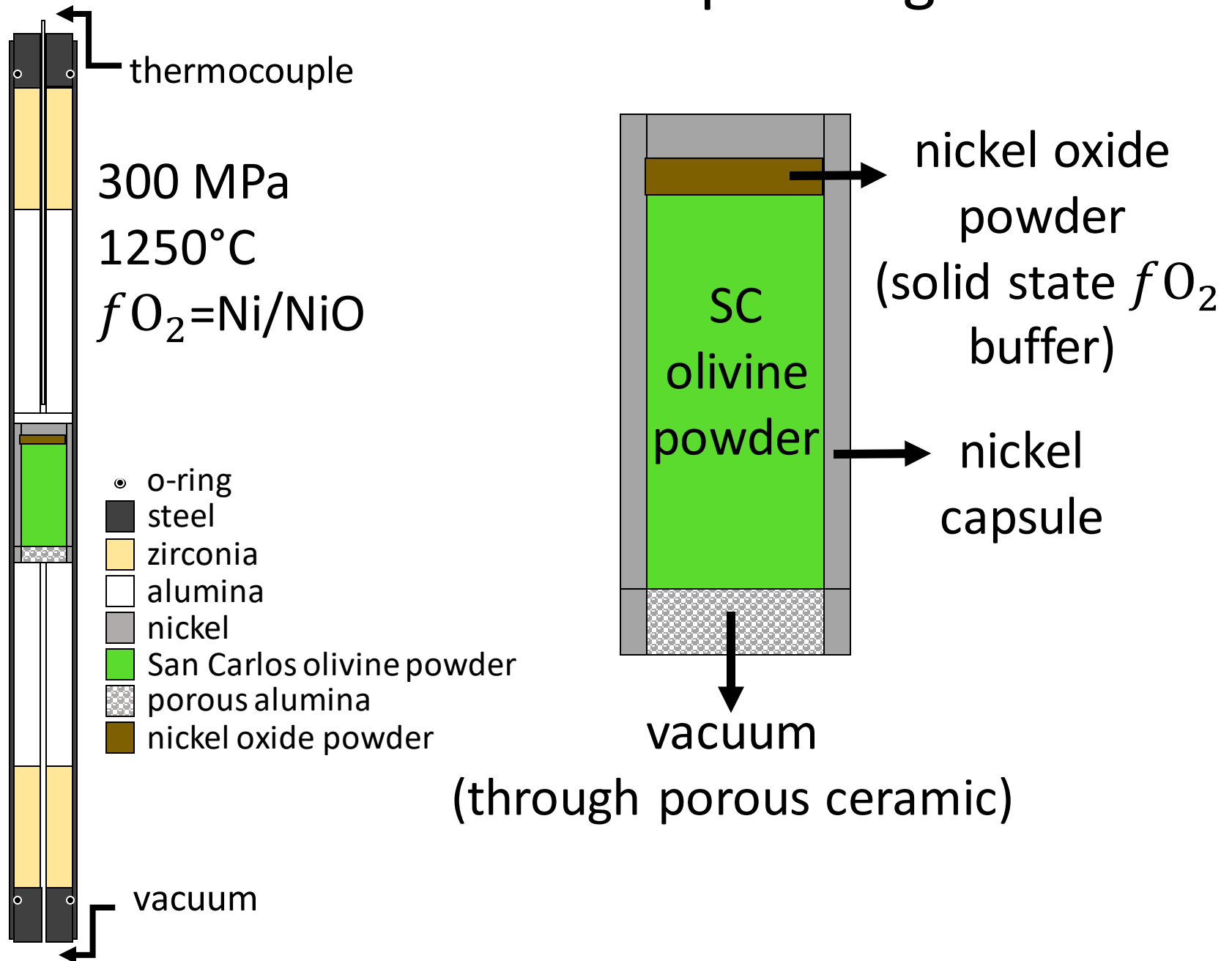
Hot pressed at 300 MPa, 1250°C, roughly 4 h in a sealed Ni capsule (~98% dense)



## Stages of densification

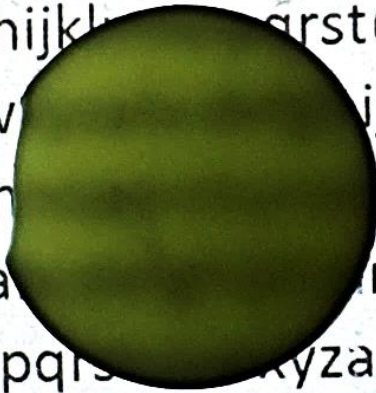


# Evacuated hot-pressing



# Conventional hot-pressed San Carlos olivine

nopqrstuvwxyzabcdefghijklmnop  
bcdefghijklmnopqrstuvwxyz  
qrstuvwxyzabcdefghijklmnop  
ghijklmnopqrstuvwxyzabcde  
vwxyzabcdefghijklmnopqrstu  
vwxyzabcdefghijklmnopqrstu  
vwxyzabcdefghijklmnopqrstu



nopqrstuvwxyzabcdefghijklmnop  
vwxyzabcdefghijklmnopqrstu  
mnopqrstuvwxyzabcdefghijklmnop  
abcdefghijklmnopqrstuvwxyz  
vwxyzabcdefghijklmnopqrstu  
ghijklmnopqrstuvwxyzabcde  
vwxyzabcdefghijklmnopqrstu  
stuvwxyzabcdefghijklmnopq



# Evacuated hot-pressed San Carlos olivine

rstuvwxyzabcdefghijklmnop  
ghijklmnopqrstuvwxyzabcde  
vwxyzabcdefghijklmnopqrstu  
vwxyzabcdefghijklmnopqrstu  
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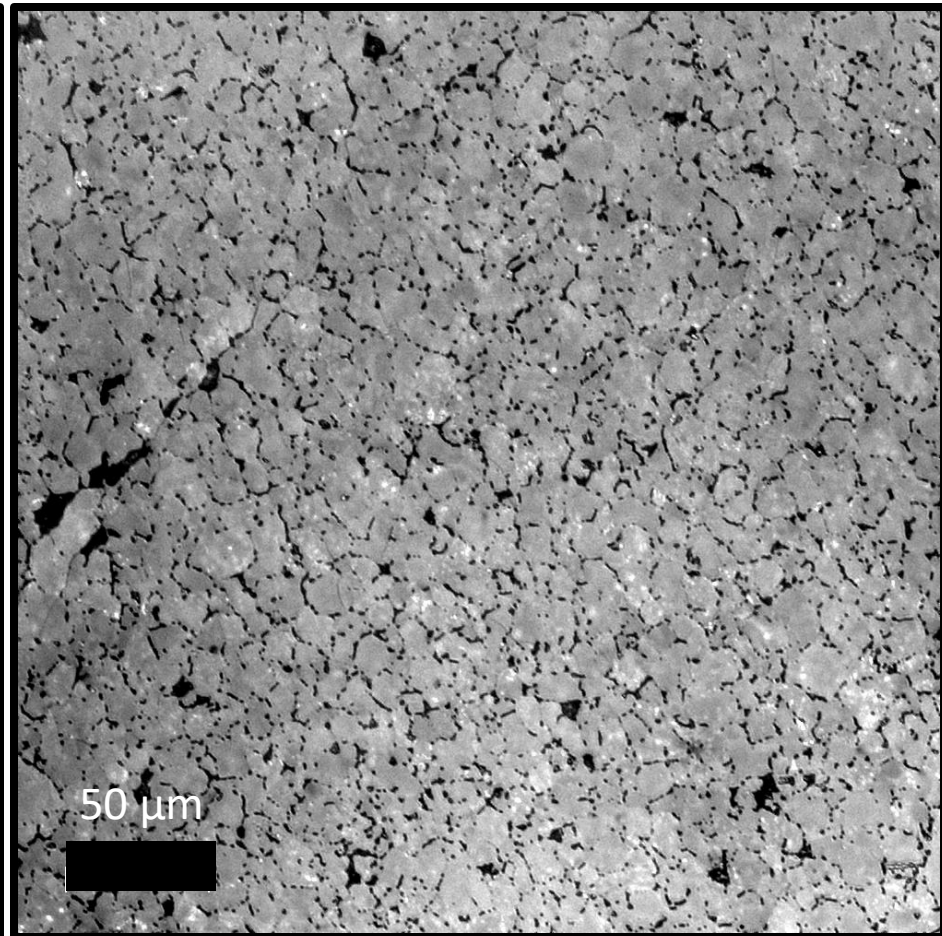
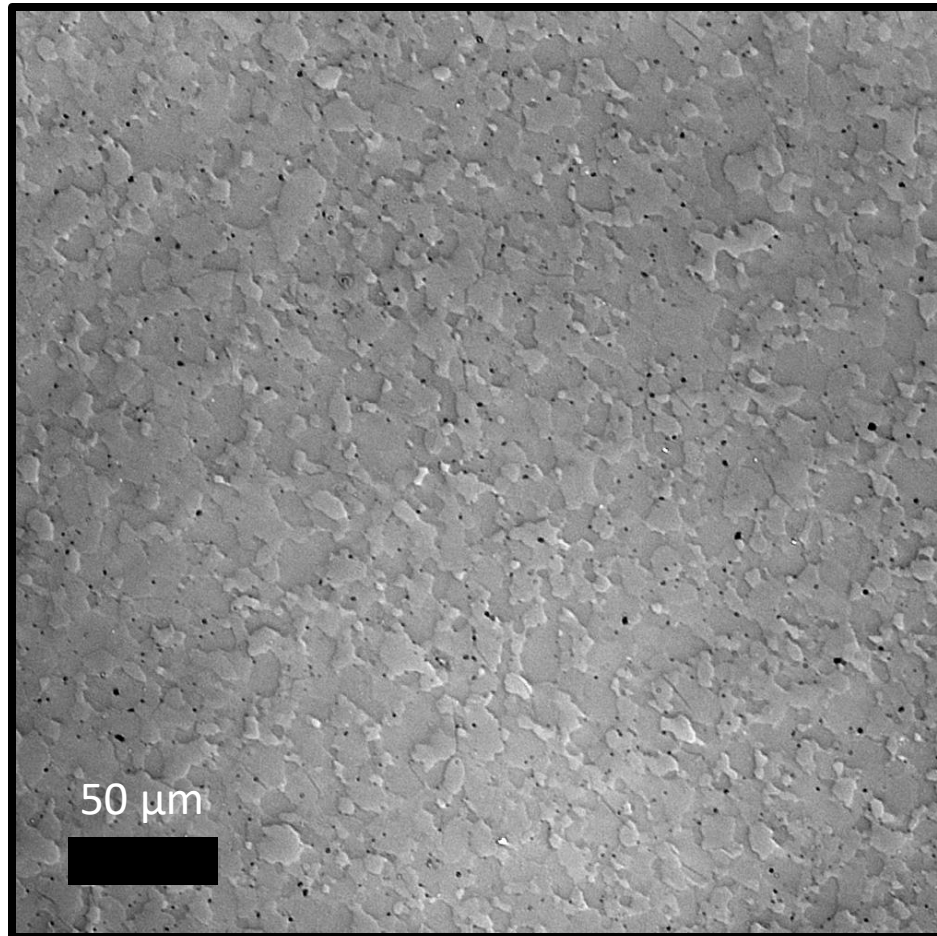


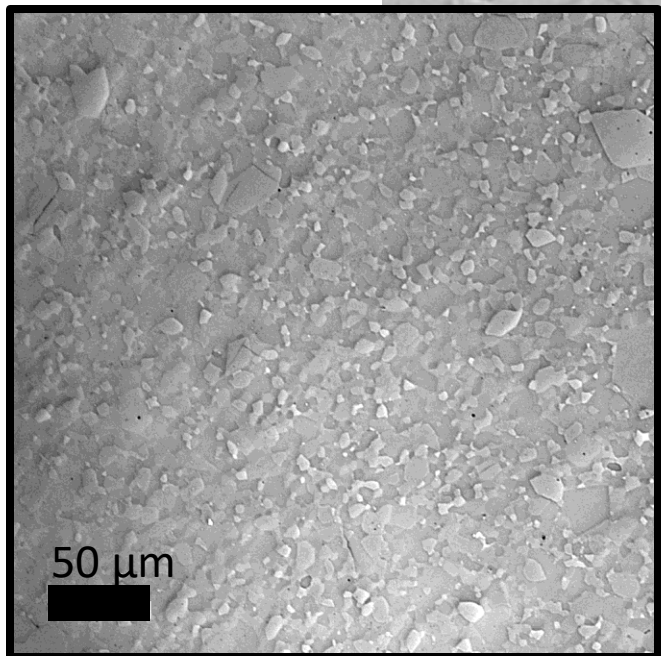
*transmitted light through 1 mm thickness; 12 mm diameter*

# Static annealing of conventional hot-press at 1 atm

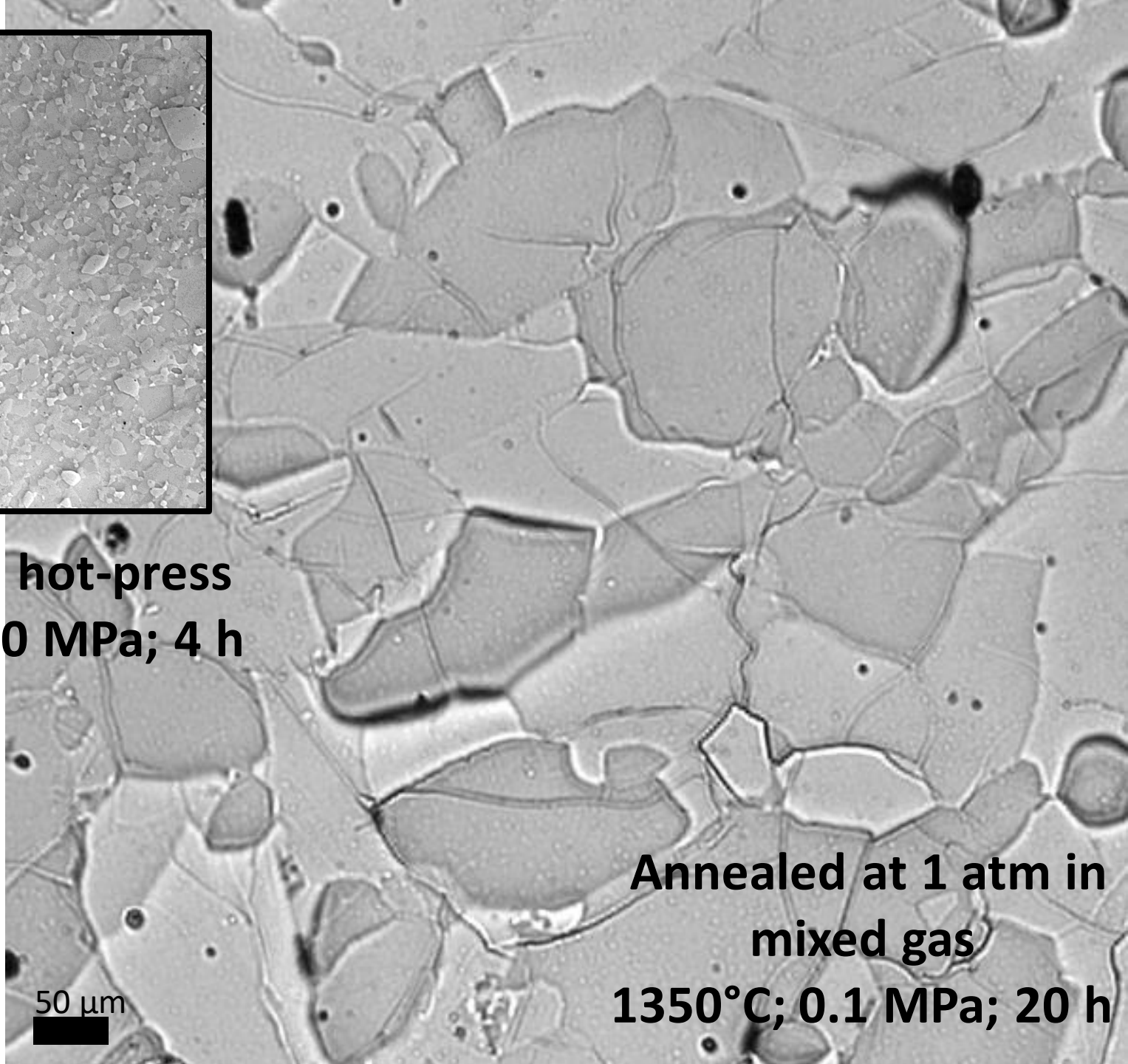
Conventional hot-press  
1250°C; 300 MPa; 4 h

Annealed in mixed gas  
1350°C; 0.1 MPa; 20 h



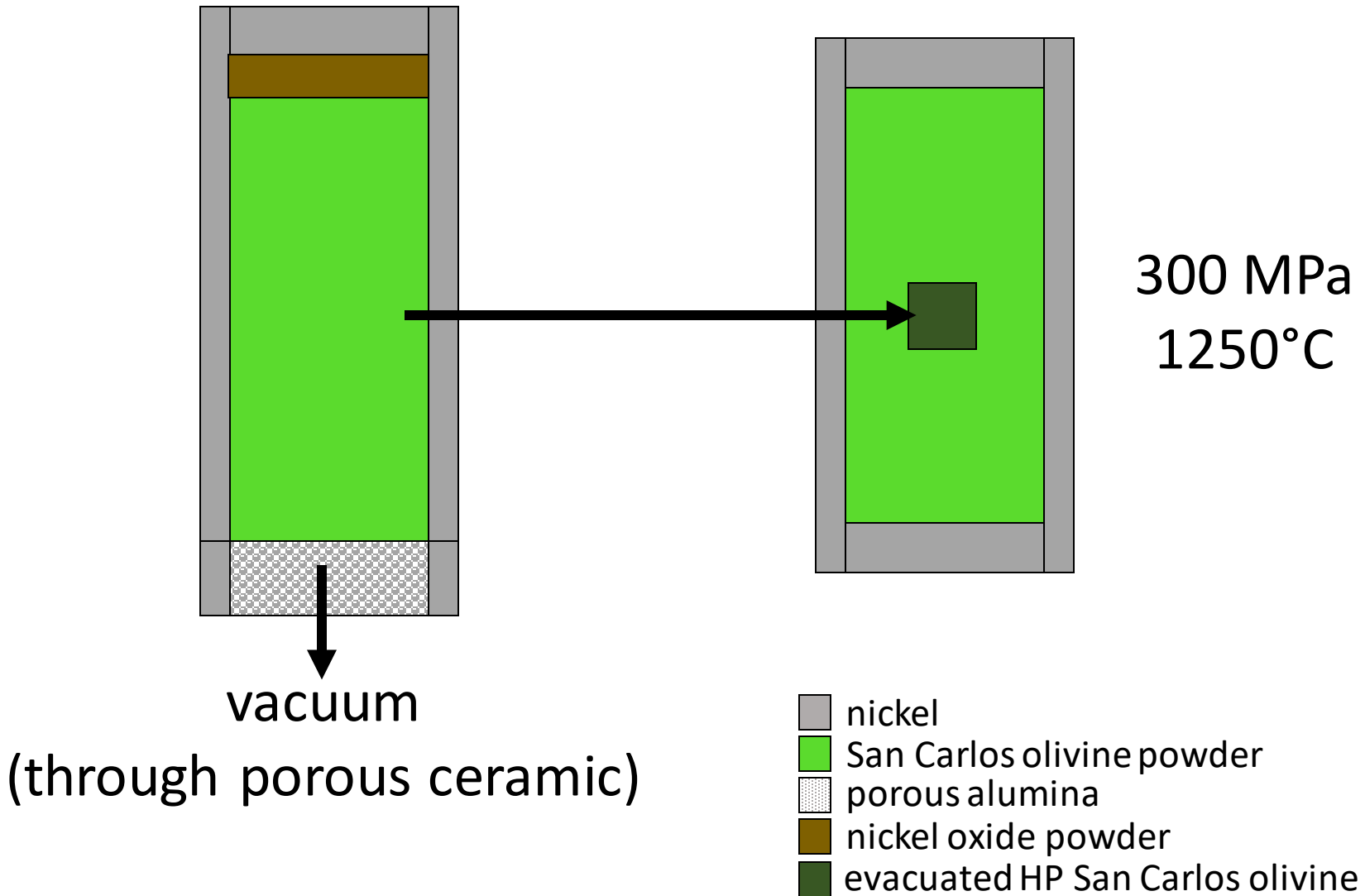


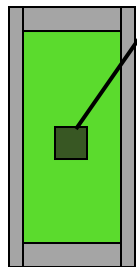
**Evacuated hot-press  
1250°C; 300 MPa; 4 h**



**Annealed at 1 atm in  
mixed gas  
1350°C; 0.1 MPa; 20 h**

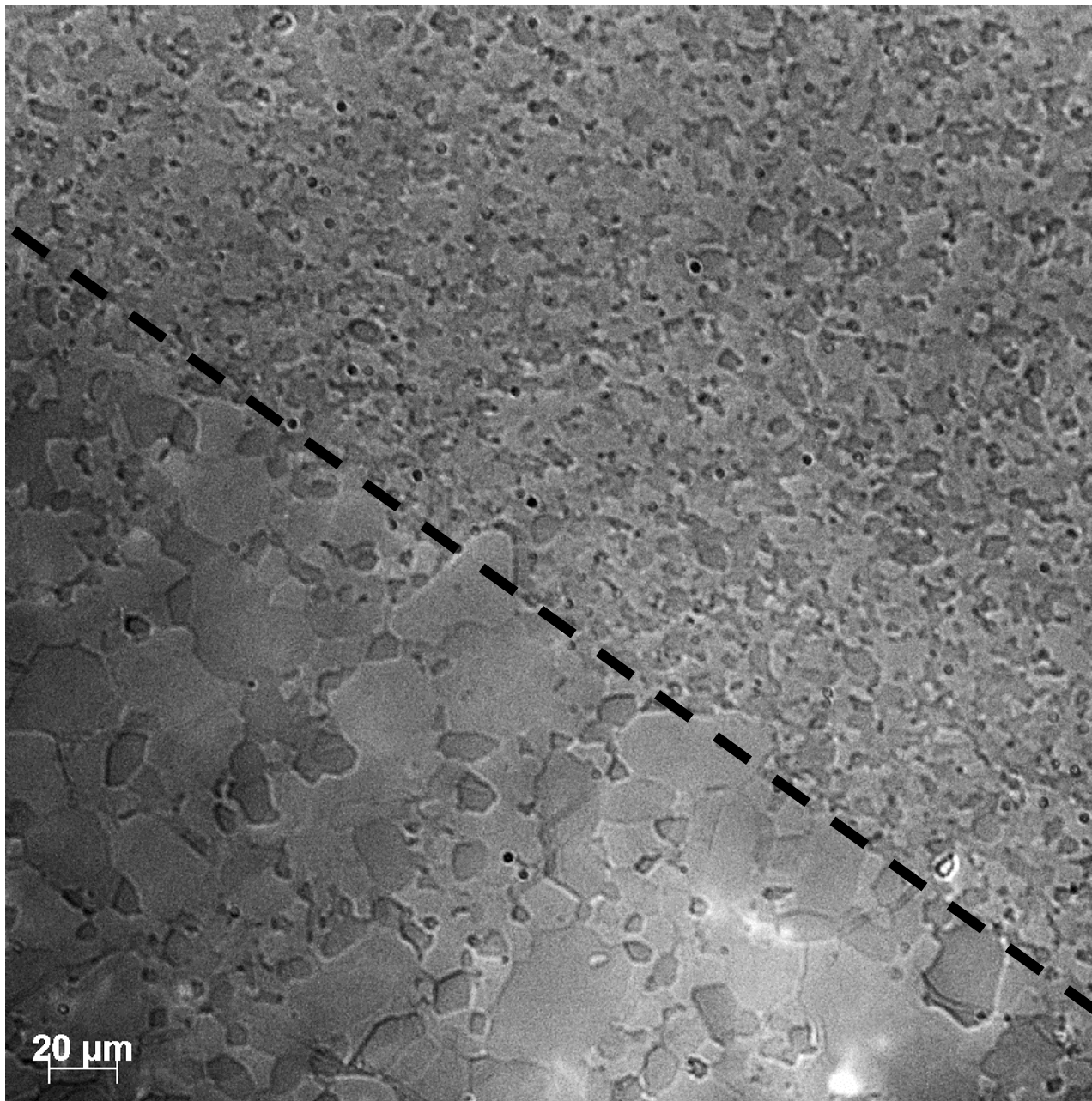
# Static annealing of evacuated hot-press at 300 MPa





**1250°C**  
**300 MPa**  
**4h**

Static anneal  
of **evacuated**  
**hot-press**



**Conventional**  
**hot-press** of  
surrounding  
evacuated  
hot-press

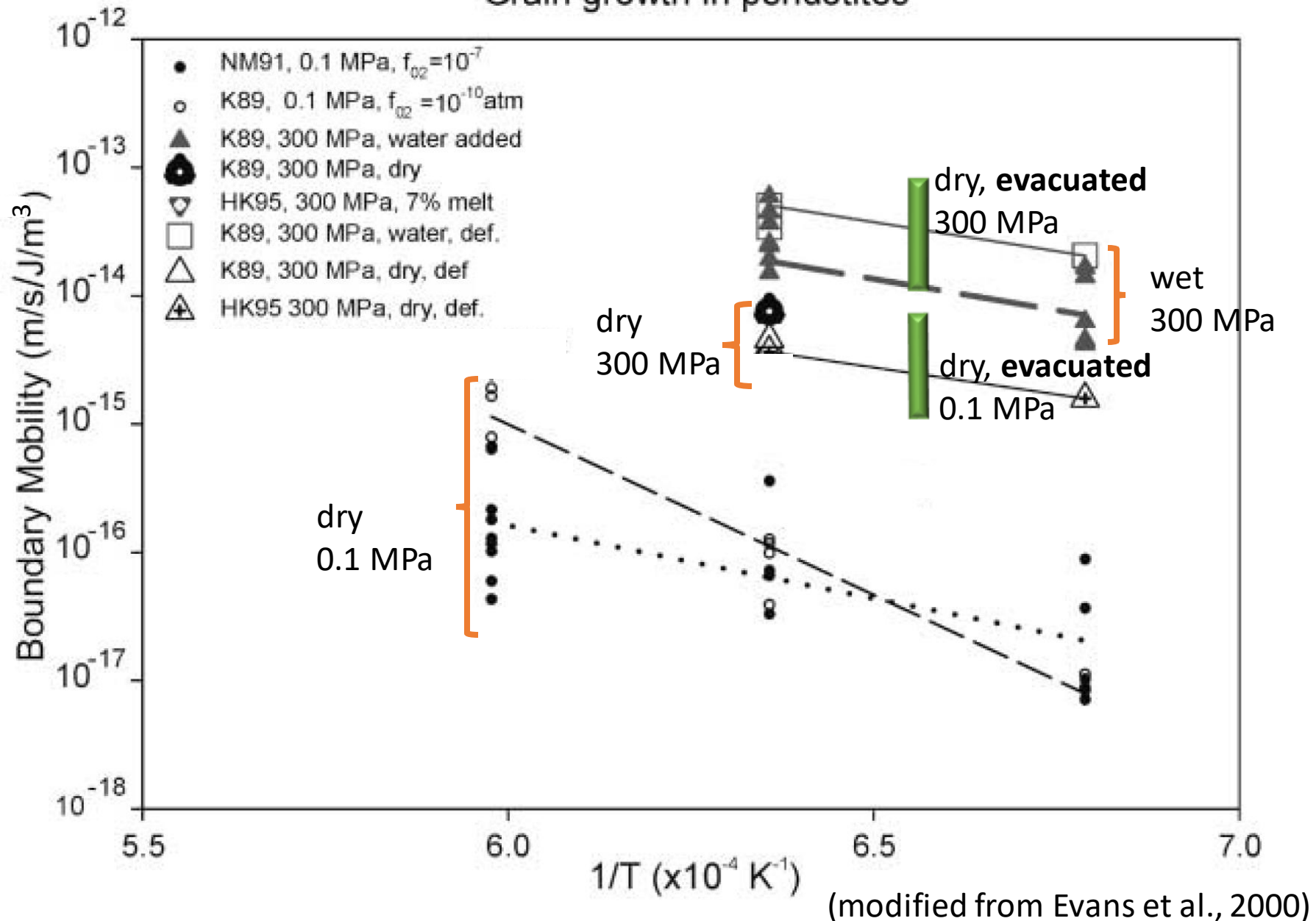
(same starting  
powder)

20  $\mu\text{m}$



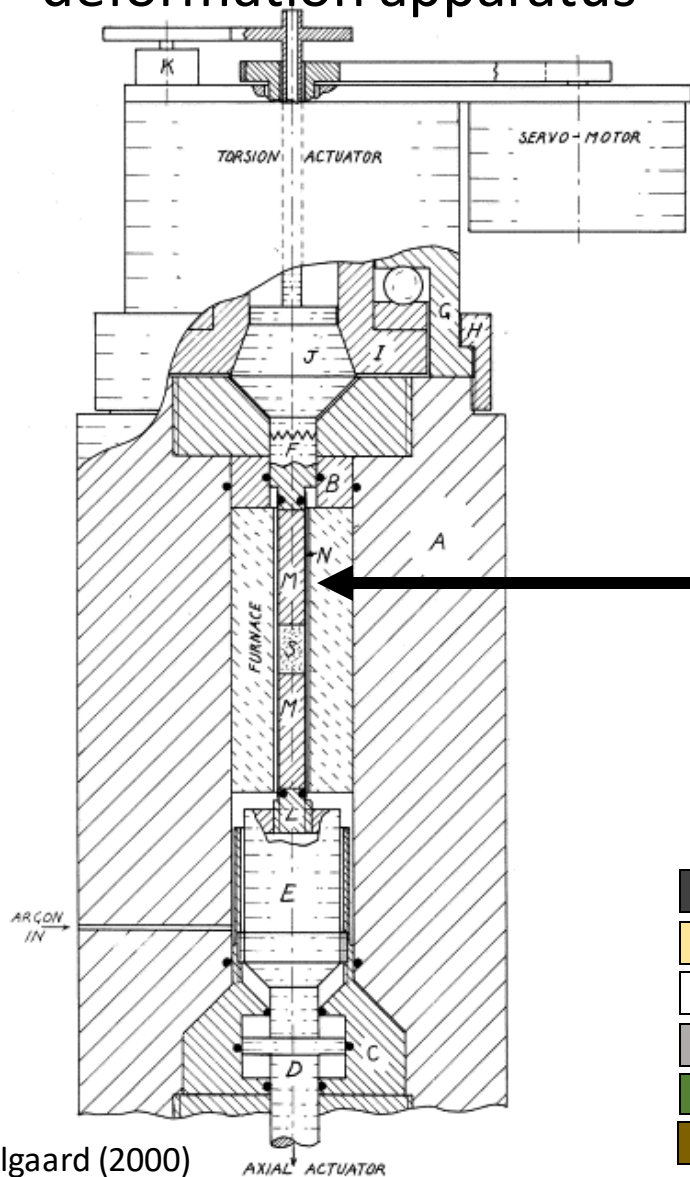
# Comparison with previous work

## Grain growth in peridotites



# High-strain torsional deformation experiments

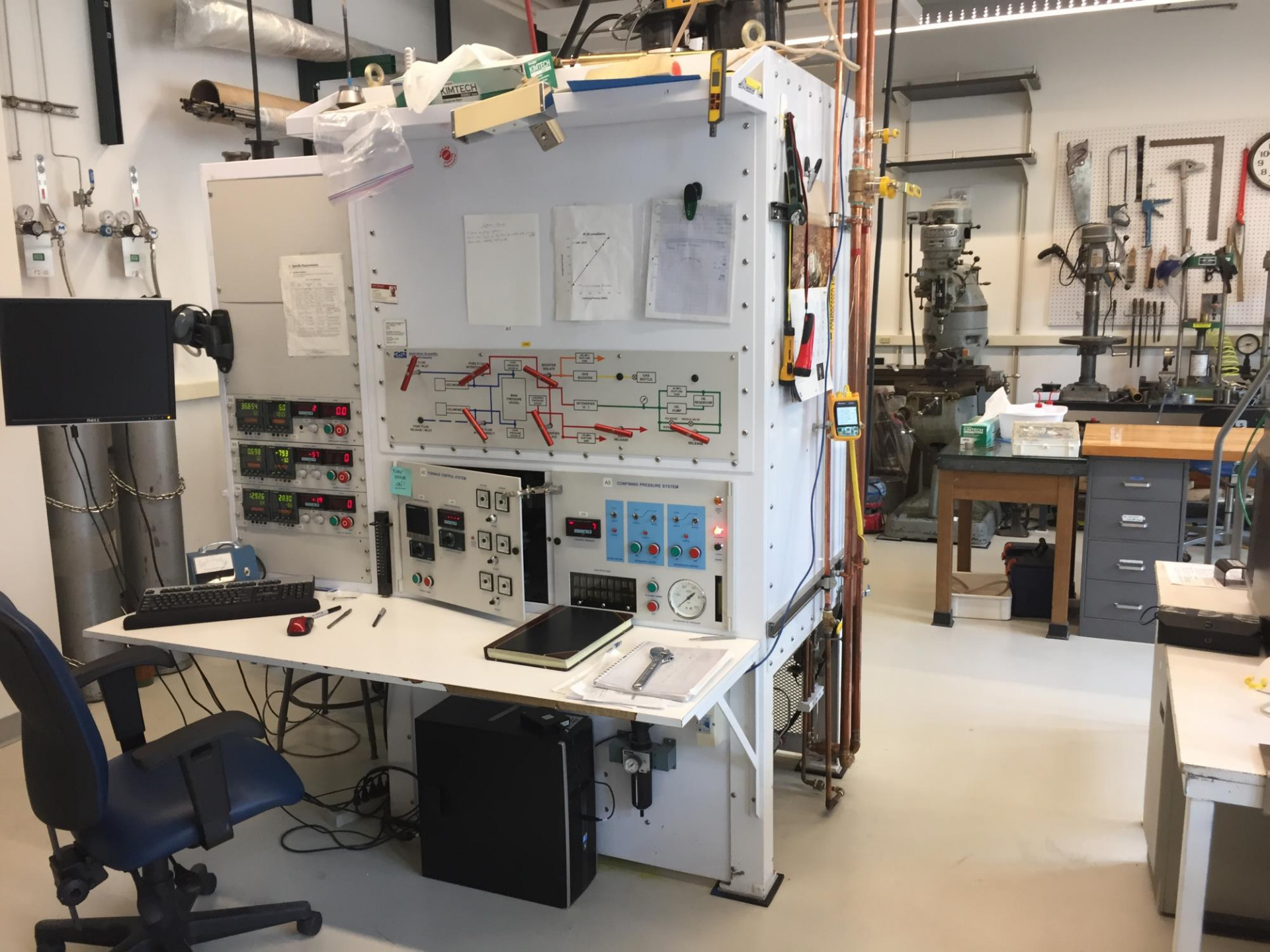
High-resolution gas-medium deformation apparatus



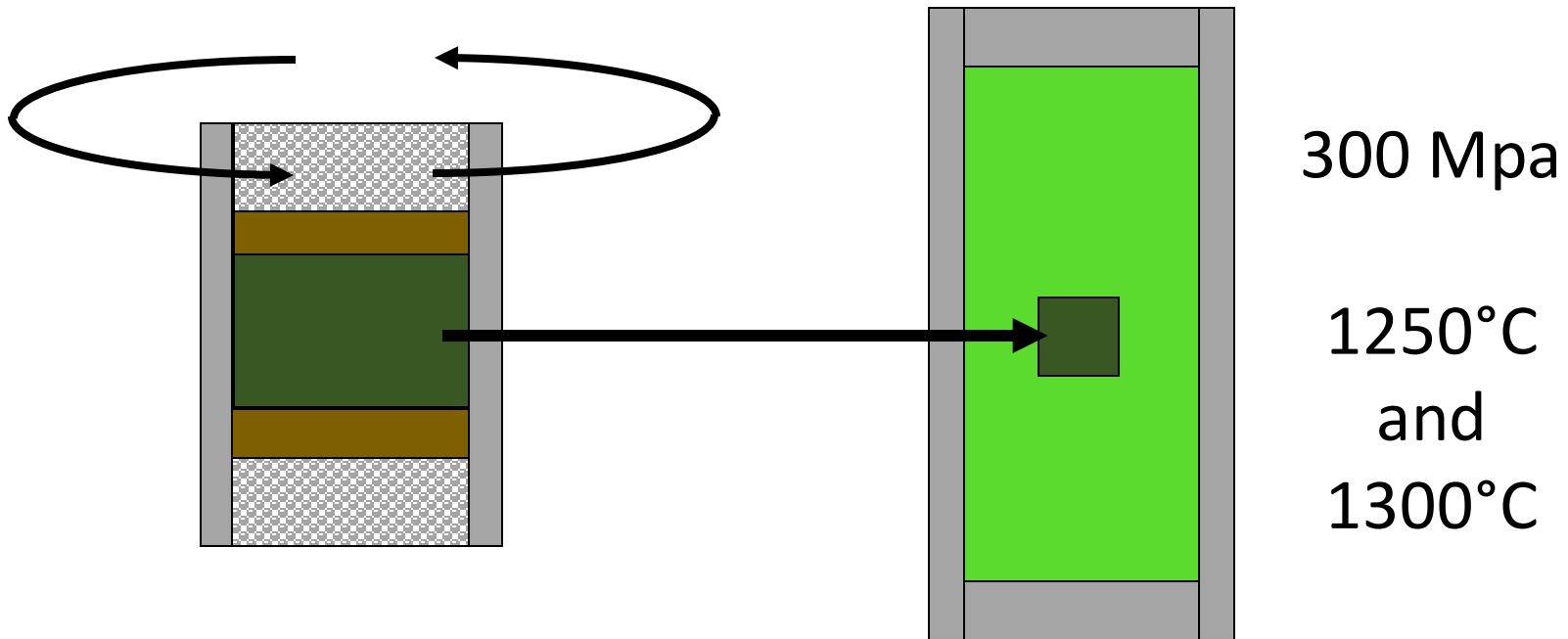
thermocouple

1250°C  
300 Mpa

- o-ring
- steel
- zirconia
- alumina
- nickel
- evacuated HP olivine
- coarse-grained natural dunite



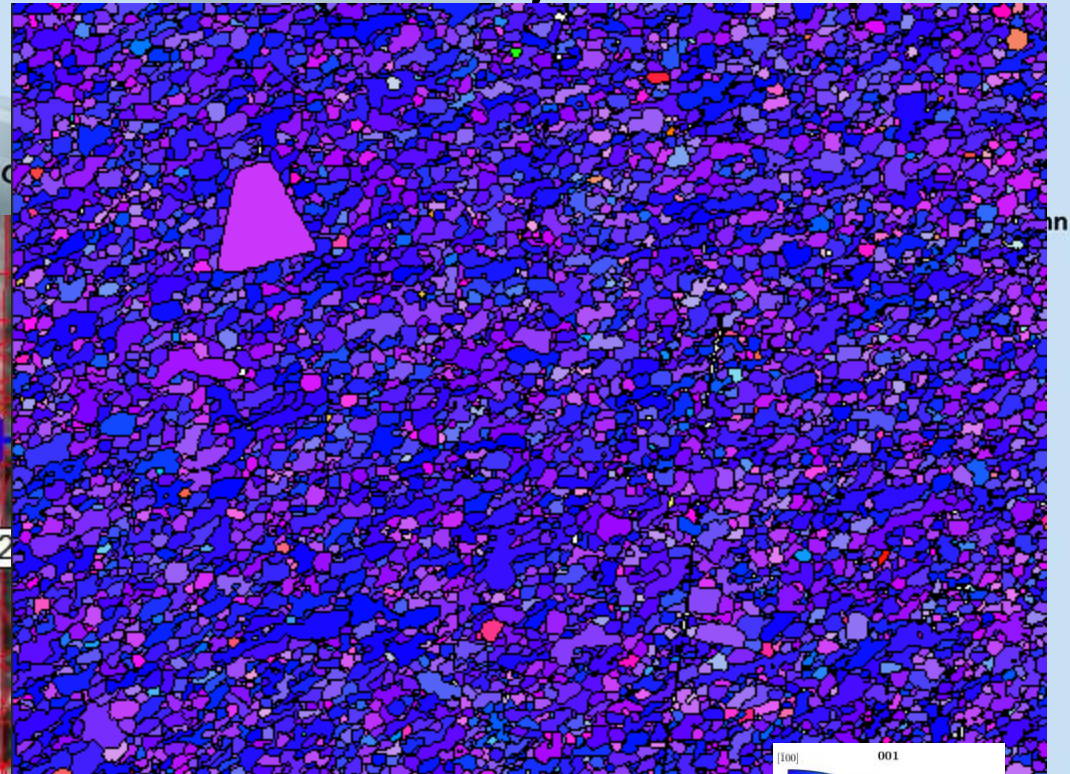
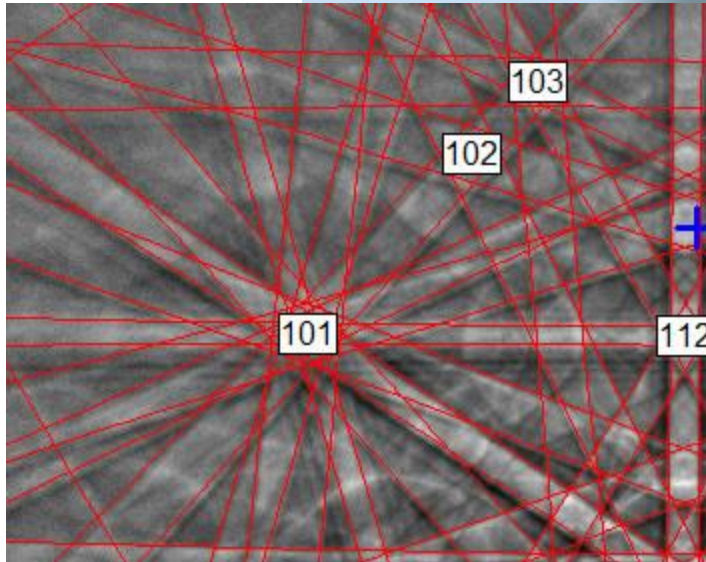
# Static annealing of deformed specimens at 300 MPa



Torsional high-strain  
deformation  
experiment

- nickel
- San Carlos olivine powder
- porous alumina
- coarse-grained natural dunite
- evacuated HP San Carlos olivine

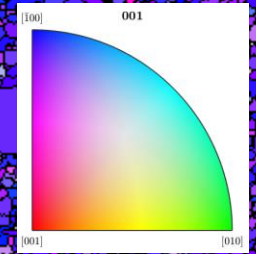
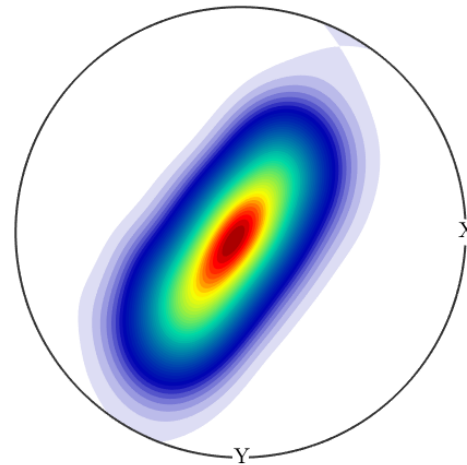
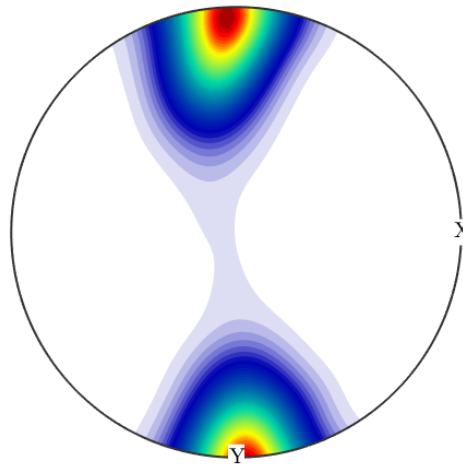
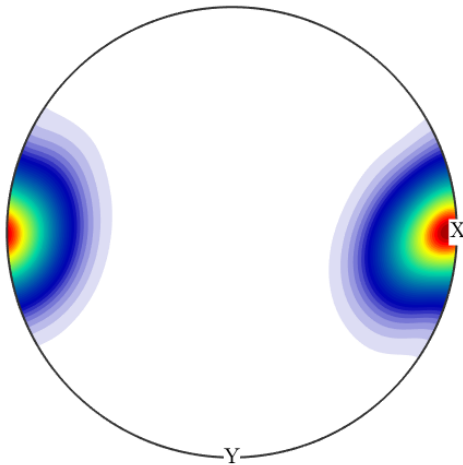
# Overview of EBSD analysis



(100)

(010)

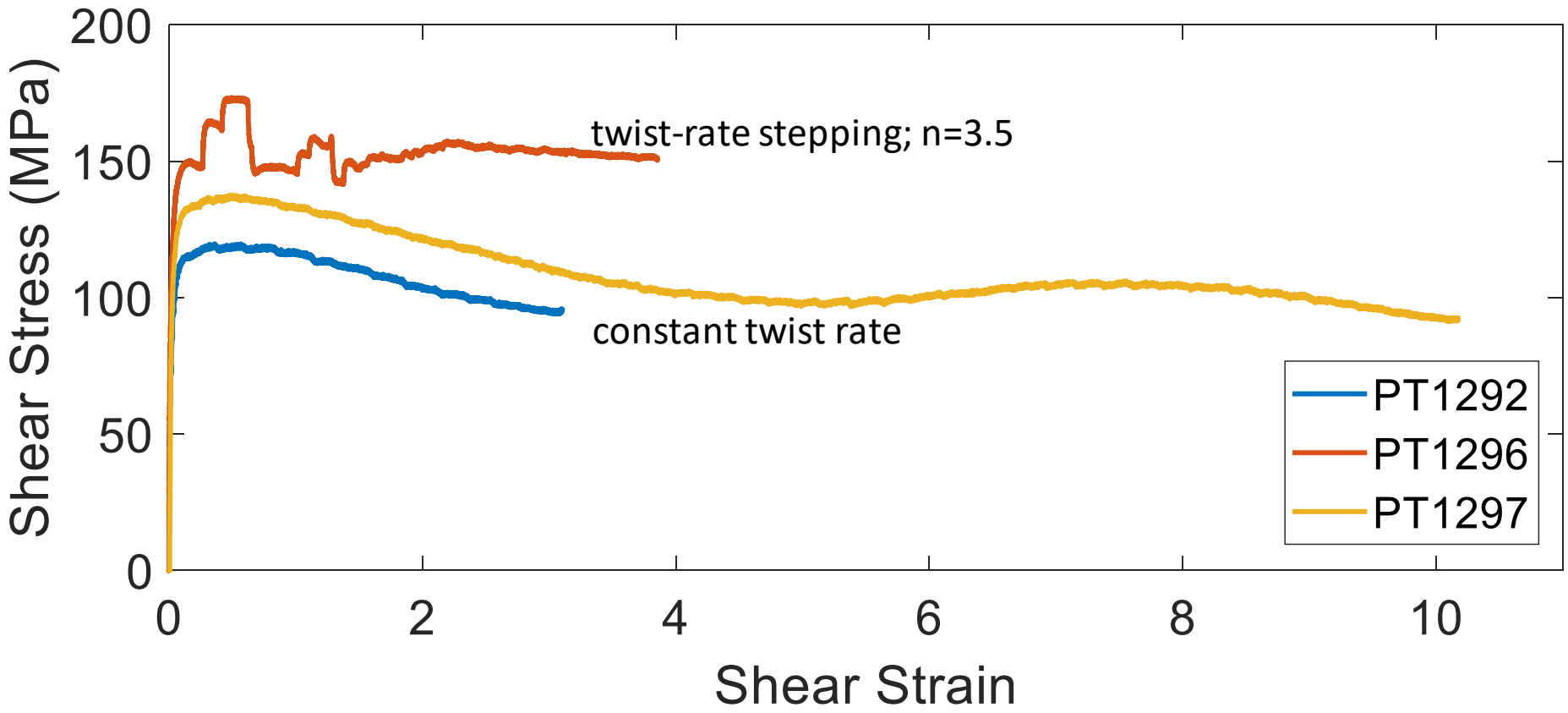
(001)



Controller

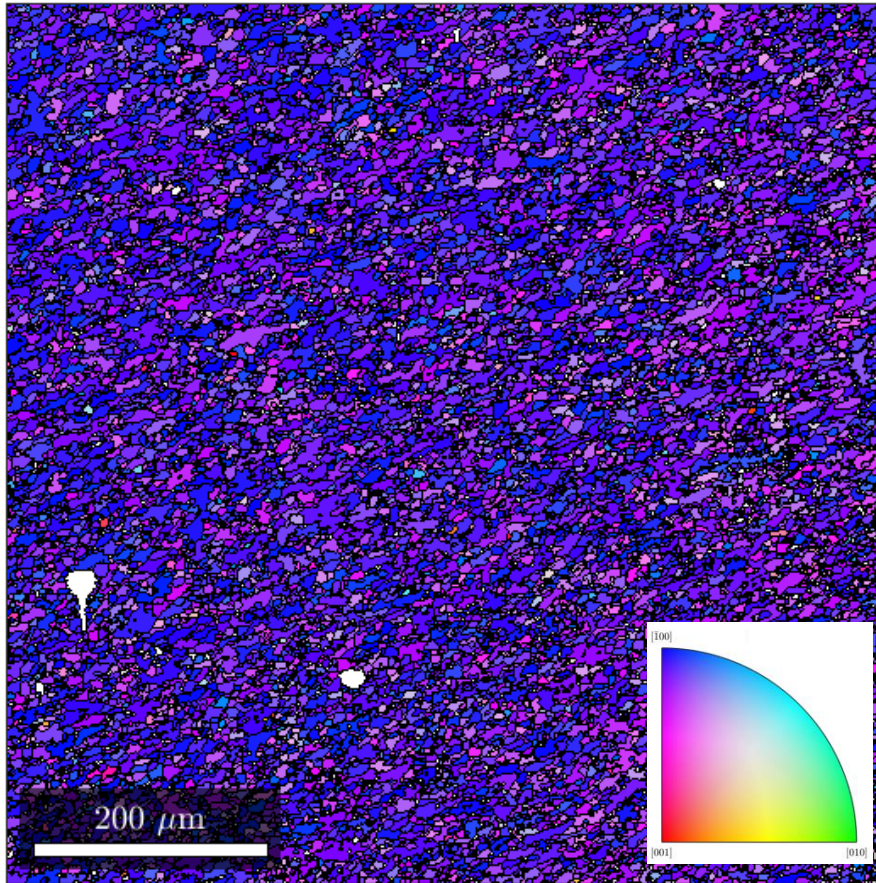
# Mechanical data from high-strain torsion experiments

Stress and strain calculated at edge of specimen

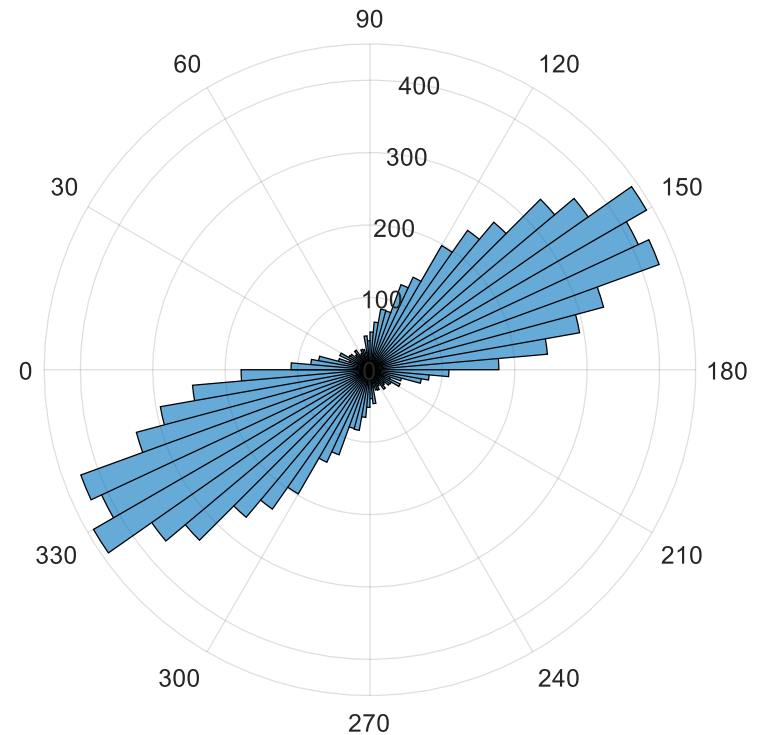


# Tangential section of high-strain torsion samples

## Inverse Pole Figure False Color Map

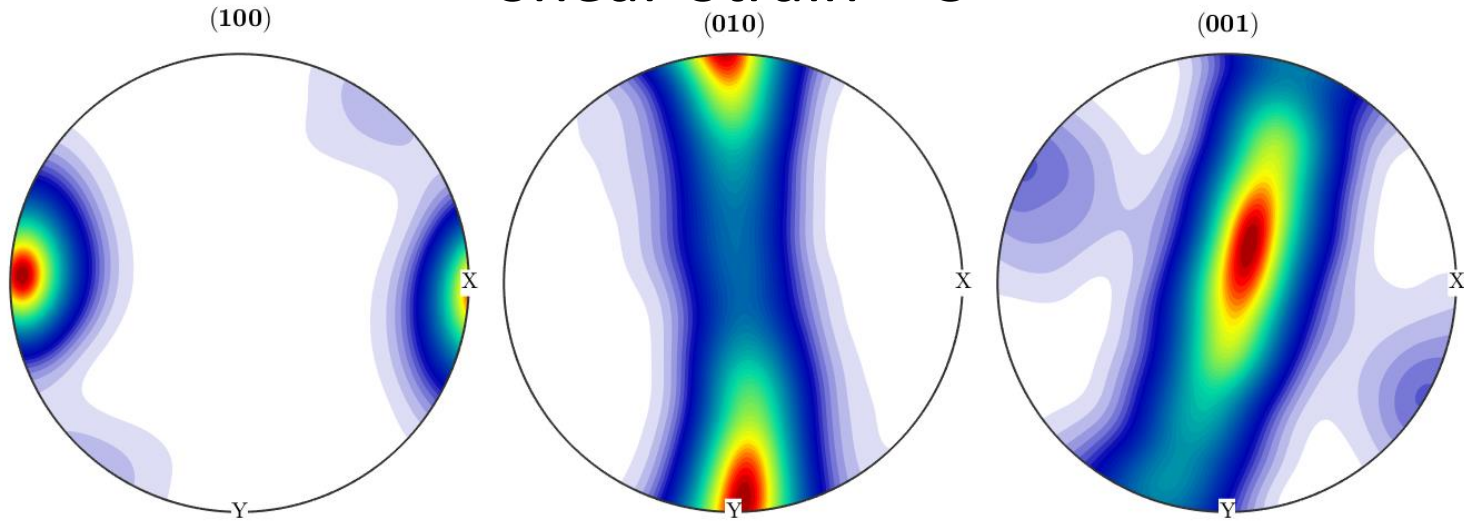


## Grain Shape Orientation

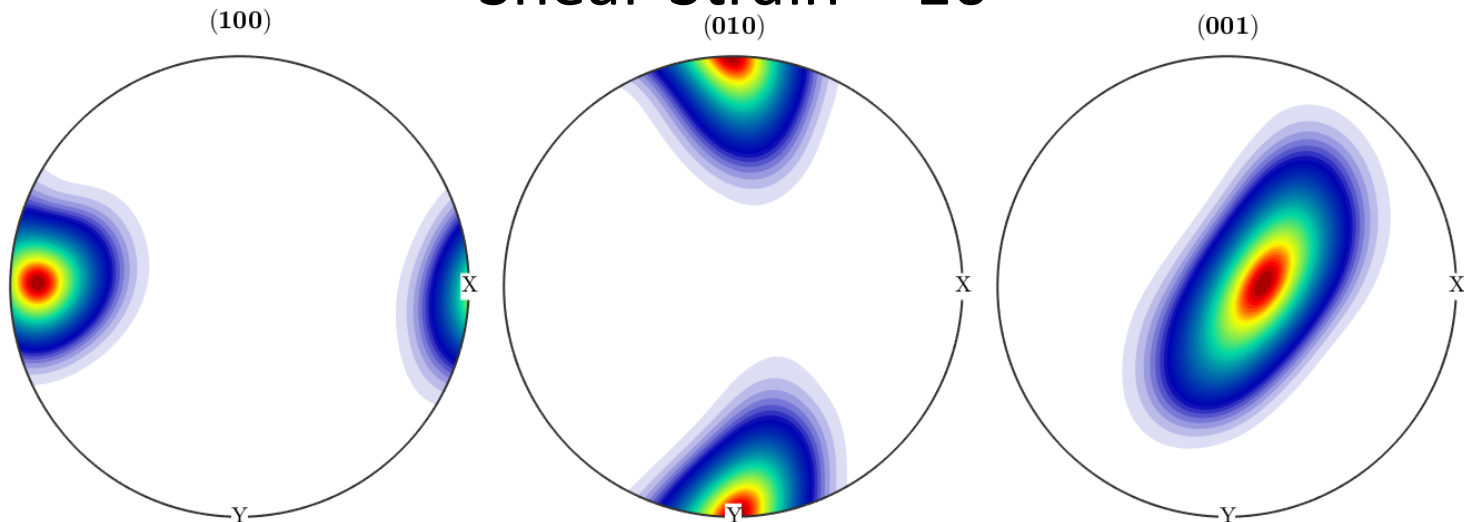


# CPO of samples deformed in torsion to high-strain

Shear Strain = 3



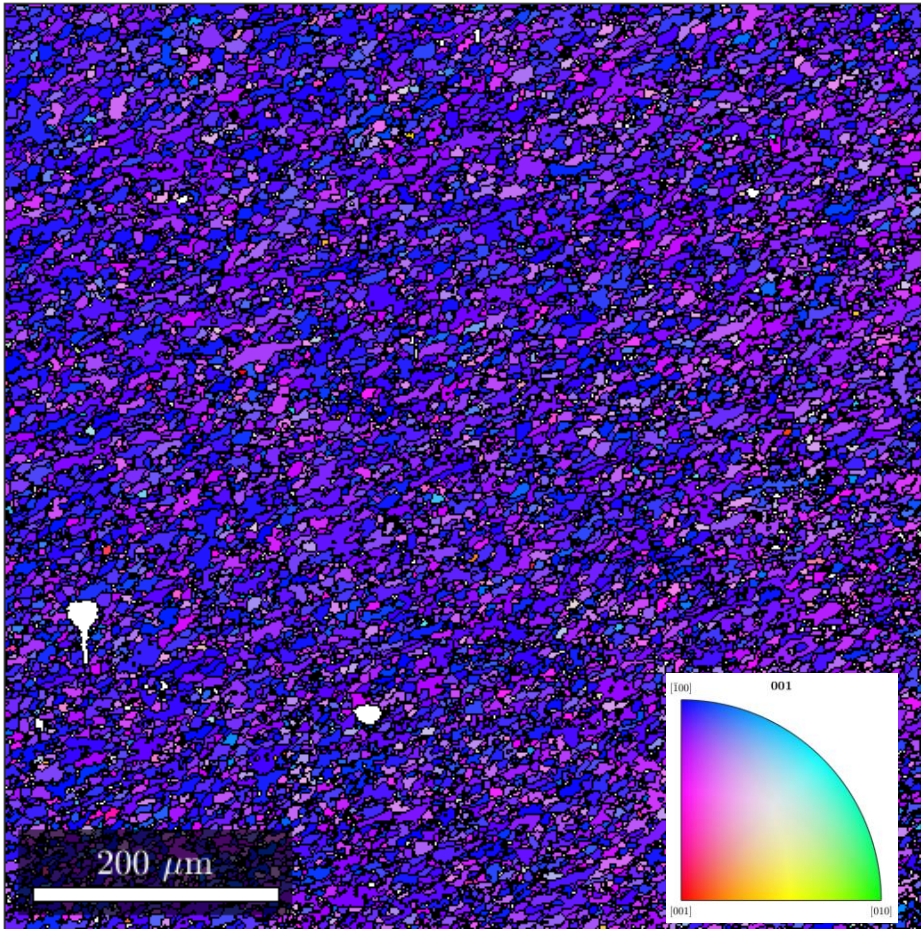
Shear Strain = 10



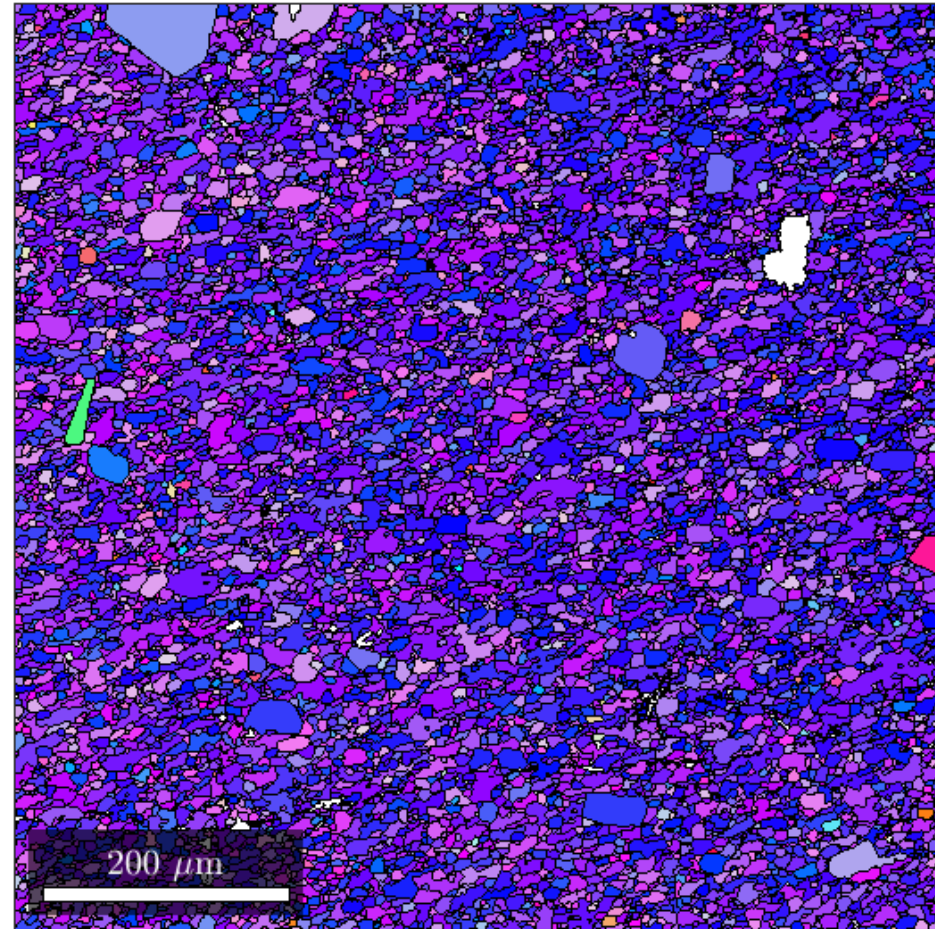


# Microstructures of annealed tangential sections

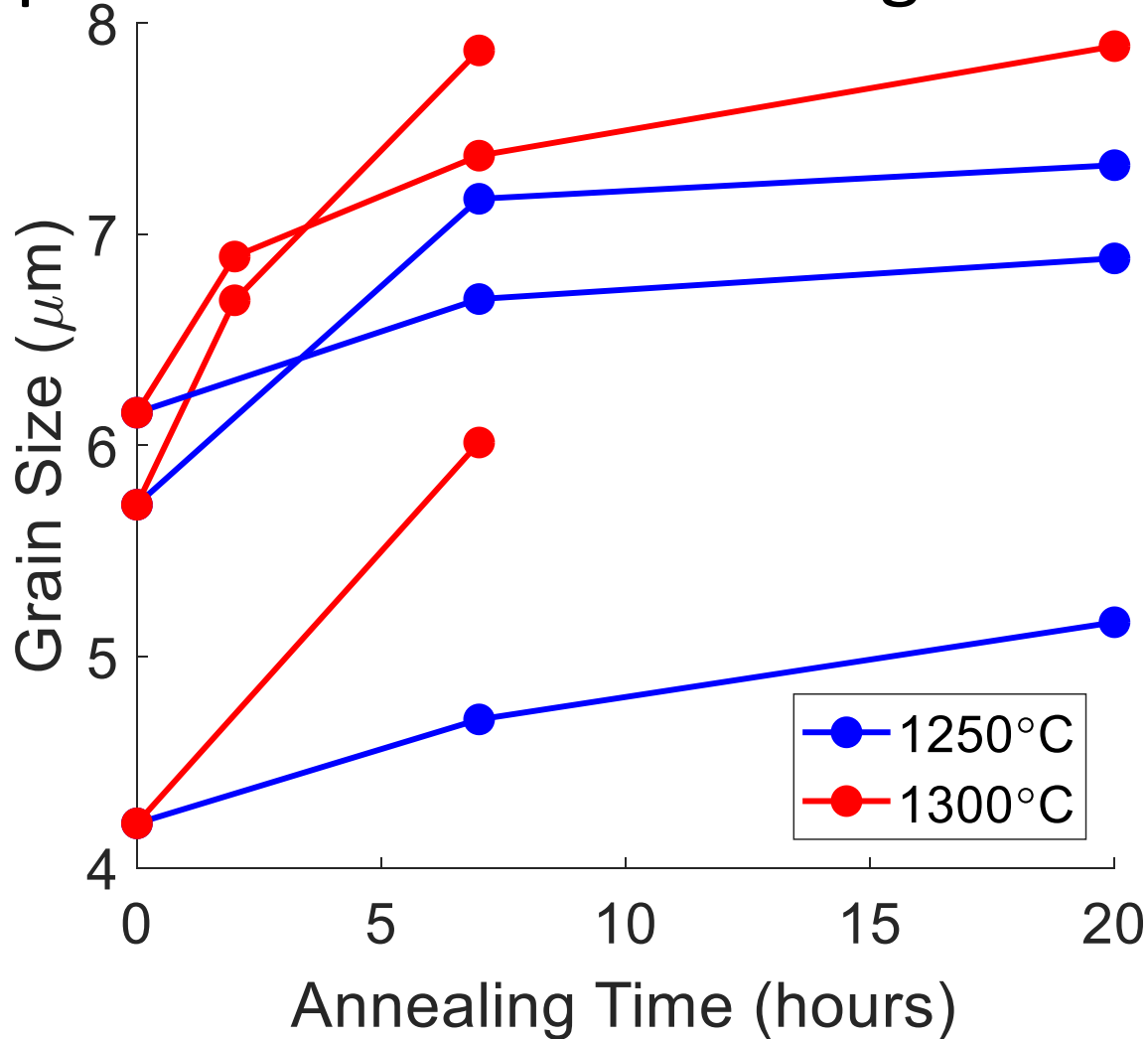
After Deformation



Annealed at 1300°C, 300 Mpa, 7hrs

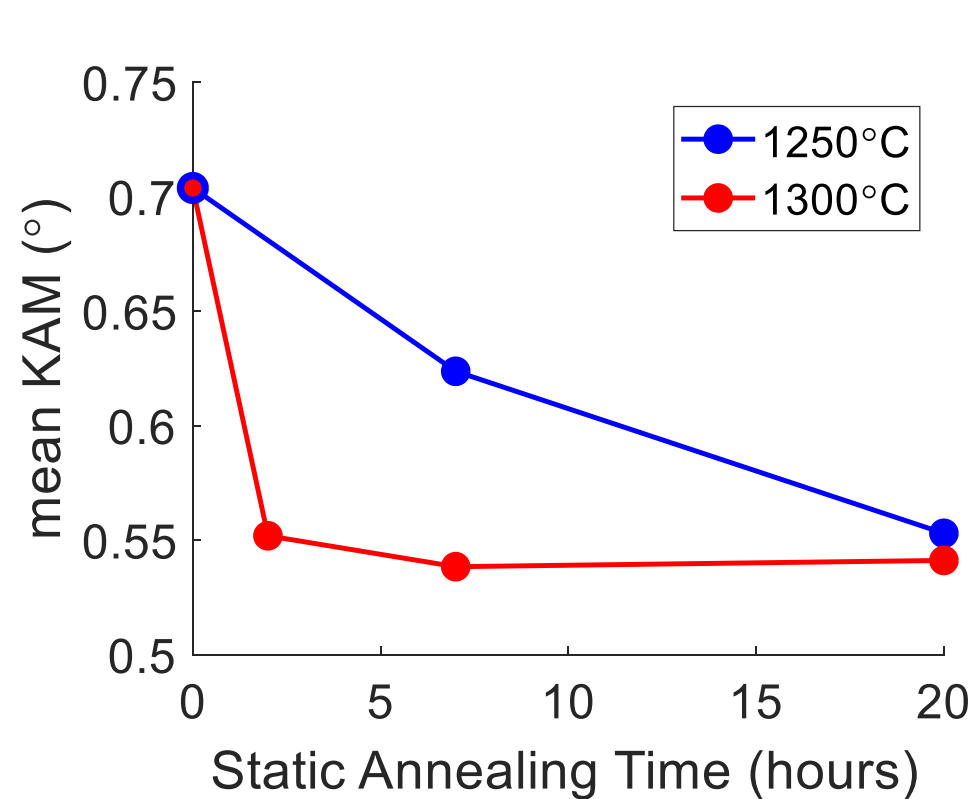
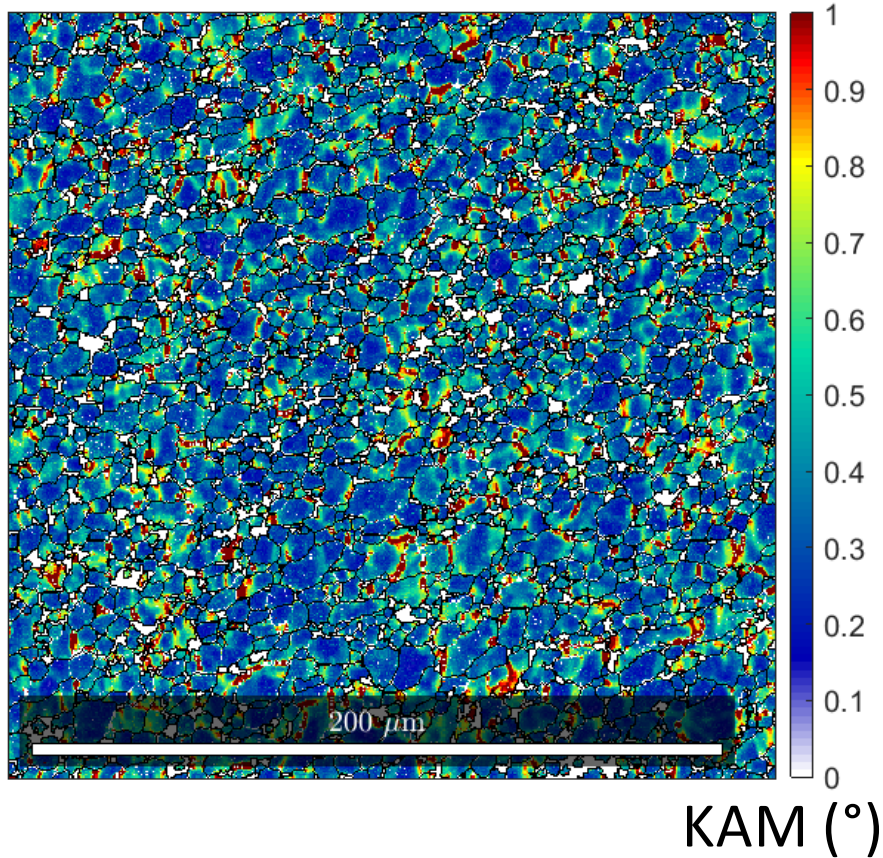


# Grain growth in tangential sections of specimens deformed to high shear strain



Normal Grain Growth Law:  $d_{final}^m - d_{initial}^m = kt$ ;  $k = k_o \exp\left(\frac{-Q}{RT}\right)$

# Intragranular Crystallographic Distortion: Kernel Angular Misorientation



# Overview

- Evacuated hot-pressing reduces contamination filled porosity to the point that one can easily read through a 1-mm thick slice.
- Grain boundary mobility is enhanced in evacuated hot-presses relative to conventional hot-presses of the same powder.
- High-strain torsion of evacuated hot-pressed olivine aggregates leads to grain-size refinement, intragranular crystallographic distortion, development of a strong shape preferred orientation (SPO), and development of a strong crystallographic preferred orientation (CPO)
- During static annealing of samples deformed to high strains, grains become progressively equiaxed and intragranular crystallographic distortion is reduced, while CPO geometry and strength remains relatively constant

Thanks to:

National Nuclear Security Administration Stewardship Science Graduate Fellowship

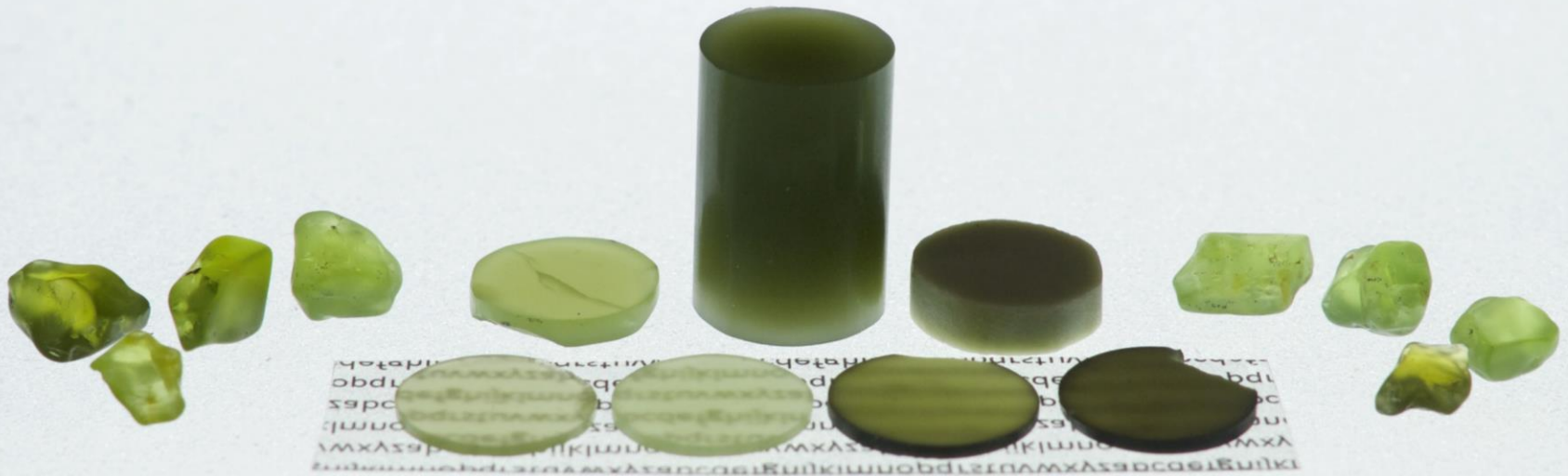
National Science Foundation

University of Minnesota Characterization Facility

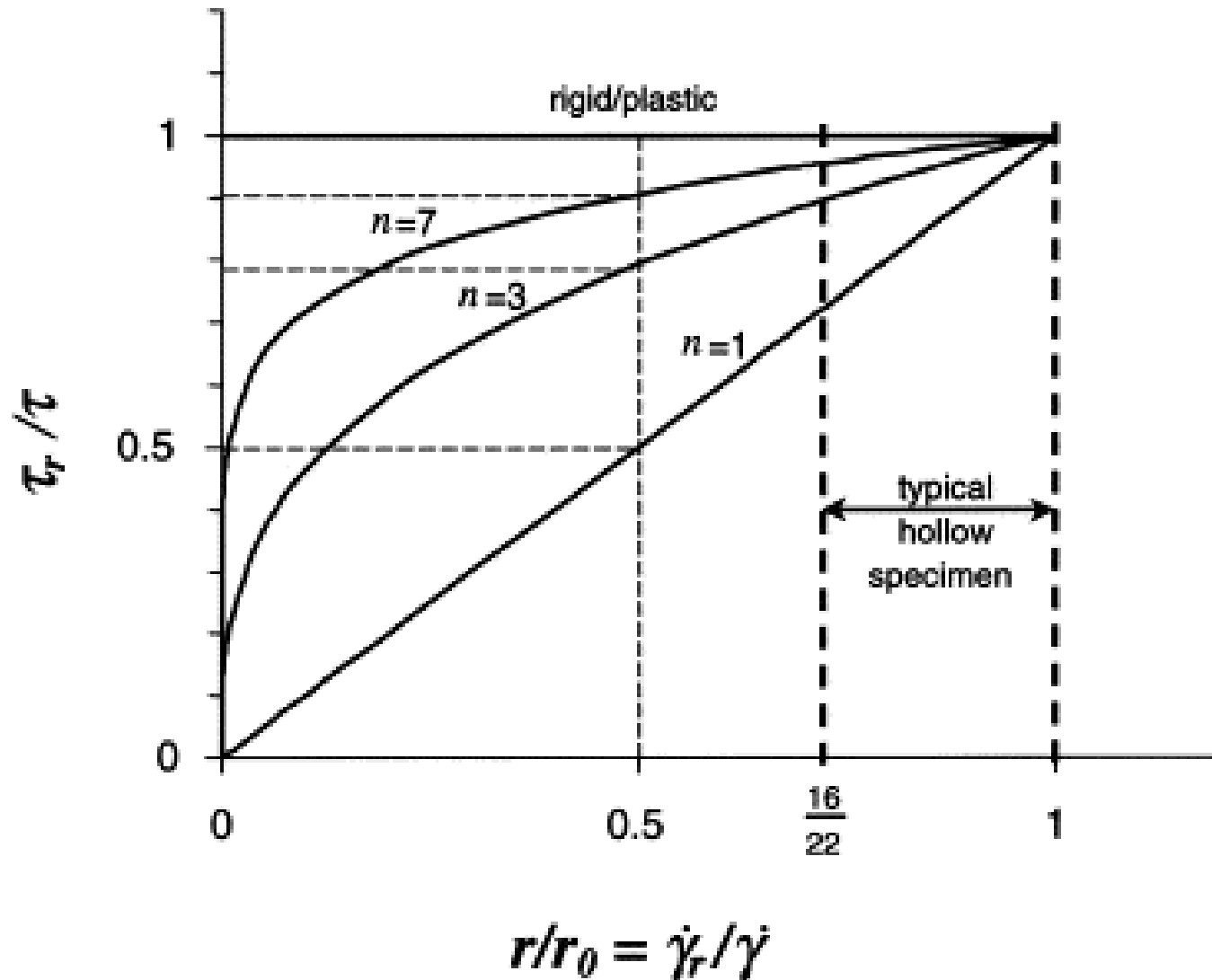
Minnesota Nano Center

Members of the Kohlstedt Research Group

Kruckenberg Research Group



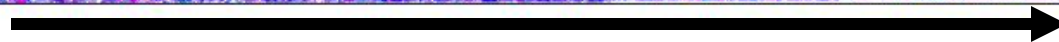
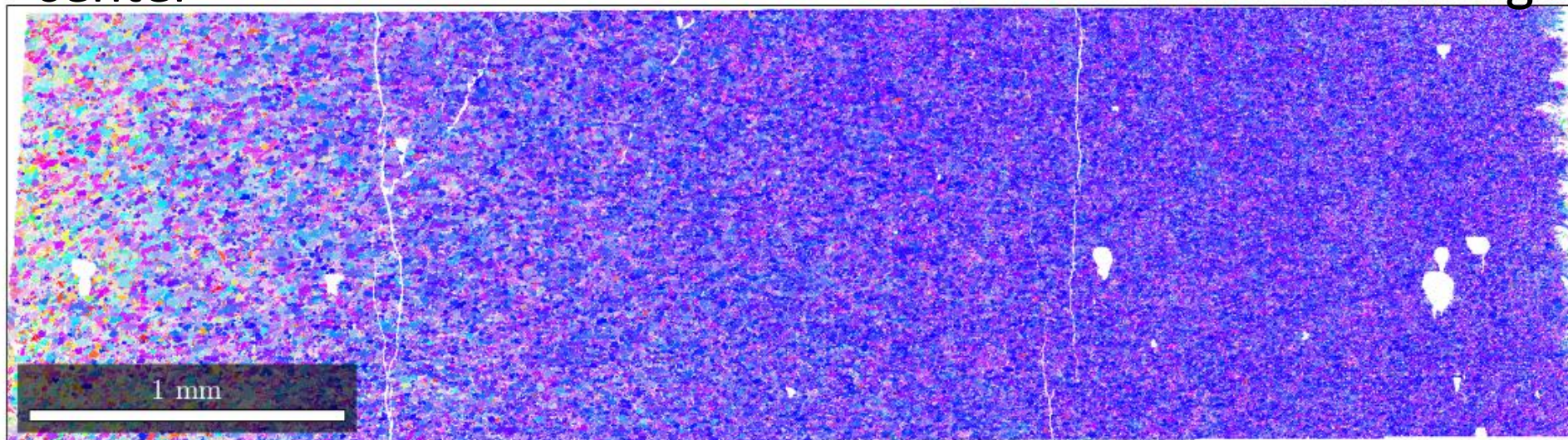
# Radial stress and strain gradient in torsional deformation



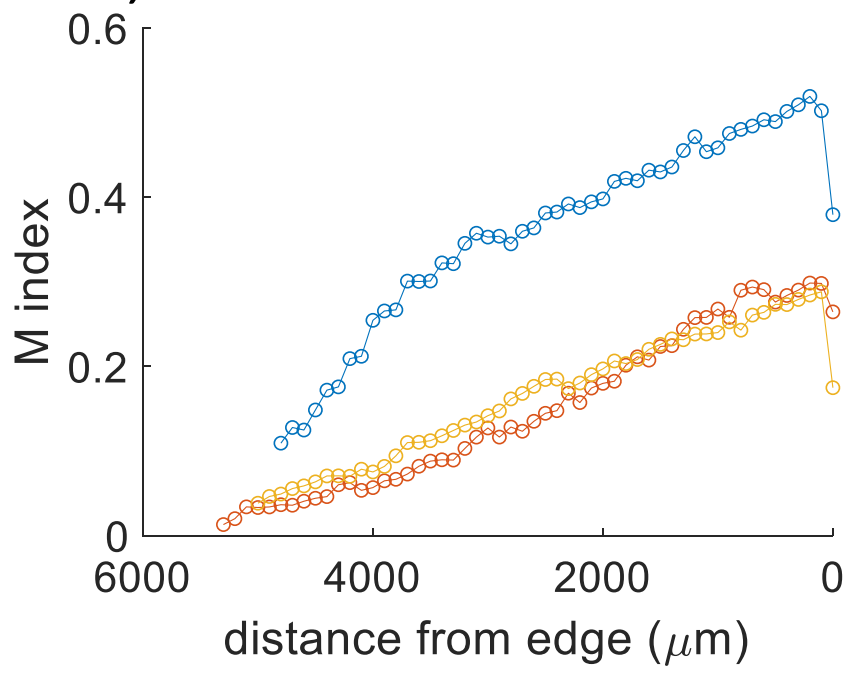
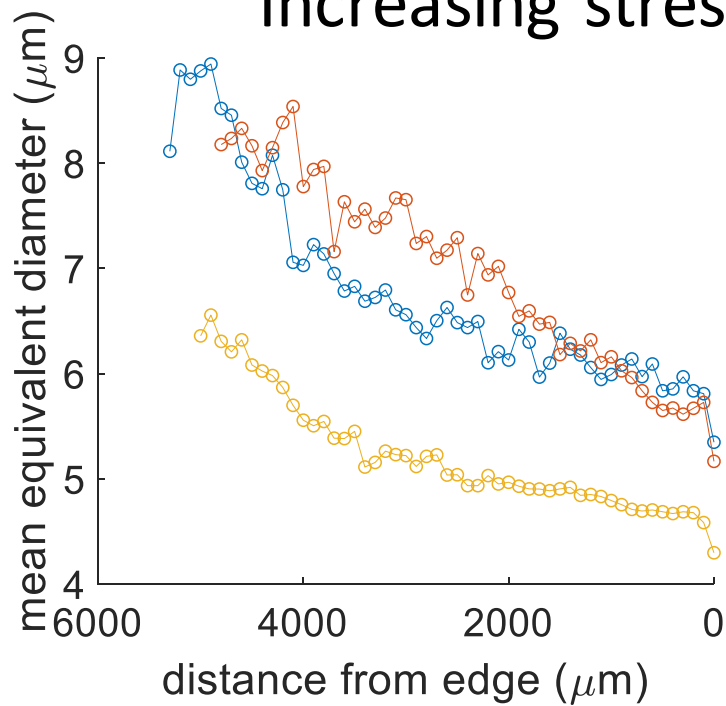
# Microstructures of Radial Sections

center

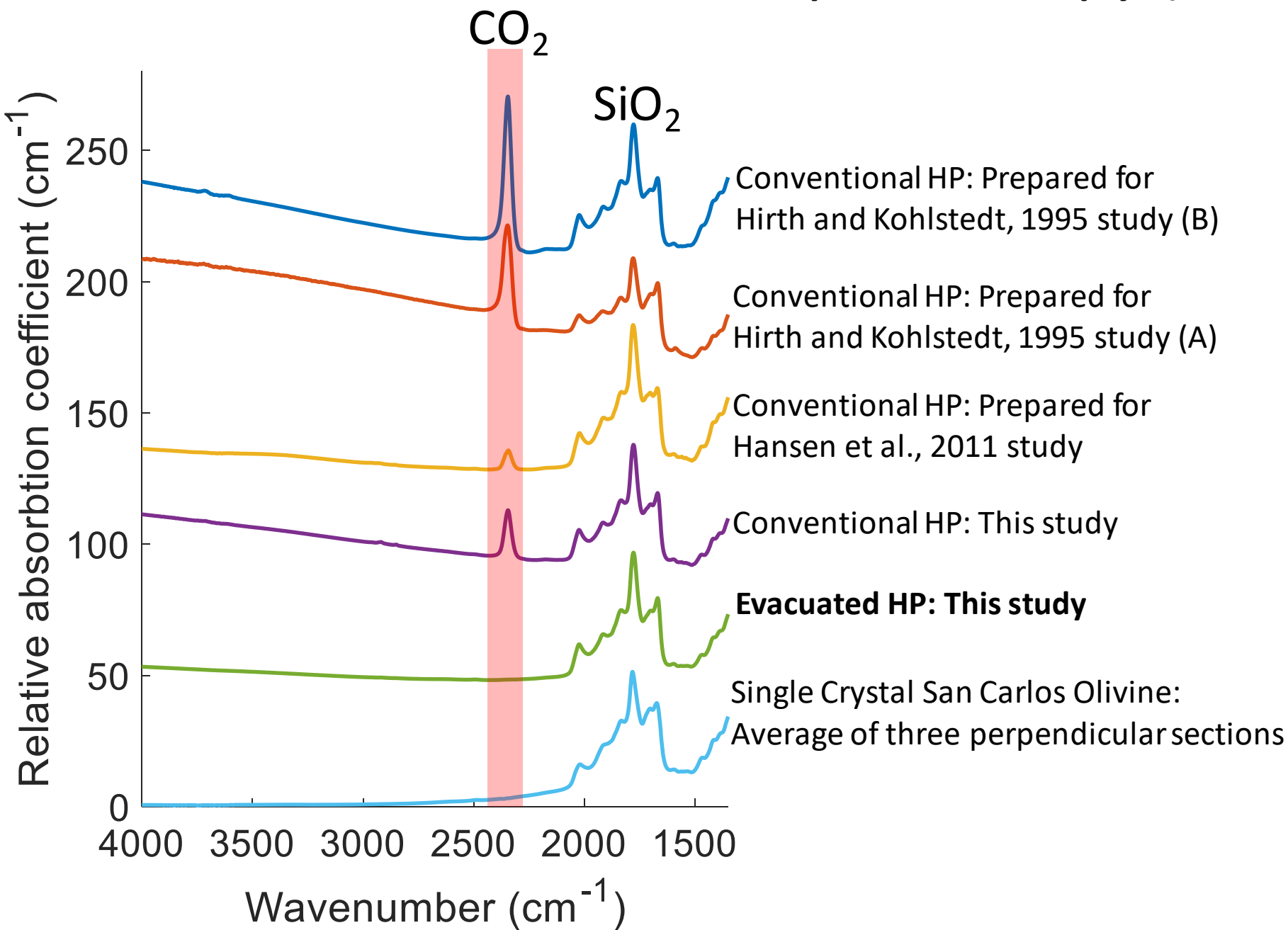
edge



Increasing stress, strain, strain-rate



# Fourier transform infrared spectroscopy (FTIR)





# Confocal Raman spectroscopy

