Dislocations in Ceramics: Opportunities and challenges in quantitative 3D imaging

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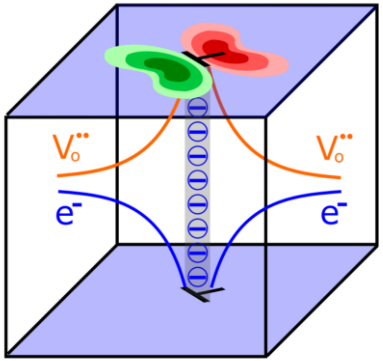
Dark Field X-ray Microscopy at the ESRF-EBS, May 6th 2021



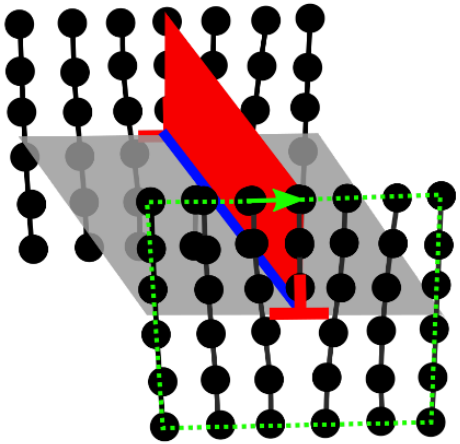
Content:

- Overview of dislocations in ceramics
- ID06 data set
- Questions in plasticity of ceramics

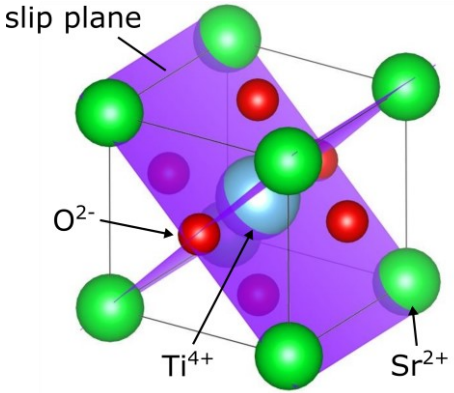
Photograph by Viviane Seidel

Introduction:
Fundamentals

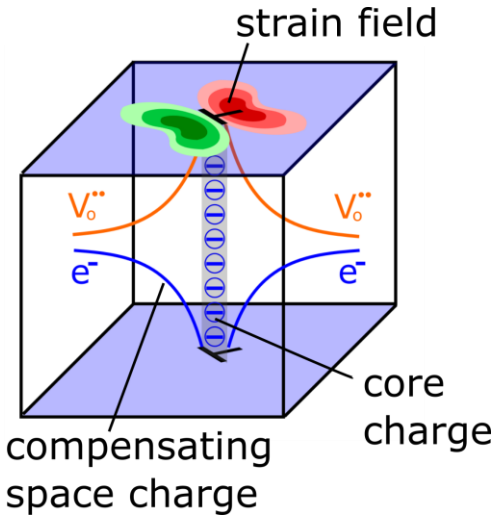
Dislocations are well known for metals



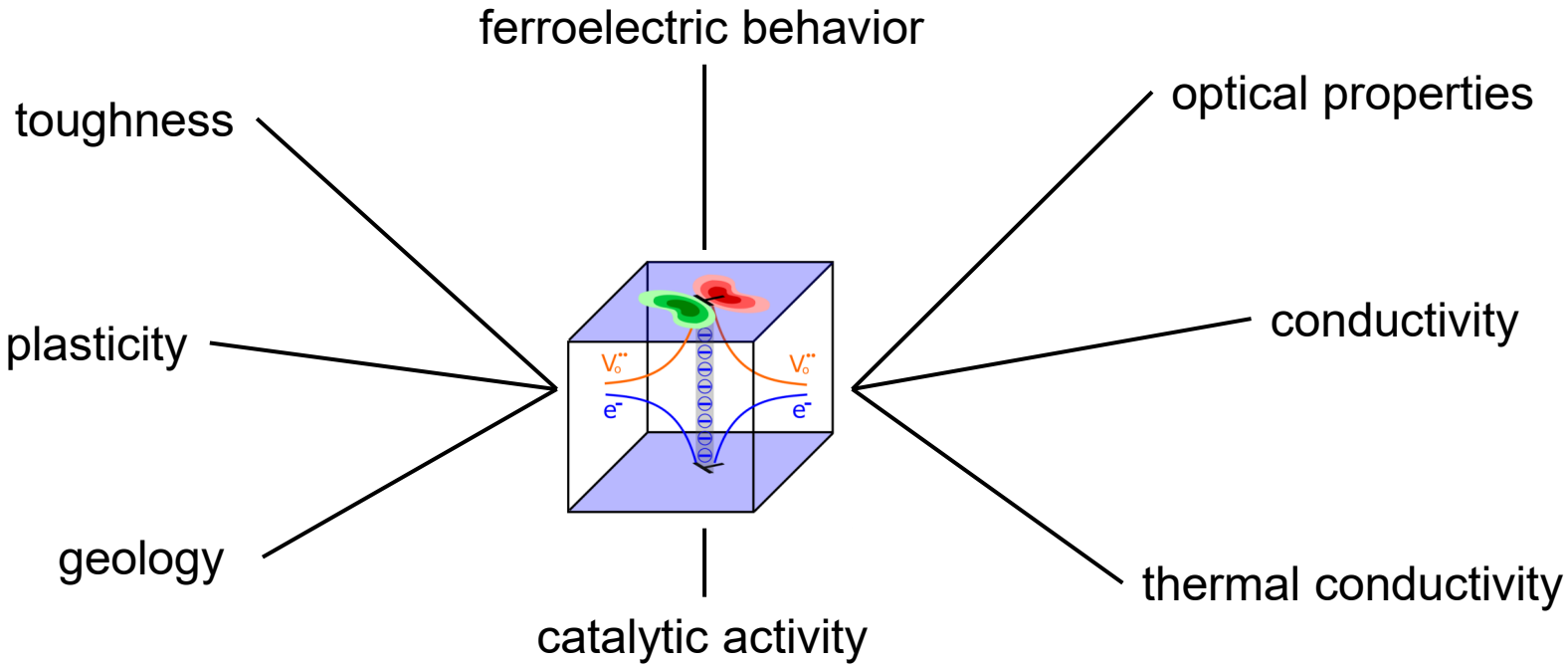
Dislocation mechanics is much less understood for ceramics



Dislocations can strongly effect functional properties



Why dislocations in ceramics?



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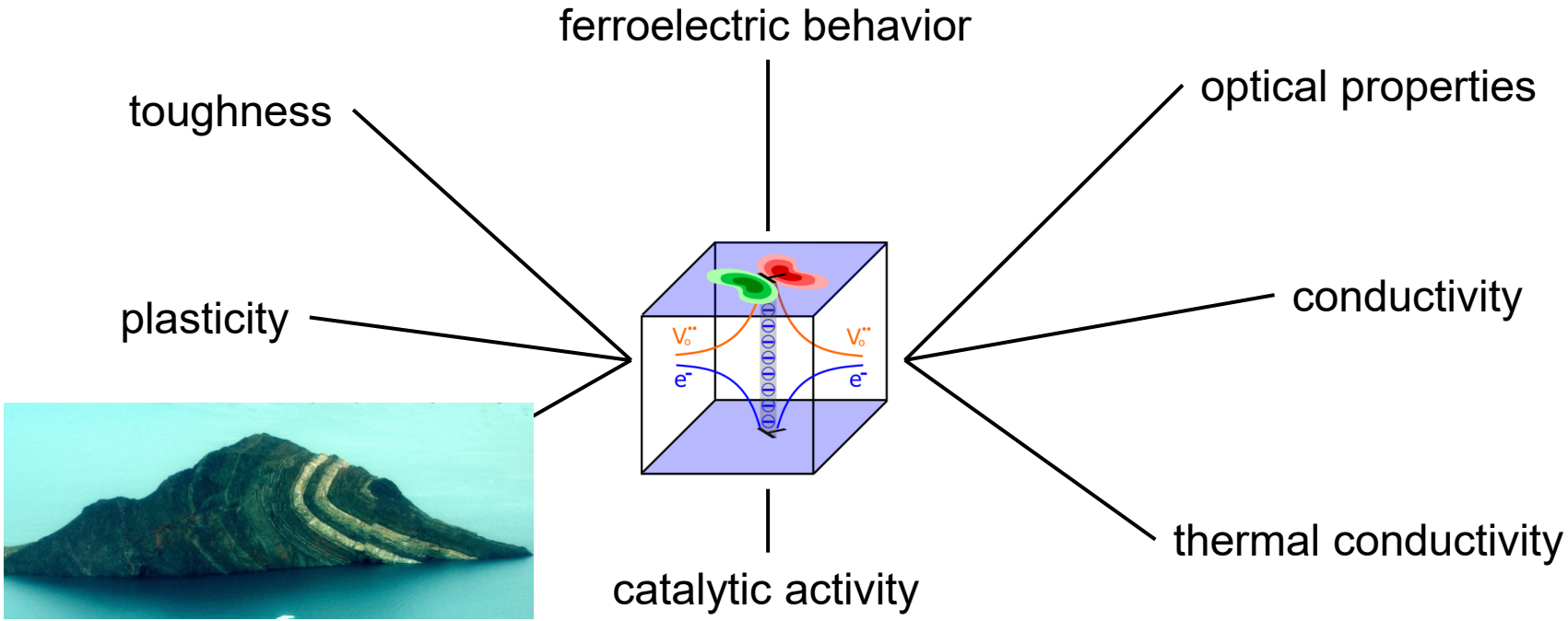
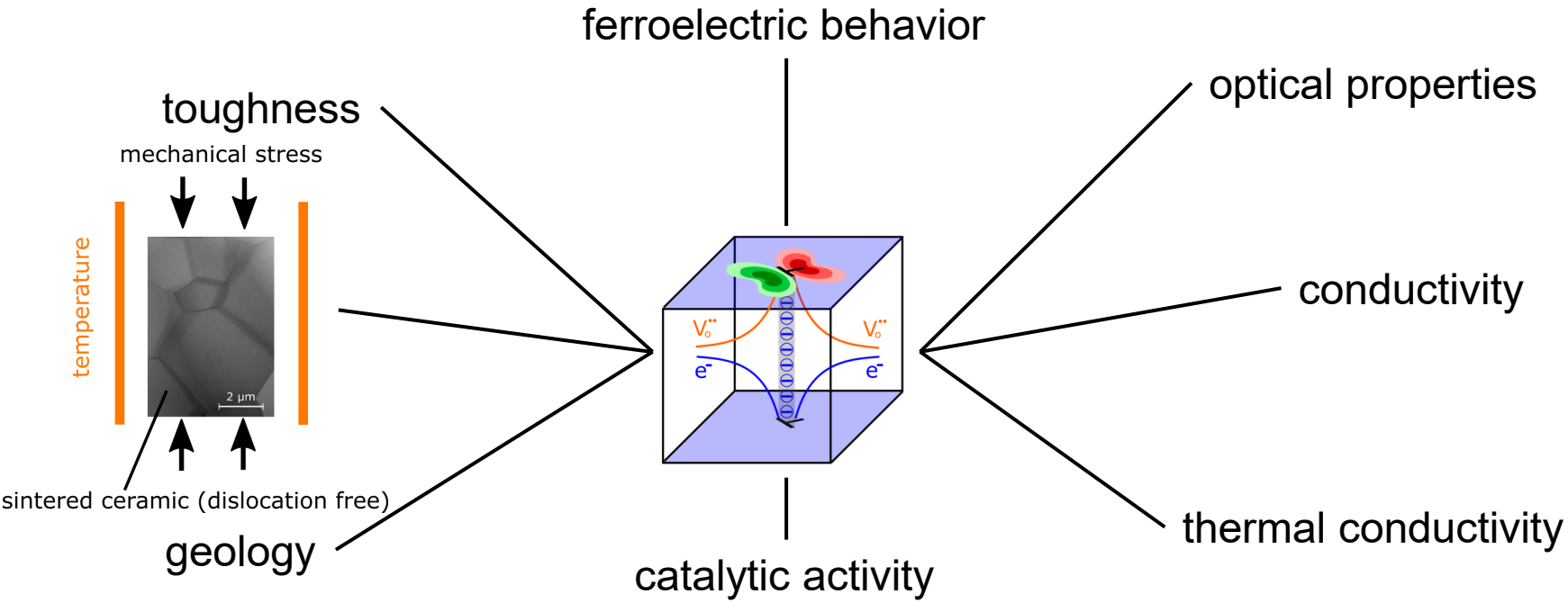
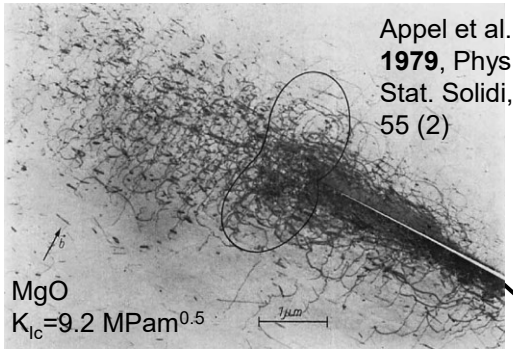


Foto by Håvard Berland

Why dislocations in ceramics?



Why dislocations in ceramics?

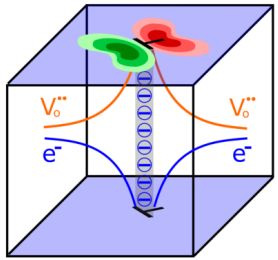


ferroelectric behavior

optical properties

plasticity

conductivity



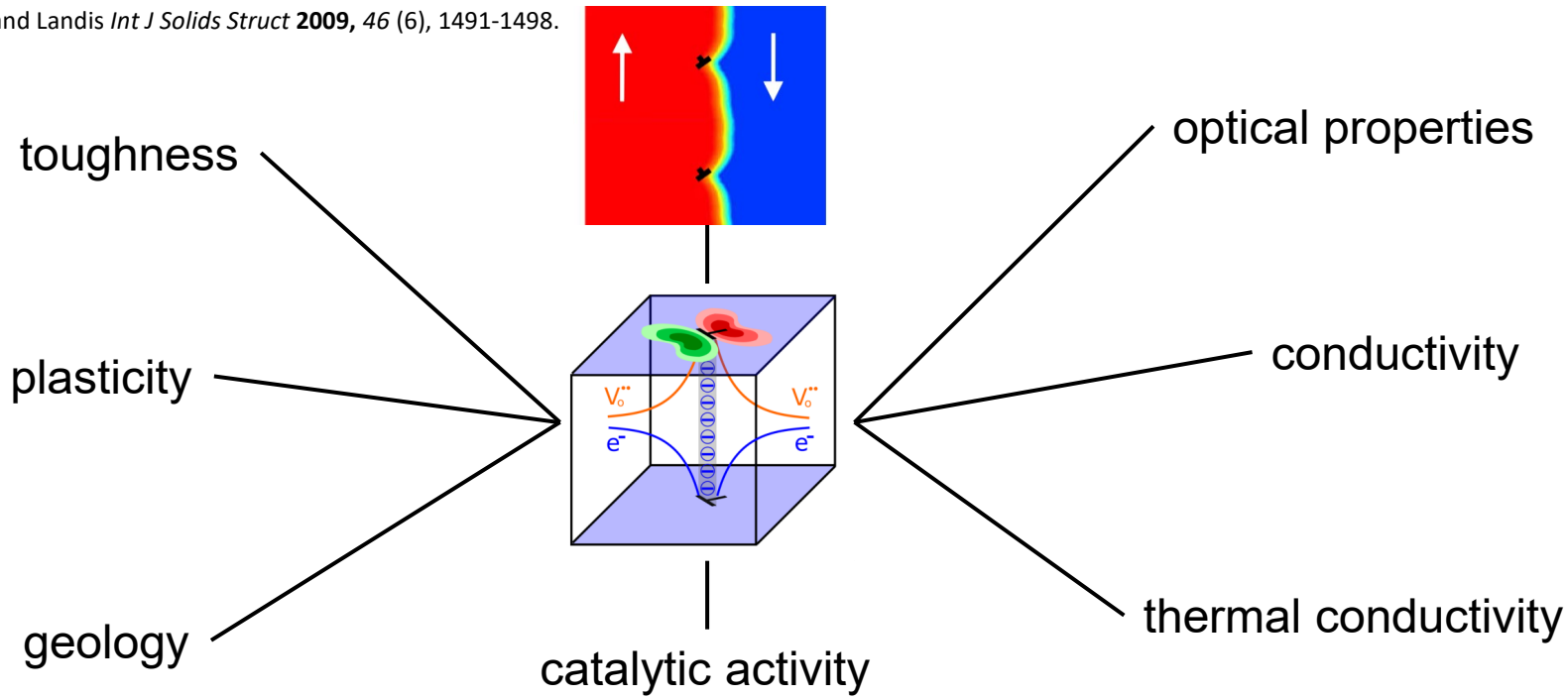
geology

catalytic activity

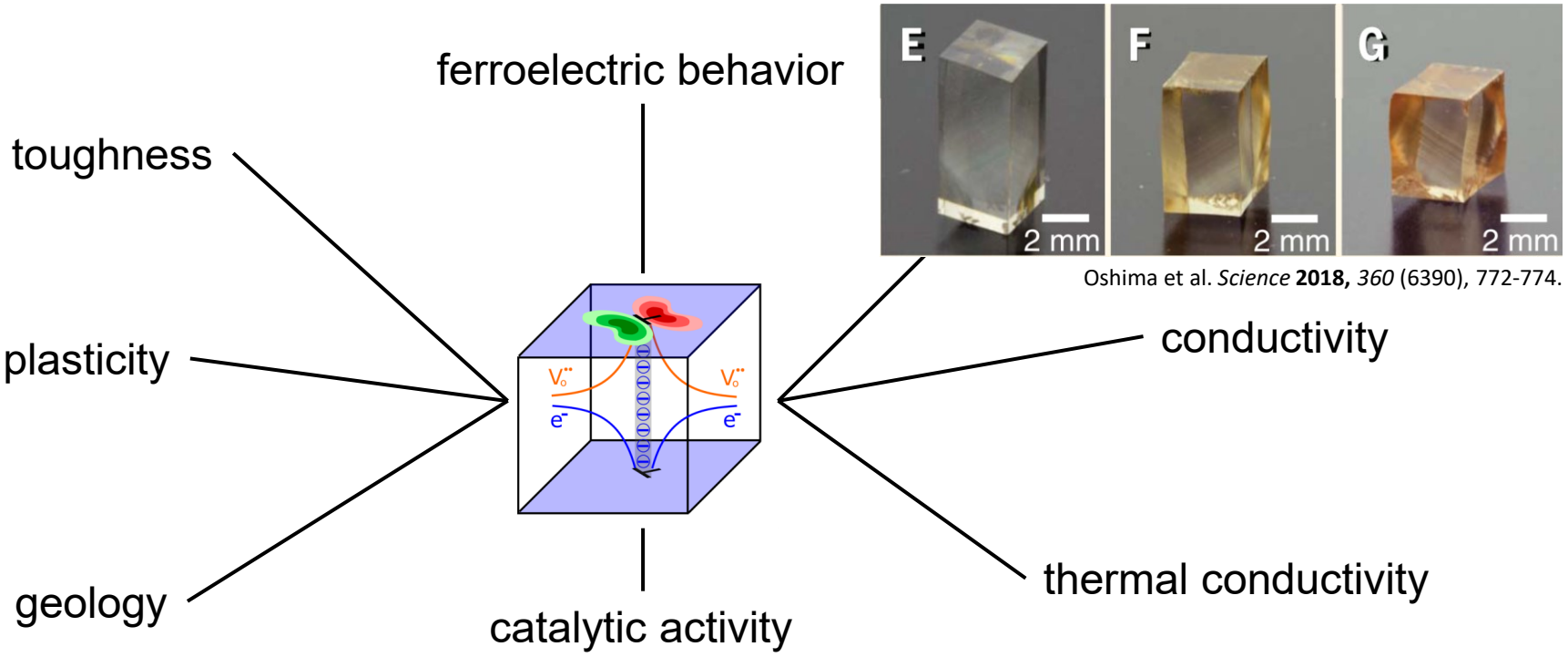
thermal conductivity

Why dislocations in ceramics?

Kontsos and Landis *Int J Solids Struct* **2009,** *46* (6), 1491-1498.

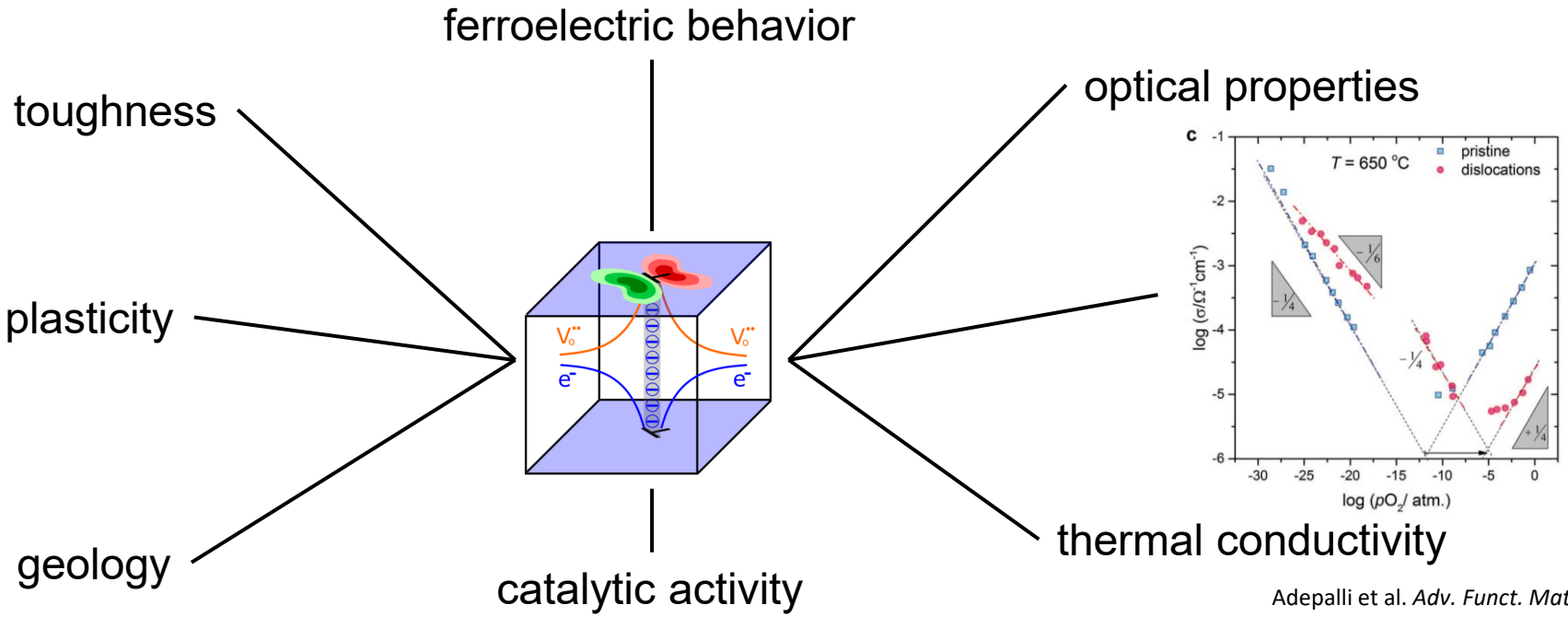


Why dislocations in ceramics?



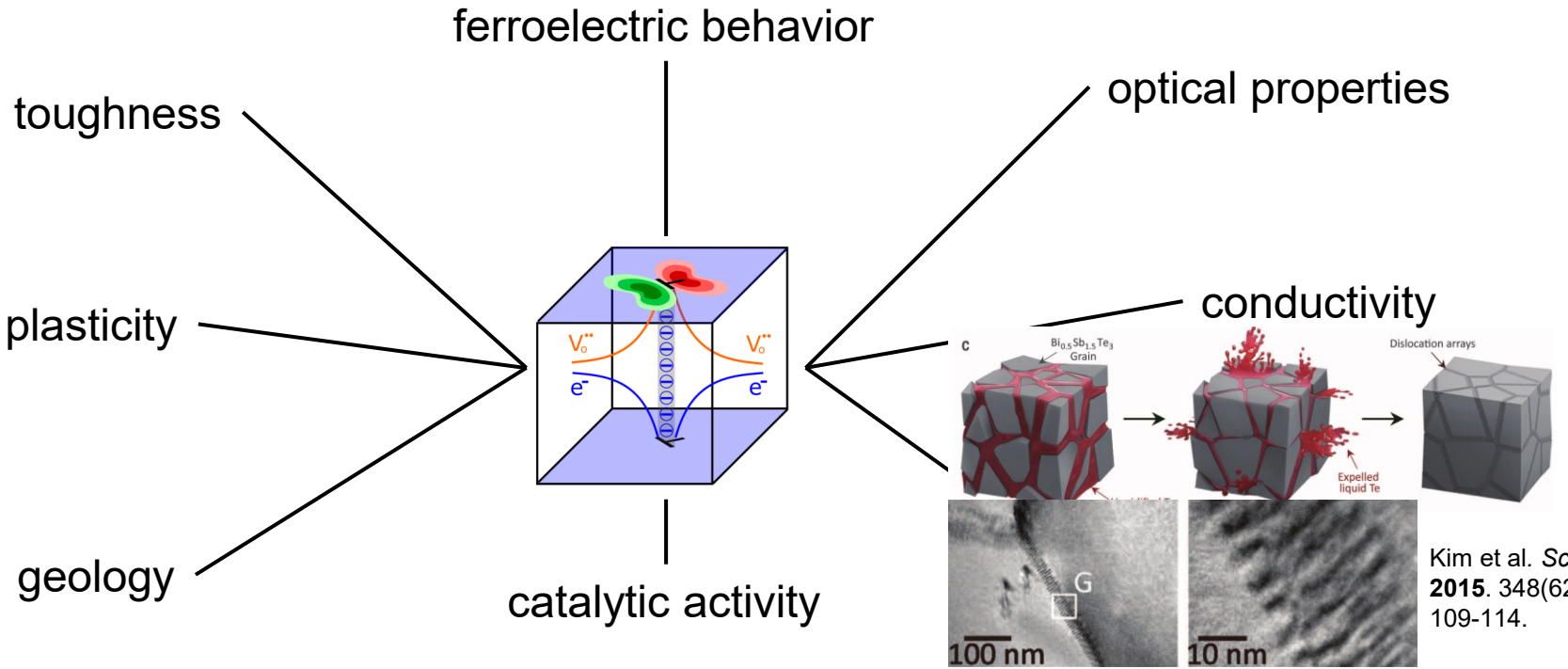
Oshima et al. *Science* **2018,** *360* (6390), 772-774.

Why dislocations in ceramics?



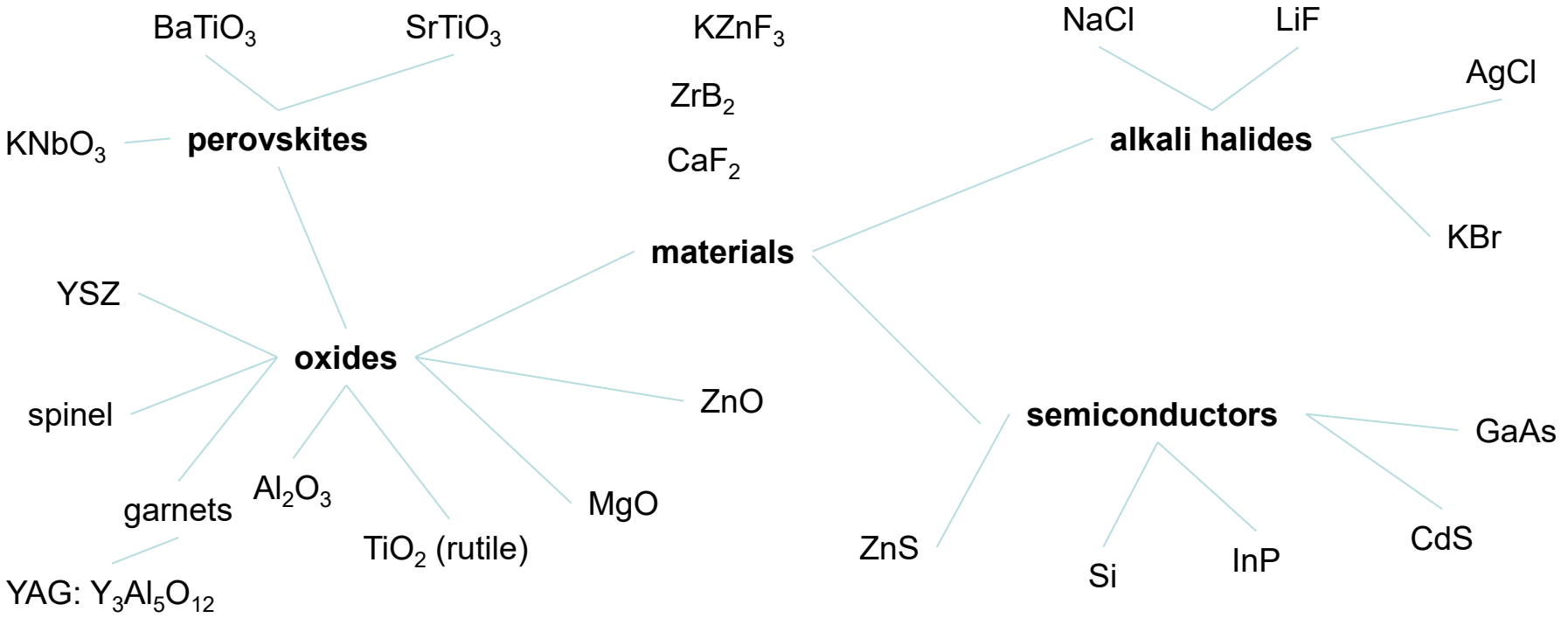
Adepalli et al. *Adv. Funct. Mater.* **2017,** *27* (22), 1700243.

Why dislocations in ceramics?

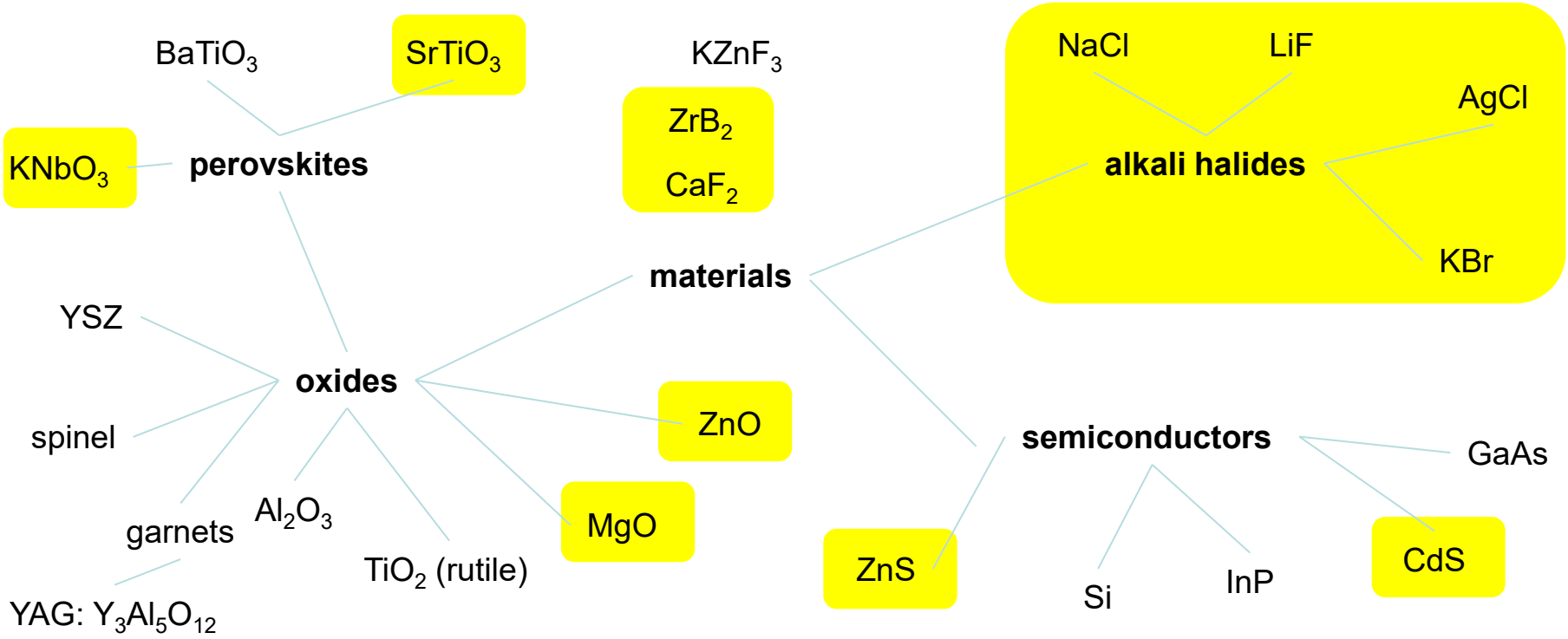


Kim et al. *Science*, **2015**. 348(6230), 109-114.

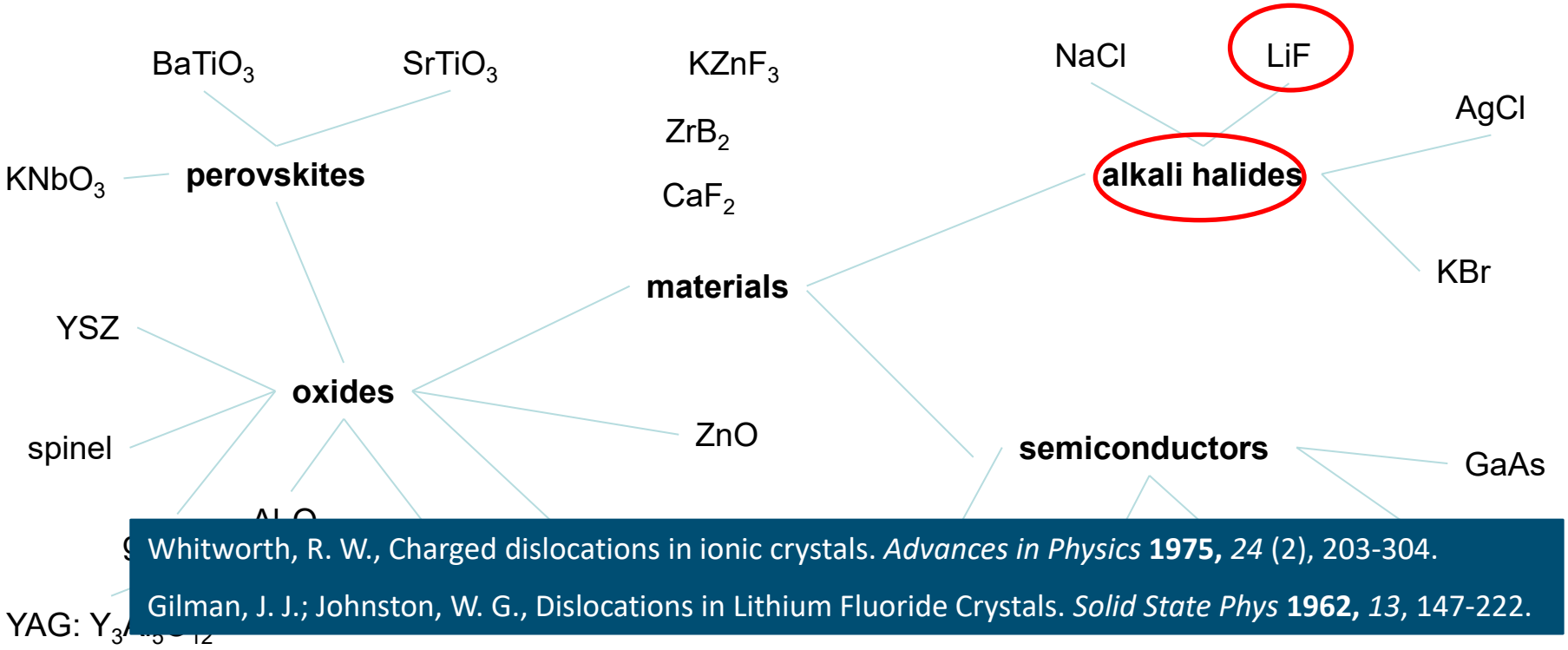
Materials – an overview



Materials with dislocation mobility at 25 °C



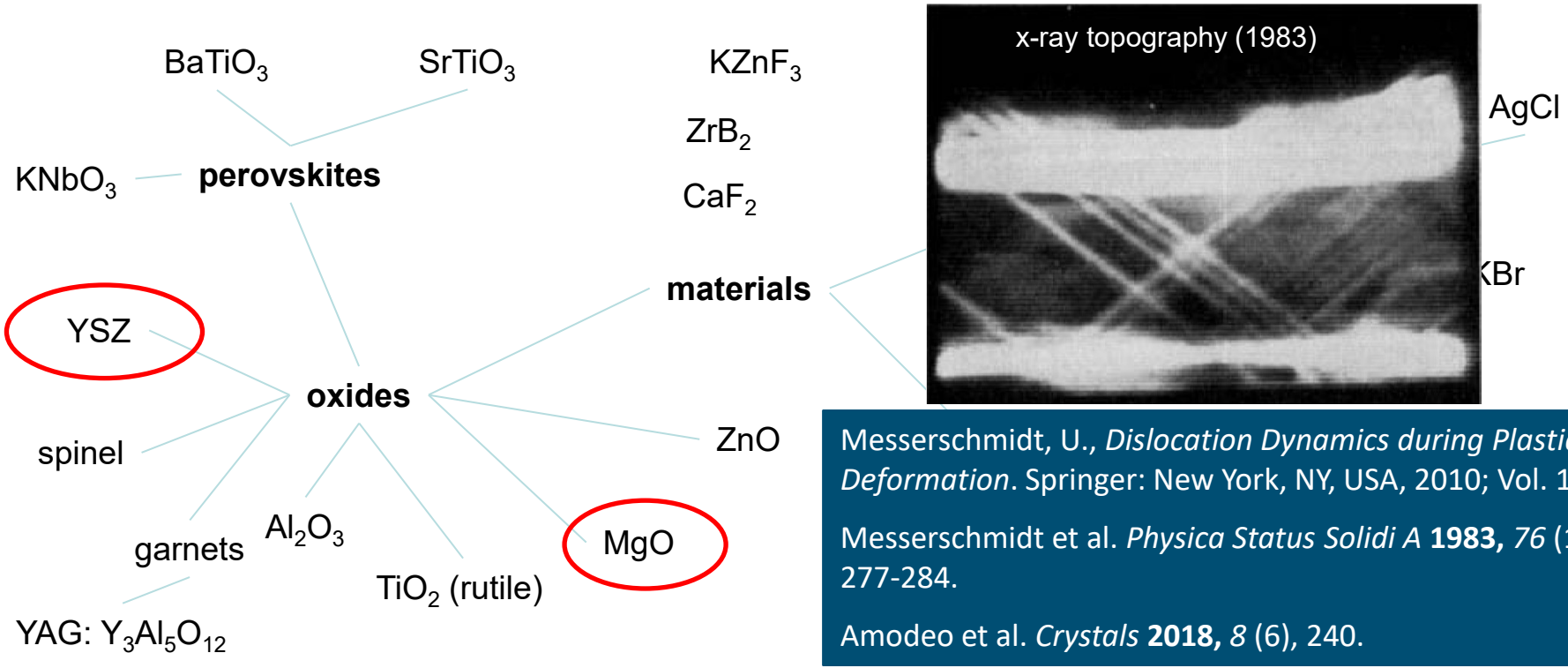
Role model materials



Whitworth, R. W., Charged dislocations in ionic crystals. *Advances in Physics* **1975,** *24* (2), 203-304.

Gilman, J. J.; Johnston, W. G., Dislocations in Lithium Fluoride Crystals. *Solid State Phys* **1962,** *13*, 147-222.

Role model materials



Messerschmidt, U., *Dislocation Dynamics during Plastic Deformation*. Springer: New York, NY, USA, 2010; Vol. 129.

Messerschmidt et al. *Physica Status Solidi A* **1983,** *76* (1), 277-284.

Amodeo et al. *Crystals* **2018,** *8* (6), 240.

Materials

Activity in Darmstadt

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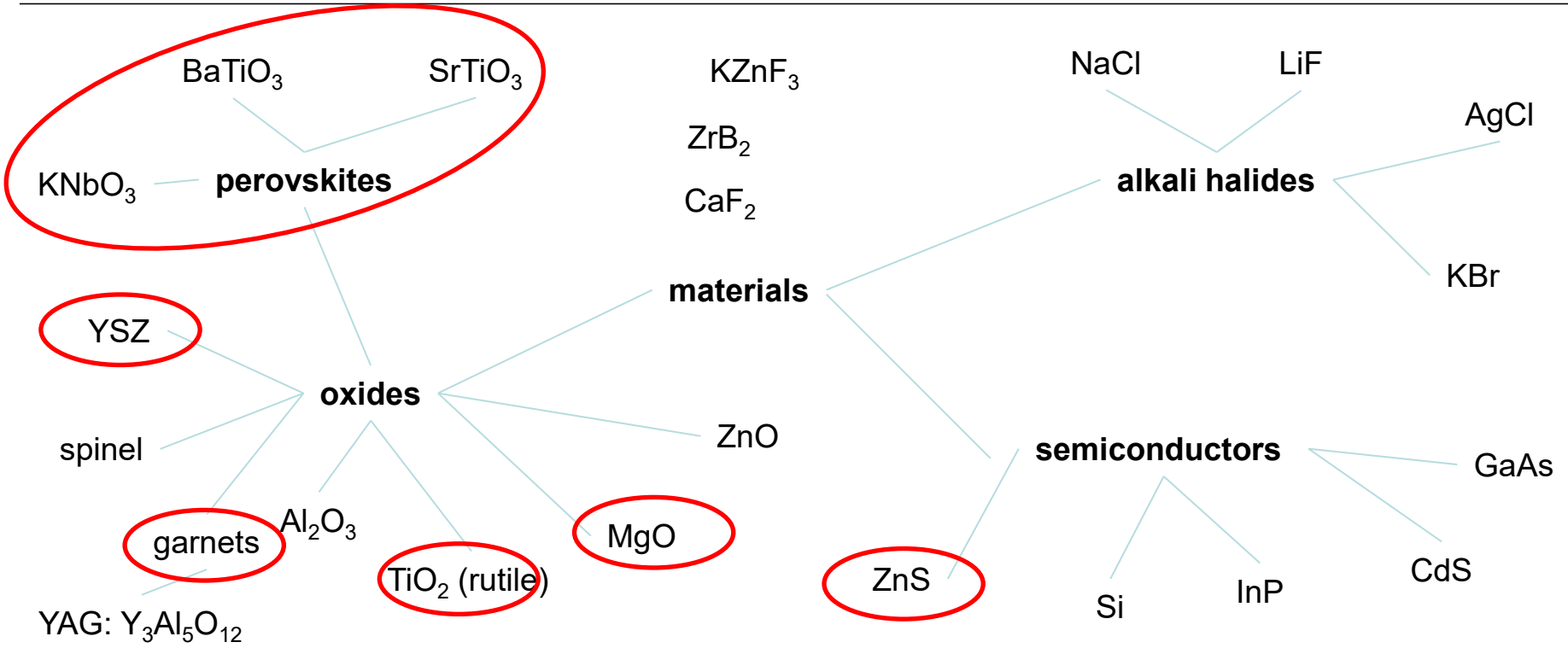
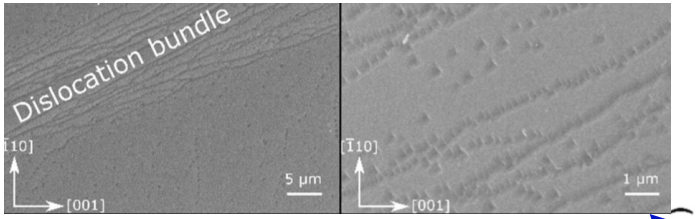
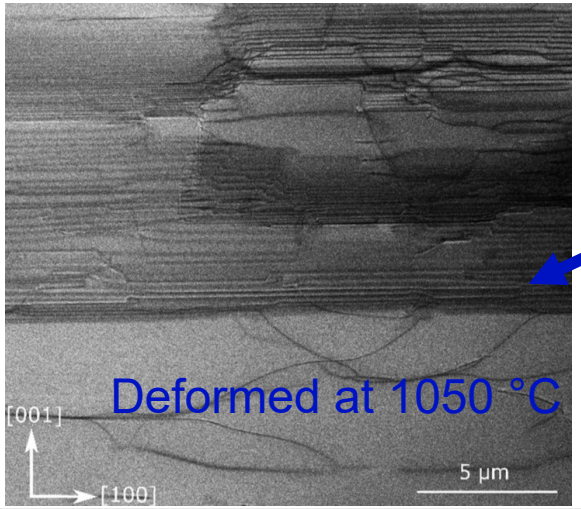
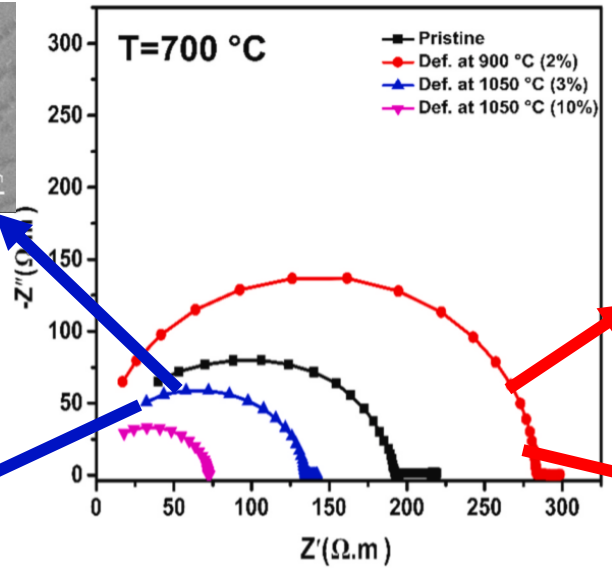


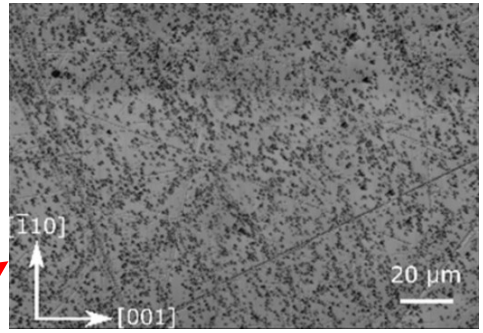
Dislocation-tuned conductivity:
Donor- and acceptor-like self-doping in TiO_2

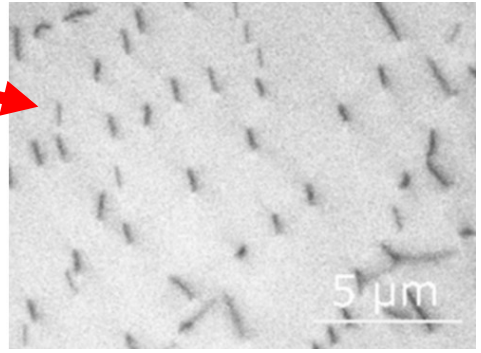






Deformed at 900 °C





Muhammad, Q. K.; Porz, L. et al. *Nano Energy* **2021,** *85*, 105944.
Porz, Frömling, et al. ACS nano **2020**, DOI: 10.1021/acsnano.0c04491

Dislocation-tuned conductivity:
Donor- and acceptor-like self-doping in TiO_2

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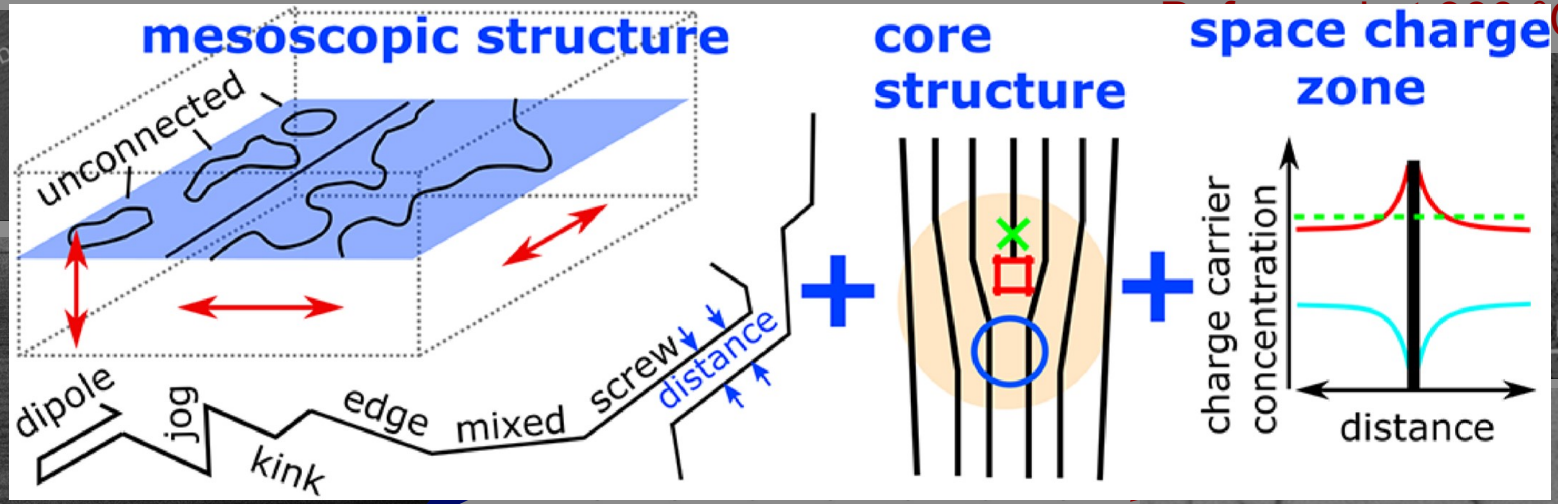
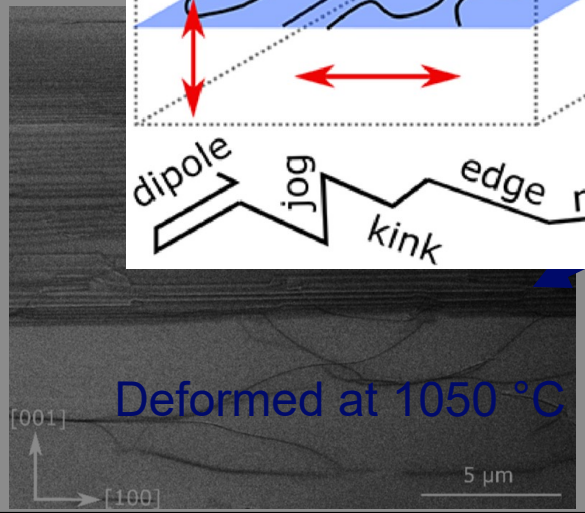
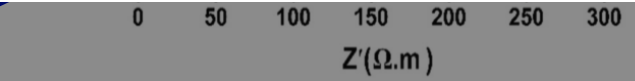
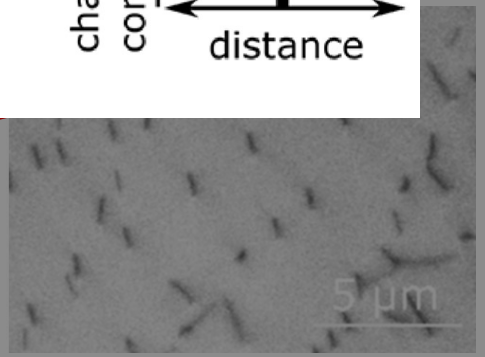


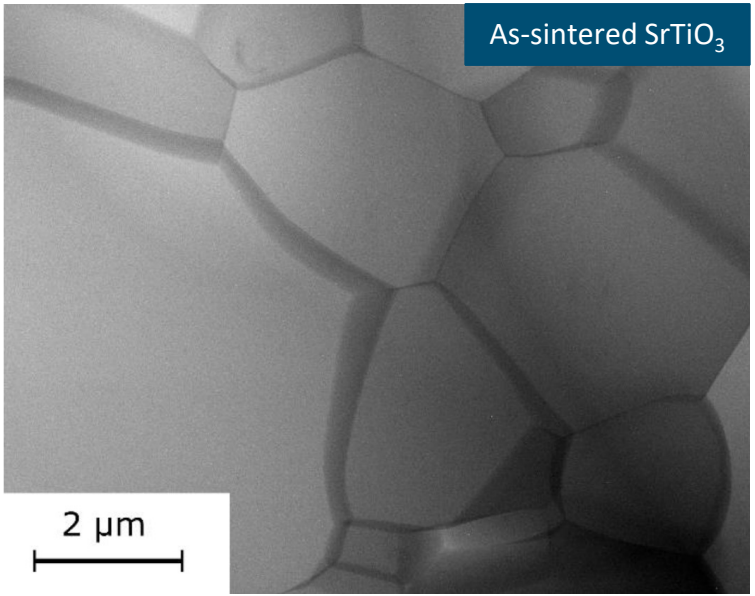




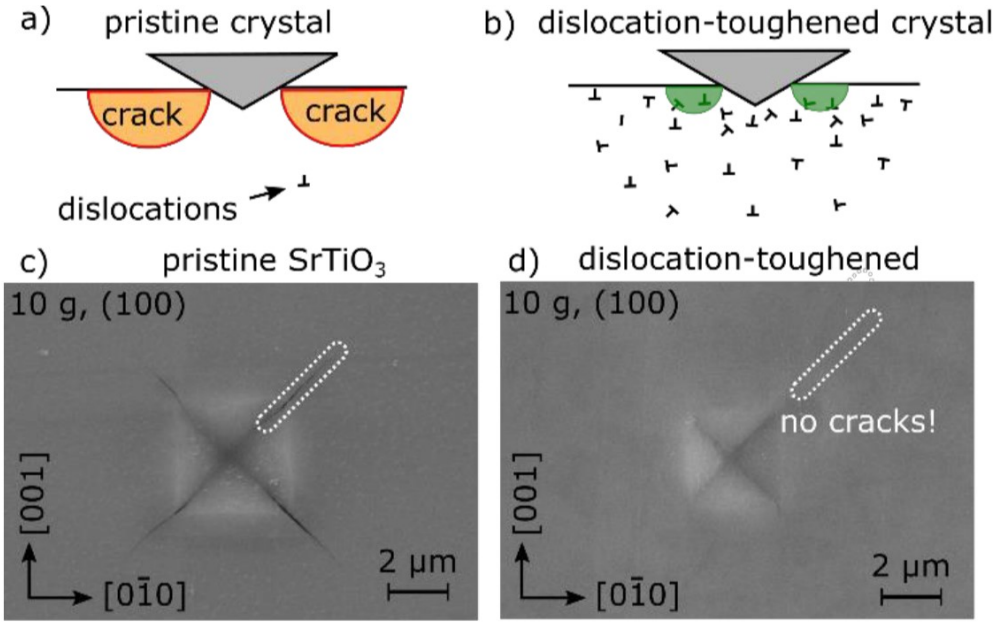


Muhammad, Q. K.; Porz, L. et al. *Nano Energy* **2021,** *85*, 105944.
Porz, Frömling, et al. ACS nano **2020**, DOI: 10.1021/acsnano.0c04491

Why are ceramics brittle?
Dislocation-toughened ceramics



UHVEM images by Prof. Atsutomo Nakamura

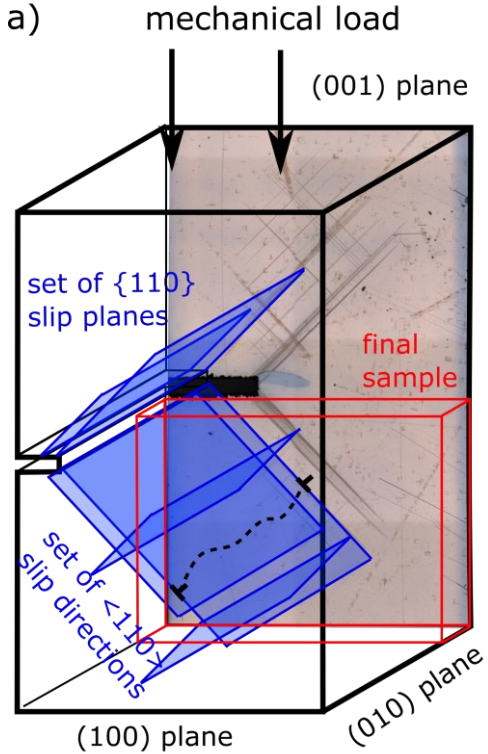


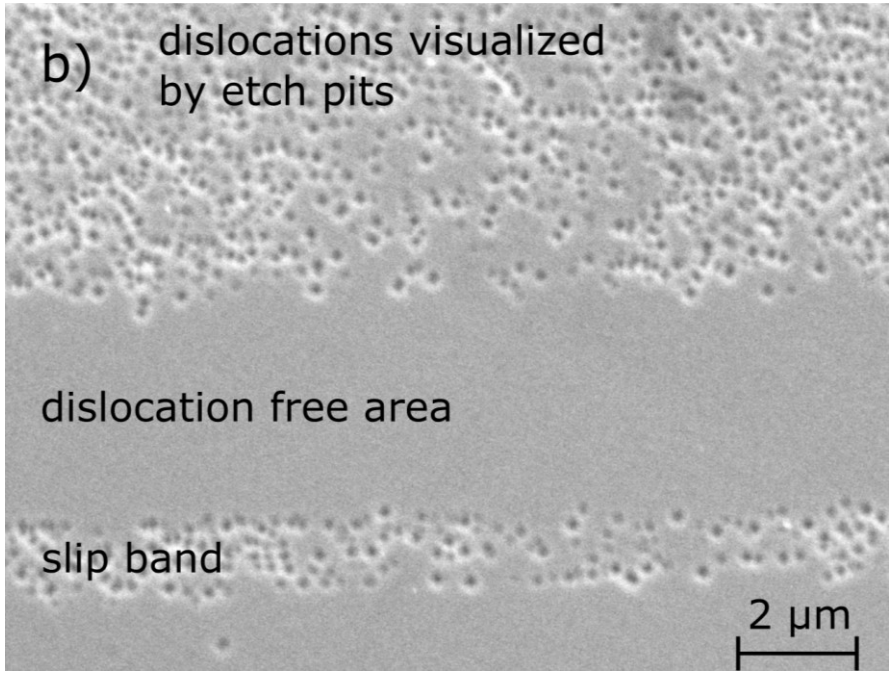
pristine

with a density of $5*10^{14}$ m^{-2}

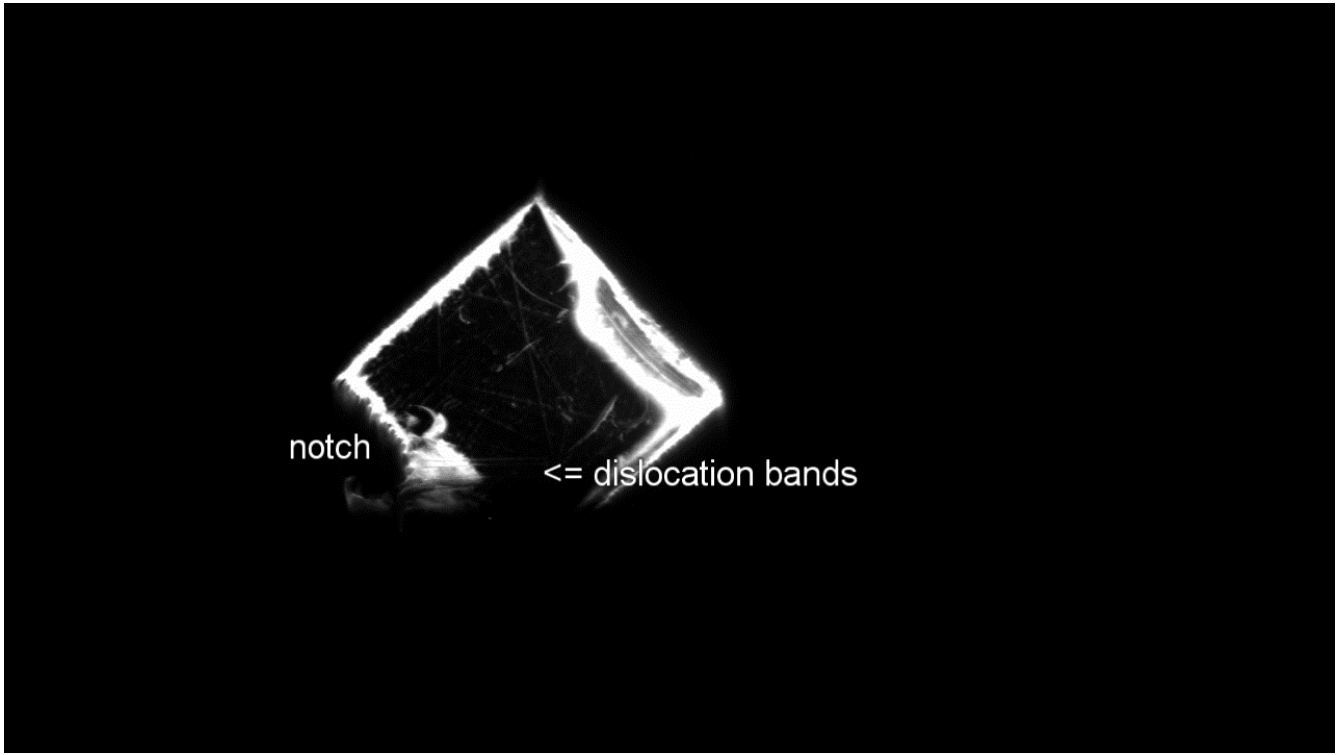
Porz et al. **2021**, Materials Horizons, 10.1039/d0mh02033h

Standard experimental setup





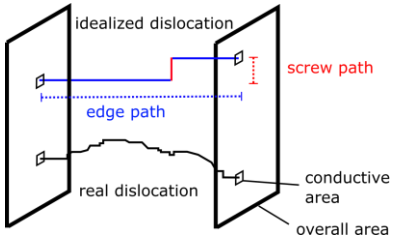
DFXM: Visualization of the multiscale structure



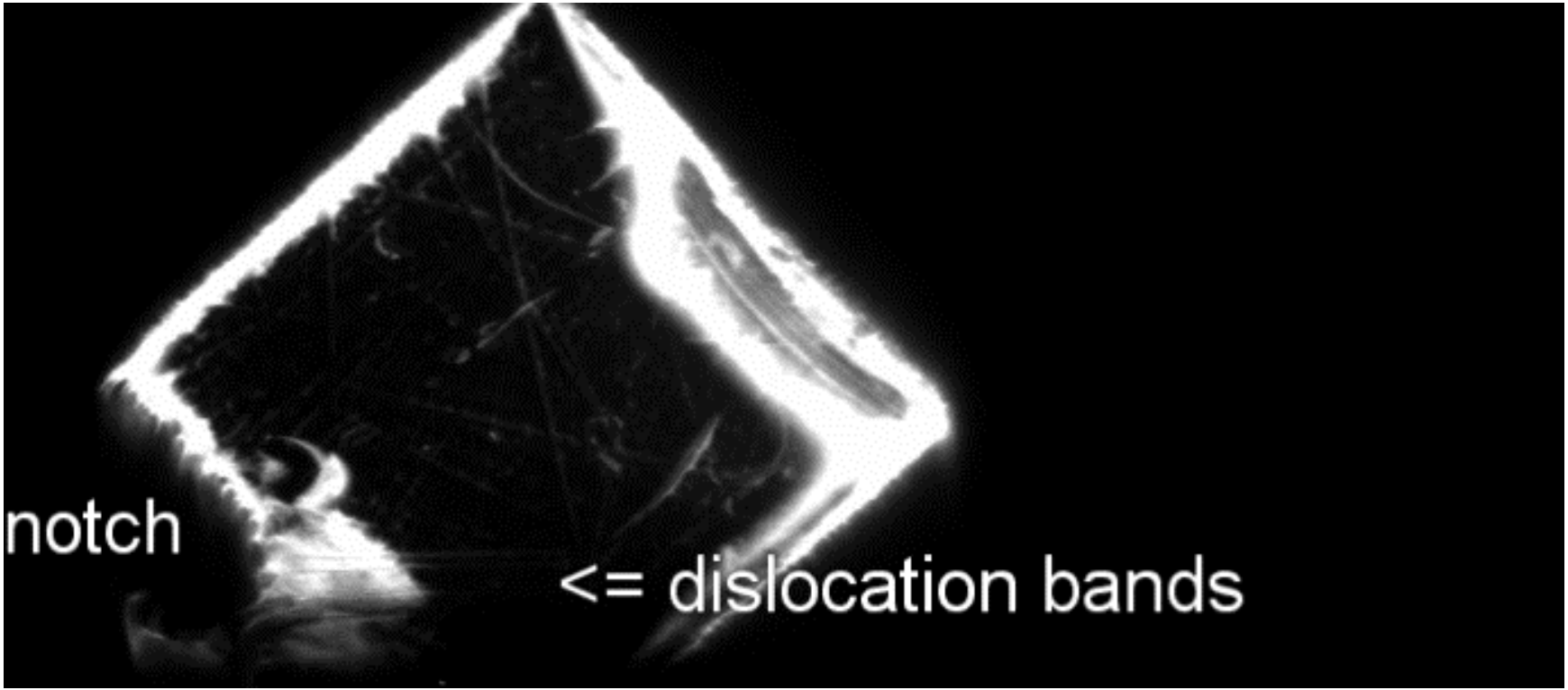
Dark-field x-ray Microscopy

ID-06 at the European Synchrotron Radiation Facility

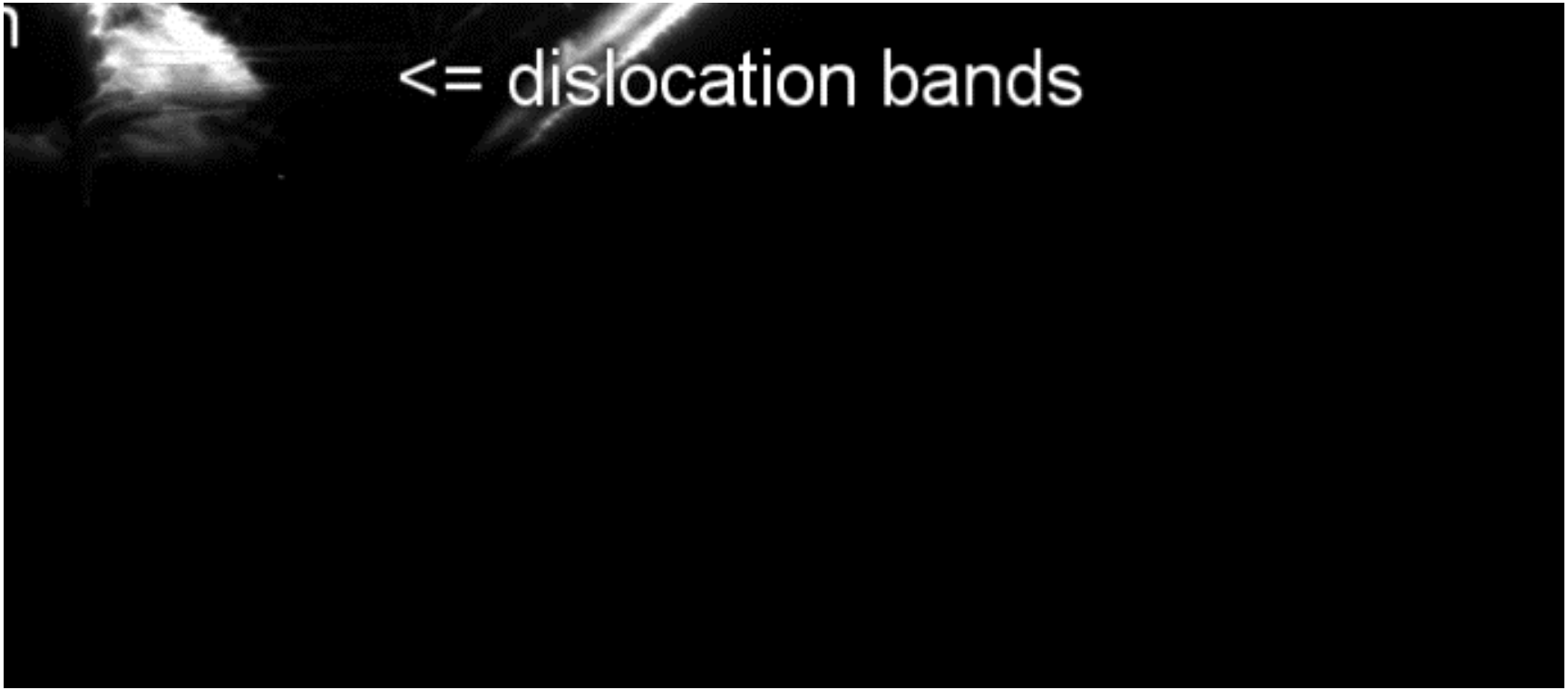
Porz et al. **2021**, Materials Horizons, 10.1039/d0mh02033h



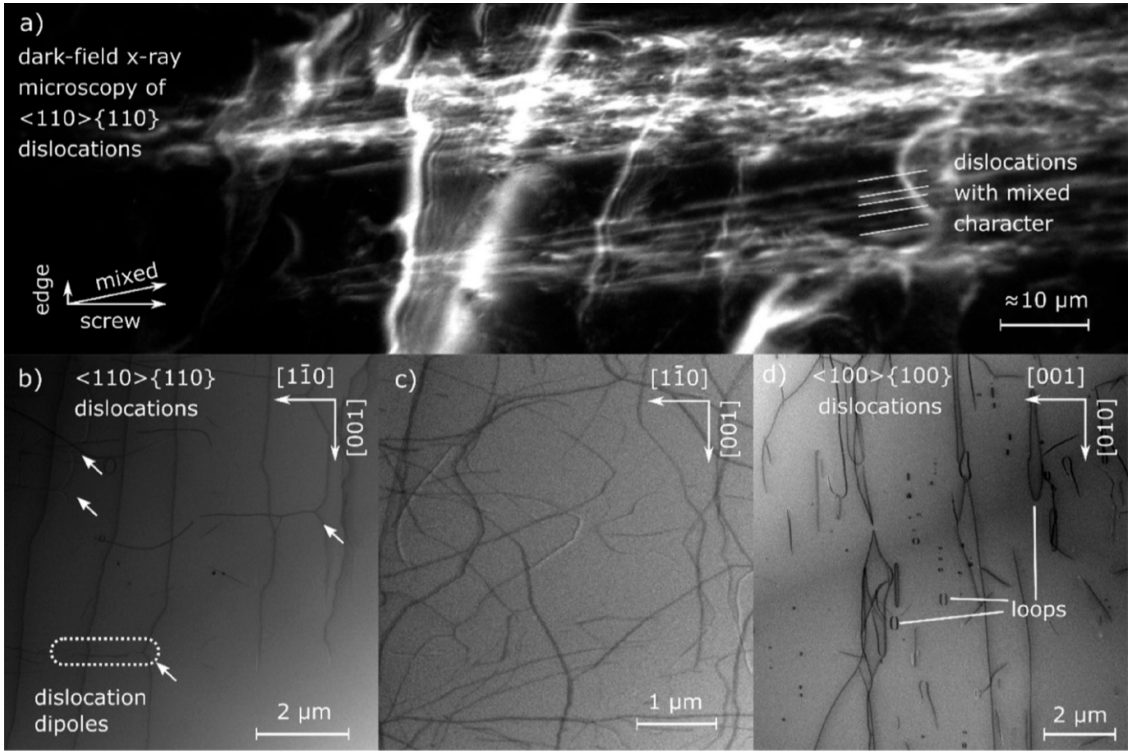
Visualization of the multiscale structure



Visualization of the multiscale structure



Results of visualization

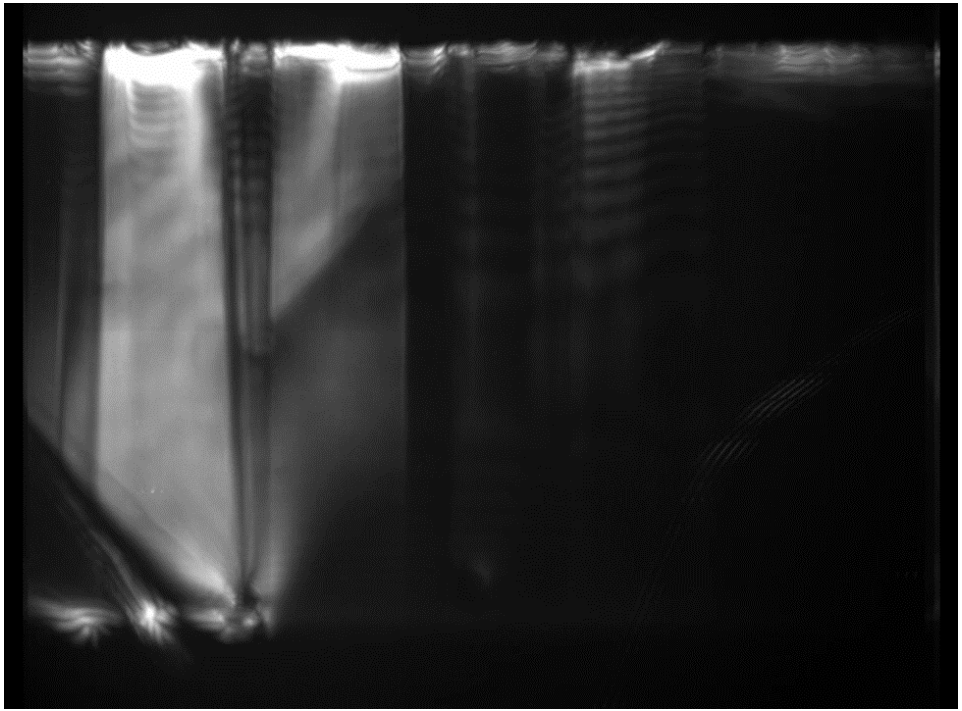


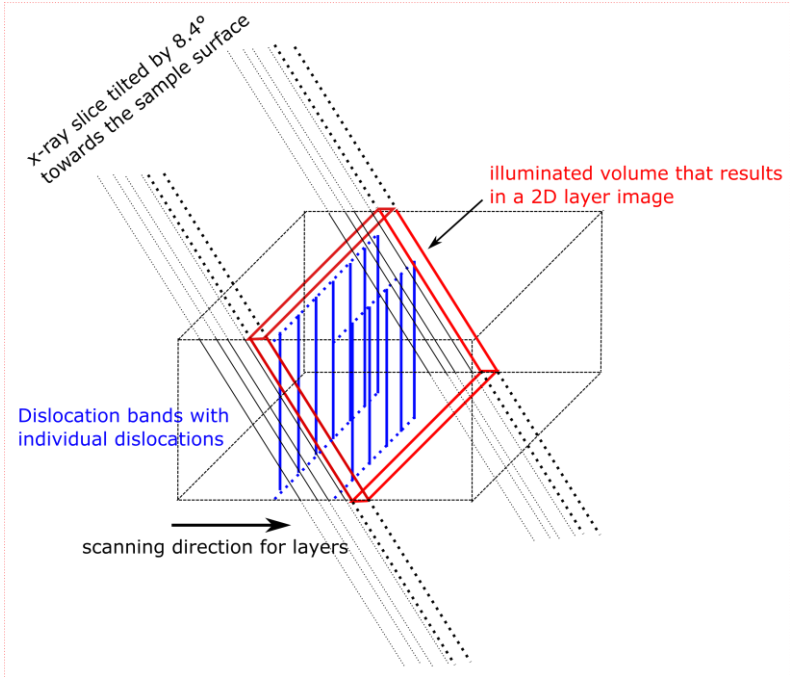
- Low density areas can be nicely visualized
- High density areas are a mess even in TEM
- Dislocations of the <100>{100} high temperature slip system are much more suitably spaced

Porz, Frömling, et al. ACS nano **2020**,
DOI: 10.1021/acsnano.0c04491

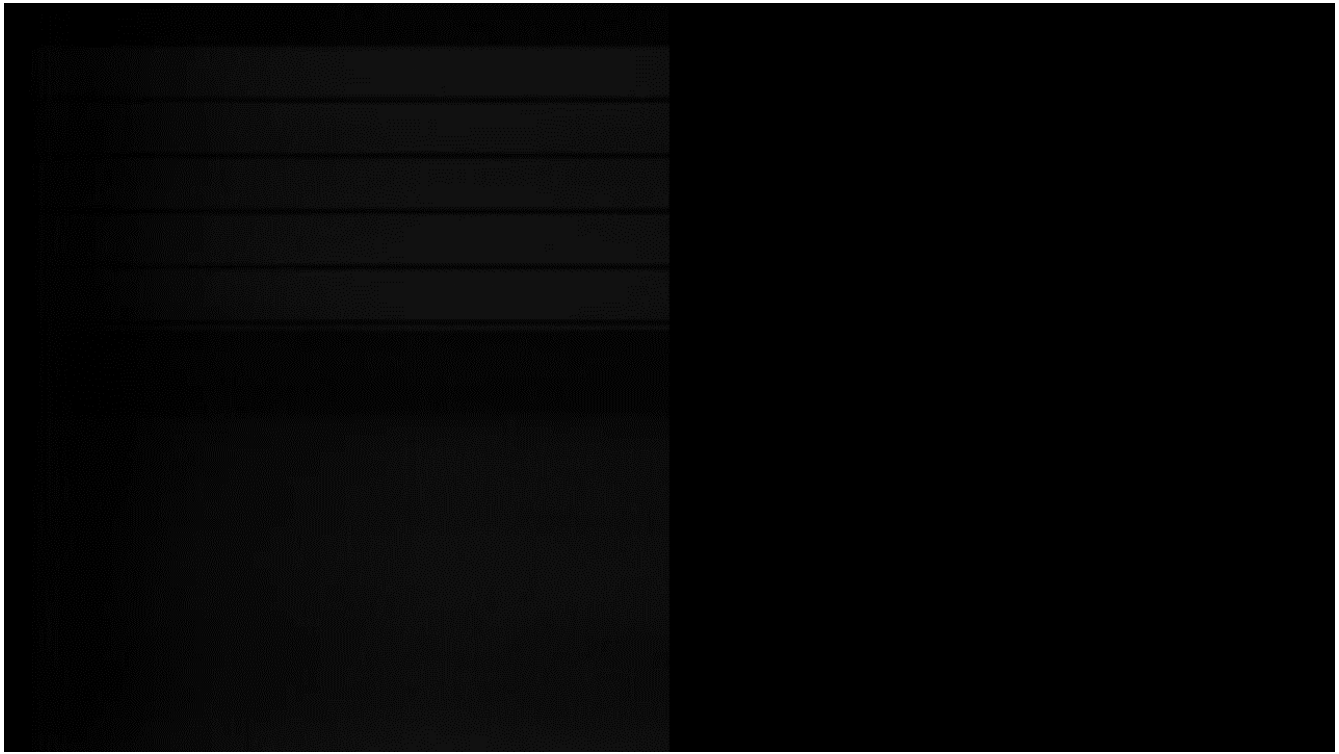
UHVEM images by Prof. Atsutomo Nakamura

Intensity (layer-by-layer)





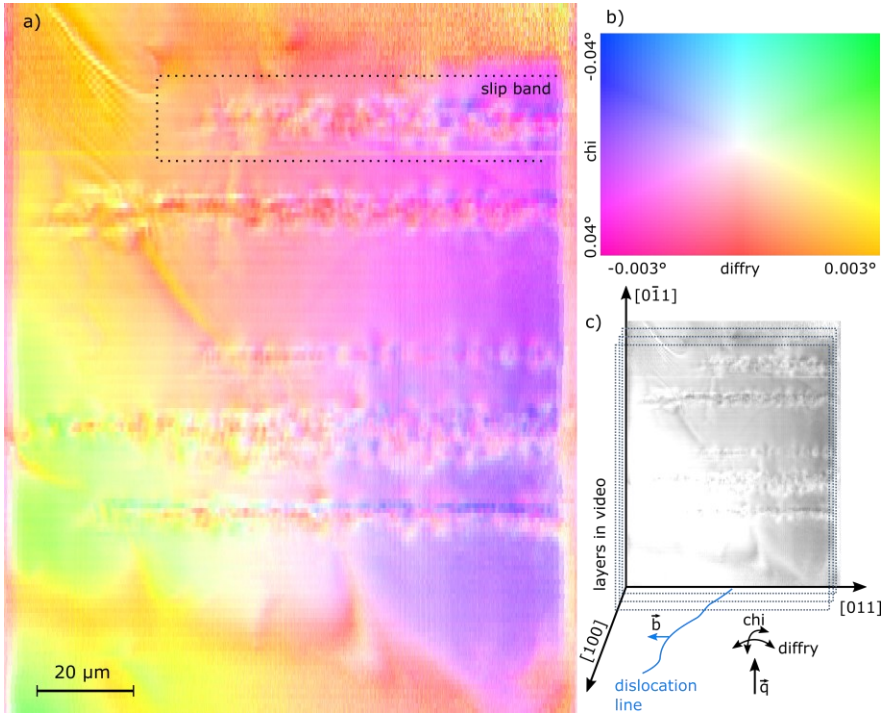
Intensity (top view)



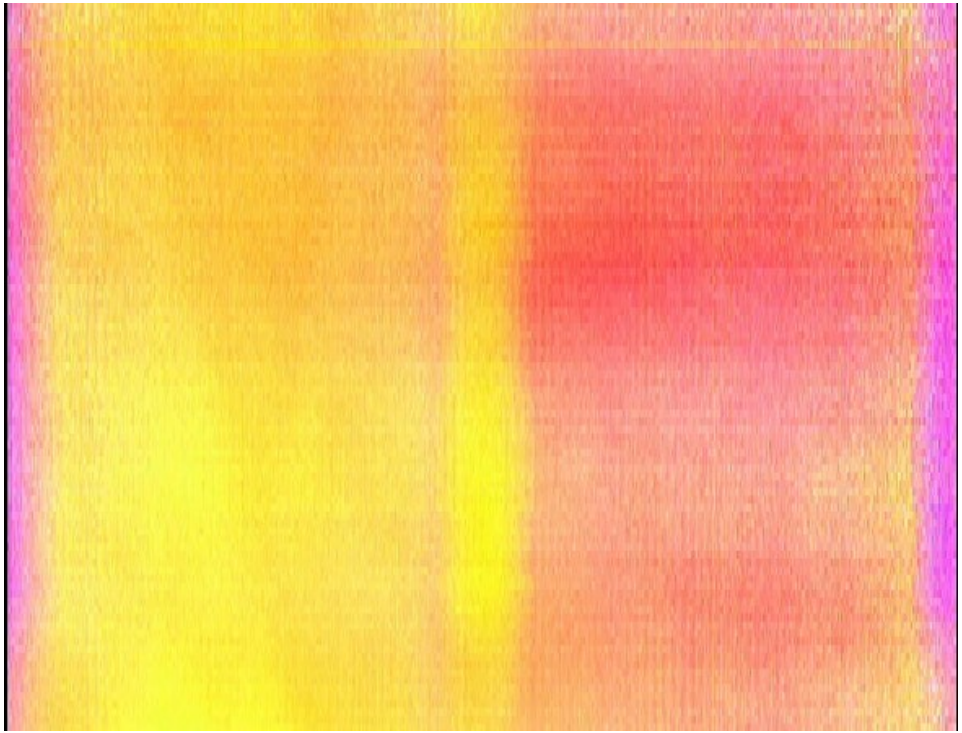
Lessons learned

- Surface damage “shines all the way through”
- Slicing direction should be closer to the line vector than to the Burgers vector
- Slicing distance should be as small as possible

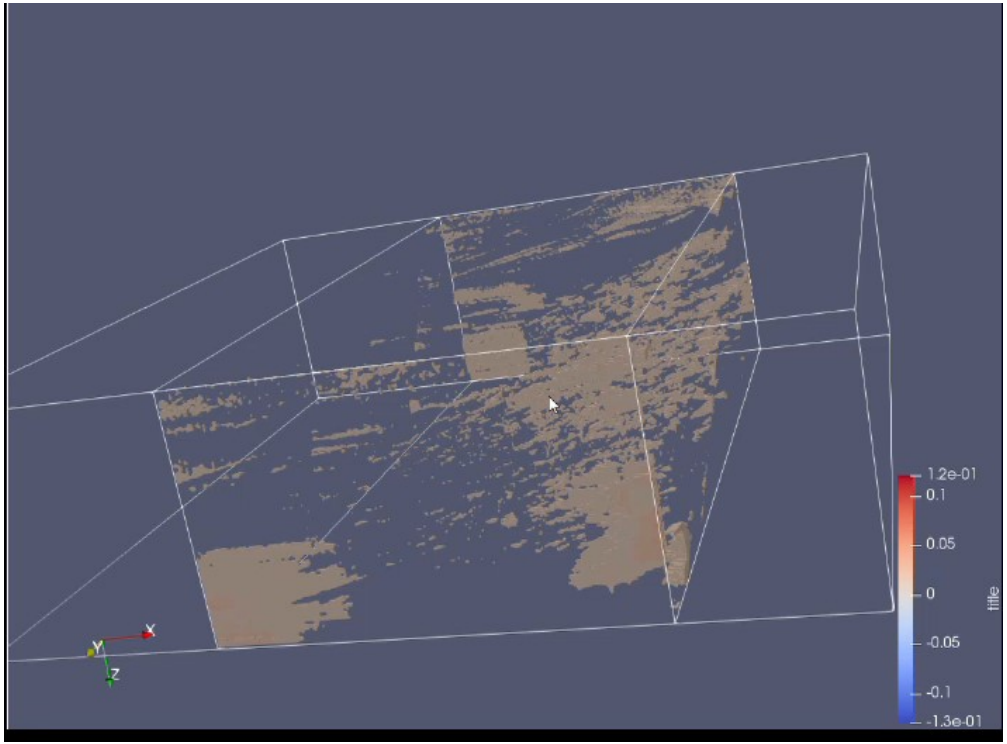
3D imaging of mosaicity and strain



Mosaicity top view



3D reconstruction



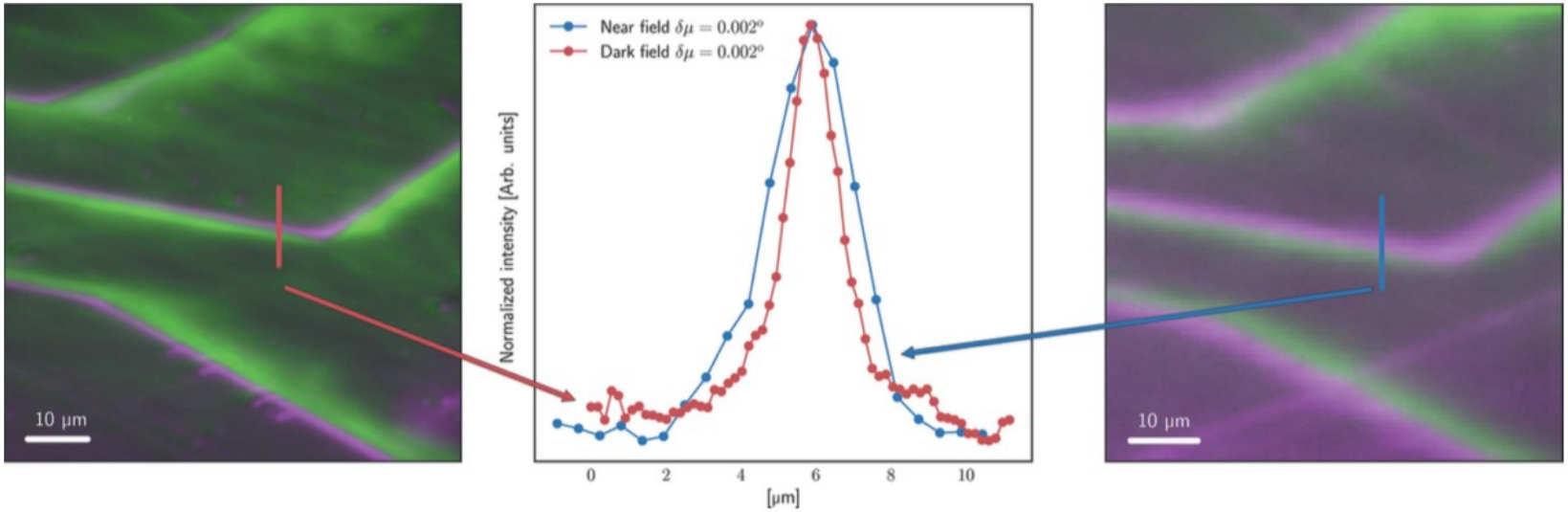
Here:

- threshold in intensity or threshold in tilt gradient

Challenges:

- Better criterion for identifying dislocations
- >1 dislocation per voxel

Indexing of dislocations?

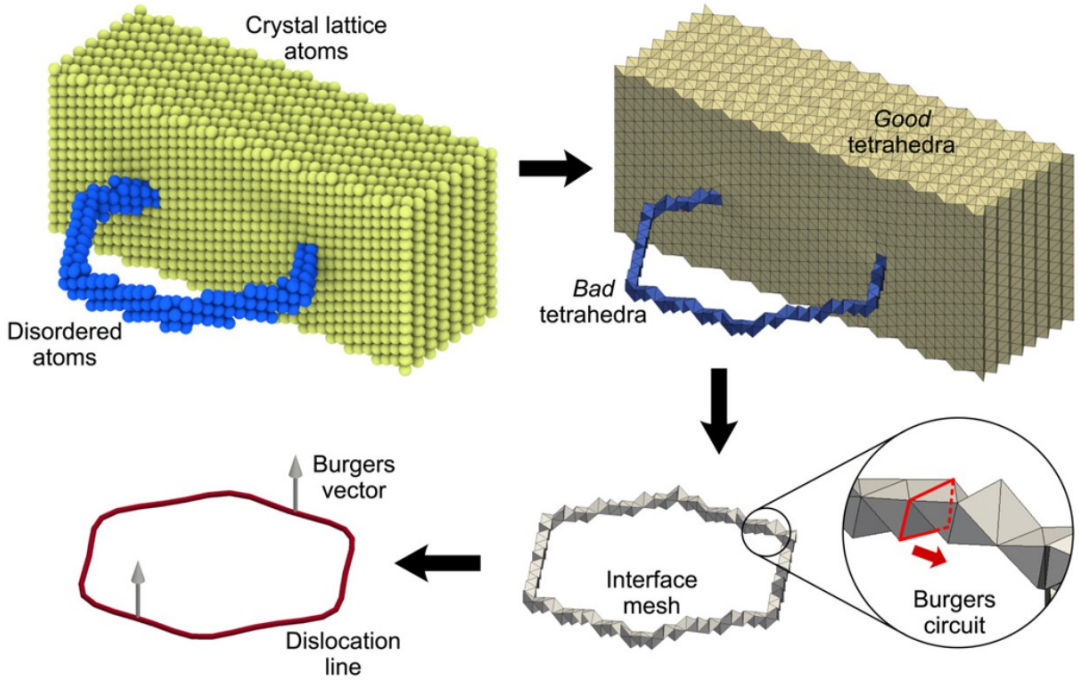


Purple: +0.002°
Green: -0.002°

Jakobsen et al. *J. Appl. Crystallogr.* **2019,** *52*, 122-132.

Can we extract dislocation line positions form automated analysis of the strain/mosaicity field data?

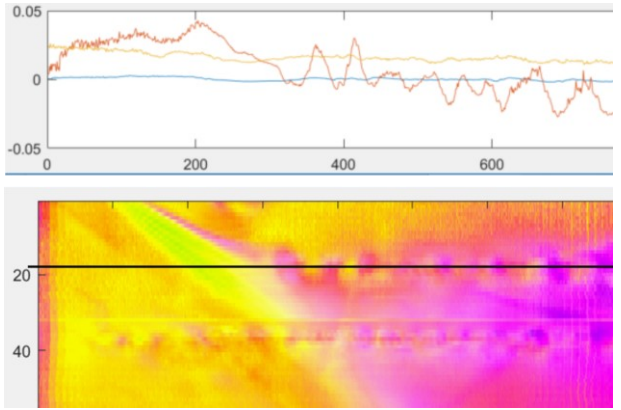
Automated indexing



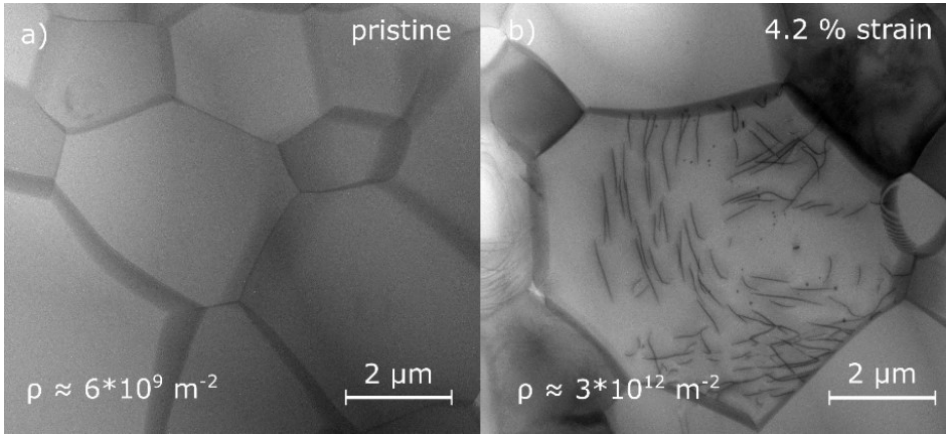
Stukowski et al. *Modell. Simul. Mater. Sci. Eng.* **2012,** *20* (8), 085007.

Stukowski *Simul. Mater. Sci. Eng.* **2010,** *18* (1), 015012.

strain: yellow
dim1: blue
dim2: red



High-temperature plasticity of ceramics



M. Johanning, L. Porz et al. Appl. Phys. Lett. 2020, 117.

Rice, R. W., Deformation, Recrystallization, Strength, and Fracture of Press-Forged Ceramic Crystals. *J. Am. Ceram. Soc.* **1972,** 55, (2), 90-97.

High-temperature deformation:
Contrast between metals and ceramics

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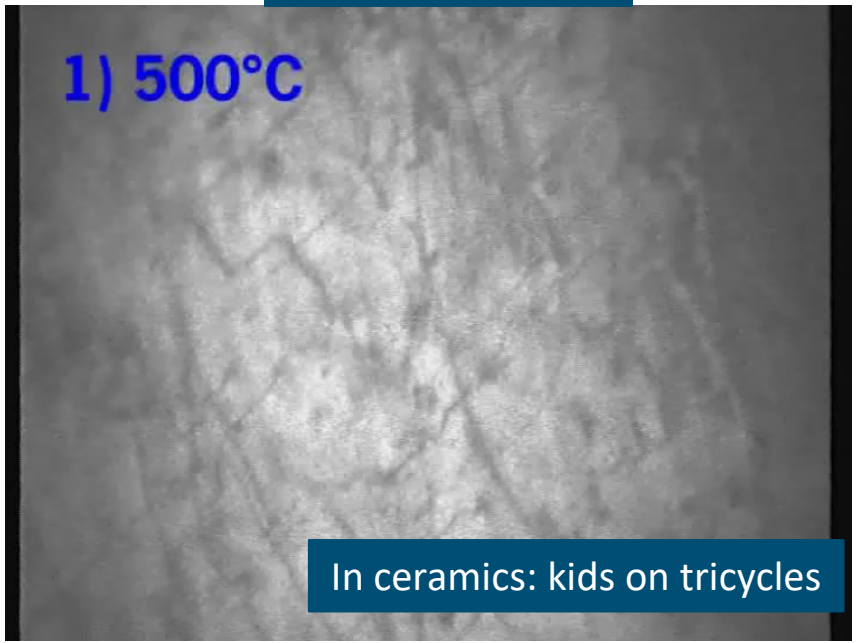
Limited by obstacles

Limited by velocity



Video Material from: U. Messerschmidt, *Dislocation Dynamics during Plastic Deformation*, Vol. 129, Springer, New York, NY, USA 2010.

High-temperature deformation:
Quantification of plastic flow

Multitude of mecha

- Strain softening
- Strain hardening

- Mobility limitatio

- T-dependent yield stress
- (No) dislocation creep

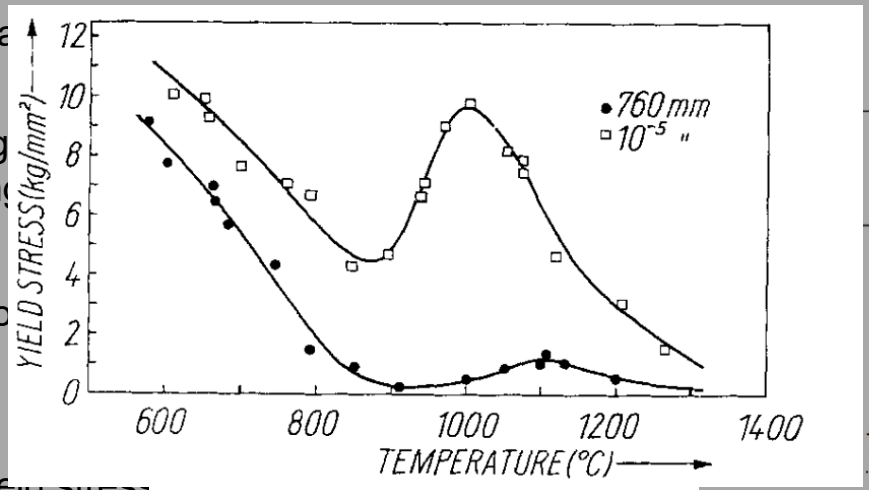
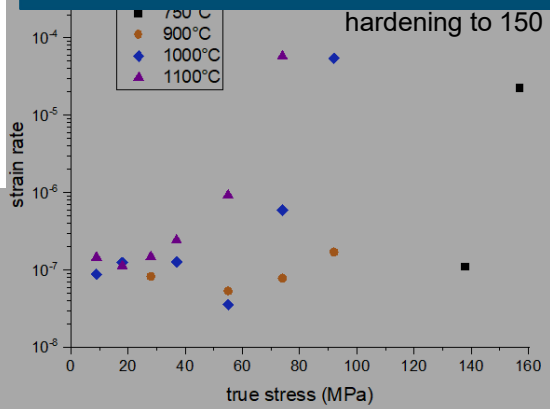


Fig. 5. Temperature-dependence of the yield stress at 760 mm and 10^{-5} mm air pressures

K. H. G. Ashbee and R. E. Smallman, *Phys Status Solidi*, 1964, **4**, 289-298.

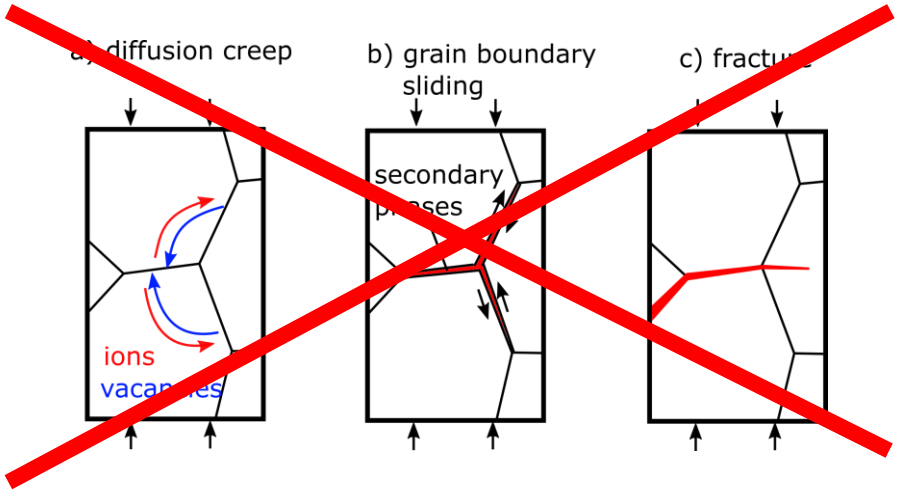
hardening to 150 MPa

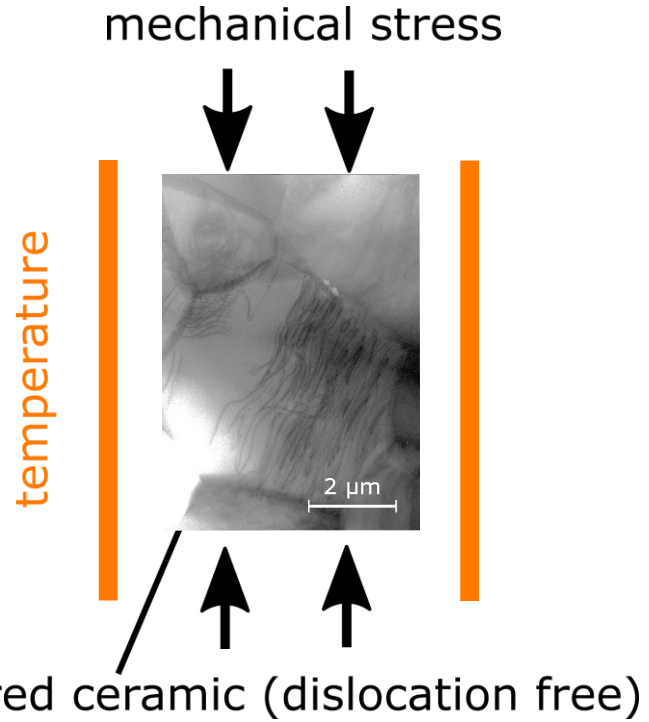


Thanks to my students Sabrina Kahse and Johannes Puy

High-temperature deformation:
The key idea

Temperature makes ceramics ductile(?)





Rheinheimer, W.; Hoffmann, M. J., *Scripta Mater.* **2015,** 101, 68-71.

High-temperature deformation:
Increases of dislocation density

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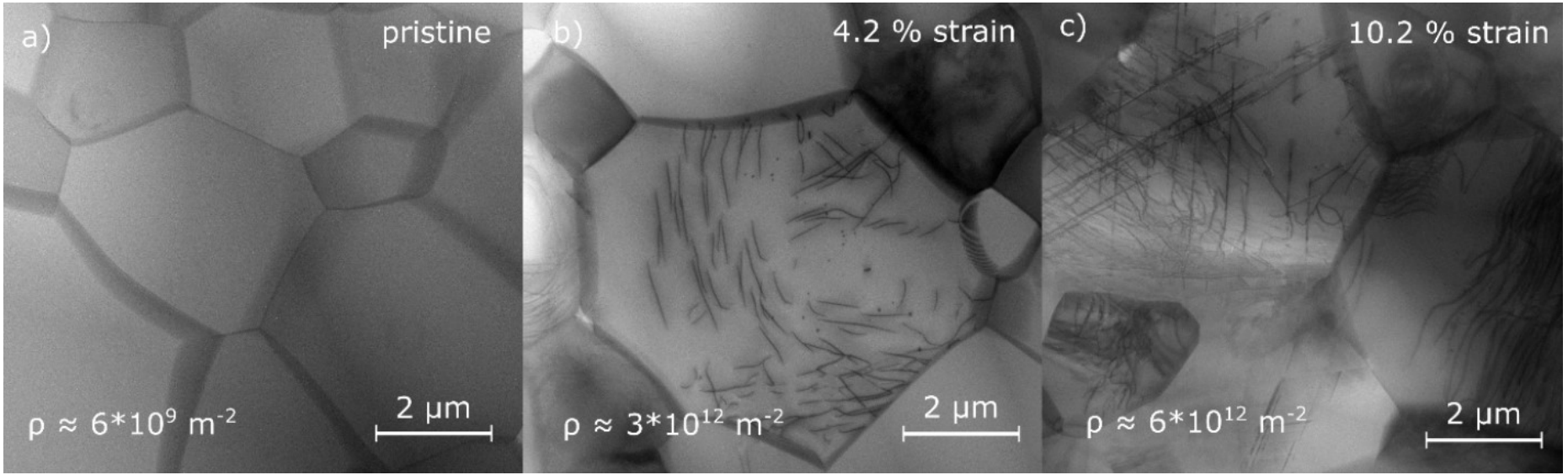
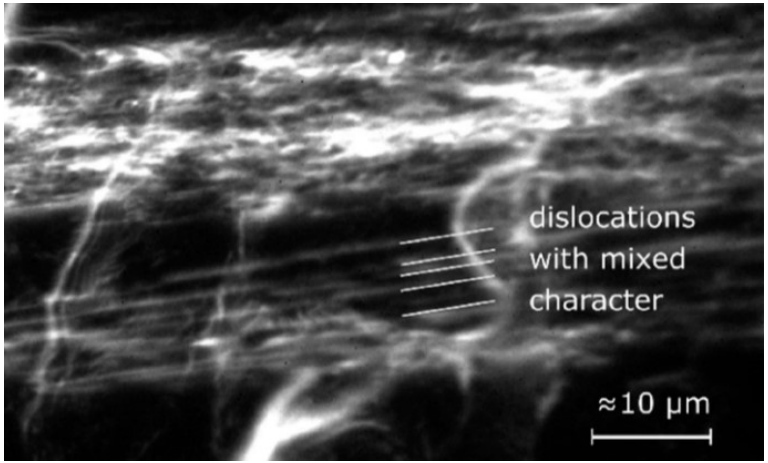
>25% deformation possible at 1150 °C



M. Johanning, L. Porz et al. Appl. Phys. Lett. 2020, 117.

Opportunities and challenges for DFXM for dislocations in ceramics

- Density is lower, but not always -> visualization becomes easier



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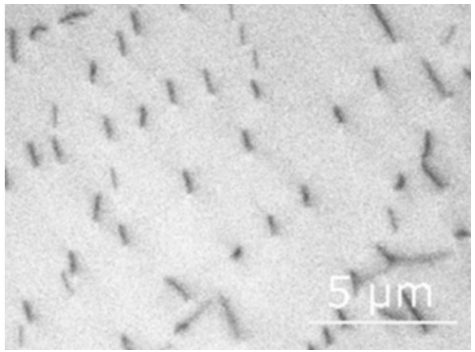

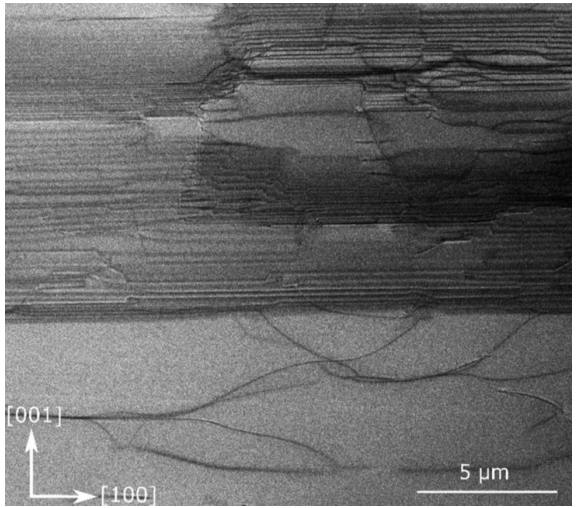

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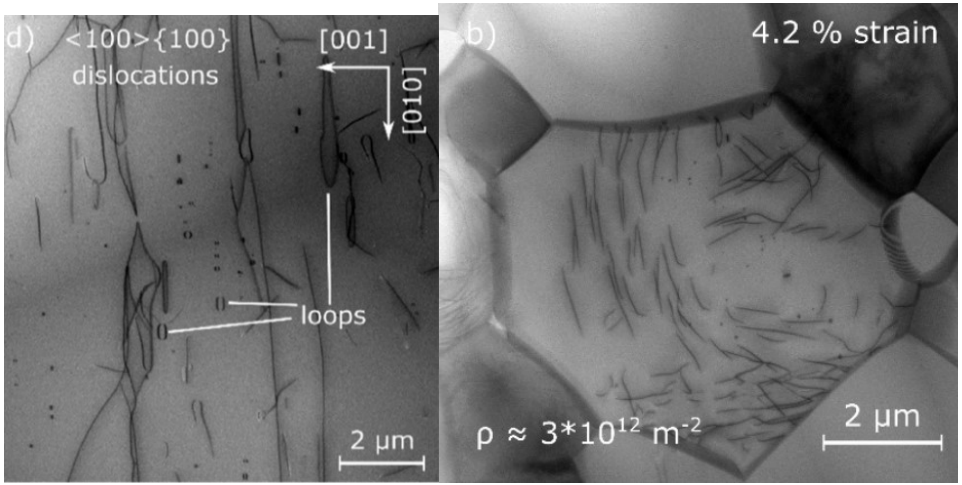
  - In-situ deformation (time resolved)
  - At about 1100 °C or 900 °C at least



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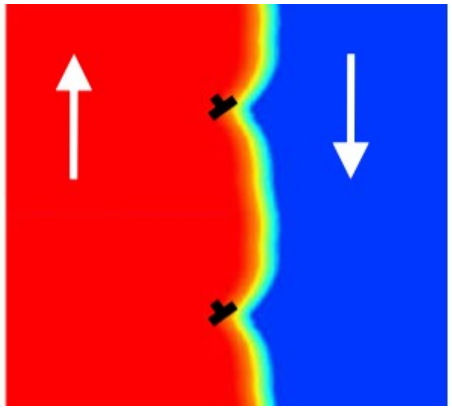

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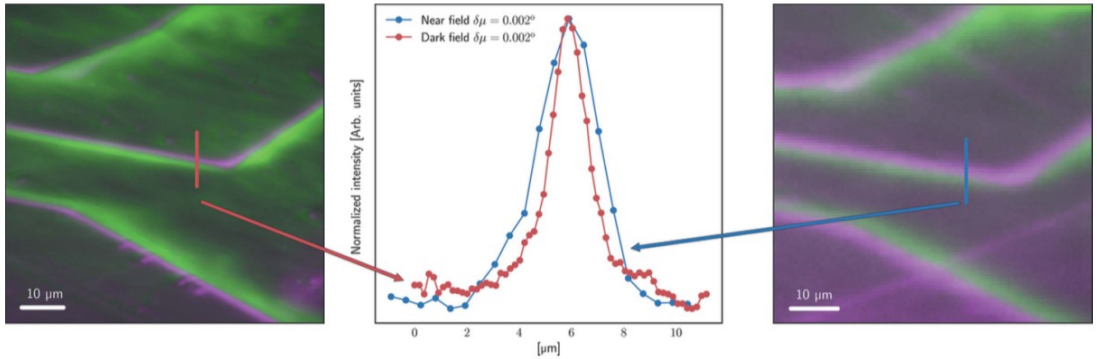
- Dislocation indexing



The dislocation team:

Lukas Porz

Marion Höfling

Nonmetallic- Inorganic Materials
Department of Materials and Earth Sciences
Technical University of Darmstadt
Germany

Jürgen Rödel

Qaisar Muhammed

Michael Scherer

Fangping Zhuo

Shuang Gao

Xufei Fang