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Developing Radiation Resistant Ceramics through Microstructural Engineering

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Personal

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Research Group: Structural Materials in Extreme Environments Group (SMEE)

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Facilities

Frederick Seitz Materials Research Laboratory (MRL)

Sandia National Laboratories

Center for Integrated Nanotechnologies (CINT)

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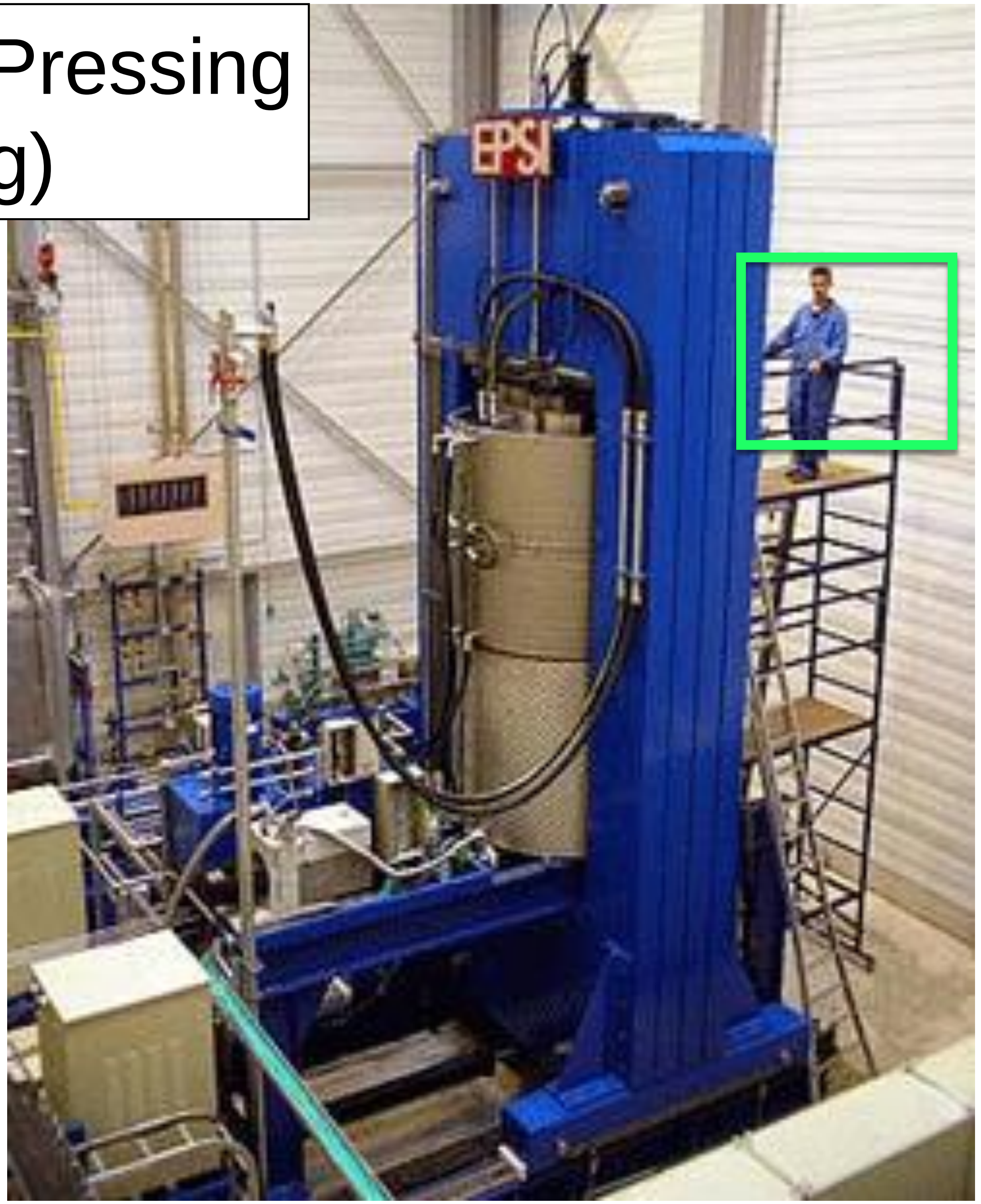
MRL THE FREDERICK SEITZ MATERIALS RESEARCH LABORATORY

Porosity…

Good or Bad?

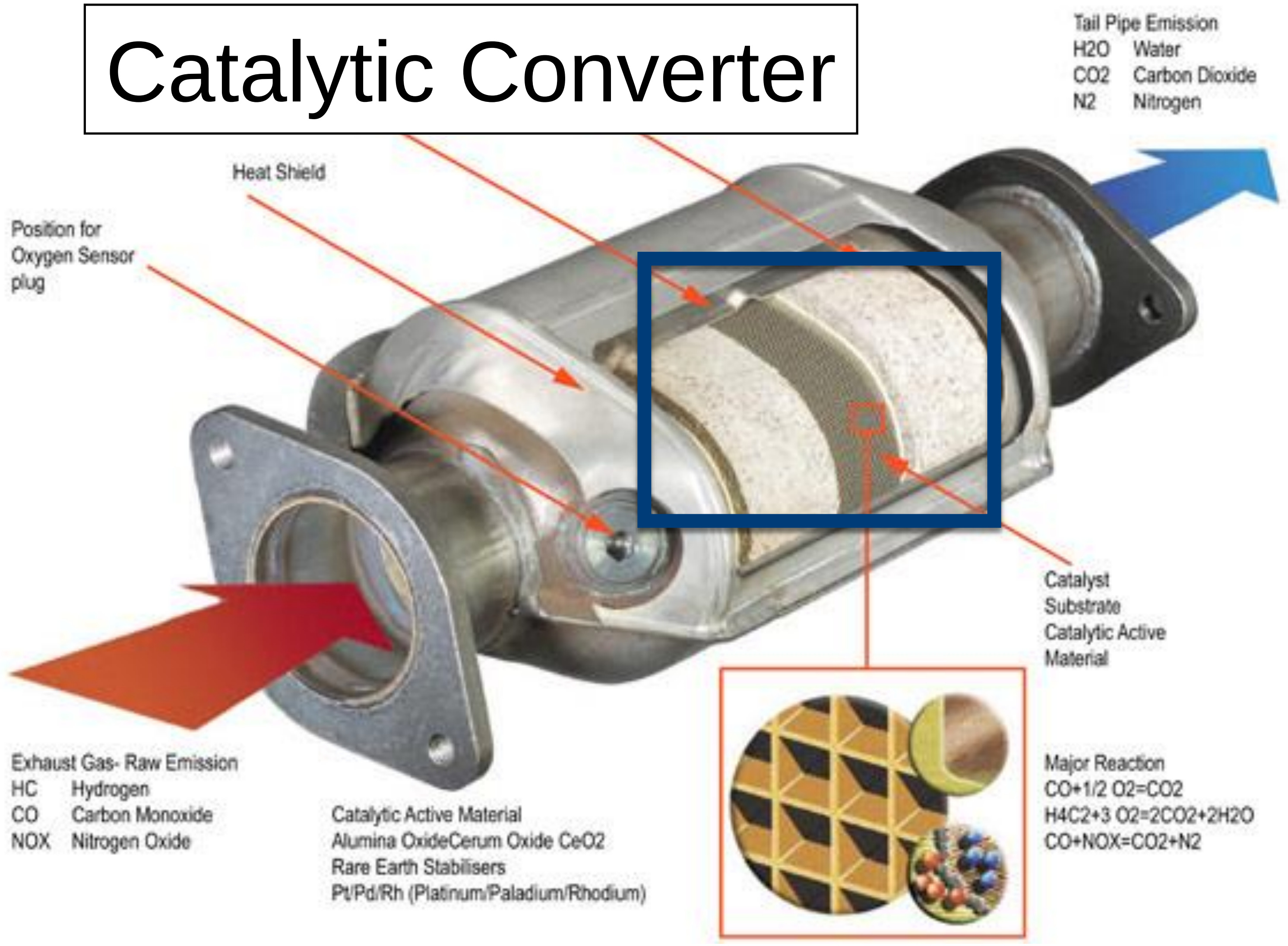
Hot Isostatic Pressing (HIPing)



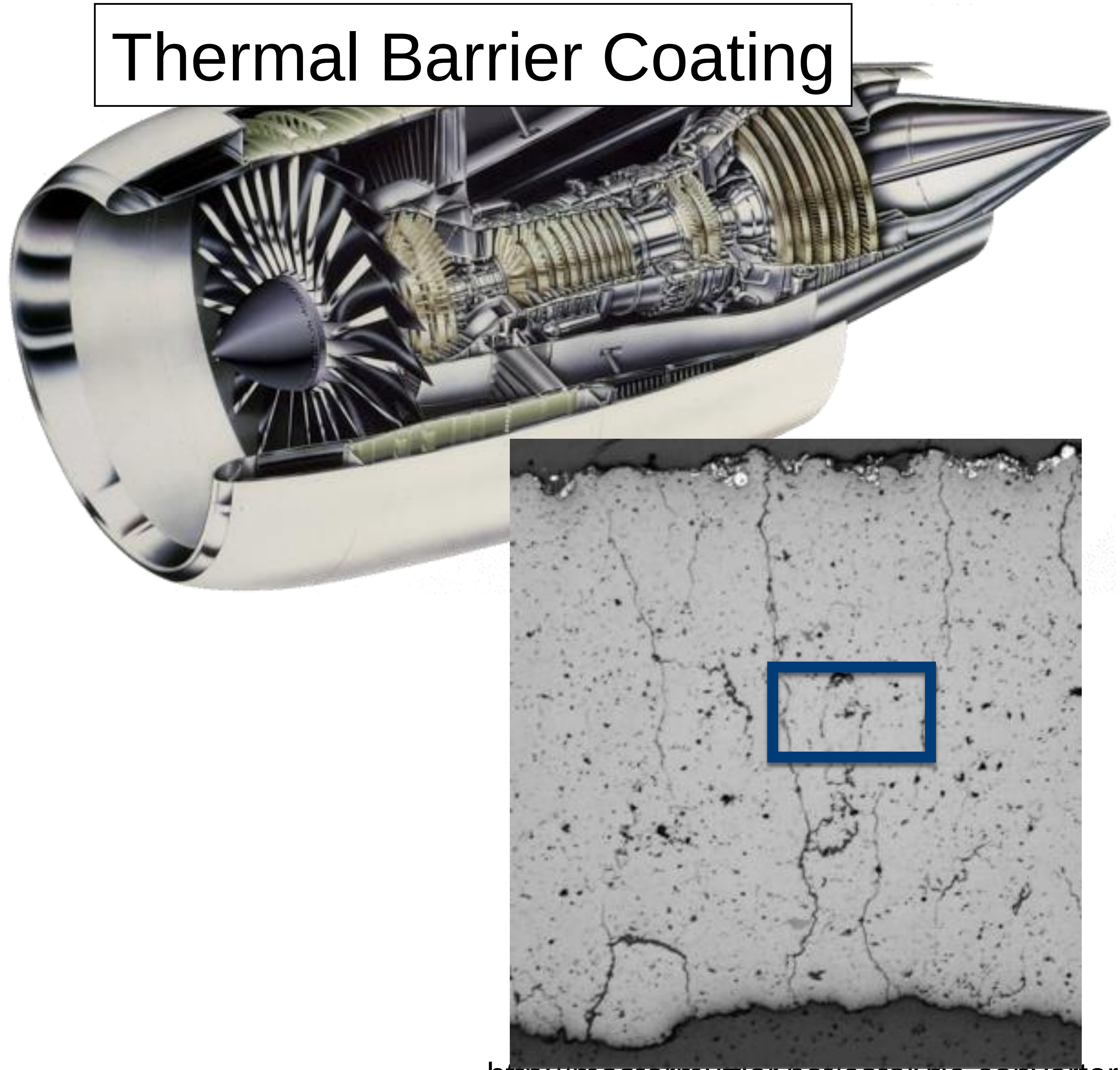
A lot of time, effort and money has been spent to reduce or eliminate porosity

Many applications take advantage of porosity

Catalytic Converter

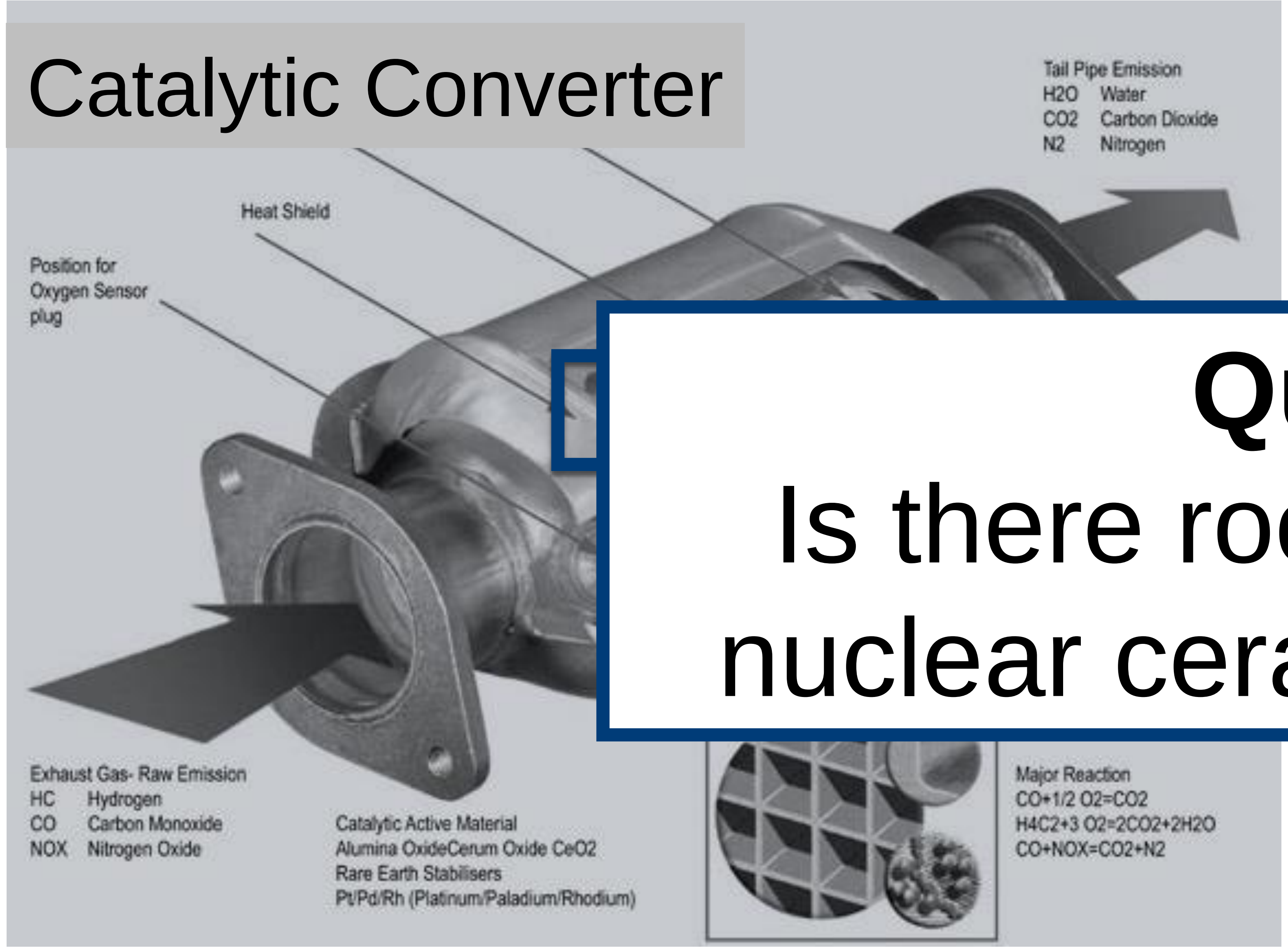


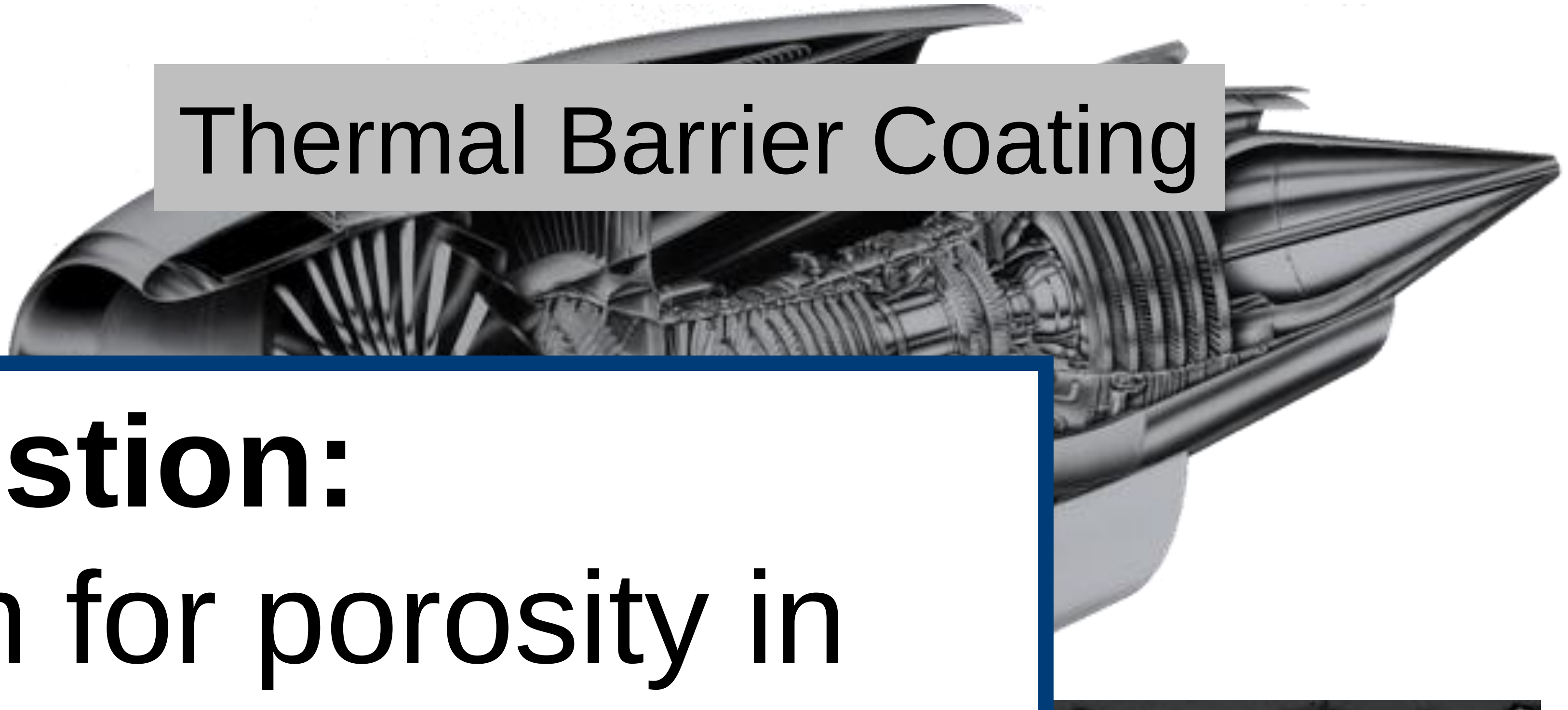
Thermal Barrier Coating



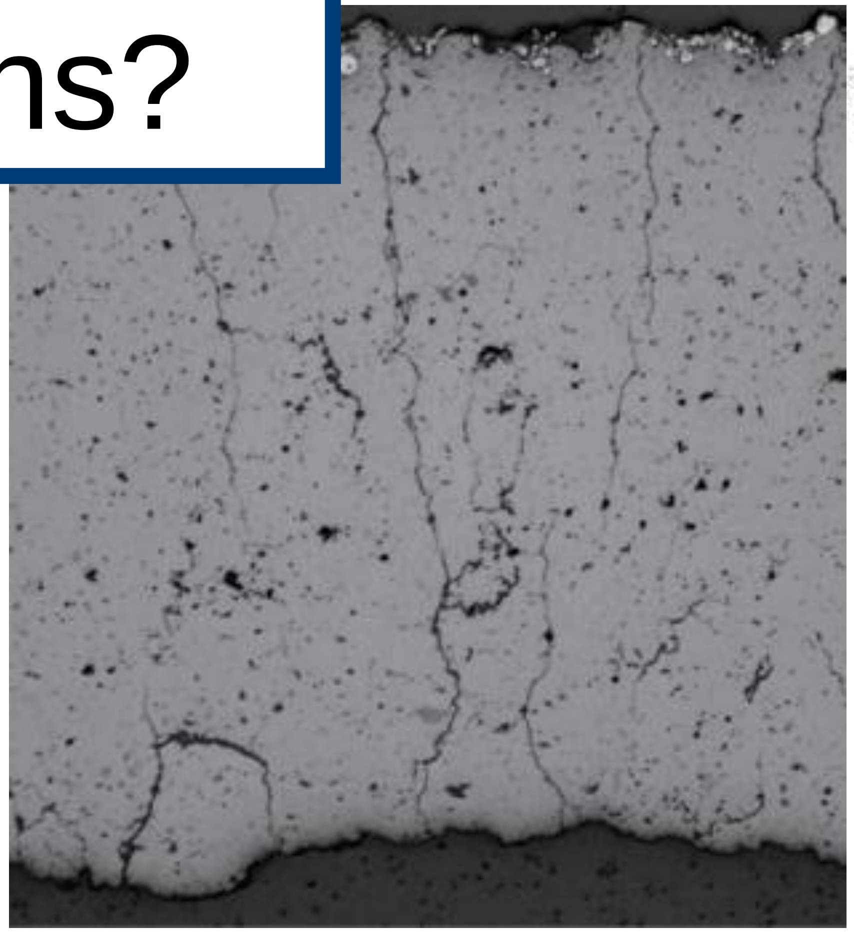
http://mastermuffler.net/catalytic-converter/
http://people.seas.harvard.edu/

Many applications take advantage of porosity





Question:
Is there room for porosity in nuclear ceramic applications?



http://mastermuffler.net/catalytic-converter/
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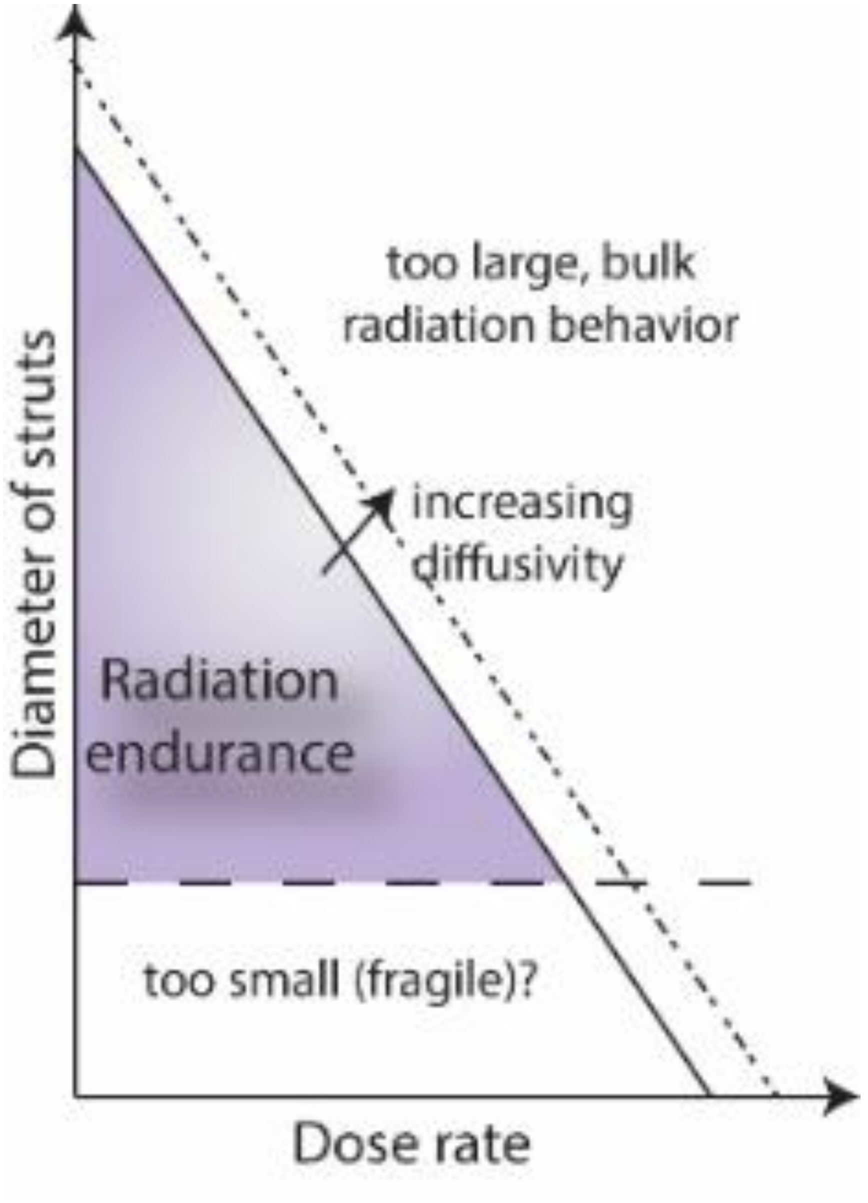
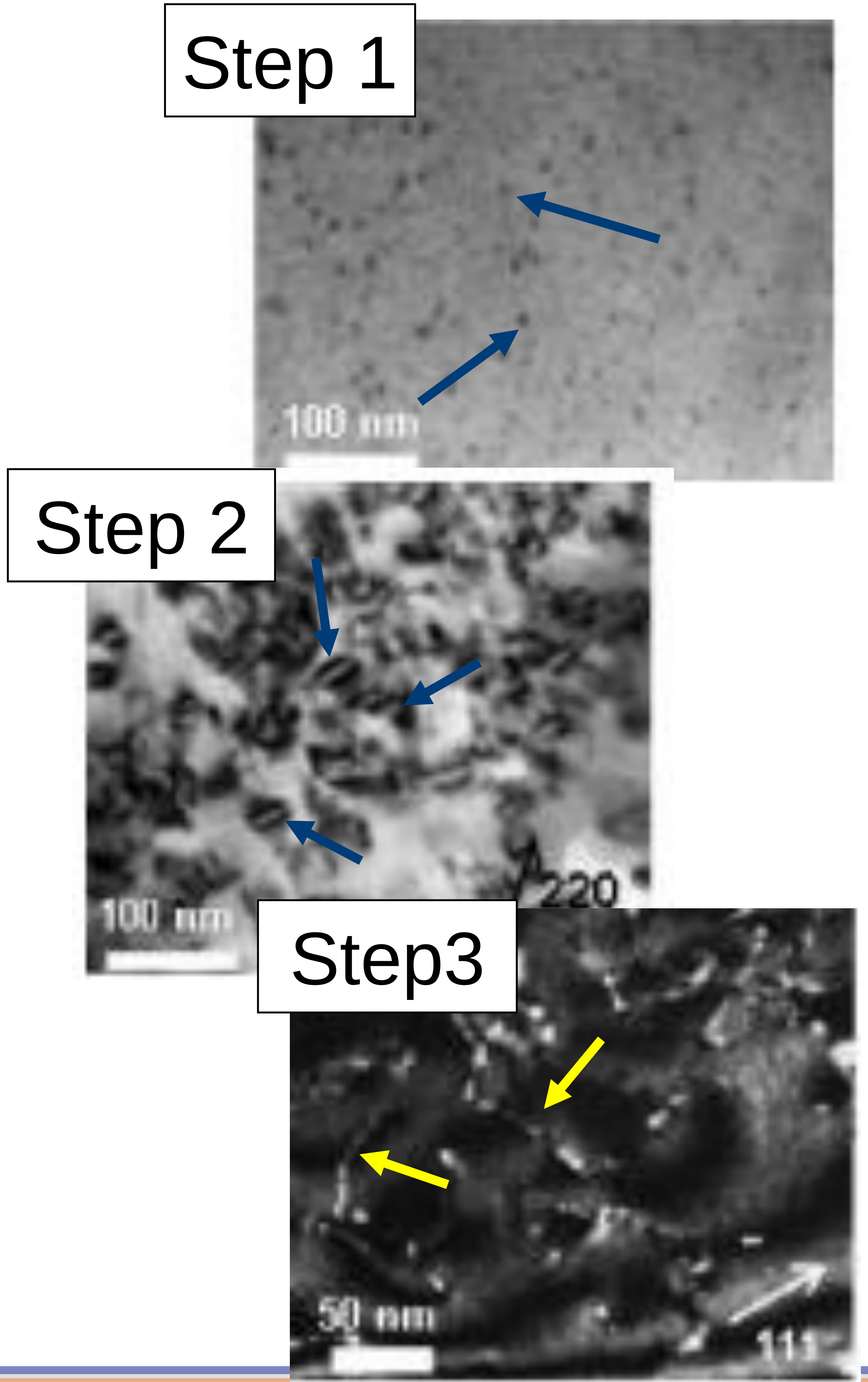
Outline of Presentation

1. Yttria stabilized Zirconia (YSZ) as nuclear ceramic and microstructural strategies for mitigating radiation damage



2. Preliminary in-situ observations

Irradiation of single crystal cubic YSZ



Damage accumulation in fluorite-structured YSZ occurs in **three different stages**

1. Formation of isolated defects
2. Rapid damage accumulation as the defects link or coalesce
3. Final saturation stage were ordering occurs and **defects begin to disappear**

Adapted from Thomé et al._2011_Acta Physica Polonica A

How do you increase the radiation of cubic YSZ?

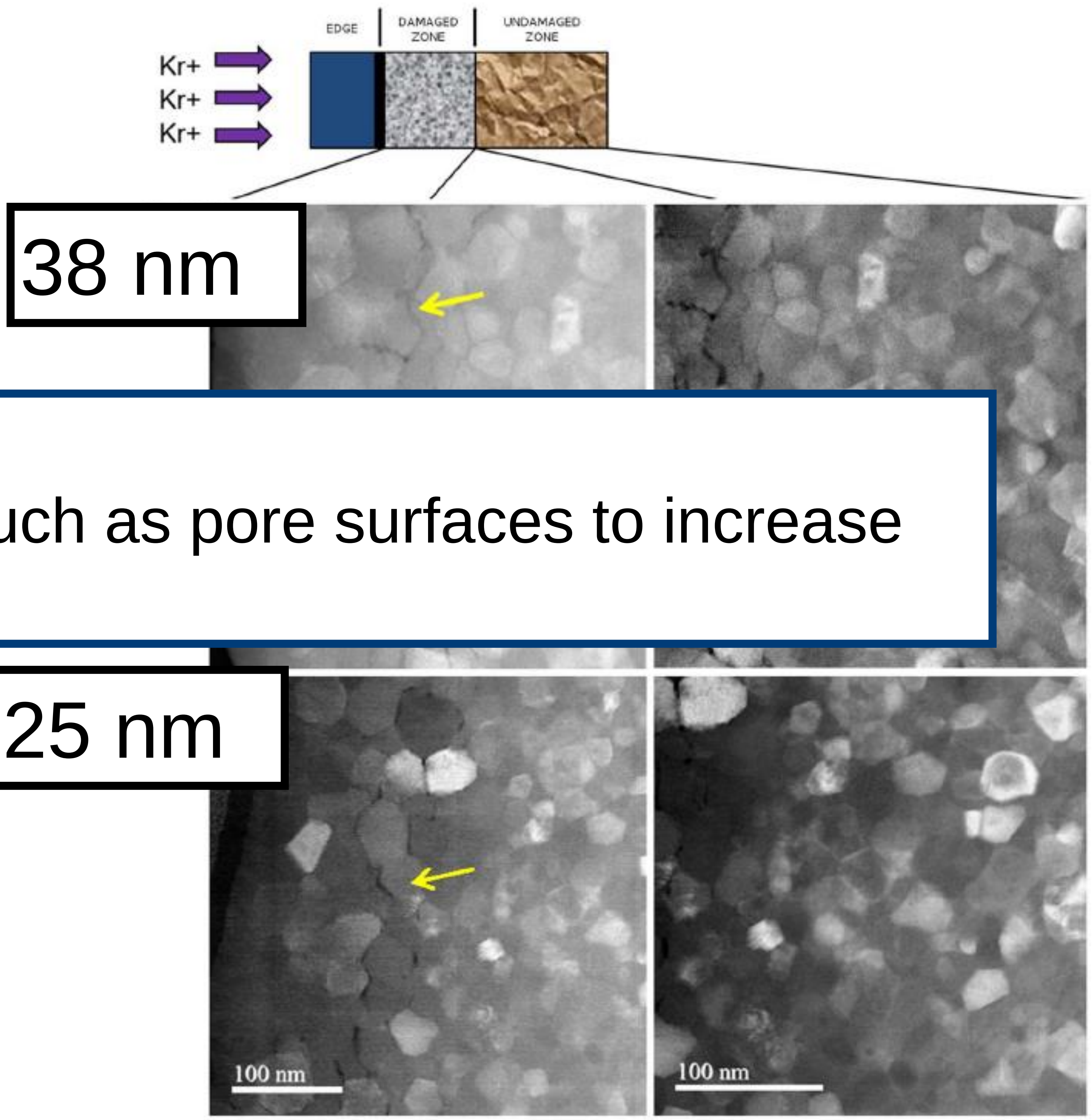
Increase the amount of interfaces

STEM DF Images

10YSZ Irradiated
with 400 keV Kr

Fl

R

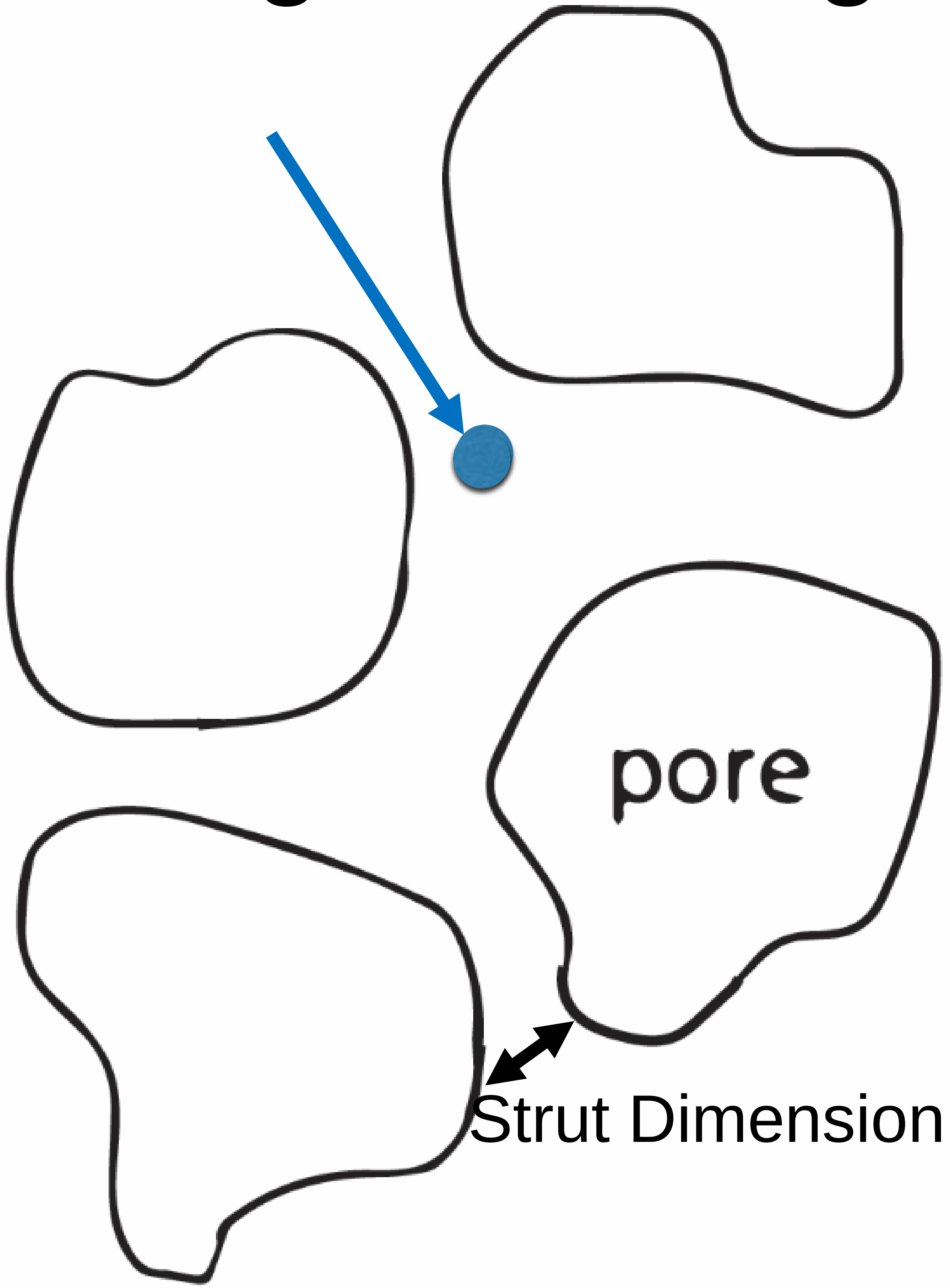


Question:
Can we use a different interface such as pore surfaces to increase radiation tolerance?

Major Observations
- Fewer defects observed in nanograin YSZ than bigger grain YSZ
- Yellow arrows indicate intergranular crack due to coarsening of grains

Adapted from Dey *et. al.* Scientific Reports (2015) 1-9

Thoughtful design of microstructure is needed



Example microstructure

Cascade Event cause by radiation

Defects must be able to diffuse to a pore surface (removes defect)

Strut dimension is the parameter in the microstructure that must be controlled

By controlling the critical parameter microstructure radiation tolerance can be improved

Theoretical Radiation Endurance Zone

Y-axis

Diameter of Struts

X-axis

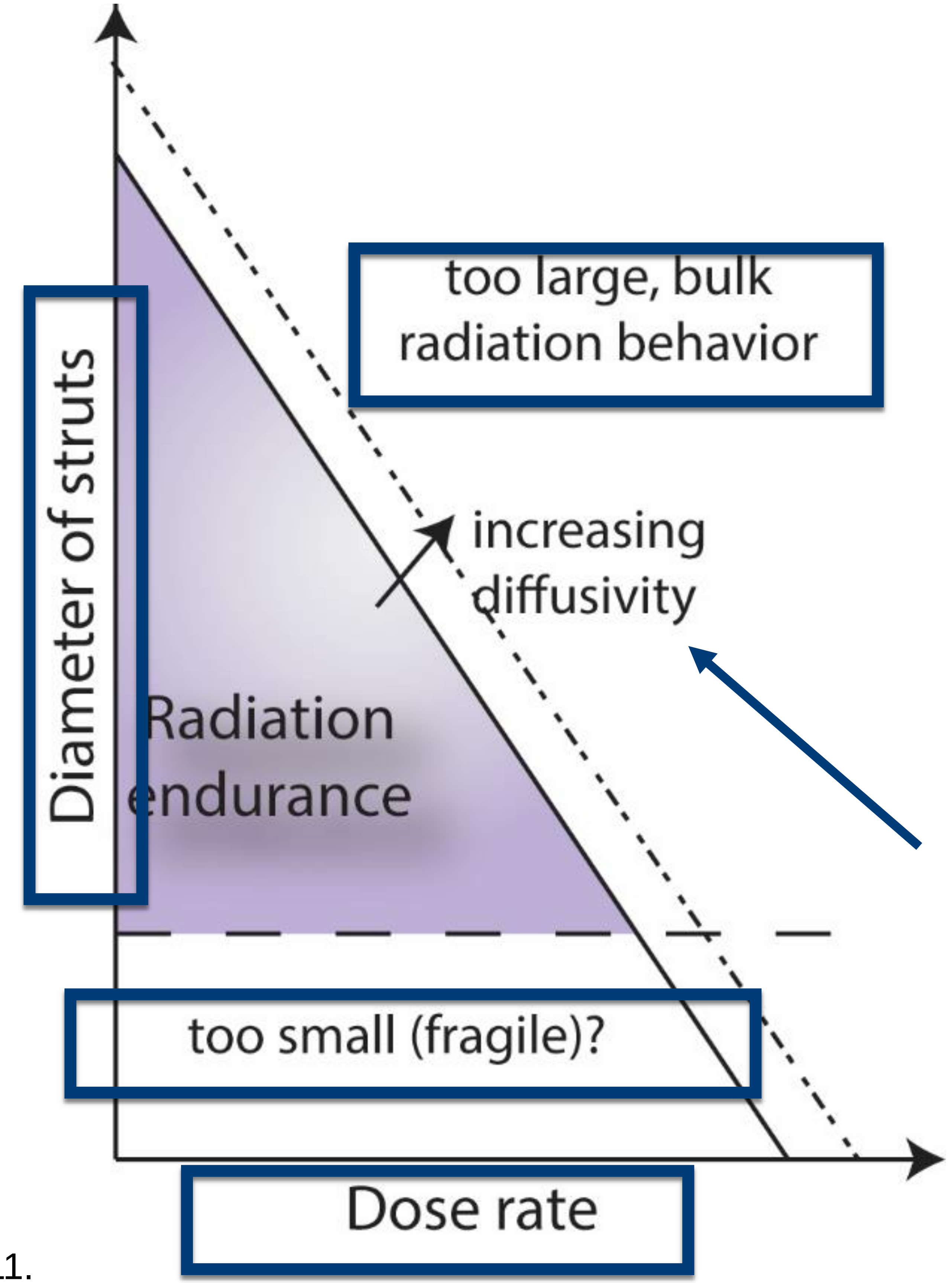
Dose rate (ion current)

Line at an angle (function of defect diffusion)

If x-axis and y-axis are balanced, material becomes is in radiation endurance zone

If **dose rate** is **too high** or **diameter** of struts **too big,** bulk radiation behavior

If diameter of strut too small, the materials is not mechanically stable



Adapted from E. Bringa *et. al.* Nano letters, vol. 12, no. 7, pp. 3351–3355, 2011.

Theoretical Radiation Endurance Zone

Y-axis

Diameter of Struts

X-axis

Dose rate (ion current)

Line at an angle (function of defect diffusion)

If x-a…

mate…

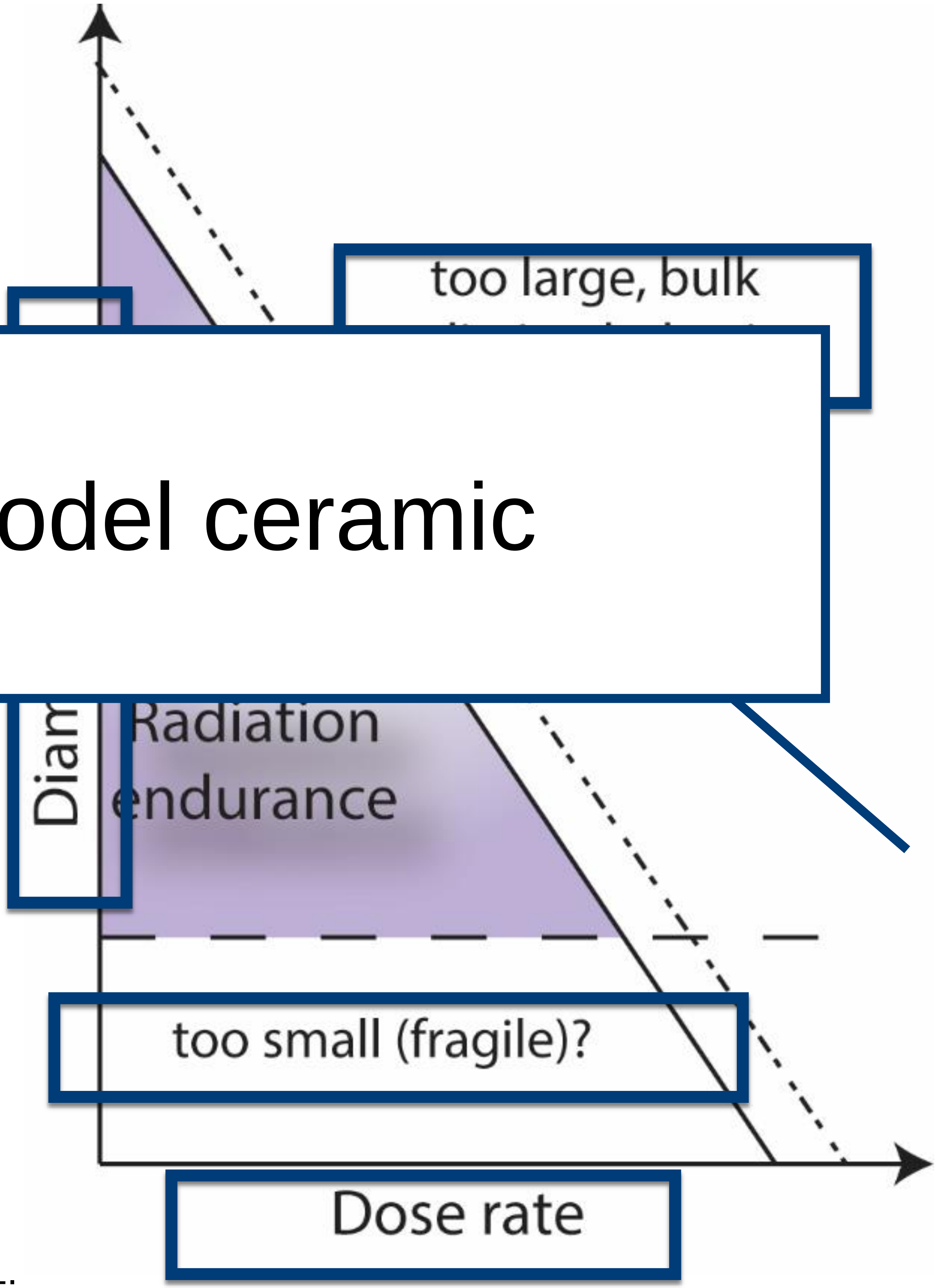
endu…

Question:

What does this look like in the model ceramic system?

If **dose rate** is **too high** or **diameter** of struts **too big,** bulk radiation behavior

If diameter of strut too small, the materials is not mechanically stable



Adapted from E. Bringa *et. al.* Nano letters, vol. 12, no. 7, pp. 3351–3355, 2011.

In-Situ Observation of Defect Evolution

Sandia National Laboratories

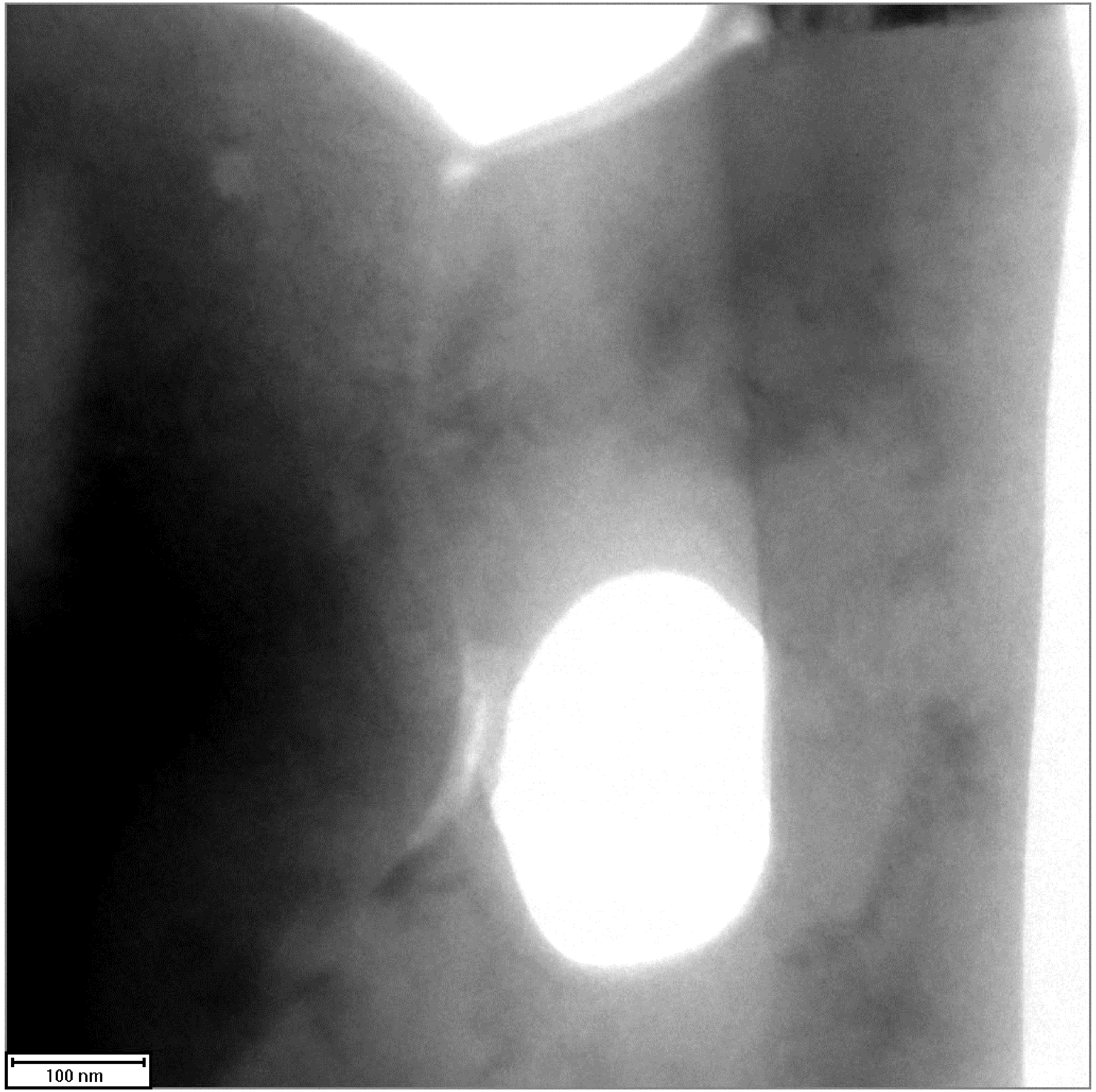
I3TEM = In-Situ Irradiation capabilities

Allows for direct observation of defect migration to free surface

energy.gov

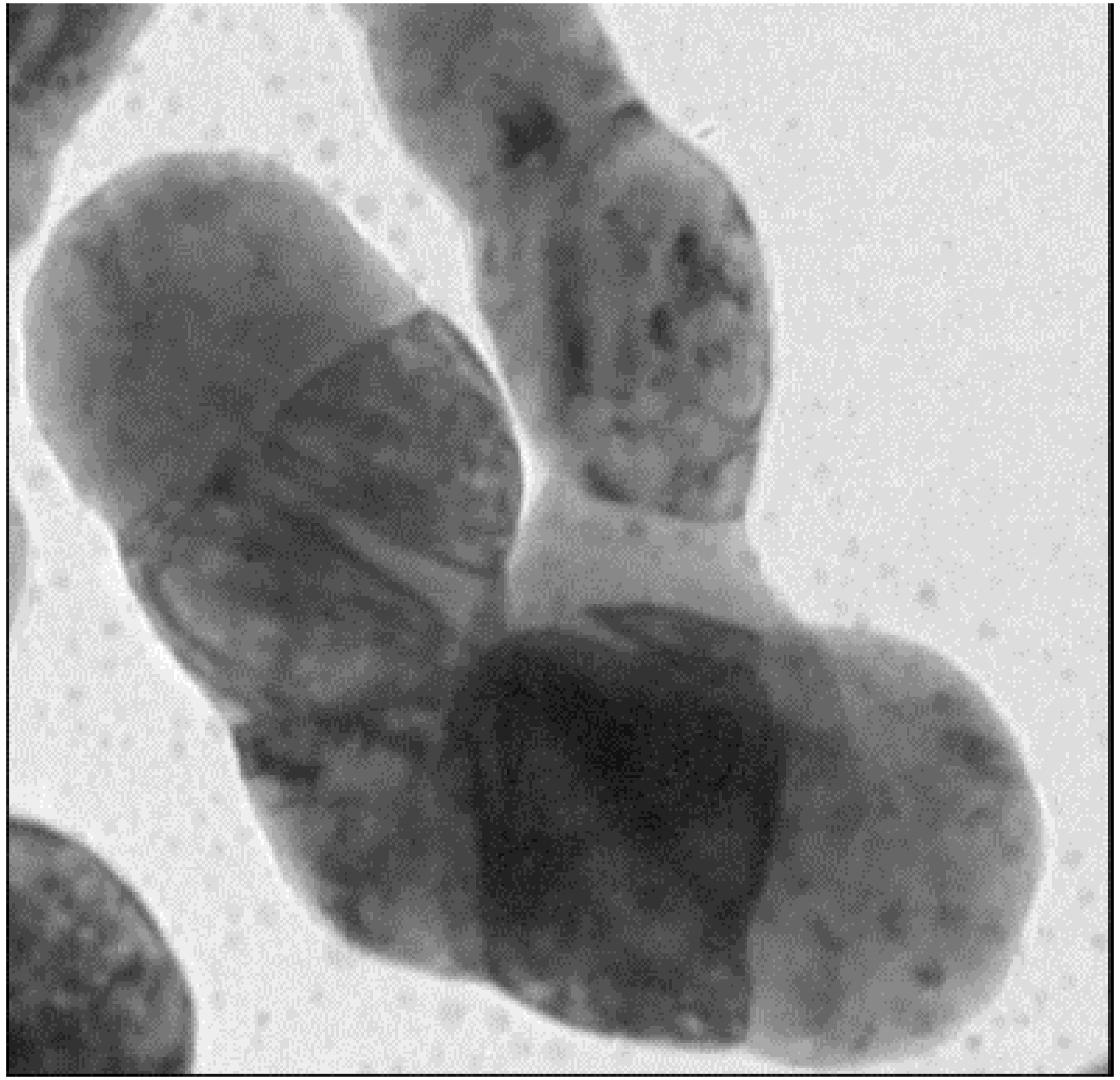
K. Hattar et al. / Nuclear Instruments and Methods in Physics Research B 338 (2014) 56–65

In-Situ Observation of Defect Evolution



- Images taken every 5 minutes
- Defects come in and out
- Microstructural changes
- Allows for direct observation of defect migration to free surface

This is an established method

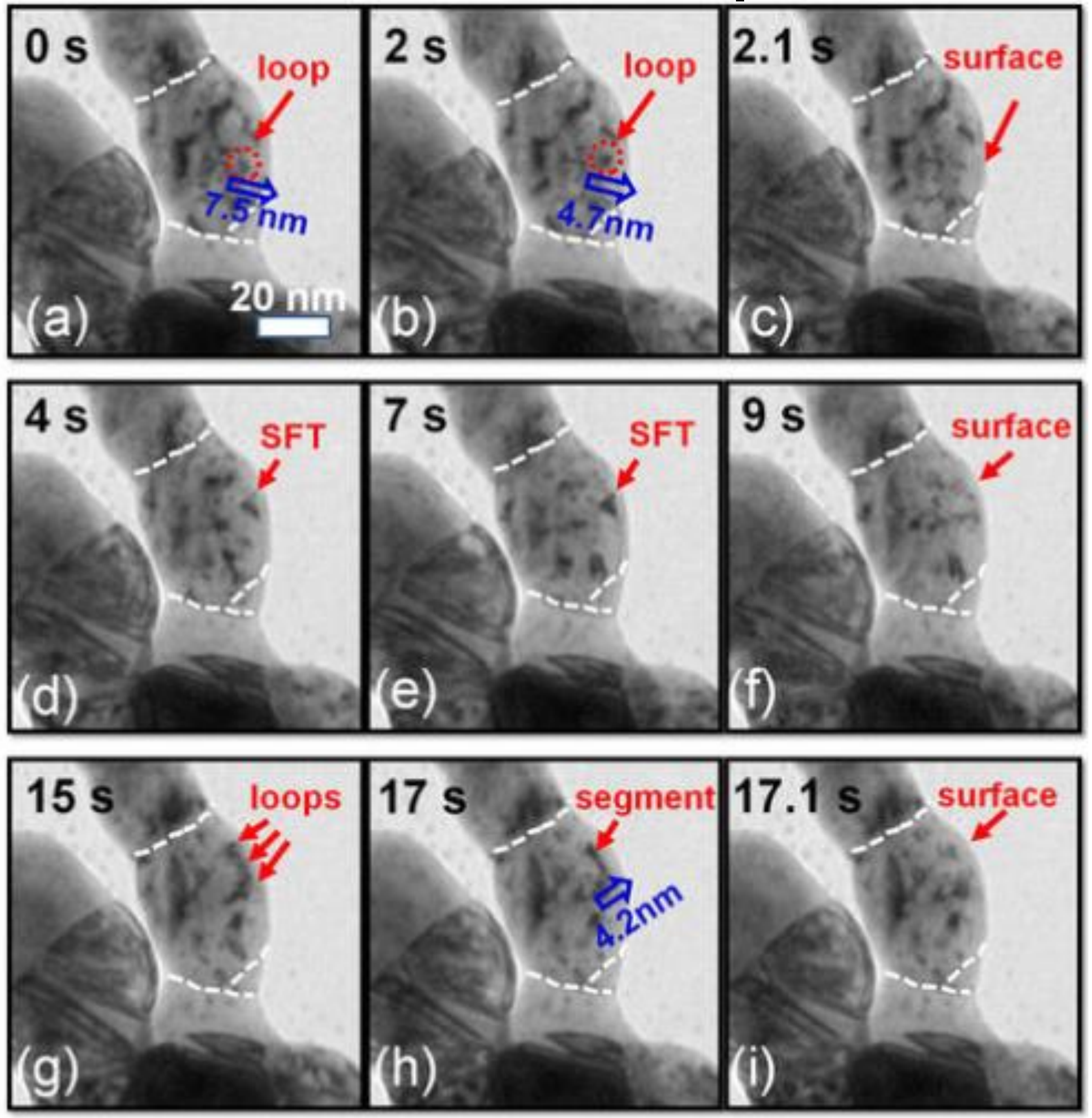


In-Situ video showing capture events

Ag irradiated with Kr

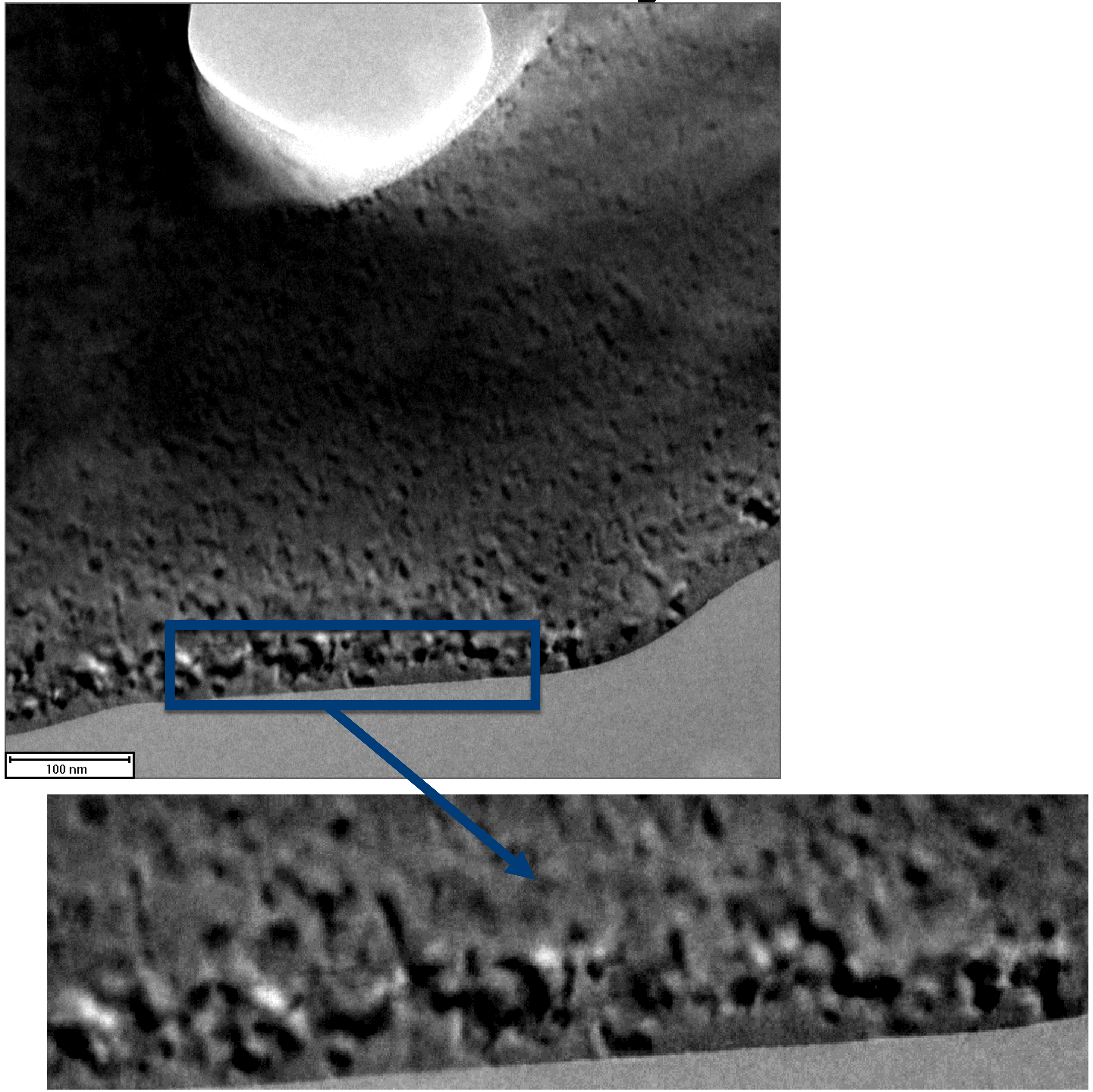
Room Temperature

1.18 – 1.27 dpa



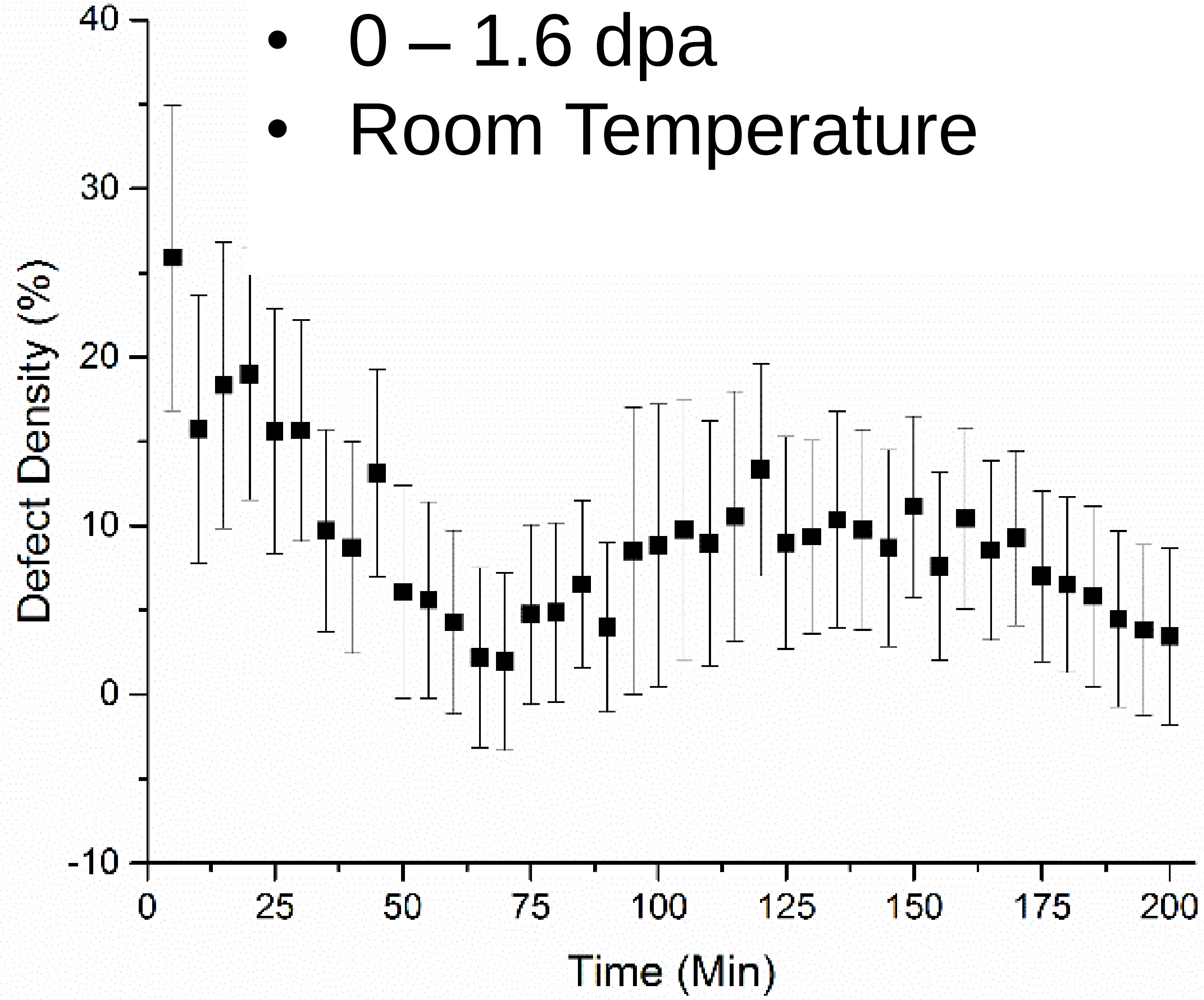
Adapted from C. Sun et. al. *Scientific Reports* **4**, Article number: 3737 (2014)doi:10.1038/srep03737

“Defects Density” near a free surface

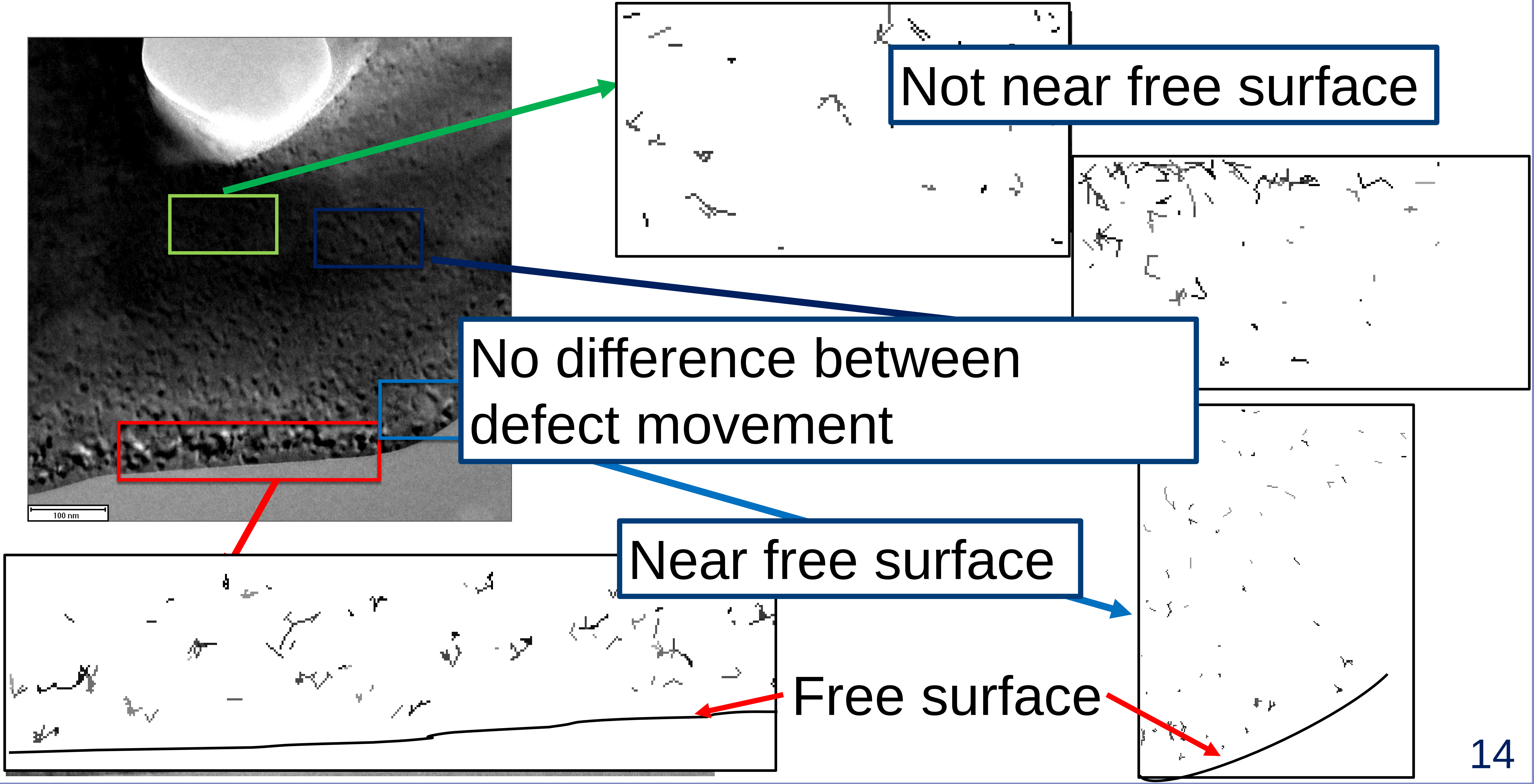


Irradiation Conditions

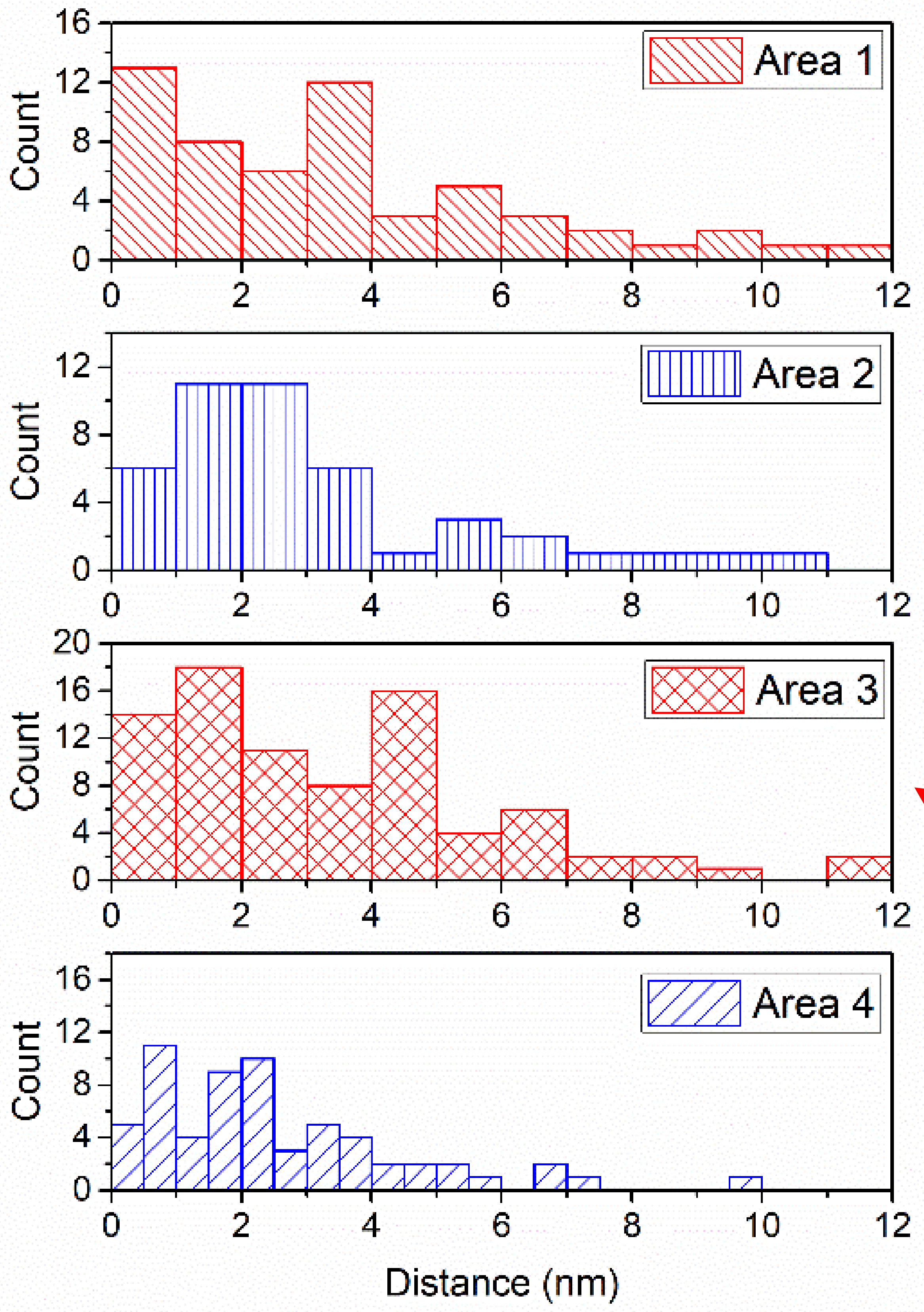
- Energy of ion: 2.8 MeV Au ions
- Ion Current: 1.5 nA
- 0 – 1.6 dpa
- Room Temperature



Defect Movement as function of time



Histograms for total displacement



Not near free surface

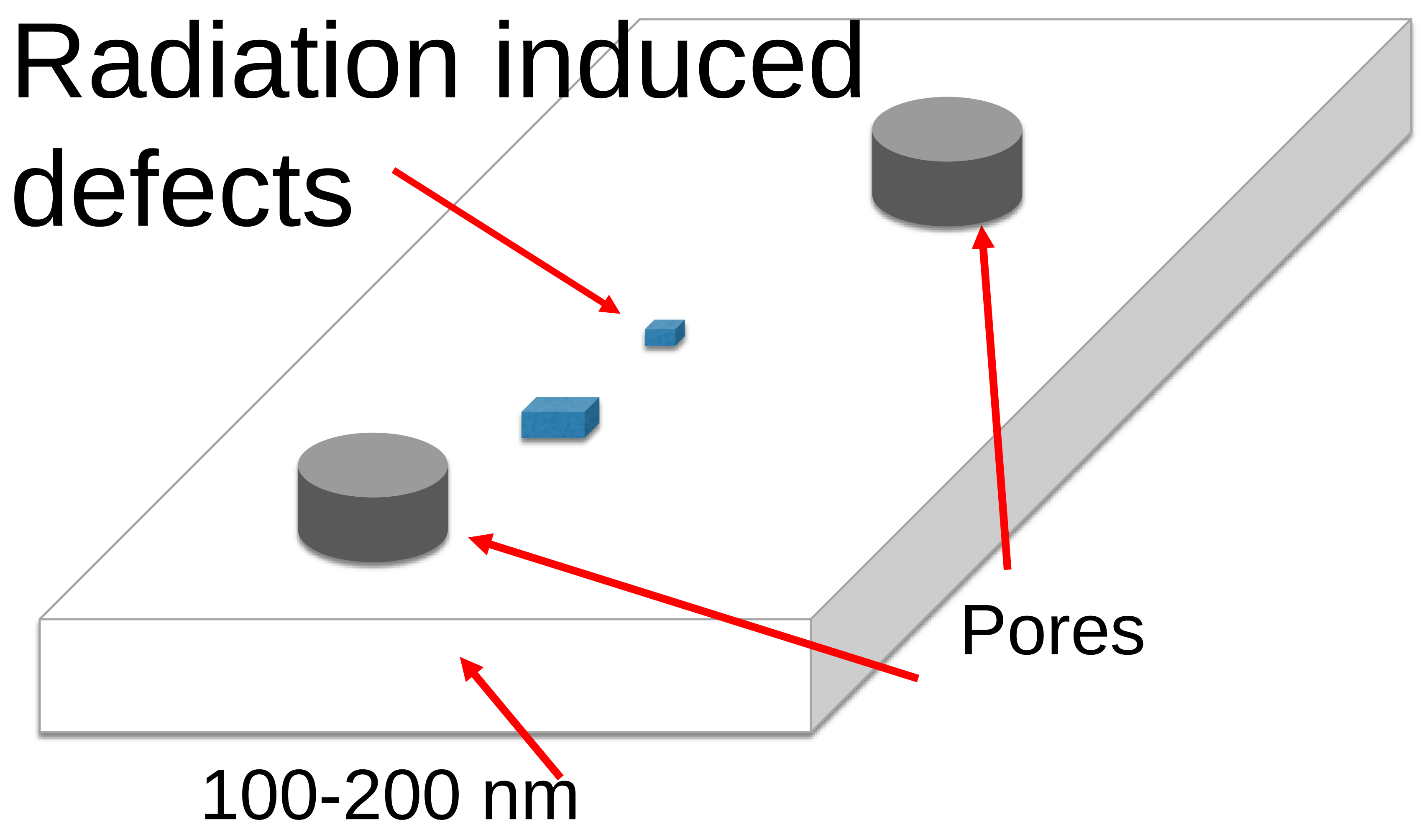
Confirms that there is no net movement toward free surface

near free surface

Discussion

Defects tend to move to the free surface of the TEM lamella versus the surfaces of pores

TEM lamella



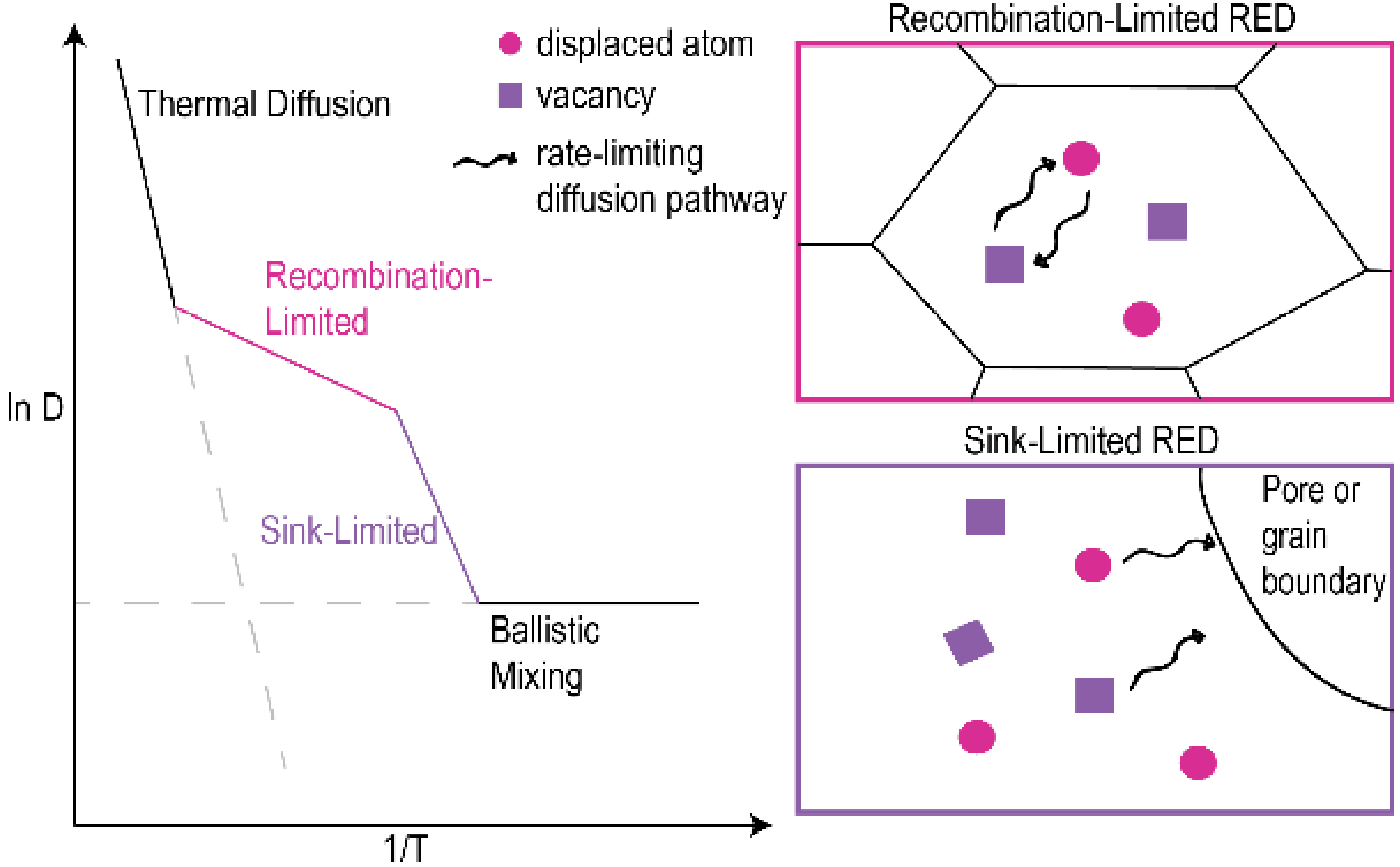
Future Work

Objectives

(i) identify critical microstructural dimensions as a function of temperature

(ii) confirm RED mechanisms in YSZ (qualitative)

(iii) correlate the first two objectives with the evolution of microstructural features.

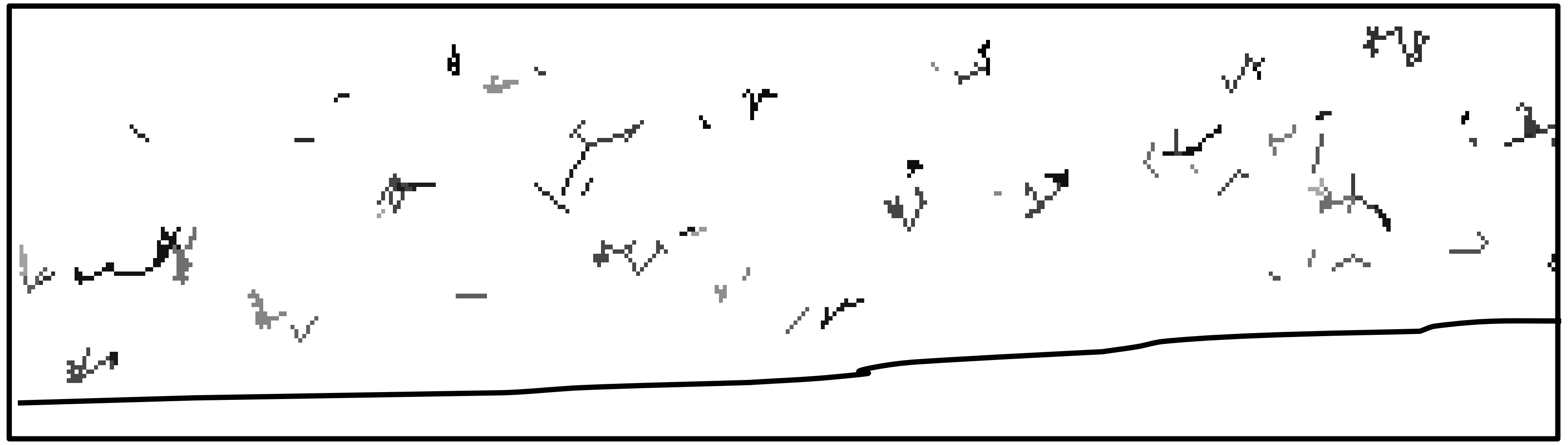


Rate limiting defect annihilation mechanisms as a function of temperature

Exploring different Kinetic regimes for damage recovery

CONCLUSION

1. Yttria stabilized Zirconia (YSZ) as nuclear ceramic
2. Our microstructural strategies for mitigating radiation damage (using pores)
3. Developed baseline defect behavior for in-situ TEM experiments at room temperature



SMEE Group Website
Krogstad.matse.Illinois.edu

Backup Slides

Initial Proposed Experiments

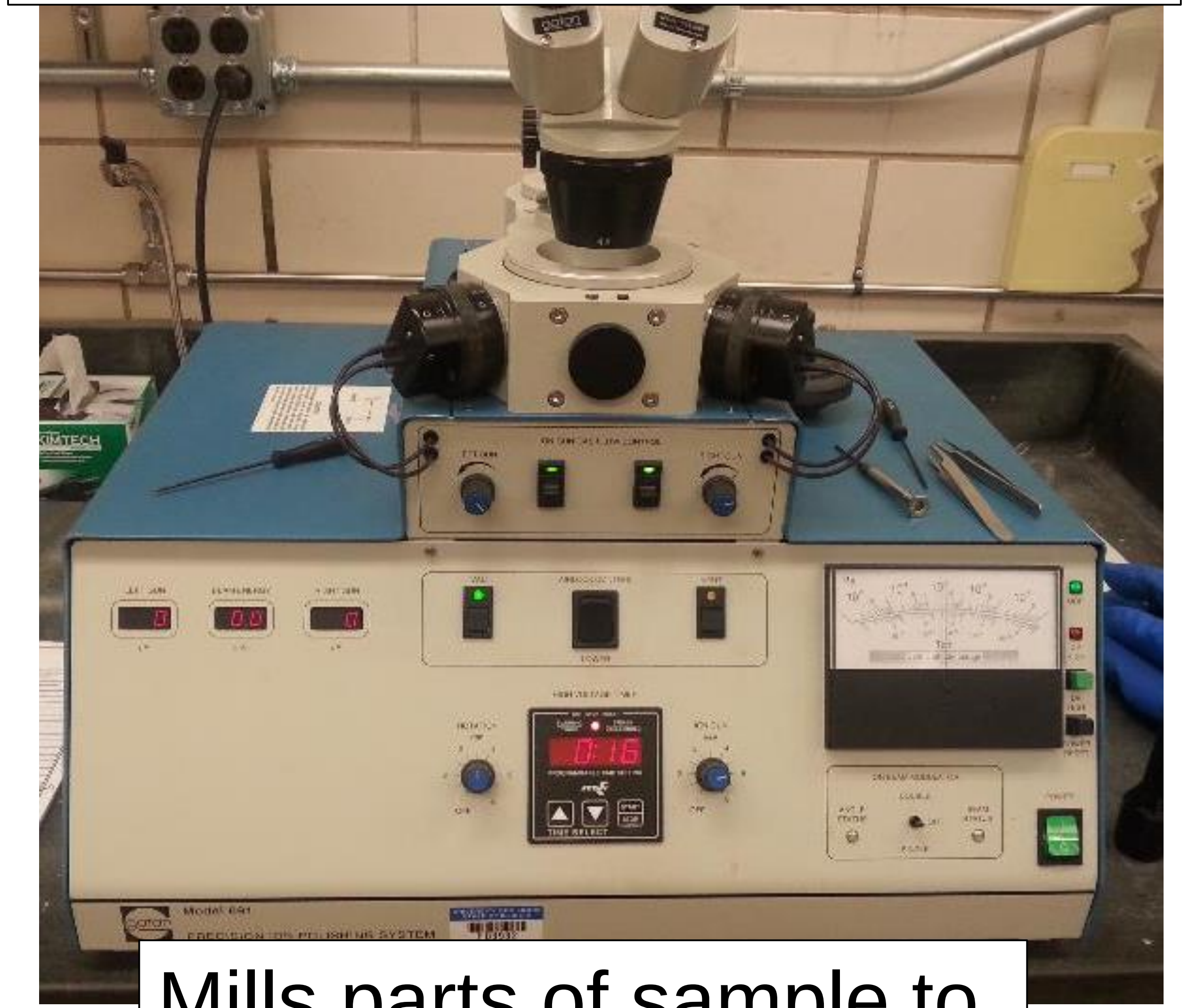
The first on-site experiments will utilize the heating stage to probe three initial temperature regimes: low (~300ºC), intermediate (~700ºC) and high (~1000ºC) under the similar ion beam conditions

Sample Preparation

Multiprep => Mechanical Polish

Section pellets and then thin 30 to 70 μm

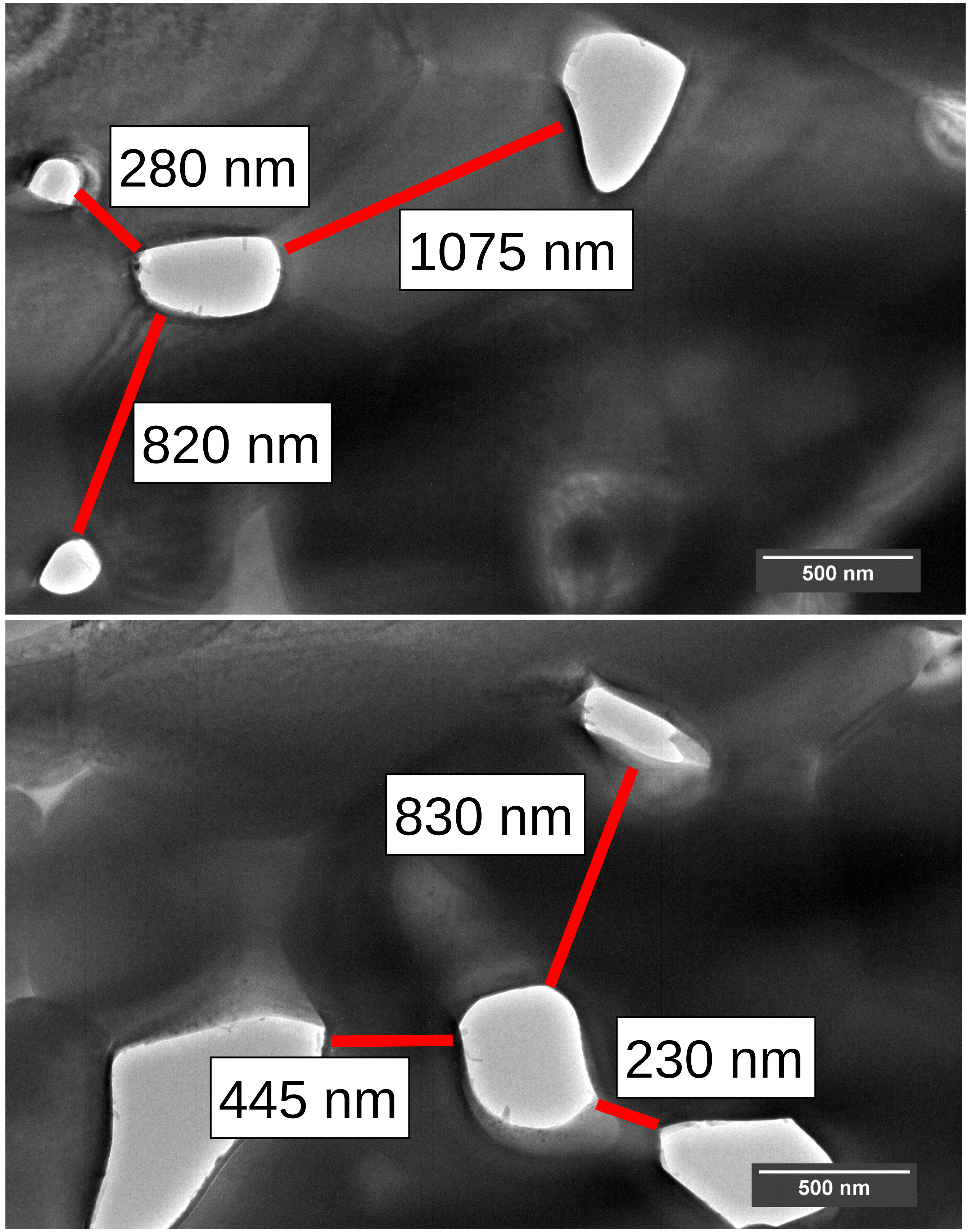
PIPs => Low energy ion milling



Mills parts of sample to electron transparency

PIPsed for 5 hours at 5 keV with $\pm 5°$ with a rotation of 3 rpm

Real Microstructures



Ceramic pellets where produce through traditional ceramics processing

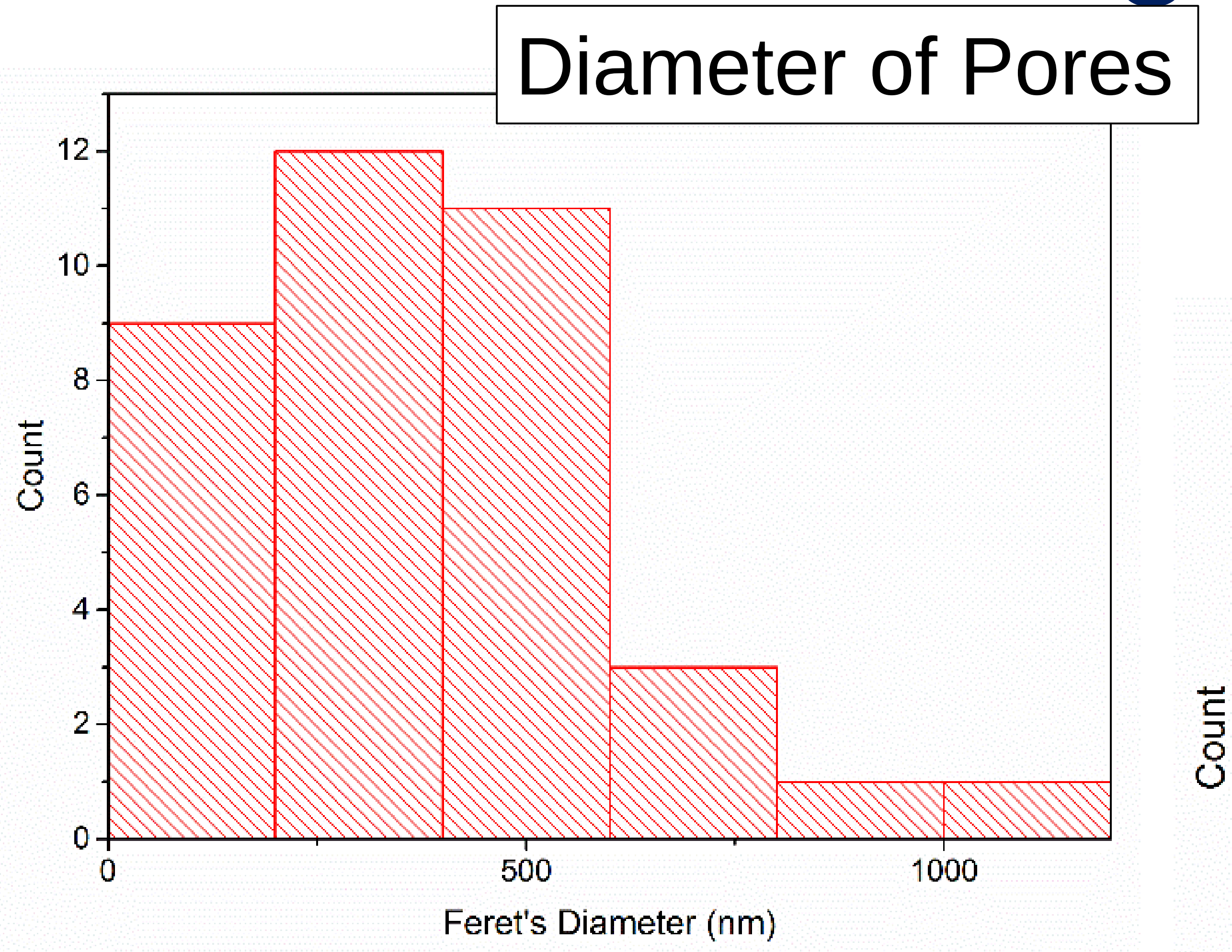
- Ceramic powder produced through precipitation
- Dry pressed and sintered in air

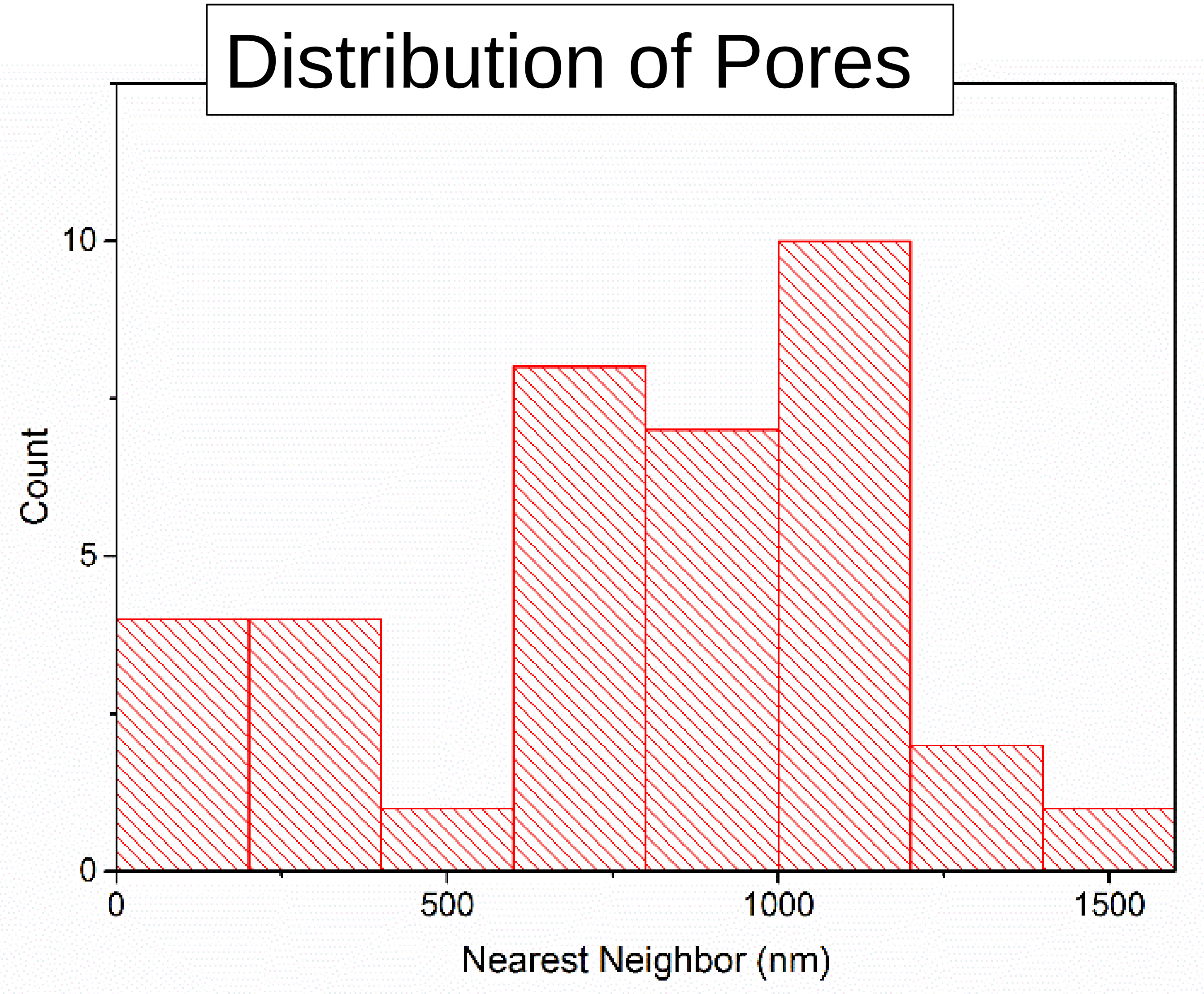
TEM samples prepared by:

- Sectioning pellets with diamond saw
- Thin cross-section followed by low energy ion polish

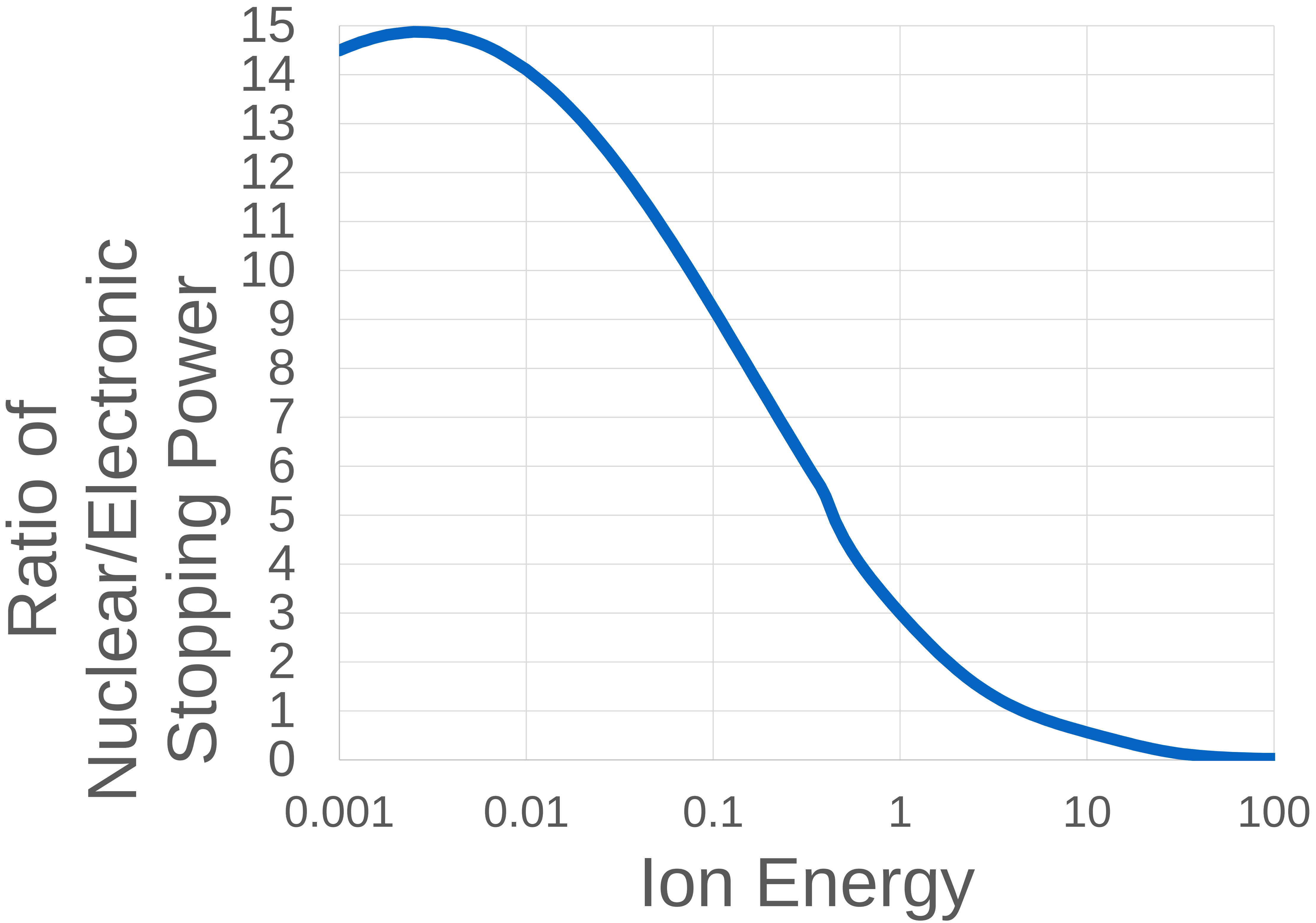
Range of strut lengths in microstructure

A little bit of statistics on pores from TEM micrographs



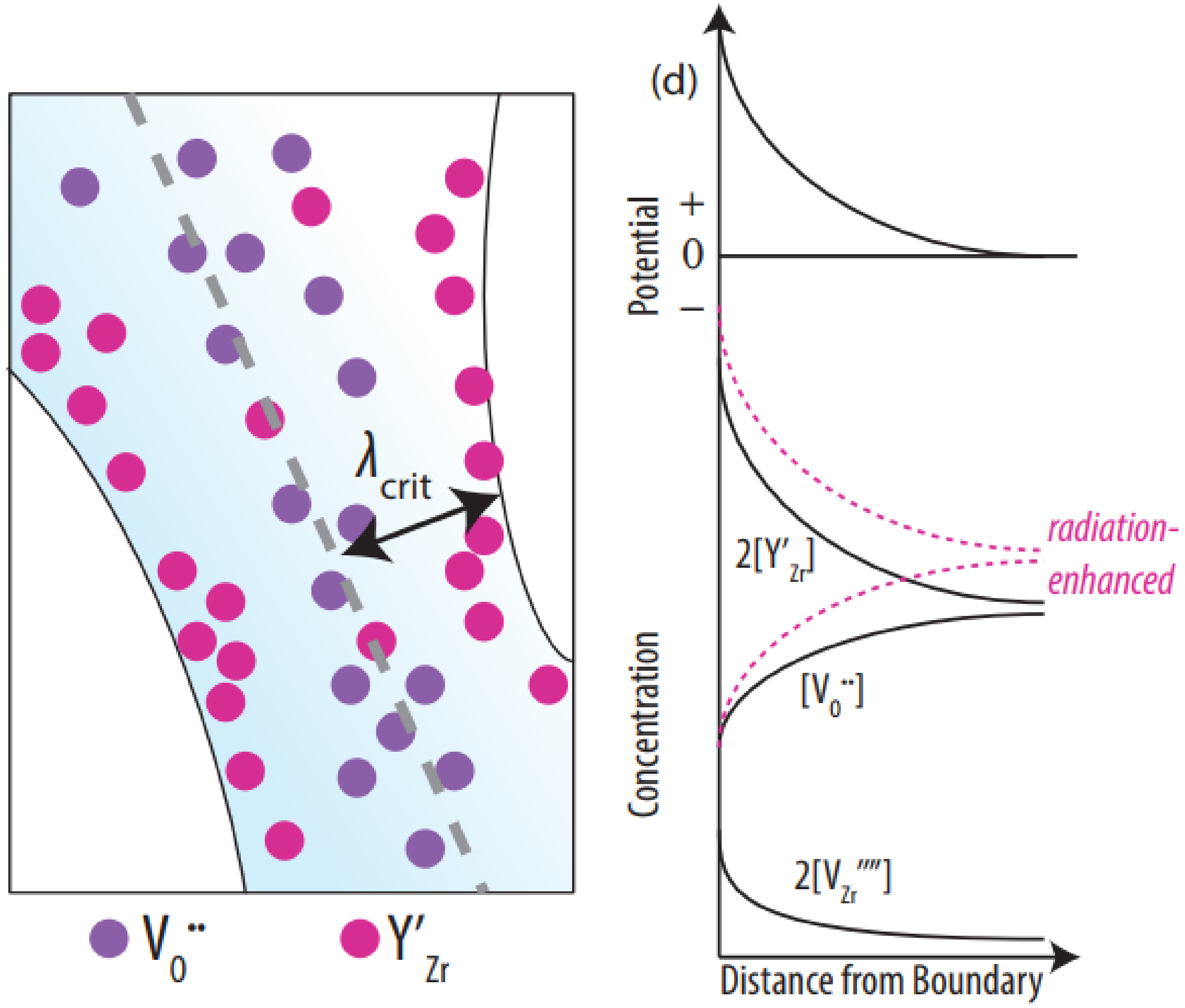


Stopping Power



Gold in YSZ Ratio of Stopping power is ~1.5

But, the porous microstructure must be stable…



Naturally occurring space charge enhancement of aliovalent dopants near surfaces may be further enhanced by RED, effectively pinning the boundaries