Microstructural Evolution of Nanocrystalline Nickel Thin Films due to High-Energy, Heavy-Ion Irradiation

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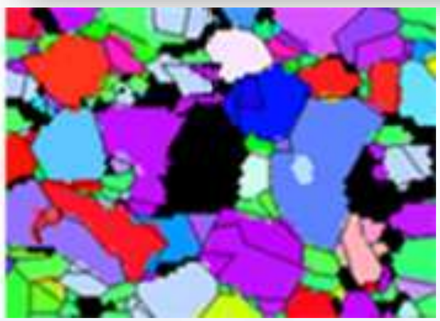
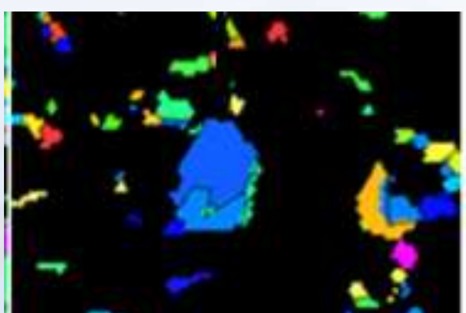
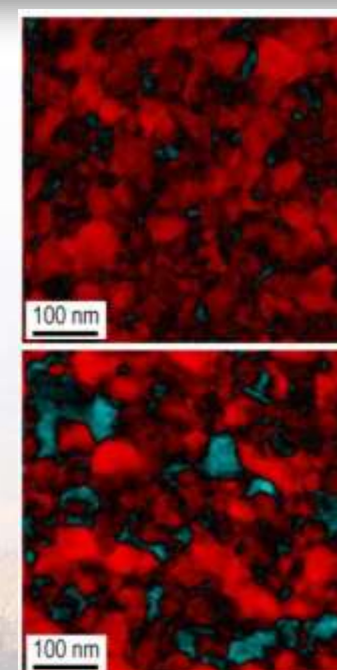




Outline

- Nanostructured metals provide the potential for radiation tolerant design
- Far-from-equilibrium nanograined metals can exhibit very unique and unexpected defect structures
- Thermal, mechanical, and radiation properties are probably related
- Precession microscopy provides a way to characterize grain orientation and grain boundary at the smallest scale
- *In situ* ion irradiation provides the potential to directly watch the evolution



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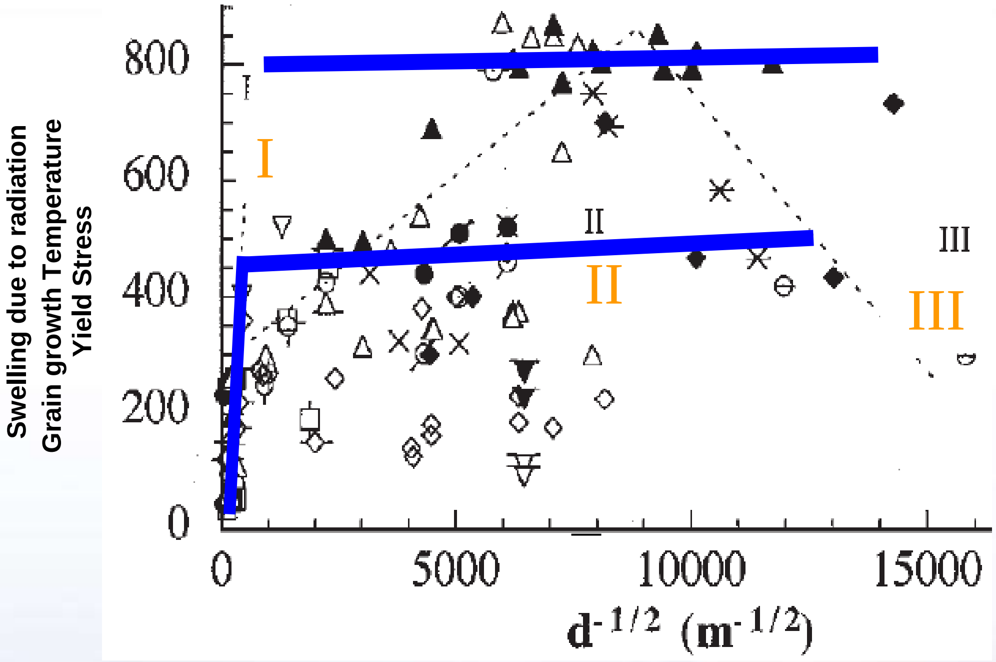
Properties and Mechanisms Active in Nanograined Metals



Conrad, Metallurgical and Materials Transactions A: 2004. 35 p. 2681

Due to the variations in:

- Production methods

→ Range of microstructures

- Testing methods

→ Range of experimental uncertainty

Key overlooked factors

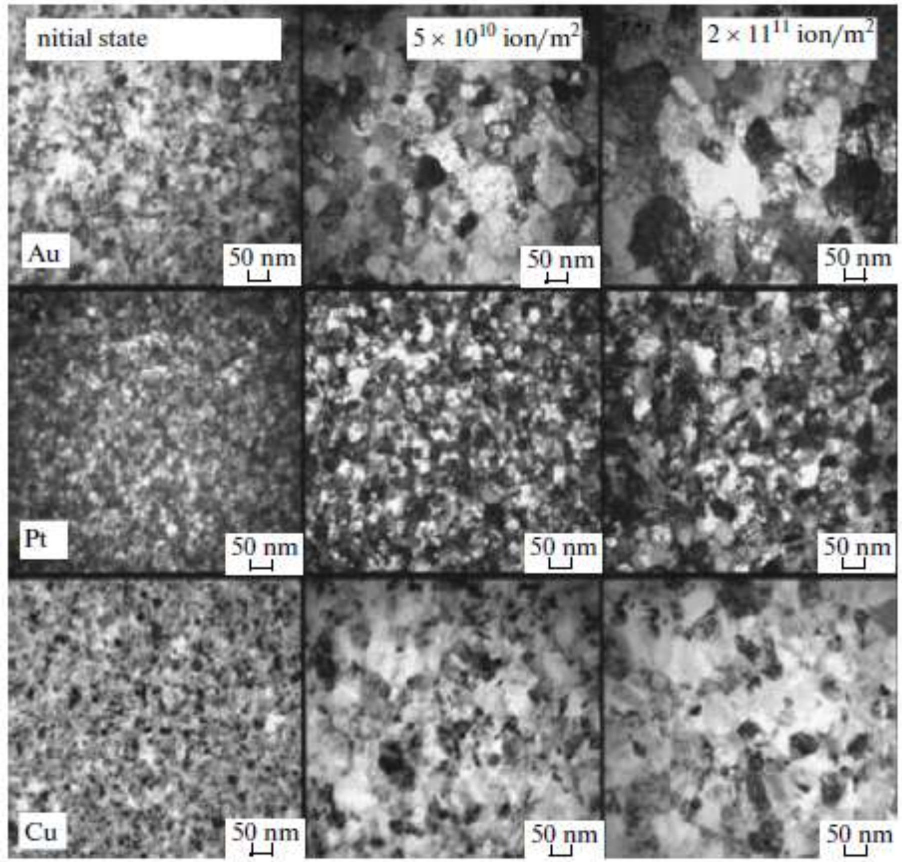
- Grain boundary type
- Relative grain orientation
- Grain boundary energy
- Etc.

1) What are the thermal and mechanical properties and how do they effect the radiation tolerances?

2) Beyond just grain size, what is the microstructure?

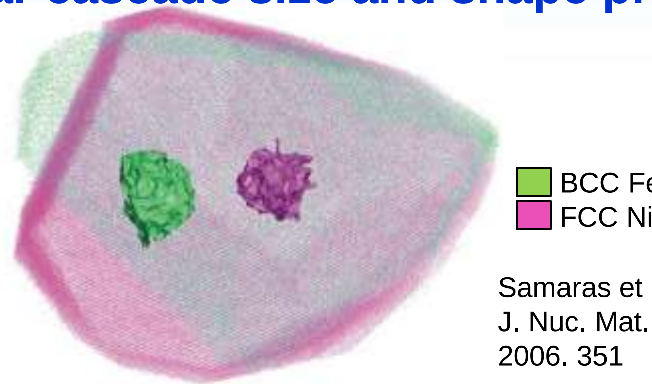
Radiation Tolerance from Nanograined Metals

Variation in radiation tolerances



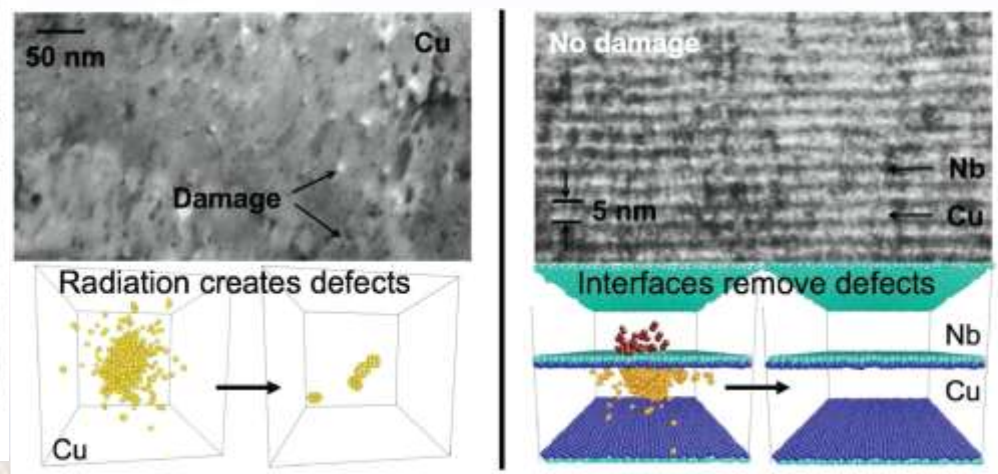
Kaomi et al., JAP: 2008. 104 073525

Similar cascade size and shape predicted



Samaras et al., J. Nuc. Mat.: 2006. 351

Nanolamellars are radiation tolerant

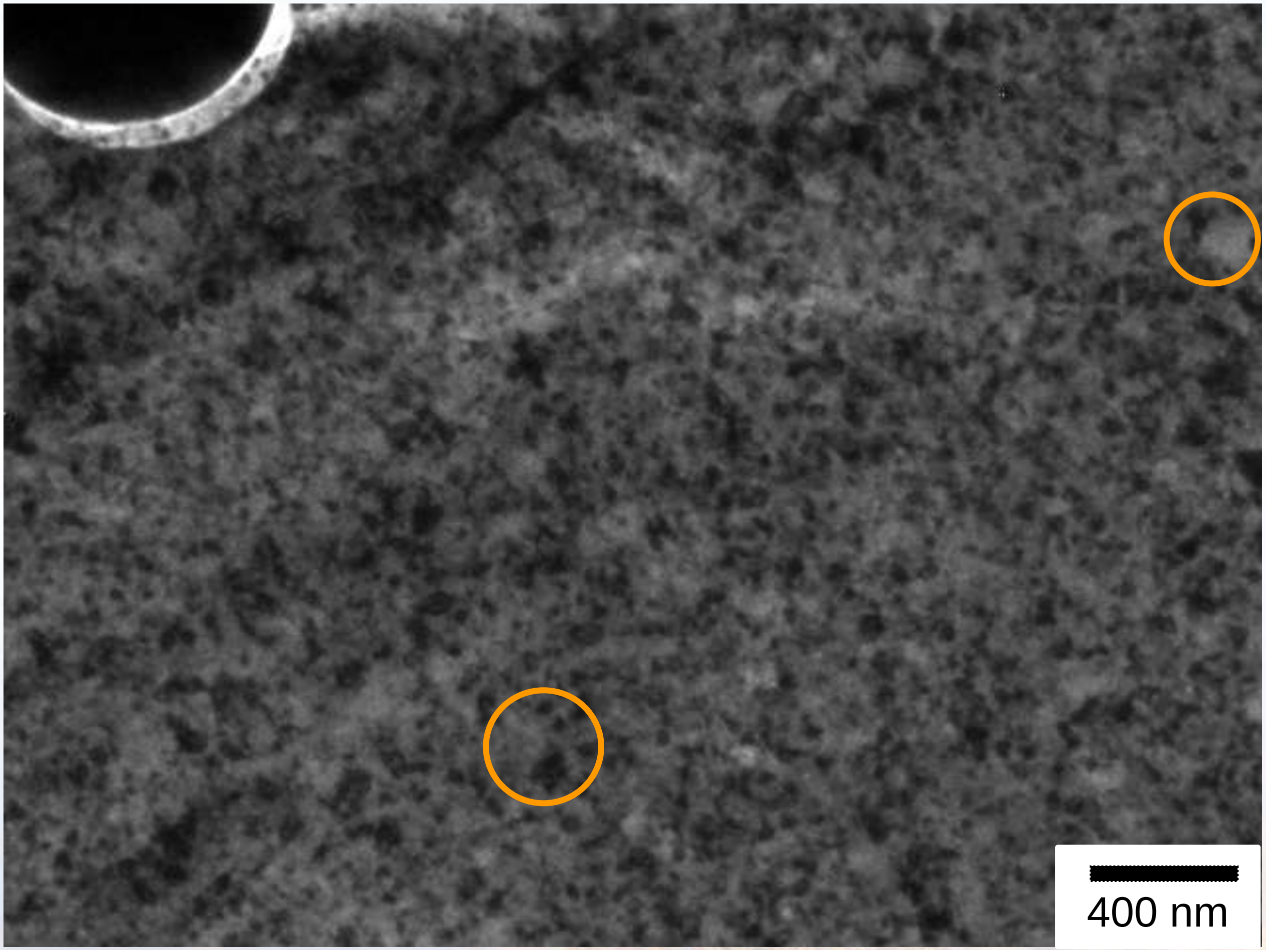


Demkowicz et al., MRS Bulletin: 2010. 35

To a first order mean grain size comparison, these reports appear conflicting.

This may not be the case if initial microstructural details and associated properties are considered

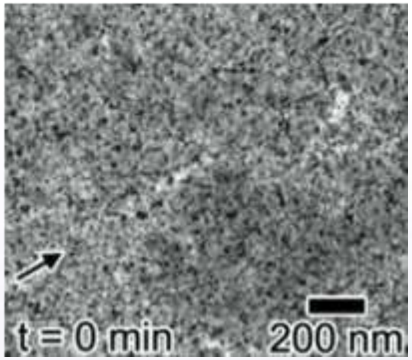
Thermal Stability of Nanocrystalline Materials

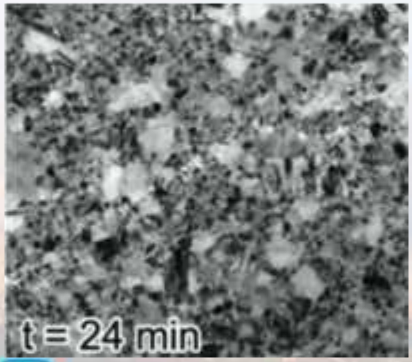


90 nm-thick PLD Ni during annealing at 350 ºC

Abnormal Grain Growth is a function of:

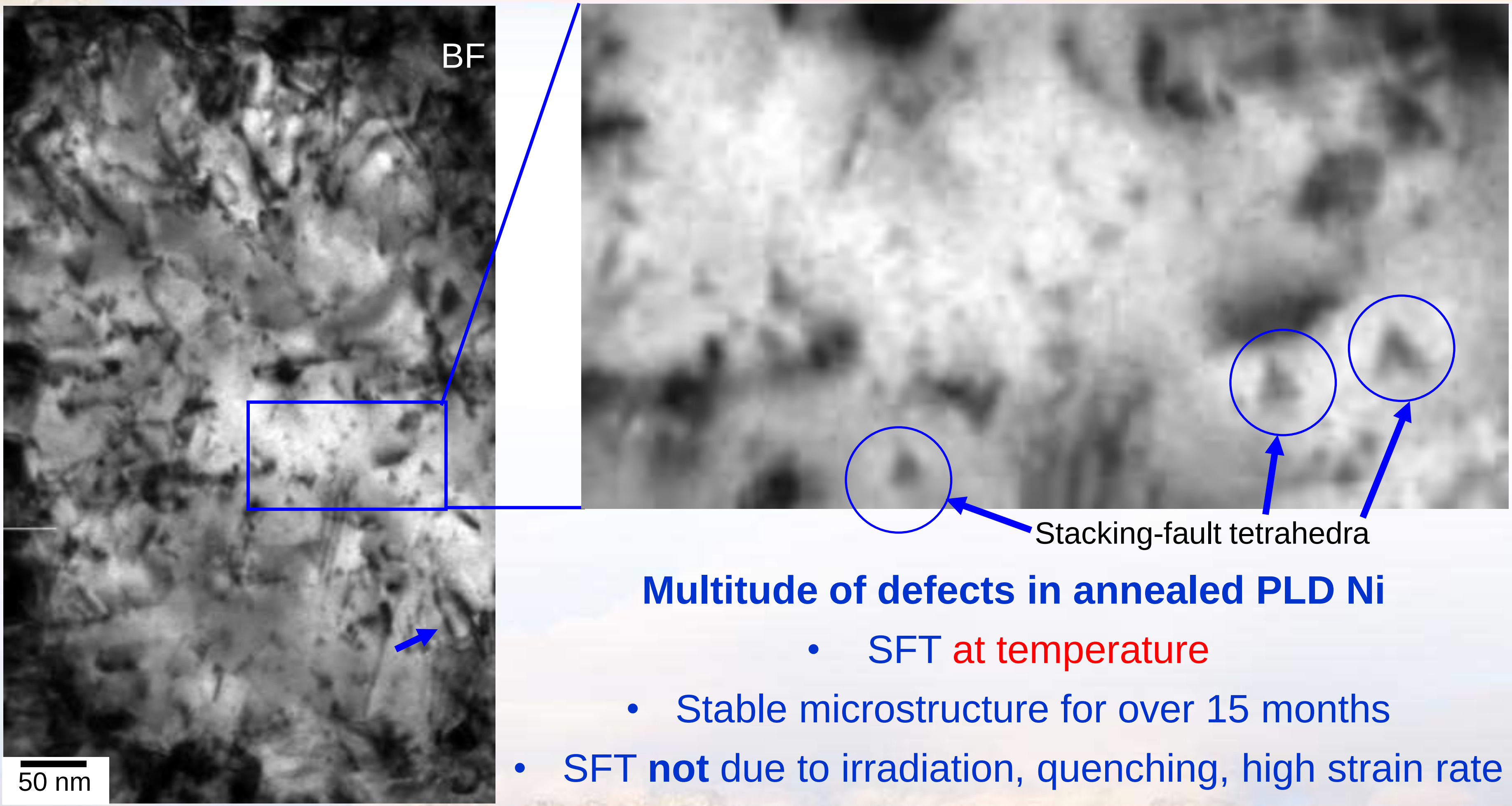
- Time
- Temperature
  - e^- beam
- Film thickness
- 4 years of Aging
- Surface Abnormalities
  - Ledges
  - Splats (No effect)





A few select grains grow at the expense of the remaining matrix

A Variety of Unexpected Defect Structures in Ni



230 nm thick PLD Ni Annealed at 225 ºC for 14 hrs.

Multitude of defects in annealed PLD Ni

- SFT at temperature
- Stable microstructure for over 15 months
- SFT **not** due to irradiation, quenching, high strain rate
- SFT are theorized to be formed by rapid grain growth through the high free-volume at the initial grain boundaries

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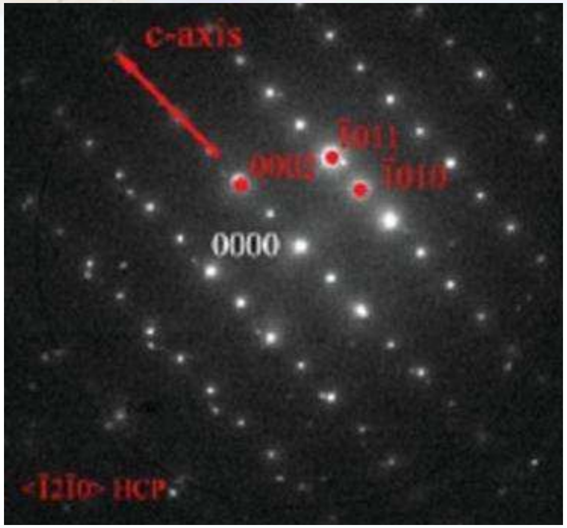
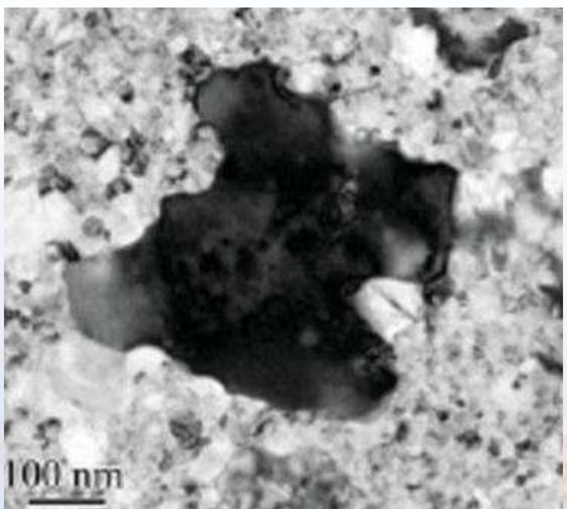
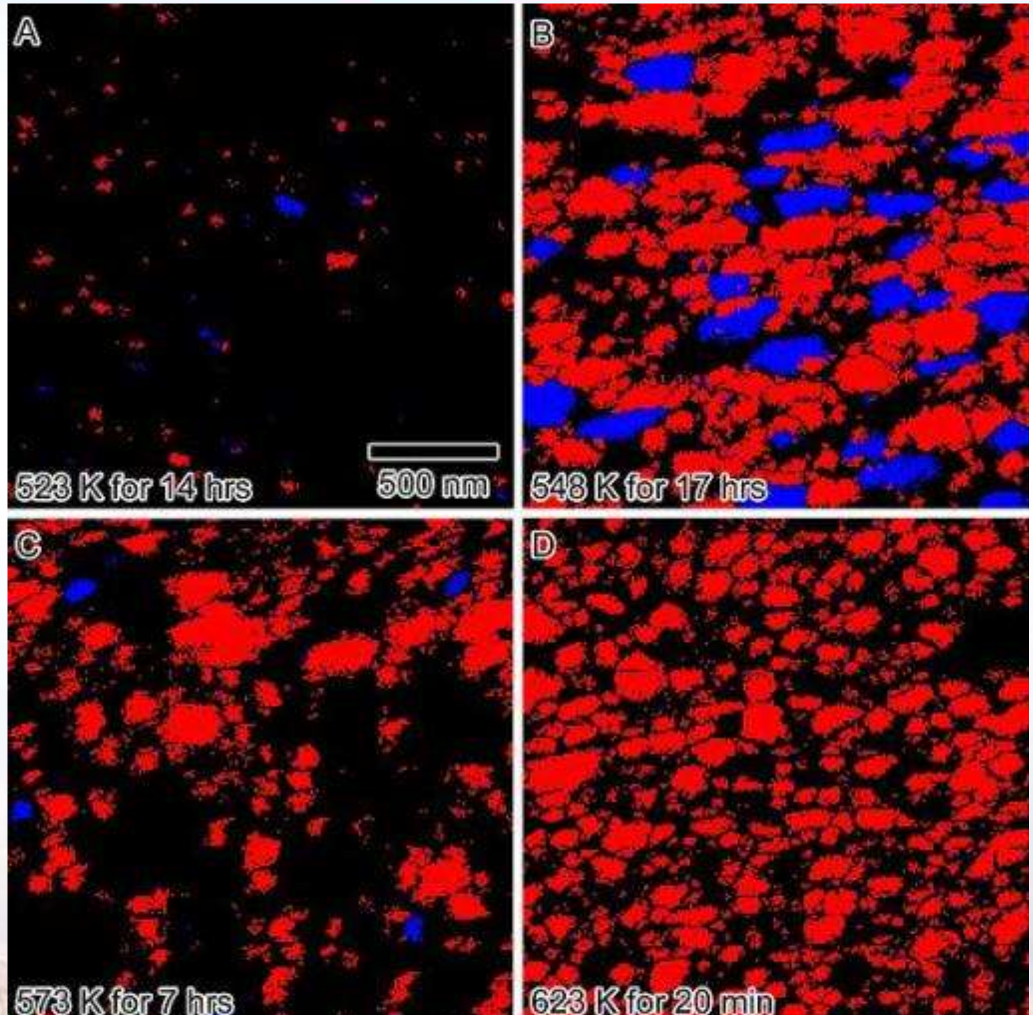
Thermal Stability of Nanocrystalline Materials: Evidence of *HCP* Phase Grains







FCC phase

HCP phase

However, Spatial resolution limits detailed analysis of the *HCP* phase evolution

EBSD and SAD confirm the presence of ***HCP*** phase in some abnormal grains

L. N. Brewer, D. M. Follstaedt, K. Hattar, J. A. Knapp, M. A. Rodriguez, I. M. Robertson, Adv. Mater. 22 (2010), 1161

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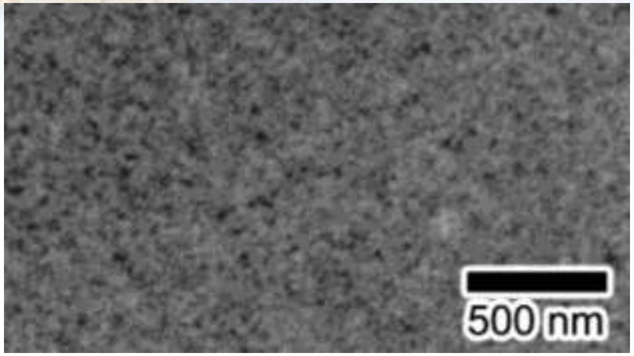
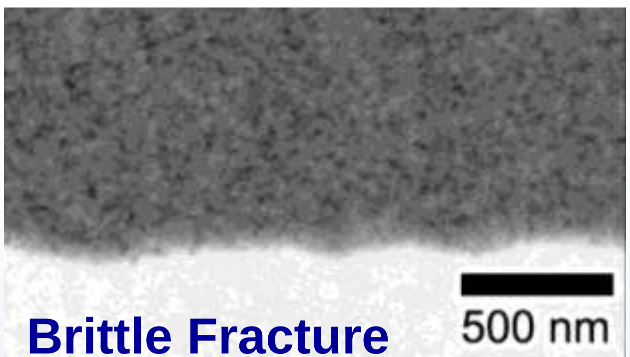
Failure Analysis of Strained PLD Ni

Nanograined

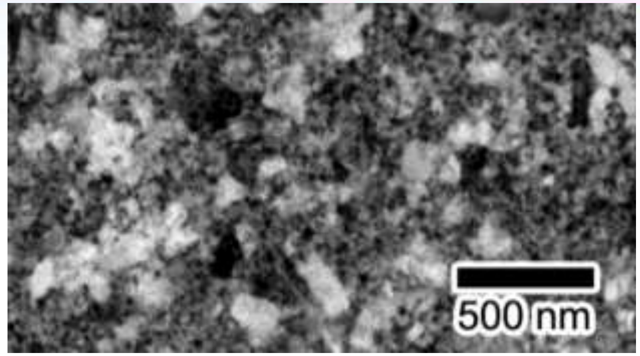


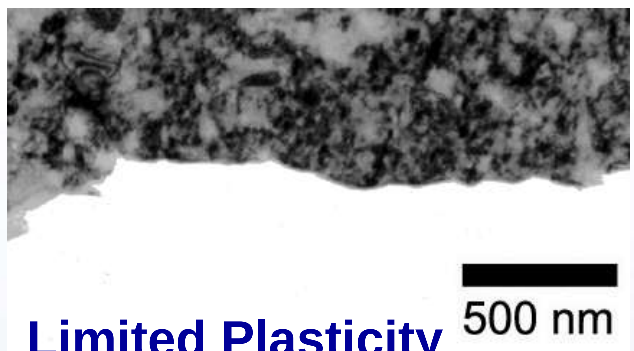


Brittle Fracture

- No observation of global plasticity

Bimodal

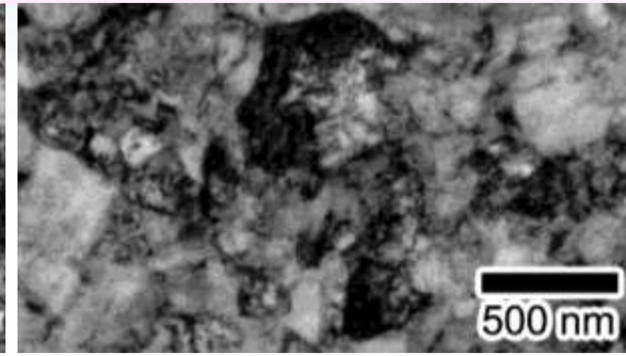




Limited Plasticity

- Dislocation pile-up
- Local shear

Ultra-fine grained

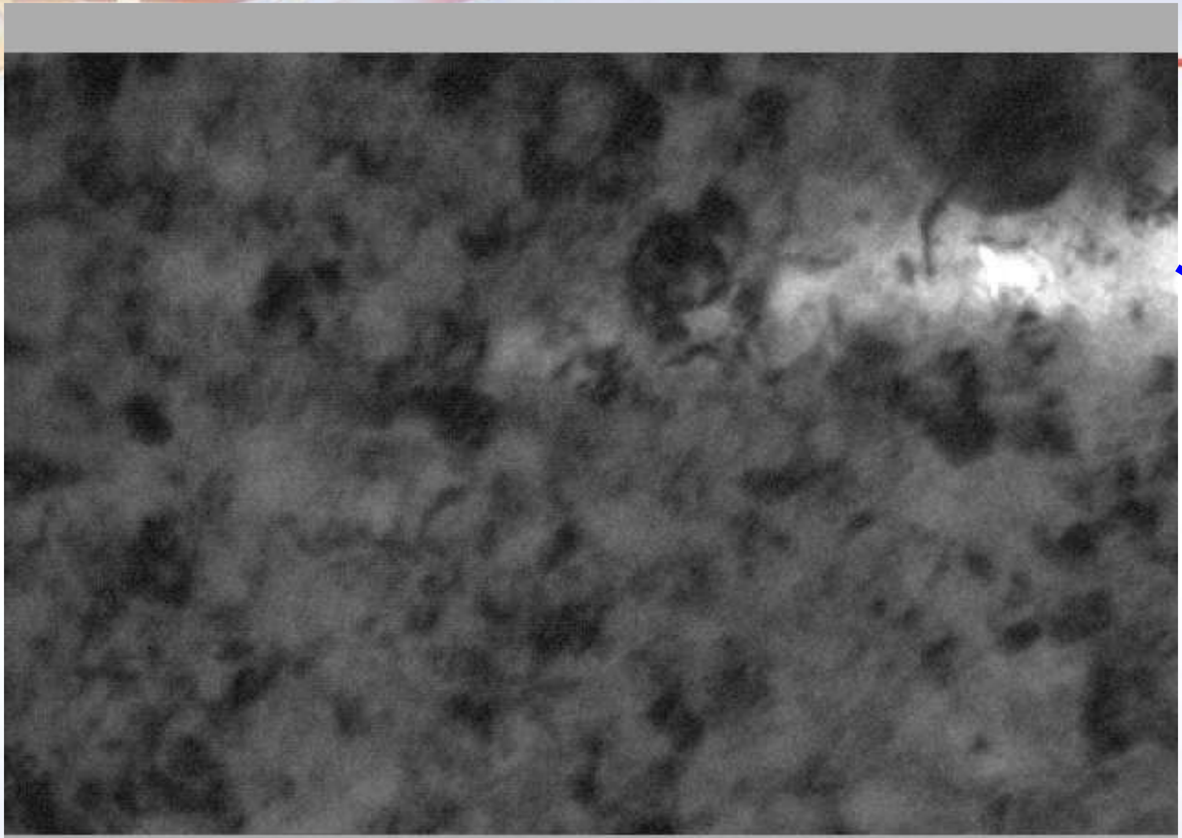


Shear Failure

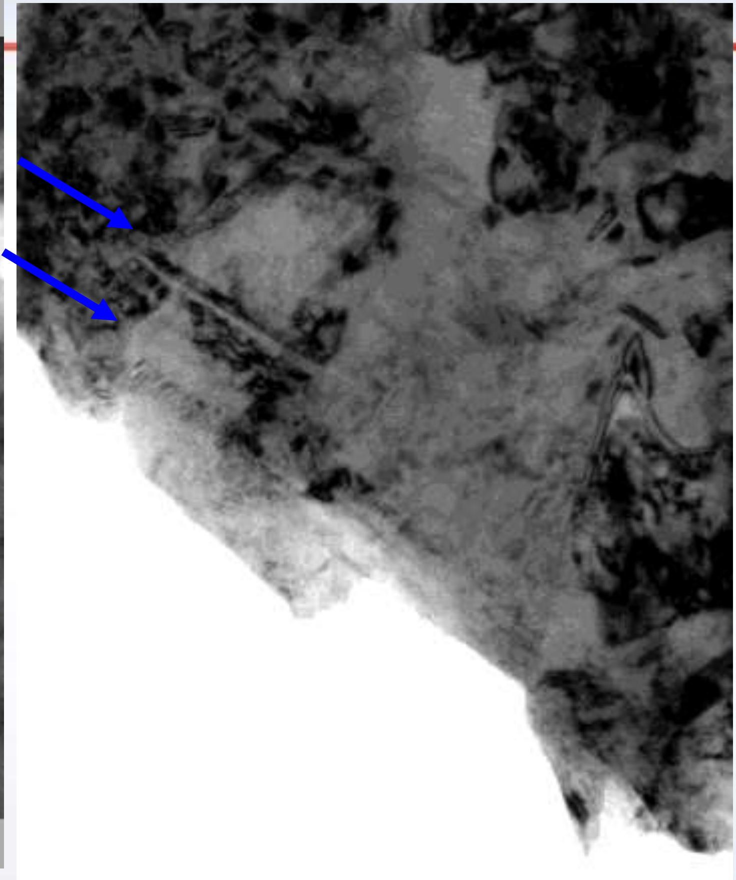
- Shear teeth
- Dislocation structure

Fracture surfaces provide insight to deformation processes

Deformation and Failure in Bimodal PLD Ni



80 nm-thick PLD Ni annealed at 275 ºC



Throughout the film

- Elastic strain
- Limited dislocation slip

In the plastic zone

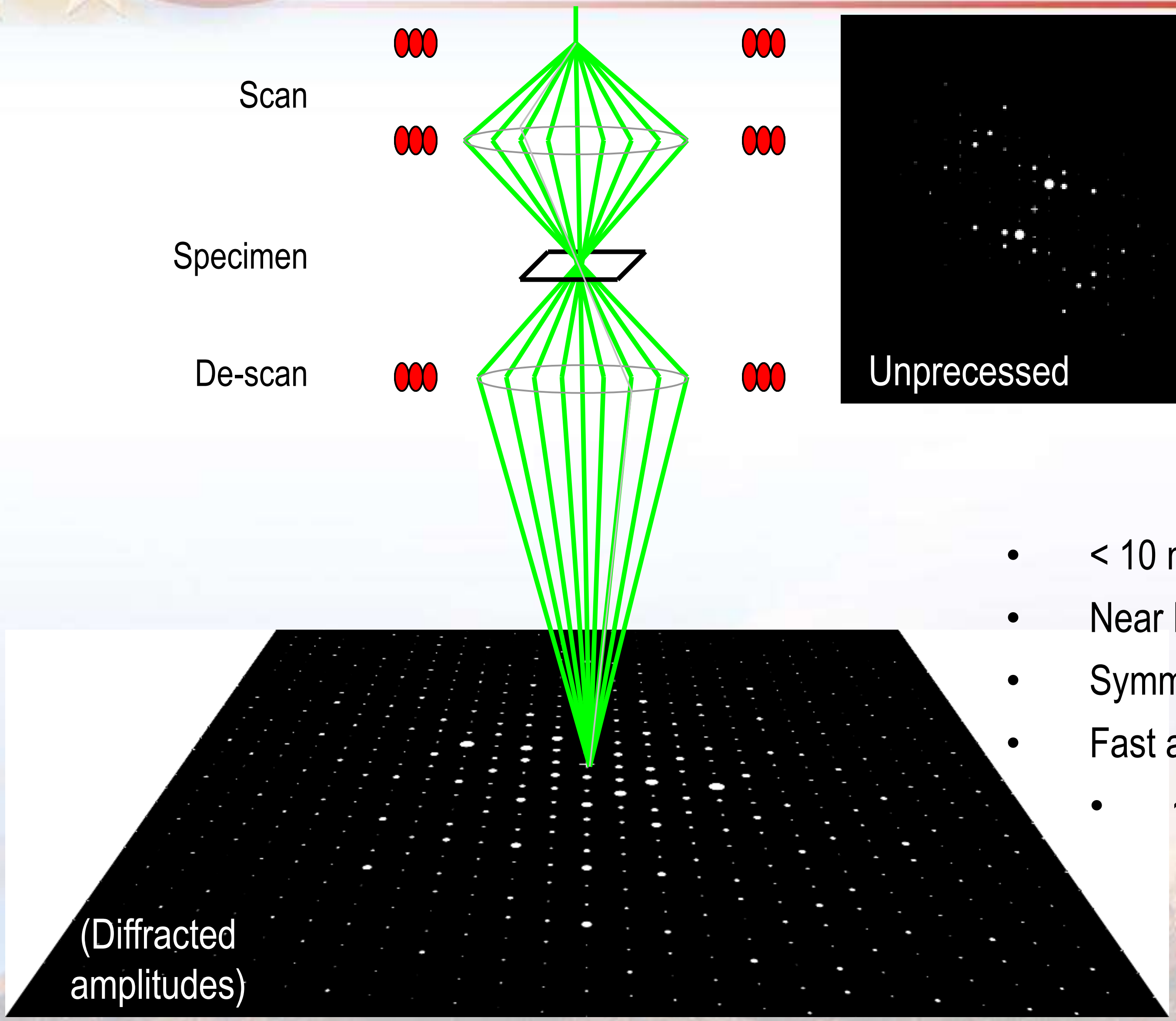
- Extensive dislocations slip
- Twinning

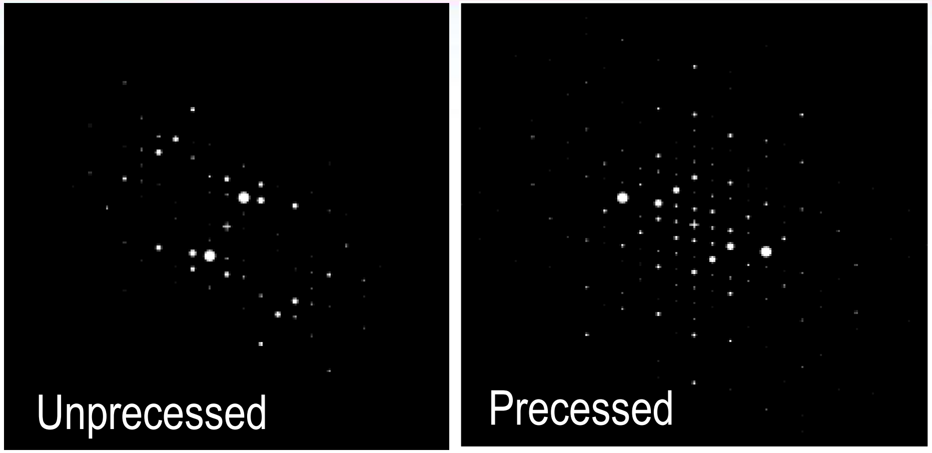
At Crack Tip

- Necking
- Grain agglomeration

We have some insight into the unique thermal and mechanical mechanisms and properties. What is the initial nanostructure that causes this?

Precession Electron Diffraction Microscopy



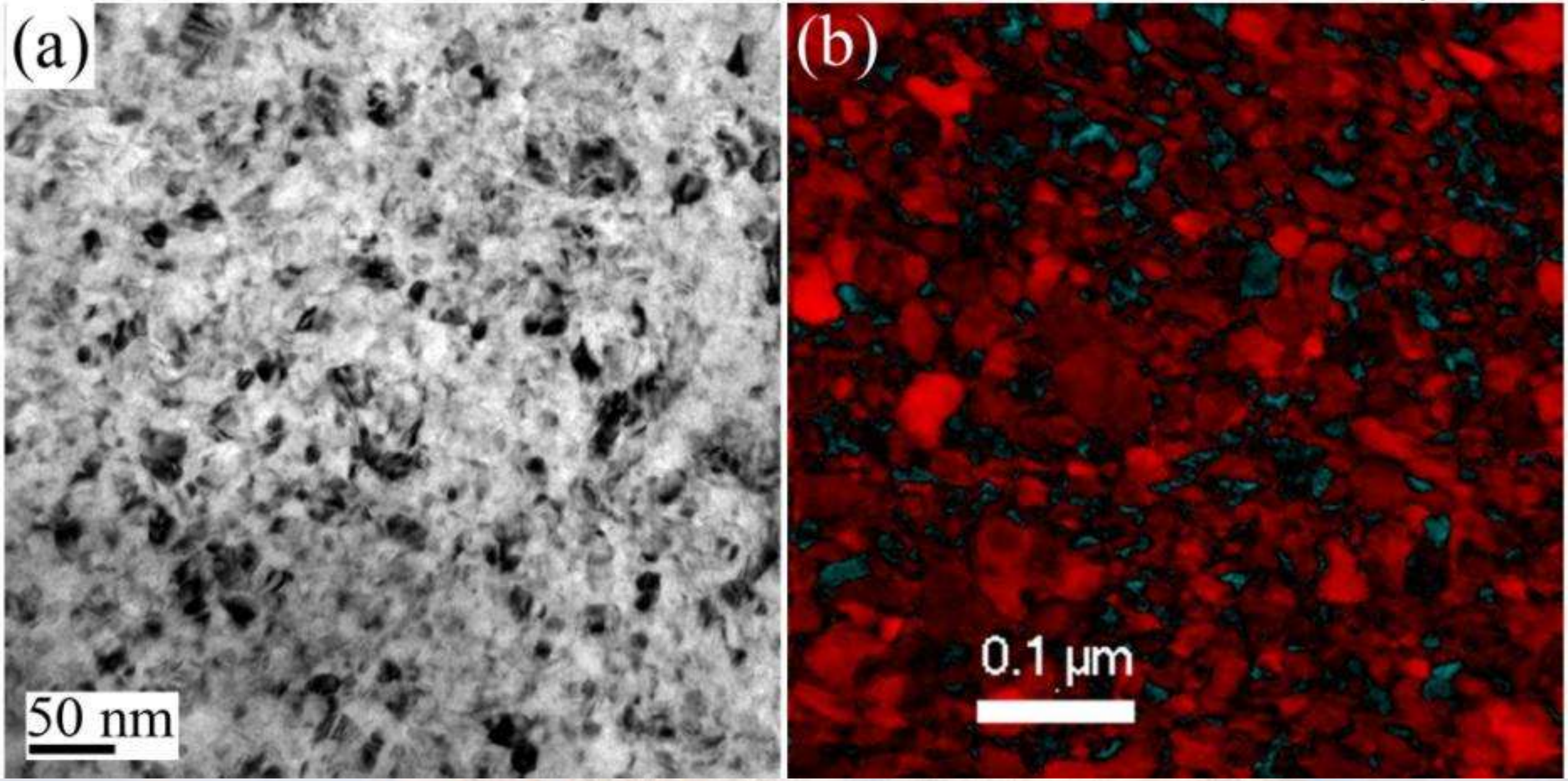


Advantages:

- < 10 nm spatial resolution
- Near kinematical electron diffraction
- Symmetry ambiguities are resolved
- Fast and automated acquisition
  - ~200 grains in 15 min.

Phase Determination in 50 nm As-deposited Ni Film

BF – TEM | **Re-constructed phase and reliability map**



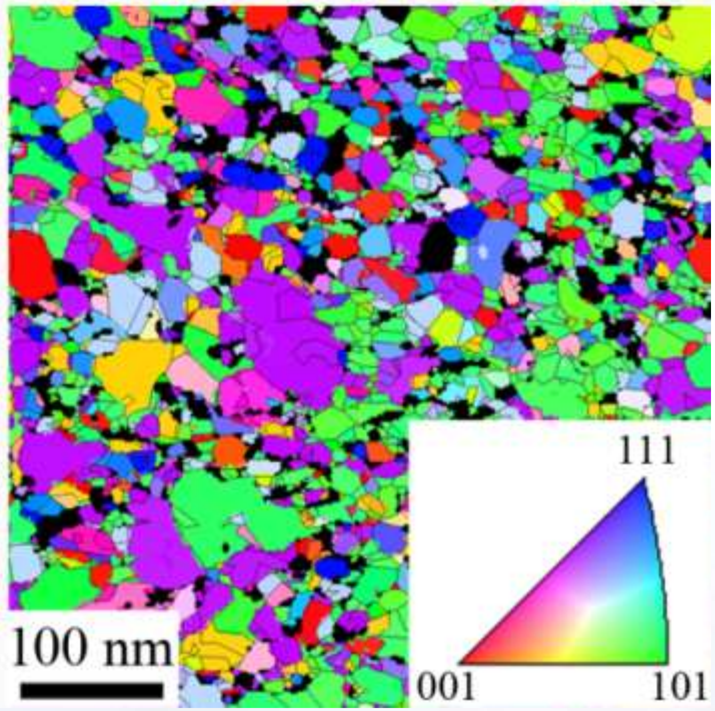
- 1,124 HCP phase grains (in 1.5 μm^2)
- Mean HCP grain size : 8.1 $\pm$ 0.3 nm
- Mean HCP phase percentage: 6.0%

Clear observation of morphology in nanocrystalline films

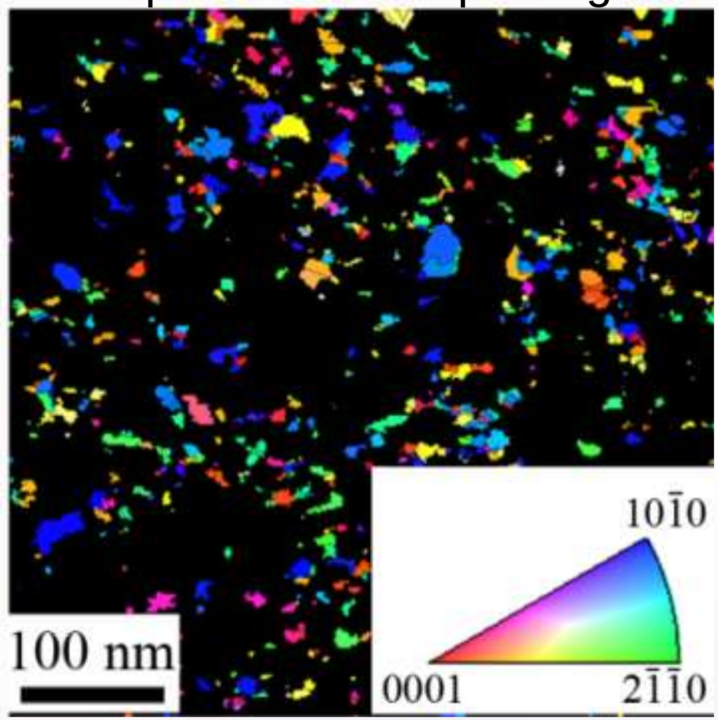
FCC phase
HCP phase

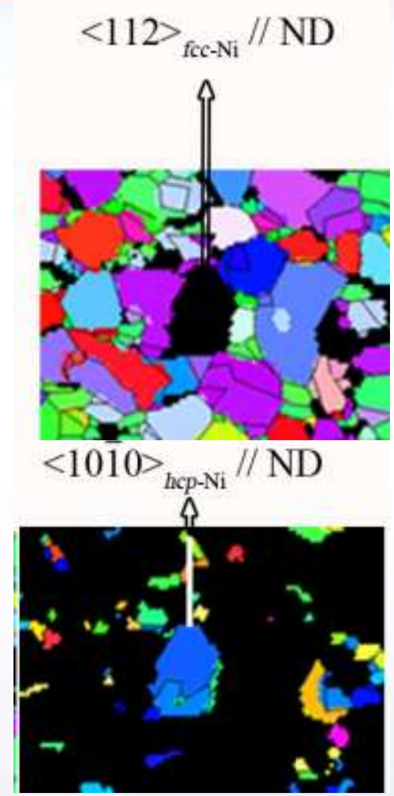
FCC and *HCP* Texture Determination in 50 nm As-deposited Ni Film

FCC phase inverse pole figure

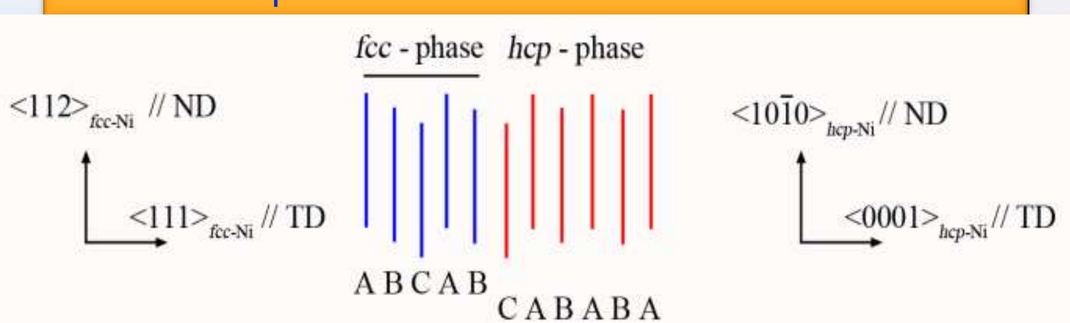


HCP phase inverse pole figure





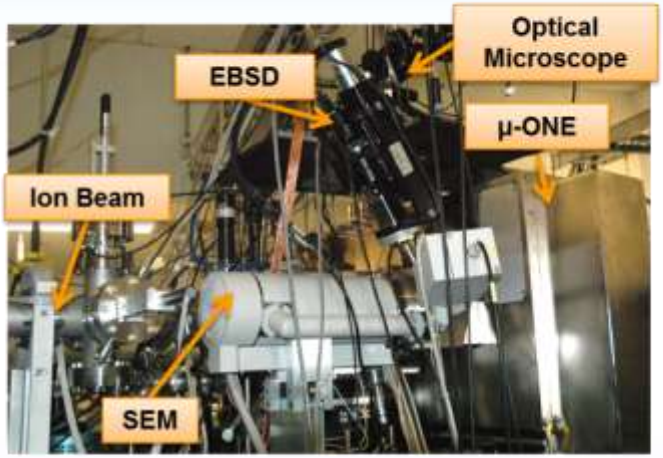
Texture maps at the nanoscale obtained from a TEM

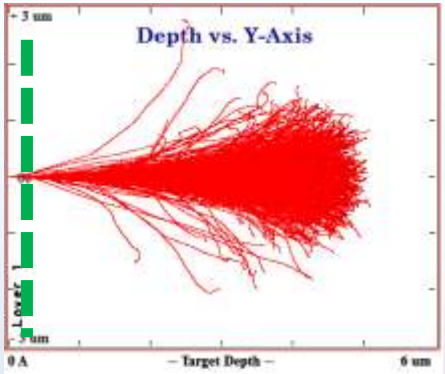


<112> $_{FCC-Ni}$ // ND results in an in-plane <111>$_{FCC-Ni}$ // TD

High energy PLD may introduce stacking faults leading to a <0001>$_{HCP-Ni}$ // TD

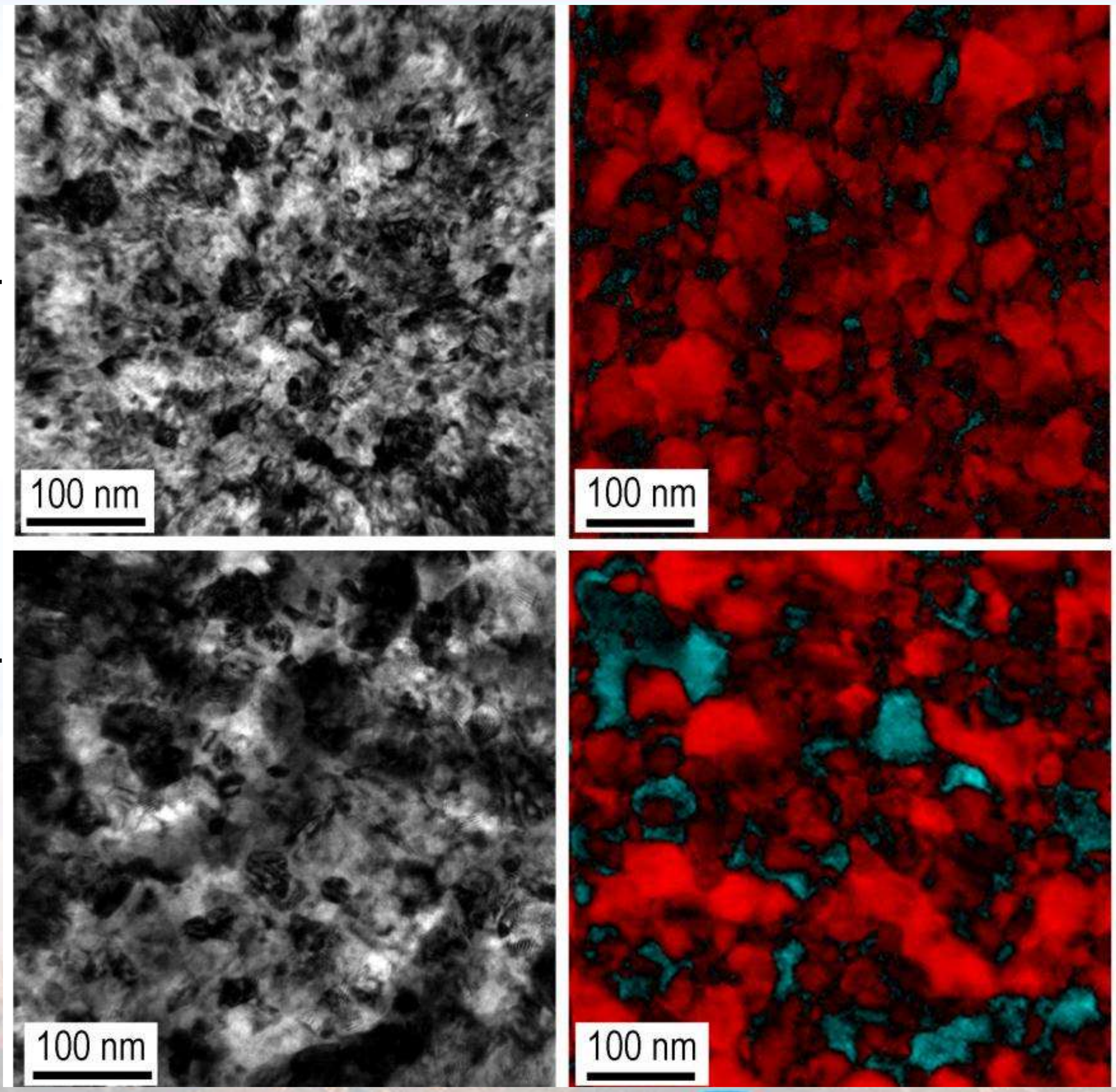
FCC and *HCP* Phase Evolution after 35 MeV Ni Irradiation





As-deposited

35 MeV Ni ~ 10 dpa

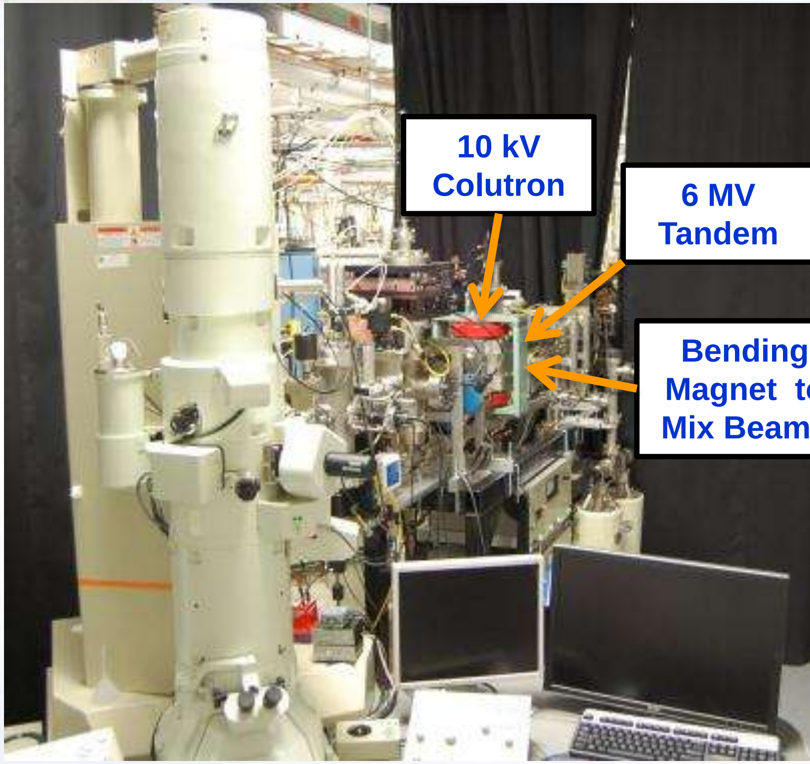


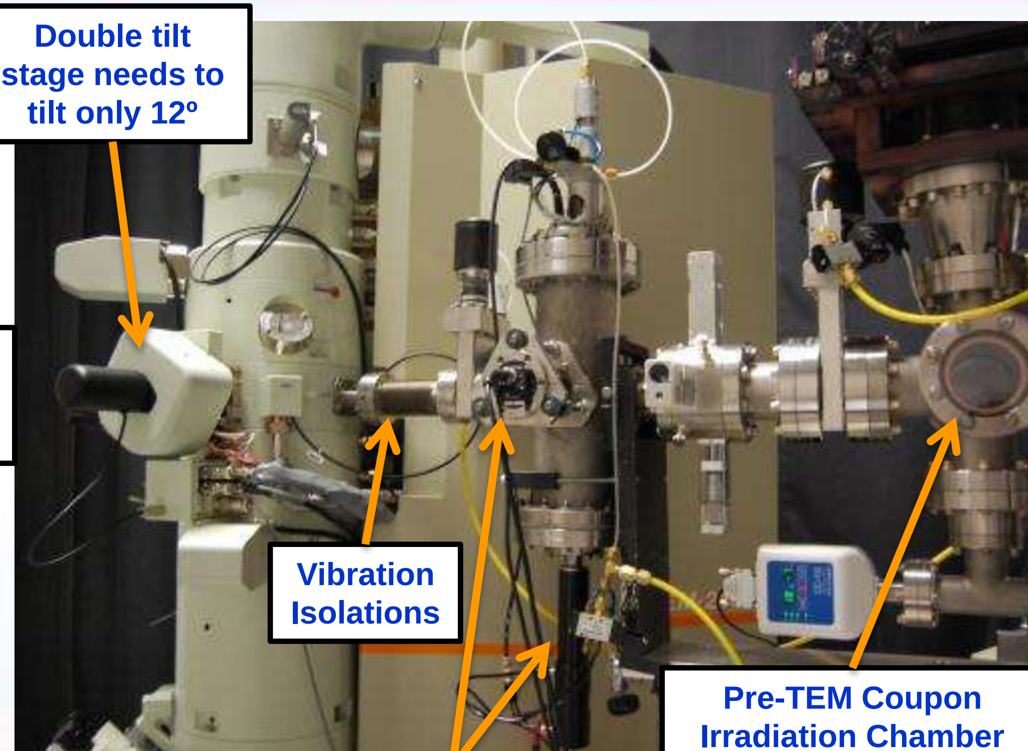
FCC phase *HCP* phase

Despite the minimal interaction predicted in 100 nm film, grain growth was observed and increased *HCP* phase resulted

In situ TEM Beamline

Collaborators: D. Buller and J.A. Scott





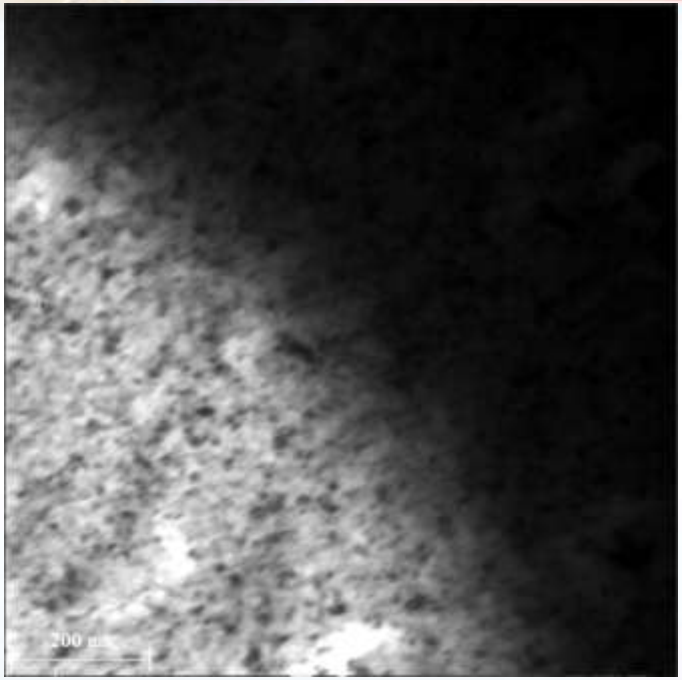
Beam burn from 14 MeV Si

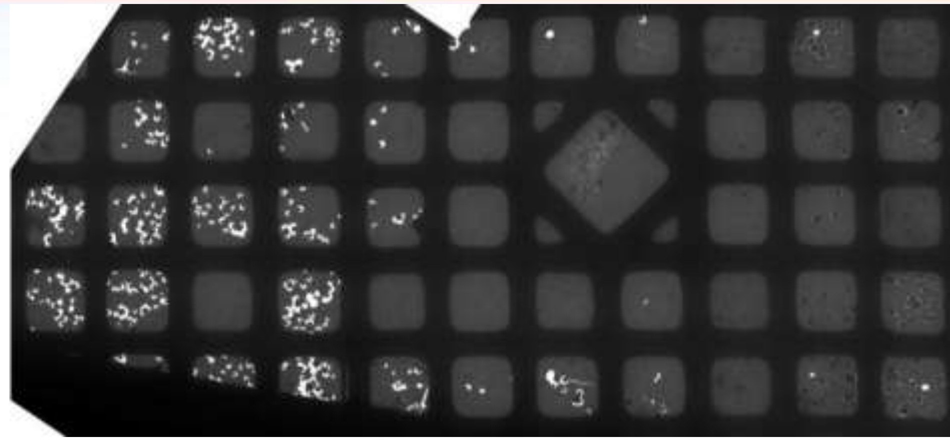
Tandem beamline into the TEM is completed and operated regularly

Colutron beamline is assembled, under vacuum, and baked out

We hope to have concurrent heavy and light ion irradiation facility operational in 2012

Initial *In situ* Ion Irradiation Results of PLD Ni



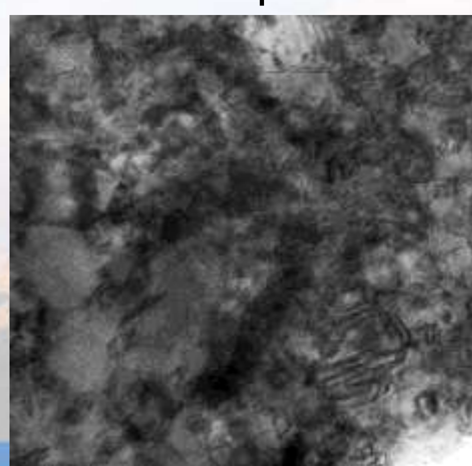


- 80nm PLD Ni deposited on salt and transferred to Cu grid
- Annealed 2 hrs at 250 ºC
- 60 nA 5 MeV Si^{3+} beam with d_{beam} ~ 3mm
- Sample irradiated 45˚ normal to ion beam

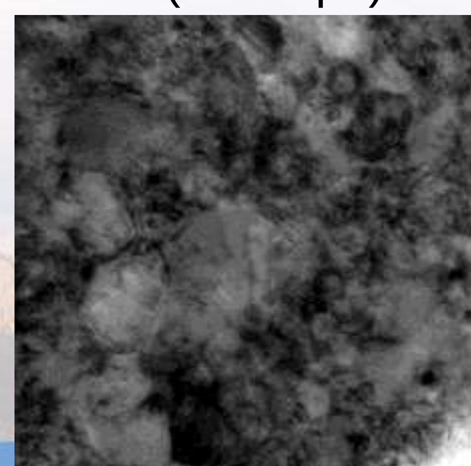
High energy heavy ion beams alter the electron beam when not grounded

Some structural changes observed during *in situ* TEM

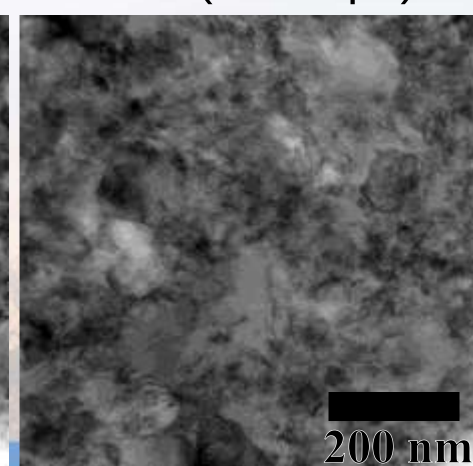
0 dpa



5 min (~.02 dpa)



10 min (~.04 dpa)



Collaboration with: A. Kinghorn and B. Yates

Conclusions

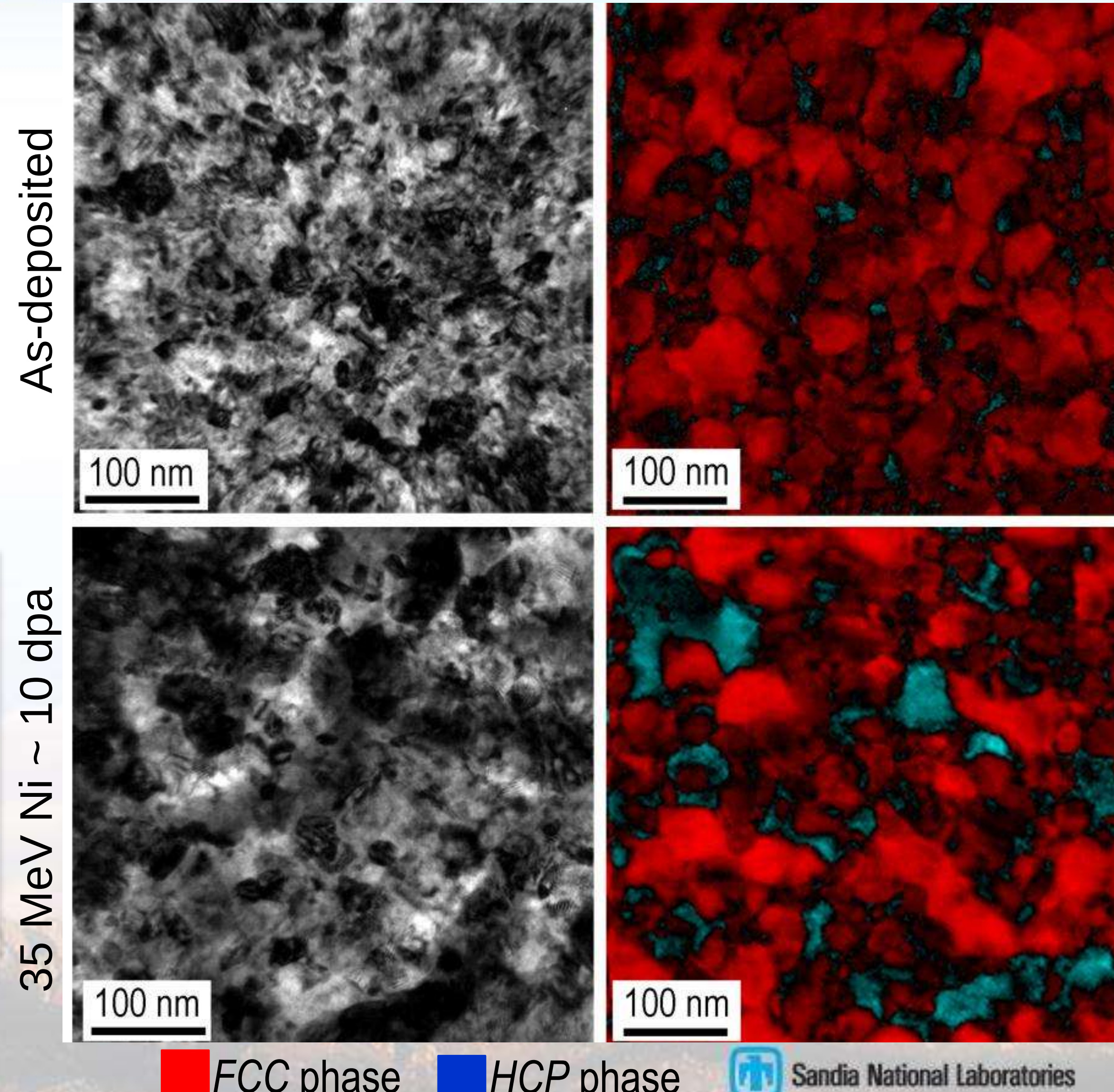
- **Unique structures result from nanograined processing**
- **Thermal, mechanical, and radiation stability of nanograined metals are probably intertwined**
- **Precession microscopy provides a unique tool to study the grain orientations and boundary relationships**

Ion Irradiation

\+

<u>Precession Microscopy</u>

Greater insight into structural evolution due to radiation damage



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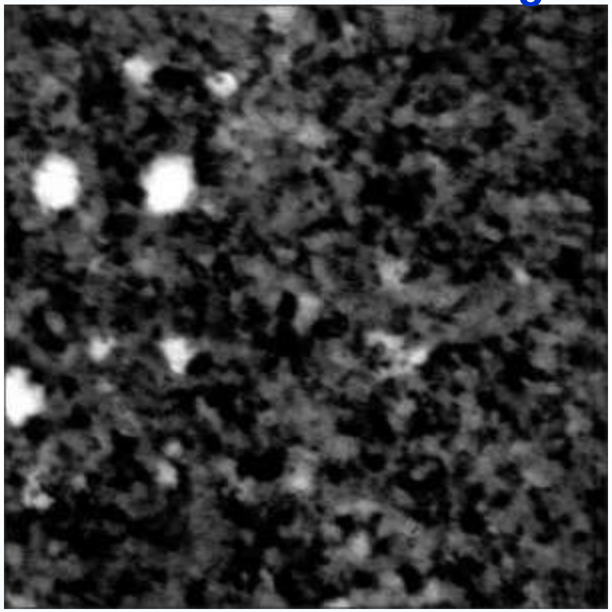
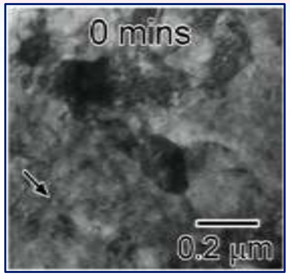
Interesting New *In situ* TEM Directions…

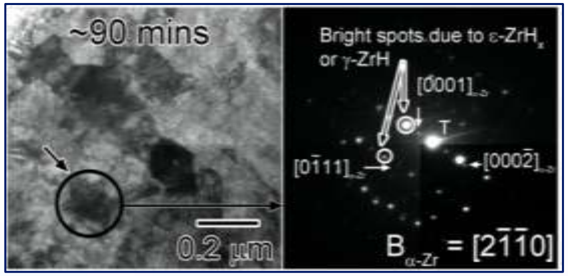
Acetic acid flown over ng-Fe

Collaboration with: D. Gross, J. Kacher, and I.M. Robertson



Hydride formation in Zirlo exposed to pure H_2

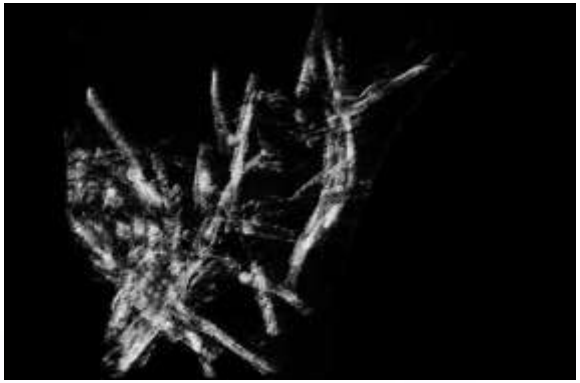




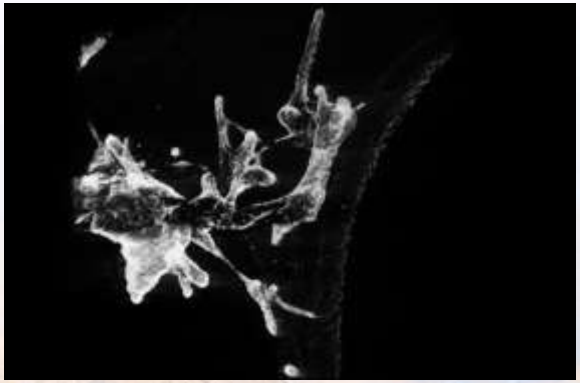
Collaboration with: S. Rajasekhara and B.G. Clark

Irradiation stability of scintillators

Initial $CdWO_4$ model



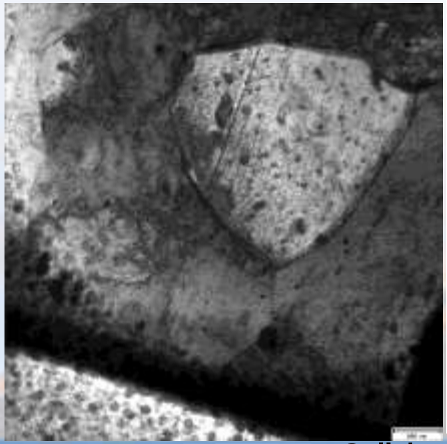
Irradiation with 20 nA, 3 MeV Cu^+



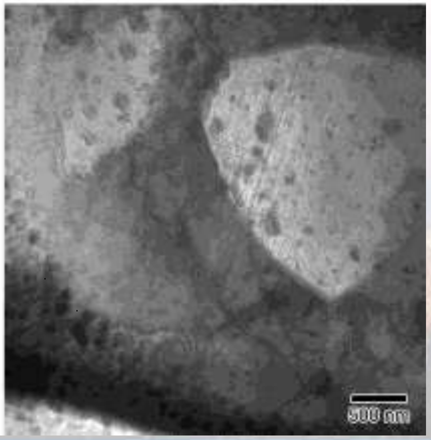
Collaboration with: S. Hoppe, B.A. Hernandez-Sanchez

Zircaloy heating in air

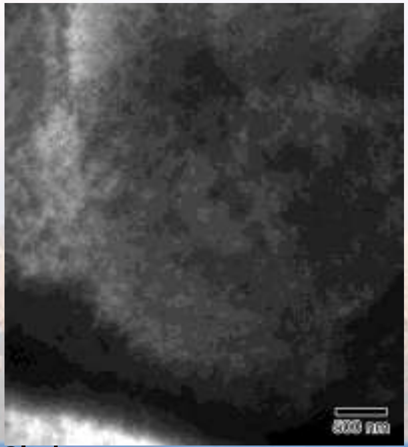
<u>Vacuum & Single Window</u>



<u>~1 atm & Two Windows</u>



<u>~1 atm & ~1200 C</u>



Collaboration with: S. Rajasekhara and B.G. Clark