Understanding Abnormal Grain Growth in Nanograined Nickel through the Combination of in situ TEM and Precession Microscopy

SAND2012-7538C

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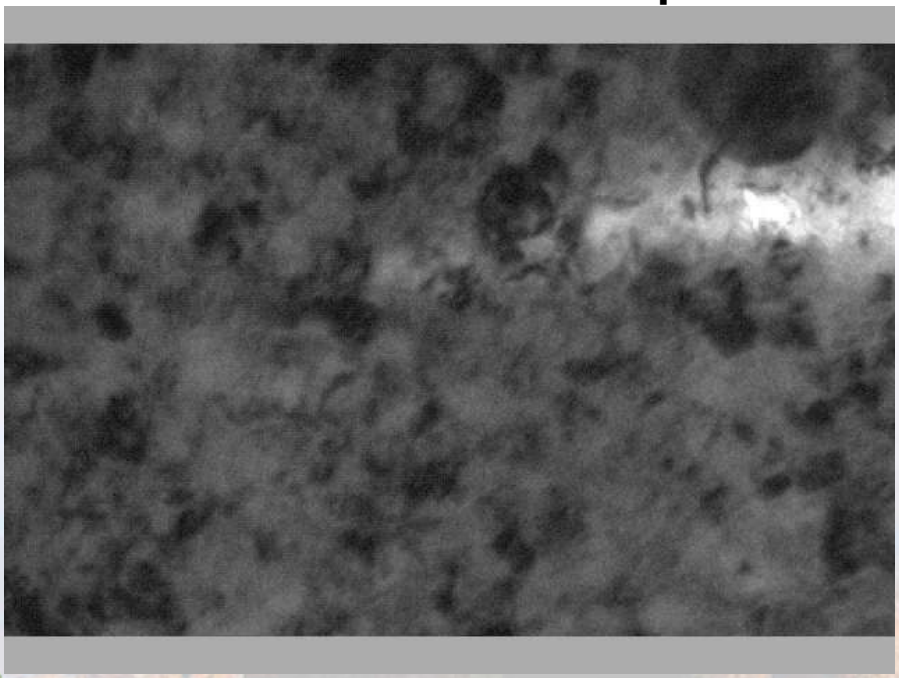
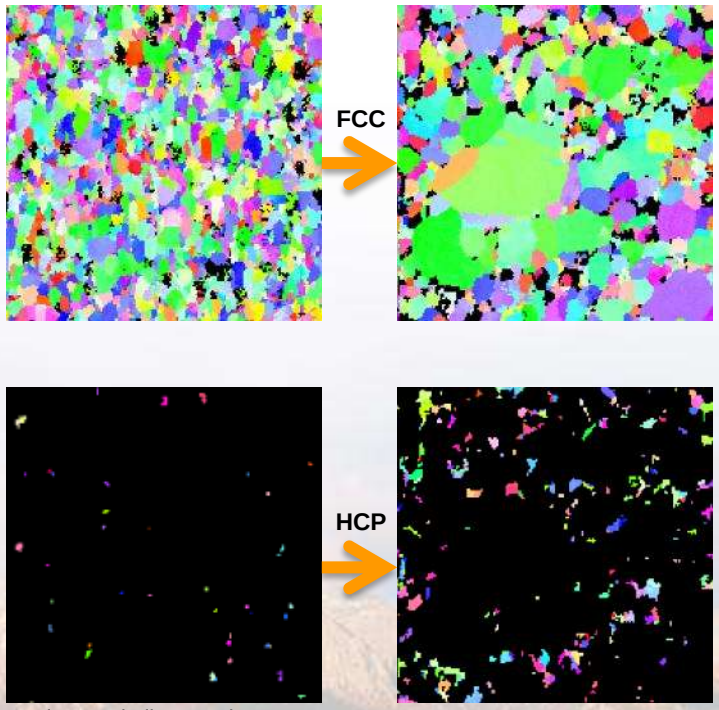
September 18, 2012





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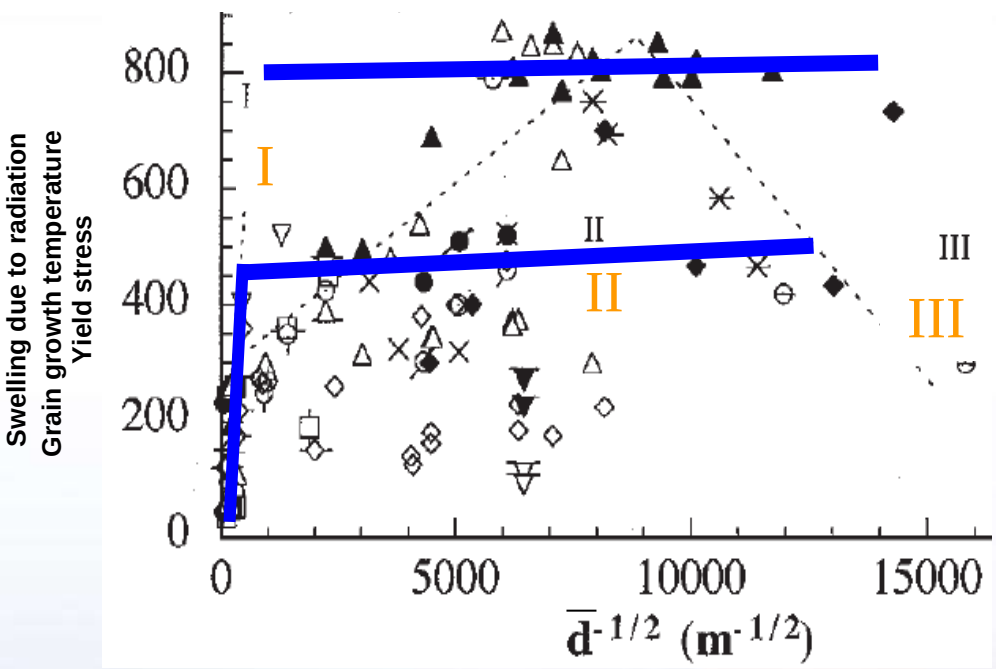
Properties and Mechanisms Active in Nanograined Metals



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

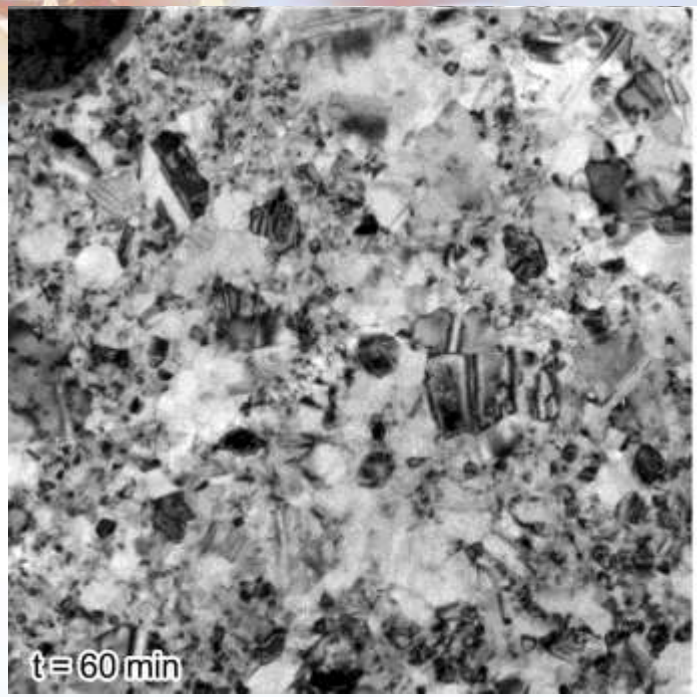
Key overlooked factors

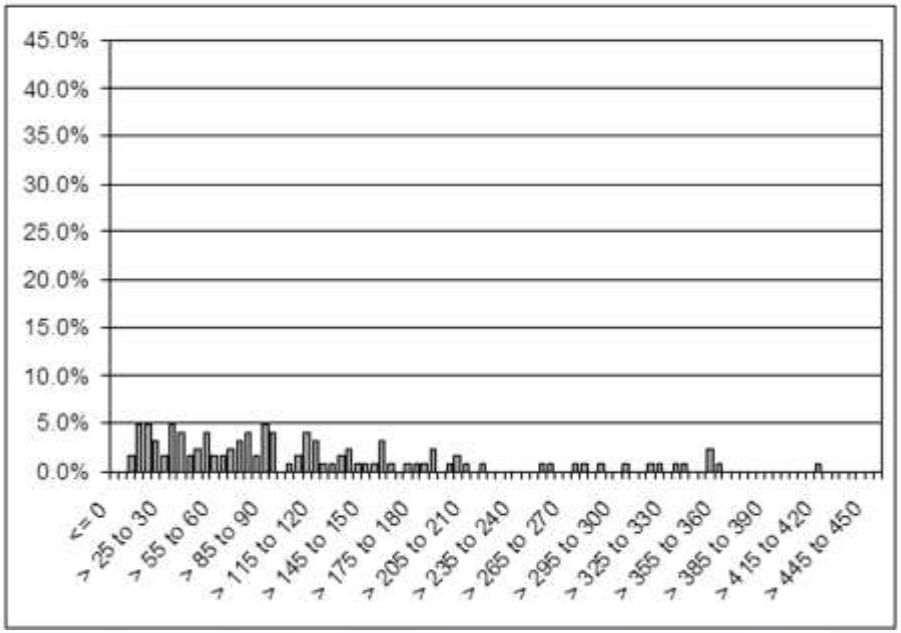
- Grain boundary type
- Relative grain orientation
- Grain boundary energy
- Etc.

1) What effect does grain boundary stability play in the reported thermal, mechanical, and radiation properties?

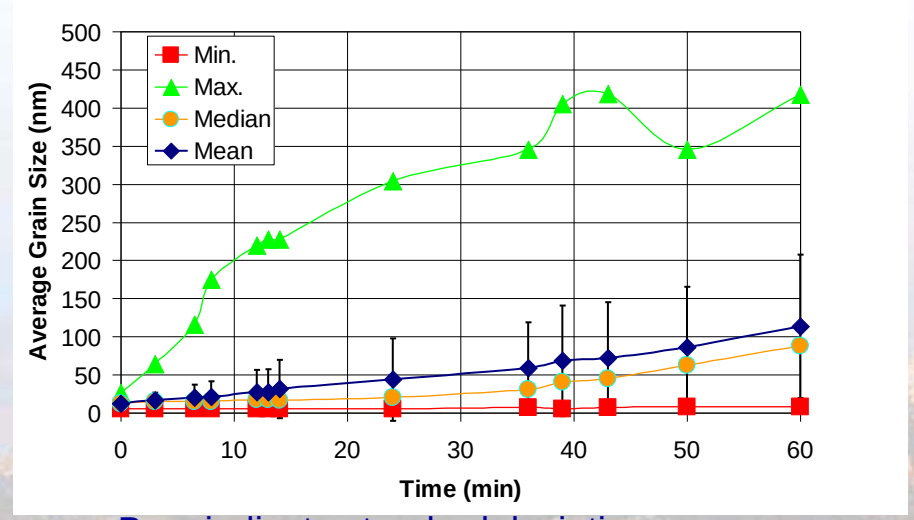
2) Beyond just grain size, what is the microstructure?

Kinetics of Abnormal Grain Growth in PLD Ni





50 nm-thick PLD Ni
Aged 4 yrs.
Annealed at 350 ° C



Bars indicate standard deviation

Microstructures of abnormal grain growth

- Epitaxial nanograined
  - Bimodal dominated by nanograins
  - Bimodal dominated by ultra-fine grains
  - Ultra-fine grains

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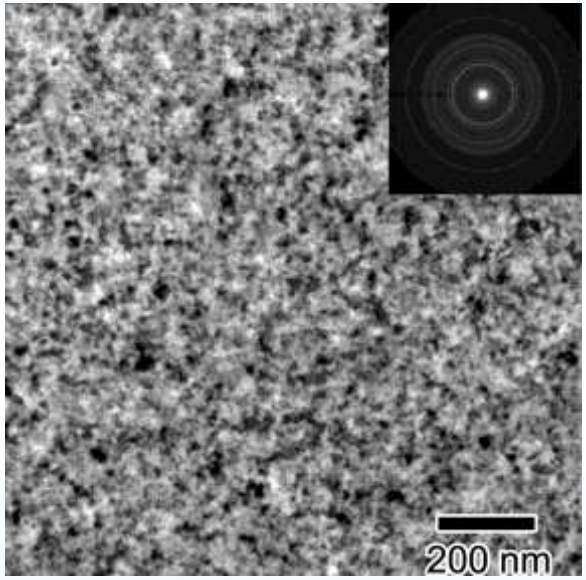
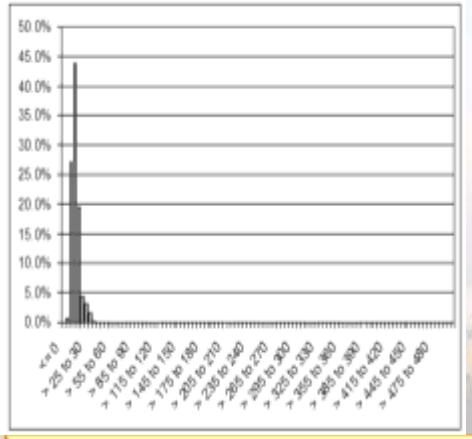
Thermal Stability of Nanograined Ni Films

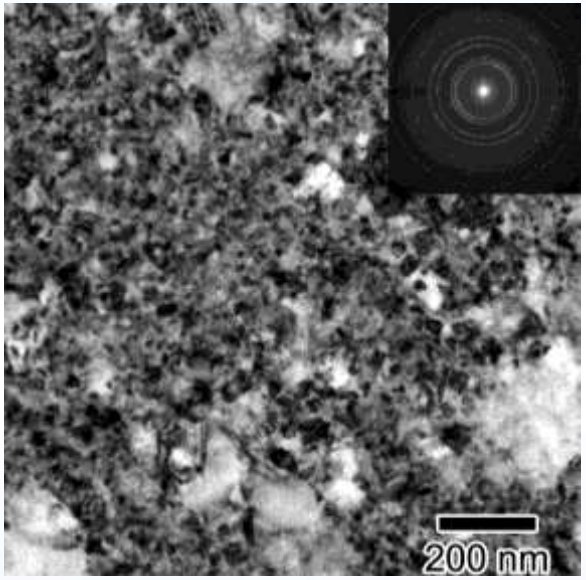
Nanograined



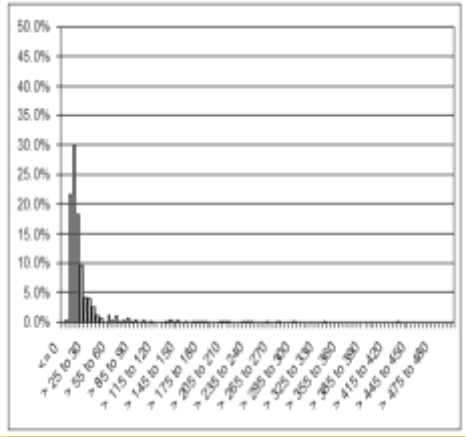
80 nm-thick As-deposited on Si



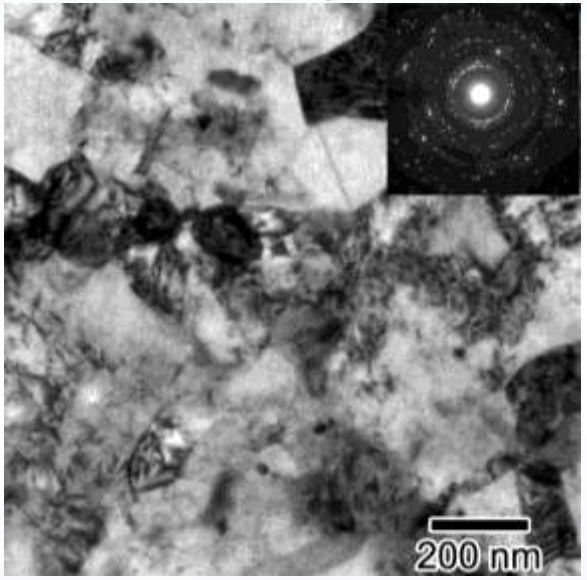
Bimodal



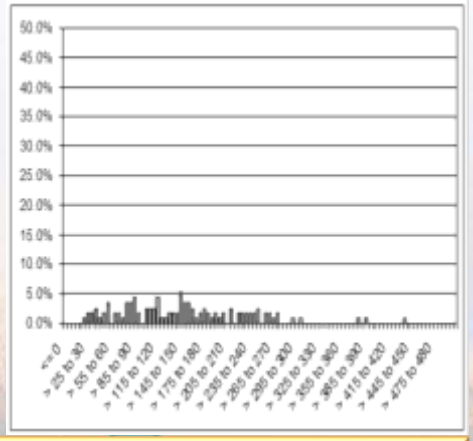
1 hr at 275 ° C



Ultra-fine grained

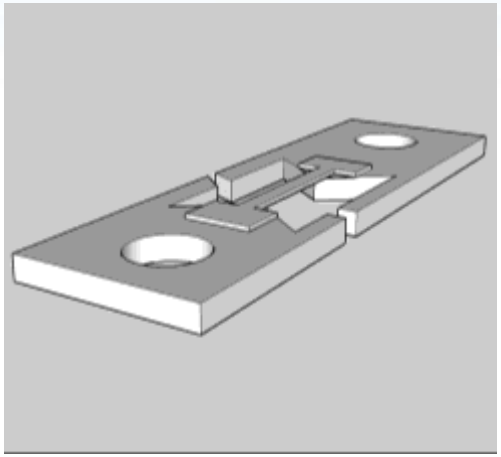


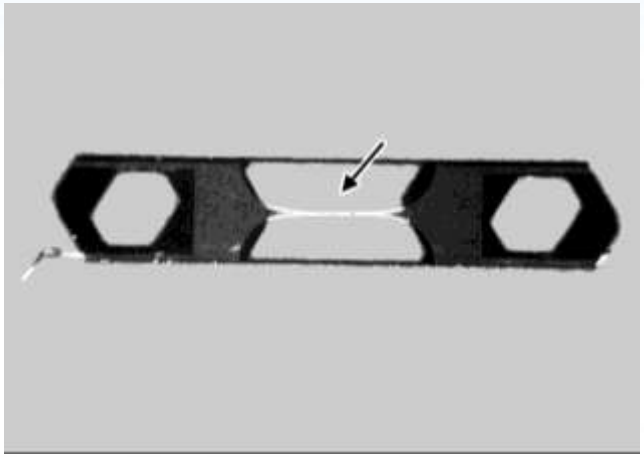
1 hr at 375 ° C

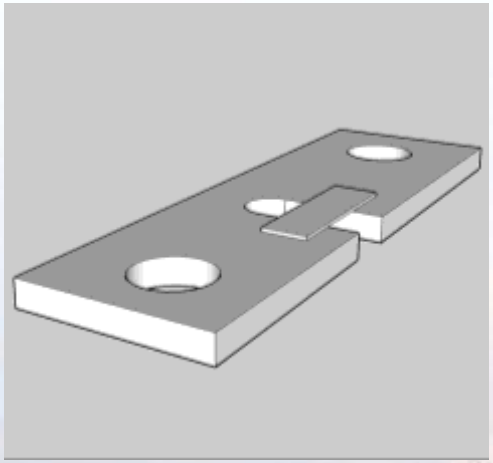


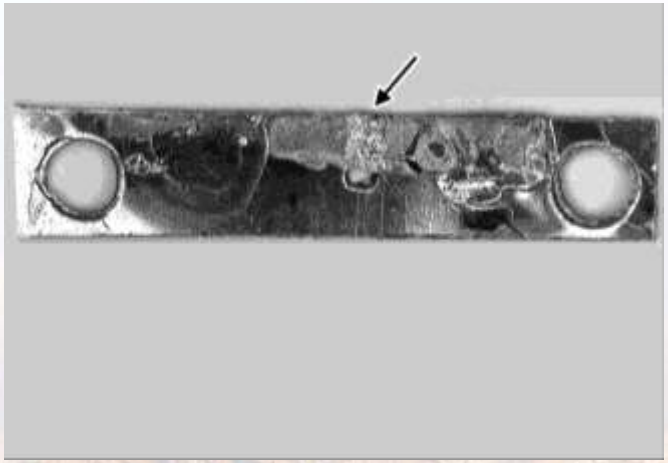
A few select grains grow at the expense of the remaining matrix

Development of TEM straining devices for PLD Ni









Microfabricated Sample

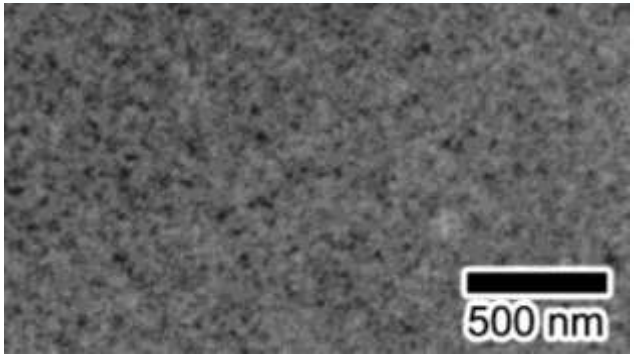
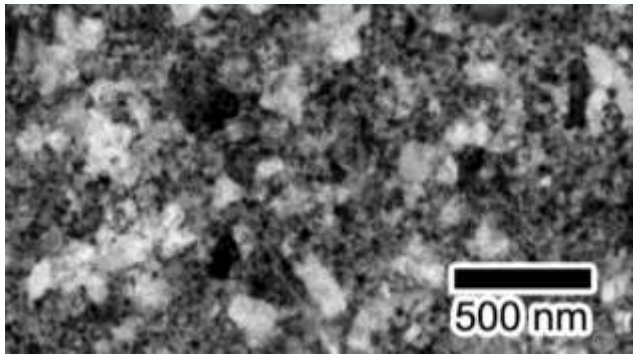
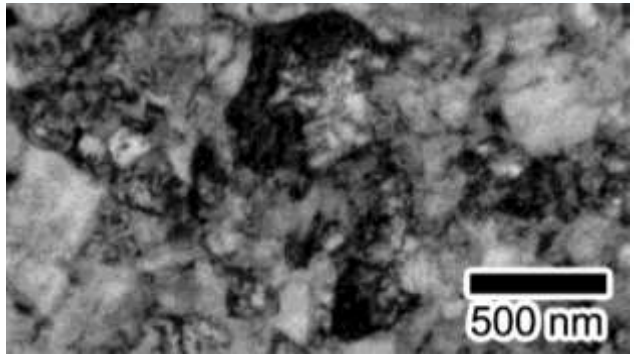
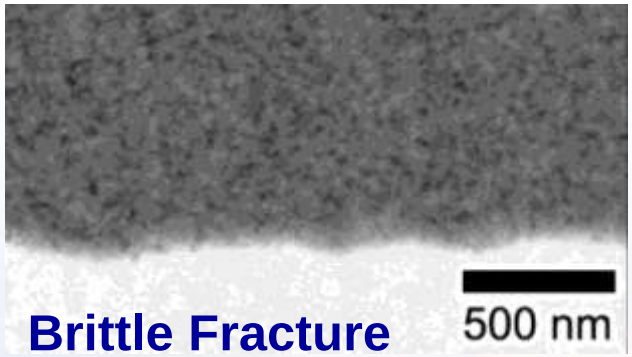
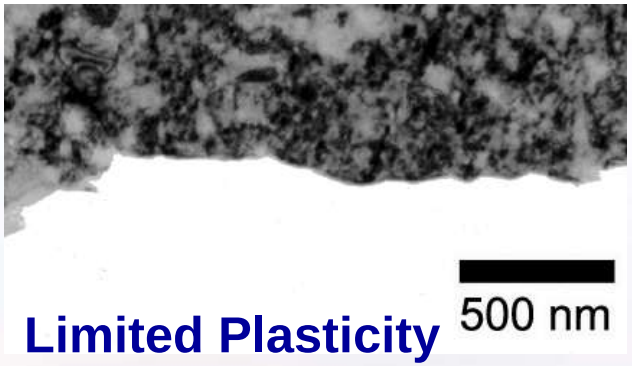
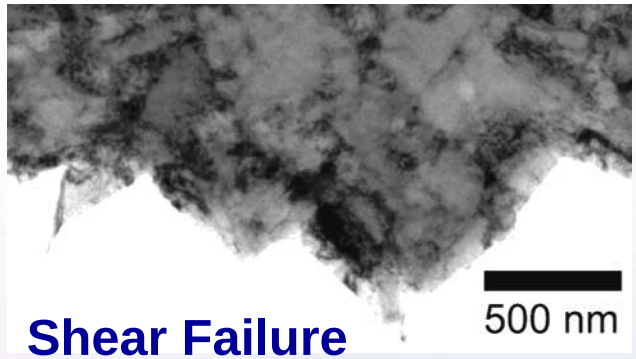
- No stress-strain
- Uniform dimensions
- Significant beam bending
- Si support beam must be fracture prior to strain

Home-made Jig

- Delicate sample preparation
- Non-uniaxial tension
- Epoxy can slip or contaminate

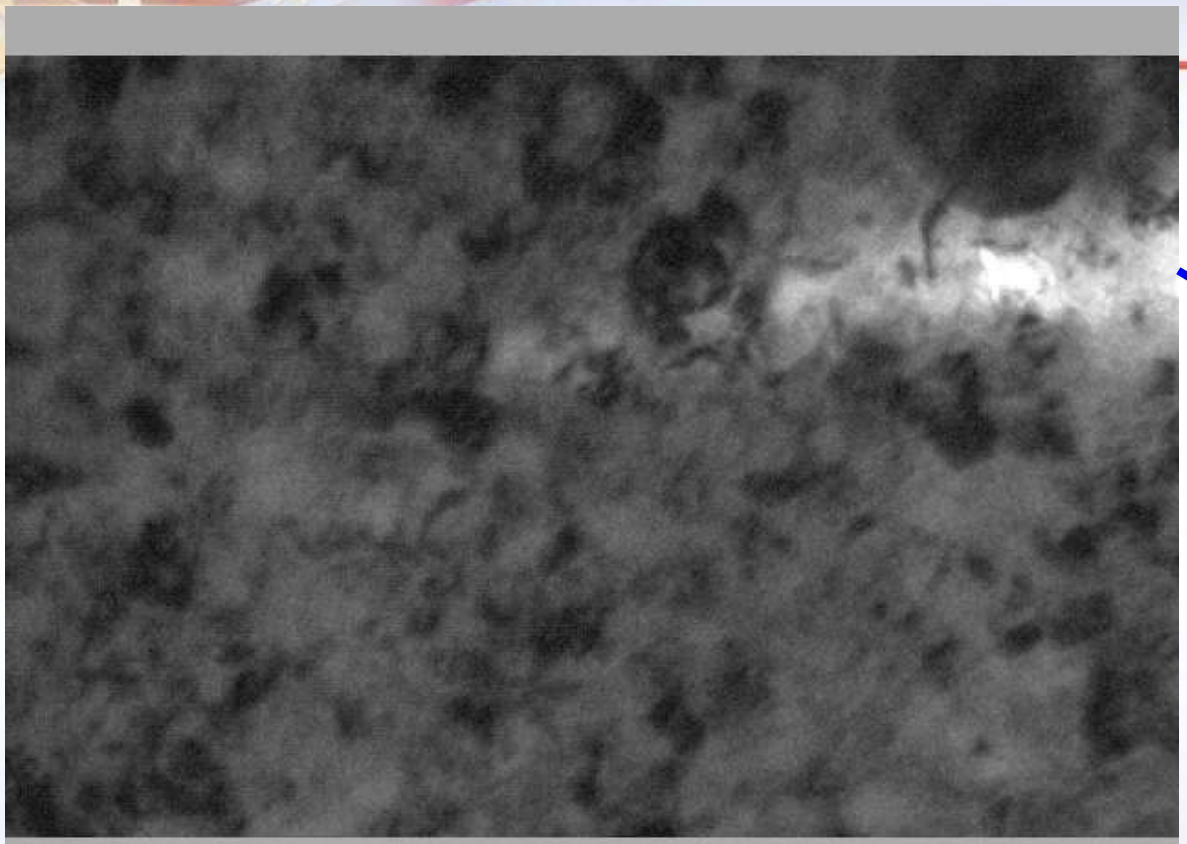
Developed different techniques to strain uniform thickness free-standing films

Failure Analysis of Strained PLD Ni

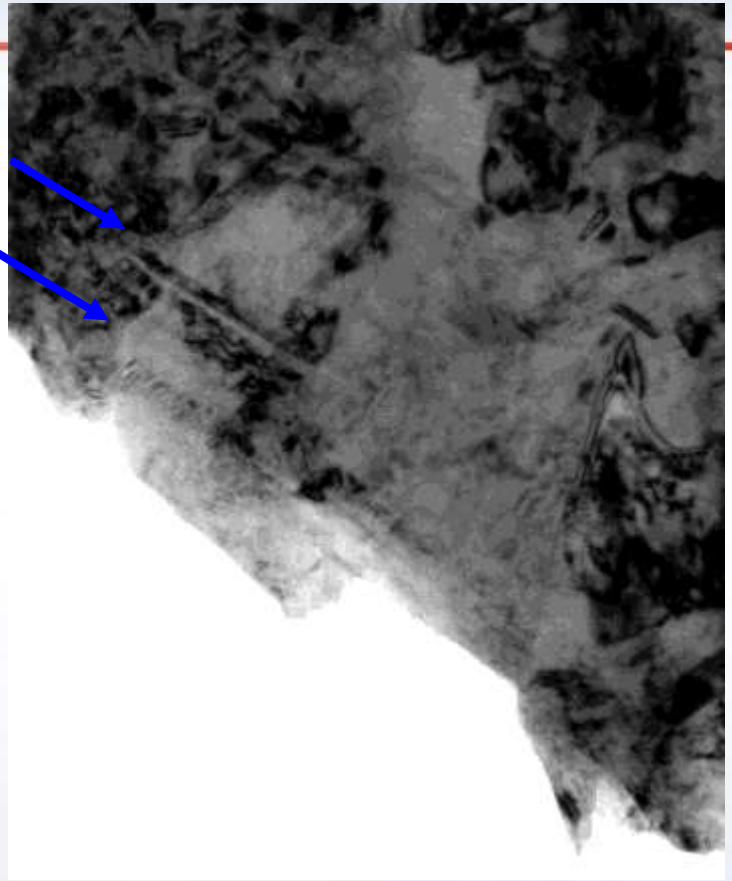
Nanograined	Bimodal	Ultra-fine grained
		
 **Brittle Fracture**	 **Limited Plasticity**	 **Shear Failure**
▪ No observation of global plasticity	▪ Dislocation pile-up ▪ Local shear	▪ Shear teeth ▪ Dislocation structure

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

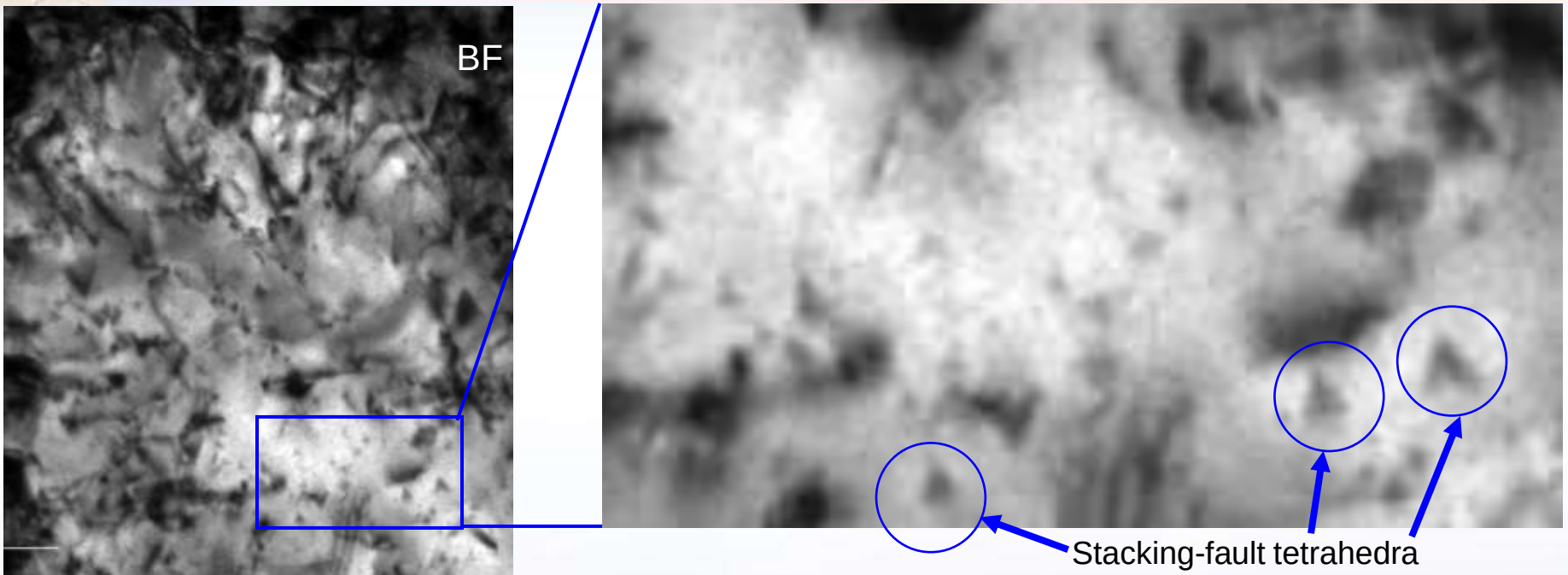
- Extensive dislocations slip
- Twinning

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?

A Variety of Unexpected Defect Structures in Ni



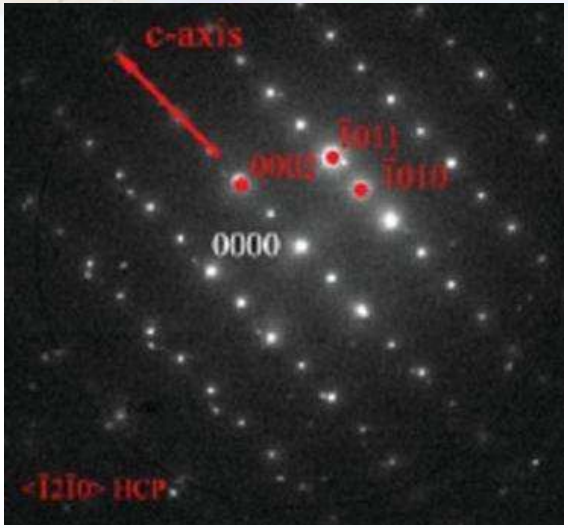
230 nm thick PLD Ni Annealed at 225 ºC for 14 hrs.

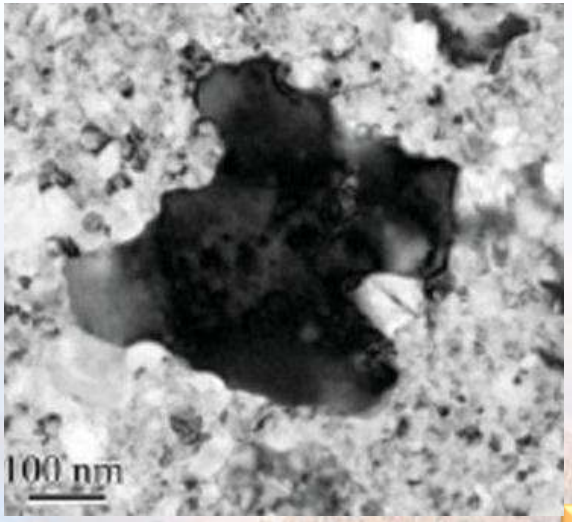
Multitude of defects in annealed PLD Ni

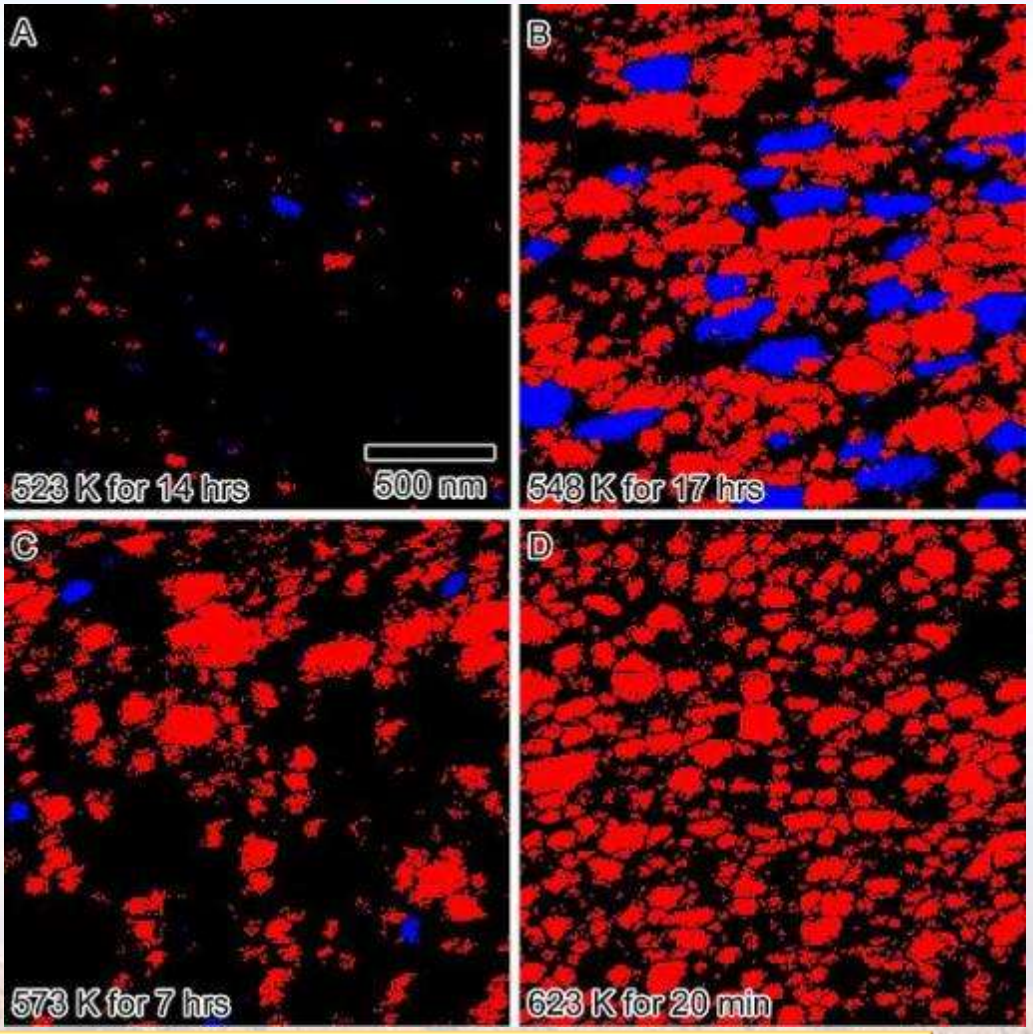
- SFT at temperature
- Stable microstructure for over 15 months
- SFT **not** due to irradiation, quenching, high strain rate
- SFT are theorized to be formed by rapid grain growth through the high free-volume at the initial grain boundaries

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

Collaboration with: L.N. Brewer, J.A. Knapp, D.M. Follstaedt, and I.M. Robertson







FCC phase

HCP phase

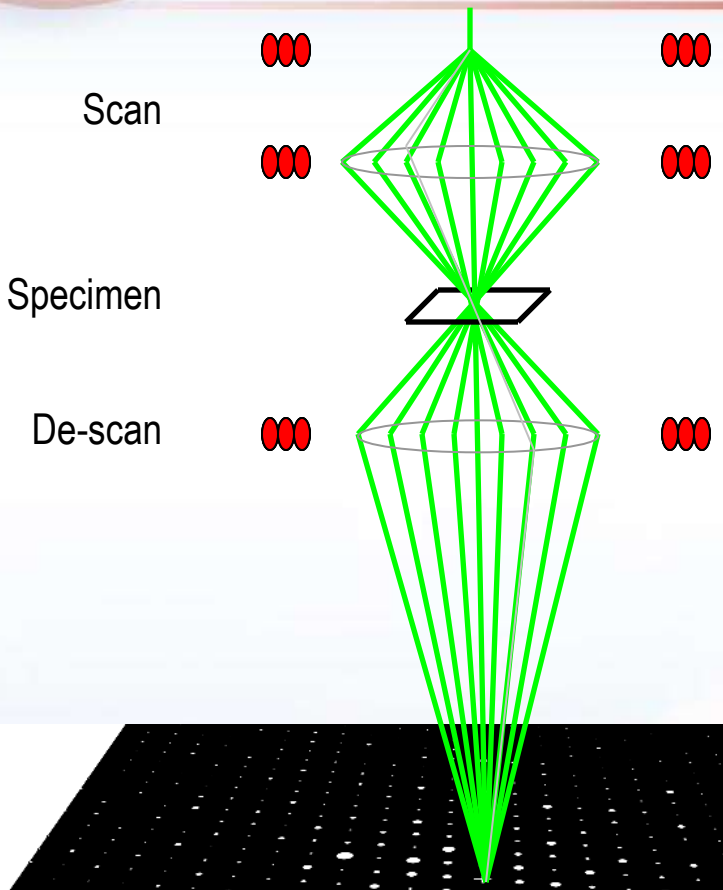
However,

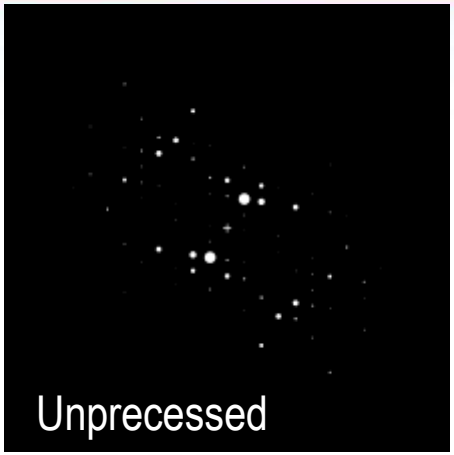
Spatial resolution limits detailed analysis of the *HCP* phase evolution

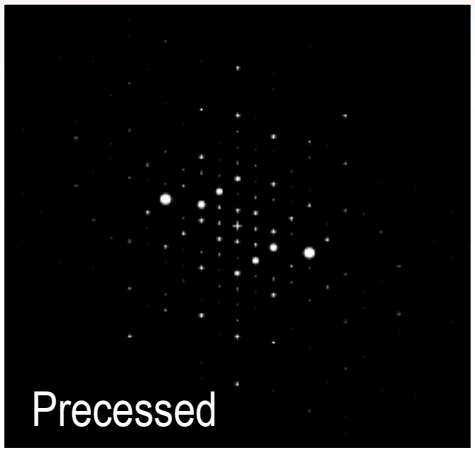
L. N. Brewer, D. M. Follstaedt, K. Hattar, J. A. Knapp, M. A. Rodriguez, I. M. Robertson, Adv. Mater. 22 (2010), 1161

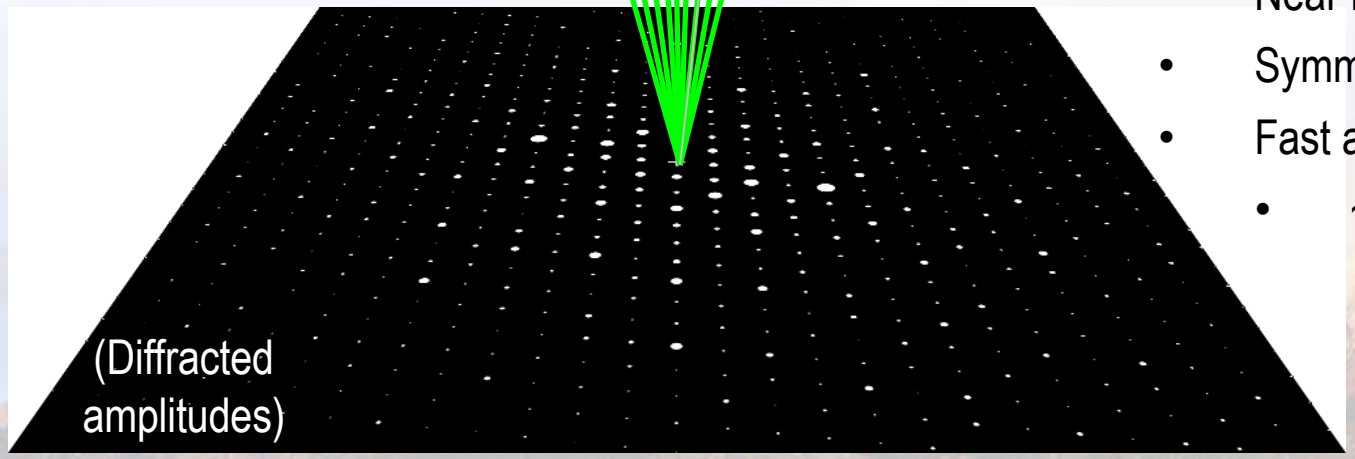
EBSD and SAD confirm the presence of ***HCP*** phase in some abnormal grains

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.

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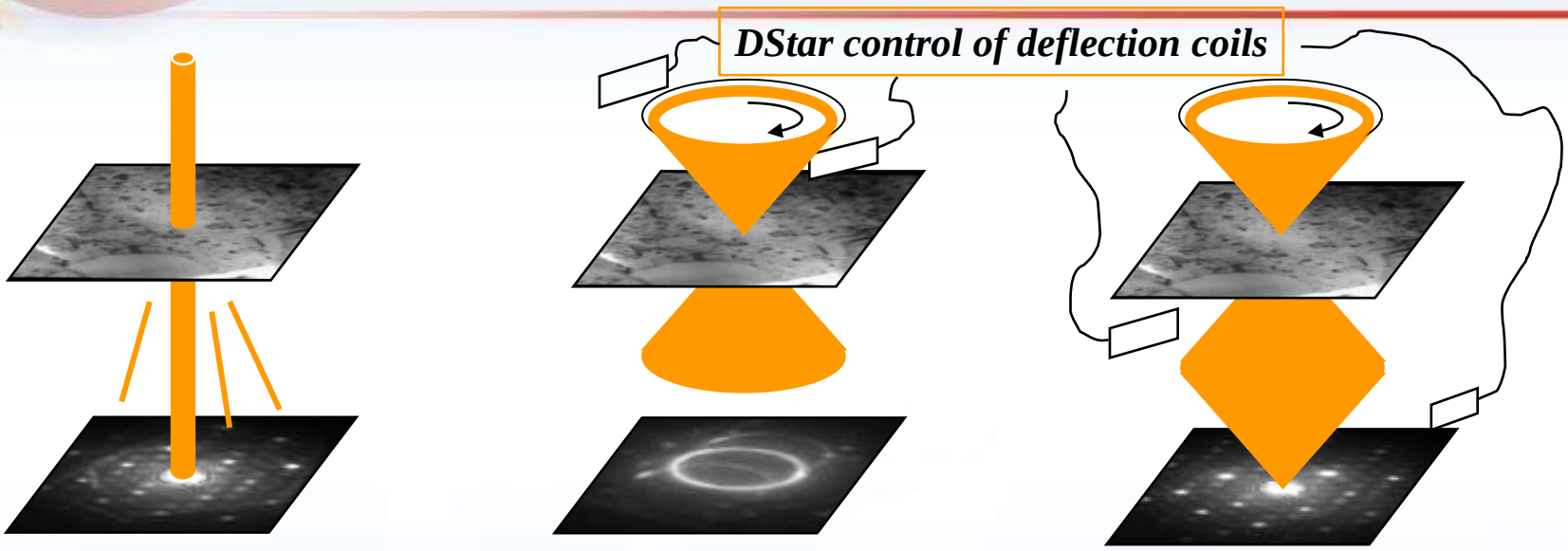
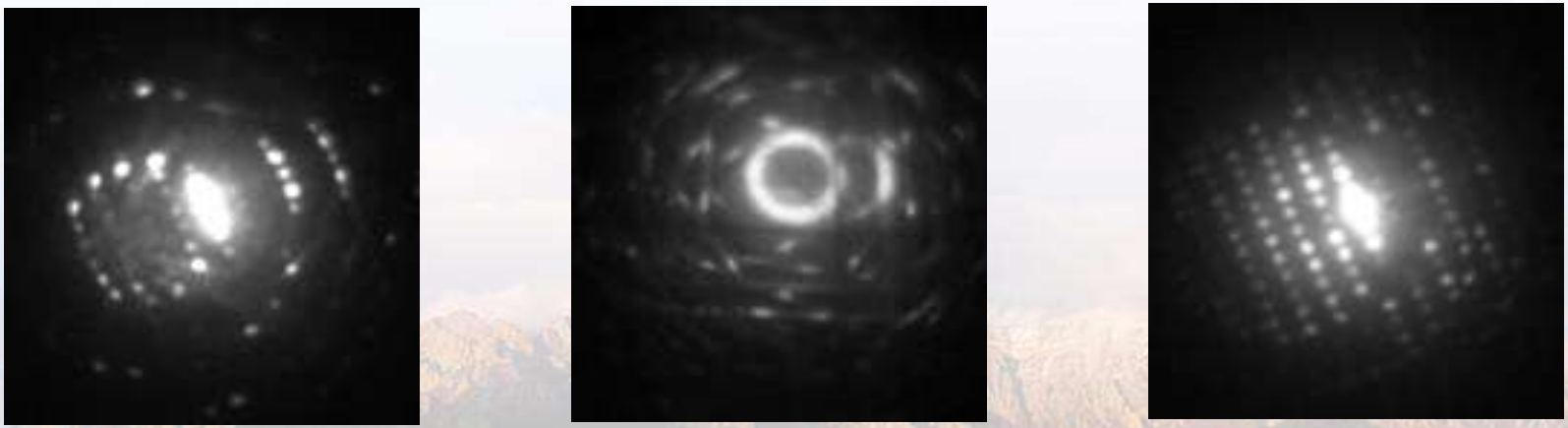
Precession Electron Diffraction Microscopy



Standard setting

½ Precession

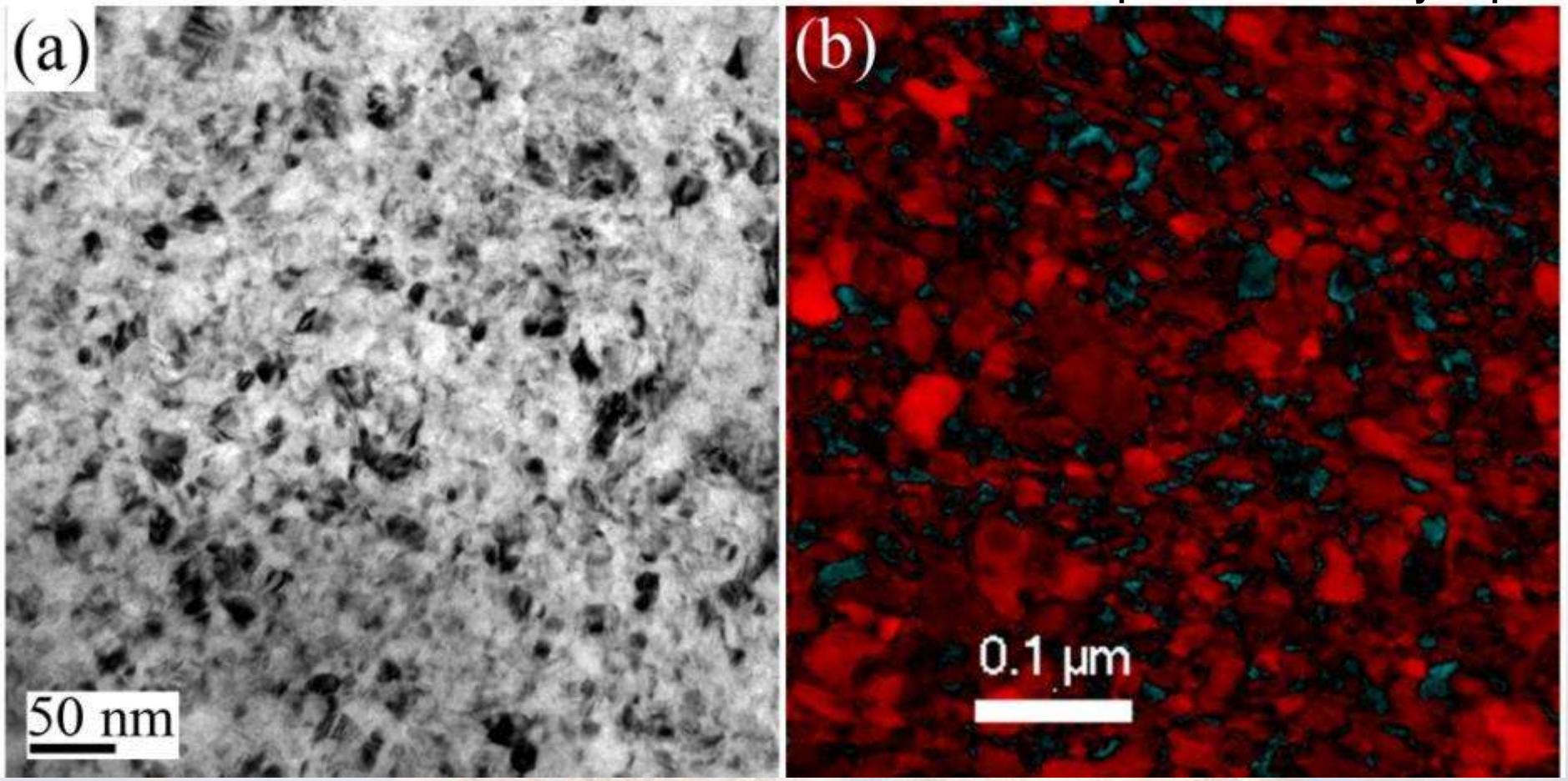
Full precession



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

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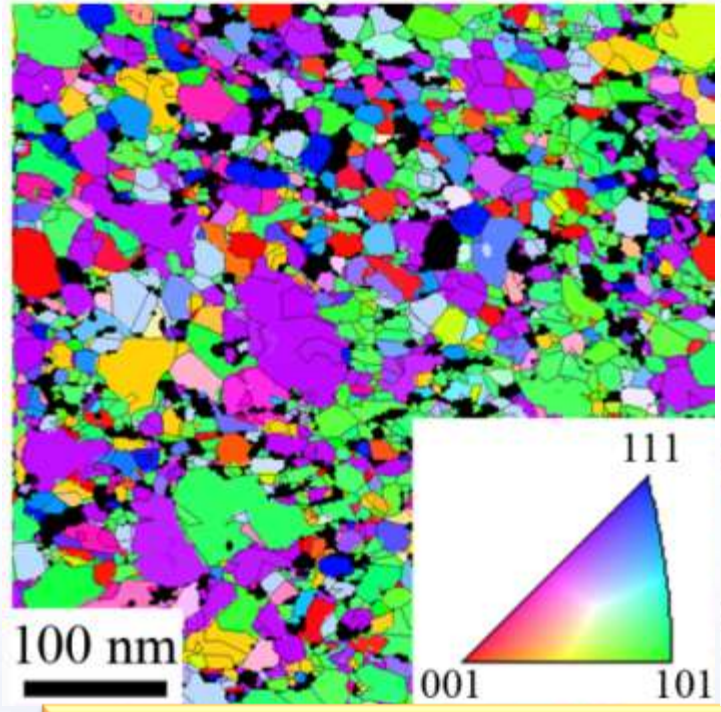
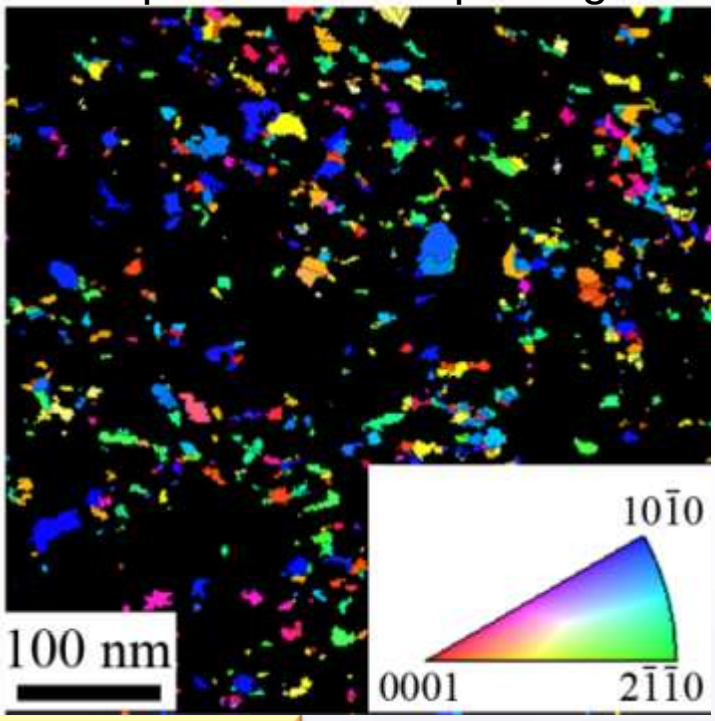
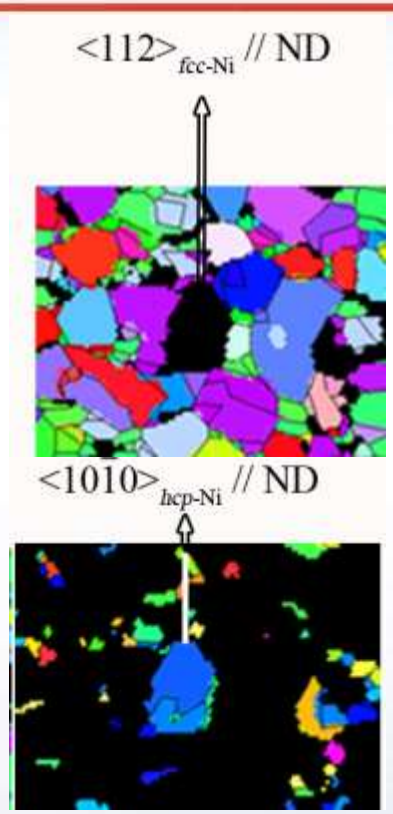
FCC and *HCP* Texture Determination in 50 nm As-deposited Ni Film

FCC phase inverse pole figure

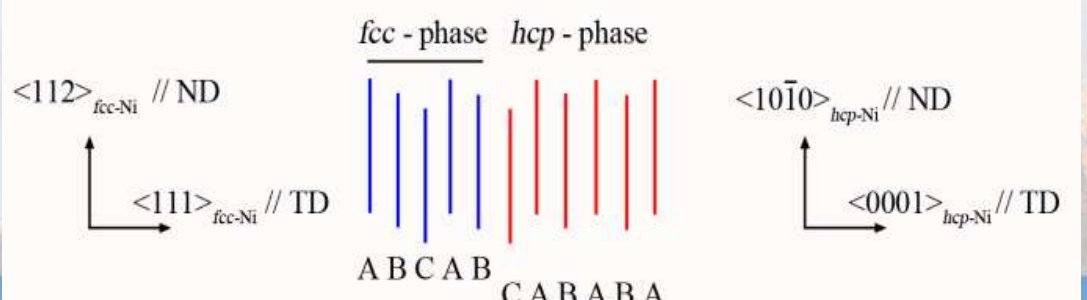


HCP phase inverse pole figure





Texture maps at the nanoscale obtained from a TEM



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

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

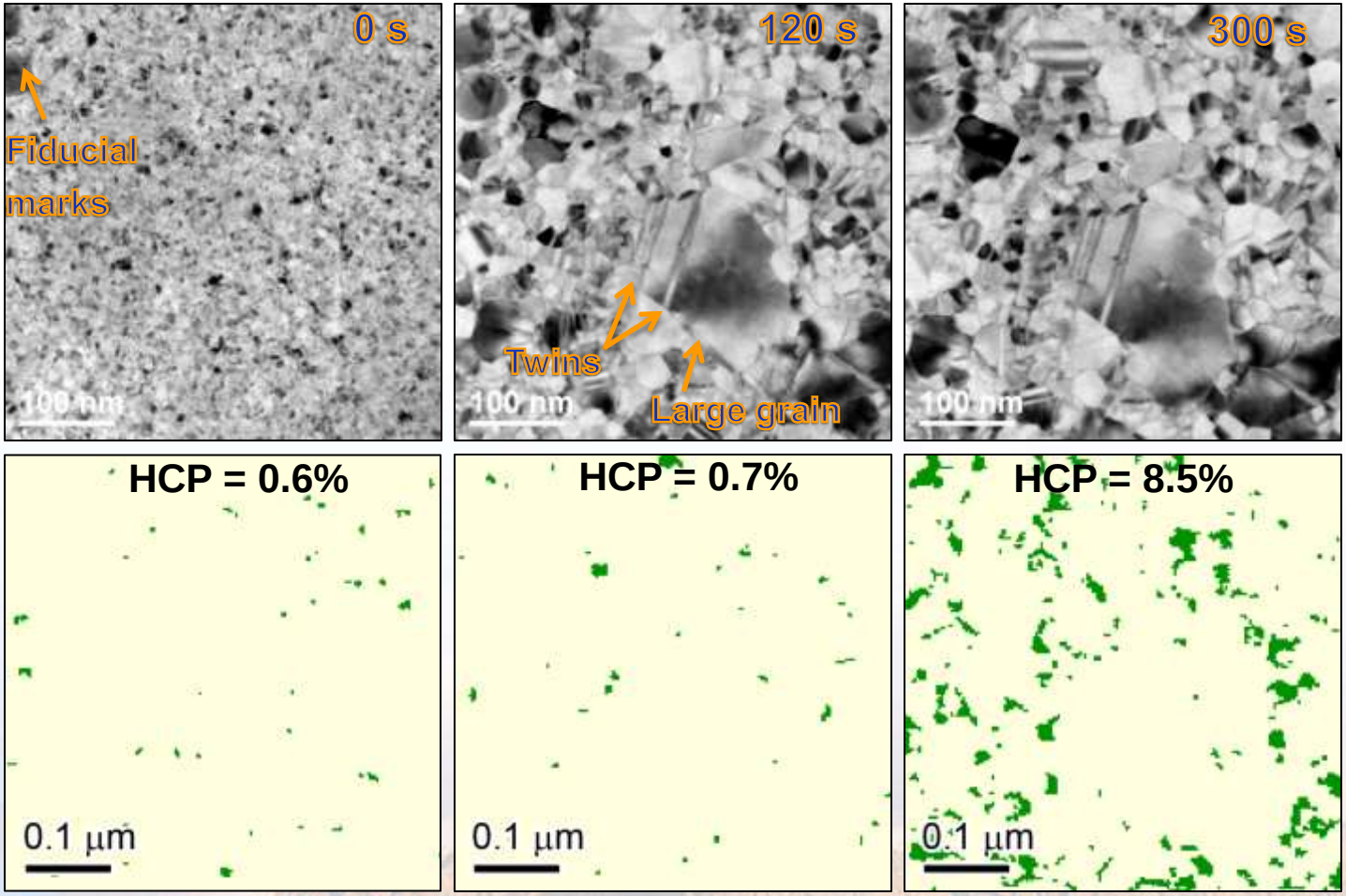
Recent Advancements in TEM heating Stages

Manufacturer	Stage	Image	Maximum Temperature	Approximate Thermal Volume of Sample
Gatan	628		1300 ºC	~1 x 10^{-2} mm^3
Hummingbird	Heating		1000 ºC	~1 x 10^{-2} mm^3
Hysitron	PI-95 MEMS heater		400 ºC	~1 x 10^{-7} mm^3
Protochips	Aduro		1200 ºC	~1 x 10^{-11} mm^3

Decreasing thermal volumes and other improvements have minimized drift and increased heating and cooling rates

In situ observation of microstructure and phase evolution

700 °C



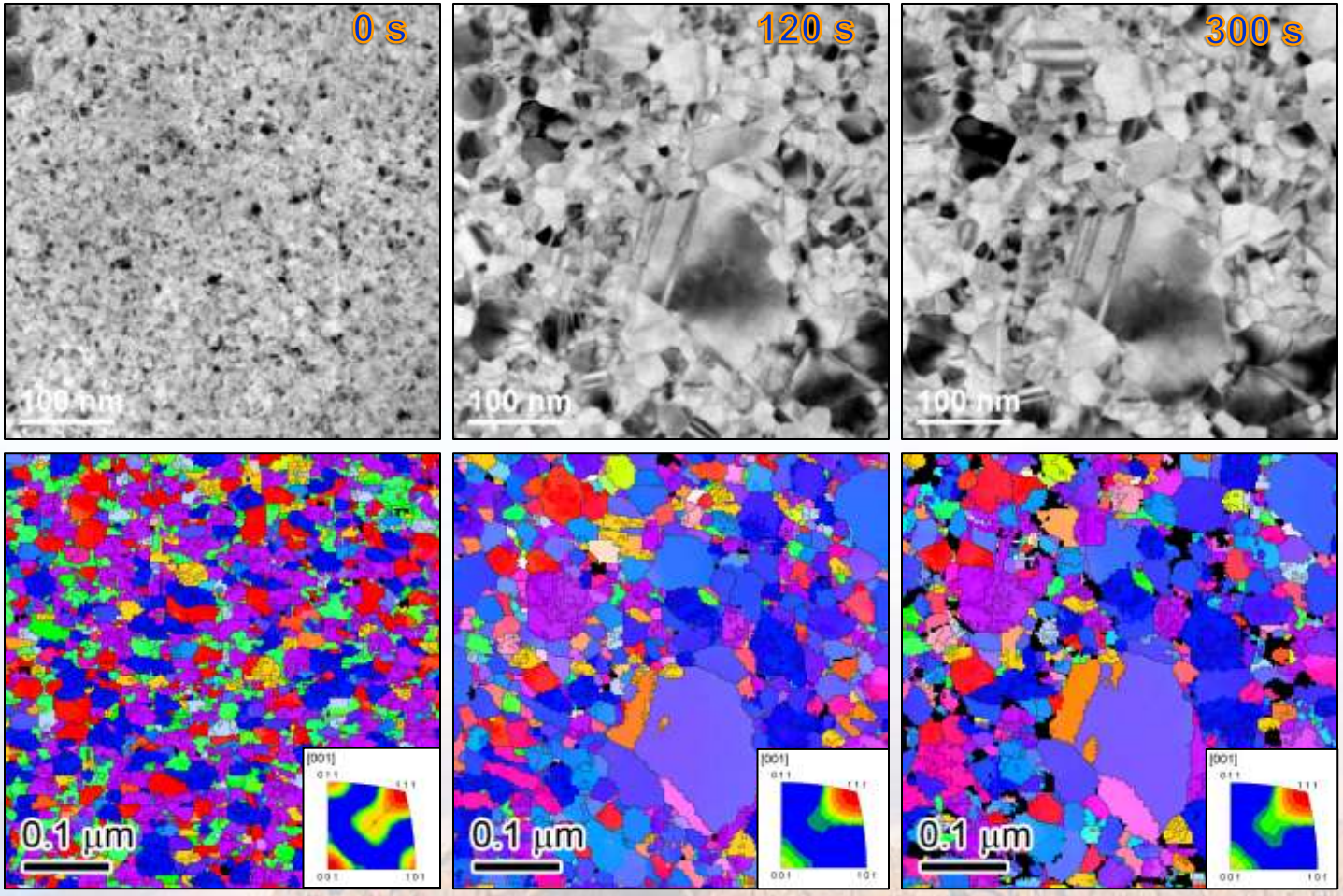
- The largest grain (~ 24156 nm^2) forms in 2 mins.
- Annealing twins form despite SFE of 125 mJ/m^2

~Number of Grains	0 s	300 s
FCC	370	150
HCP	30	150

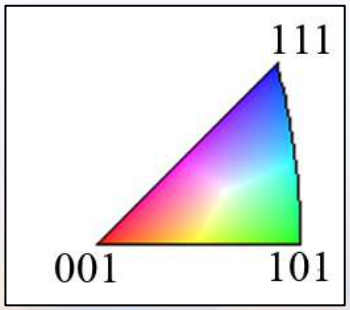
New HCP grains are detected during annealing

In situ observation of *FCC* phase texture evolution

700 °C



***FCC* texture evolves from nearly random to <111> during annealing**



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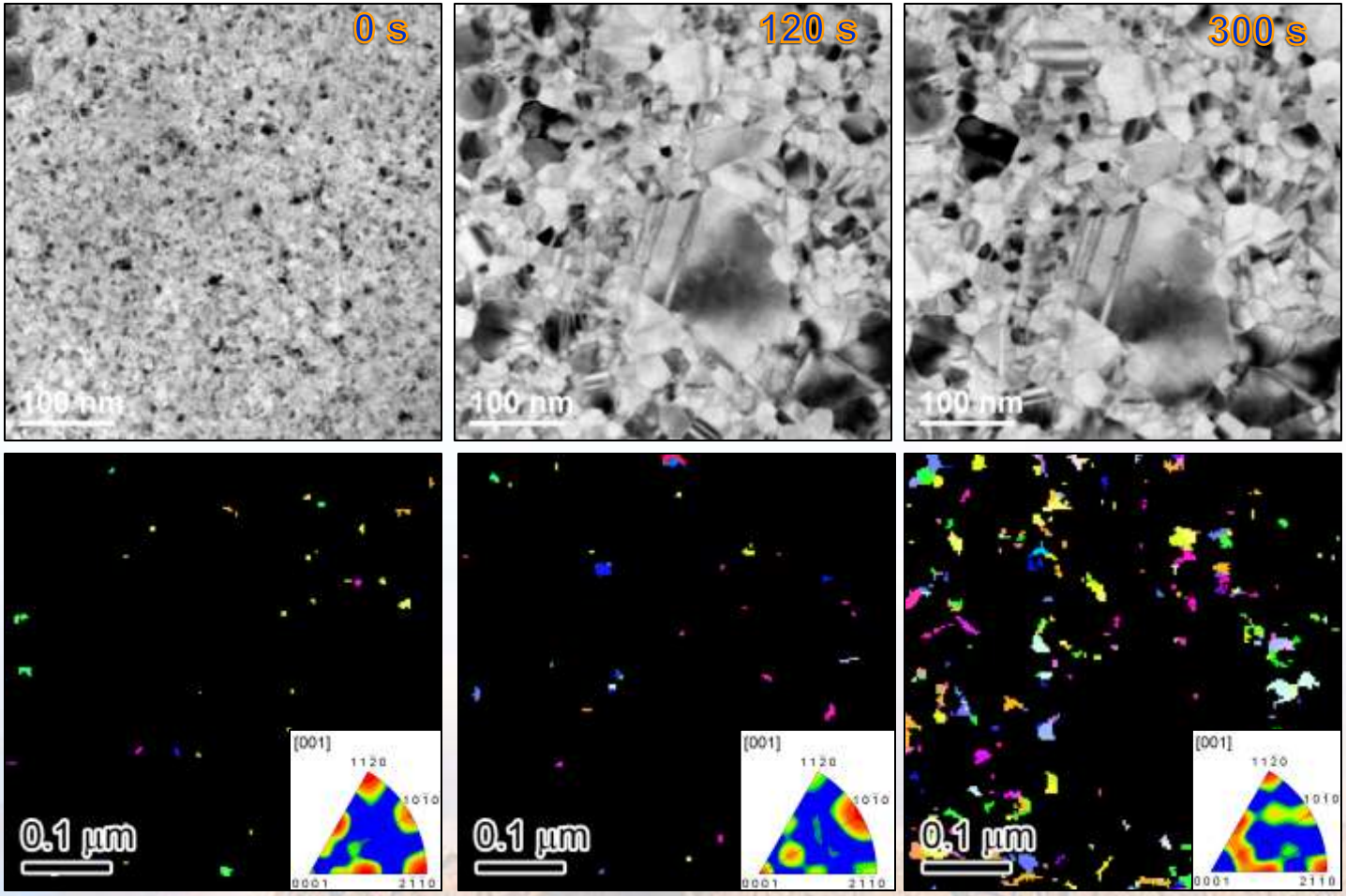
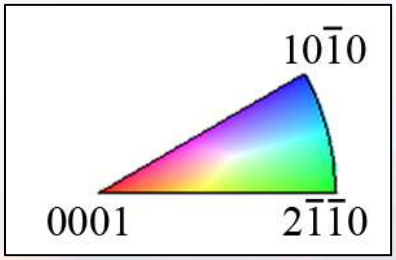
In situ observation of *HCP* phase texture evolution

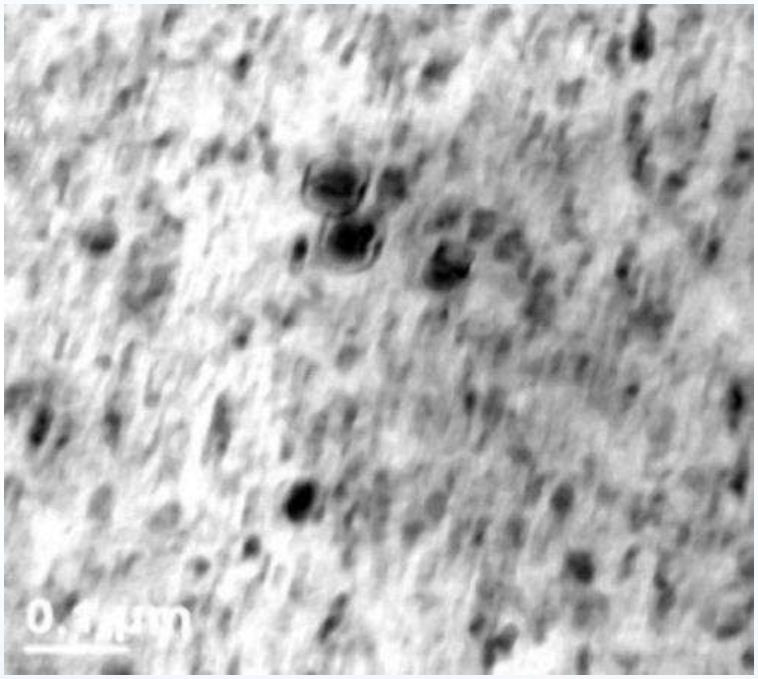
700 °C

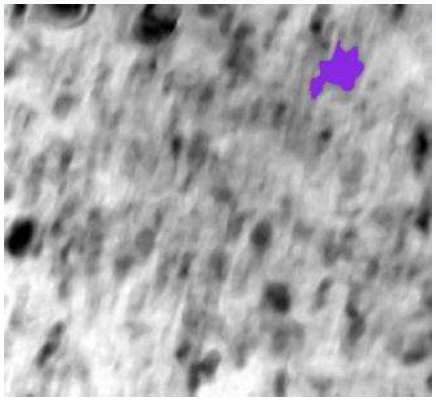


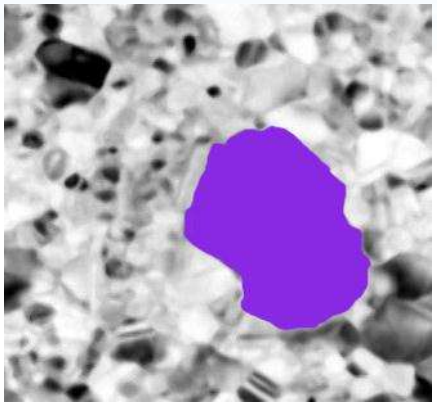
Texture evolution is complex and still under study

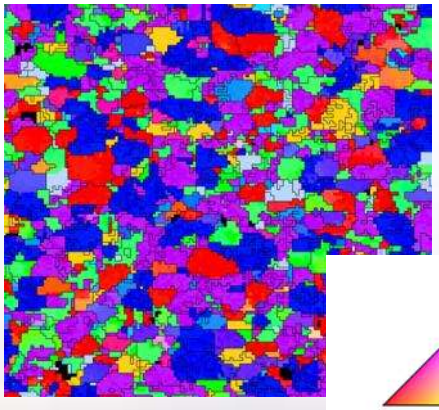


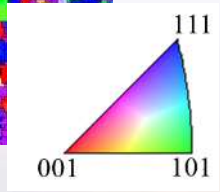
Tracking of Individual Grain Evolution

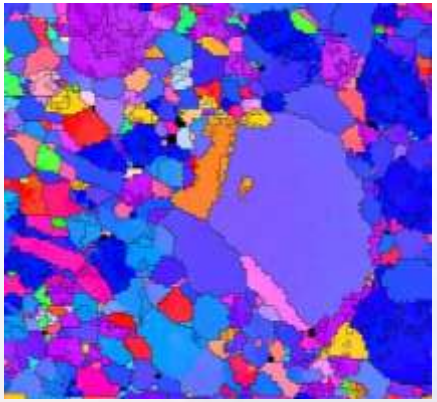


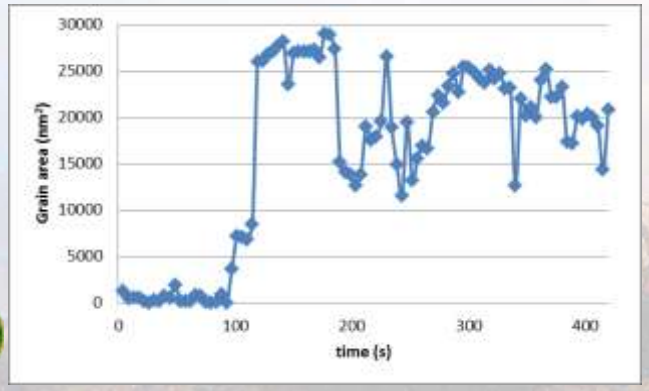






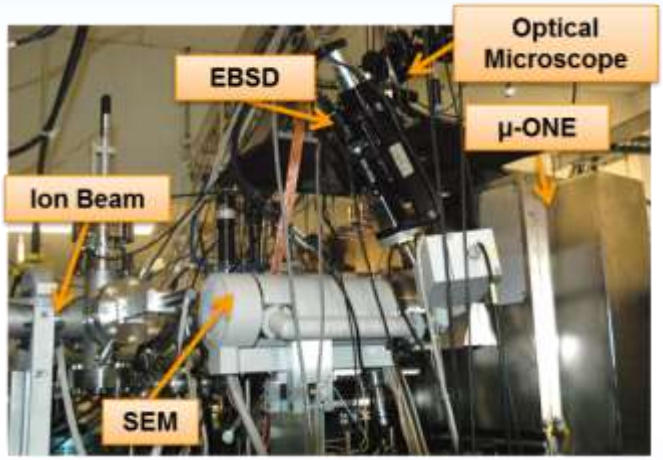


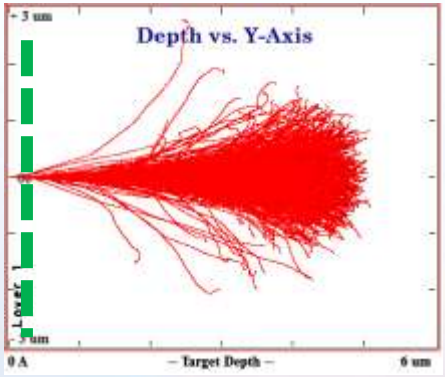




In situ TEM heating and precession microscopy permit tracking of texture evolution

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

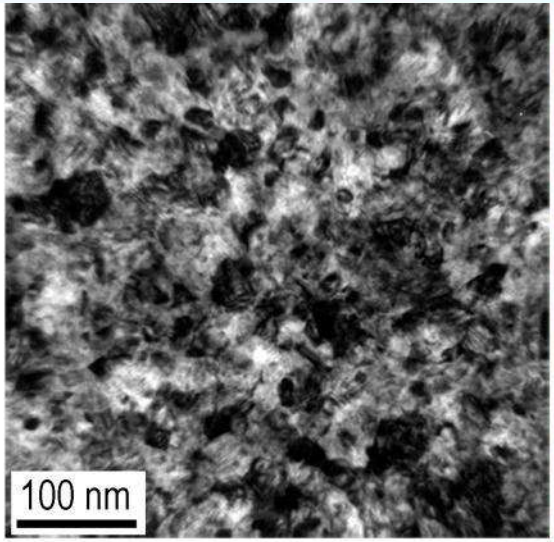


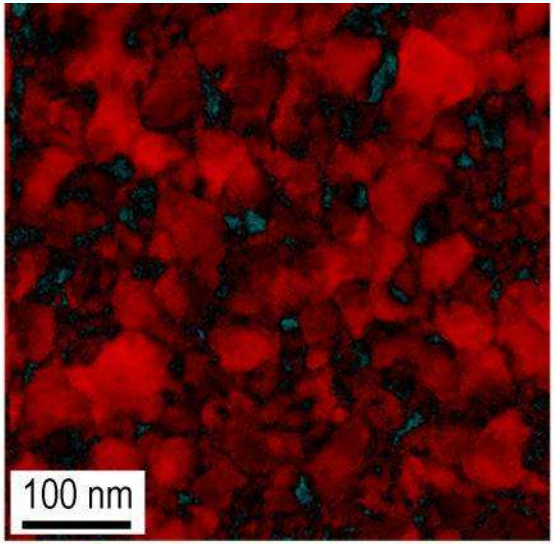


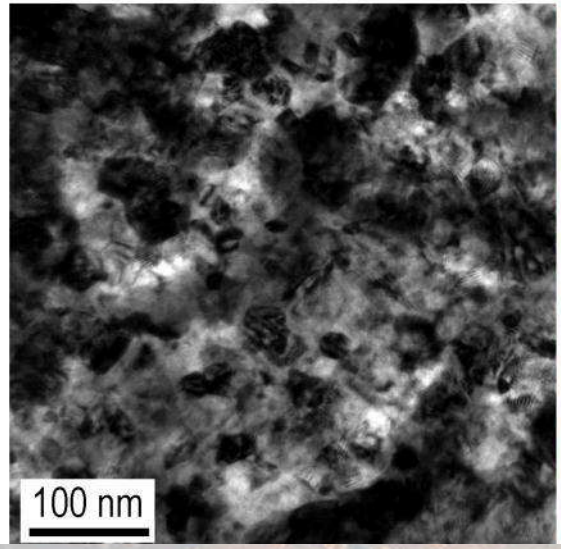
Despite the minimal interaction predicted in 100 nm film, grain growth was observed and increased *HCP* phase resulted

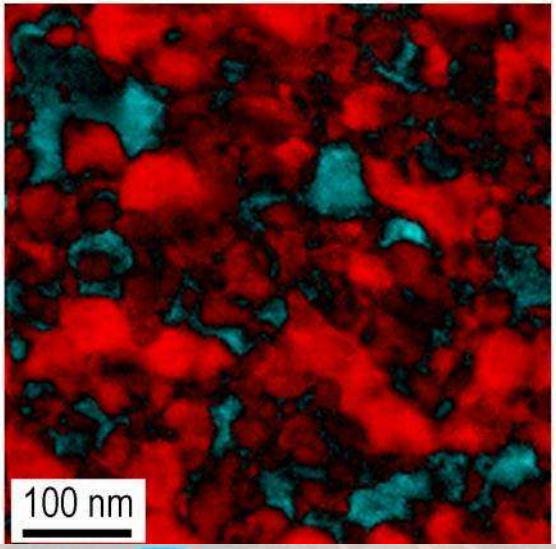
As-deposited

35 MeV Ni 3 x 10^{14} cm^{-2}







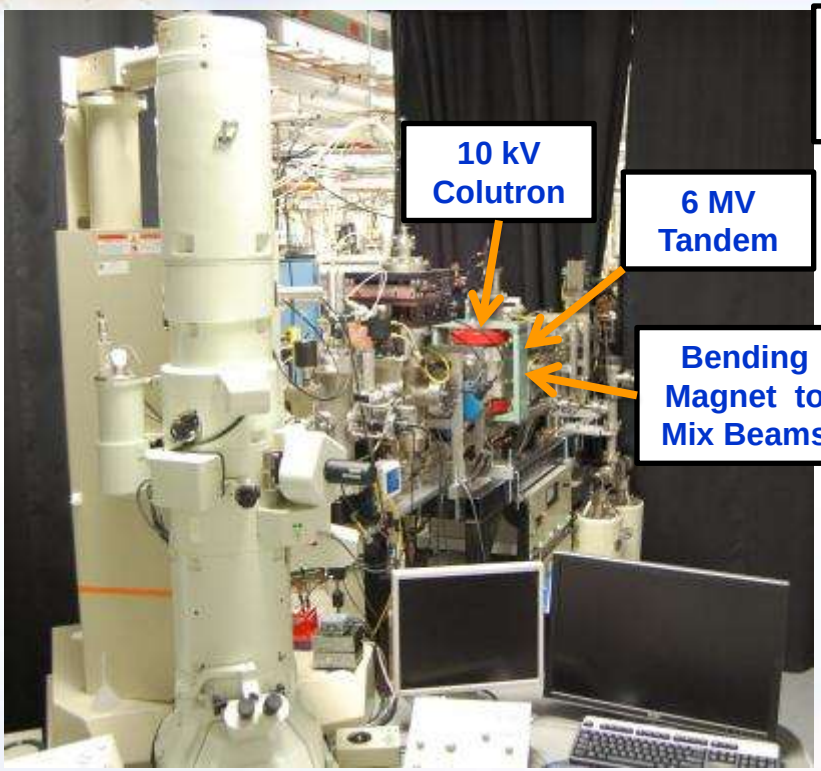


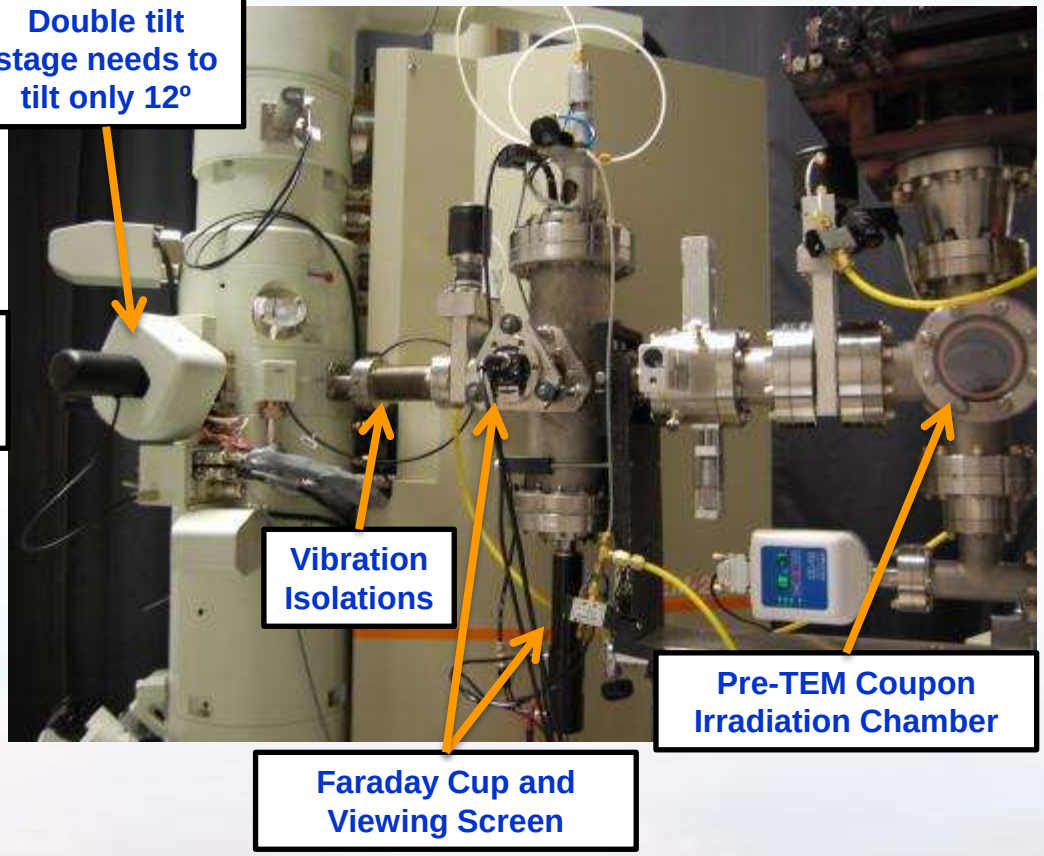
FCC phase *HCP* phase

Sandia National Laboratories

In situ TEM Beamline

Collaboration with: D. Buller, J. A. Scott, and B.L. Doyle





Beam burn from
14 MeV Si

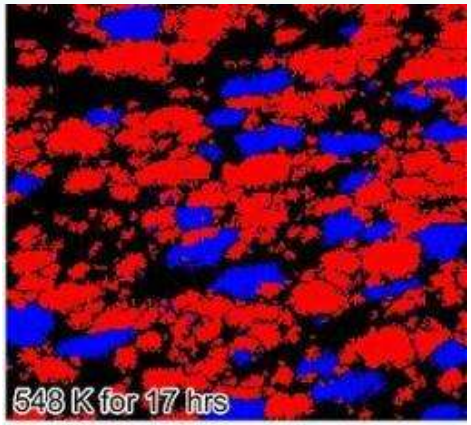
Tandem beamline into the TEM is completed and operational

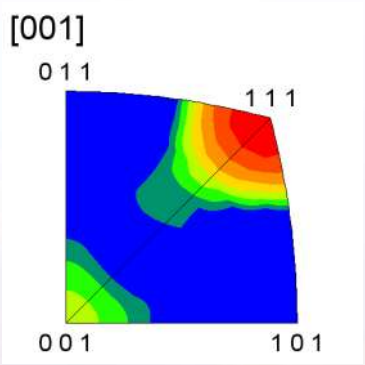
Colutron beamline is assembled and operational

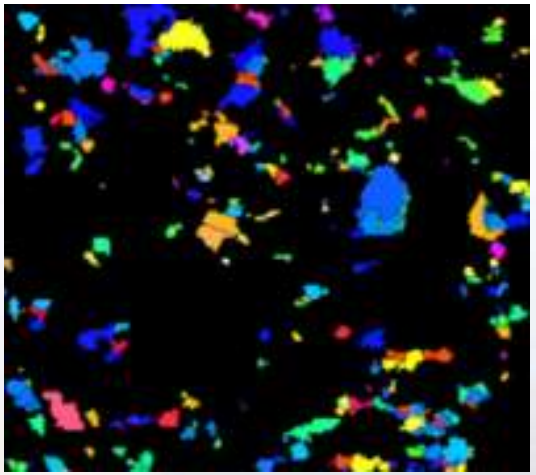
We hope to have concurrent heavy and light ion irradiation facility operational in 2012

Conclusions

- **Unique and unexpected structures can result from nanograined processing**
  - SFT and metastable HCP phase
- **Thermal, mechanical, and radiation stability of nanograined metals are probably intertwined**
- ***In situ* TEM provides ability to determine active mechanisms in a system during thermal, mechanical, or radiation loading**
- **Precession microscopy provides a unique tool to study the grain orientations and boundary relationships of nanostructured thin foils**







In situ TEM heating, straining, or irradiation + precession microscopy = Greater insight into structural evolution

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