

Ion Beam Modification of Nanomaterials *via* *In Situ* Ion Irradiation TEM

SAND2012-7100C

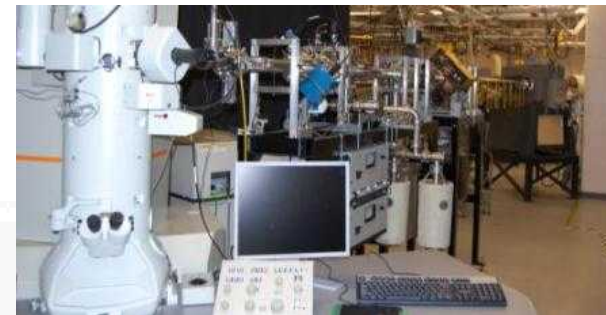
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IBMM 2012
September 3rd, 2012

Sandia's Ion Beam Laboratory



In situ Ion Irradiation TEM



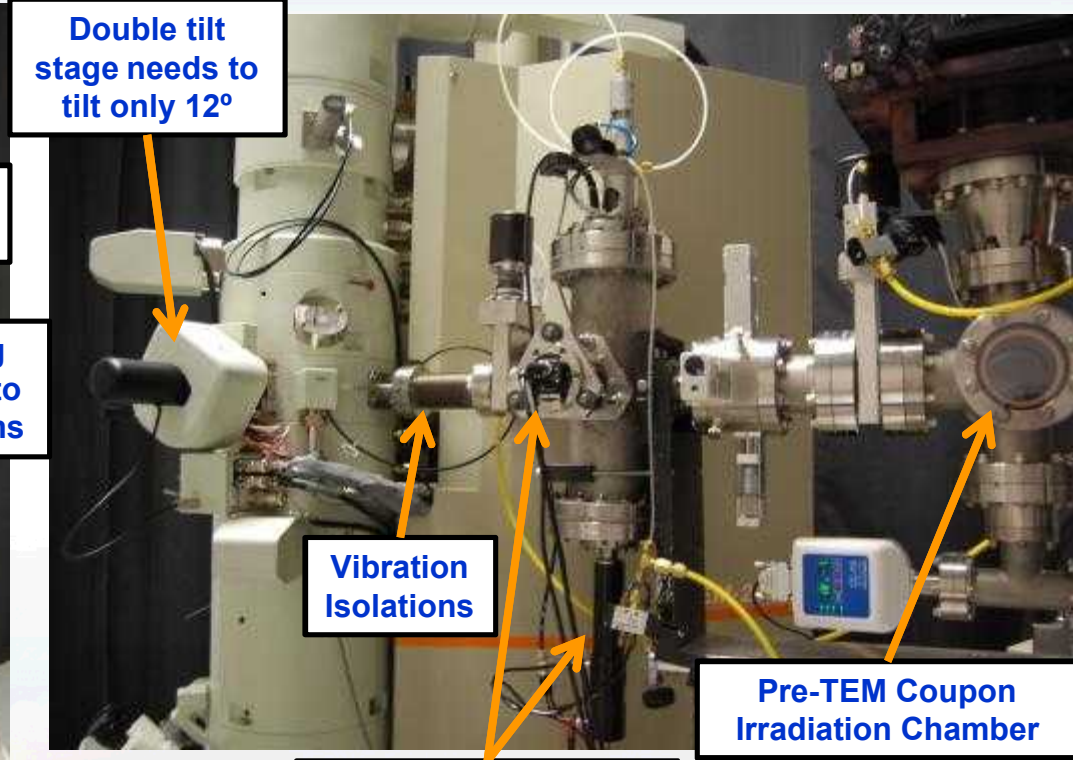
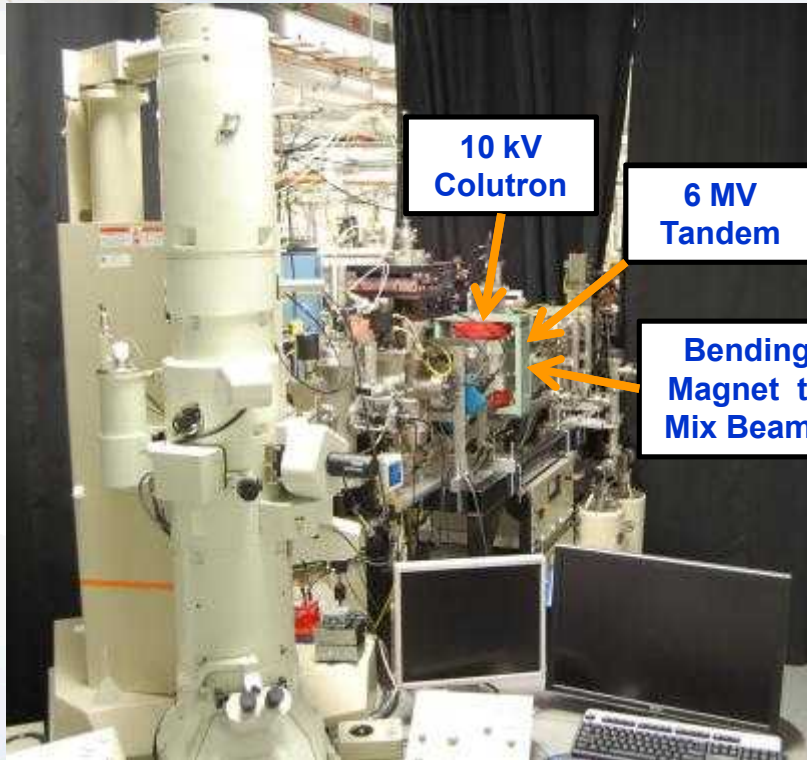
Outline

A versatile *in situ* TEM capability to
characterize radiation effects in materials
4D evolution of irradiated Au nanoparticles
Microstructure in PLD nanograin Ni



Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000. This work was partially supported by DOE, Office of Science, Office of Basic Energy Sciences.

In situ Ion Irradiation TEM (I³TEM)



Beam burn from 14 MeV Si

TEM and all stages fully operational
MeV beams from Tandem routinely used
keV beams from Colutron a work in progress
Light collection for luminescence demonstrated

TEM and Beam Line Layout

JEOL 200kV LaB₆ TEM

Tandem Beams:

3 MeV He⁺ Si³⁺ Cu³⁺ Au³⁺

14 MeV Si³⁺

Coultron Beams:

10 keV D²⁺

10 keV He⁺

Simultaneous MeV/keV beams

Electron tomography stage

Heating stage

Gas heating stages

Liquid stage

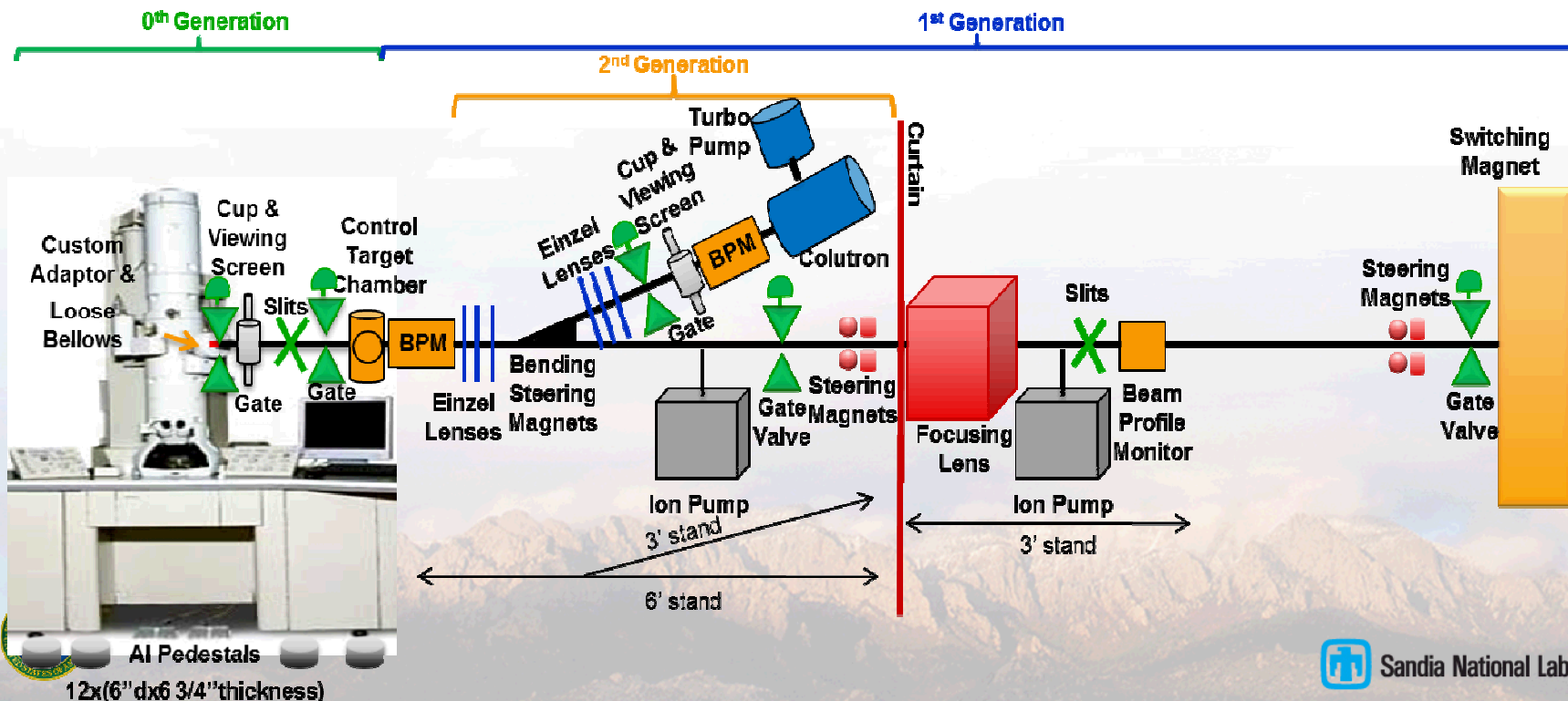
In situ optical properties (PL, CL, IL)

Recently funded:

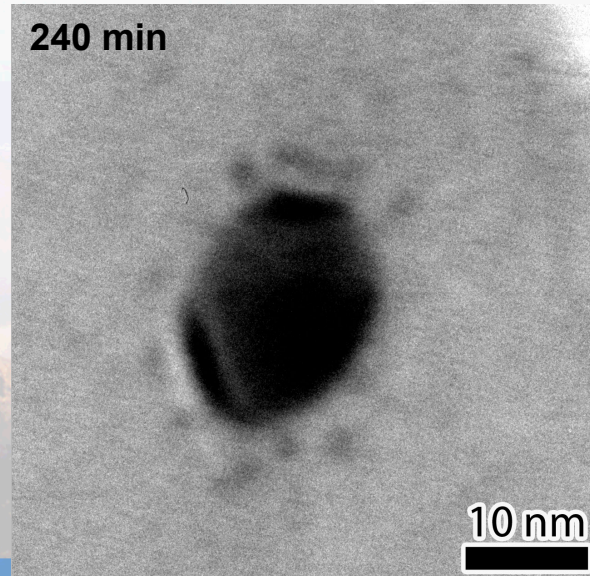
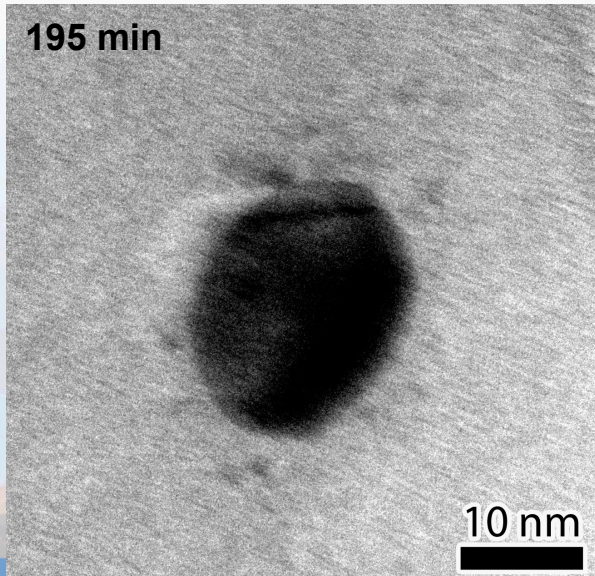
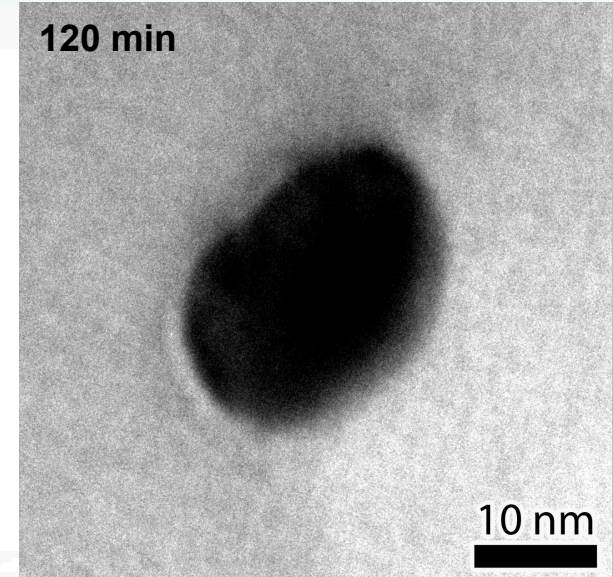
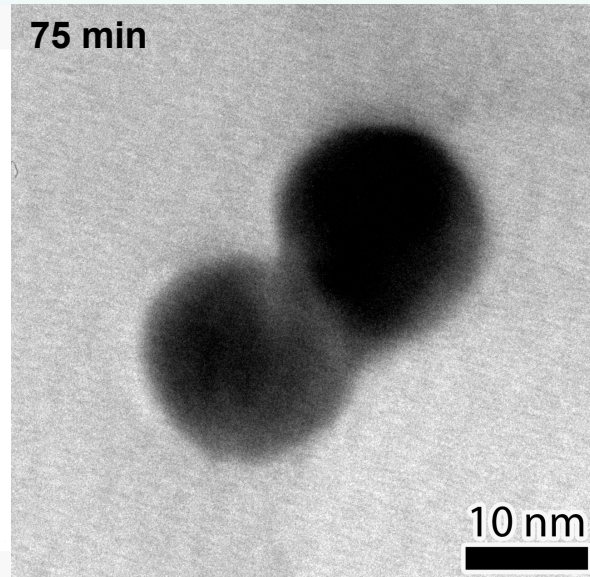
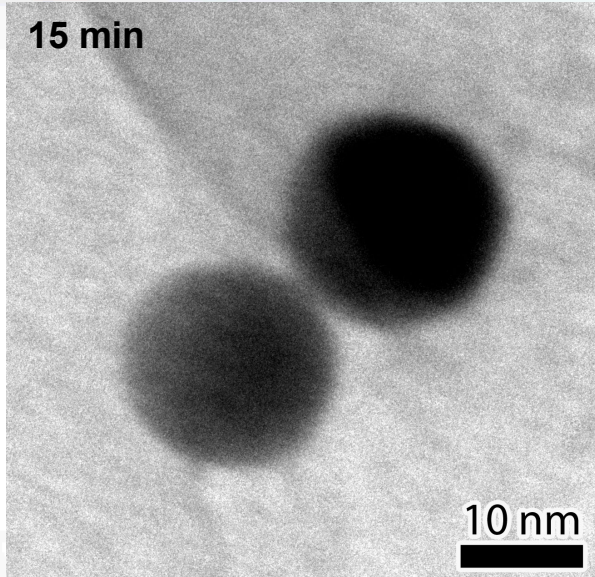
Electrical measurement stage

Nanomechanical indent/straining stage

Unique combination of *in situ* TEM and ion beam capabilities permit investigation of synergistic effects



Au Nanoparticles irradiated with 100 nA of 3 MeV Cu^{3+}



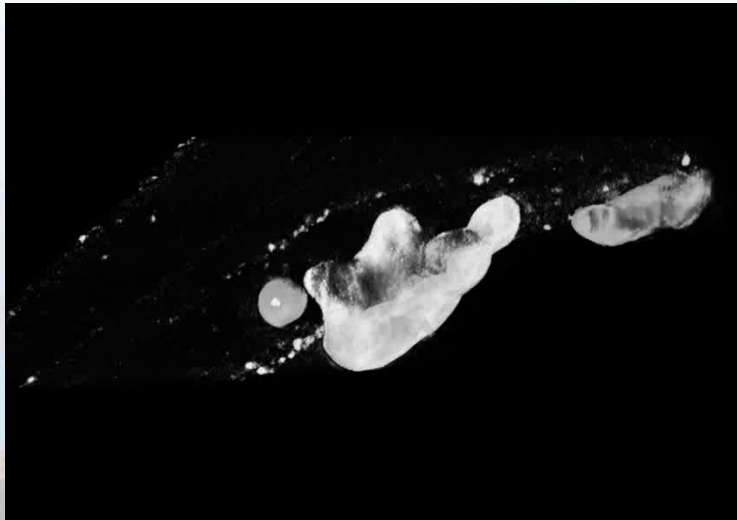
Over 4 h, two spherical particles coalesced, then smaller particle began to sputter off creating a halo around parent particle.

Tomography of Au nanoparticles irradiated with 80nA 3 MeV Cu³⁺

Unirradiated

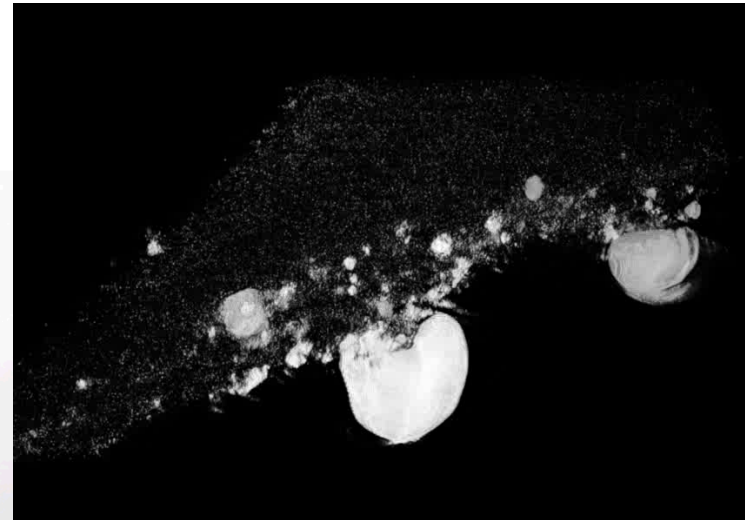


30 minutes



Recent advancements in TEM control and reconstruction software permit collection and production of 3D model of the “transmission” micrograph.

60 minutes



Tilt series were collected after each dose of irradiation resulting in 4D tomography with 3D reconstructions showing radiation damage over time.

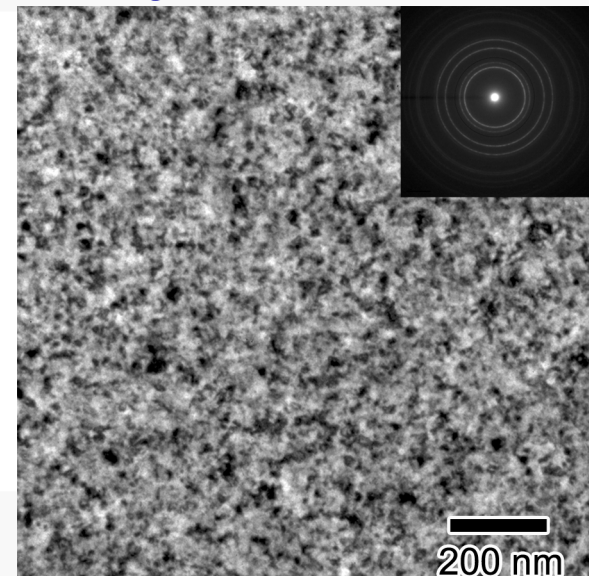
Abnormal Grain Growth in Nanocrystalline Ni

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

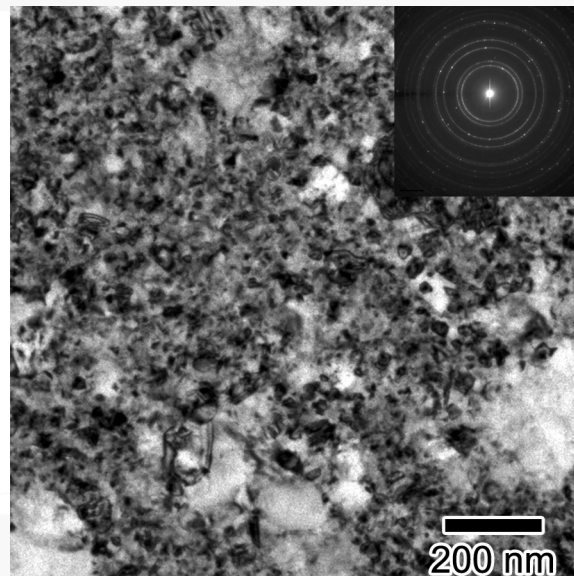
Nanograined

Bimodal

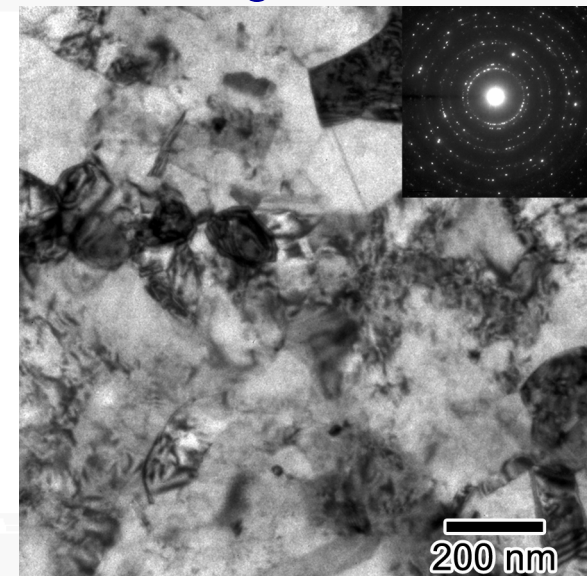
Ultra-fine grained



80 nm-thick As-deposited on Si



1 hr at 275 °C



1 hr at 375 °C

Pulsed laser deposition of thin, nanograined Ni films

Abnormal grain growth regularly observed in nanograined materials

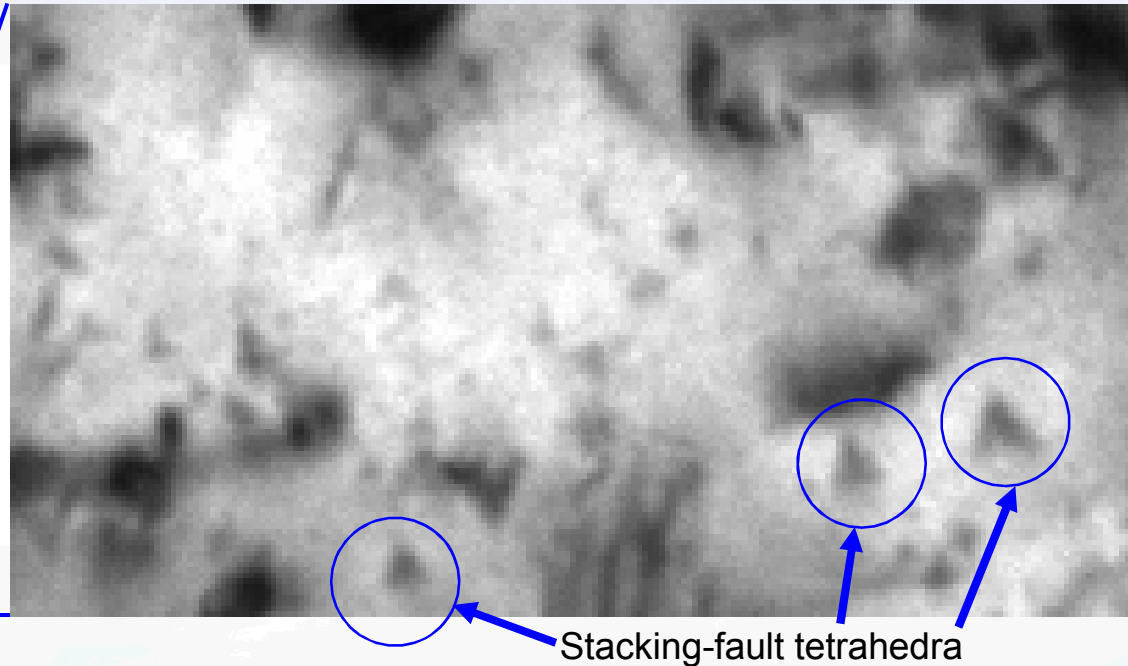
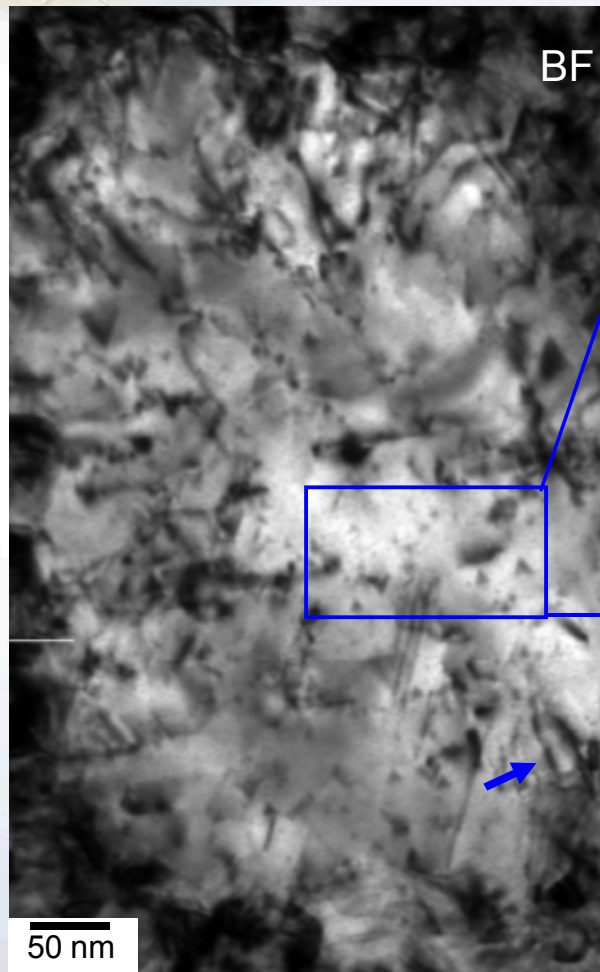
Thermodynamics vs kinetics of the process – what controls it?

The role of defects, grain boundaries, and phase boundaries



In Situ Annealing of PLD Ni (no irradiation)

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



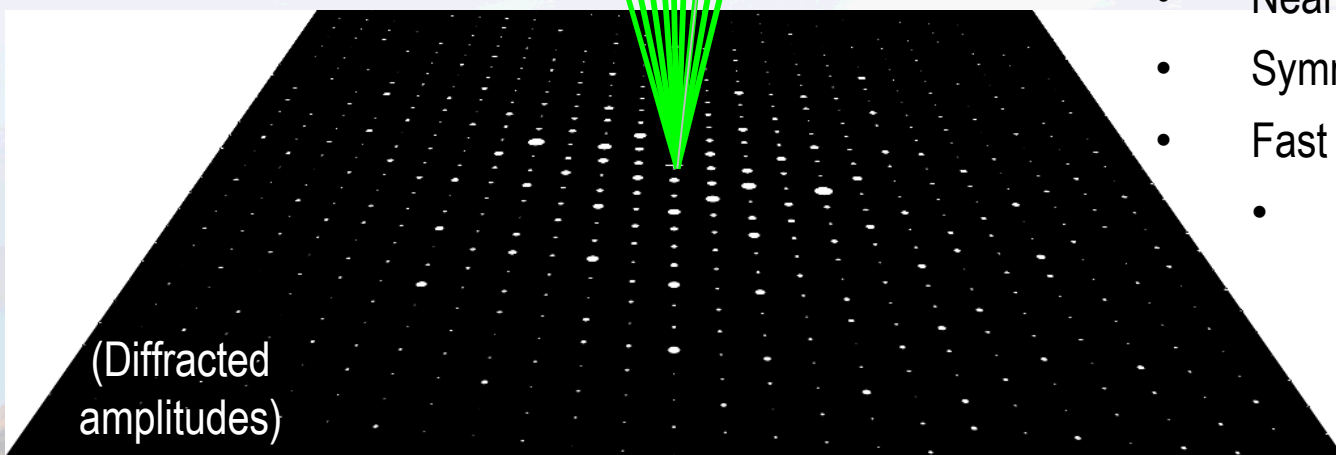
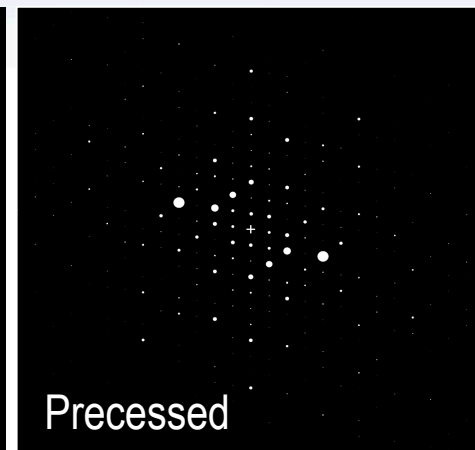
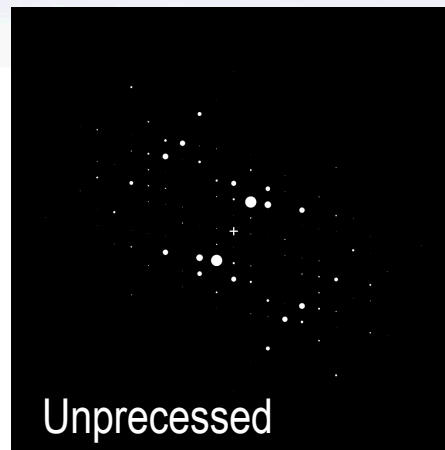
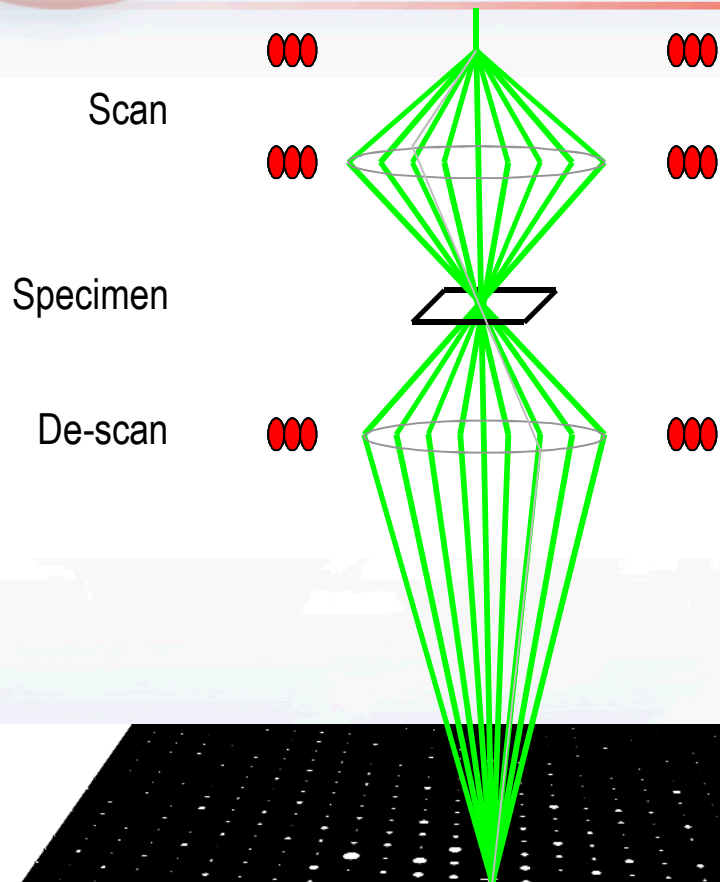
Multitude of defects in annealed PLD Ni

- SFT form **at temperature**
- 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 boundaries

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



Nanograin phase identification and orientation using Precession Electron Diffraction Microscopy at UT



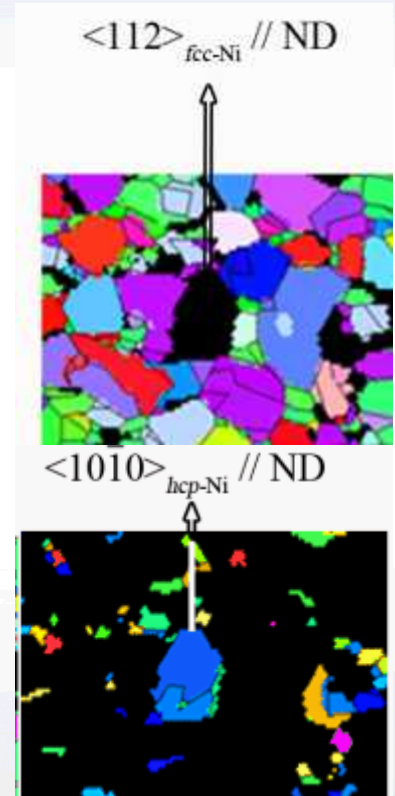
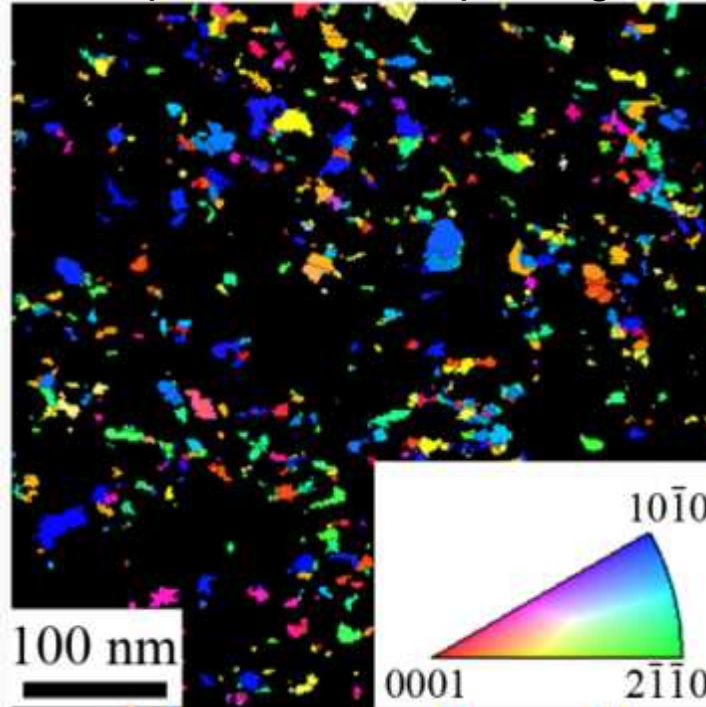
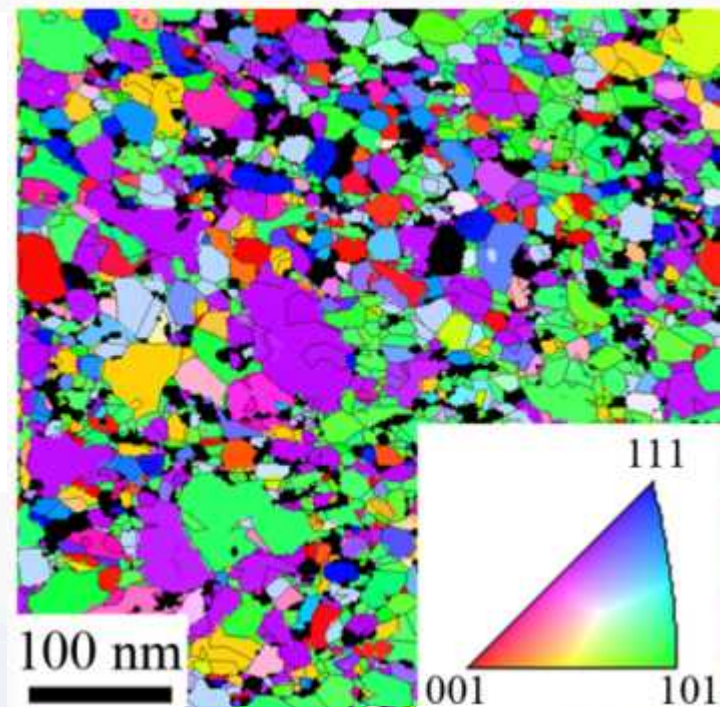
Advantages:

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

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

FCC phase inverse pole figure

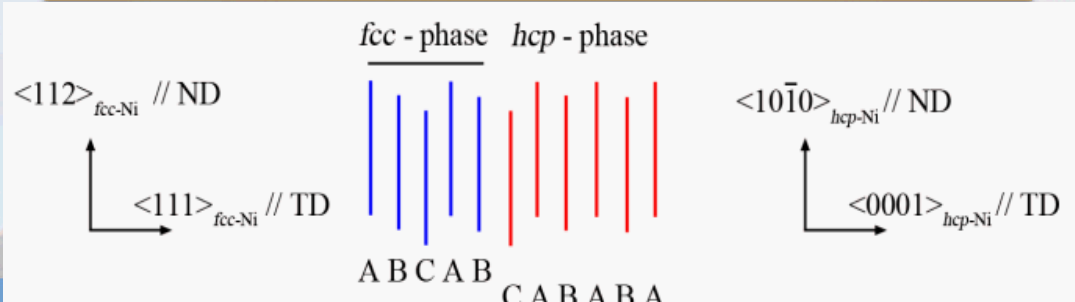
HCP phase inverse pole figure



Texture maps at the nanoscale obtained via precession electron diffraction in a TEM

$\langle 112 \rangle_{\text{FCC-Ni}} // \text{ND}$ results in an in-plane $\langle 111 \rangle_{\text{FCC-Ni}} // \text{TD}$

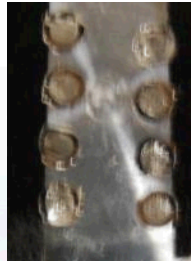
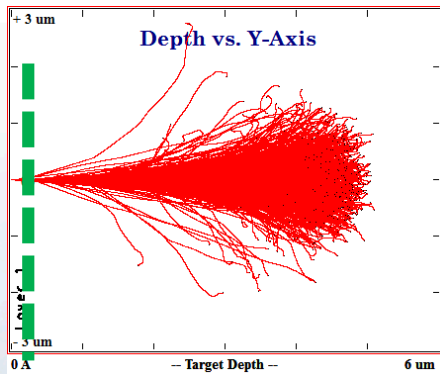
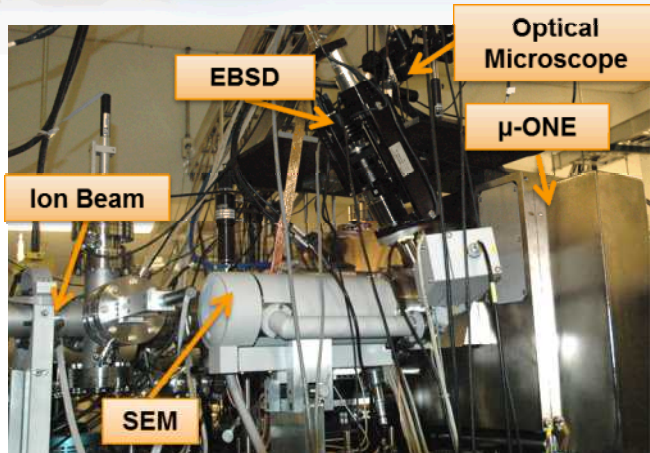
High energy PLD may introduce stacking faults leading to a $\langle 0001 \rangle_{\text{HCP-Ni}} // \text{TD}$



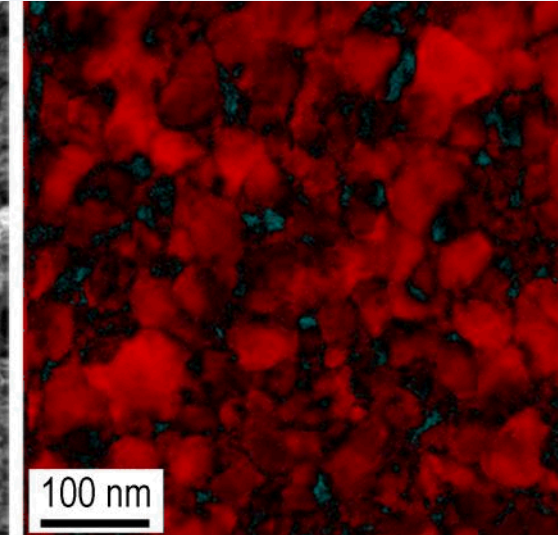
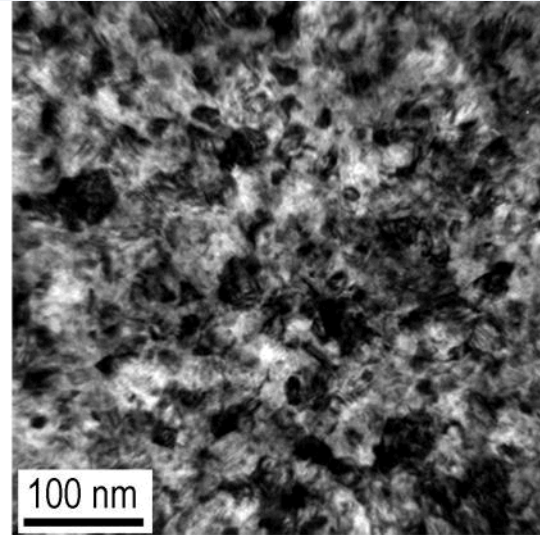
FCC and HCP Phase Evolution after 35 MeV Ni Irradiation

Microbeam irradiation at SNL

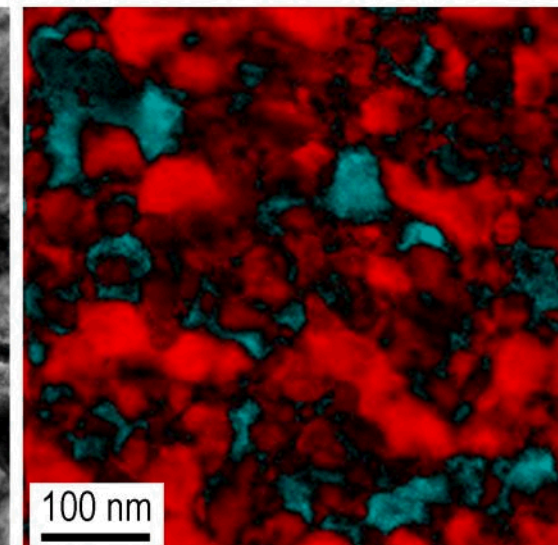
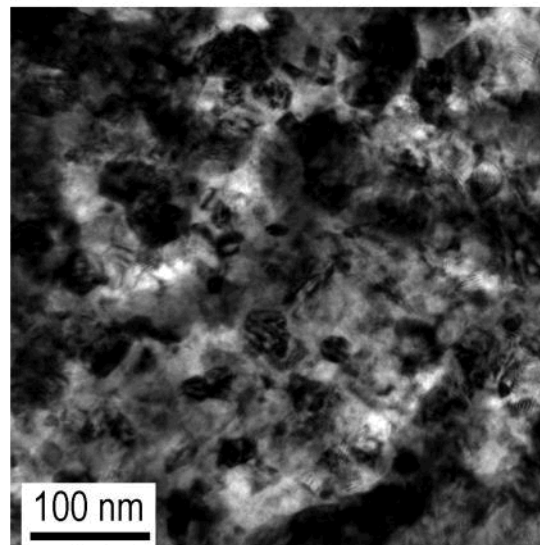
Precession TEM at UT Austin



As-deposited



35 MeV Ni 3×10^{14} cm⁻²



Irradiation increases abnormal grain growth in HCP

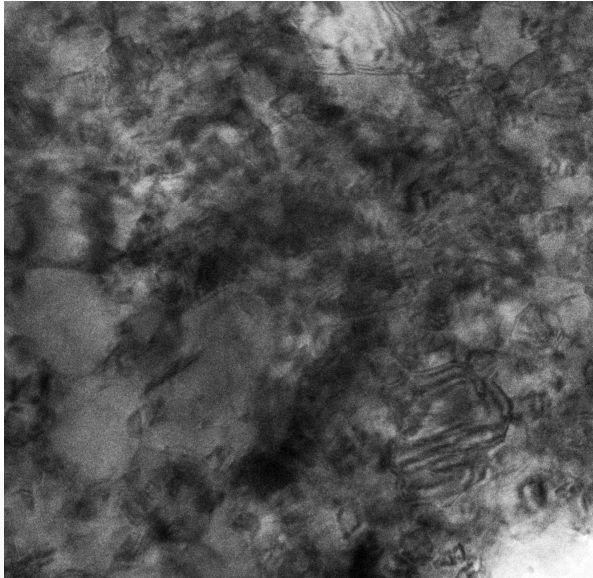
■ FCC phase ■ HCP phase

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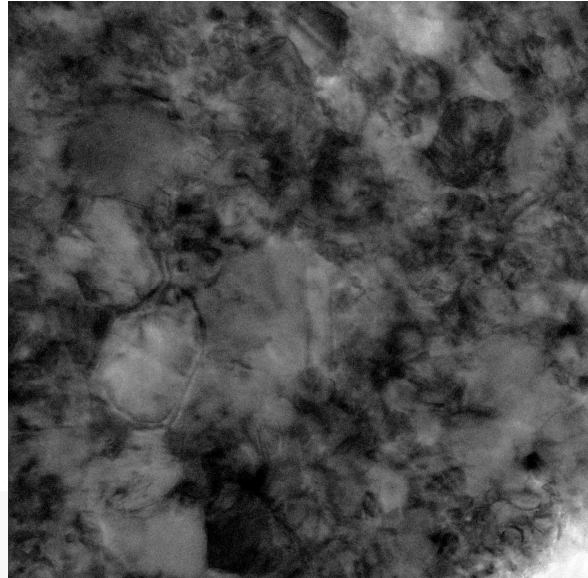


Initial I³TEM Results on PLD Ni

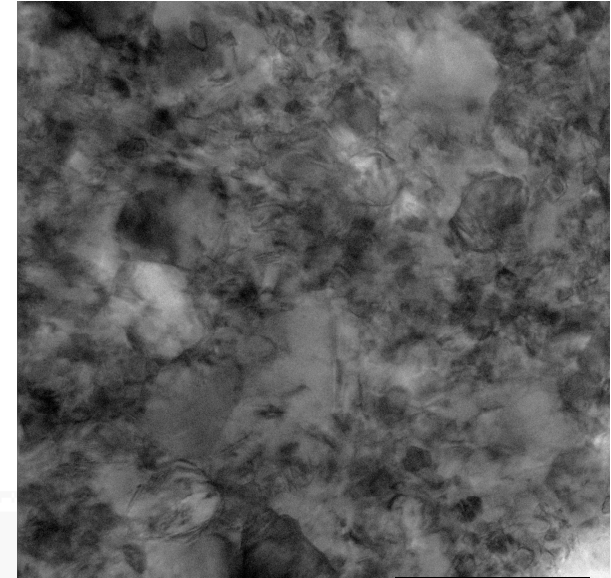
0 dpa



5 min (~.02 dpa)



10 min (~.04 dpa)



200 nm

- 80 nm PLD Ni deposited on salt and transferred to Cu grid
- Pre-annealed 2 hrs at 250°C
- Irradiated with a 60 nA 5 MeV Si³⁺ beam with $d_{\text{beam}} \sim 3$ mm (larger than TEM grid)
- Sample irradiated 45° normal to ion beam

Some structural changes observed during *in situ* TEM





Conclusions

- **Versatile and flexible I³TEM is functional at the Ion Beam Laboratory at Sandia**
- ***In situ* tomography of materials under irradiation demonstrated with Au nanoparticles**
- **Abnormal grain growth in nanograined films can now be monitored *in situ* to untangle the thermodynamics vs kinetics**

