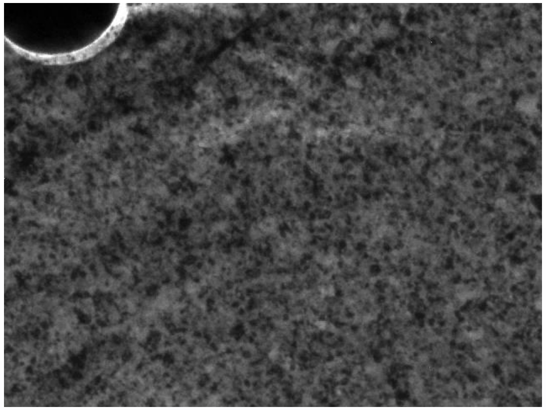
# Exploring the Thermal, Mechanical, and Radiation Stability of Nanocrystalline Metals via *In situ* TEM

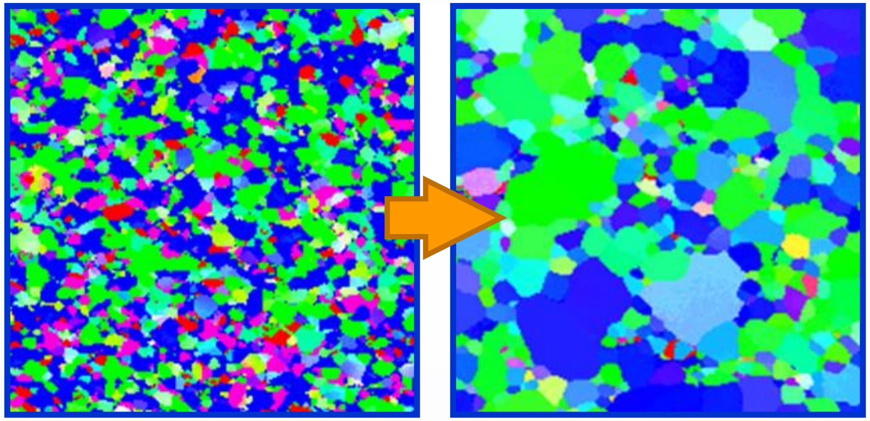
SAND2016-4807C

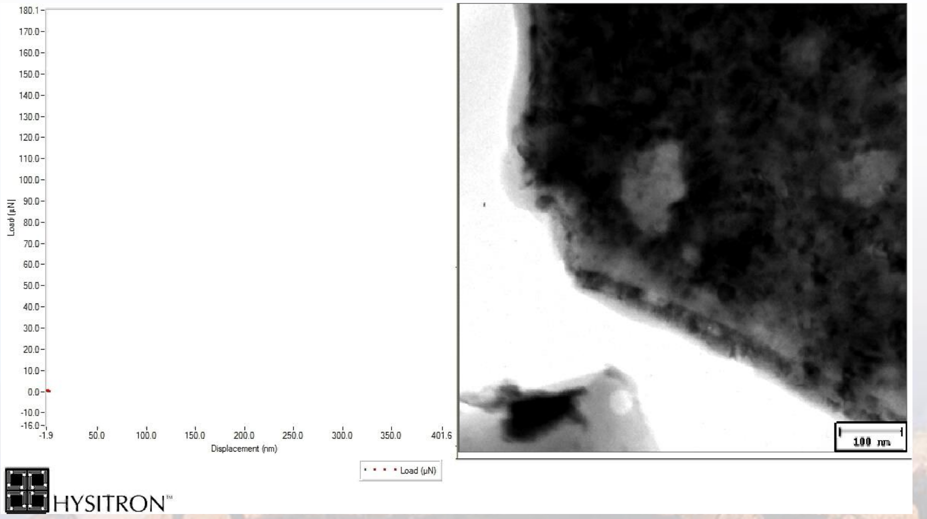
K. Hattar, D.C. Bufford, & B.R. Muntifering

Sandia National Laboratories

May 30, 2016





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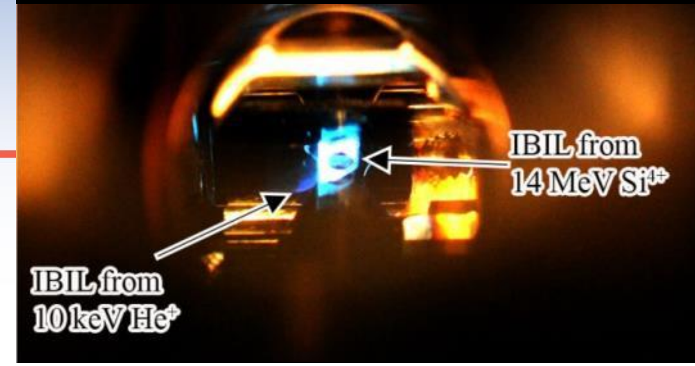
# Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

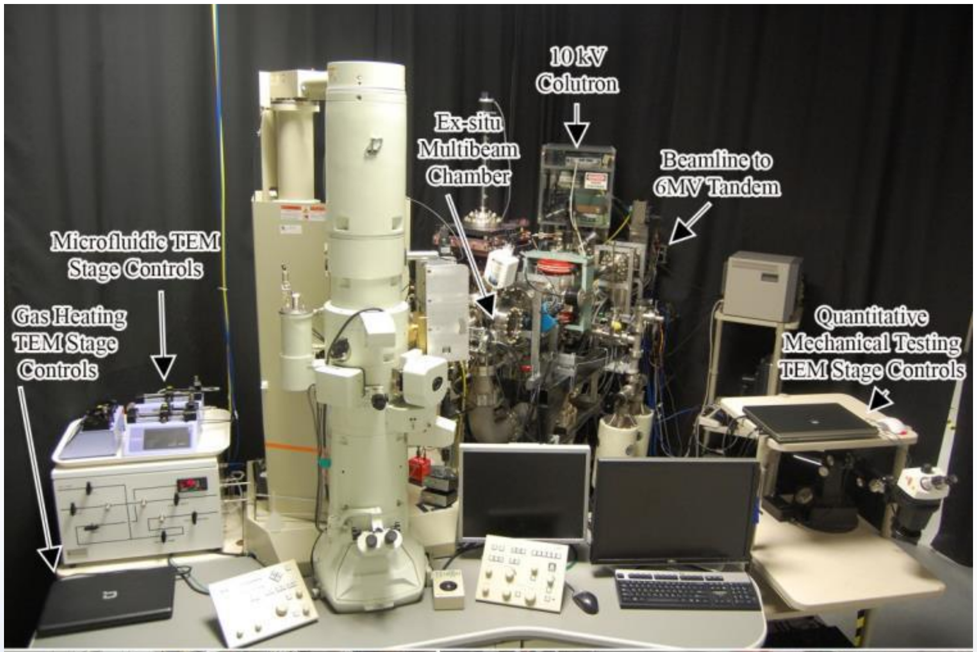
Collaborator: D.L. Buller

IBIL from a quartz stage inside the TEM



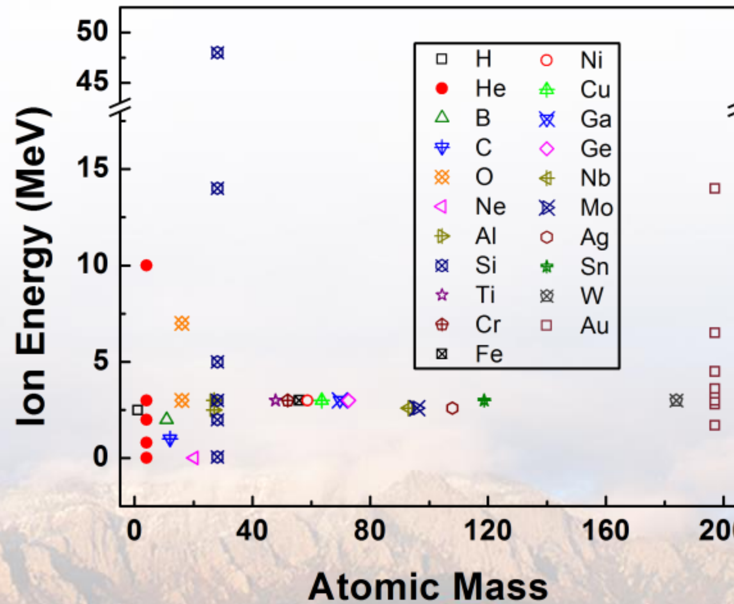

**Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution**

## 10 kV Colutron - 200 kV TEM - 6 MV Tandem





## Ion species & energy introduced into the TEM

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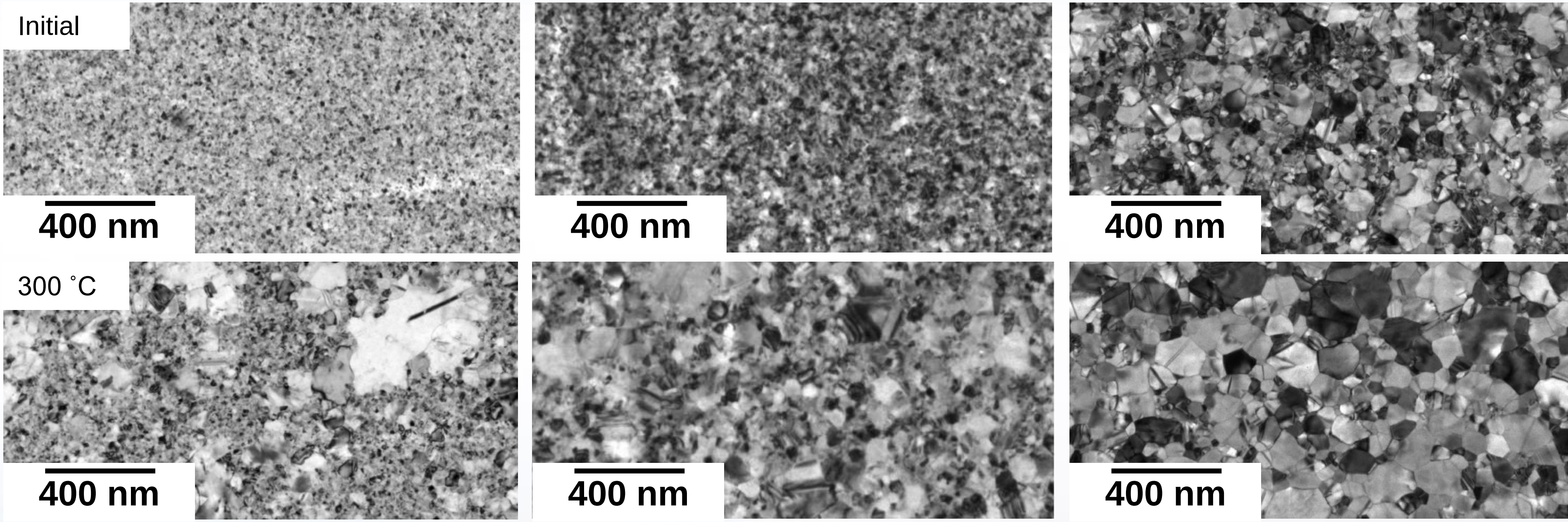
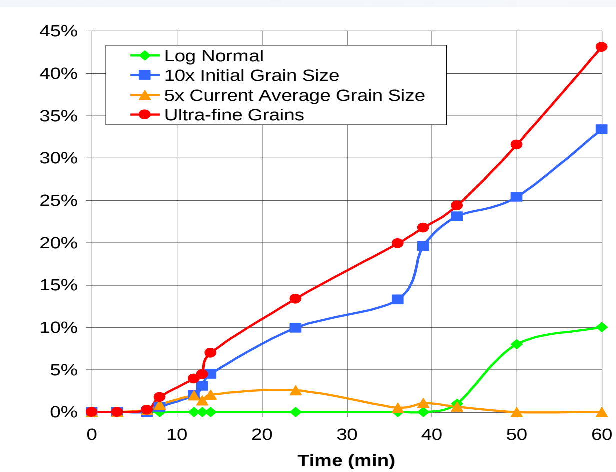
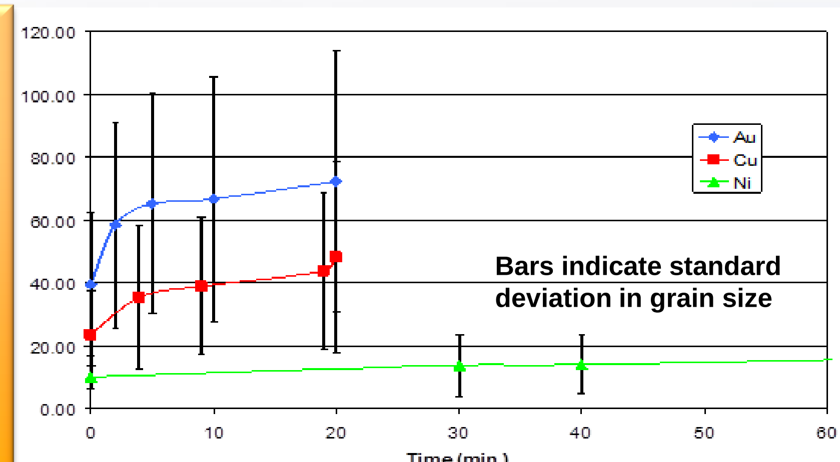
# Limitations of Classical Grain Size Analysis during *In situ* TEM Experiments

PLD Ni | PLD Cu | PLD Au

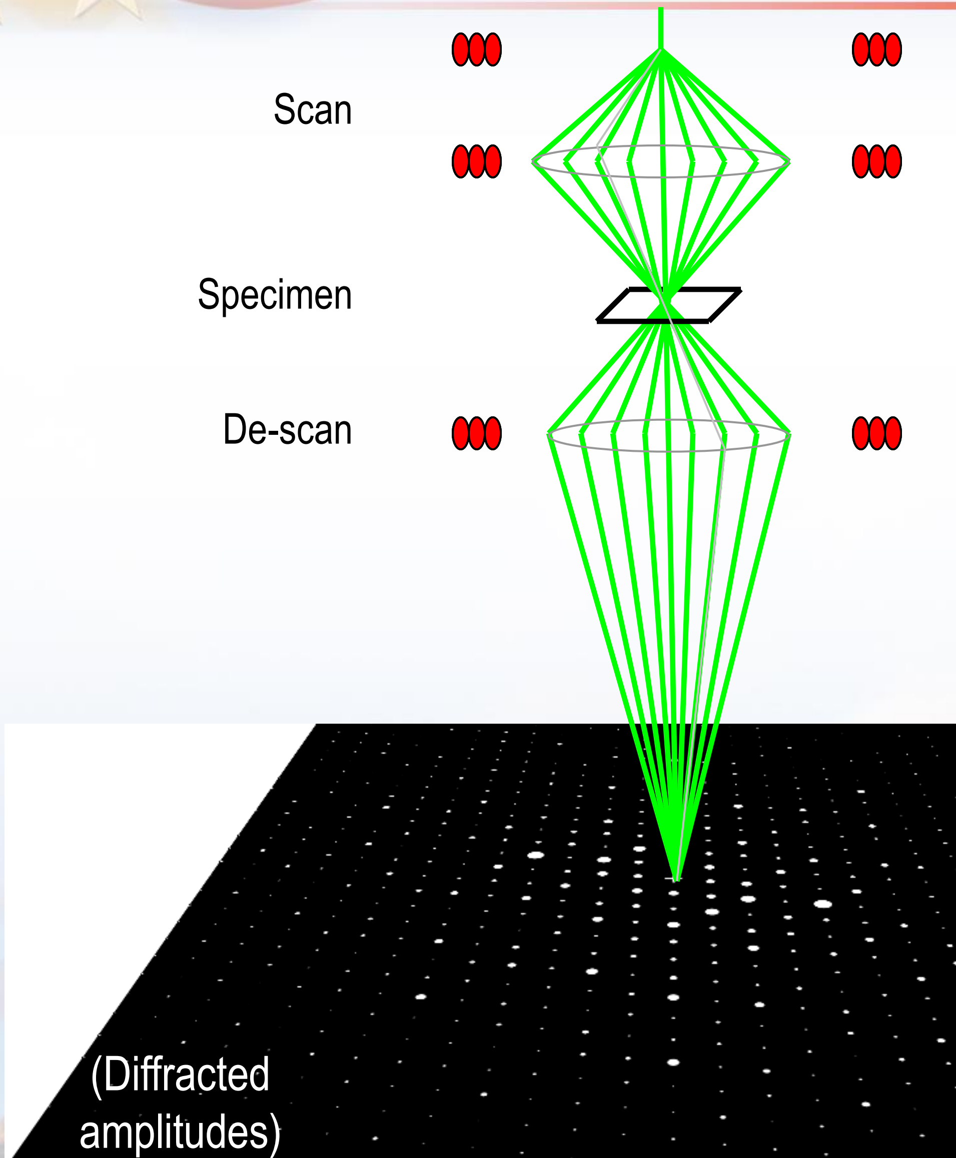


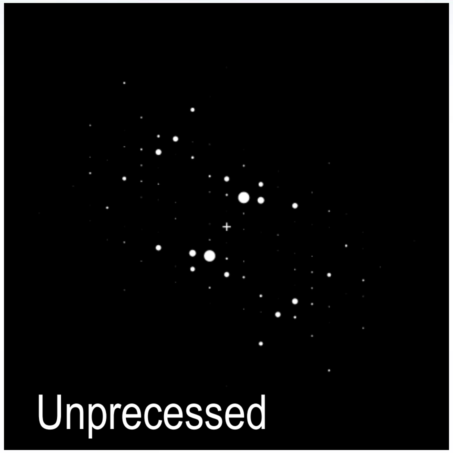



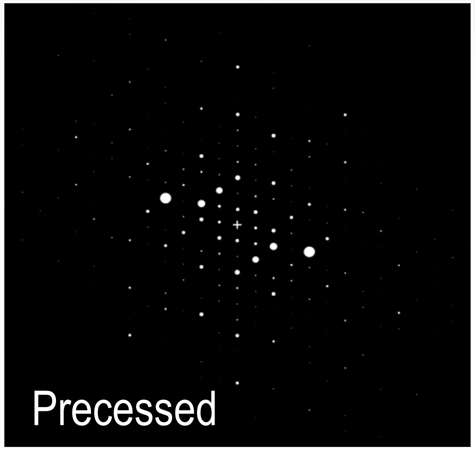

**Classical line intercept analysis provides insight into grain size and distribution, but is time intensive and provides little analysis of texture and grain boundary character**



# Precession Electron Diffraction Microscopy




**Advantages:**

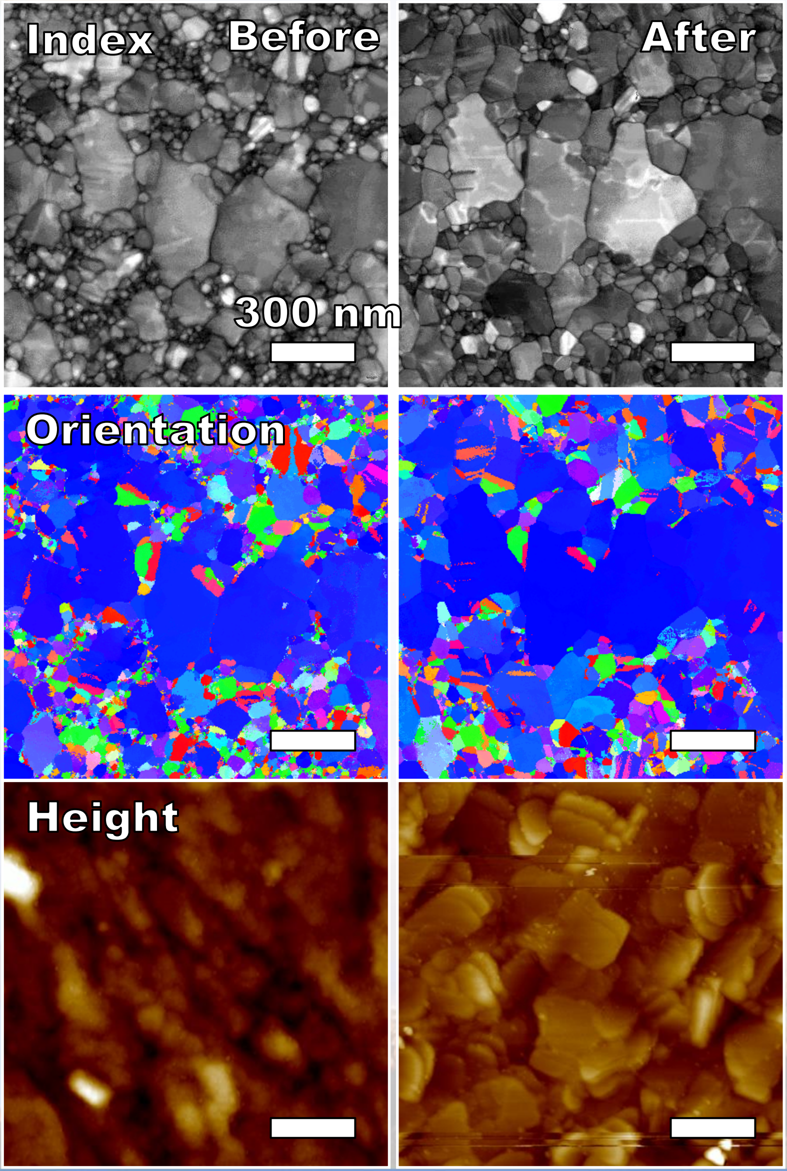
- < 10 nm spatial resolution
- Near kinematical electron diffraction
- Symmetry ambiguities are resolved
- Fast and automated acquisition
  - ~200 grains in 15 min.

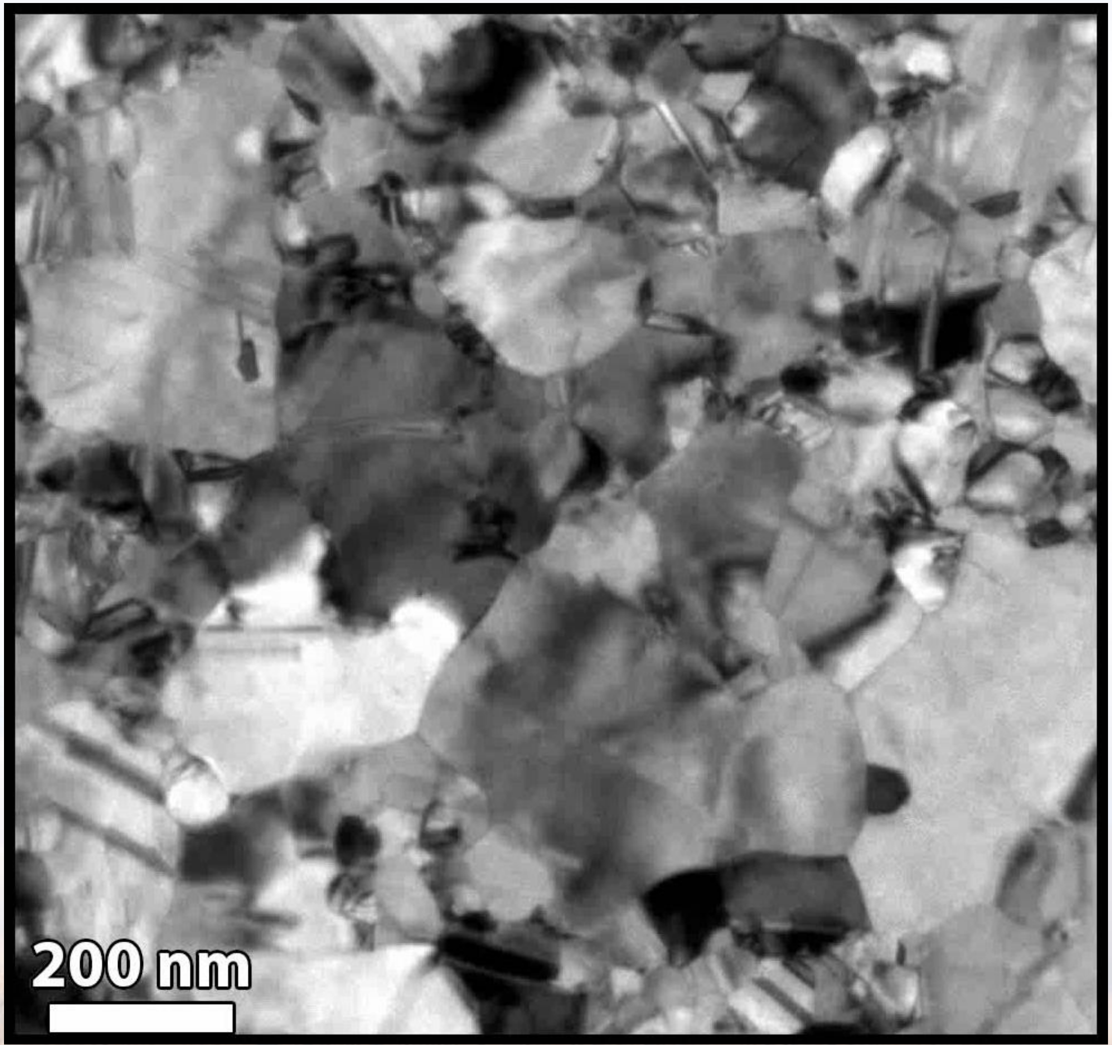
**Precession Mapping provides a wealth of crystal orientation mapping**

Chris Own, PhD Dissertation, Northwestern University, 2004

Slide courtesy : K. J. Ganesh

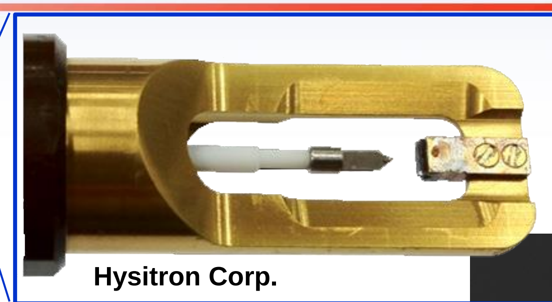
# Initial Exploration into the Role of Texture and Surface Roughness on Abnormal Grain Growth Mechanisms




**PED and AFM permit insight into texture, GB character, and surface roughness role on abnormal grain growth**

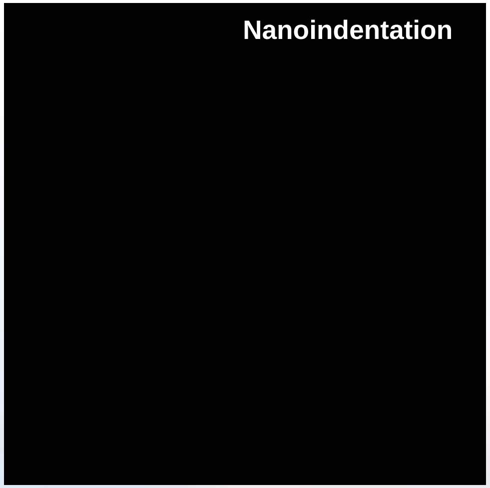
# *In situ* Quantitative Mechanical Testing

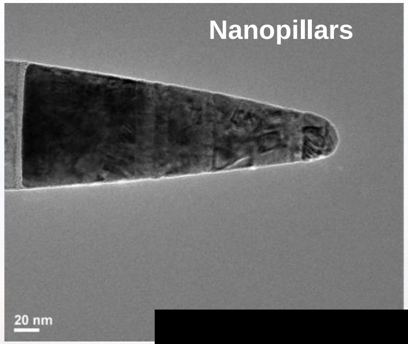


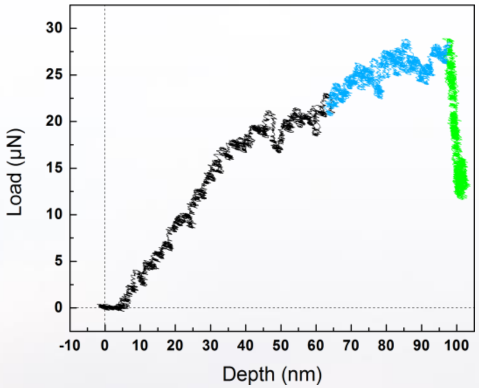

Hysitron PI95 *In Situ* Nanoindentation TEM Holder

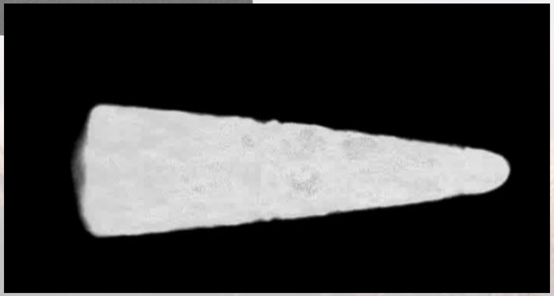
- Sub nanometer displacement resolution
- Quantitative force information with µN resolution

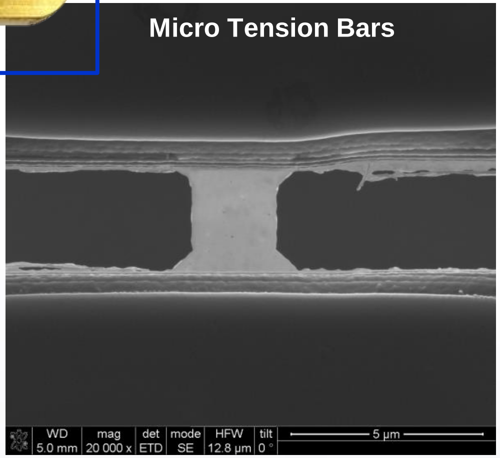
- **Concurrent real-time imaging by TEM**

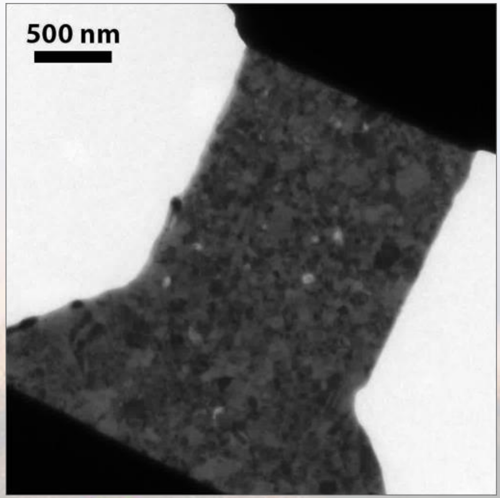




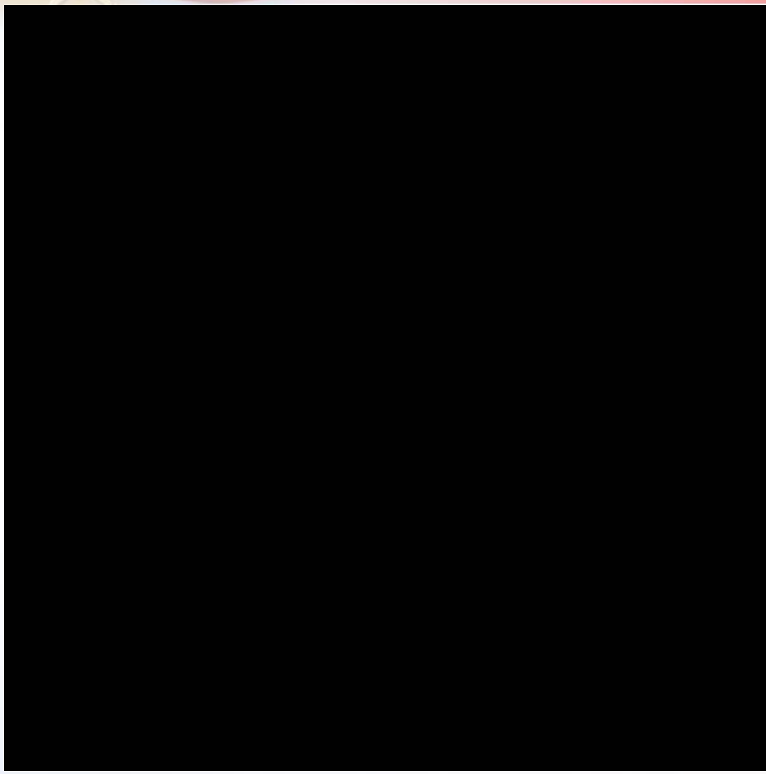



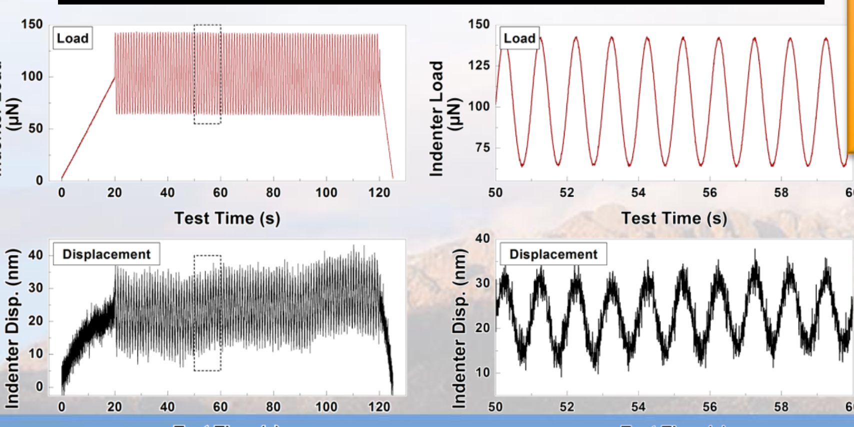

**A range of mechanical testing of nanoscale geometries are possible.**

# *In situ* TEM Quantitative Fatigue Testing

Contributors: D.C. Bufford, D. Stauffer, W. Mook

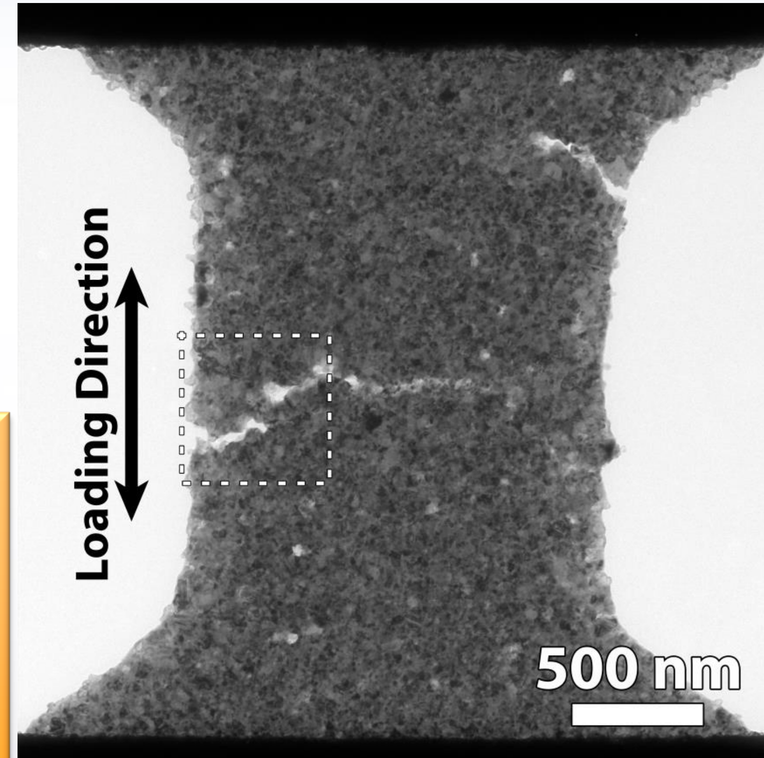
3× playback speed

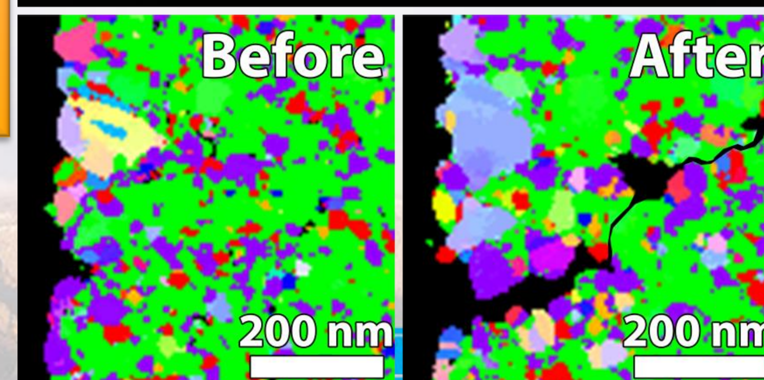





**High cycle fatigue in real time with nanometer resolution**

**Reveals abnormal grain growth ahead of a crack tip**

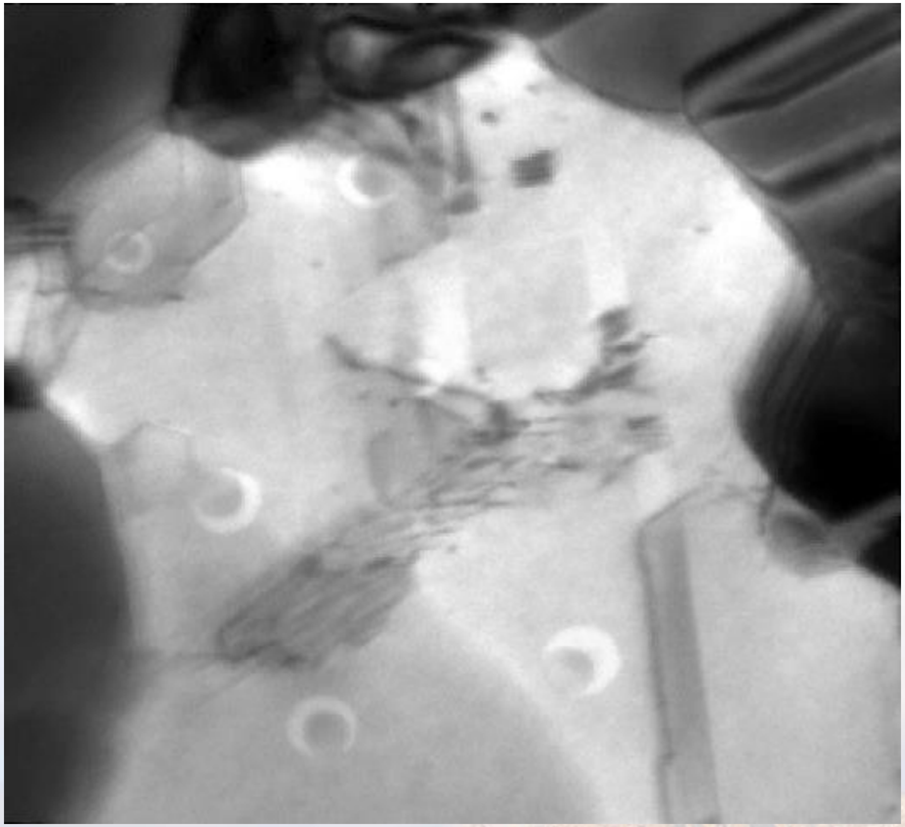


# Dose Rate Effects

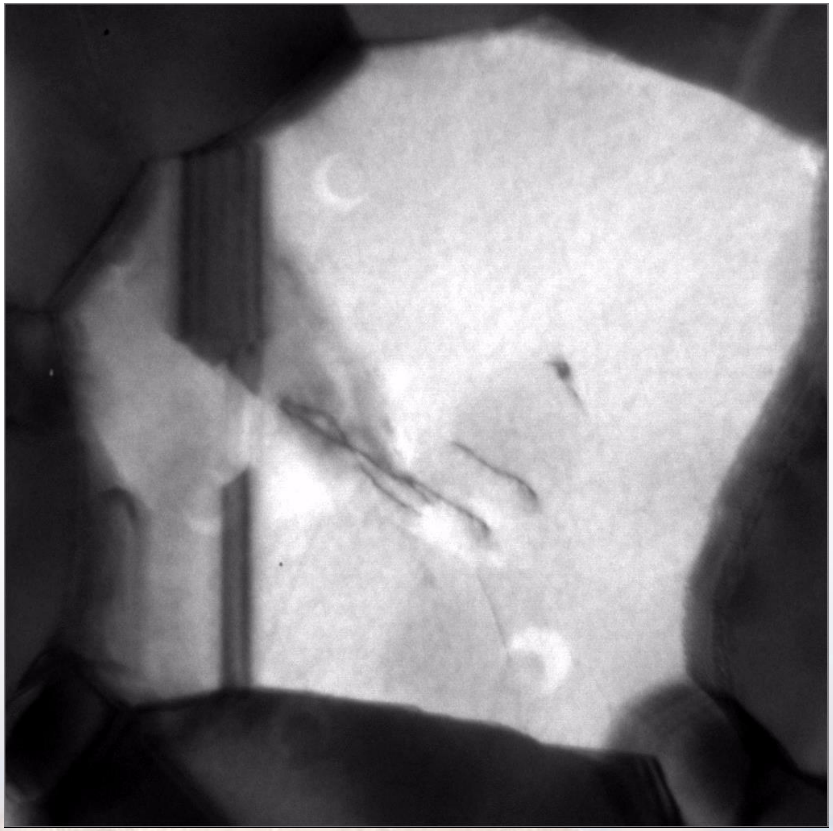
Collaborators: C. Chisholm , P. Hosemann, & A. Minor

**$7.9 \times 10^9$ ions/cm²/s**



**VS**

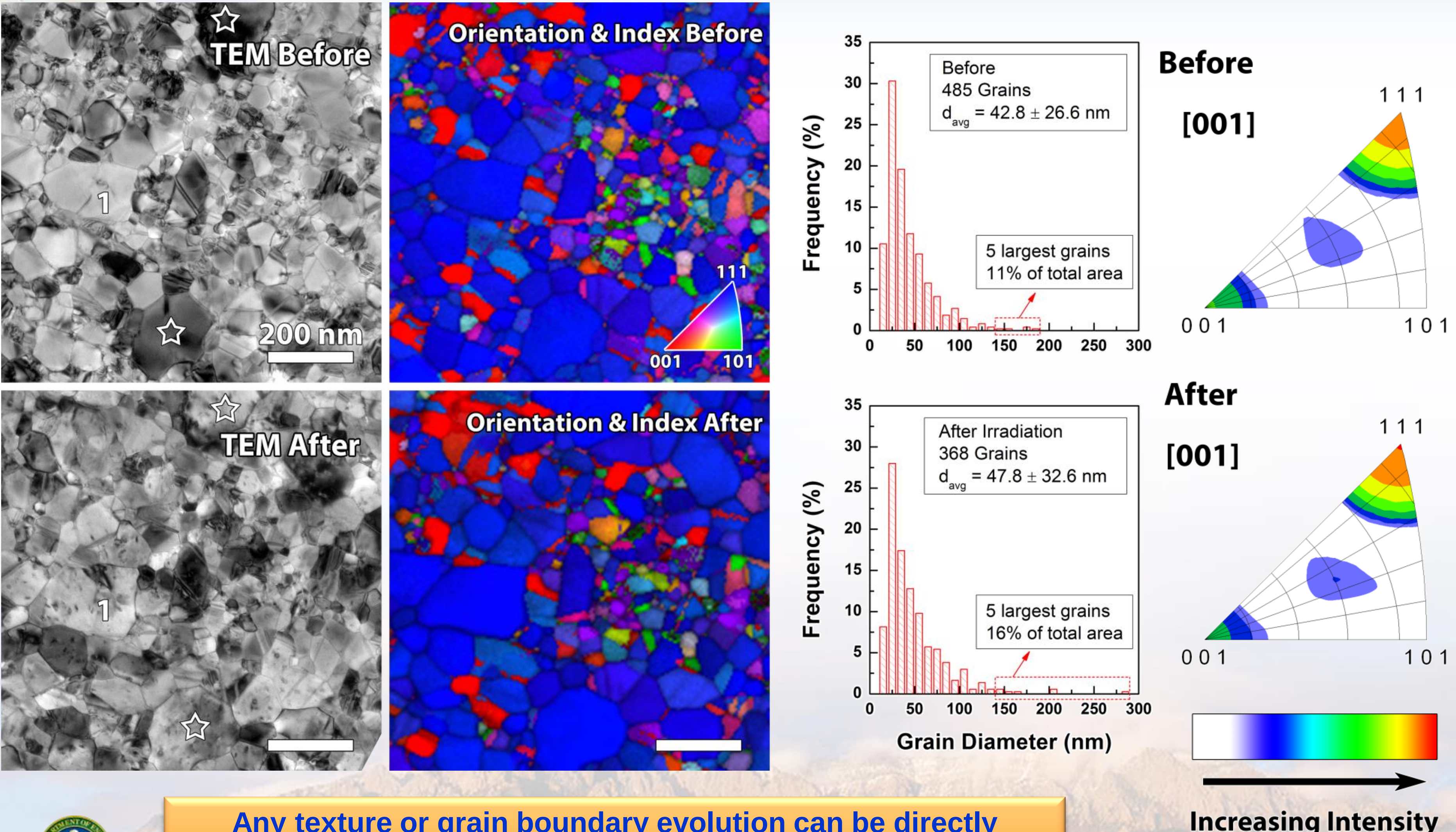
**$6.7 \times 10^7$ ions/cm²/s**



**Single Ion strikes and the resulting microstructural evolution can be directly observed with the I³TEM**

# Quantifying Stability of Nanocrystalline Au during 10 MeV Si Ion Irradiation

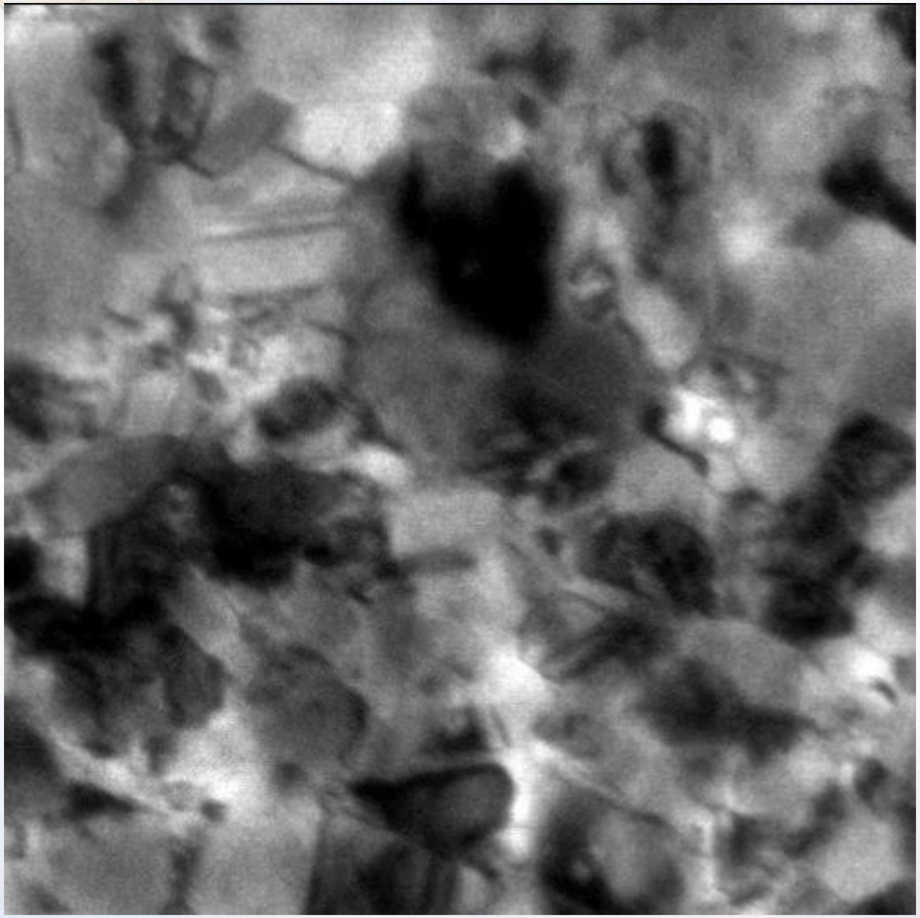
Collaborators: F. Abdeljawad, & S.M. Foiles

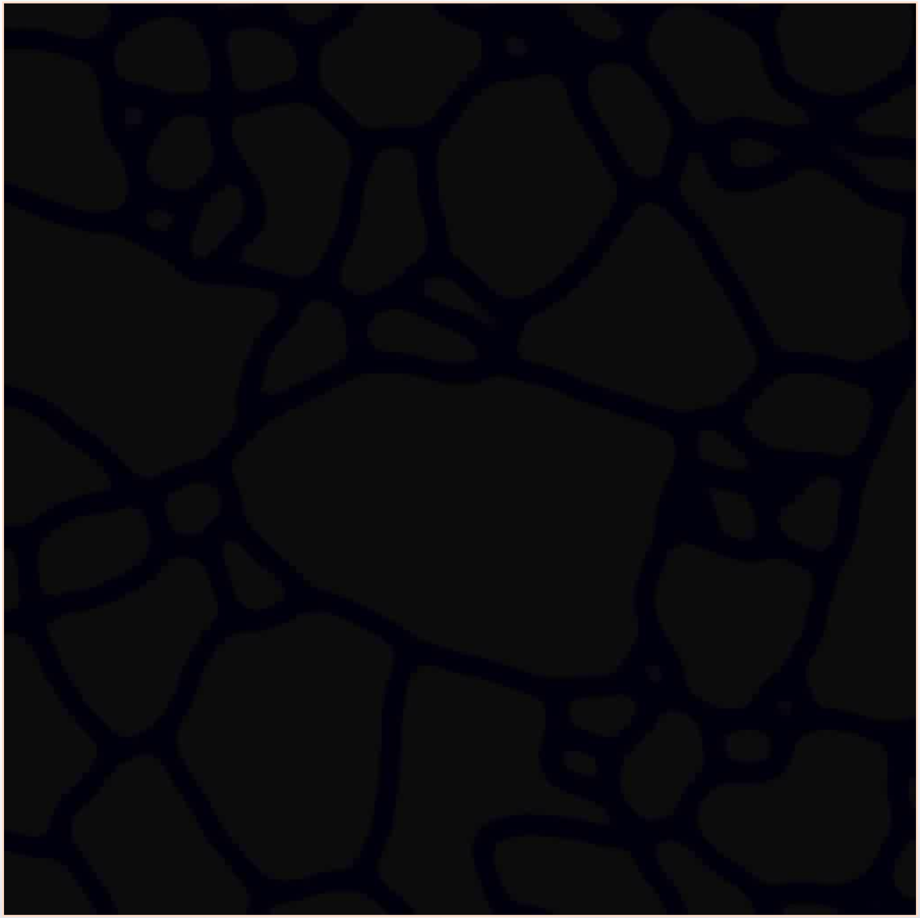



**Any texture or grain boundary evolution can be directly mapped, quantified, and associated with *in situ* dynamics**

# Comparing Experimental and Modeling Dynamics

Collaborators: F. Abdeljawad, & S.M. Foiles





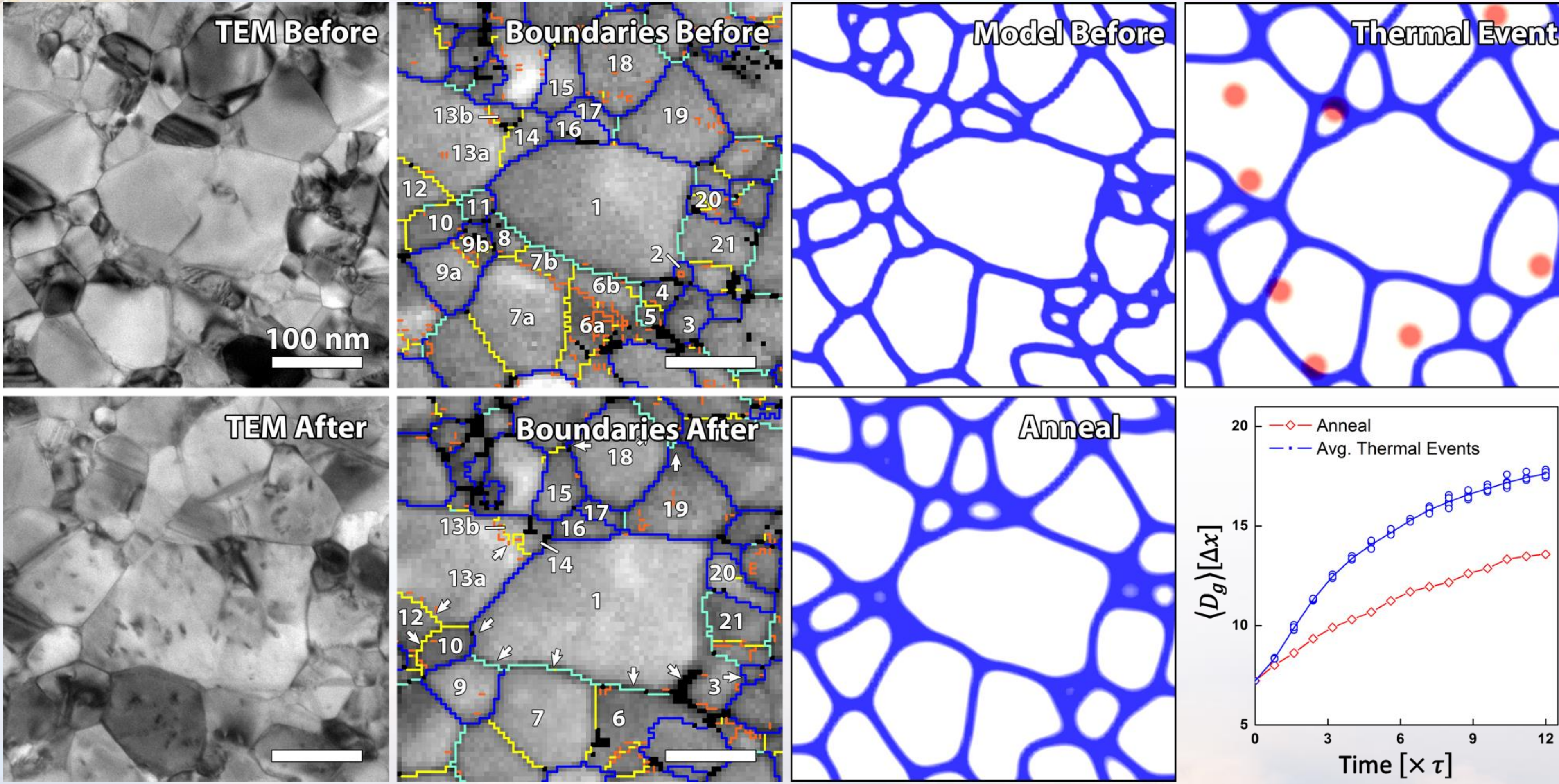
2× real time

- **Au foil during bombardment with 10 MeV $Si^{3+}$**
- **Approx. 22 s of 4000s total experiment time**

**Dynamics do not match, but show many similar effects**

# Direct Comparison to Mesoscale Modeling

Collaborators: F. Abdeljawad, & S.M. Foiles

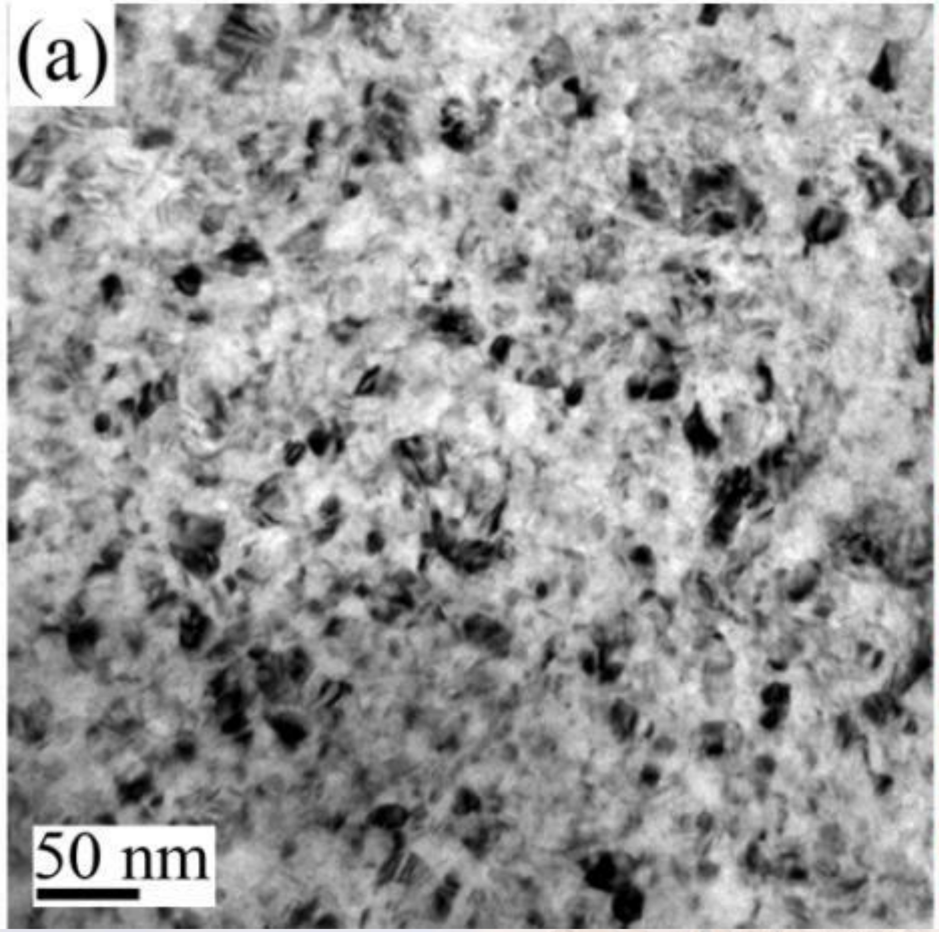



**Because of the matching length scale, the initial microstructure can serve as direct input to either MD of mesoscale models. Subsequent structural evolution can be directly compared.**

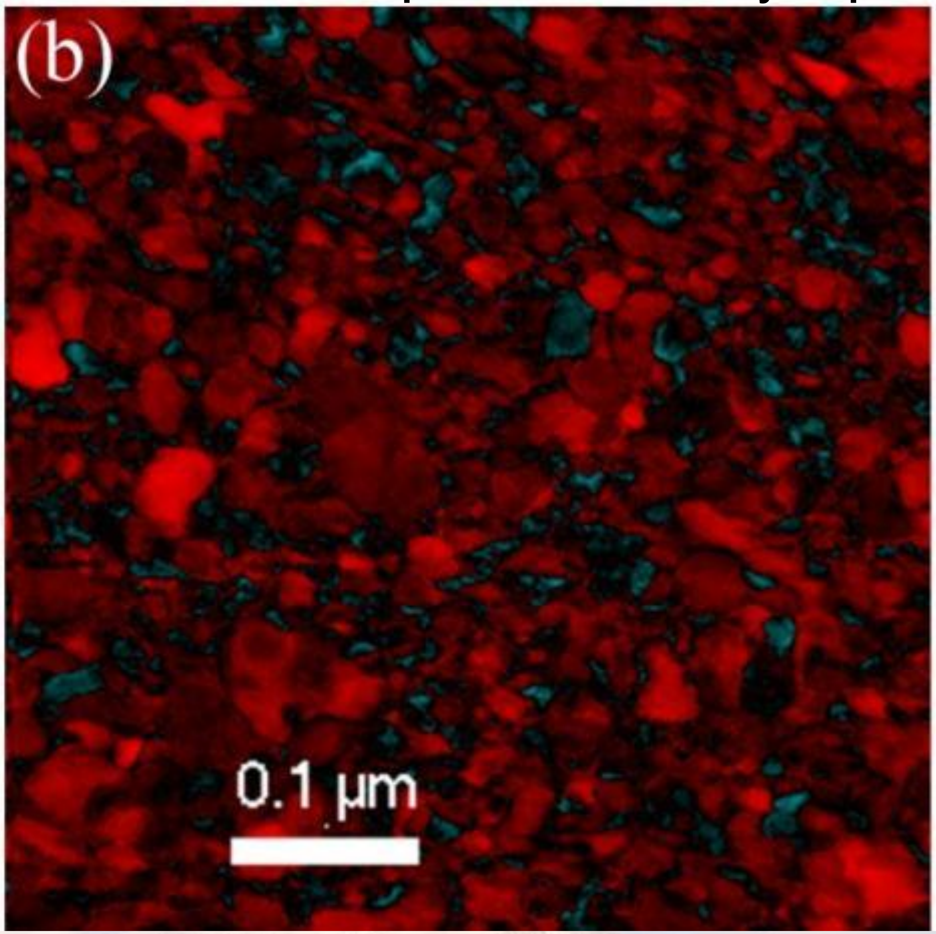
# Phase Determination in 50 nm As-deposited Ni Film

Collaborators: P. Ferreira & S. Rajasekhara

**BF – TEM**




**Re-constructed phase and reliability map**




1,124 HCP phase grains (in 1.5 $\mu m^2$)

Mean HCP grain size : 8.1 $\pm$ 0.3 nm

Mean HCP phase percentage: 6.0%

**Both FCC and HCP phase are present in as-deposited high purity PLD Ni films**

*FCC* phase

*HCP* phase

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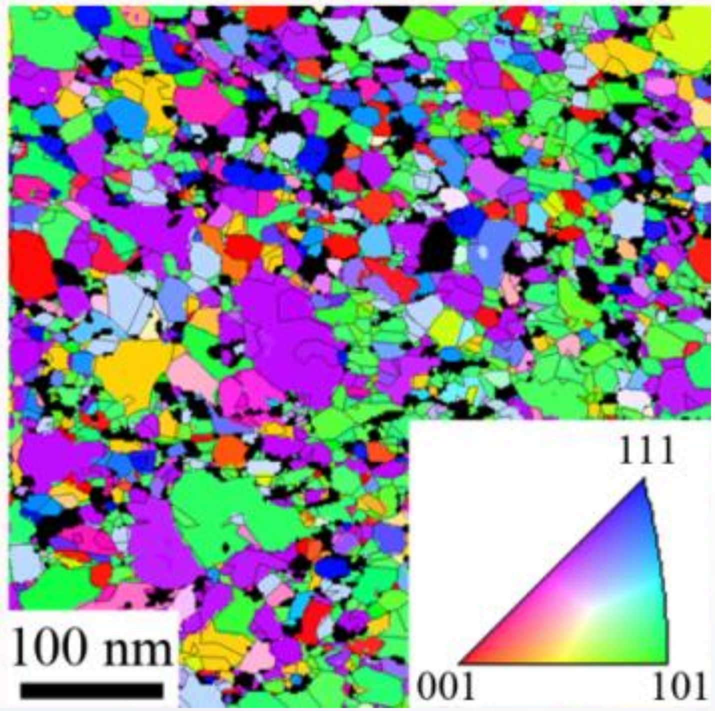
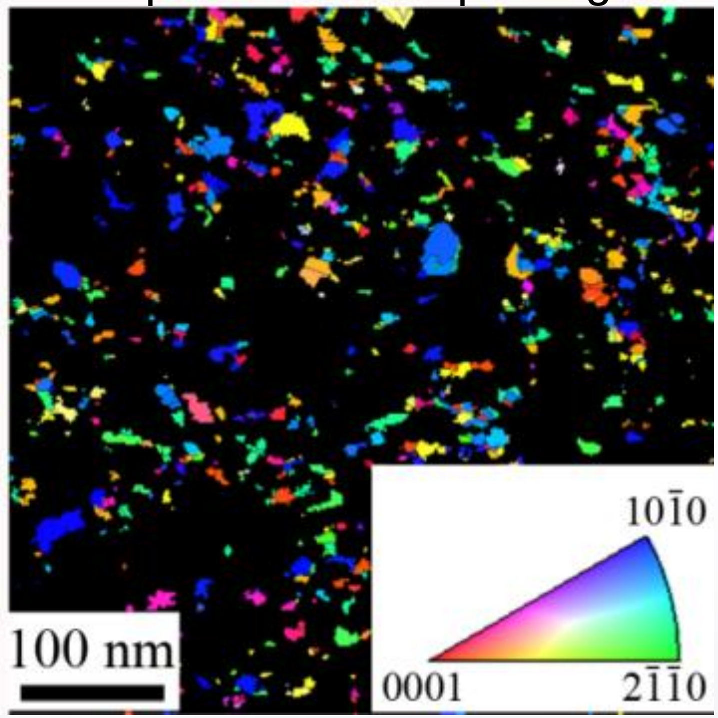
# *FCC* and *HCP* Texture Determination in 50 nm As-deposited Ni Film

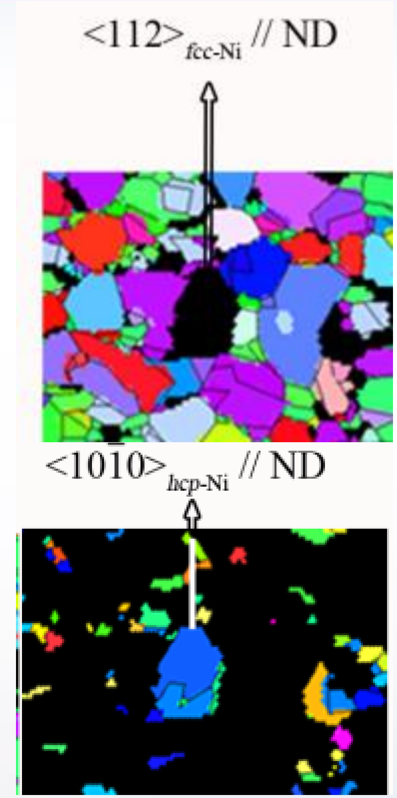
Collaborators: P. Ferreira & S. Rajasekhara

*FCC* phase inverse pole figure

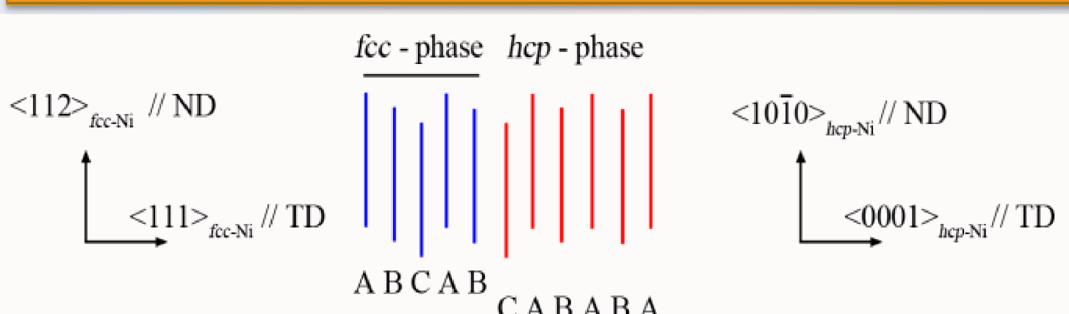



*HCP* phase inverse pole figure




**Texture maps at the nanoscale obtained from a TEM increase insight into abnormal grain growth**




**<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**

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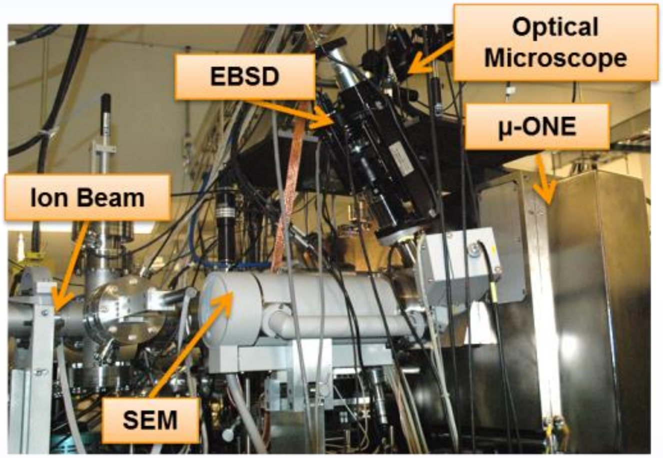
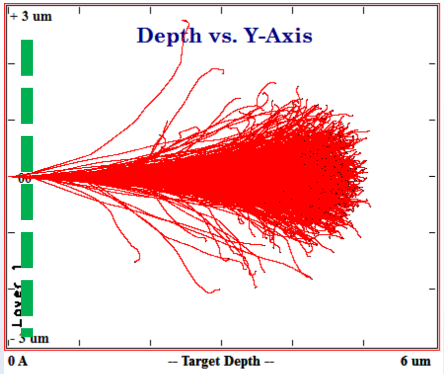
# *FCC* and *HCP* Phase Evolution after 35 MeV Ni Irradiation

Collaborators: P. Ferreira & S. Rajasekhara

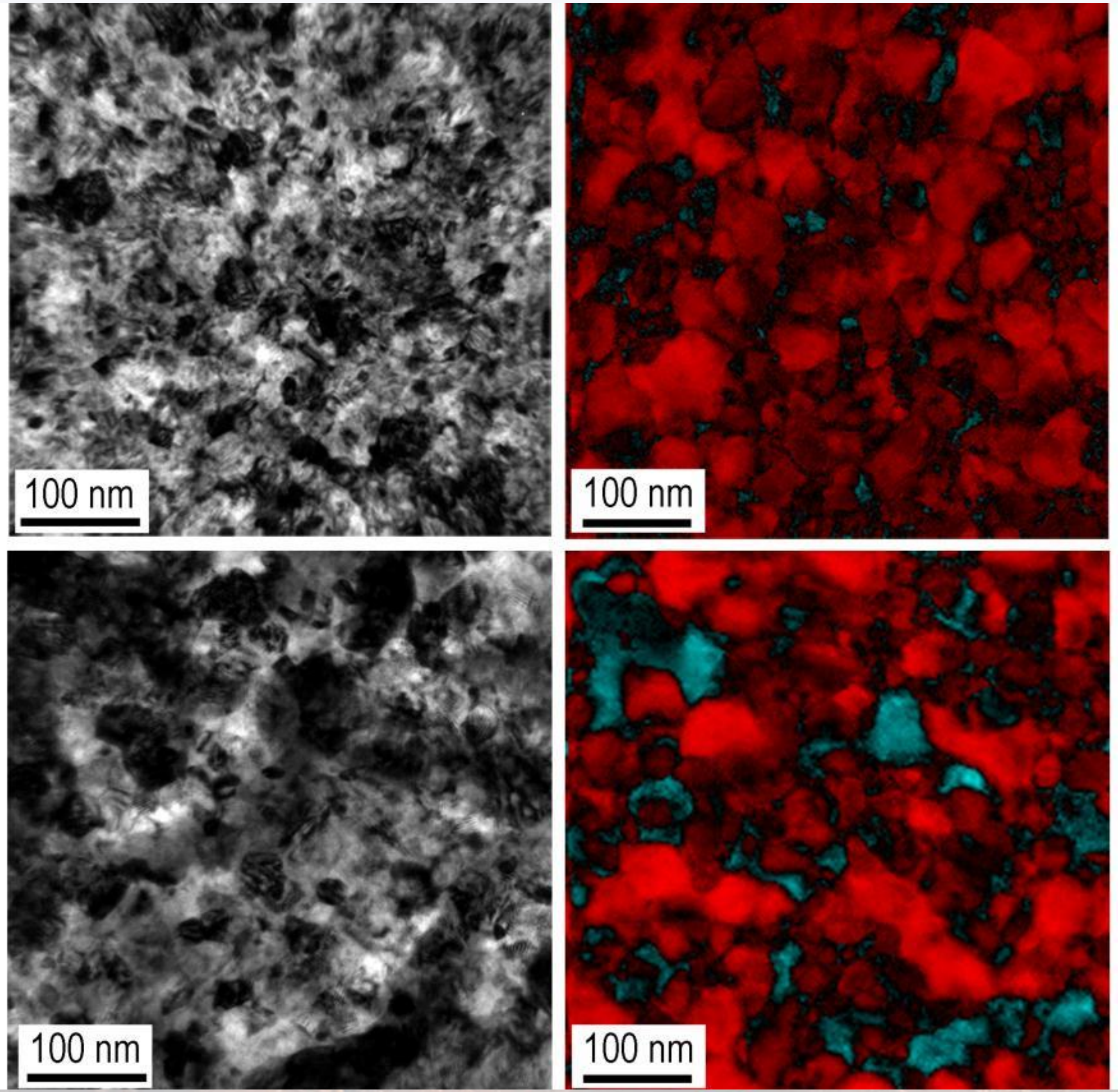



**Despite the minimal interaction predicted in 100 nm film, grain growth was observed and increased HCP phase resulted**

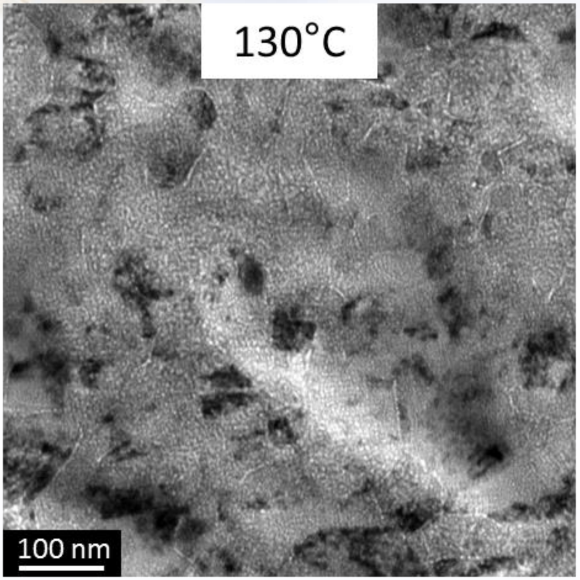
As-deposited

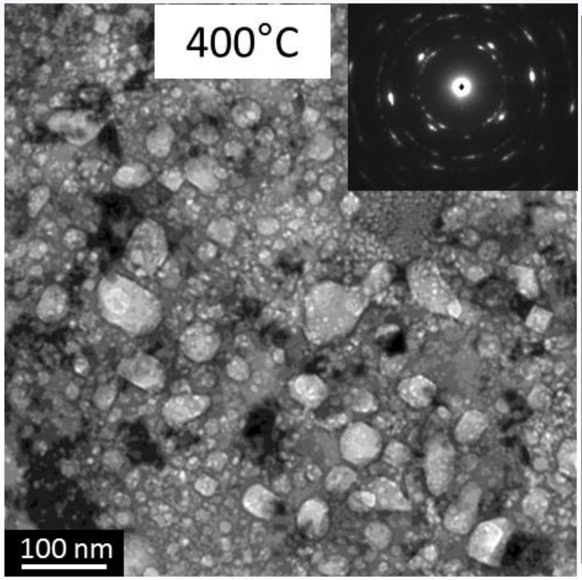
35 MeV Ni 3 x $10^{14}$ cm$^{-2}$

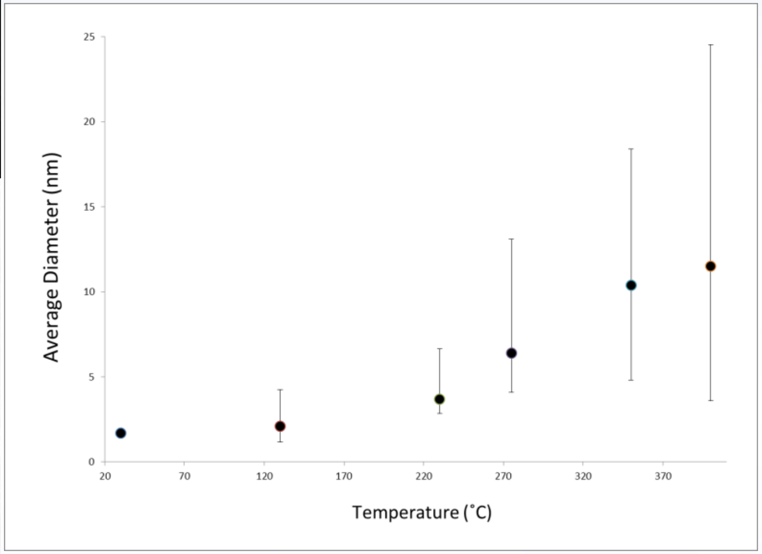



*FCC* phase *HCP* phase

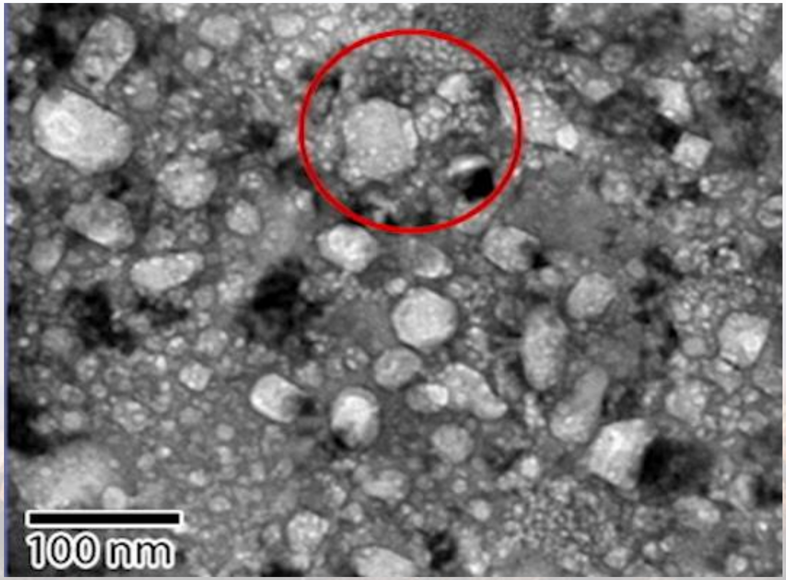
# Cavity Growth during In-situ Annealing of 10 keV $He^{+}$ Implanted and then 3 MeV Irradiated $Ni^{3+}$

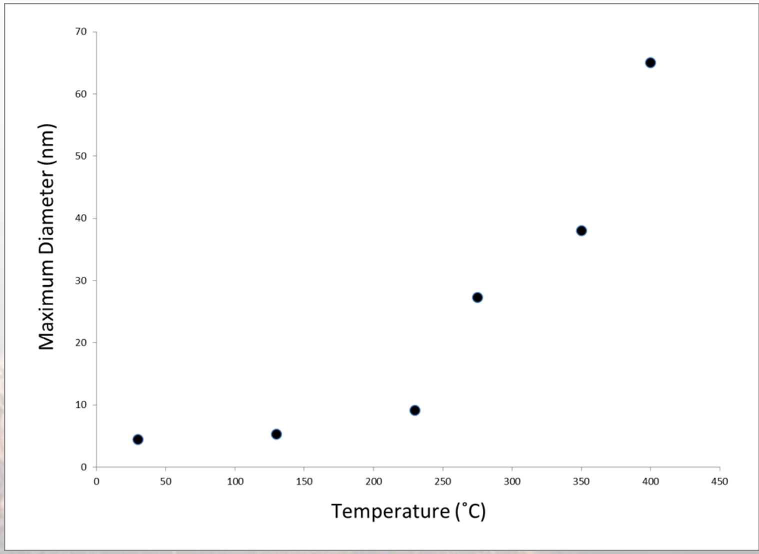



**Bubble to cavity transition and cavity evolution can be directly studied**



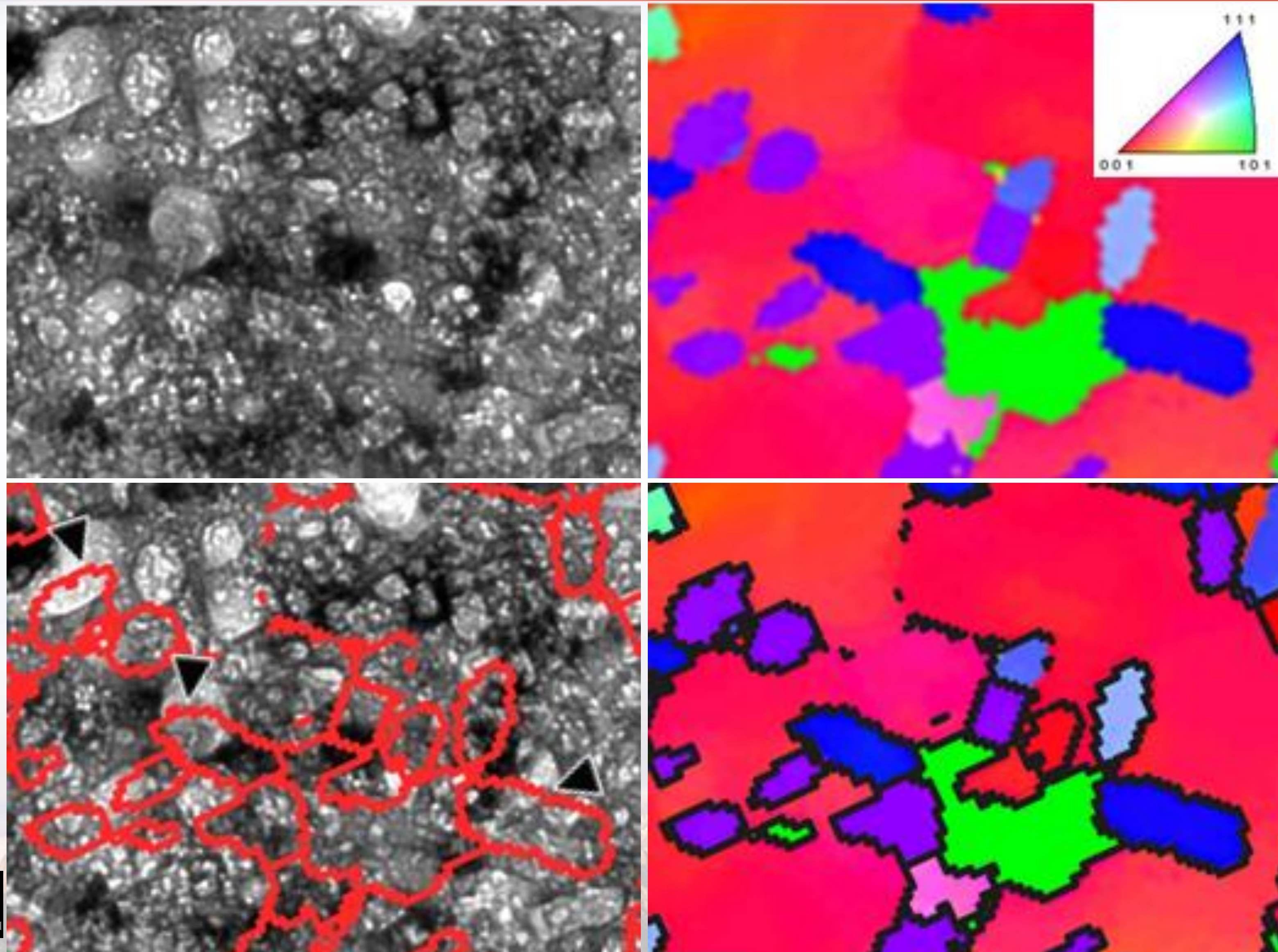



# Precession Electron Diffraction Reveals Hidden Grain Structure

Cavities in helium implanted, self-ion irradiated,

nc nickel film annealed to 400 ˚C

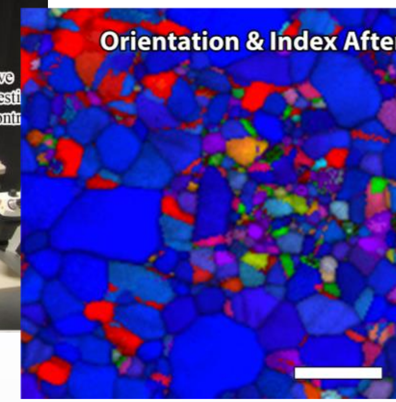
**Cavities span multiple grains at identified grain boundaries**




100 nm

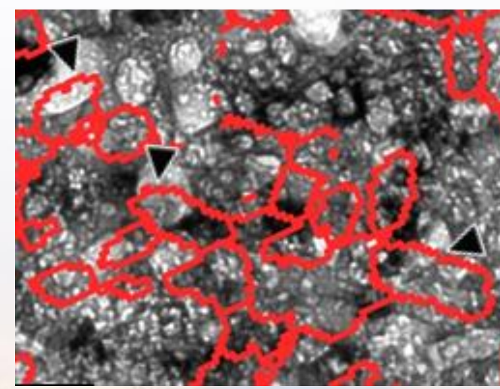
# Summary

- **Sandia's I³TEM capabilities:**
  - ***In situ*** **high energy ion irradiation from H to Au**
  - ***In situ*** **gas implantation**
  - **Heating up to 1,000 ˚C**
  - **Quantitative and bulk straining**
  - **Two-port microfluidic cell**
  - **Gas flow/heating stage**
  - **Electron tomography**
  - **Precession Electron Diffraction**


**Currently applying the current I3TEM capabilities to various material systems in sequential or combined harsh environmental conditions**

- **Structural response of metal sample to various harsh environments:**
  - **Sequence of gas implantation and cascade formation matters for the final microstructure**
  - **Concurrent gas implantation and irradiation permits the deconvolution of environmental parameters**
  - **PED permits the correlation of bubble evolution with grain texture and boundary type**
  - **Not limited to vacuum environments**



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