Task: Mechanics of Nanostructured Materials

SAND2007-4319P

David M. Follstaedt, James A. Knapp and Sean J. Hearne
Sandia National Laboratories, Albuquerque, NM 87185-1056

Goals: To develop fundamental understandings of the Mechanical Properties and Thermal Stabilities of Nanostructured Metals

We do this by detailed characterization of nanostructures including *in situ*, by using special synthesis methods, and with numerical analyses.

→ *We interact within Sandia and collaborate with major U.S. institutions.*

Key Results: *Both* Dislocation and Grain-boundary Deformation Processes operate in Nanocrystalline Ni (~10 nm).

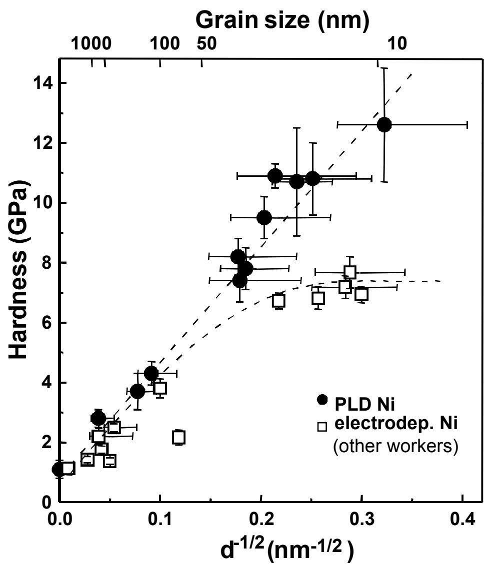
Abnormal Grain Growth in Nanocrystalline Ni produces grains that are *not* defect free, even containing *Vacancy-related Defects*.

He bubbles in Ni appear “shearable”, but in fact *Bind Dislocations Strongly*, leading to Orowan Hardening.

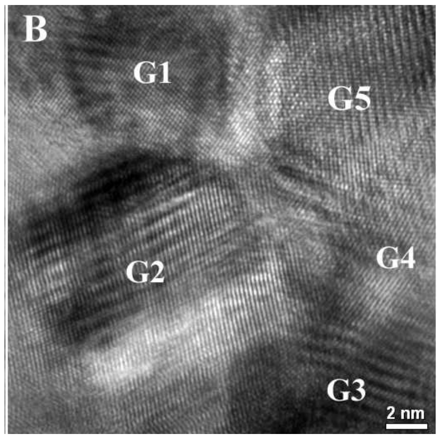
Nanocrystalline Ni exhibits Grain-boundary Deformation Processes

With S.X. Mao, J. Wiezorek (U. Pittsburgh), Z. Shan (now Hysitron), & E.A. Stach (NCEM, now Purdue)

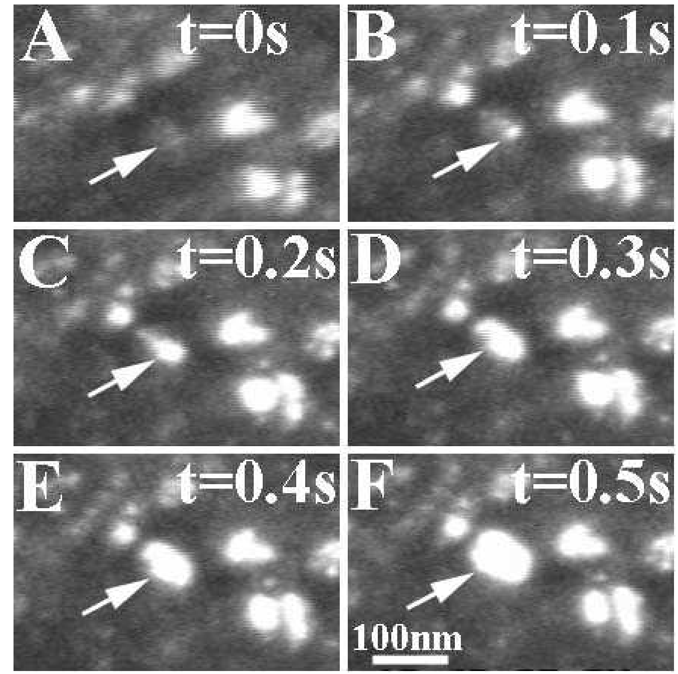
Earlier, we had shown:



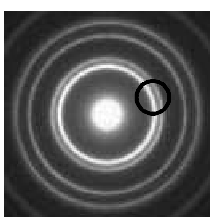
We and our collaborators have not found voids:



In-situ **TEM**: Thinning area just ahead of a propagating crack in 10 nm Ni



Our PLD Ni appears to be Ideal Nanocrystalline Ni.



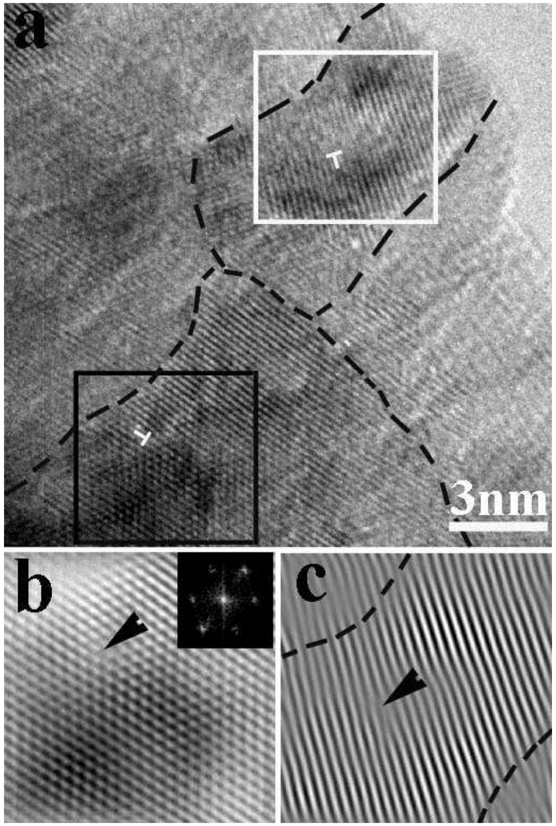
Dark-field TEM selects a specific crystal orientation; particle grows by nanograins rotating into alignment.

Shan et al, Science 305, 654 ('04)

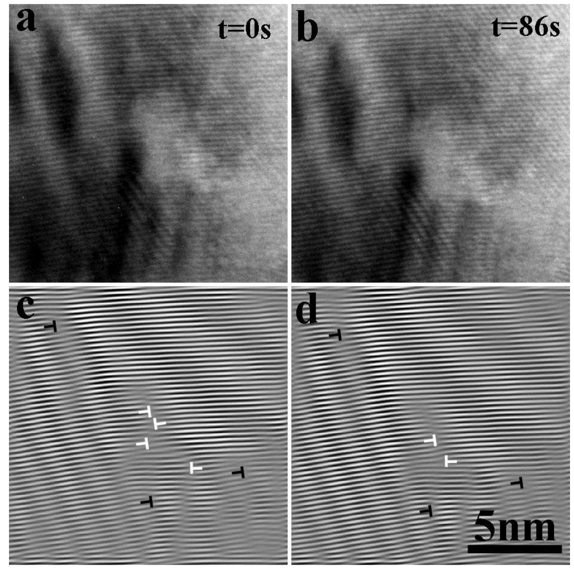
- **A very clear demonstration of Grain-boundary Deformation in nanocrystalline metal**

Dislocations observed within Tensile-strained Ni Nanocrystals

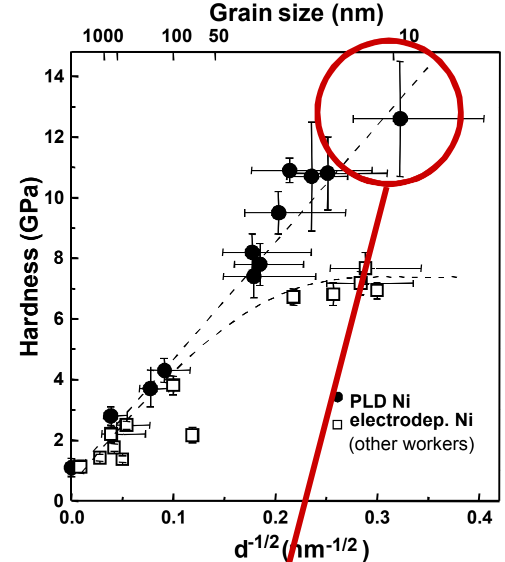
In-situ HREM, strain maintained:



Another area:



Extended observation shows dynamic dislocation processes.



10-nm Ni may give peak H-P strength. Dislocation and Grain-boundary processes are *both* prominent!

Whole Dislocations, not partials!

- HREM can be used to characterize dislocations during tensile straining.
- Dislocations were found within nanograins that were maintained under strain.
- Dislocation dynamics can be observed during long-term strain relaxation.

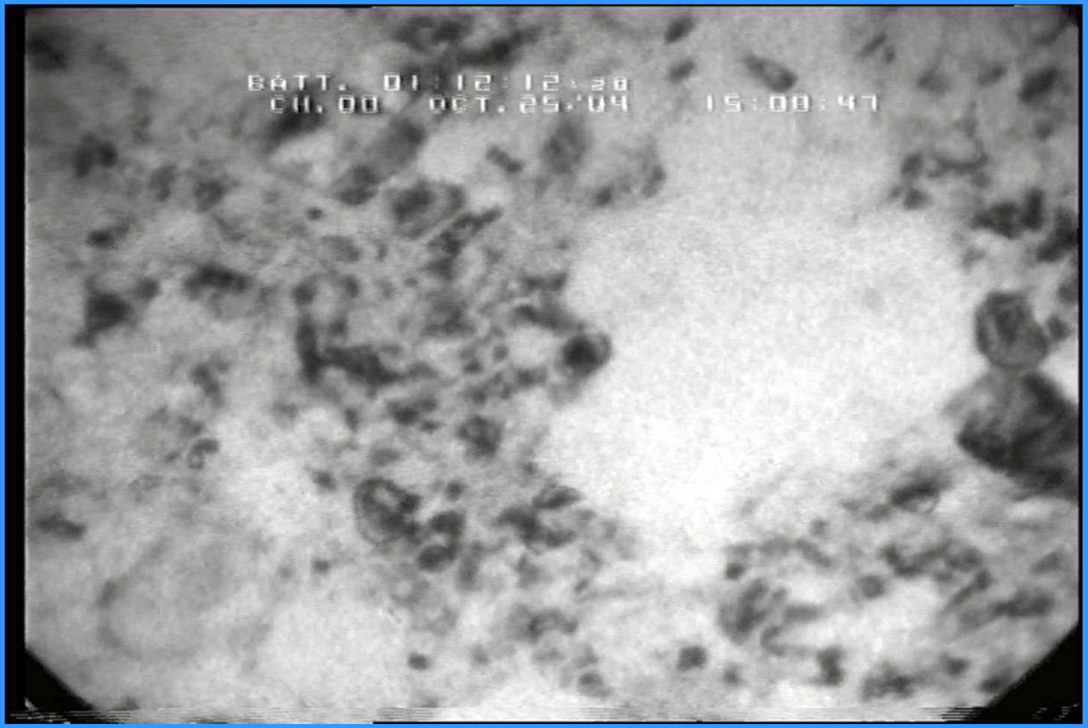
Shan et al, PRL 98, 095502 '07

Nanocrystalline Ni films undergo Abnormal Grain Growth

With I.M. Robertson, K. Hattar – Univ. Illinois Urbana-Champaign

150nm Ni/SiO_2, 300°C; in situ TEM at 350 kV

5x time compression



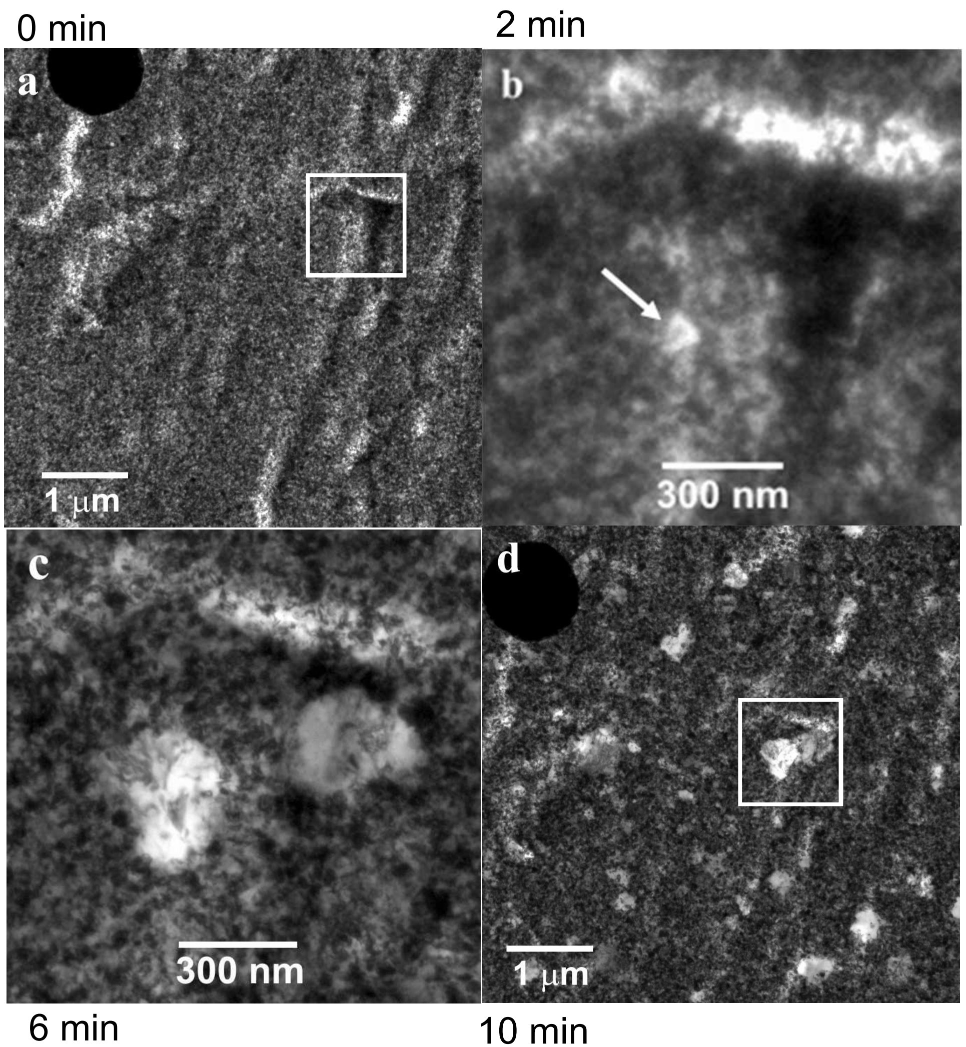
Ni1-3-500x.avi

250nm

Nano grains loose contrast as they are engulfed; rotation was not detected.
Growth appears erratic; direction unpredictable.
Sometimes a nanograin appears surrounded, then is engulfed.
Note that even the larger nano-grain reacts, but slowly.

Nanocrystalline Ni films undergo Abnormal Grain Growth

150 nm thick, annealed 275ºC; 200 kV "still" TEM images:



Reaction can also be followed with individual images (better resolution).

Such sequences provide essential properties of abnormal growth:

- Abnormal grains exhibit weak diffraction contrast; a specific orientation is not readily apparent.

- Thicker films react faster.

- Rate of Transformation

- Density of Sites

This is key information to be compared with theoretical modeling being done in the Task on Theory of Microstructures (E.A. Holm)

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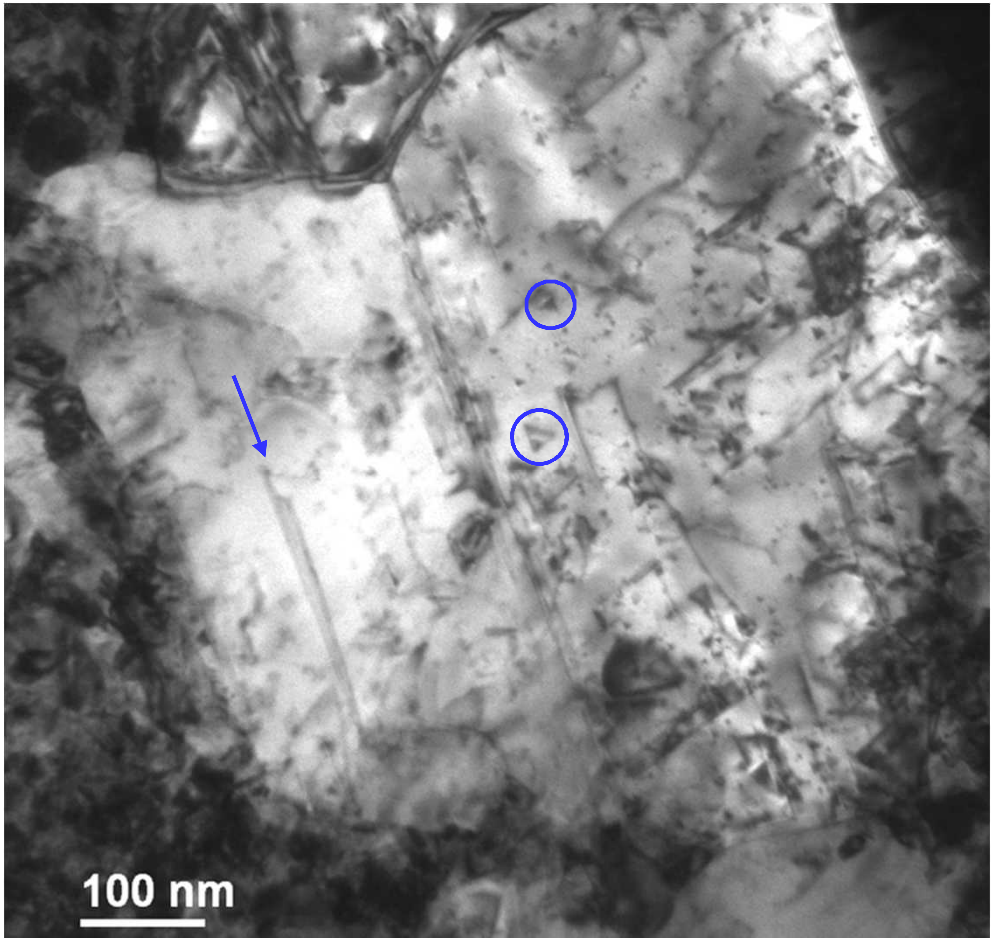
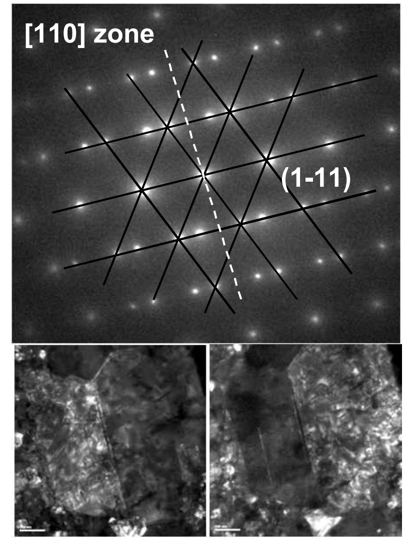
Key Test of understanding Grain Growth in Nanocrystalline Ni: Lattice Defects

150-nm film, annealed at 275°C (200 kV TEM):
Stacking Fault Tetrahedra, Twinned Inclusions, Stacking Faults, & Dislocations



Post mortem



Important Details:

SFTs are Vacancy Defects (fcc)

SFTs are seen in all our annealed Ni

SFTs seen at the anneal temperature - *Not Quenched-in !*

TEM at 200 kV - *Not Radiation Damage !*

Vacancies are incorporated in Abnormal Grains; we suggest from the Numerous Lower-density Grain Boundaries.

→ Critical Test for Models of Abnormal Grain Growth

Hattar et al, submitted to Acta Mat.

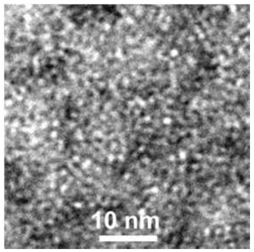
Follstaedt et al, Metal. Mat. Trans. 34A, 935 '03

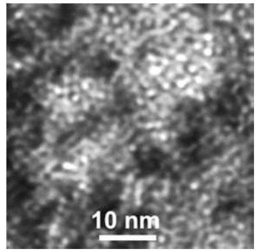
Can "Shearable" Nanoprecipitates strengthen like Oxides ?: He-implanted Ni

Myers/Follstaedt determined strong binding of dislocations to cavities – JAP 86, 3048 '99

Room Temperature Implantation

1 at.% He

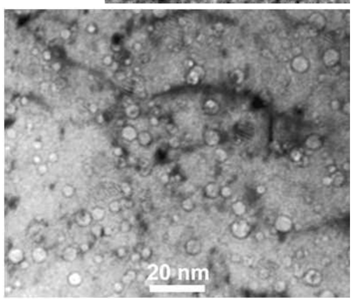


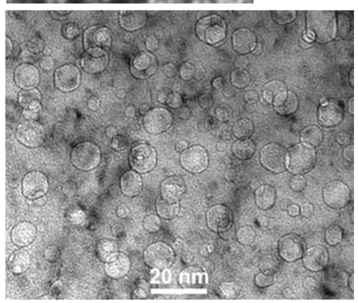


1.1 nm bubbles (both)

5 at.% He

500°C

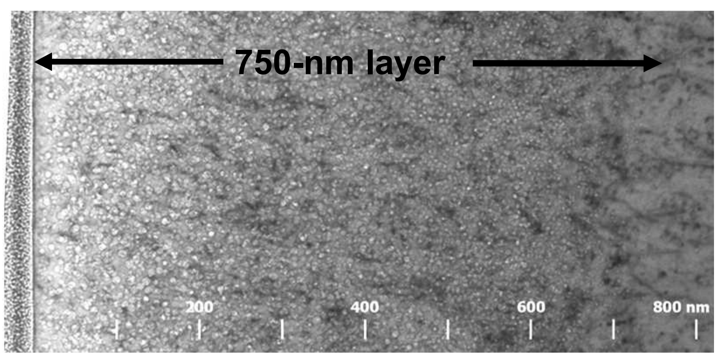




3.1 ± 0.9 nm

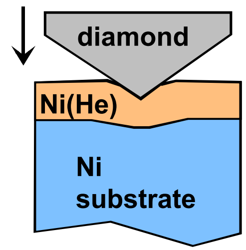
6.4 ± 2.0 nm

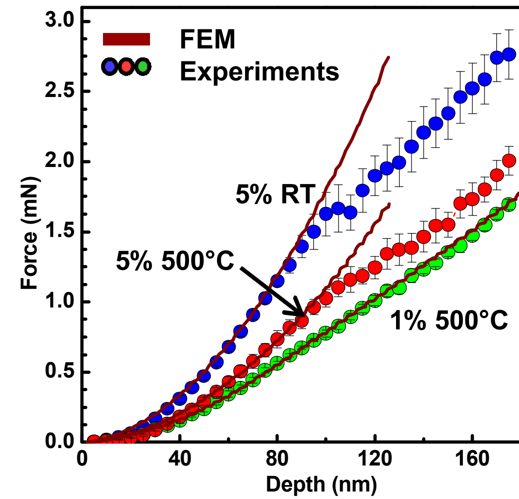
5 at.% He at 500°C



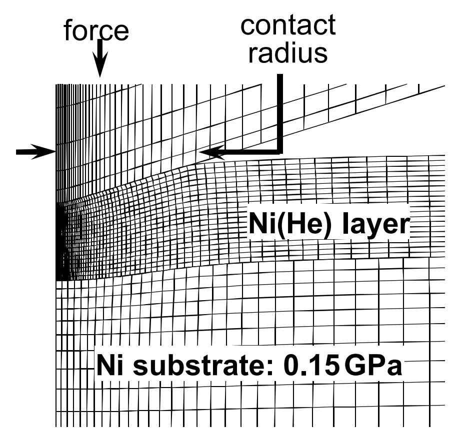
Evaluate Layer Strengths *(as developed here!)*:

Nanoindentation





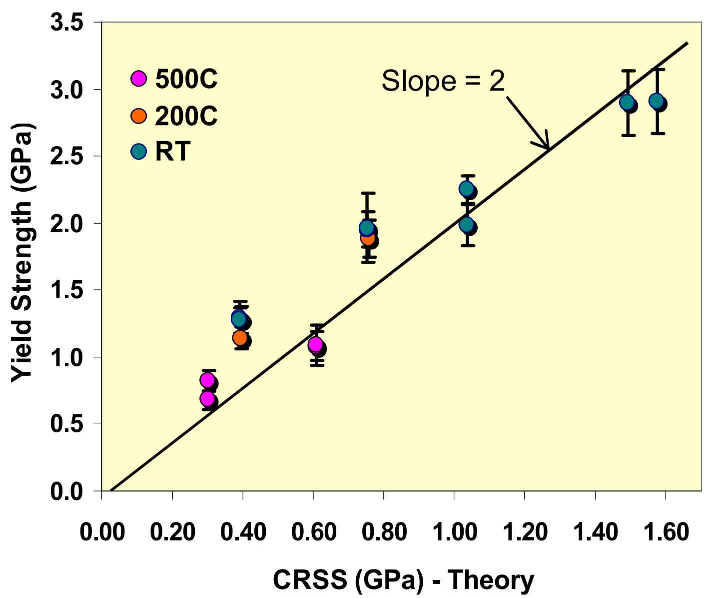
Finite-element Modeling (ABAQUS)



Yield Strengths (GPa)	
1 at.% RT	1.29
5 at.% RT	2.25
1 at.% 500°C	0.82
5 at.% 500°C	1.09
10 at.% RT	2.91

- **He Bubbles greatly strengthen Ni**

Strengthening by He Bubbles is understood Quantitatively and Mechanistically



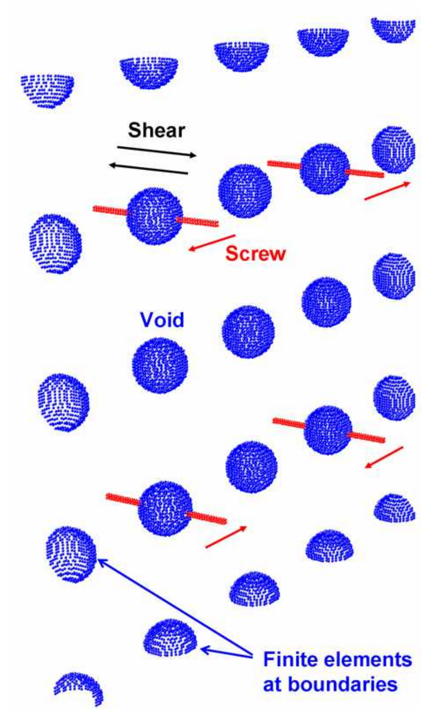
$$\tau = \frac{(0.9)\,\mu b}{2\pi(1-\nu)^{1/2}\,L}\;\frac{(\ln(2/b(1/L + 4/\pi d)))^{3/2}}{(\ln(L/b))^{3/2}}$$

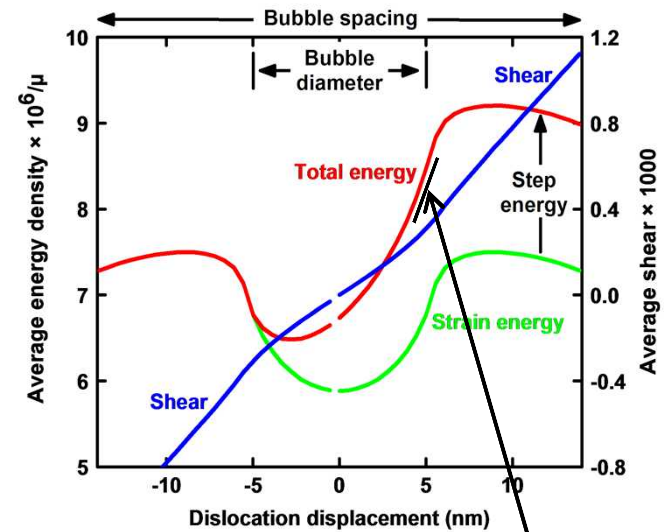
Orowan theoretical formalism includes:

Dislocation Bowing & Interactions

Statistical Positions of Bubbles

Finite-element calculations of Dislocation Energy
S.M. Myers -Sandia





Slope gives shear stress needed to move dislocation through the bubble:
1 nm cavities 2.8 nm apart: 2.4 GPa
10 nm cavities 28 nm apart: 0.58 GPa

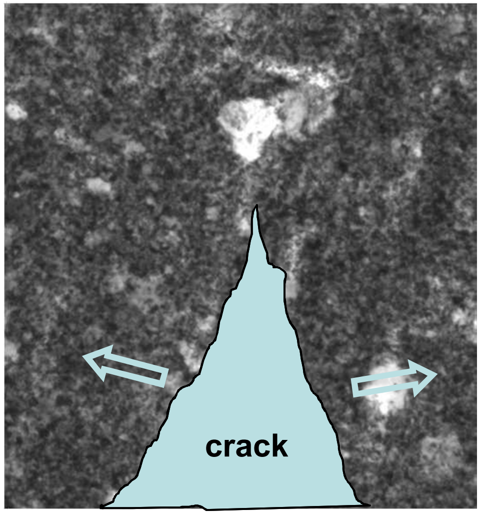
- **Strain Energy and Step Energy pin Dislocations to Cavities at observed Shear Stresses**

Manuscript in Preparation

- **Orowan hardening accounts for Yield Strength over Full Range of Bubble Nanostructures**

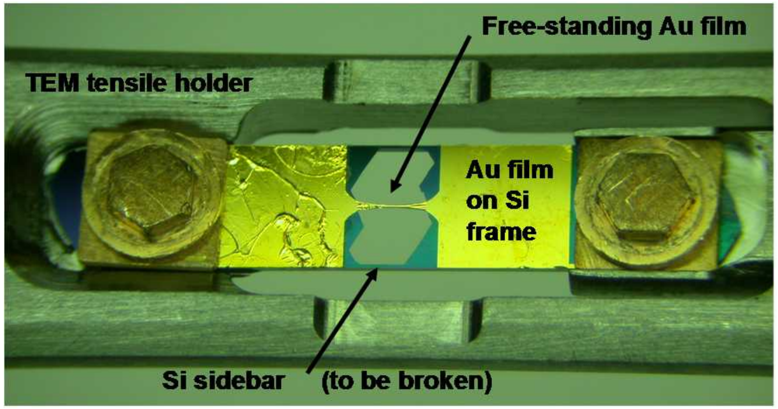
Mechanical Properties of Nanostructured Metals: Future Investigations

Use Abnormal Grain Growth to tailor *Bimodal* Nanocrystalline Ni films for *in-situ* tensile testing (with U. Illinois)



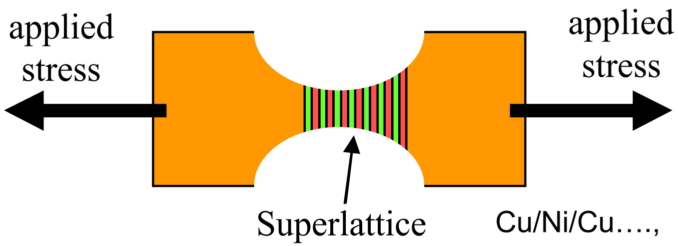
Literature results suggests "bimodal" nanocrystalline Ni may be more ductile, perhaps tougher. *How does a crack propagate when it encounters such a grain ?*

We have developed Si frames with free-standing Au and Ni films. They will allow us to:



Examine the *Tensile* Strength of Nanocrystalline Ni to compare with strength found by nanoindentation

Use *in situ* X-ray diffraction (synchrotron) to examine dislocations in "un-thinned" foils during straining (with H. Van Swygenhoven – PSI)



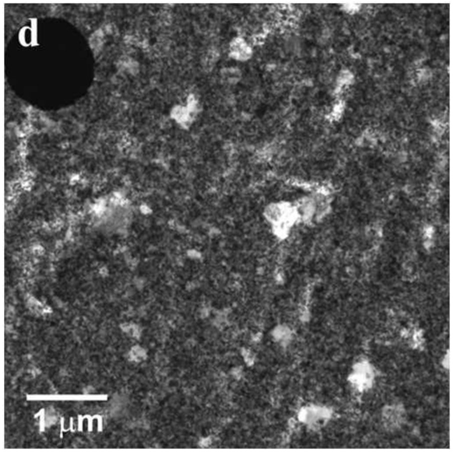
Cu/Ni/Cu…., Cu/Co/Cu….

We can now use electrodeposition to form "lateral superlattices". *For the first time*, superlattice properties can be determined *by deforming along the stacking axis*. The structure allows for *in-situ* SEM and TEM.

Abnormal Grain Growth in Nanocrystalline Metals: Future Investigations

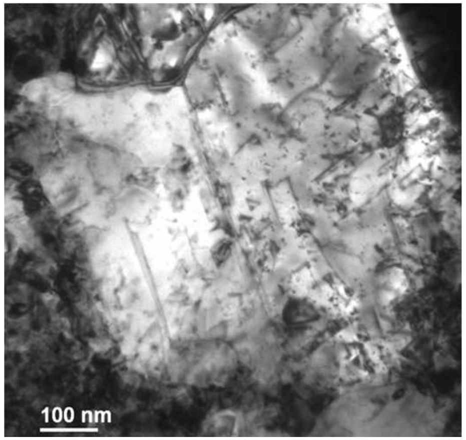
We will examine the Time-Temperature dependence of Abnormal Grain Growth in Ni

- Characterize transformed fraction
- Determine density of sites
- Compare with Theoretical Modeling (Task by E. Holm) to develop a thorough understanding of the driving force and kinetic mechanisms



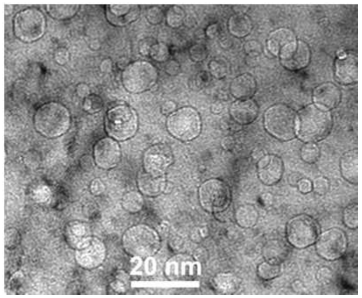
We will pay special attention to the Defects in Abnormal Grains

- Dislocation Loops coming out of Grain Boundaries
- Characterize the Stacking Fault Tetrahedra (vacancy content) (Ni, and metal with a lower SF energy – Cu, Ag ?)
- Measure the rate of grain growth into surrounding matrix
- Compare these features with Atomic Scale Modeling:
  Does it predict Vacancy Entrapment ?
  (such modeling is being done at Sandia by S. Foiles/E. Holm)



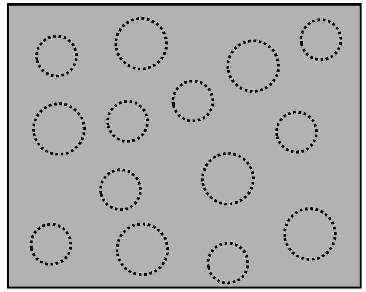
Strengthening by Nanoprecipitates: Future Investigations

Cavities/bubbles in Ni



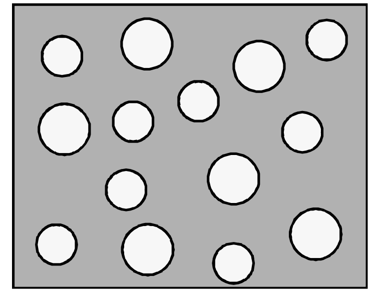
Dislocation Binding
$\propto (\mu_{Ni} - 0)$

"Ni Precipitates" in Ni (Pure Ni)



Dislocation Binding
$\propto (\mu_{Ni} - \mu_{Ni}) = 0$

Ag Precipitates in Ni



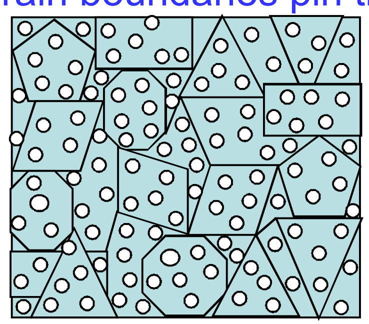
Expected Dislocation Binding
$\propto (\mu_{Ni} - \mu_{Ag}) = 77.8 - 25.6 = 52.2$ GPa

Soft metal precipitates are "shearable", but their binding can be predicted.

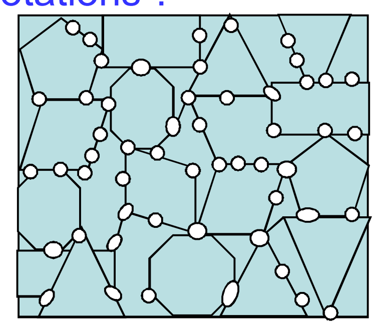
We will examine soft metal precipitates in Ni.

We will explore mechanical properties of nanocrystalline Ni with nano-scale oxide precipitates (NiO).

- Will both mechanisms combine to strengthen the material further?
- Will very high-strength grains promote grain-boundary processes ?
- Will oxides in grain boundaries pin them & prohibit rotations ?



nm



nm