SAND2010-1663P

Nanomechanics and Nanometallurgy of Boundaries
(formerly: Mechanics at Small Length Scales)

BES Triannual Review, March 2010

Brad L. Boyce
Multiscale Metallurgical S&T Department
Materials and Process Science Center
Sandia National Laboratories
Albuquerque, NM

Key Personnel

Current PI Team:

- Brad L. Boyce, Program Leader, experimental mechanical properties studies
- Blythe G. Clark, in-situ TEM studies
- Stephen M. Foiles, molecular dynamics studies
- Khalid M. Hattar, ion irradiation effects and in-situ studies
- Elizabeth A. Holm, mesoscale grain growth modeling studies
- James A. Knapp, deposition/processing of nanocrystalline metal thin films

Recent Team Members:

- John P. Sullivan, dynamic properties of MEMS materials (left to lead EFRC effort)
- David M. Follstaedt, in-situ TEM studies (retired)

Key Interactions

TEM studies

- Paulo Ferreira, Univ. Texas - Austin
- Scott Mao, Univ. Pittsburgh
- Ian Robertson, Univ. Illinois (UIUC)
- Eric Stach, Purdue Univ.

Mechanical behavior studies

- Daniel Gianola, Univ. Pennsylvania
- Apurva Mehta, Stanford Synchrotron (SSRL)
- Amit Misra, Los Alamos National Lab.

Grain growth and deformation modeling

- Phillip Duxbury, Michigan State Univ.
- Anthony Rollett, Carnegie Mellon Univ.
- Luke Brewer, Sandia National Labs.

Key Science Questions

- *CORE MISSION: Understand structure-property relationships in nanocrystalline metals to control behavior (strength, toughness, ductility, etc.)*

- *What deformation mechanisms operate in nanocrystalline metals to provide unique mechanical properties? What are the critical length scales for mechanism transitions?*

- *How are the unusual properties of nanocrystalline metals connected to grain and structural instabilities?*

- *What is the underpinning mechanism of abnormal grain growth?*

- *What is the process by which mechanical grain growth occurs? What is the coupling (if any) between thermal and mechanical grain growth? What are the connections between monotonic and cyclic mechanical grain growth?*

- *Can we stabilize nanocrystalline grain boundaries against normal and abnormal grain growth?*

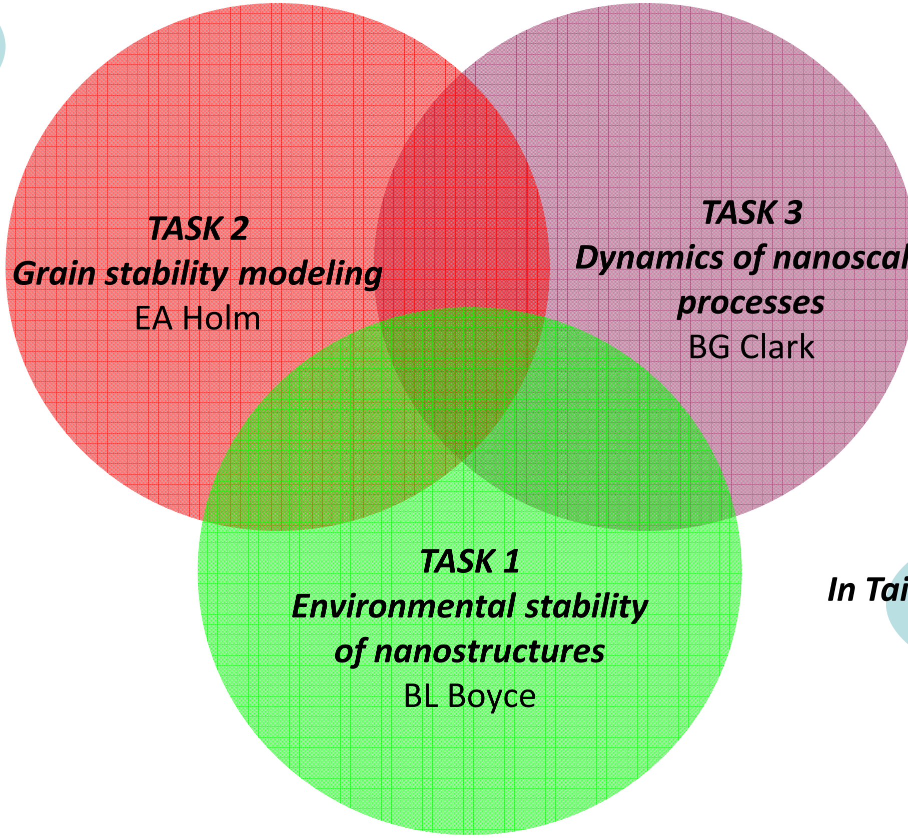
Nanomechanics and Nanometallurgy of Boundaries

Grain Boundary Science
Foiles / Holm/Brewer

LDRD ended FY09

In-Situ Stage Development
Clark / Hattar

BES proposal
Under development



Polycrystalline Dislocation Dynamics
Boyce / Robbins

Ongoing LDRD

Fatigue and Wear In Tailored Nanocrystalline Alloys
Boyce / Prasad / Hall

BES proposal
Submitted October 2008

Ion Irradiation
Hattar

Ongoing LDRD

MEMS platforms
Boyce

CINT interaction

How does Sandia fit into Nanomechanics Discovery?

• Sandia is emerging as a in-situ TEM powerhouse with 3 in-situ specialists (Blythe Clark, Khalid Hattar, Jianyu Huang).

• Extensive materials modeling capabilities with powerful supercomputers & class-leading codes (LLAMPS, SPPARKS)

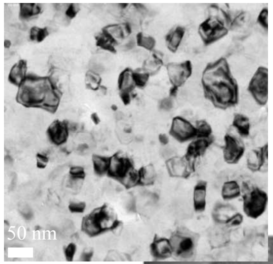
• CINT provides key tools for nanomechanics discovery in an 'open' user facility.

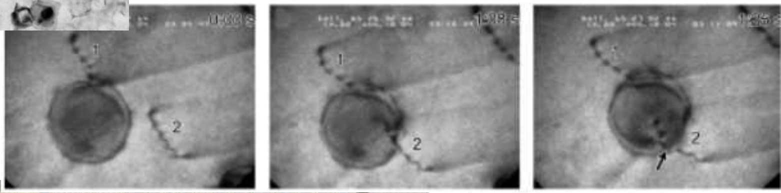
• Sandia's MEMS capabilities provides microfabrication facilities and discovery 'platforms'.

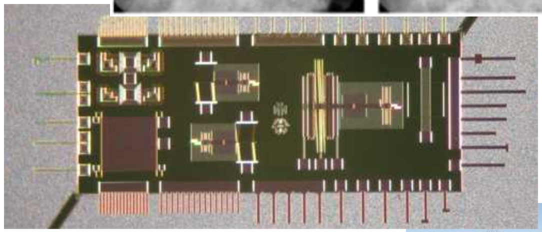
• Sandia provides extensive ion irradiation facilities, including the new Ion Beam Laboratory.

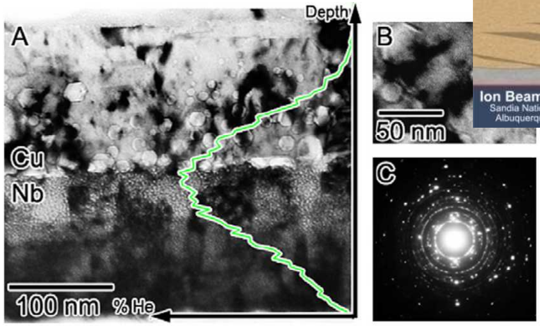
• Pulsed laser deposition (Jim Knapp) provides a source of pure nanocrystalline metal thin films.

• Collaborations are in place or under development with other key research institutions (e.g. LANL / Amit Misra / nanolaminates).

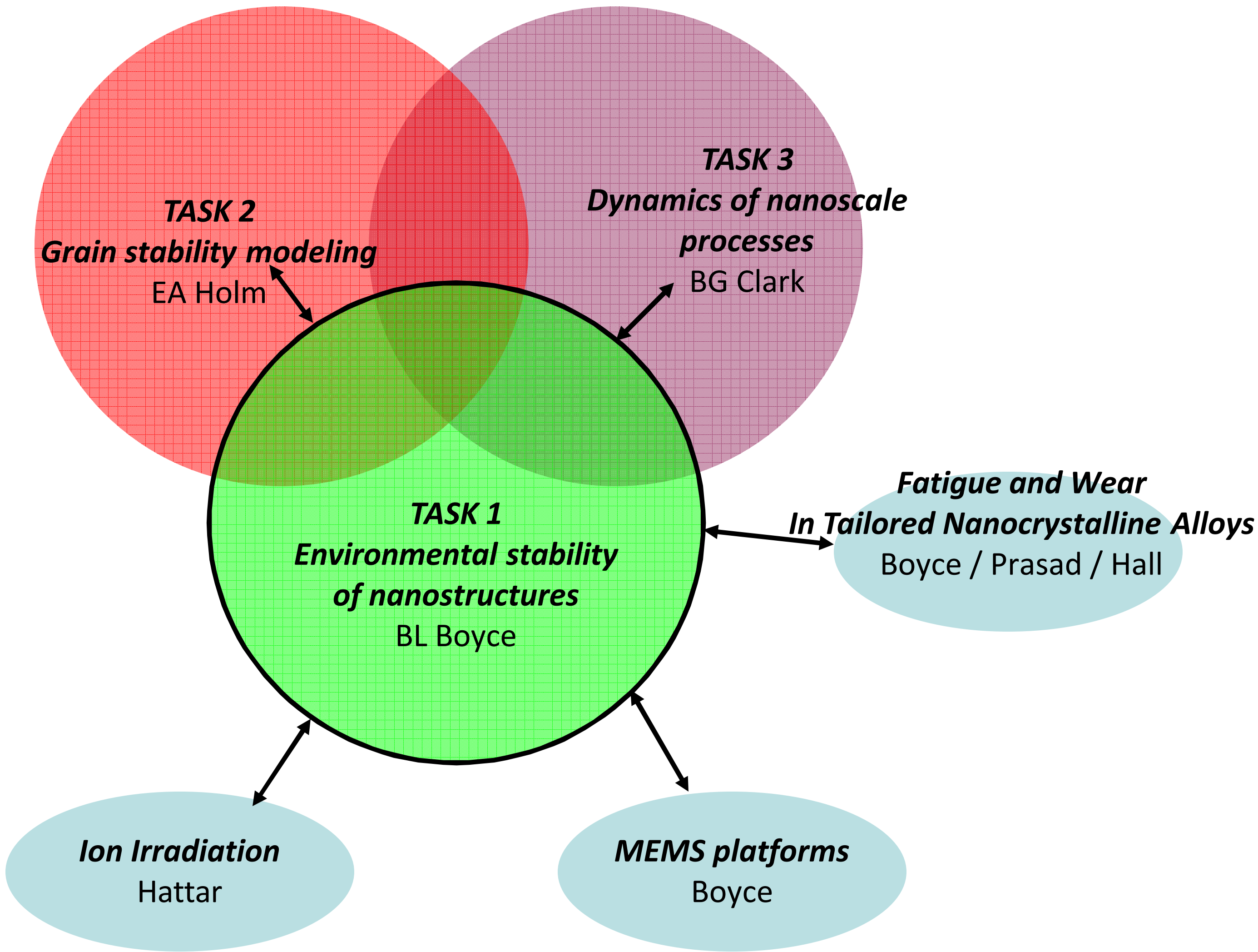




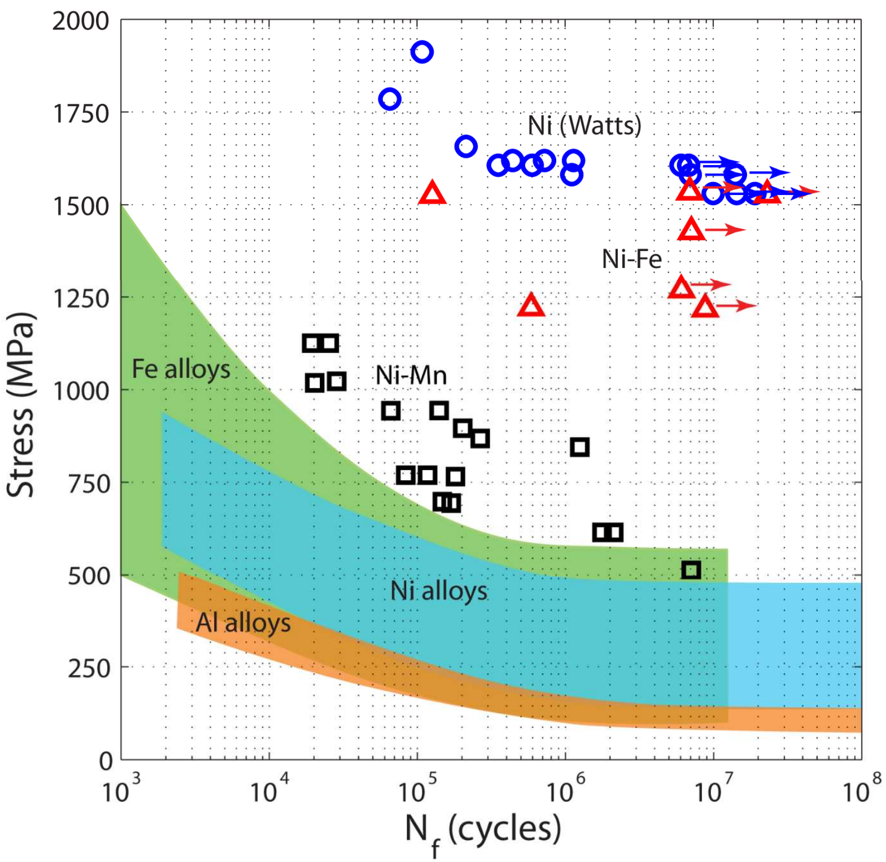




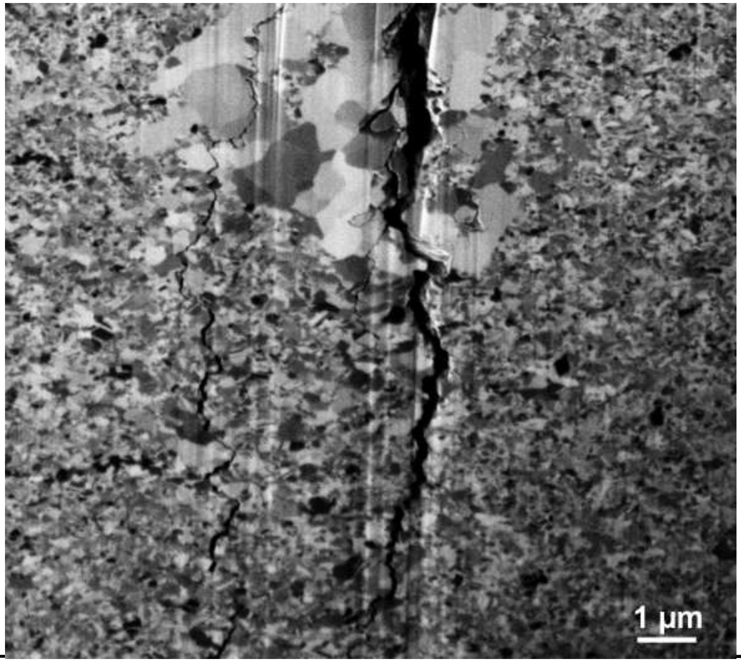
Nanomechanics and Nanometallurgy of Boundaries



Fatigue of Nanocrystalline Alloys



- Nanocrystalline alloys exhibit anomalous fatigue resistance because they exceed yield-strength scaling.
- In our recent observations on 3 NC Ni-alloys, *crack initiation is always preceded by fatigue-induced localized grain growth!*
- Microstructural stabilization is key to shutting down traditional PSB-driven crack initiation.



See poster by B.L. Boyce

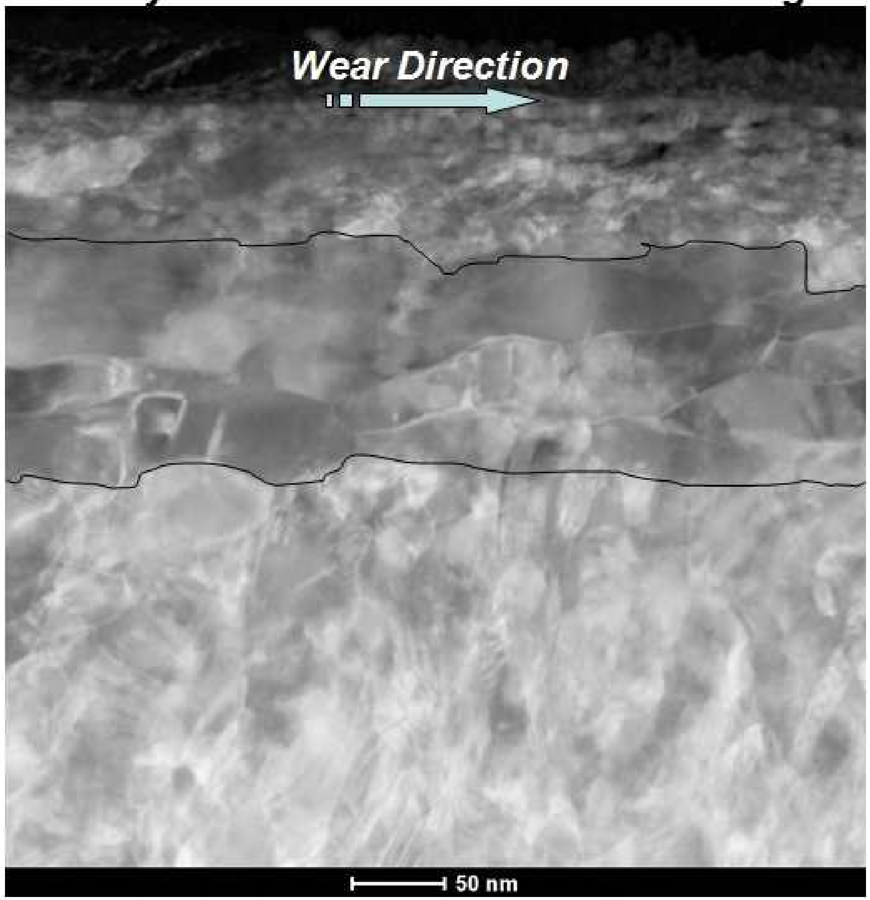
Frictional Processes in Nanocrystalline Alloys

Nanocrystalline Ni ***Annular DF Image***

Zone 1
Ultra Nanocrystalline

Zone 2
Grain Growth + Texture

Zone 3
Parent (Bulk) Microstructure



Ni-3C-A1 #1 051202D Track 19, 10g, 20rpm, 600cycles

- Nanocrystalline alloys exhibit *unusually low friction coefficients (μ<0.3) under some conditions,* associated with the formation of a tribological bilayer.
- Grain-growth in Zone 2 is thought to provide a confined shear accomodation layer.
- *Friction behavior is highly rate sensitive (due to grain-boundary sliding mechanisms?)*

Proposed Work: Task 1

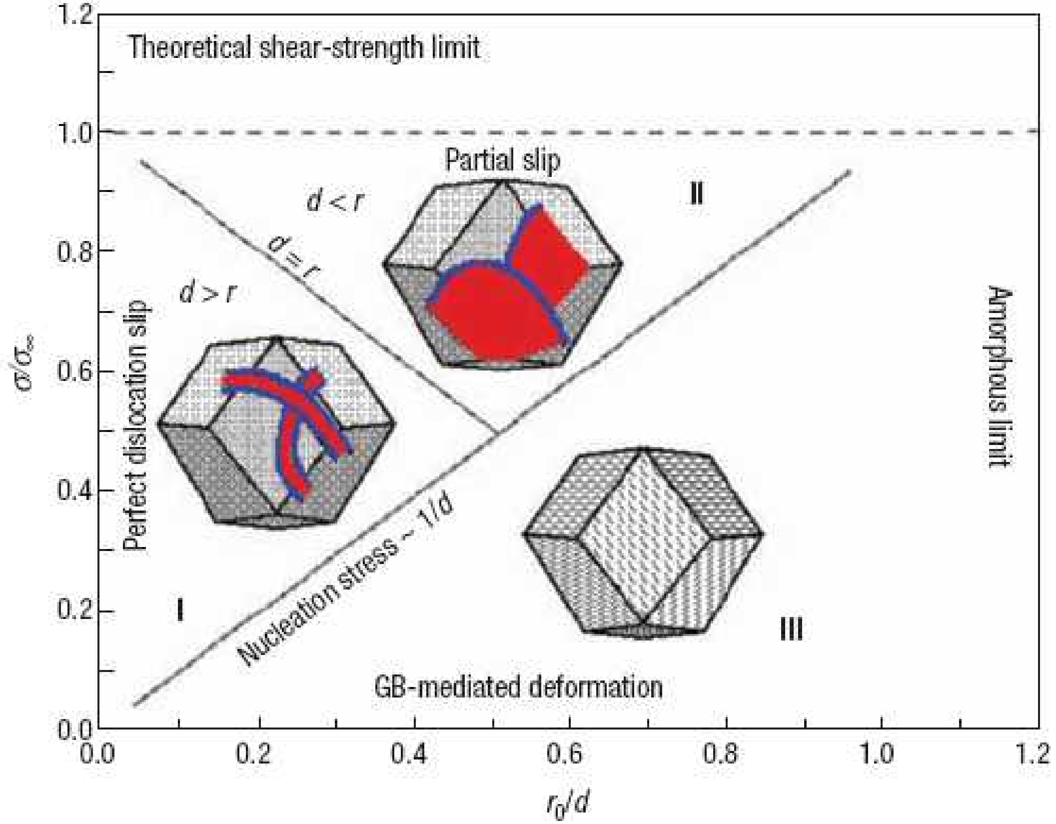
TASK 1
Environmental stability of nanostructures
BL Boyce

A. Construct a 'deformation-mechanism map' for coupled thermal+mechanical nanostructural evolution as a function of stress, temperature, grain size, and alloy content.

B. Observe grain growth kinetics of various nanocrystalline metallurgical systems through diffraction peak broadening and direct observation using a newly developed thin film thermal+mechanical loadframe.

C. Investigate the stabilizing (or destabilizing?) effect of ion irradiation damage on thermal and mechanical grain growth processes.

A 'Deformation Mechanism Map' for Nanocrystalline Grain Stabilility?

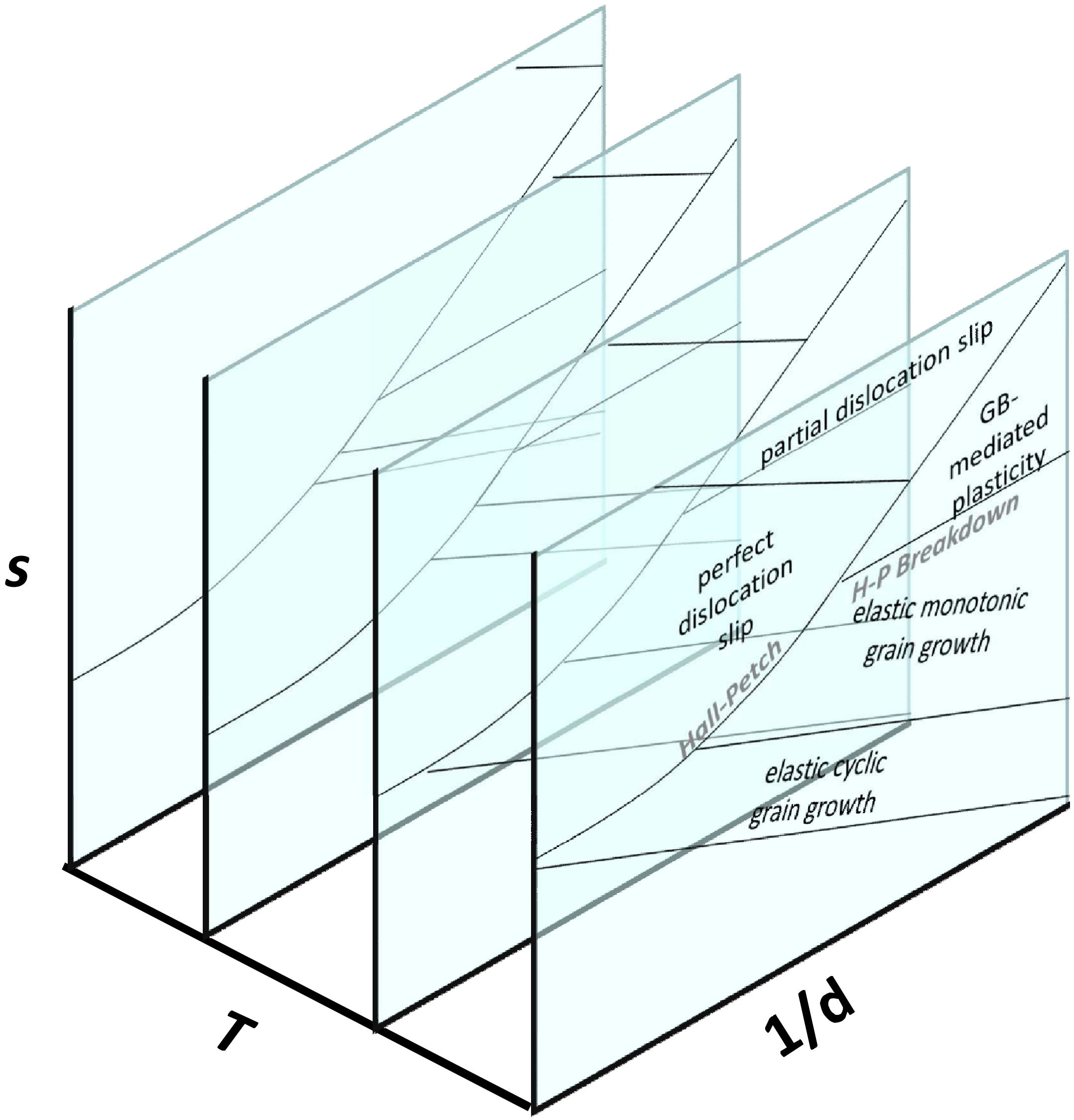


Deformation-mechanism map for nanocrystalline metals by molecular-dynamics simulation

V. YAMAKOV[1,†], D. WOLF*[1], S. R. PHILLPOT[1,‡], A. K. MUKHERJEE[2] AND H. GLEITER[3]

nature materials | VOL 3 | JANUARY 2004

A 'Universal Mechanism Map' for exploring stress – temperature – grain size space



PLASTICITY:
- dislocation processes
- grain-boundary sliding
- grain rotation
- nanotwinning

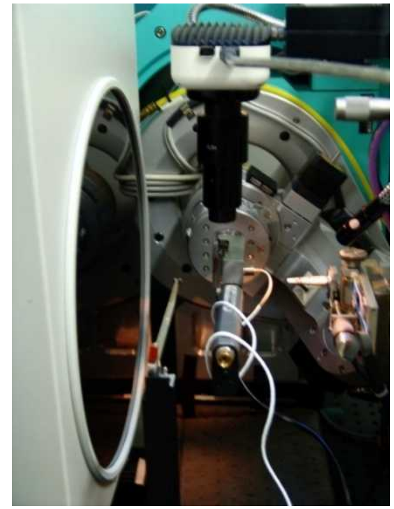
CREEP:
- GB vs. bulk diffusion
- power-law vs. diffusive creep

GRAIN GROWTH:
- abnormal vs. normal growth
- thermal vs. mechanical
- stress vs. strain driven
- monotonic vs. cyclic

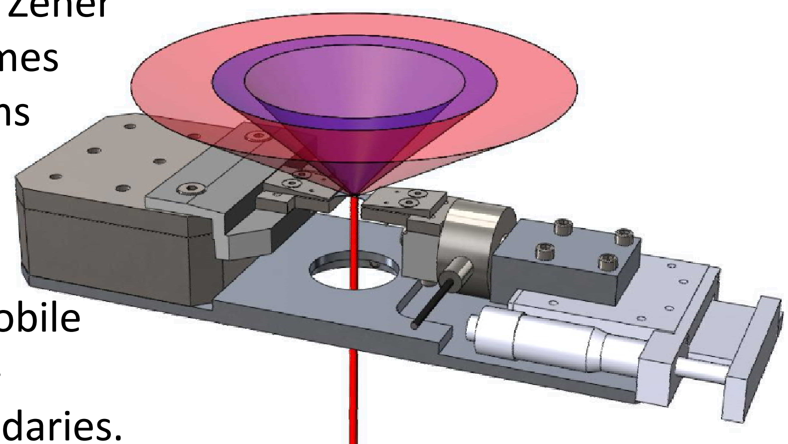
Key Experiments for Environmental Stability of Nanocrystalline Alloys

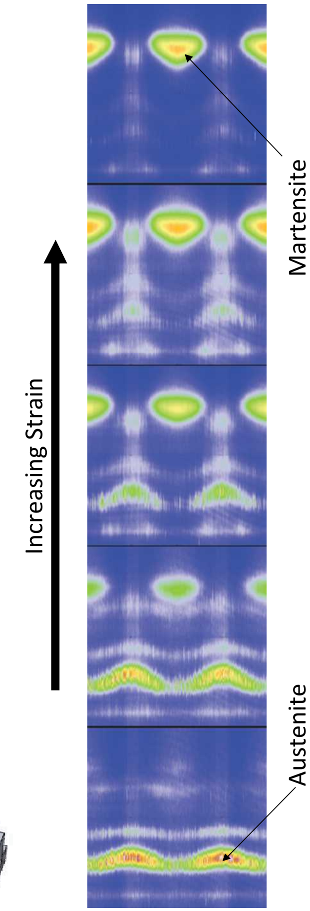
Digital Image Correlation Strain-field Mapping and Transmission monochromatic polycrystalline diffraction

- In an unalloyed pure metallic system (PLD Ni), study the kinetics of mechanical and thermal grain growth.
- Establish a stress-temperature-grain size mechanism map using in-situ XRD, FIB, TEM as well as ex-situ techniques.
- Examine the alloying effects of grain-stabilization through solute drag and Zener pinning. Do these stabilization schemes affect each of the growth mechanisms similarly?
- In FIB, perform 3D EBSD on highly mobile grain boundaries to determine full 5-dimensional character of those boundaries.



SSRL beamline 11.3





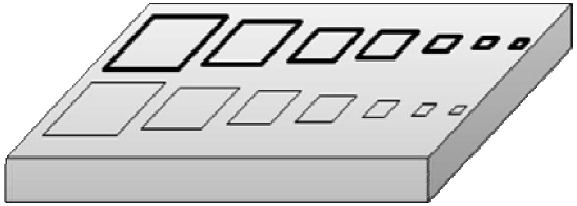
Ion-Damage Mediated Grain Growth Studies

Ion Beam Laboratory Facilities

Focused ion nanoimplanter (FIN):
- liquid metal ion source (LMIS)
- 10 nm ion beam resolution at 1 pA
- 1-100 kV accelerating voltage ion column
- Ga, Si, Be, and Au + other low T_{mp} eutectics
- ***E*** x ***B*** filter

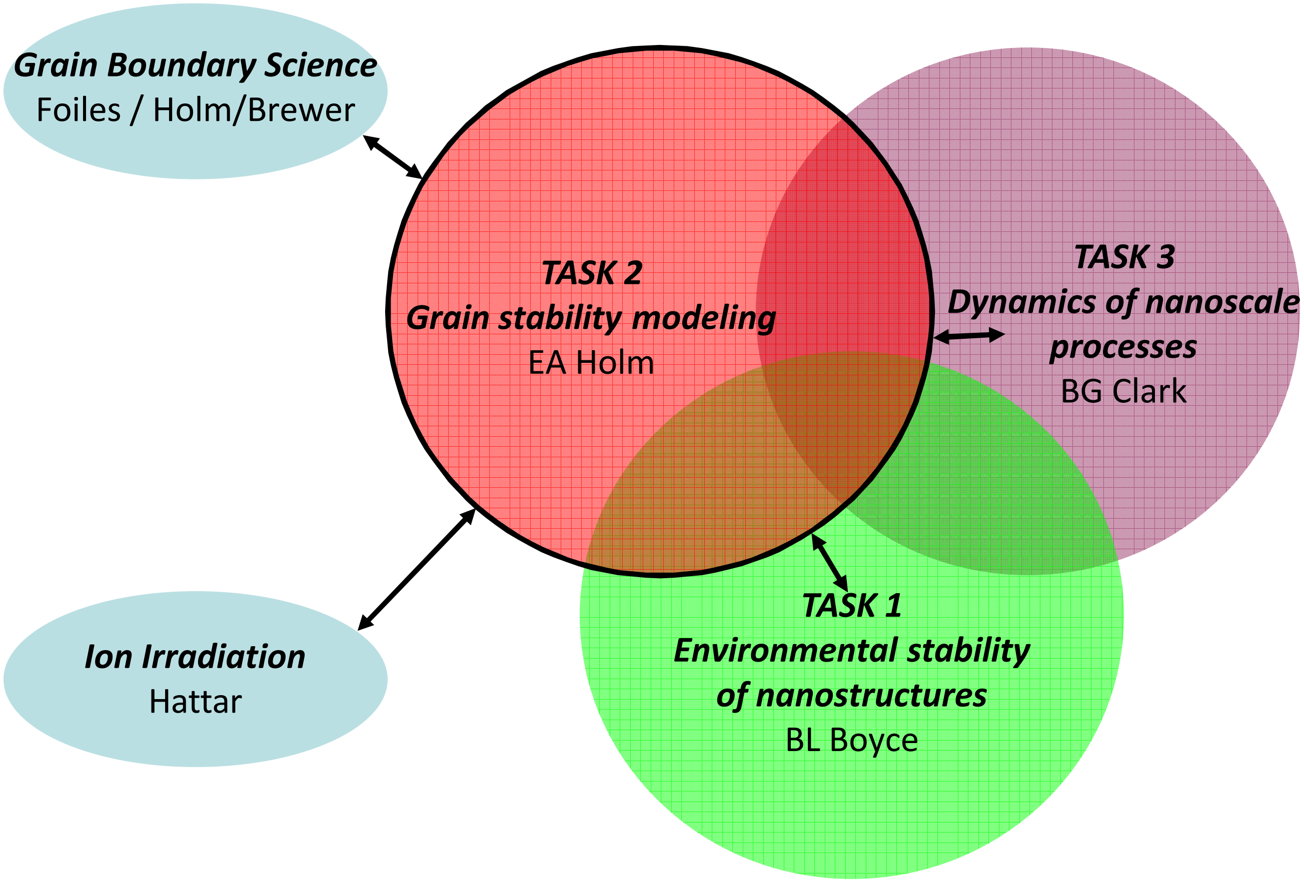
Micro-ONE implanter:
- 10 MeV accelerating voltage (Tandem accelerator)
- Micron scale resolution
- Implantation deeper than 1 μm possible
- Self-implantation (Ni in Ni) or alloying (Fe in Ni)

Experiment:
- in-situ thermal and/or mechanical loading in FIB before and after ion irradiation.
- *observe grain growth & deformation as a function of local ion irradiation:*
  - Impart local alloying or self-ion damage
  - Graded implantation lines allow a combinatorial-style examination of dose effects
  - Locally stablize (or induce?) grain-boundary motion.
  - Patterned irradiation to study size effects (to nm scale)

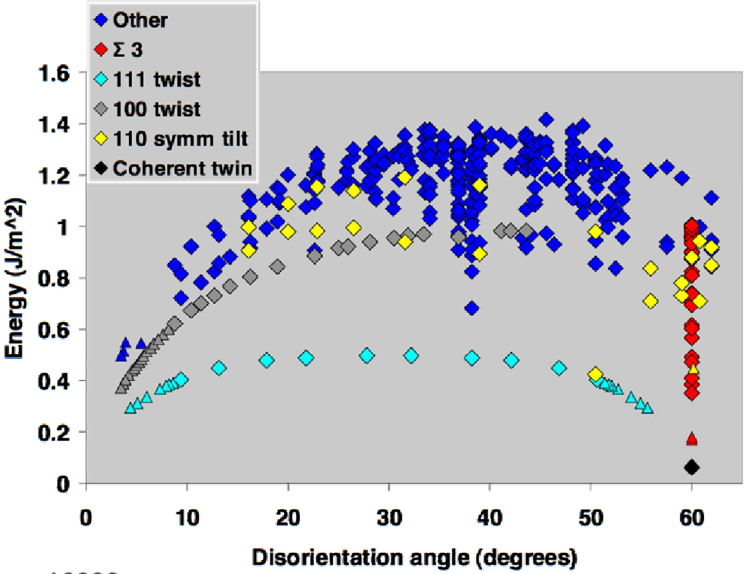


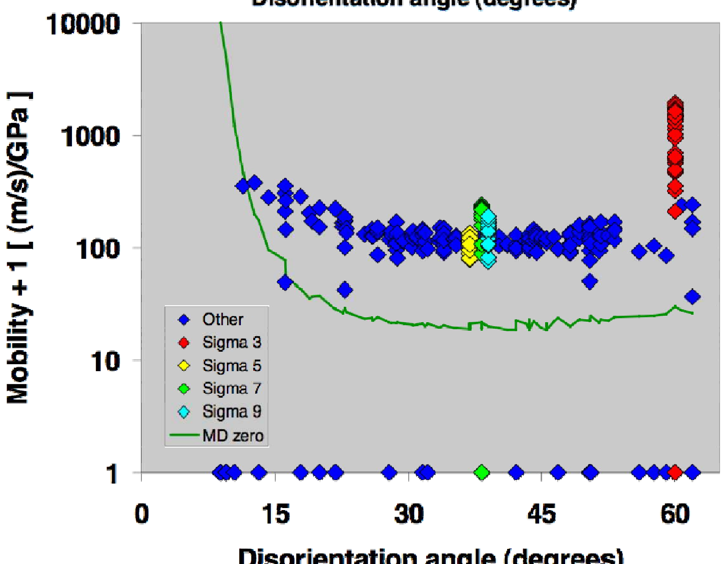
See poster by K.M. Hattar

Nanomechanics and Nanometallurgy of Boundaries



Theory of Microstructures accomplishment: Atomic-scale simulations of grain boundary properties

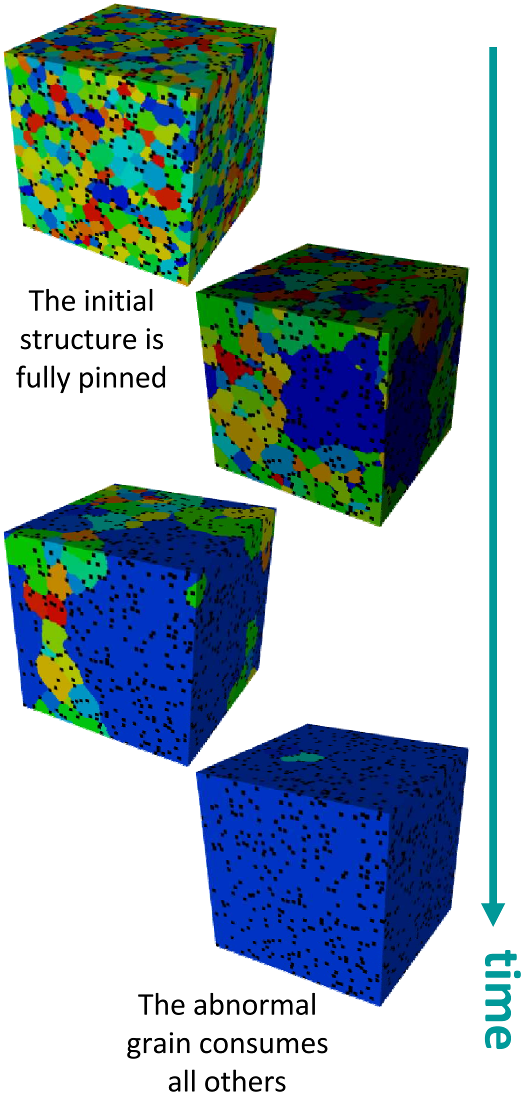




- Developed high throughput MD method for calculating grain boundary energy and mobility
- Determined energy and mobility for a catalog of 388 grain boundaries in 4 materials
- Results provide a wealth of new insights:
  - *Pervasive shear-coupled boundary motion*
  - *Existence of non-activated motion*
  - *Broad range of properties for Σ3 boundaries*
  - *Wide range of boundary roughening temperatures*
  - *Importance of grain boundary plane*
  - *Crystallographic correlations*
  - *…*
- Subject of 5 journal papers since 2006.

Highlight Talk by E.A. Holm

Theory of Microstructures accomplishment: Mesoscale simulations of abnormal grain growth



- Abnormal grain growth scenarios studied with a mesoscale grain growth model.
- In polycrystals that contain static particles, **particles that inhibit normal grain growth actually cause abnormal grain growth.**
  - *Previous theories relied on local dissolution of particles, which is not experimentally supported.*
  - *Once grain boundaries thermally fluctuate off o f particle sites, they move unimpeded.*
  - *This mechanism explains decades of observations of AGG in superalloys, ceramic composites, aluminum alloys, steels, and others.*
- Subject of 5 invited presentations since 2006.

See poster by E.A. Holm

Proposed Work: Task 2

TASK 2
Grain stability modeling
EA Holm

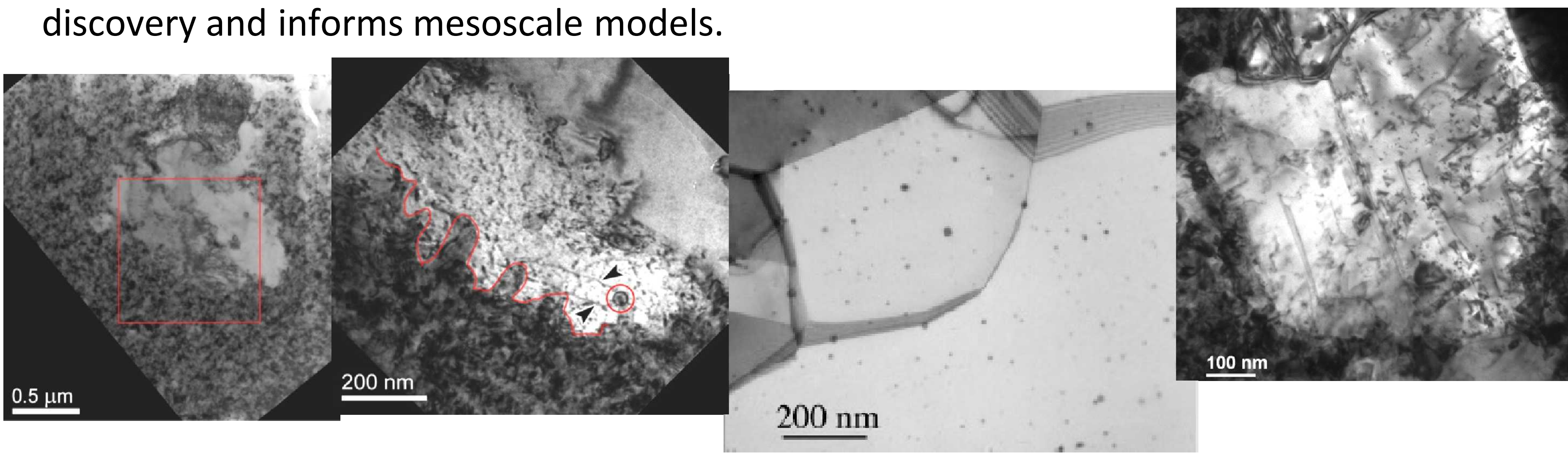
A. Develop a high-throughput MD computational framework to investigate grain boundary interactions with defects, particles, voids, etc.

B. Develop a comprehensive survey of abnormal grain growth (AGG) mechanisms building towards a uniform theory that can explain effects of solute, temperature, particle pinning, film effects, and mechanical coupling.

C. Develop phenomenology to couple mechanical state to microstructural evolution, including elastic stress coupling, plasticity effects, and crystallographic directionality effects.

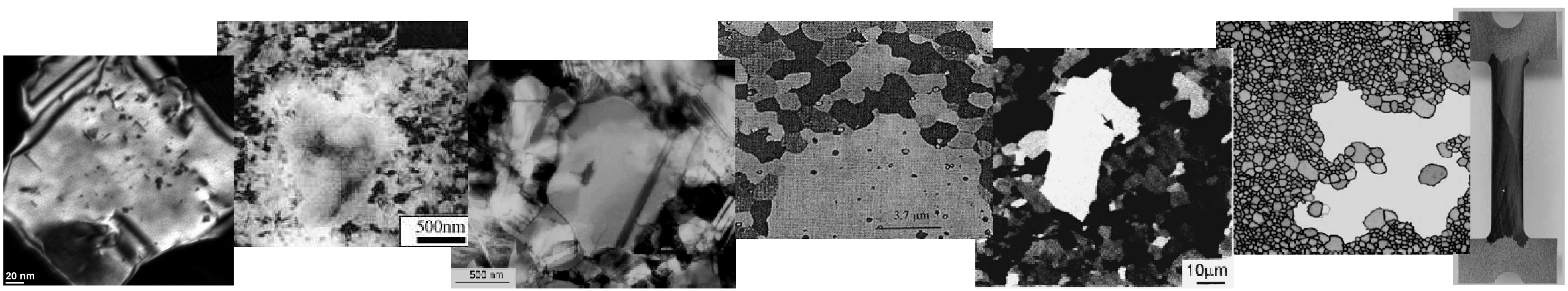
Theory of Microstructures future direction: High throughput atomistics for grain boundary interactions

- Interactions between grain boundaries and other microstructural features govern a variety of microstructural processes, yet we lack quantitative phenomenology:
  - *Defect generation during nanocrystalline grain growth*
  - *Solute drag inhibiting boundary motion*
  - *Boundary pinning by second phase particles or voids*
  - *Dislocation/boundary interaction during plasticity*
- Building on our previous studies of grain boundary properties, we will develop high-throughput methods for grain boundary / defect interactions. This permits mechanistic discovery and informs mesoscale models.



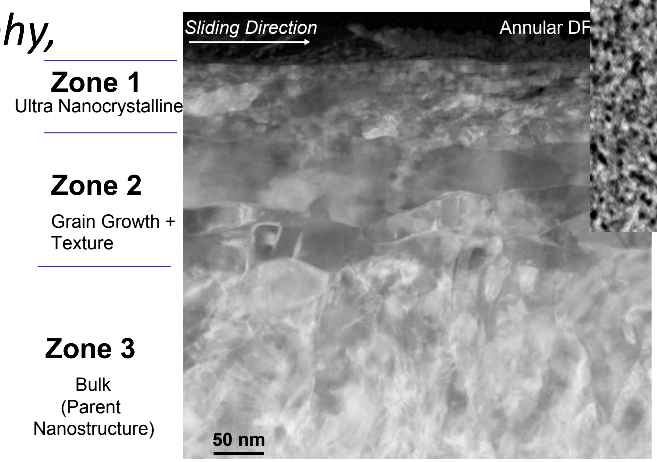
Theory of Microstructures future direction: Solving the mysteries of abnormal grain growth

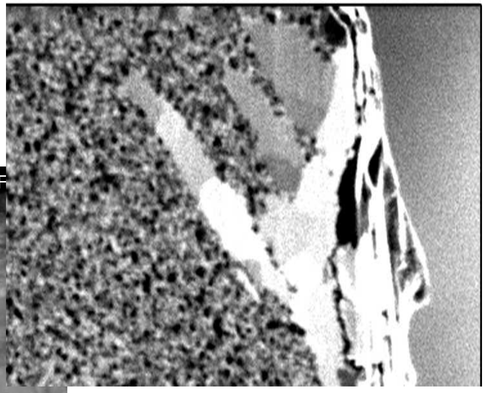
- Abnormal grain growth (AGG) occurs in diverse systems:
  - *Pure, textured systems*
  - *Pure, untextured systems*
  - *Systems with precipitates, near solvus*
  - *Systems with precipitates, far from solvus*
  - *Spatially varying systems*
  - *Strained systems*
  - *Polycrystalline films*
  - *Anisotropic systems*
  - *Refractories under fatigue*
  - *Nanocrystalline systems*
- Current AGG models are empirical and case-by-case (lacking universal phenomenology)
- Building on our expertise in AGG, we will develop a comprehensive catalog of AGG mechanisms and predictive models that can be applied to diverse materials systems.
- *Our goal is to provide the first systematic and physically-based understanding of AGG as one phenomenon with many causes.*



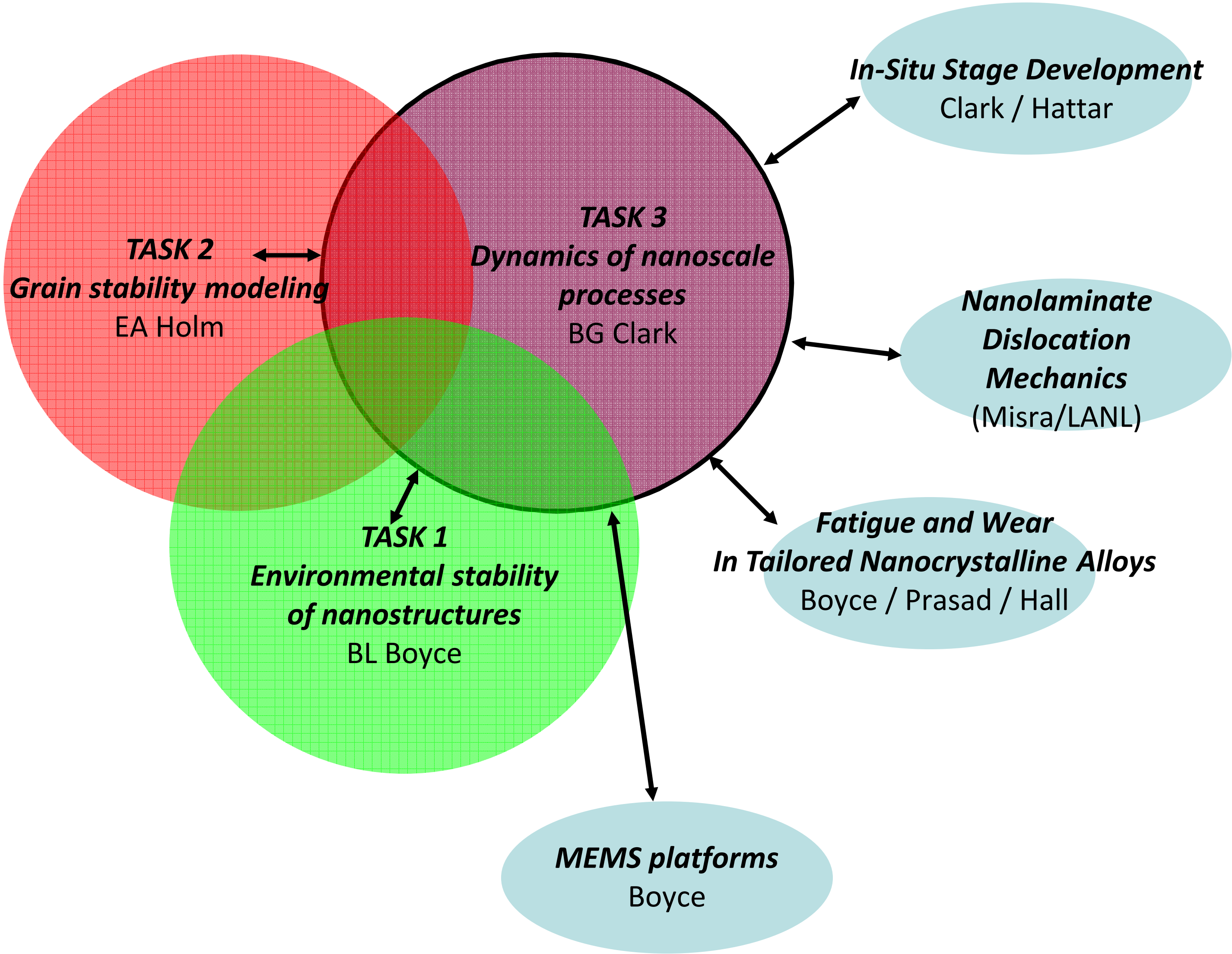
Theory of Microstructures future direction: Coupling mechanical state to microstructural evolution

- Microstructure and micromechanical state are interdependent.
  - *Primary and secondary recrystallization*
  - *Stress or strain induced grain boundary motion (SIGBM)*
  - *Recovery and subgrain formation*
  - *Normal and abnormal grain growth*
- In collaboration with Task 1 and 3 experiments, we will develop a microstructural-scale model for the evolution of grain structure in elastically and plastically strained systems.
- Our goals are:
  - *To understand the complex coupling between stress state, crystallography, and grain growth.*
  - *To inform experiments on systems where stress and microstructure interact.*

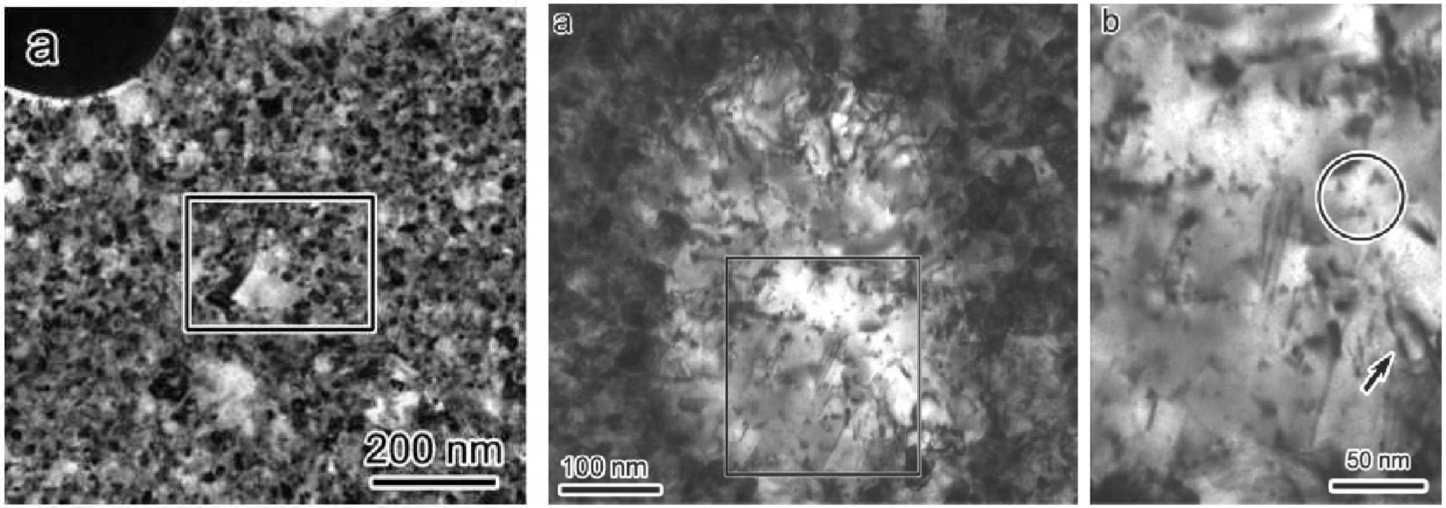




Nanomechanics and Nanometallurgy of Boundaries



In-situ Observation of Defect Residue During Anomalous Grain Growth in PLD Ni

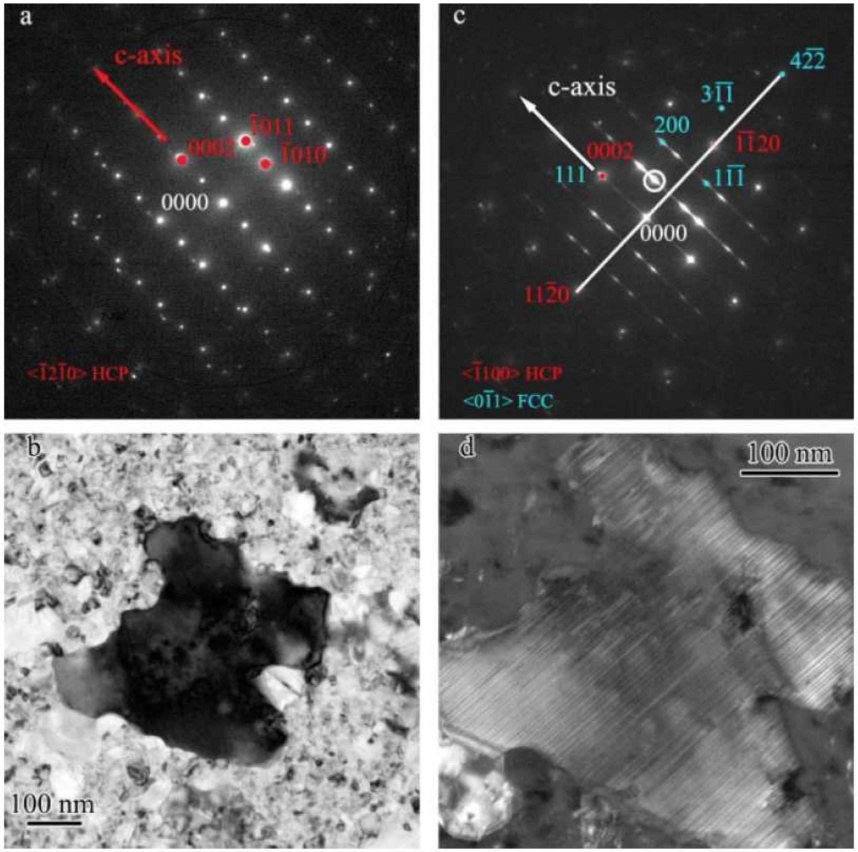


- Catastrophic anomalous grain growth observed during in-situ heating at 498K for 14h.
- Stacking fault tetrahedra, dislocation lines, loops, and twins present
- Defects give insight into boundary migration process. For example, tetrahedra are likely caused by excess free volume in deposited grain boundaries.

Hattar, Follstaedt, Knapp, Robertson, *Acta Materialia*, 2008

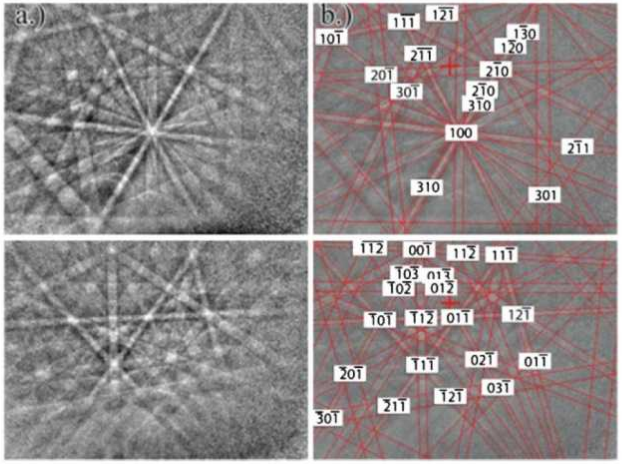
See poster by S. Foiles

Competitive HCP and FCC Ni Abnormal Grain Growth



Brewer, Follstaedt, Hattar, Knapp, Rodriguez, and Robertson, *Advanced Materials*, 2010

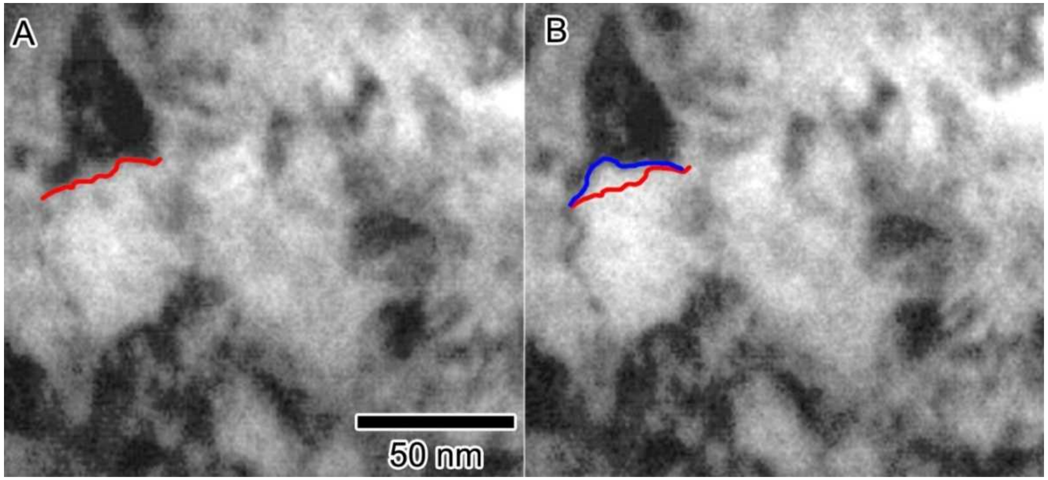
- Faulted grain structures found in large grains after abnormal grain growth at 548K for 17h.
- EBSD confirms presence of metastable HCP phase.
- First time observed during heating of PLD Ni
- Both FCC and HCP abnormal grains appear to grow competitively, presumably due to competing mechanisms of accommodating excess free volume of consumed boundaries.



In-Situ TEM Nanomechanics: Recent Results FY07-09

Stress-Driven Grain Growth in PLD Ni at Room Temperature

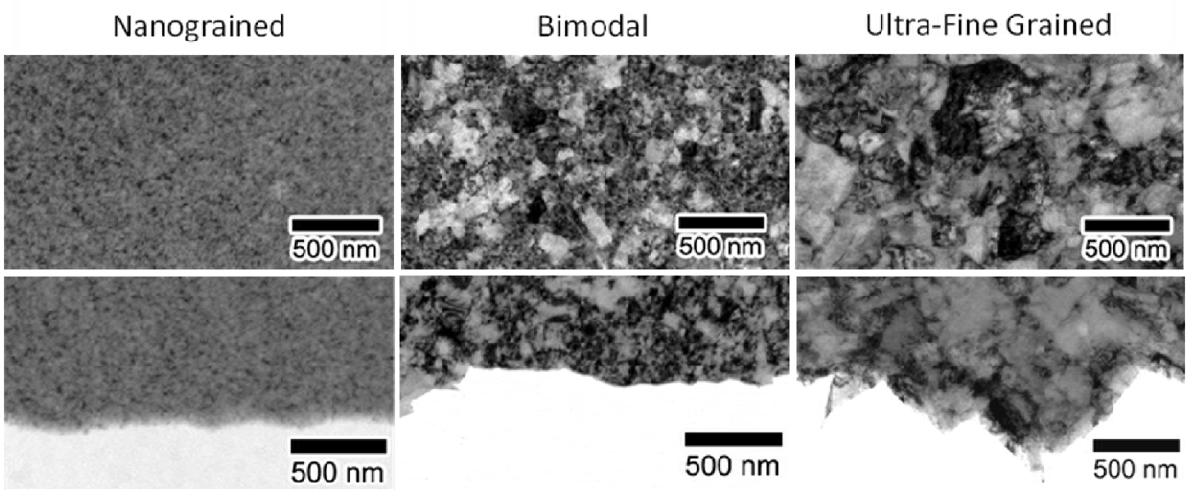
- Observed during in-situ TEM straining of PLD Ni
- Boundary motion driven by stress
- First time directly observed in PLD Ni at room temp



See poster by B.G. Clark

In-Situ TEM Nanomechanics: Recent Results FY07-09

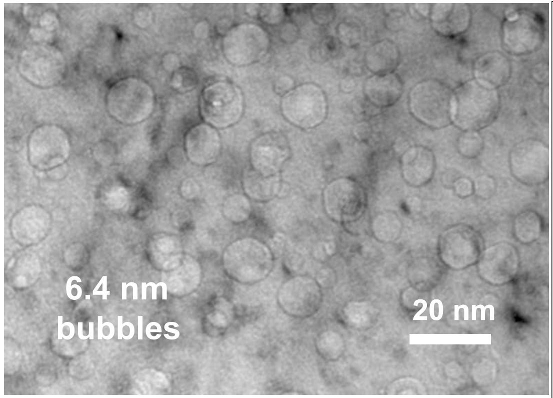
Comparison of Deformation Modes for Different Nanostructures

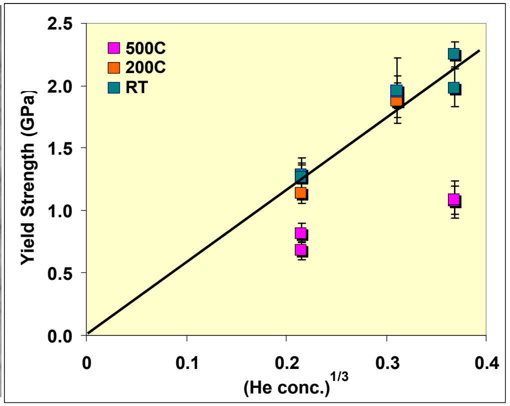


- In-situ TEM straining of free-standing PLD Ni
- Comparison shows failure modes related to grain structure
- Bimodal grain structure → enhanced toughness

Mechanical Properties of Nanograined Ni with He Bubbles

- Ni films implanted with He via ion irradiation
- Nanoindentation data shows increase in hardness with increase in He concentration
- Strength increase scales with Orowan theory





See poster by J.A. Knapp

Proposed Work: Task 3

TASK 3
Dynamics of nanoscale processes
BG Clark

A. Observe direct thermally driven abnormal grain growth processes and develop mechanistic insight into metastable phase growth in nanocrystalline metals.

B. Evaluate the ductility, toughness, and thermal stability of particle-strengthened nanocrystalline metals.

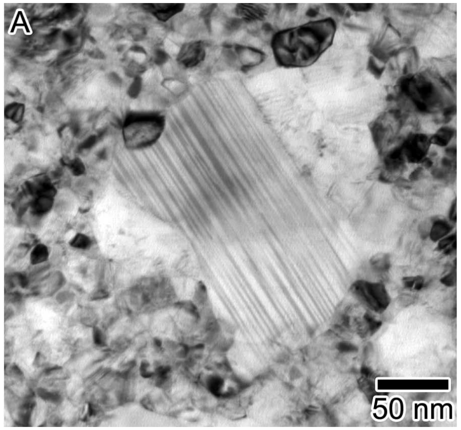
C. Elucidate phenomenology associated with confined plasticity in strained bilayer films.

D. Observe in-situ deformation and grain-growth processes during creep and fatigue loading of nanocrystalline metals.

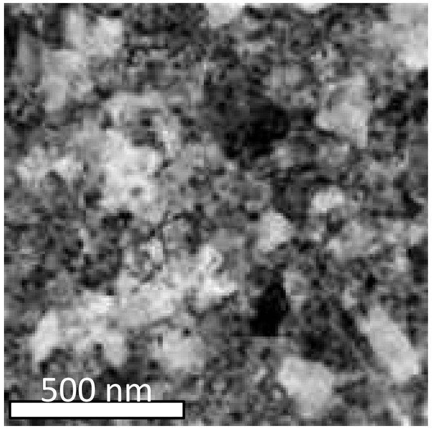
In-Situ TEM Nanomechanics: Proposed Work FY10-12

A. Competitive Grain Growth of HCP Ni in FCC Ni films

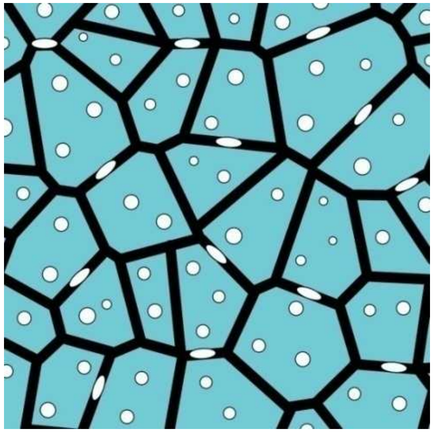
- Use in-situ TEM heating experiments to follow the formation and growth of faulted, HCP grains in PLD Ni films
- Understand the nature of how HCP competes with FCC during abnormal grain growth of Ni
- Characterize the nature of fast-moving boundaries



B. Ductility and Toughness Mechanisms in Nanostructured Materials



Bimodal grain size distribution in Ni

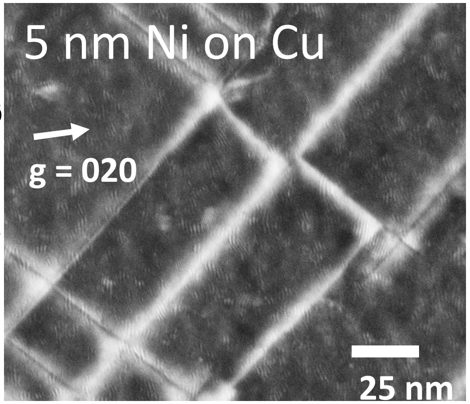

Schematic of nanoparticle-strengthened nanostructure

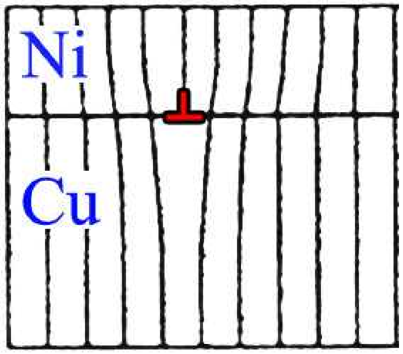
- Study ductility and toughness in nanoparticle-strengthened and bimodal nanograined materials
- Combine in-situ TEM straining (understand mechanisms) with microtensile testing (evaluate mechanical properties)

In-Situ TEM Nanomechanics: Proposed Work FY10-12

C. Confined Plasticity in Strained, Bilayer Films

Mitlin et al., Phil Mag, 2004



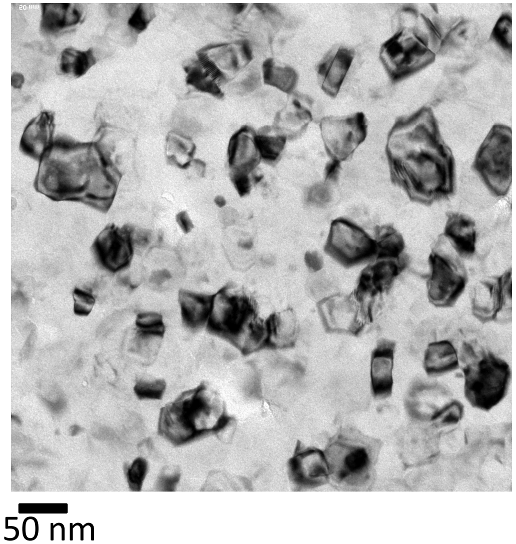


- Nucleation of dislocations not well understood for Ni-Cu
- Very thin, strained Ni layer on Cu
- Analyze defect formations and their evolution using in-situ TEM straining experiments

D. Creep and Fatigue of Nanograined Films

- Mechanisms of creep and fatigue not well understood for nanograined materials
- Use in-situ TEM heating & straining experiments to study creep deformation mechanisms
- Use in-situ TEM straining experiments to study tension-tension fatigue

Nanograined Al



Summary

- Thermomechanical grain boundary stability of nanocrystalline alloys is an issue ripe for discovery. Grain stability in nanocrystalline alloys is an issue in most mechanical phenomena from indentation behavior to fatigue performance.
- The emergence of several characterization tools (FIB, in-situ TEM, μXRD, MEMS, nanoindentation) in the past decade has enabled recent discoveries. Continued development of new instruments or hybridization of instruments will pave the way for future discoveries.
- BES provides the long-term vision and stable funding necessary for expansive, interconnected, deeply probing *discovery-class* science.

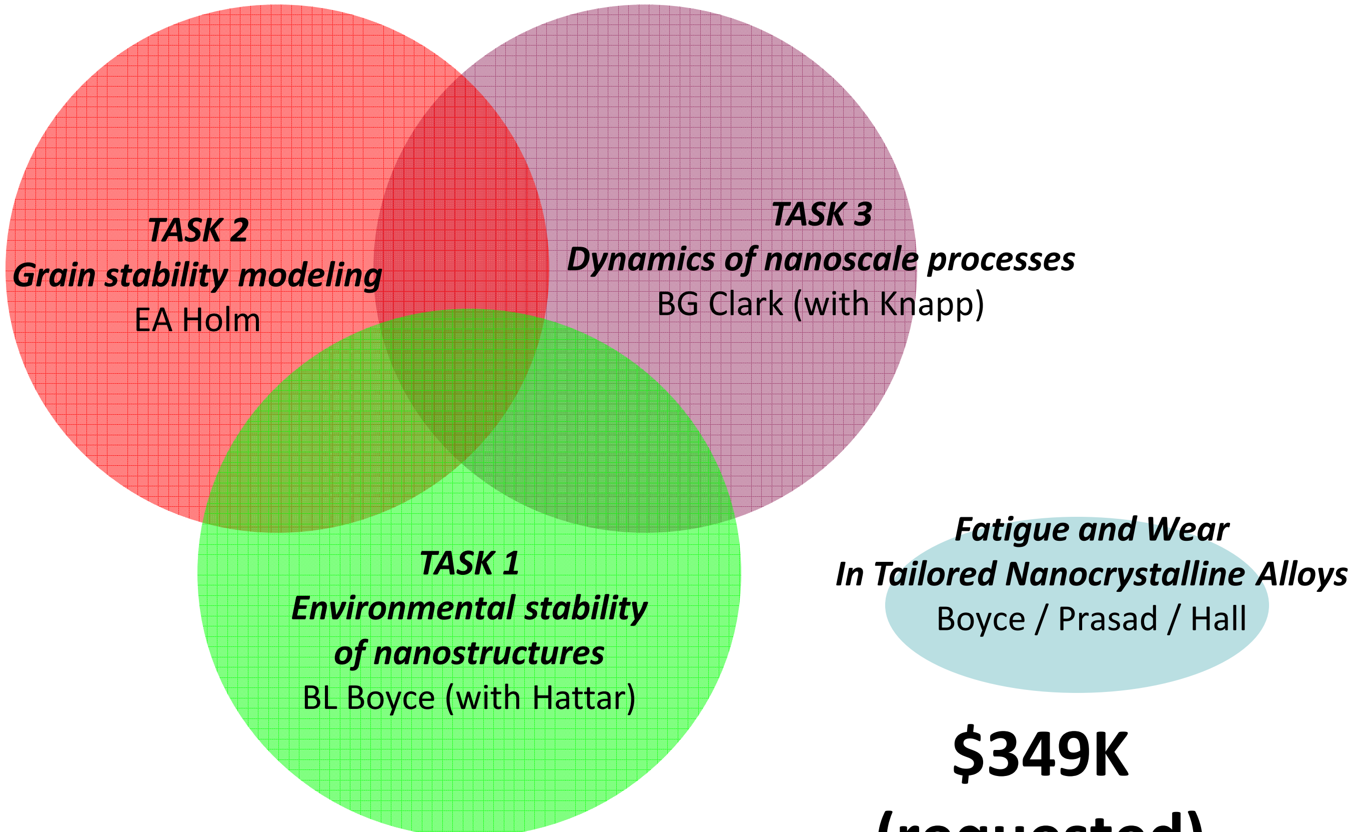
Posters

- B.L. Boyce: “Fatigue-Induced Coarsening Induces Crack Initiation in Nanocrystalline Ni Alloys”
- E.A. Holm: “Particle-assisted Abnormal Grain Growth”
- S.M. Foiles: “Molecular Dynamics Simulations of Nanocrystalline Grain Growth”
- K.M. Hattar: “Irradiation Effects on Mechanical Properties at the Nanoscale”
- B.G. Clark: “Nanoscale Deformation and Evolution Mechanisms via In-Situ TEM”
- J.A. Knapp: “Hardening by He Bubbles”

Publications 2007-2009

1. D. Olmsted, E. A. Holm, S. M. Foiles, "Survey of grain boundary properties in fcc metals: II. Grain boundary mobility," *Acta Mater.* **57** 3704–3713 (2009).
2. D. Olmsted, S. M. Foiles, E. A. Holm, "Survey of grain boundary properties in fcc metals: I. Grain boundary energy," *Acta Mater.* **57** 3694–3703 (2009).
3. S. M. Foiles, D. Olmsted, E. A. Holm, "Using Atomisic Simulations to Inform Mesoscale Simulations of Microstrucuture Evolution," *Proc. 4th Int. Conf. Multiscale Mater. Model.*, A. El-Azab (editor) (Florida State University, Tallahassee, FL, 2008, ISBN 978-0-615-24781-6) pp. 362-368.
4. A. L. Garcia, V. Tikare, E. A. Holm, "3D Simulation of grain growth in a thermal gradient with non-uniform grain boundary mobility," *Scripta Mater.* **59**[6] 661-664 (2008).
5. D. Olmsted, S. M. Foiles, E. A. Holm, "Grain boundary interface roughening transition and its effect on grain boundary mobility for non-faceting boundaries," *Scripta Mater.* **57** 1161-1164 (2007).
6. E. S. McGarrity, K. S. McGarrity, P. M. Duxbury, B. W. Reed, E. A. Holm, "Effects of grain boundary constraint on properties of polycrystalline materials," (2007 Annual Highlight Article; 577 downloads as of 8/08) *Mod. Simul. Mater. Sci. Eng.* **15**[4] S353-S360 (2007).
7. "Dislocation Dynamics in Nanocrystalline Nickel," Z. Shan, D. M. Follstaedt, J. M. K. Wiezorek, J. A. Knapp, E. A. Stach, and S. X. Mao, Phys. Rev. Lett. **98**, 095502 (2007).
8. "Competition between Compressive and Tensile Stress Creation during Constrained Thin Film Island Coalescence," A. Bhandari, B. W. Sheldon, and S. J. Hearne, J. Appl. Phys. **101**, 033528 (2007).
9. "Defect Structures Produced by Low Temperature Annealing of Pulsed-Laser Deposited Nickel," K. Hattar, D. M. Follstaedt, J. A. Knapp, and I. M. Robertson, Acta Mat. **56**, 794 (2008).
10. "Hardening by Bubbles in He-Implanted Ni," J. A. Knapp, D. M. Follstaedt, and S. M. Myers, J. Appl. Phys. **103**, 013518 (2008).
11. "Large Lattice Strain in Individual Grains of Deformed Nanocrystalline Ni," Z. Shan, J. M. K. Wiezorek, J. A. Knapp, D. M. Follstaedt, E. A. Stach and S. X. Mao, Applied Physics Letters, **92**, 091917 (2008).
12. "Inter- and Intra-Agglomerate Fracture in Nanocrystalline Nickel," Z. W. Shan, J. A. Knapp, D. M. Follstaedt, E. A. Stach, J. M. K. Wiezorek, and S. X. Mao, Physical Review Letters, **100**, 105502 (2008).
13. "Defect Structures Created during Abnormal Grain Growth in Pulsed-Laser Deposited Nickel," K. Hattar, D. M. Follstaedt, J. A. Knapp, and I. M. Robertson, Acta Materialia, **56** 794 (2008).
14. "Competitive Abnormal Grain Growth between Allotropic Phases in Nanocrystalline Nickel," L. N. Brewer, D. M. Follstaedt, J. A. Knapp, M. A. Rodriguez, K. Hattar, and I. M. Robertson, Advanced Materials, **22** 1161 (2010).
15. "Identification of hcp Phase in Abnormal-Growth Nickel Grains," L. N. Brewer, D. M. Follstaedt and K. Hattar, in preparation 2009.
16. "Unexpected Annealing Structures and Mechanisms in Nanograined fcc Metals," K. Hattar, D. M. Follstaedt, L. N. Brewer, J. A. Knapp, and I. M. Robertson, Advanced Materials, in preparation 2009.
17. "Deformation and Failure of Bimodal Grain Size Pulsed-Laser Deposited Nickel Films: Including Stress-Driven Grain Growth," K. Hattar, D. M. Follstaedt, J. A. Knapp, S. J. Hearne, and I. M. Robertson, in preparation 2009.
18. "Abnormal Grain Growth in Pulsed-Laser Deposited Nickel," K. Hattar, D. M. Follstaedt, L. N. Brewer, J. A. Knapp, and I. M. Robertson, in preparation 2009.
19. "A Comparison of Thermal Evolution and Resulting Microstructures in Pulsed-Laser Deposited Au, Cu, and Ni," K. Hattar, D. M. Follstaedt, L. N. Brewer, J. A. Knapp, and I. M. Robertson, in preparation 2009.

FY10 Budget:
Nanomechanics and Nanometallurgy of Boundaries

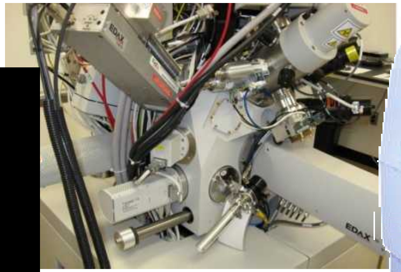


$758K (FY10)

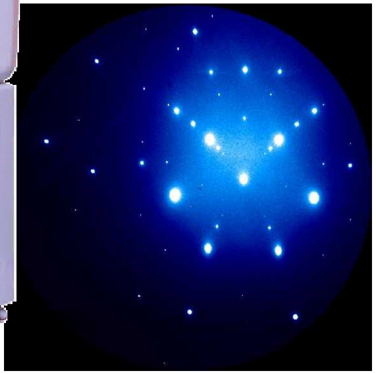
$349K (requested)

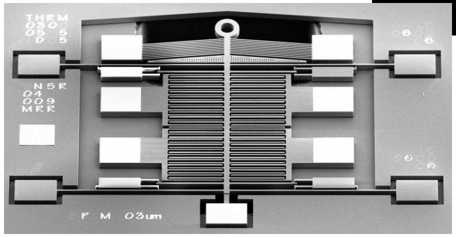
Nanomechanics Tools Drive Discovery

Many recent advances in nanomechanics can be linked to the emergence of new techniques and the hybridization of techniques.



Dual-Beam FIB



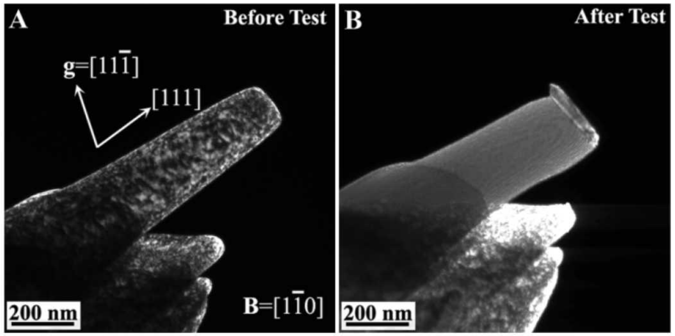


In-Situ
TEM Stages

MEMS platforms

Nanoindentation

Synchrotron
Microdiffraction



In-Situ Micropillar Compression

EBSD
AFM
…