

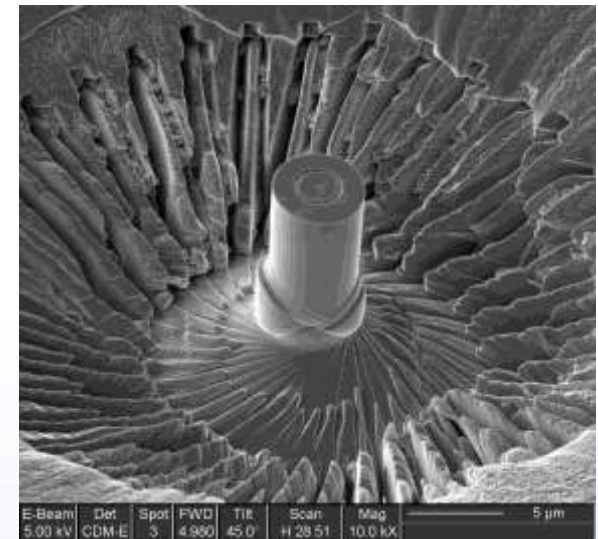
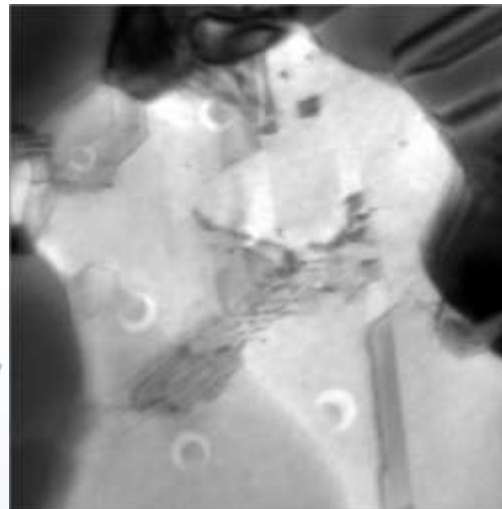
Tailoring the Properties of Metals through Ion Beam Modification

SAND2014-18513C

K. Hattar

Sandia National Laboratories

October 6, 2014



Collaborators:

- IBL: **D. Bufford, D. Buller, S. Pratt, S. Rajasekhara, J. Villone**, and all the IBL staff
- Sandia: T.E. Buchheit, B. Boyce, T.J. Boyle, F.P. Doty, P. Feng, S. Goods, B.A. Hernandez-Sanchez, **A.C. Kilgo**, P.G. Kotula, J. Puskar, **M.J. Rye, J.A. Scott**, P. Yang
- External: N. Li, A. Misra, L.N. Brewer, S. Maloy, **A. McGinnis**, P. Rossi, Protochips Inc.



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Evolution of Metal Processing

Use the Nearest Stone



to

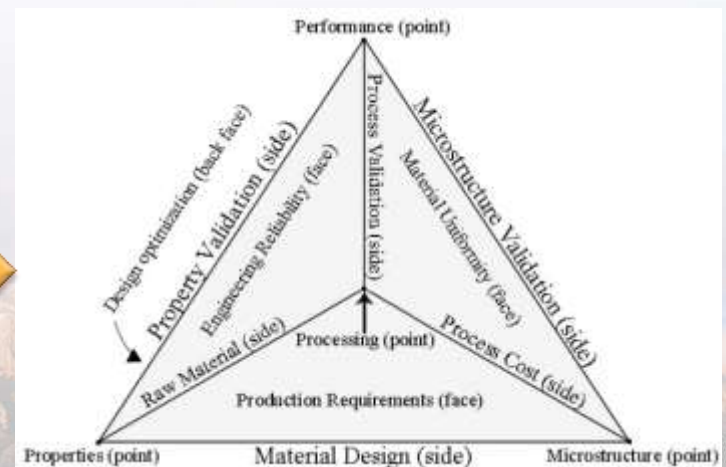
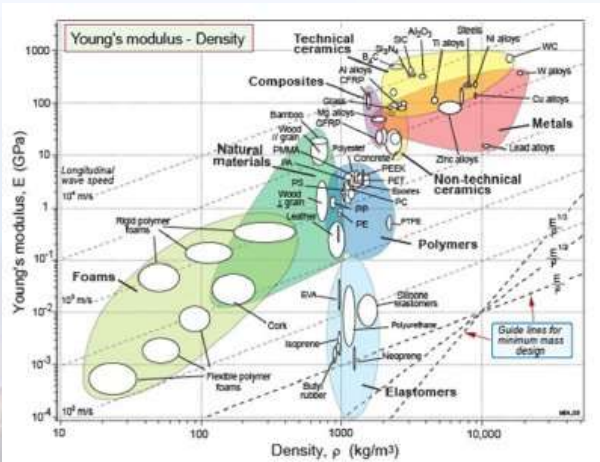


- Radar charts and Ashby plots of current material
- Accelerated and field testing
- **Scientist create a new materials. Engineers find an application.**

Materials by Design

- Physics-based approach
- Requires multiscale modeling
- **Engineers require given properties, Scientists tailor the chemistry and microstructure to achieve it.**

Great vision! We are making strides, but we are not there yet



Micropillar Compression Experiments

Collaborators: M.J. Rye, L.N. Brewer, B. Boyce

Sample Preparation:

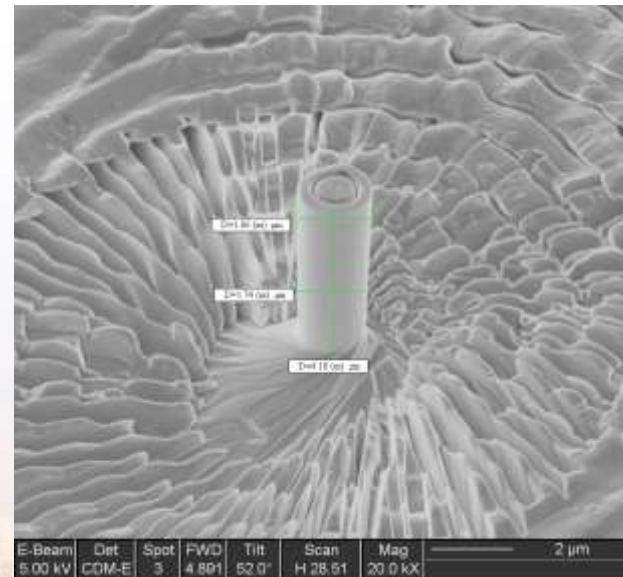
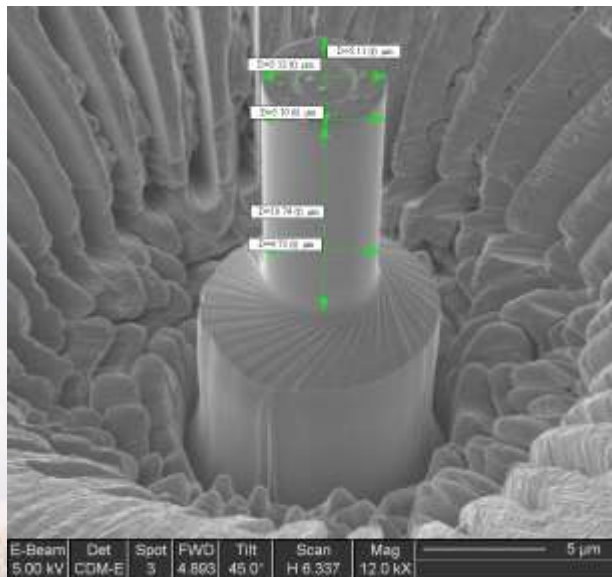
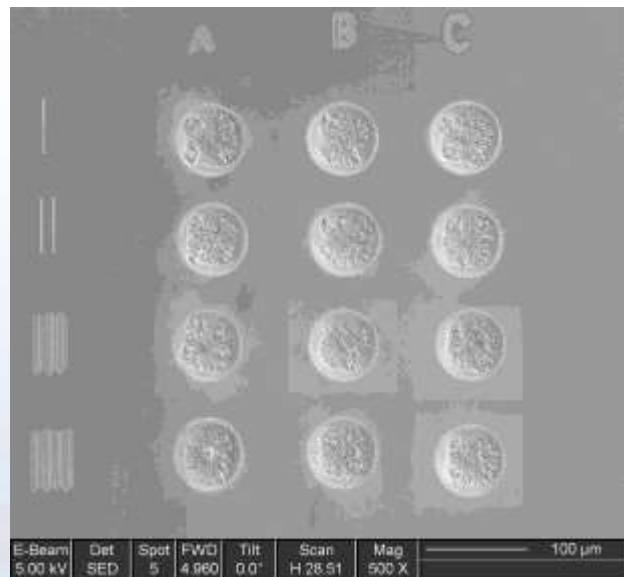
- Copper single crystals (FCC)
- Different crystallographic orientations: (100), (110), and (111)
- Self-ion Implants at 30 MeV to 0 (control), 50 dpa, and 100 dpa.

Pillar Manufacturing:

- We employed Uchic's FIB lathe machining process for straight-walled cylinders.
- Array of at least 9 nominally identical pillars tested per condition to assess statistical variability.
- Height varies from 4 μm to 10 μm

Compression Testing:

- Hysitron Performech Nanoindenter permits <1 nm and <1 μN resolution.
- 25 μm flat ended cone indenter in feedback displacement control, rather than typical force control.
- Pillars compressed 10% strain at a strain rate of 0.025 s^{-1} .

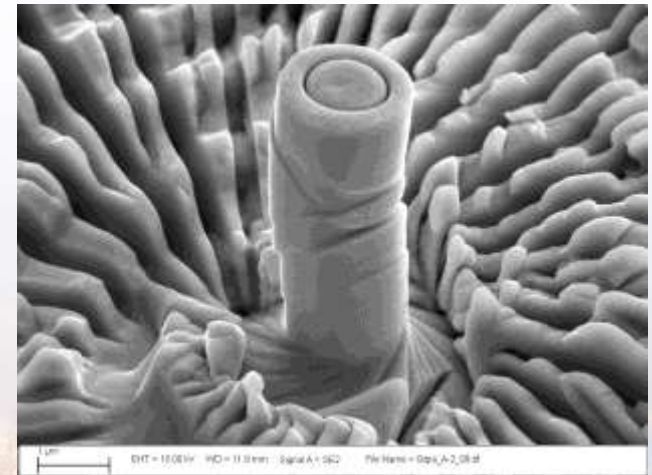
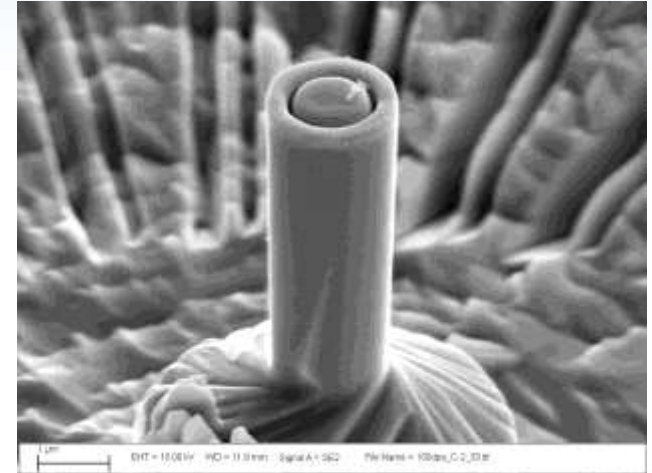
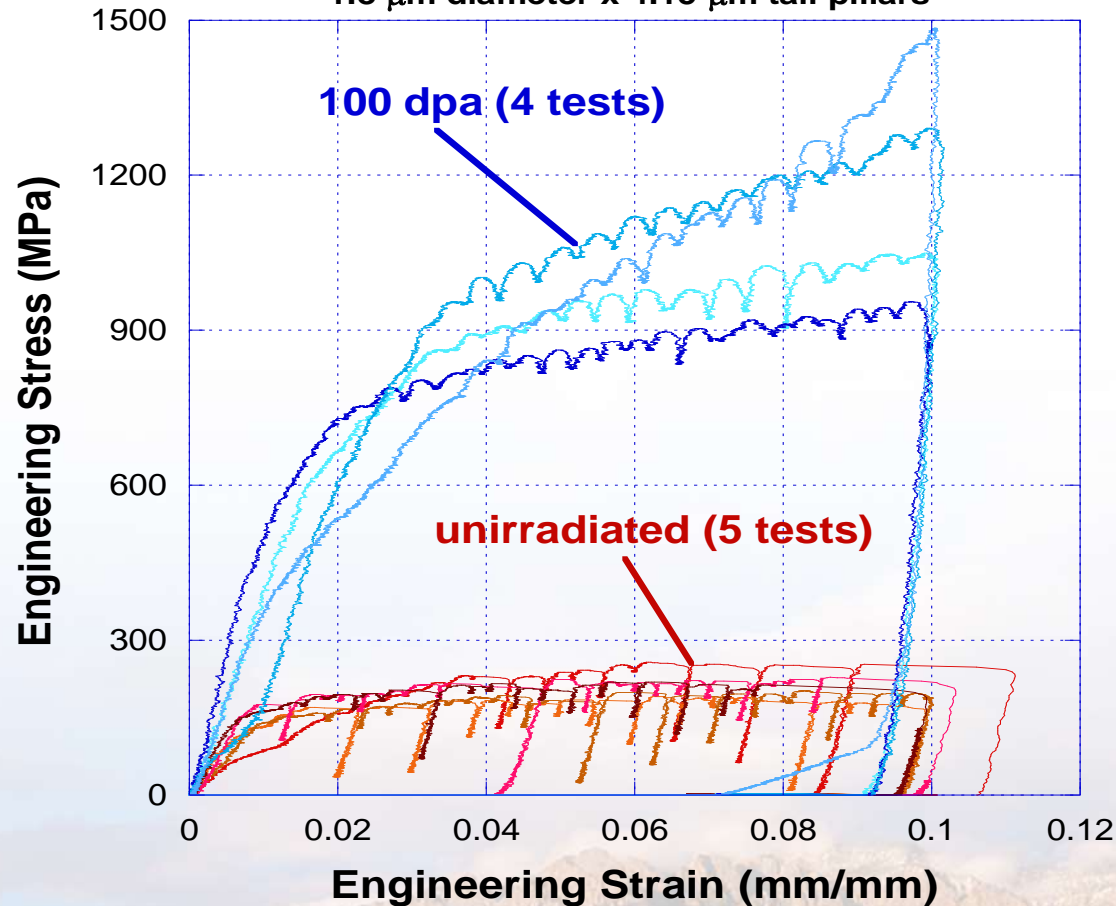


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Small Micropillar Compression

Collaborators: M.J. Rye, L.N. Brewer, B. Boyce

Single Crystal Cu - (110) orientation
1.8 μm diameter x 4.15 μm tall pillars

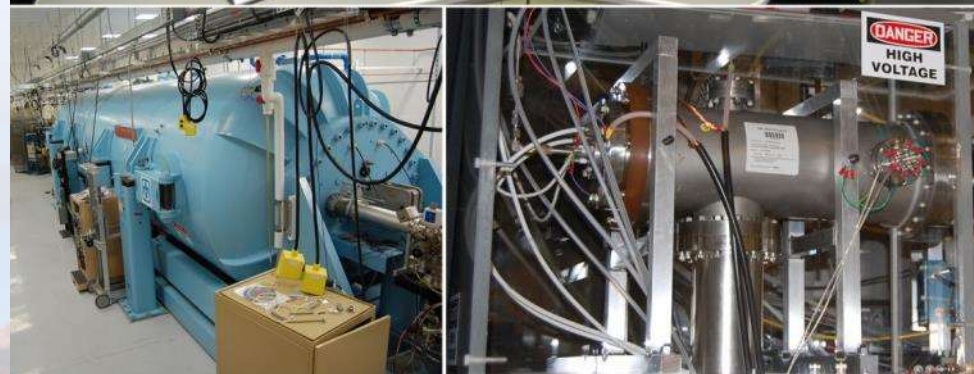


This indicates that the 4 μm -tall pillars are 5 times stronger and show no signs of slip band formation. However, predictive physics-based modeling requires deeper understanding

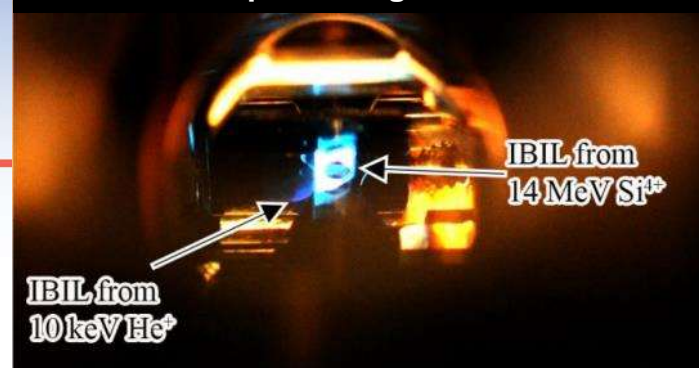
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem

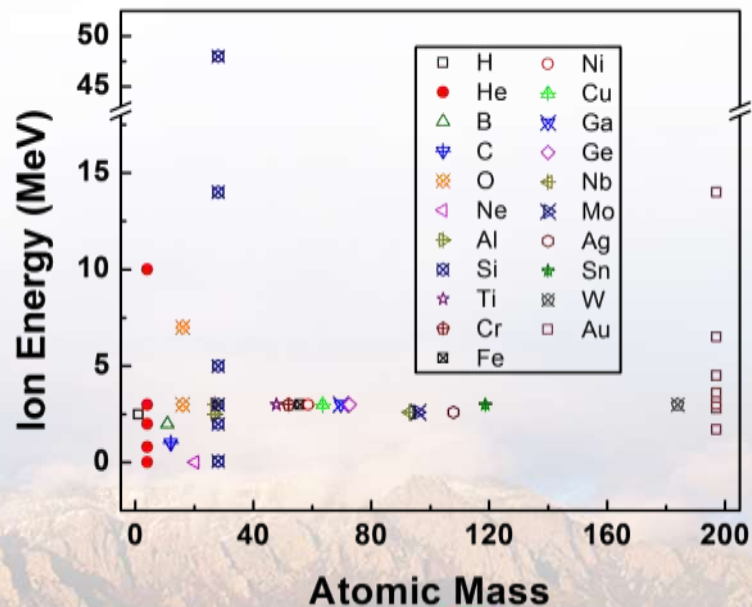


IBIL from a quartz stage inside the TEM



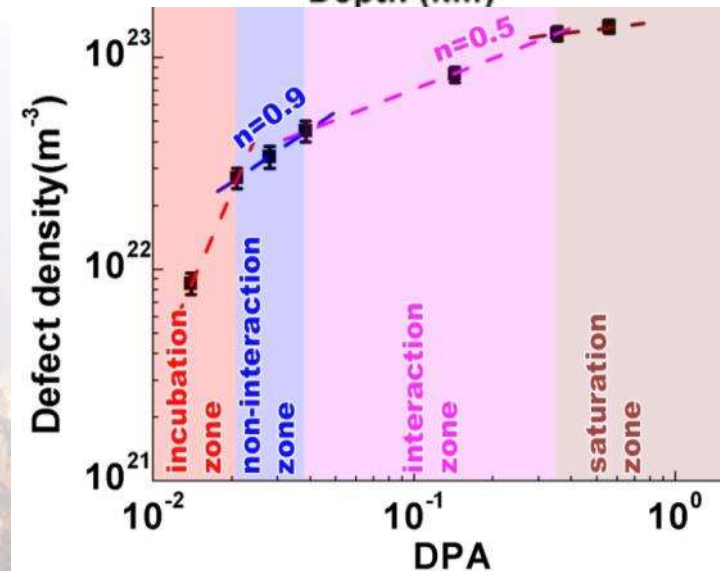
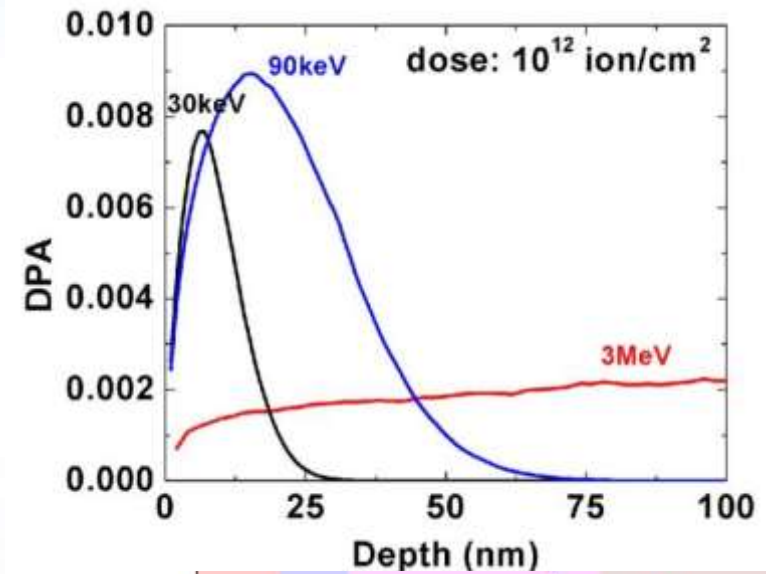
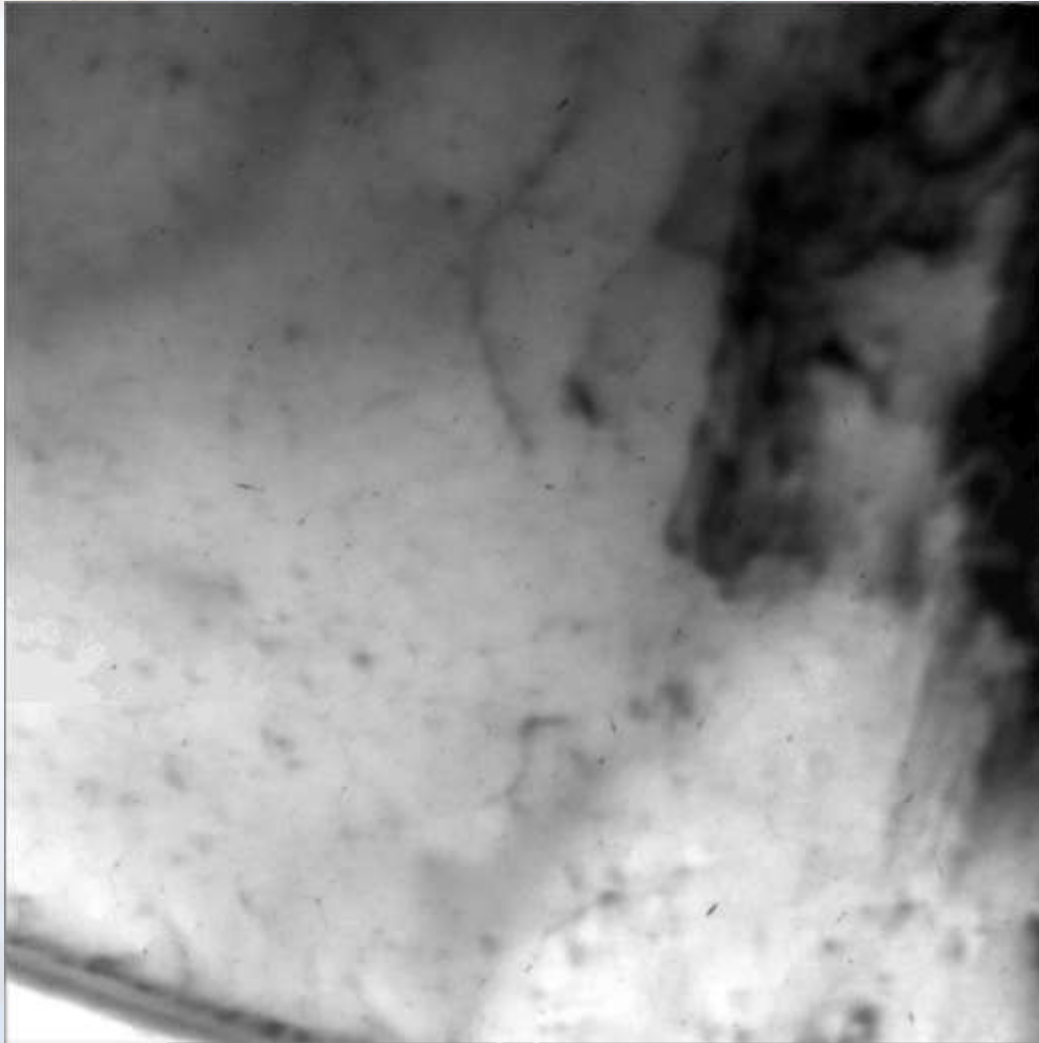
Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution

Ion species & energy introduced into the TEM



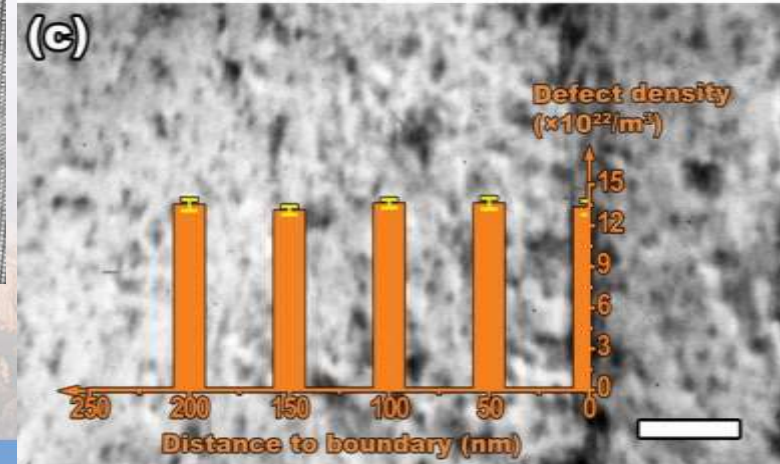
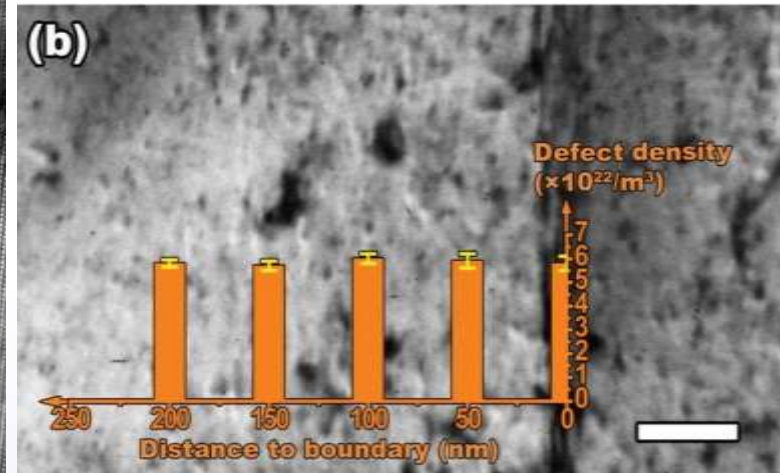
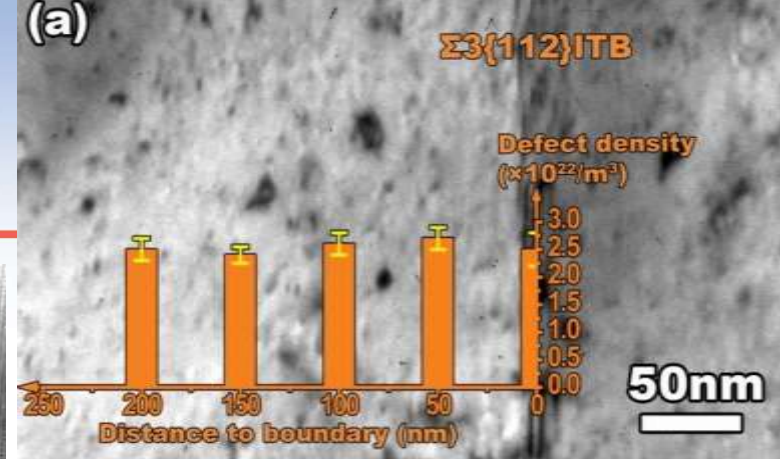
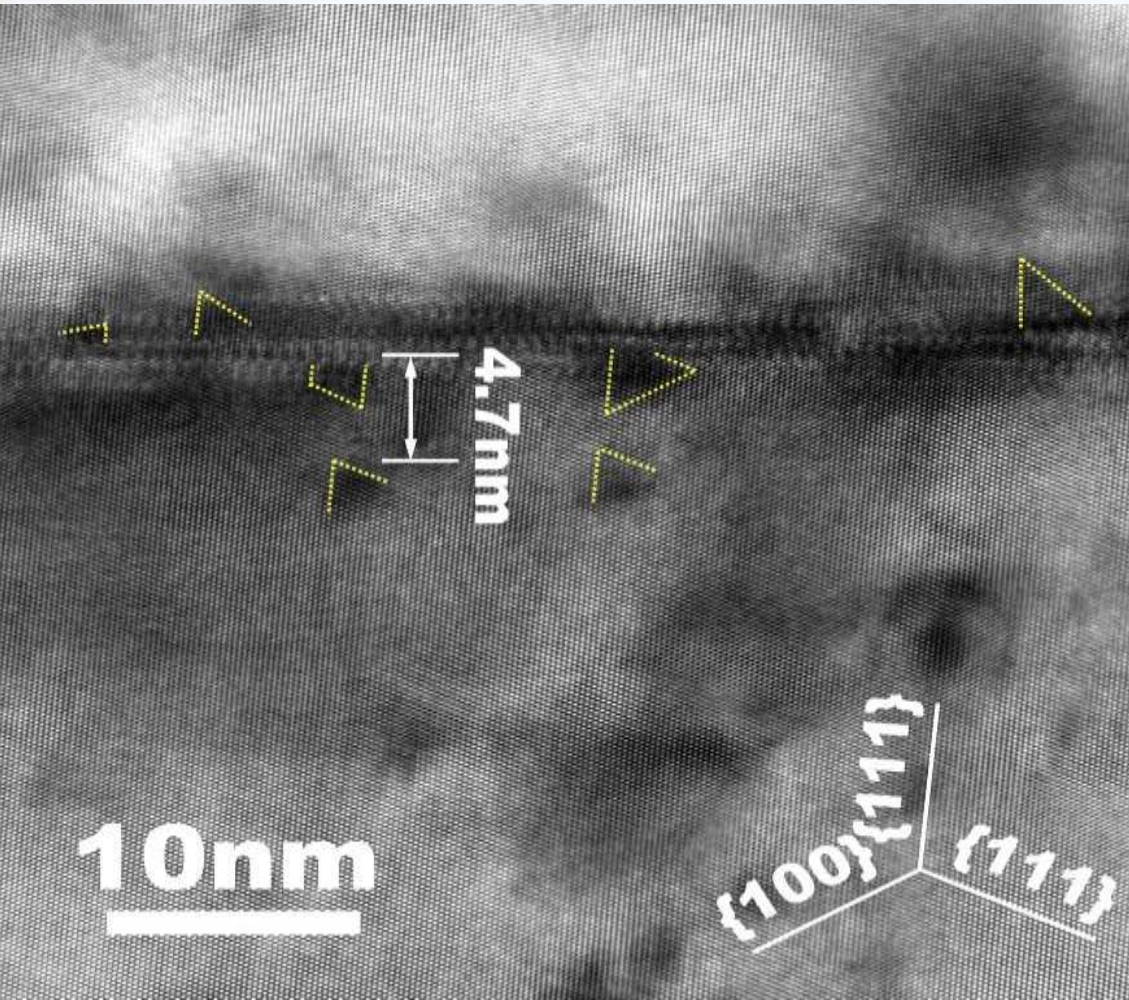
Quantifying Defect Evolution in Irradiated Cu

Collaborators: N. Li & A. Misra



Defects are Altered Little by the Presence of Grain Boundaries

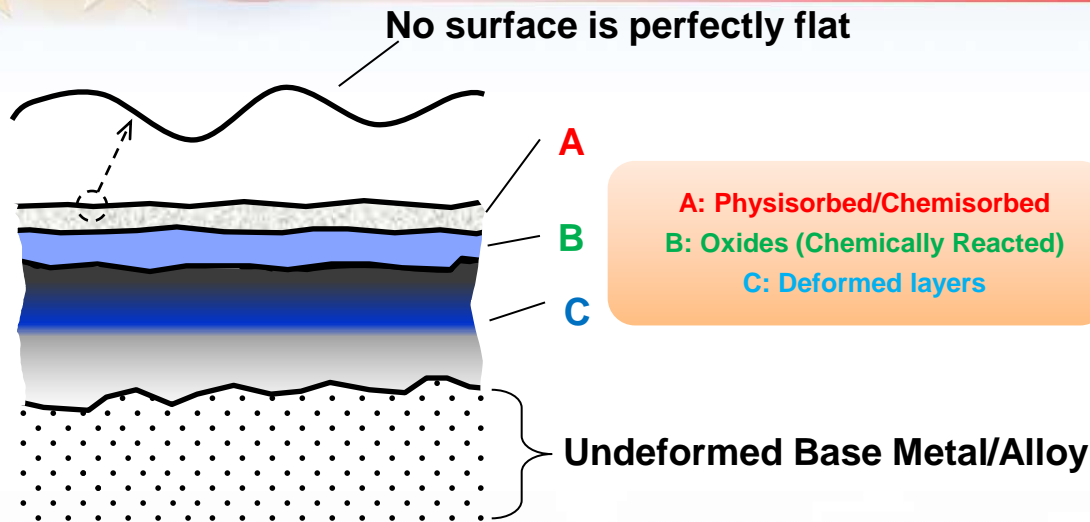
Collaborators: N. Li & A. Misra



SFT appear to be directly at GB

No change in defect density is observed near GB

Tailoring Wear Properties in Au Sliding Contacts

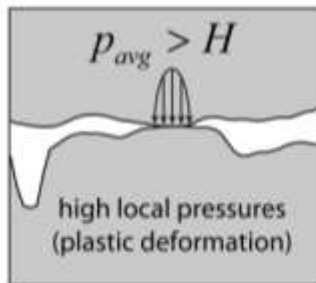


A: Physisorbed/Chemisorbed
B: Oxides (Chemically Reacted)
C: Deformed layers

*Real area of contact (A_r) to be minimized for low adhesion
(Low Adhesive Wear)
Or maximized for reduced electrical contact resistance (ECR)*

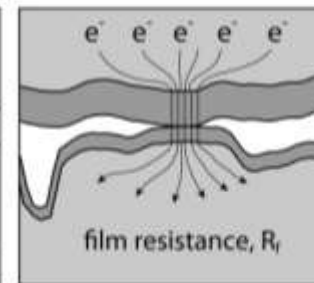
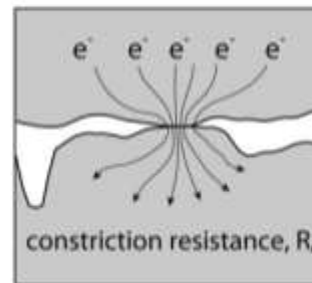
Asperity Contacts, Constriction, Asperity Contacts and Surface Films

areal sum of asperity contacts and surface films define electrical contact resistance



... for metal contacts the real area is a function of hardness and contact force (Bowden & Tabor, 1939):

$$A_r \cong \frac{F_n}{H}$$



... ECR is a function of the constriction and film resistances:

$$ECR = \sum_i (R_{c,i} + R_{f,i})$$

Archard, *Journal of Applied Physics* (1953) 24:981

R. Holm, *Electrical Contacts Handbook* (1958) Berlin: Springer-Verlag

Greenwood & Williamson, *Proc. Royal Society* (1966) A295:300

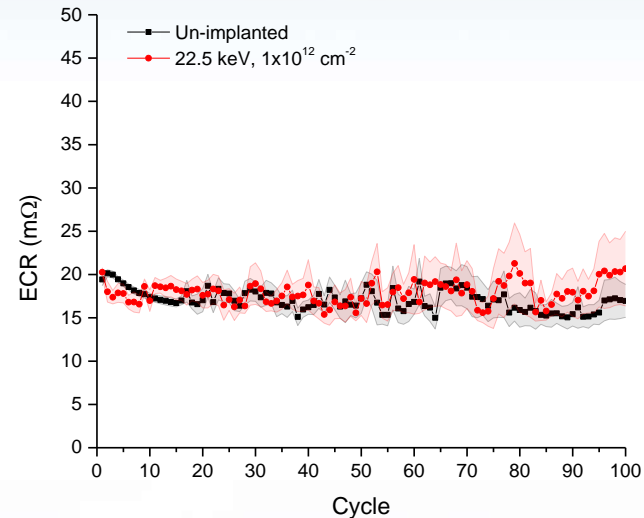
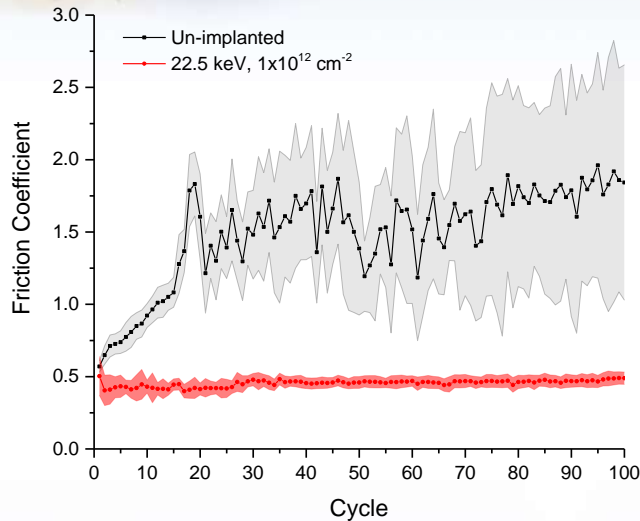
T.W. Scharf & S.V. Prasad, *Journal of Material Science* (2013) 48:511-531



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Electrical Contact Resistance and Wear Measurements

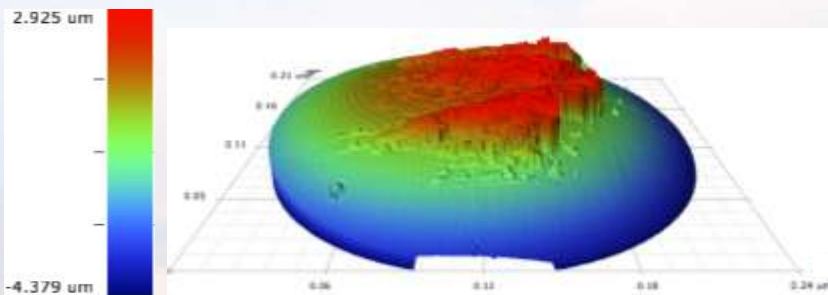
Collaborators: J-E Mogonye & S.V. Prasad



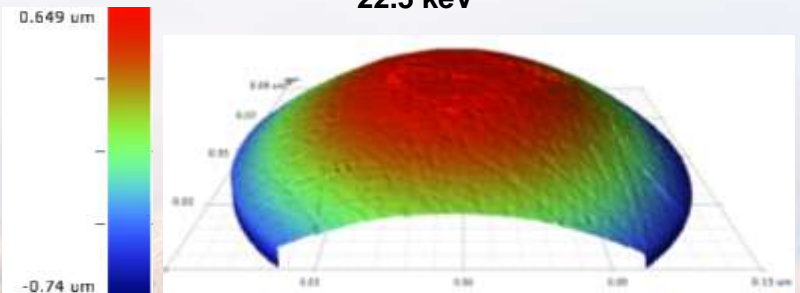
Friction is significantly reduced with ^3He implantation while maintaining ECR performance

Scanning white light interferometer topographical construction of riders after 100 cycles

Rider after 100 cycles against Un-implanted Au



Rider after 100 Cycles against Au implanted to $1 \text{E}12 \text{ cm}^{-2}$ @ 22.5 keV



Wear is significantly reduced with minimal effect in ECR



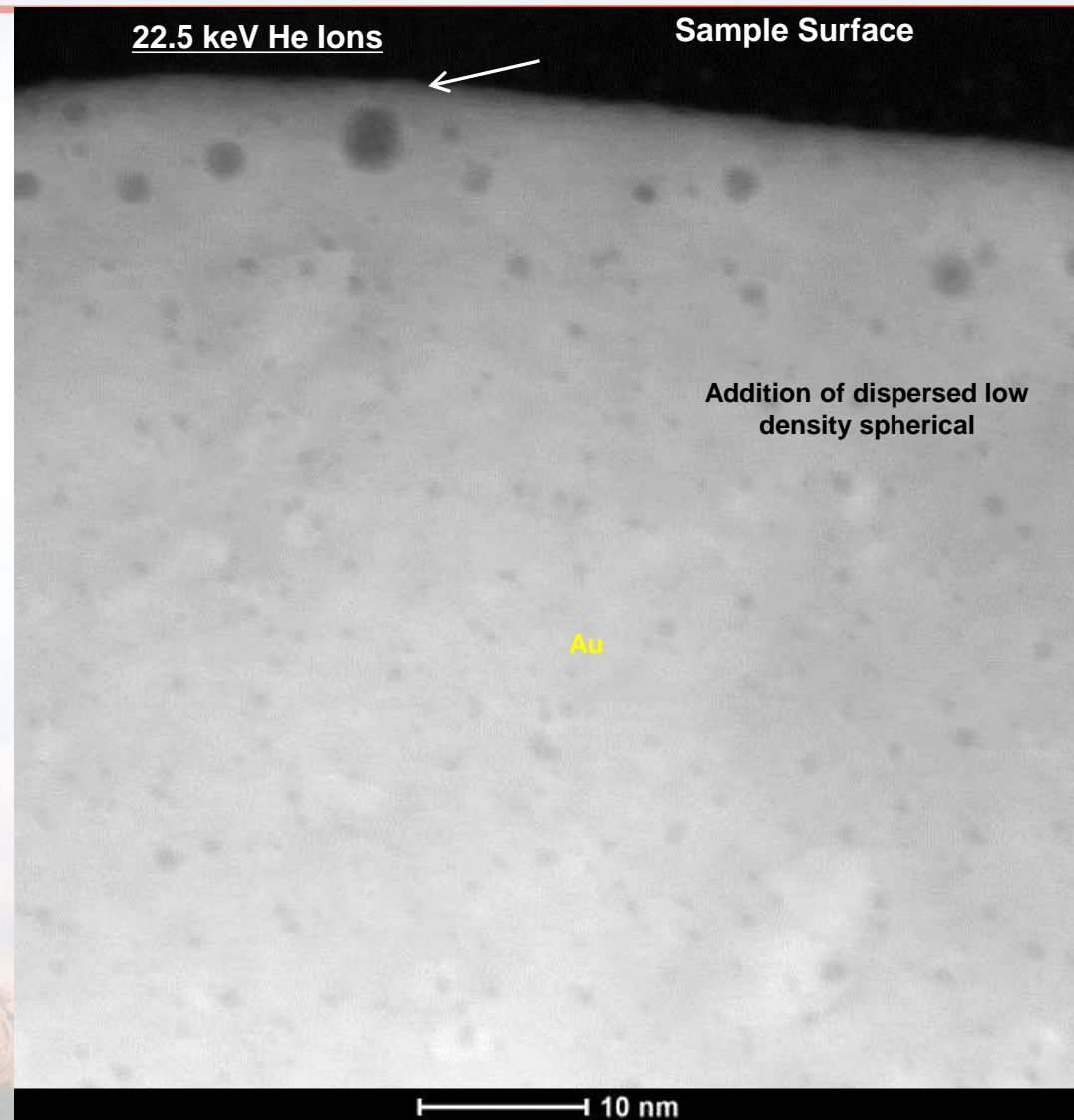
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Modeling and STEM of He Implantation

Collaborators: J-E Mogonye & S.V. Prasad

- Simulations: SRIM 2008
 - Monte-Carlo simulation of kinematic interaction based on empirical data fitted functions
 - Input variables of target material include density, AMU, and thickness.
 - Input variables of ions include AMU, energy, and angle of incidence.
 - Assumes isotropic material, thus no consideration for channeling effects
- AC-STEM used to observe the distribution of implanted bubbles
- Bubble locations are in good agreement with SRIM ion range predictions

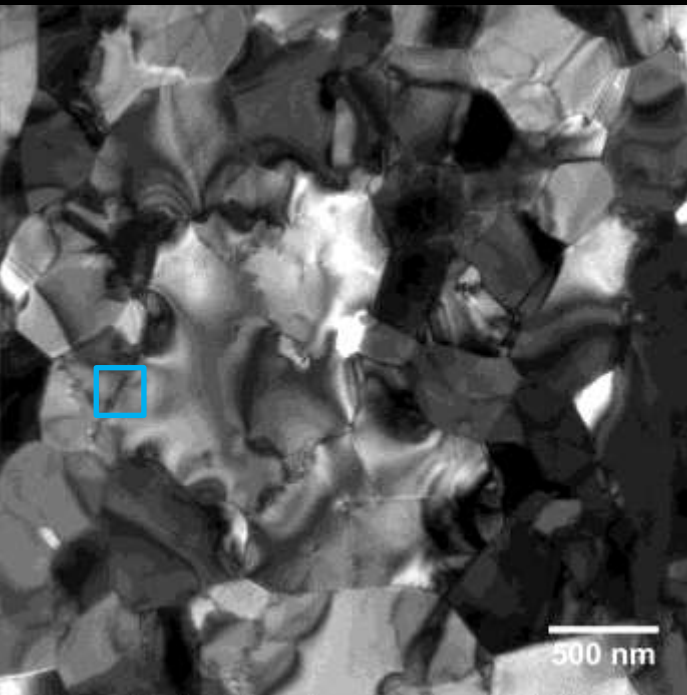
*He implantation result in small dispersed spherical structures assumed to be He bubbles.
Dispersion and depth can be tailored*



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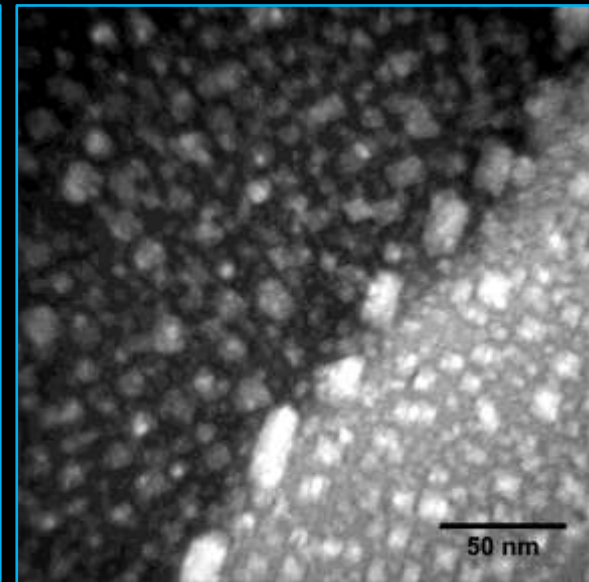
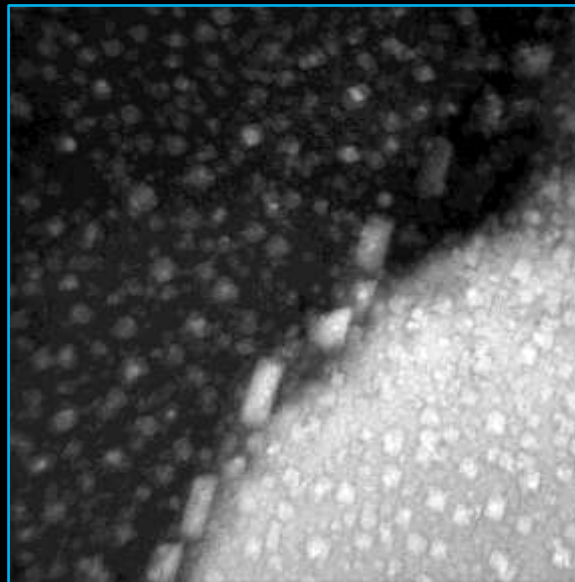
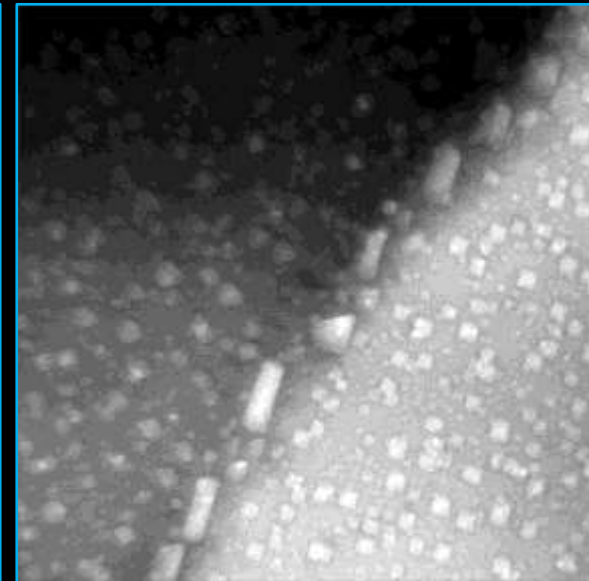
In situ Implantation

Collaborators: C. Chisholm & A. Minor



**Gold thin-film implanted
with 10keV He²⁺**

**Result: porous
microstructure**



Cumulative Effects of Ion Irradiation as a Function of Ion Energy and Au Particle Size

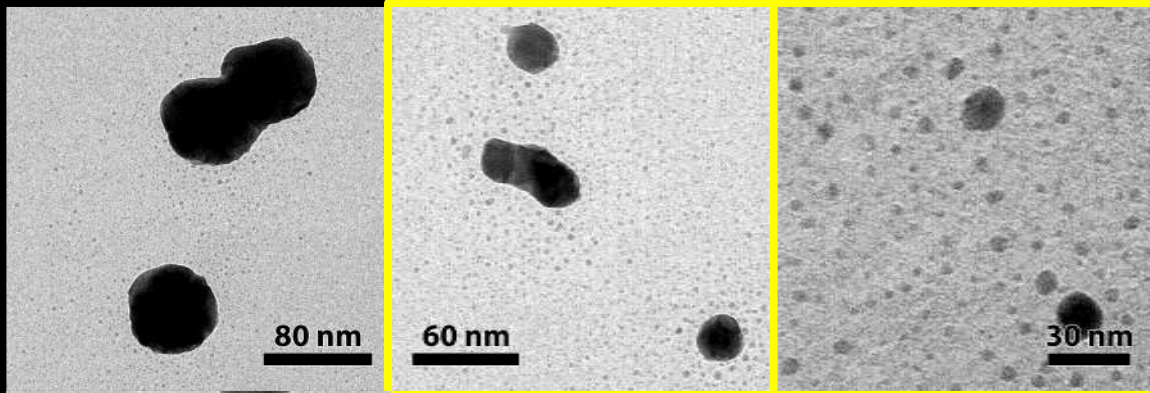
60 nm

20 nm

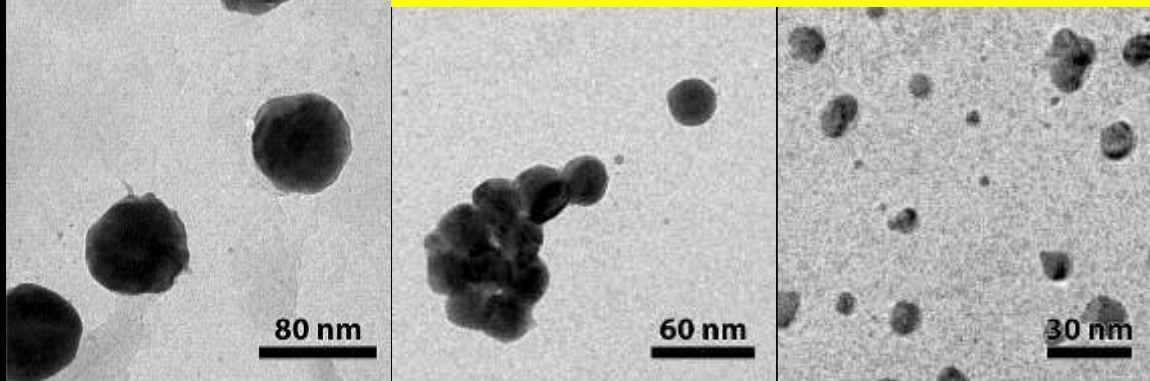
5 nm

Collaborator: D.C. Bufford

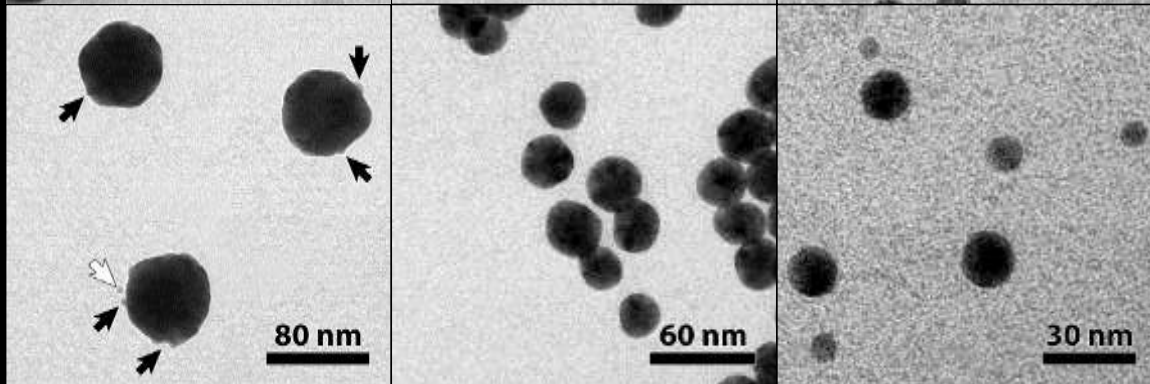
46 keV Au¹⁺
 $3.4 \times 10^{14} / \text{cm}^2$



2.8 MeV Au⁴⁺
 $4 \times 10^{13} / \text{cm}^2$



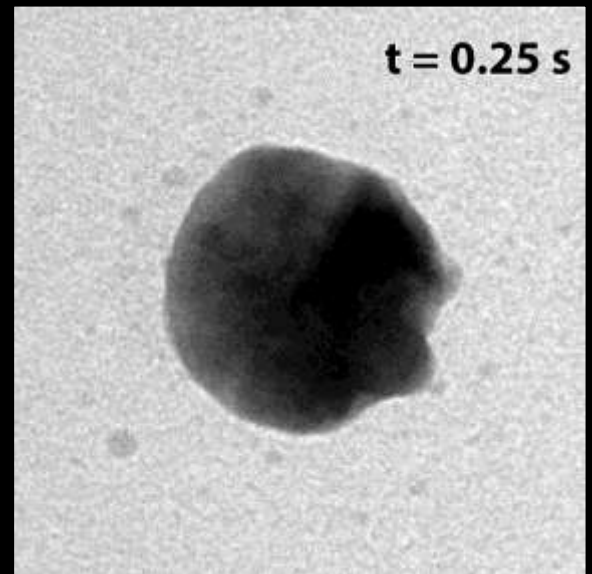
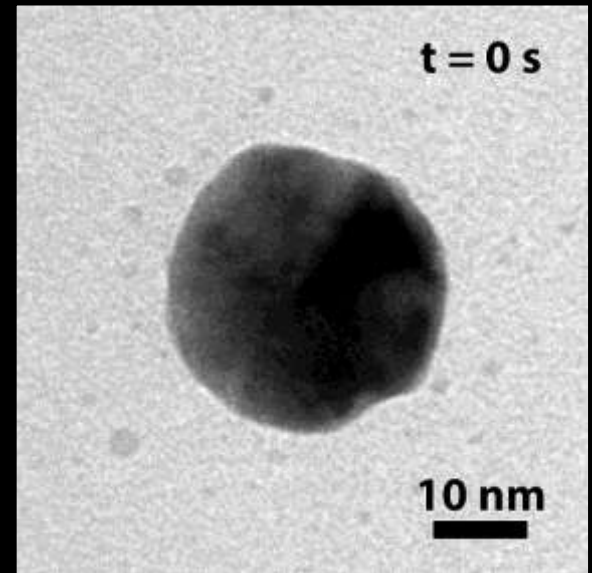
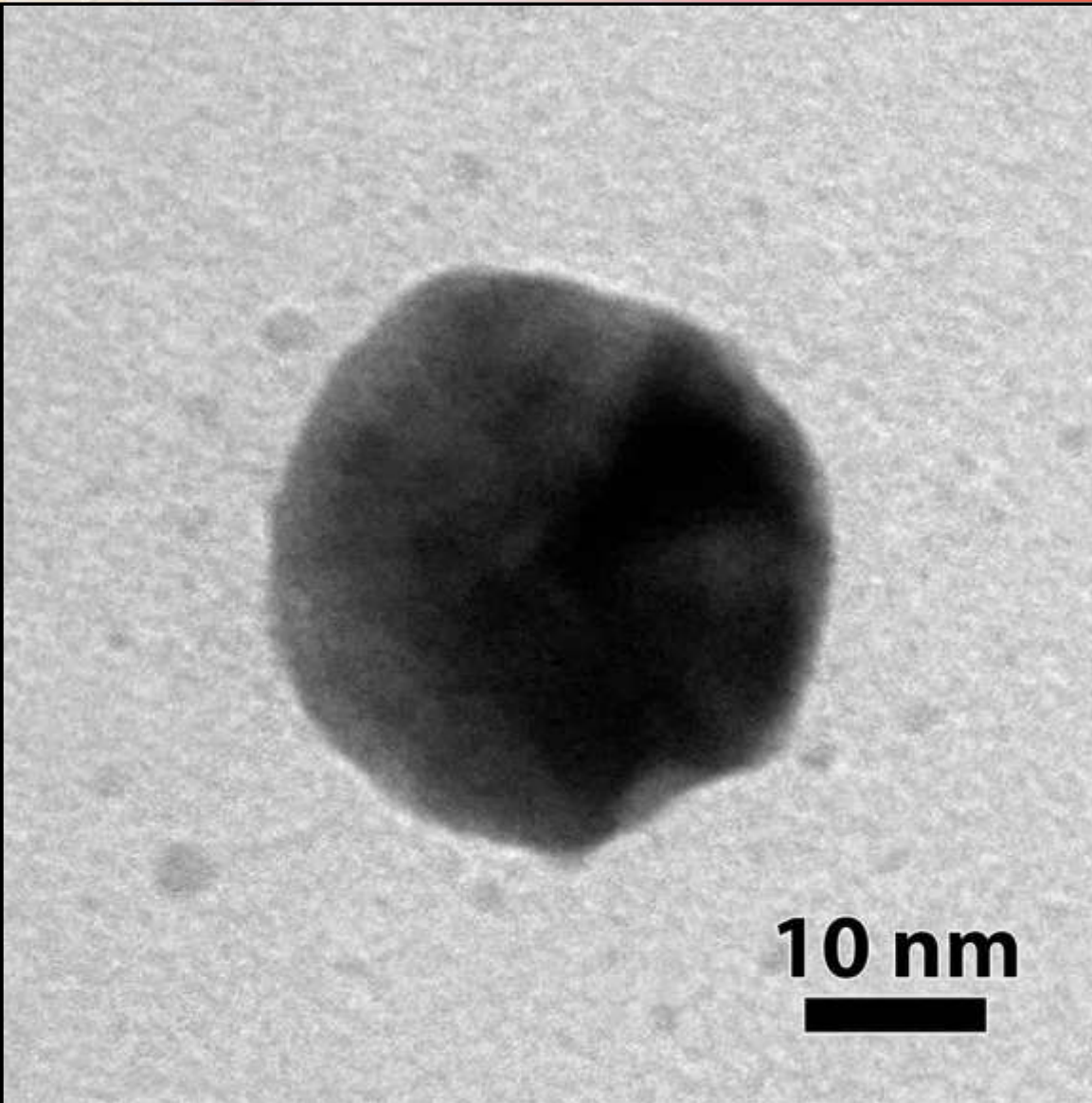
10 MeV Au⁸⁺
 $1.3 \times 10^{12} / \text{cm}^2$



Particle and ion energy dictate the ratio of sputtering, particle motion, particle agglomeration, and other active mechanisms

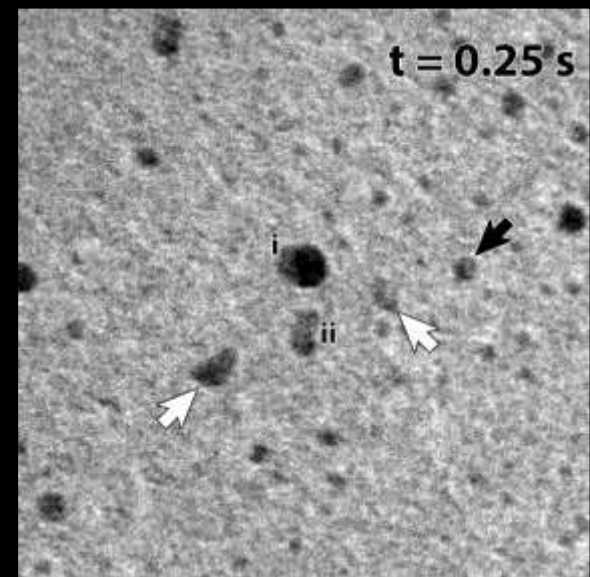
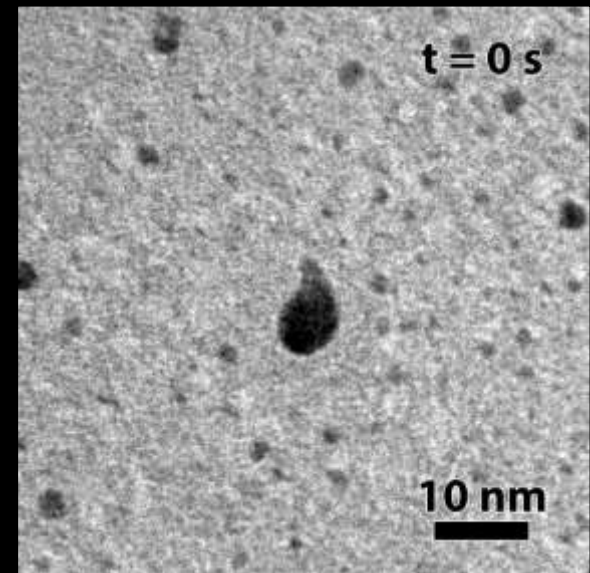
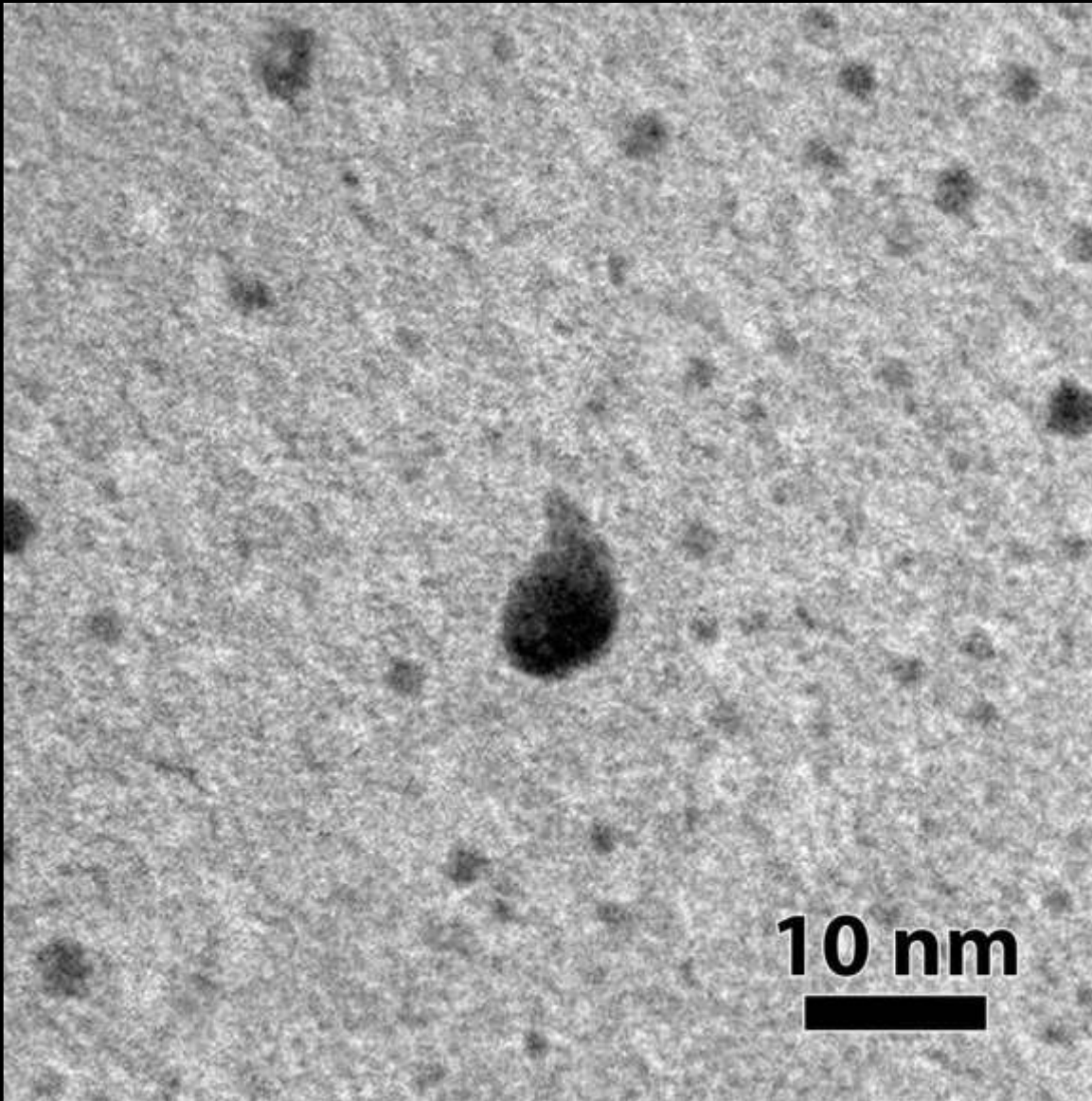
Single Ion Effects with 46 keV Au¹⁺ ions: 20 nm

Collaborator: D.C. Bufford



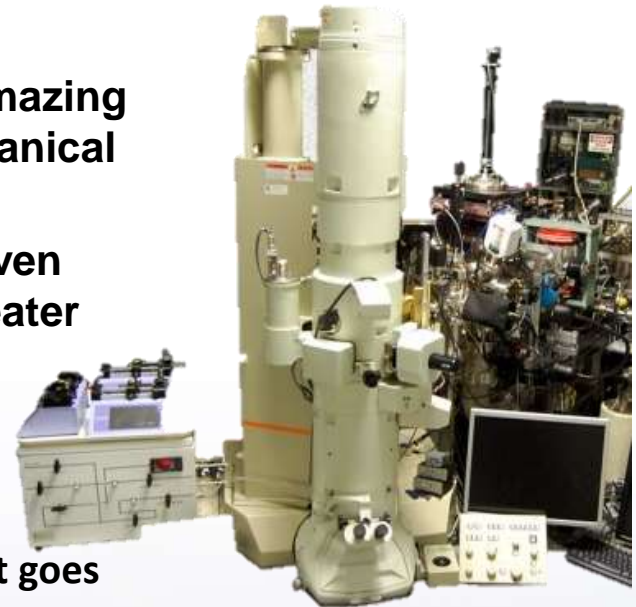
Single Ion Effects with 46 keV Au¹⁺ ions: 5 nm

Collaborator: D.C. Bufford



Summary

- Ion Beam Modification (IBM) has been shown to be an amazing tool in tailoring the thermal, optical, electrical, and mechanical properties of many material systems
- New capabilities and juxtaposing of techniques permit even greater insight into the governing physics permitting greater control of properties and performance.
- Sandia's I³TEM is one of a few in the world
 - In situ* irradiation from H to Au
 - In situ* gas implantation
 - combinations of in-situ techniques
- We are still a long way away from a complete design process that goes from fundamental physics to system engineering



Collaborators:

- IBL: [D.C. Bufford](#), [D. Buller](#), [C. Chisholm](#), B.G. Clark, B.L. Doyle, [S. H. Pratt](#), & [M.T. Marshall](#)
- Sandia: B. Boyce, T.J. Boyle, [P.J. Cappillino](#), [J.A. Scott](#), B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, E. Carnes, J. Brinker, D. Sasaki, [J.A. Sharon](#), T. Nenoff, W.M. Mook
- External: A. Minor, L.R. Parent, I. Arslan, H. Bei, E.P. George, P. Hosemann, D. Gross, J. Kacher, & I.M. Robertson