Real Time Observation of He Implantation, High-Energy Si Irradiation, and Self-Ion Irradiation of Nanostrucutred Au

SAND2014-4268C

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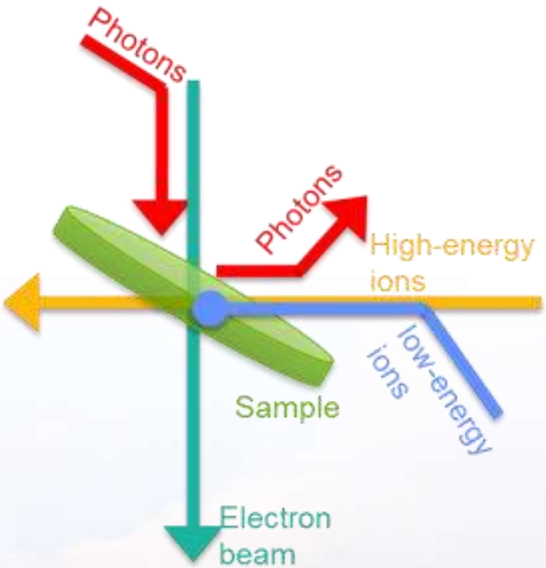
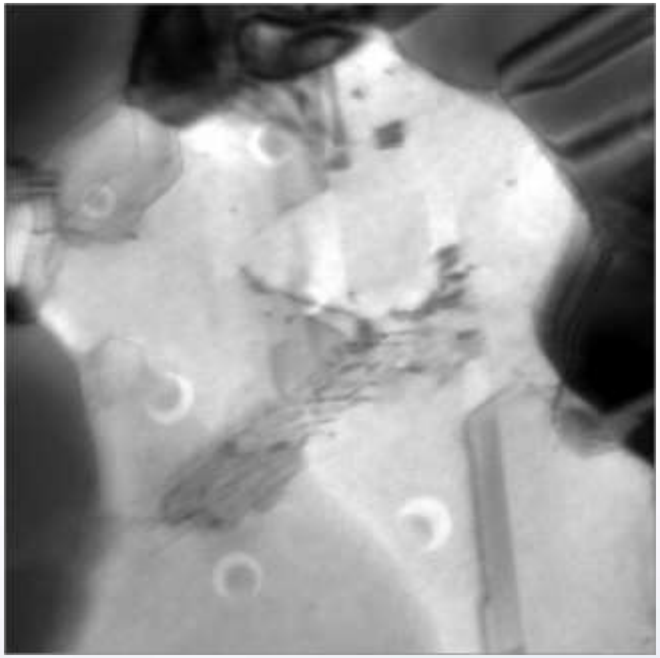
SNL, UCB, and LBNL

May 29, 2014





***In situ* Ion Irradiation TEM Facility:**

1) **Real time nanoscale observation of single ion strikes and subsequent structural evolution**
2) **Provides insight into a range of extreme environment combinations**

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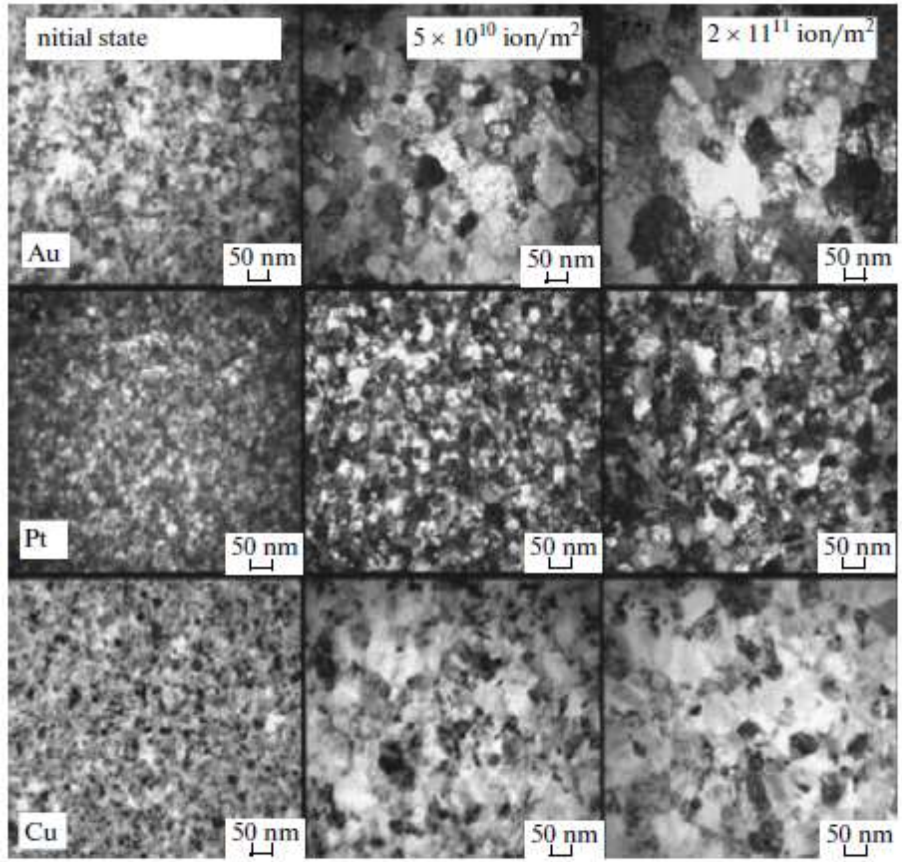
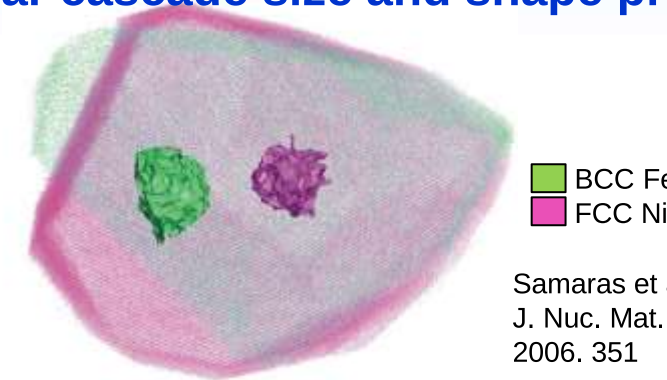
Radiation Tolerance from Nanograined Metals

Variation in radiation tolerances



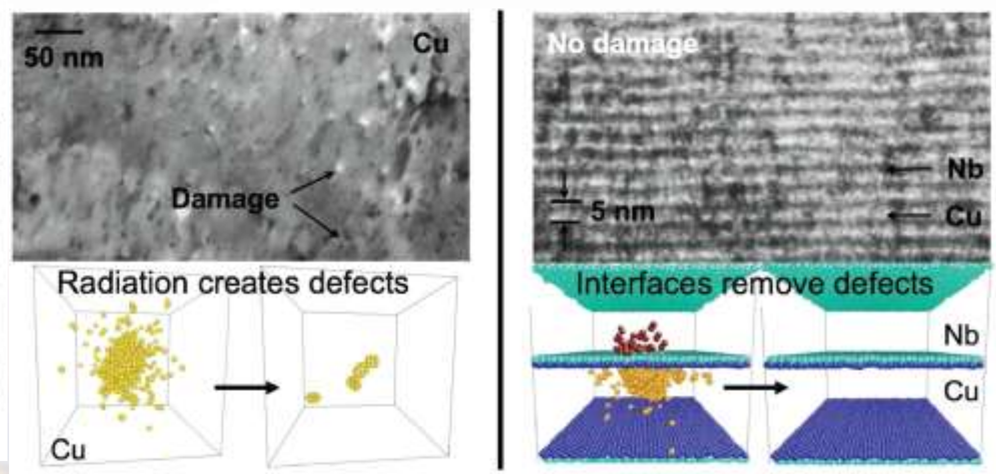
Kaomi et al., JAP: 2008. 104 073525

Similar cascade size and shape predicted



Samaras et al.,
J. Nuc. Mat.:
2006. 351

Nanolamellars are radiation tolerant



Demkowicz et al., MRS Bulletin: 2010. 35

To a first order mean grain size comparison, these reports appear conflicting.
Not necessarily the case if initial microstructural details and associated properties are considered

History of *In situ* Ion Irradiation TEM

Courtesy of: J. Hinks

1930s… | 1960s | 1970s | 1980s | 1990s | 2000s | 2010s

1931
The invention of the TEM

1961
O^- emission reported from a TEM filament by Pashley, Presland, and Meneter at TI Labs, Cambridge, UK

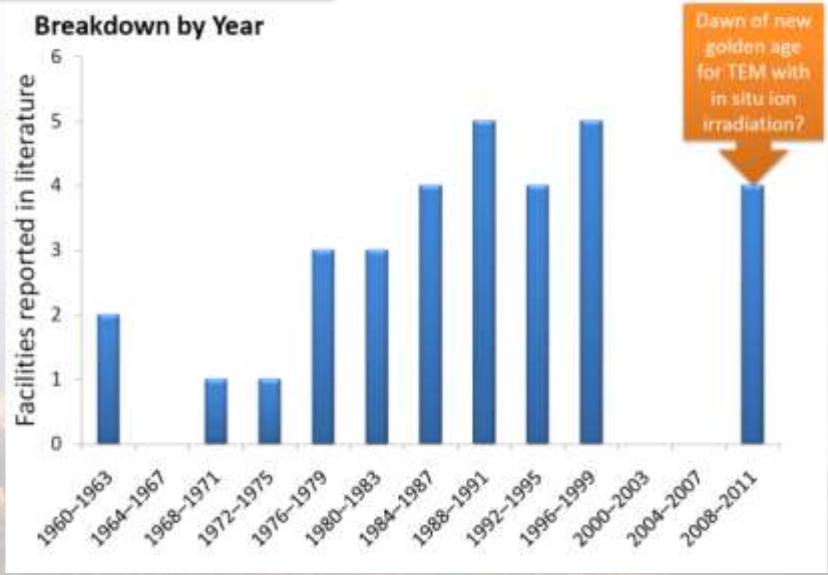
1968
First TEM beamline combination by Thackery, Nelson, and Sansom at AERE Harwell, UK

1976
First HVEM with ion irradiation at UVA, USA

1978
First in-situ ion irradiation experiments at ANL

1990s
First dual beam system developed at JAERI and NIMS, Japan

Workshop on Ion Irradiation TEM
Huddersfield, UK (2008)
Albuquerque, USA (2011)
Saporro, Japan (2013)

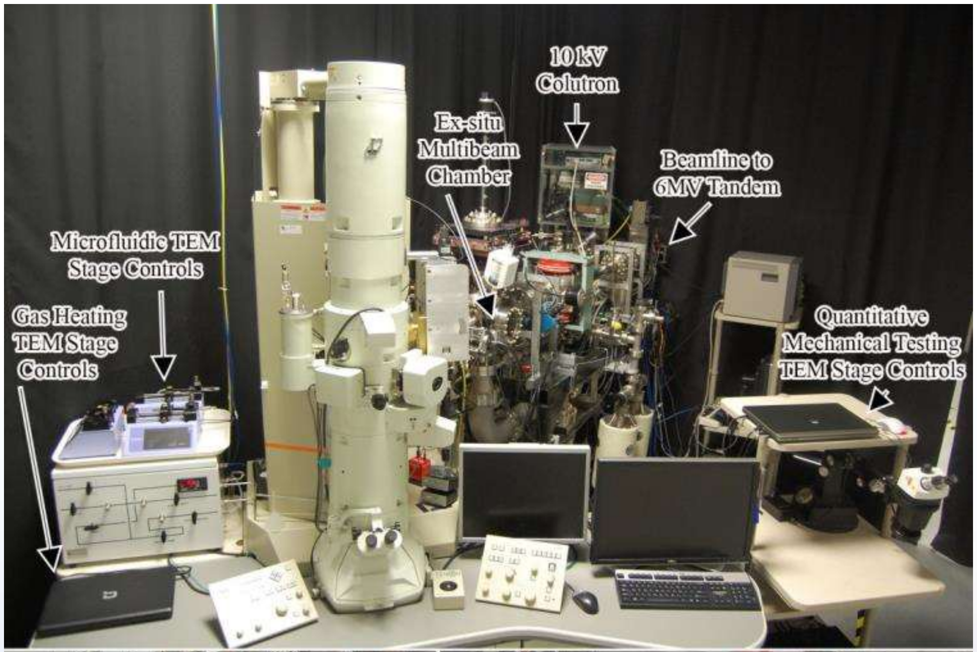


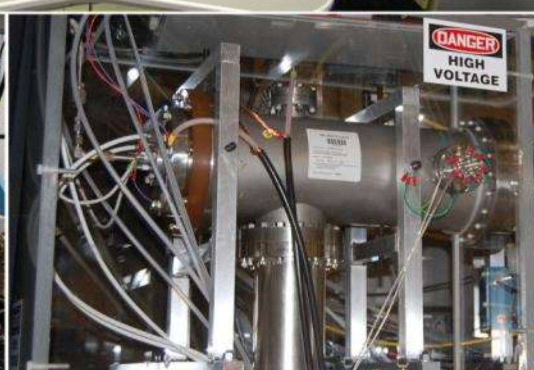
"The direct observation of ion damage in the electron microscope thus represents a powerful means of studying radiation damage"

D.W. Pashley and A.E.B. Presland Phil Mag. 6(68) 1961 p. 1003

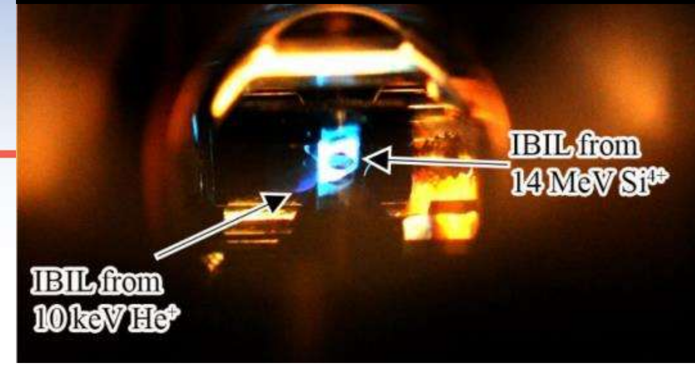
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

10 kV Colutron - 200 kV TEM - 6 MV Tandem



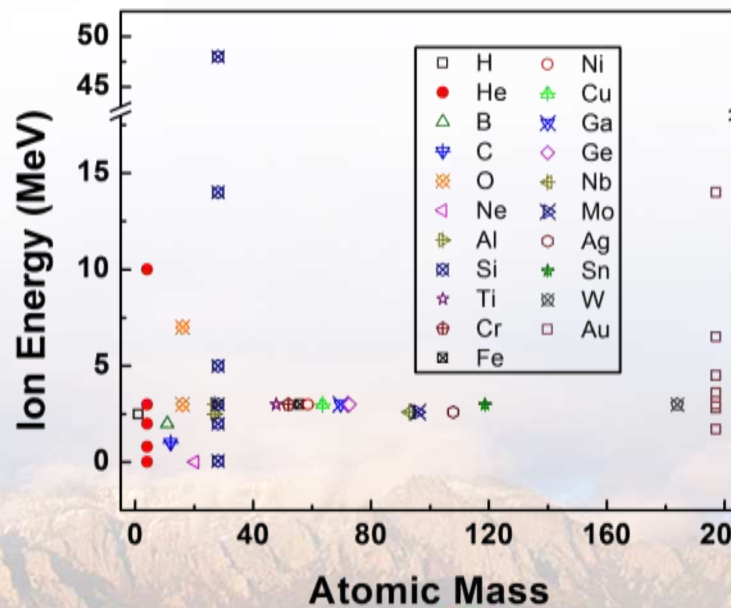


IBIL from a quartz stage inside the TEM



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

Ion species & energy introduced into the TEM



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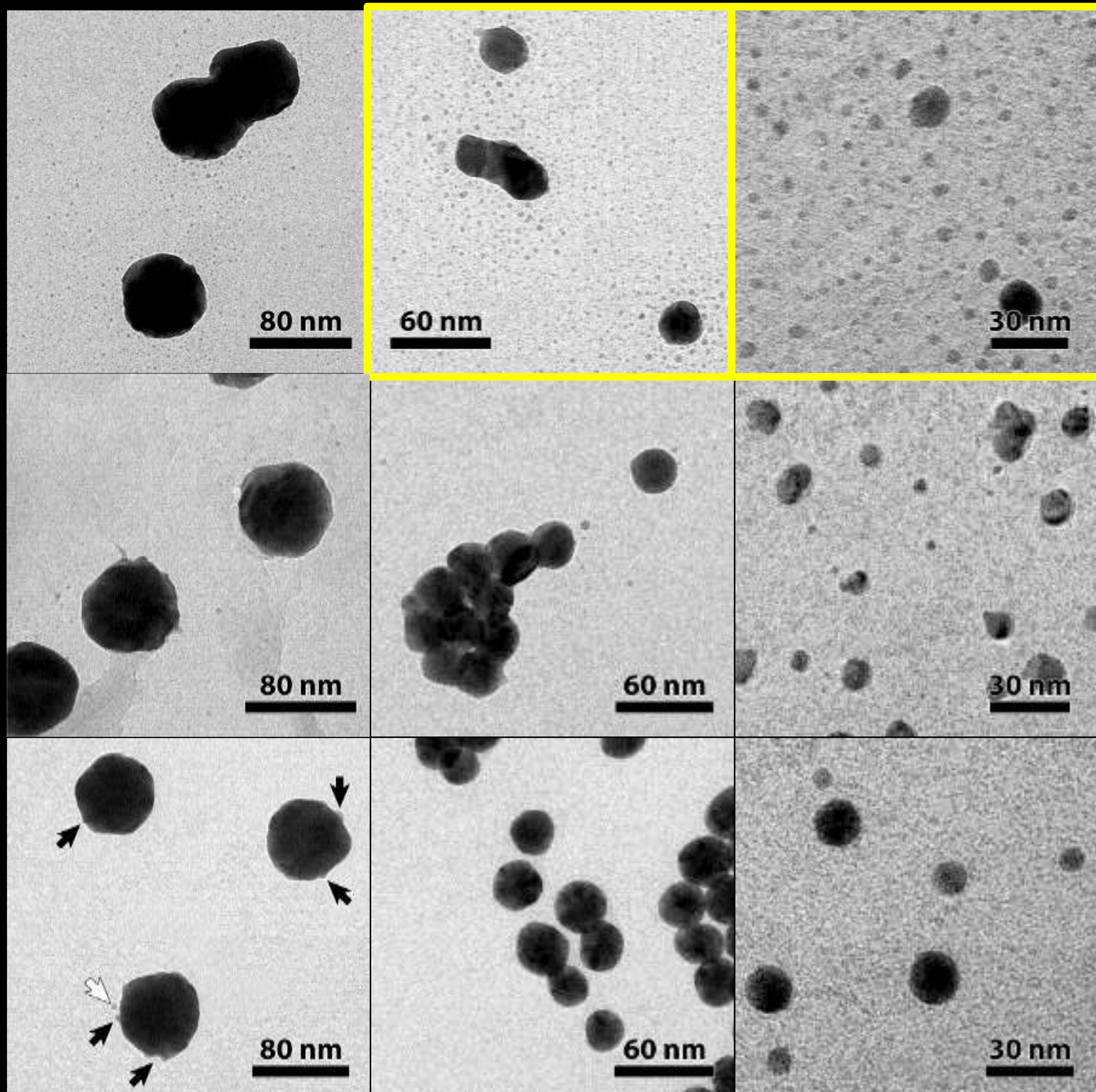
Cumulative Effects of Ion Irradiation as a Function of Ion Energy and Au Particle Size

60 nm | **20 nm** | **5 nm**

46 keV Au^{1-}
3.4×10^{14} /cm^2

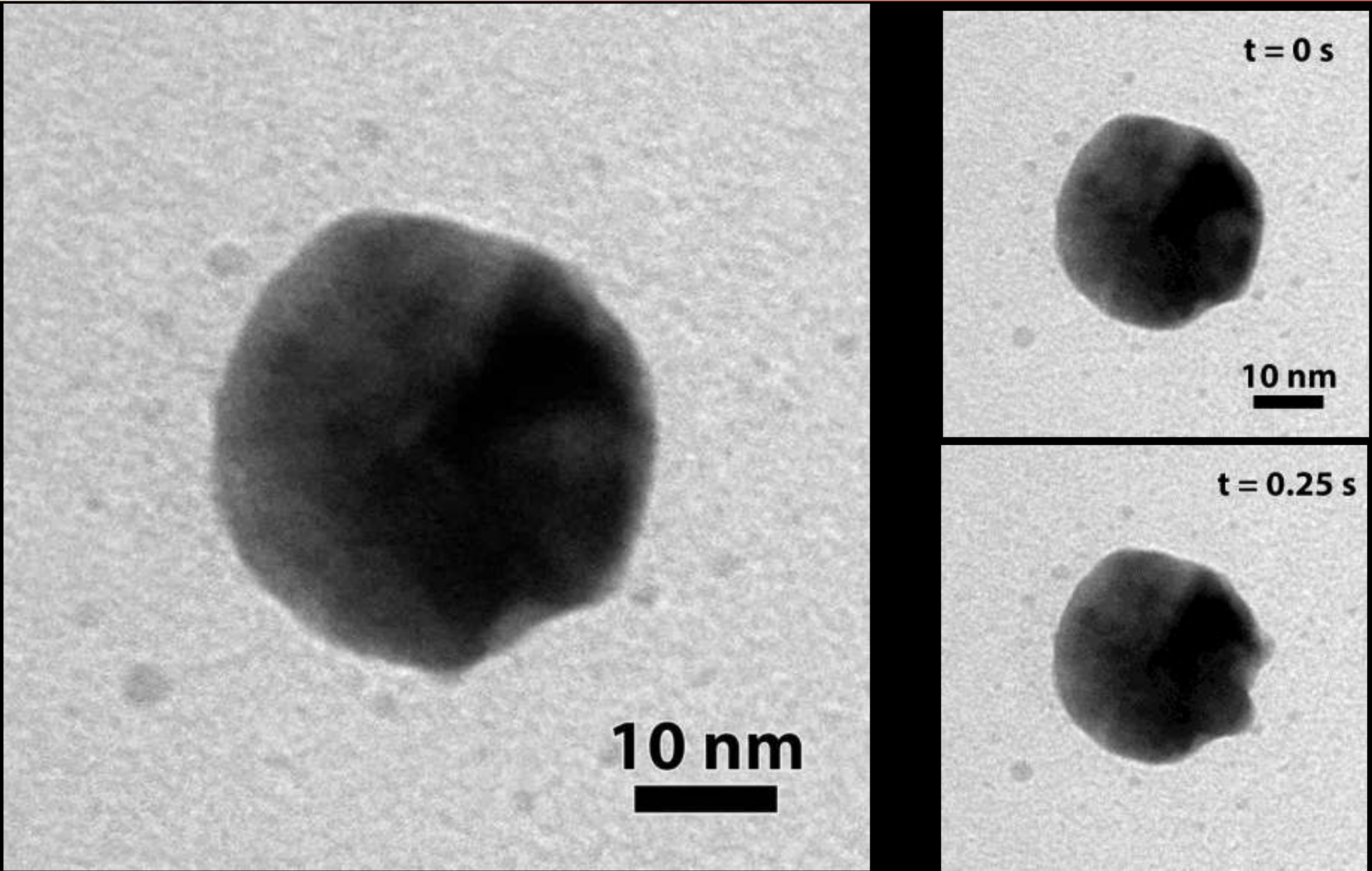
2.8 MeV Au^{4+}
4×10^{13} /cm^2

10 MeV Au^{8+}
1.3×10^{12} /cm^2

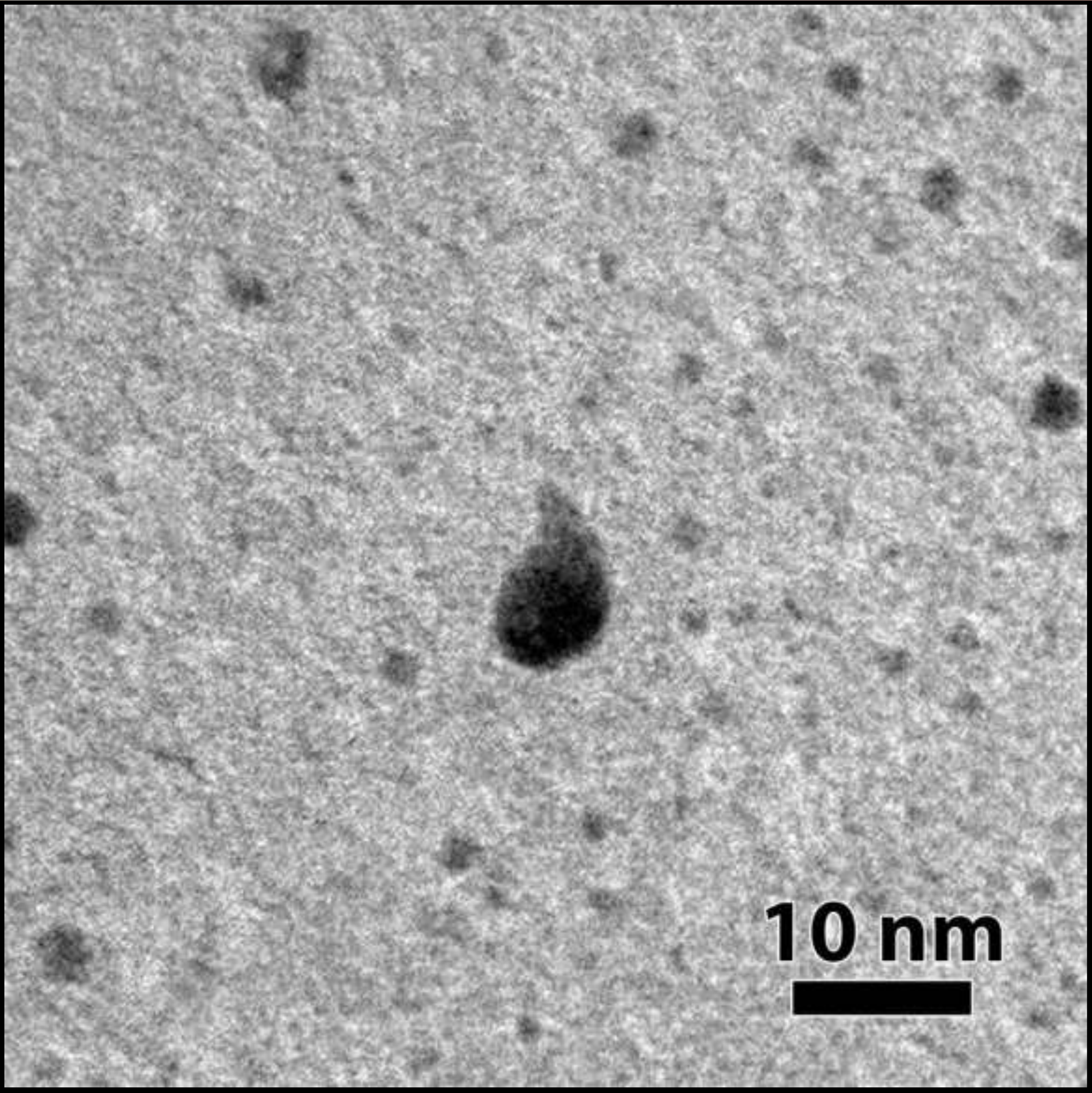


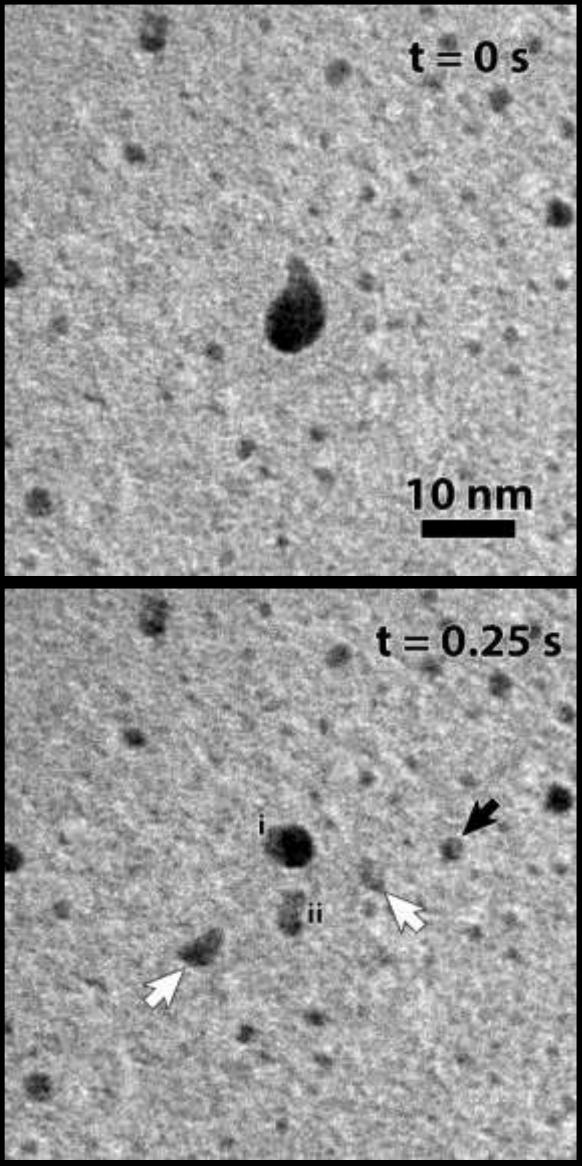
Particle and ion energy dictate the ratio of sputtering, particle motion, particle agglomeration, and other active mechanisms

Single Ion Effects with 46 keV Au^{1-} ions: 20 nm

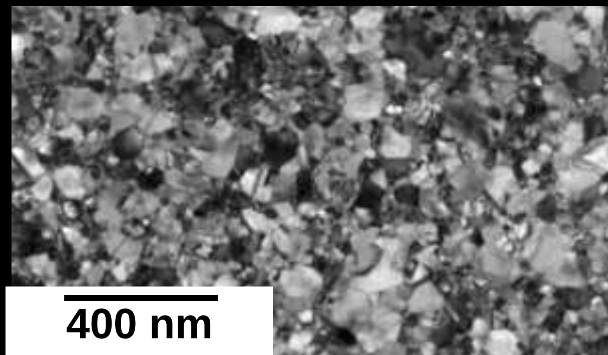


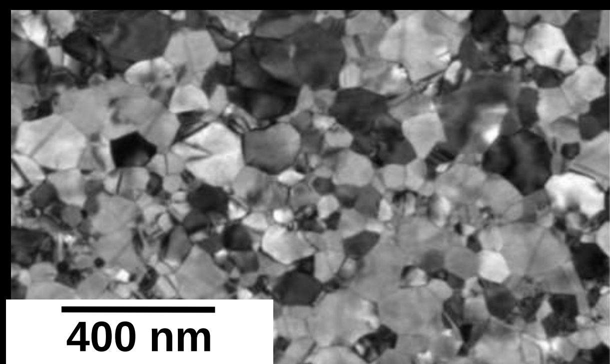
Single Ion Effects with 46 keV Au^{1-} ions: 5 nm

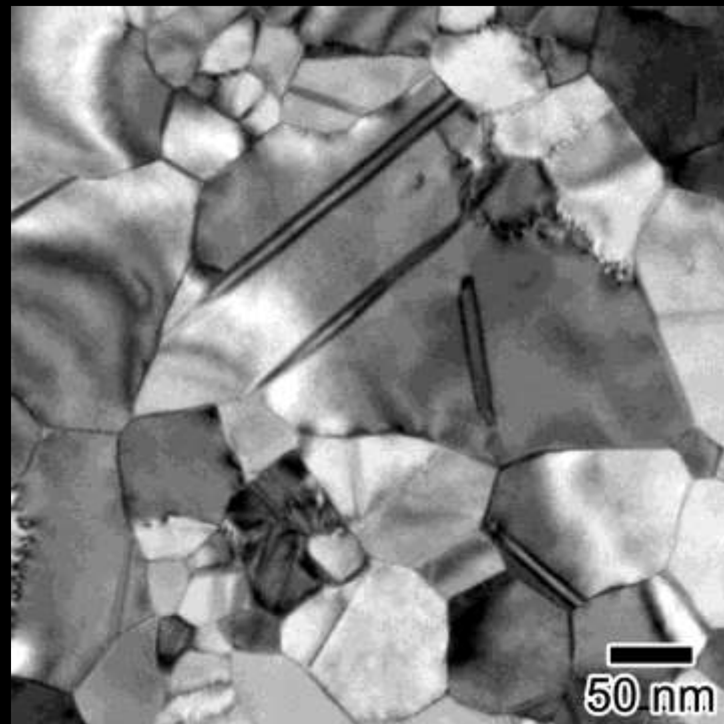




Au Microstructure Tailored through Vacuum Annealing





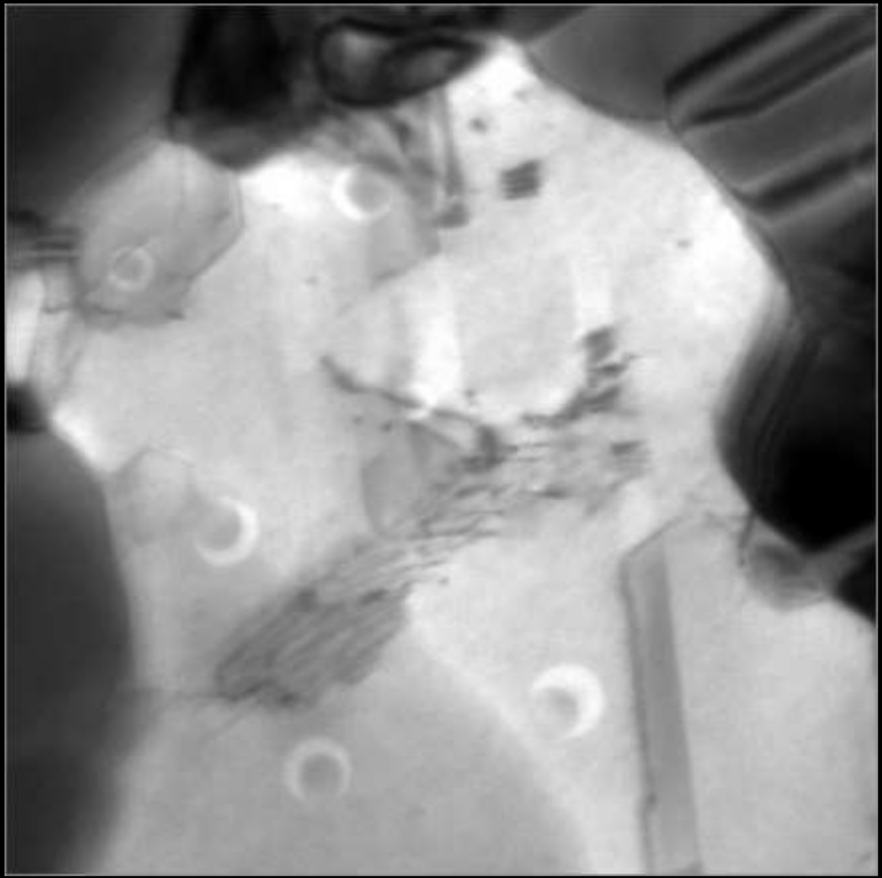


Well annealed free-standing PVD Au thin films provide an excellent model system to investigate the effects of ion beam parameters of defect structures formed from single ion effects.

Metal	Nickel	Copper	Gold
Stacking fault energy	125 mJ/m^2	45 mJ/m^2	32 mJ/m^2
Homologous temperature	0.33	0.42	0.43
Vacancy diffusivity	4.8×10^{-16} cm^2/s	1.2×10^{-11} cm^2/s	4.6×10^{-10} cm^2/s
Normalized average grain boundary area	98	42	25
Oxide layer	Yes	Yes	No

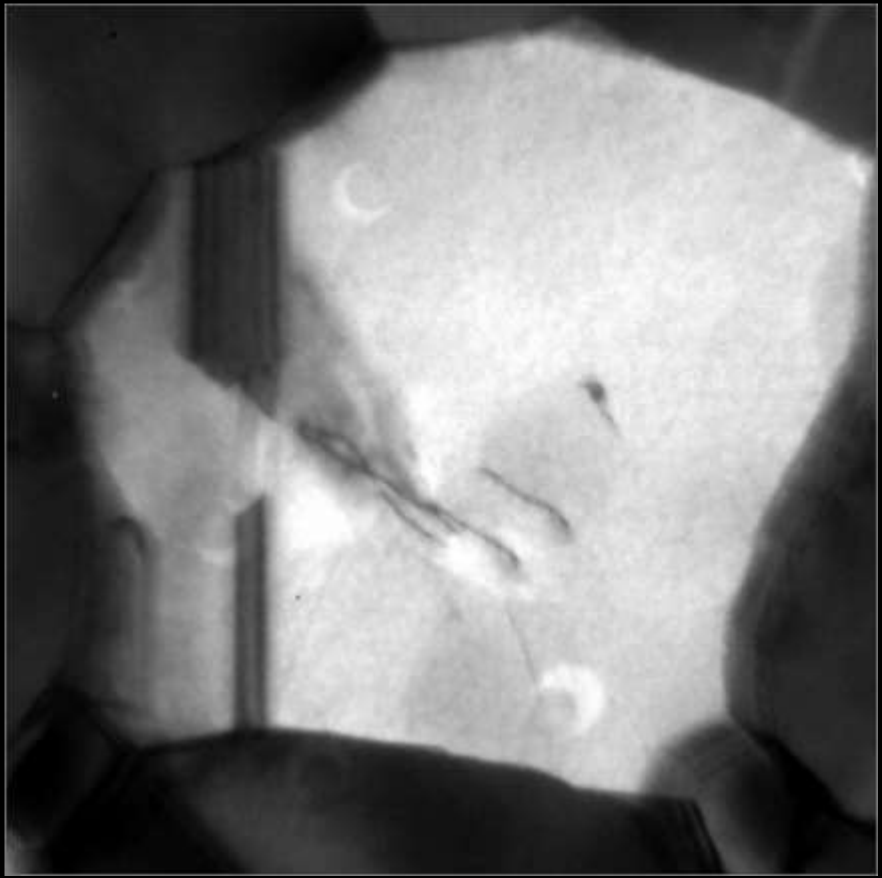
Greater than Two Orders of Magnitude Control in Dose Rate

7.9×10^9 ions/cm^2/s

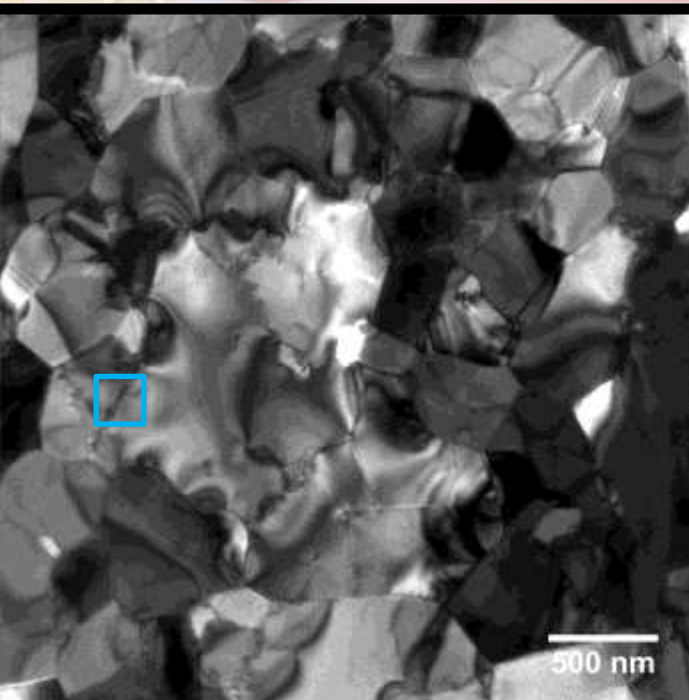


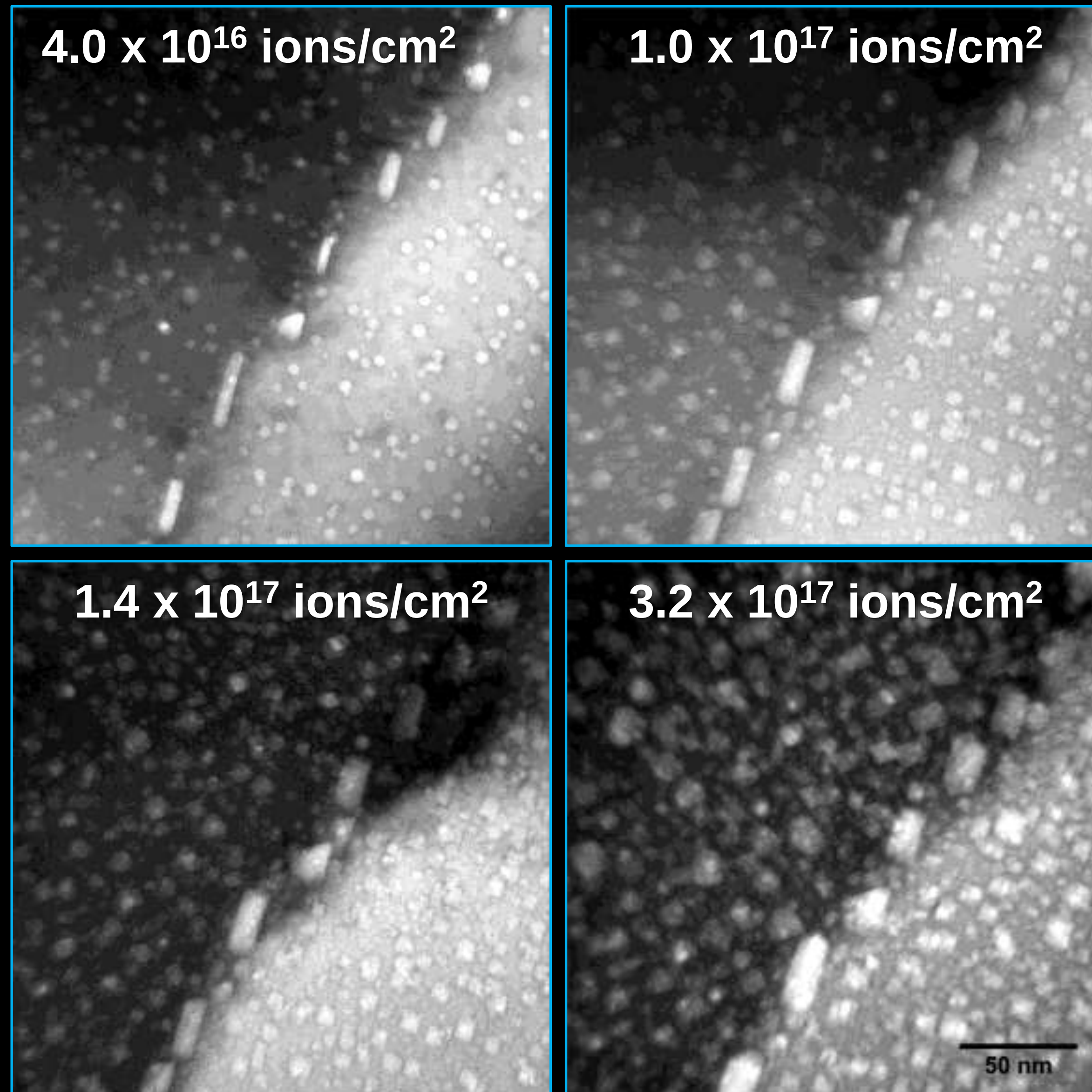
VS

6.7×10^7 ions/cm^2/s



In situ Implantation

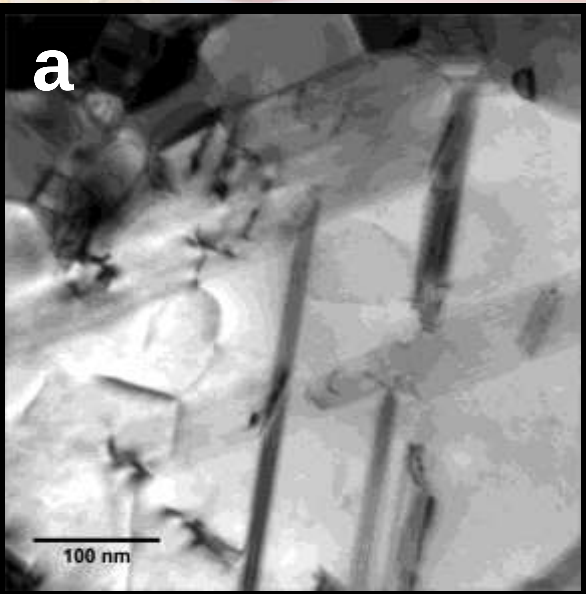


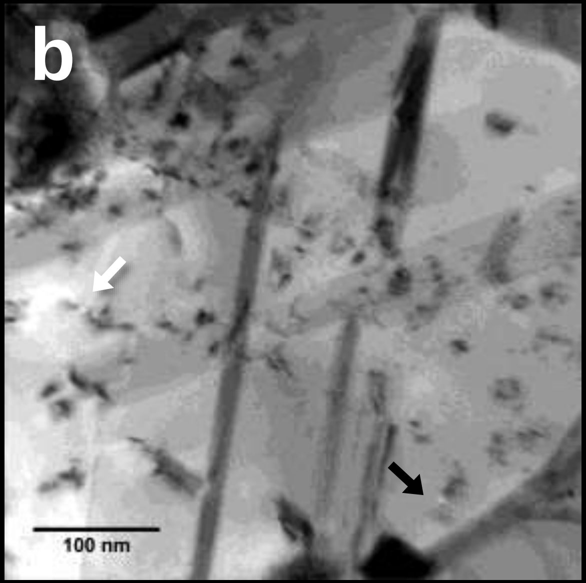


Gold thin-film implanted with 10keV He^{1+}

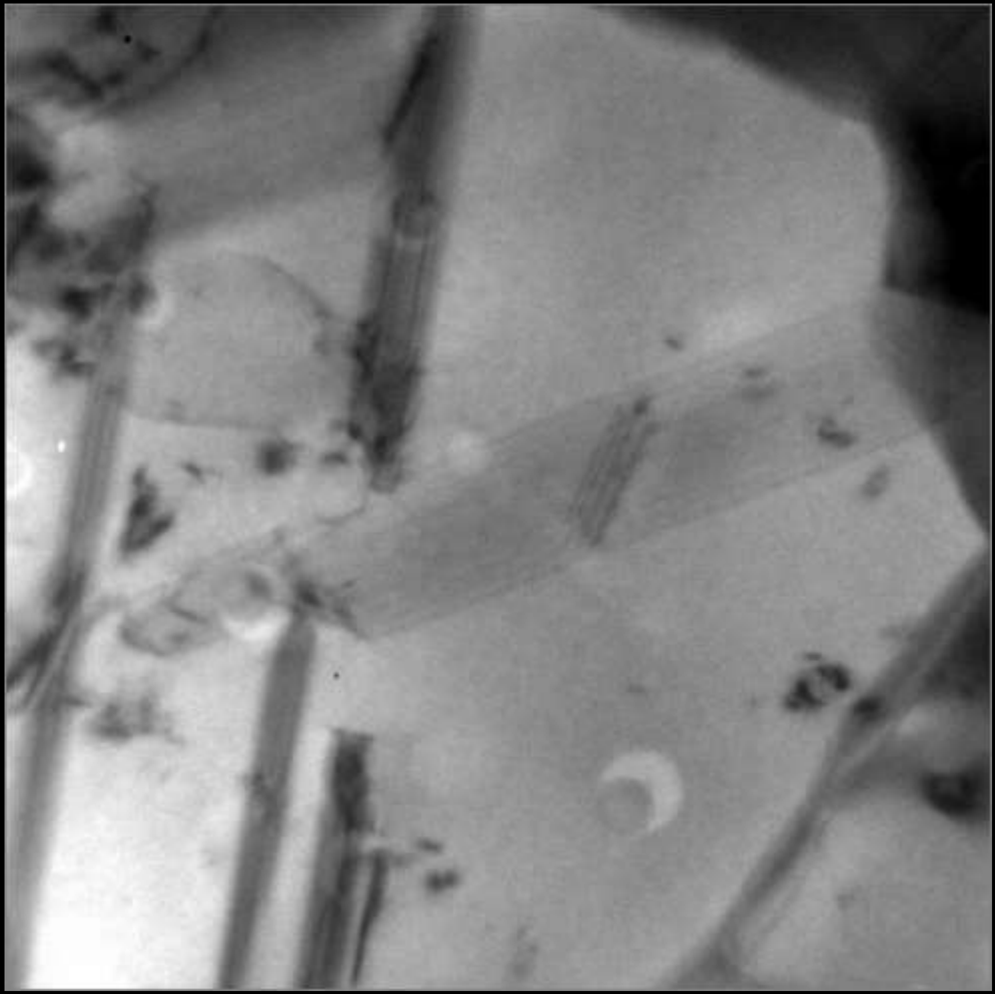
Result: porous microstructure

In situ Concurrent Implantation & Irradiation





He^{1+} implantation and Au^{4+} irradiation of a gold thin film



Single Ion Strikes During Concurrent Irradiation: Nucleation of Helium Cavities

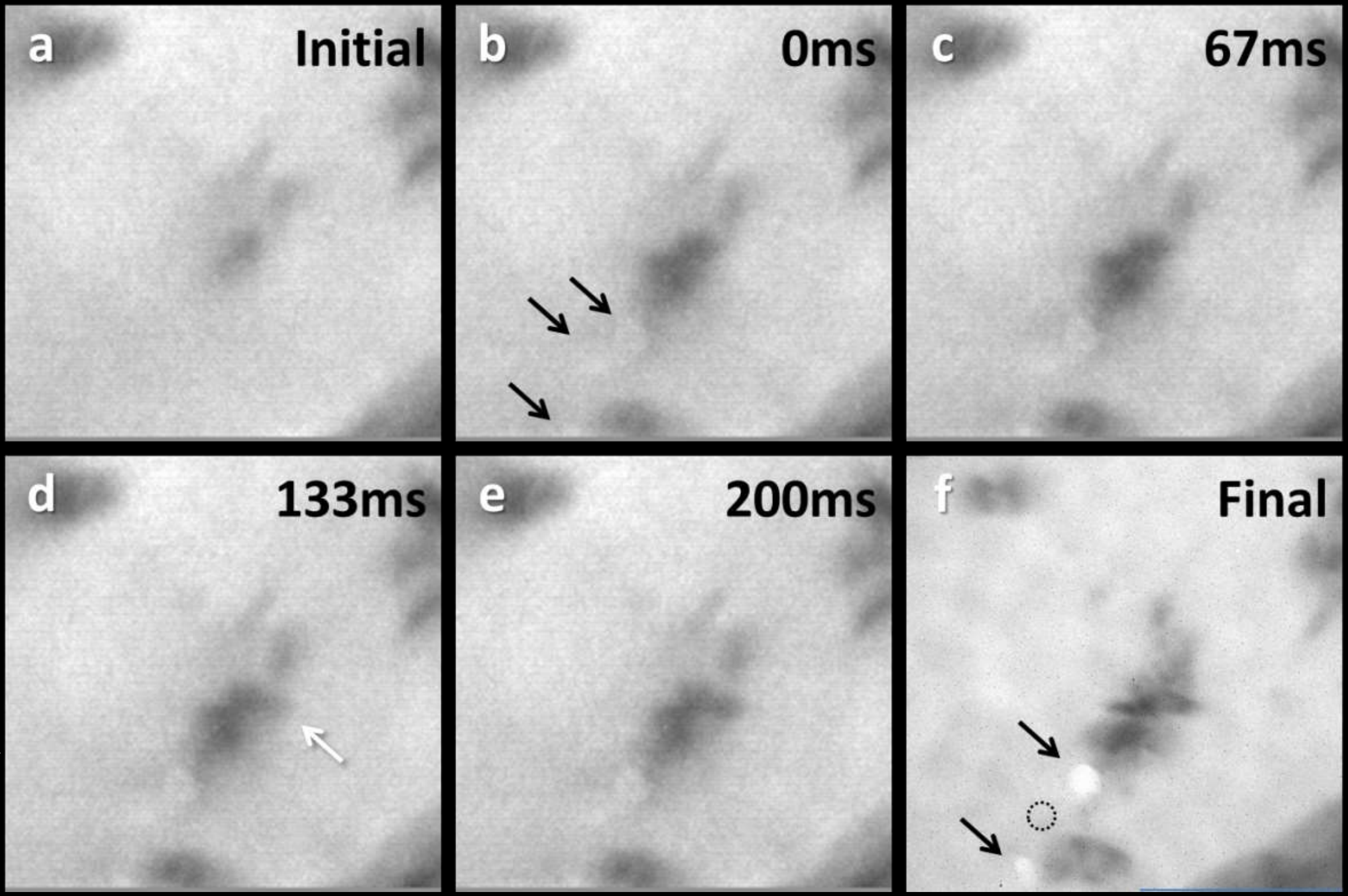
a) Initial microstructure

b) Cascade: Creation of dislocation loops, vacancy clusters, and three cavities

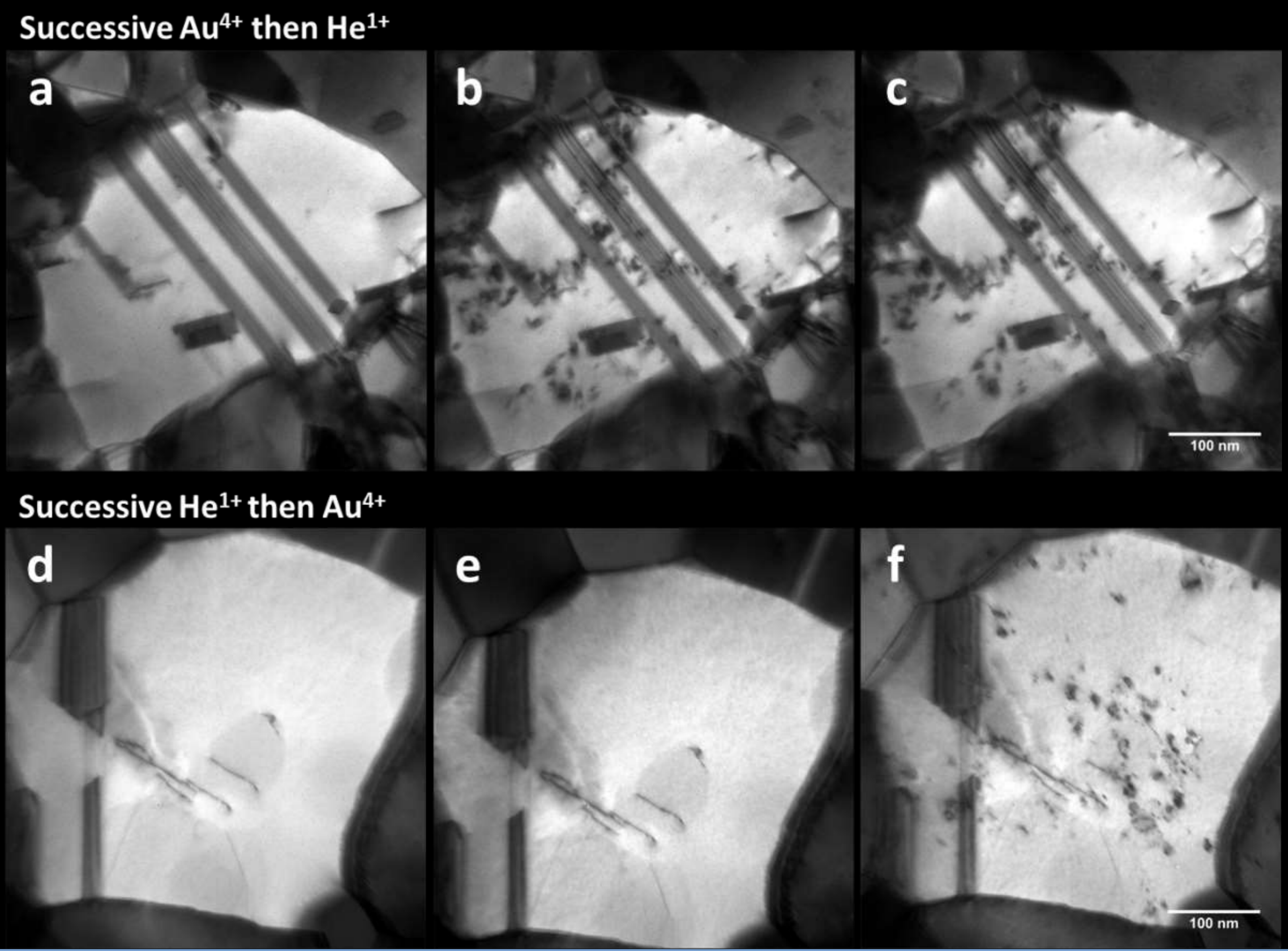
d) Cascade damage still evolving

e) Apparent stability

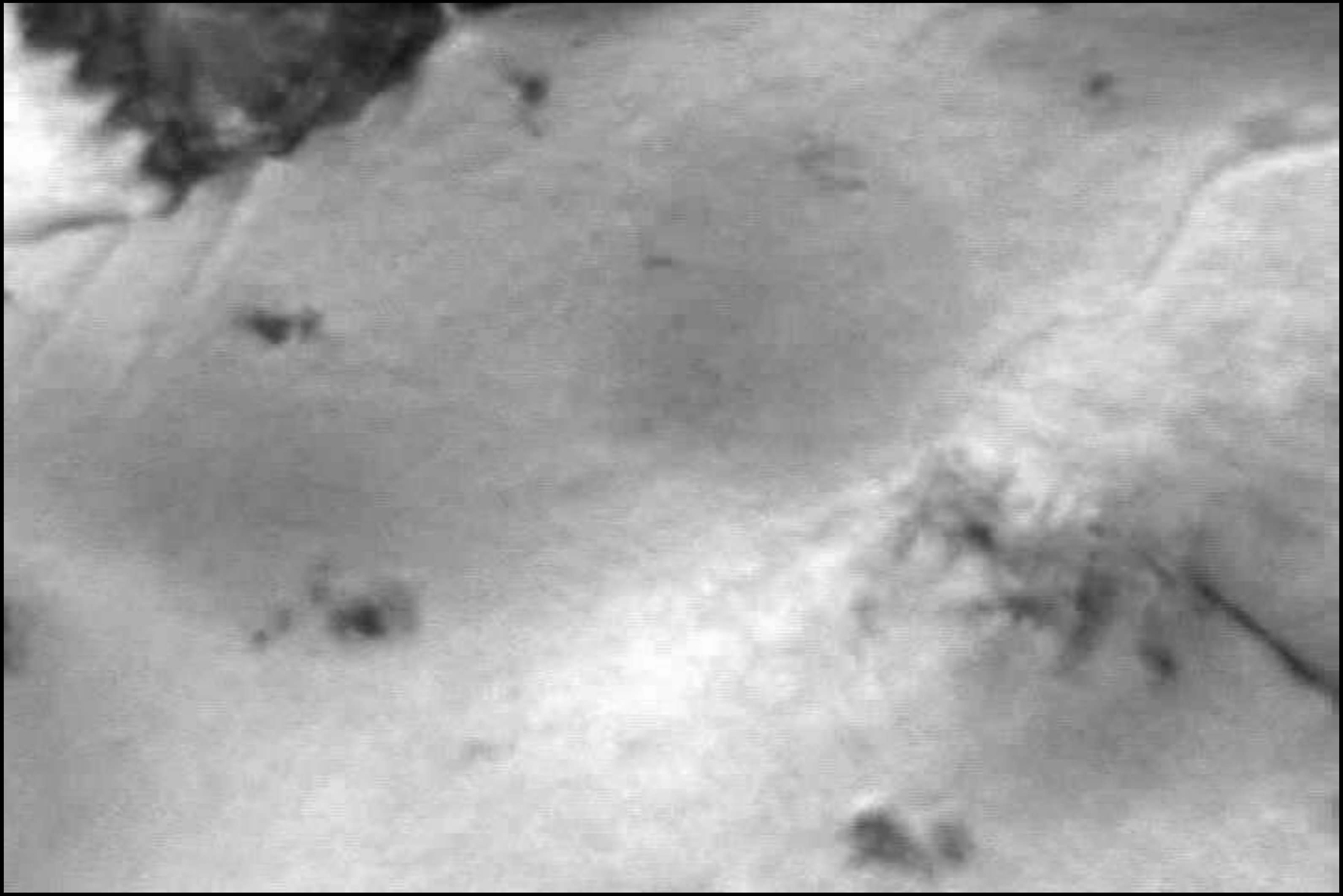
f) Final microstructure: Only two remaining cavities



In situ Successive Implantation & Irradiation



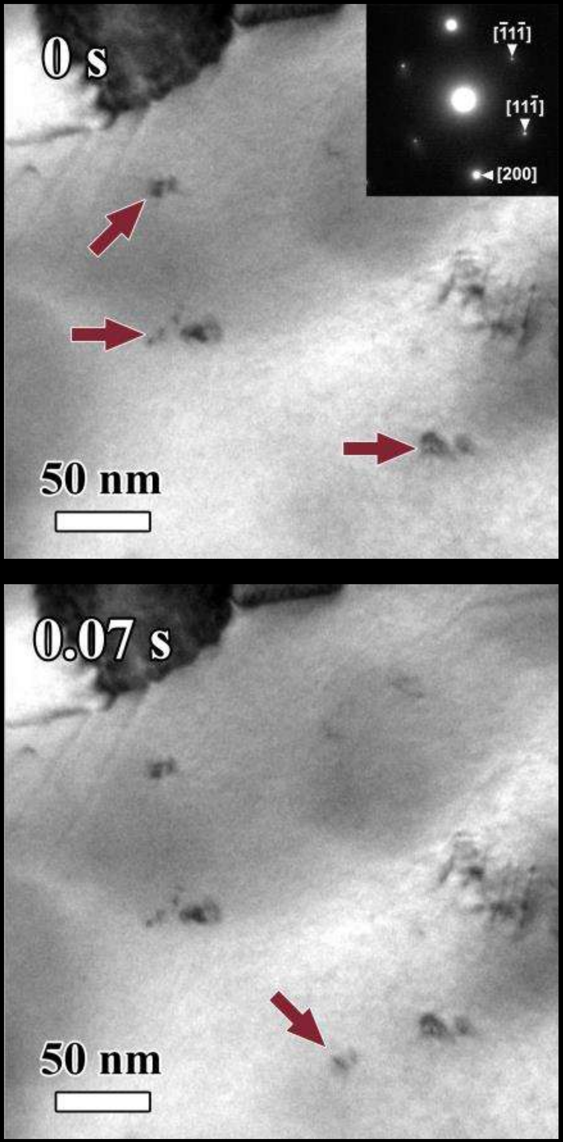
48 MeV Si into Au Thin Foil



The majority of expected Si ion strikes are not observed. Those observed result in significantly smaller defect structures.

Speed up 10x

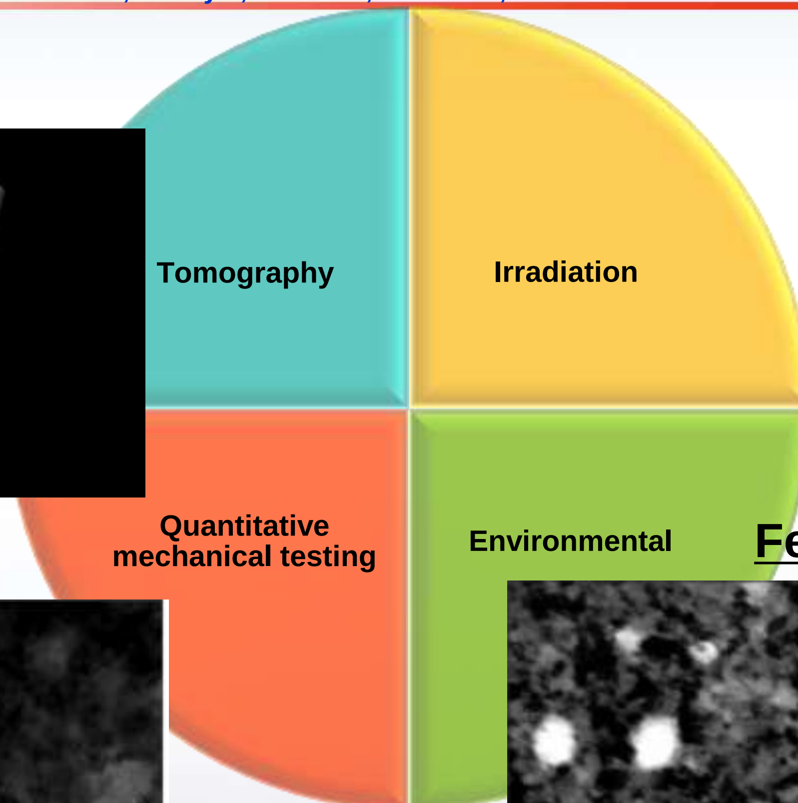
48 MeV Si into Au Thin Foil



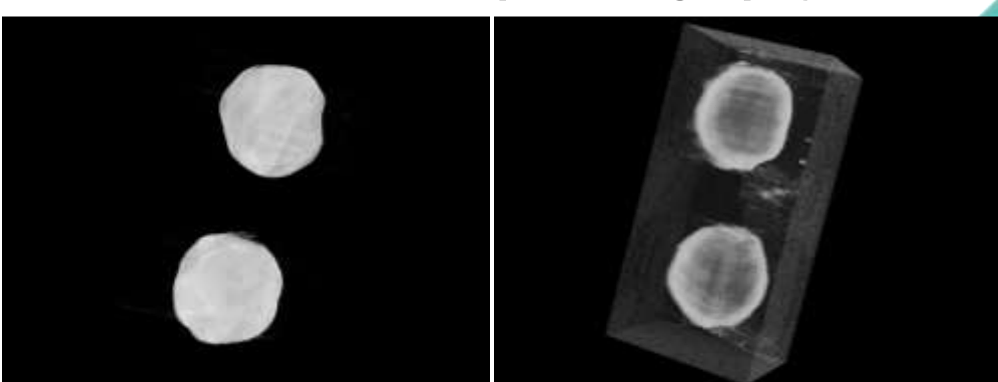
Speed up 8x

Combination of *In situ* Capabilities

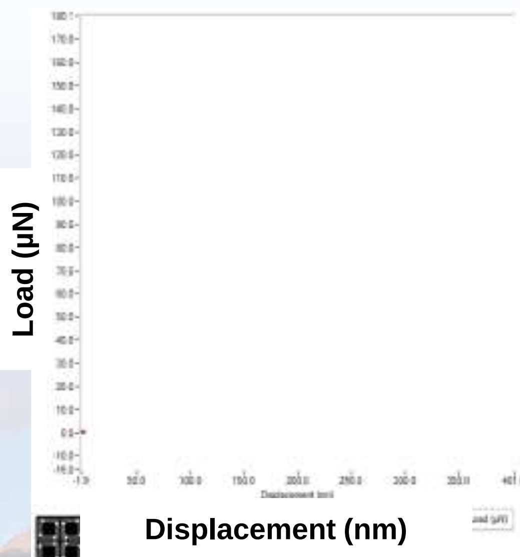
Contributors: S.M. Hoppe, B.A. Hernandez-Sanchez, T. Boyle, D. Gross, J. Kacher, & I.M. Robertson

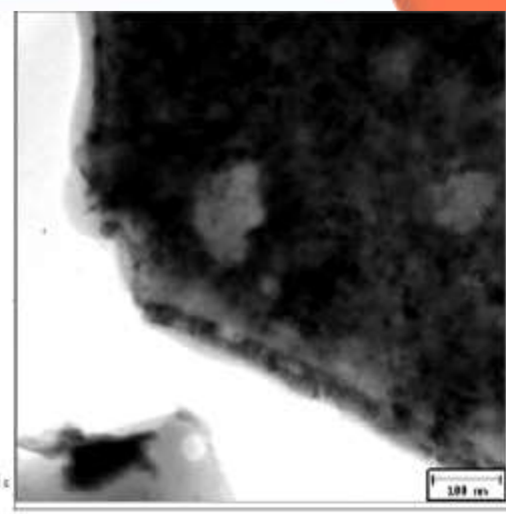


Irradiated Au np Tomography

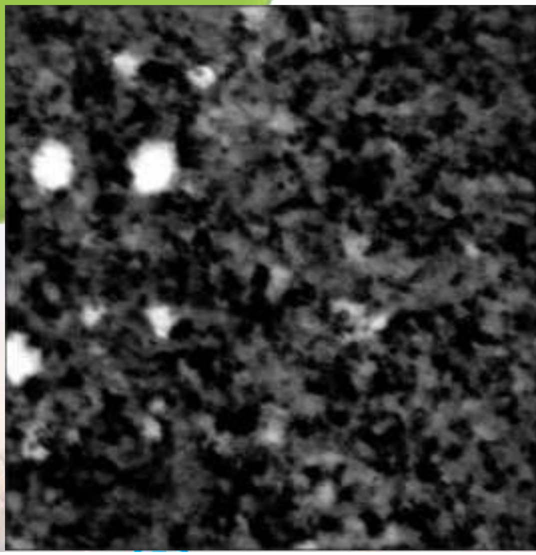


Nanoindentation of nc-Cu





Fe Corrosion

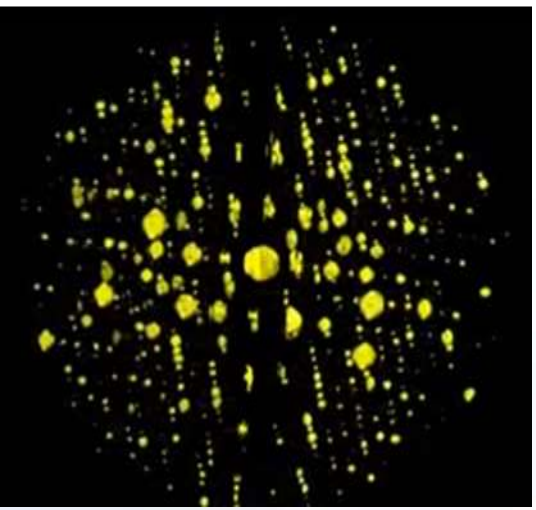


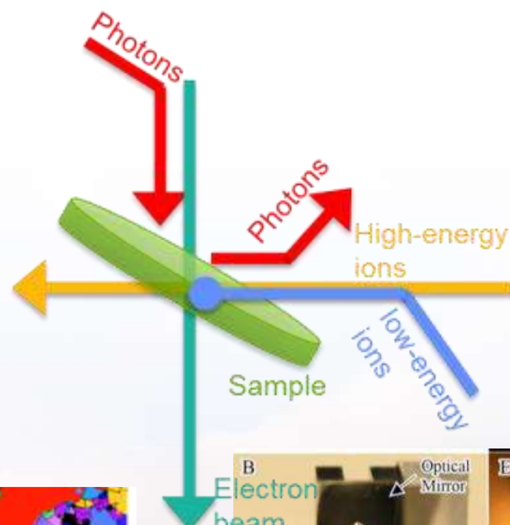
Dilute flow of acetic acid over 99.95% nc-PLD Fe

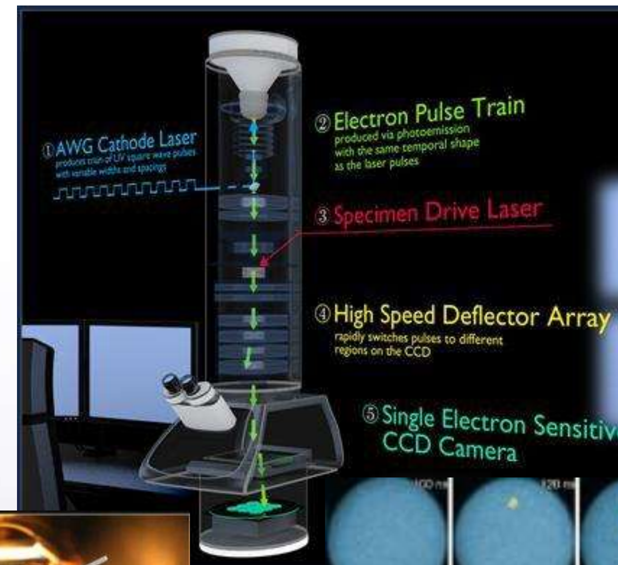
Contributors: B.G. Clark, P.J. Cappillino, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, L.R. Parent, I. Arslan, & Protochips, Inc.

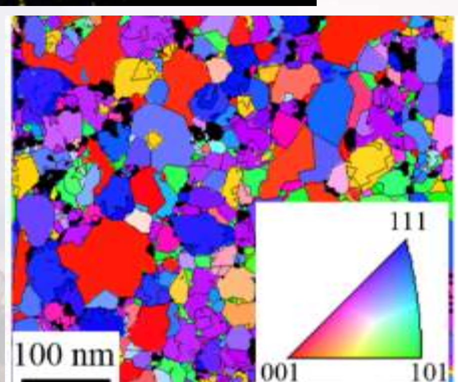
Proposed Future Directions

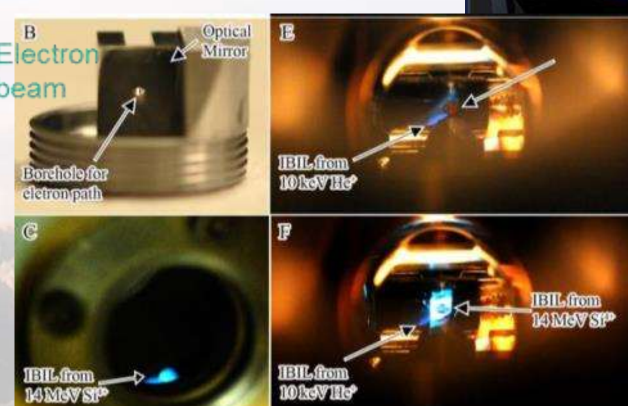
1. In-situ TEM CL, IBIL (currently capable)
2. *In situ* ion irradiation TEM in liquid or gas (currently capable)
3. DTEM: Nanosecond resolution (laser optics needed)
4. PED: Local texture characterization (proposal under review)

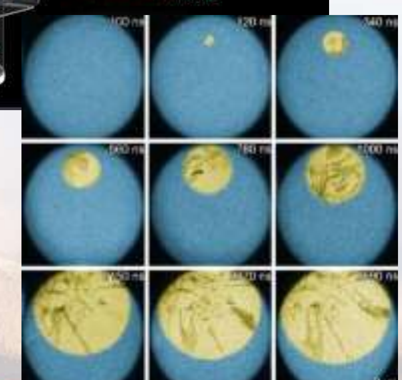












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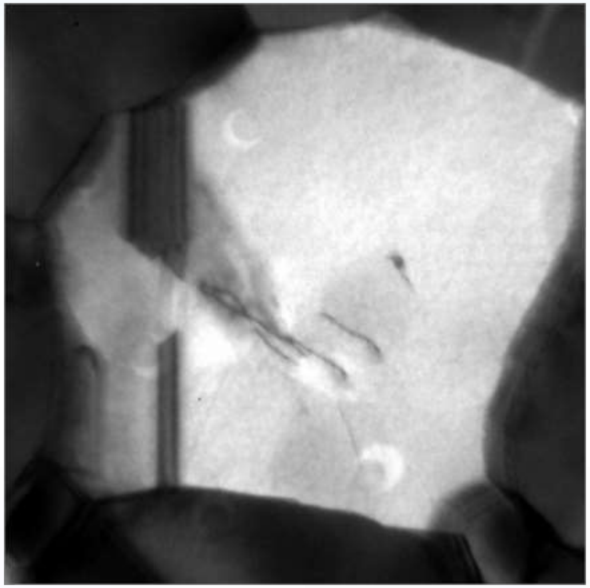
Summary

- **Single ion strikes can be observed in real time at nanometer resolution**
- **Varying Au np size and ion energy can result in cratering, nanofilaments, and particle explosion**
- **Initial studies are underway investigating ion species, energy, and dose rate in well-annealed Au thin foils**



- **Sandia's I^3TEM is one of a few in the world**
  - ***In situ* irradiation from H to Au**
  - ***In situ* gas implantation**
- **I^3TEM can provide fundamental understanding to key mechanisms in a variety of extreme conditions**
- **The I^3TEM capability are still being expanded**