Design, implementation, and characterization of a triple beam *in situ* ion irradiation TEM facility

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Motivation

- Radiation affects material properties and performance
- Synergistic effects of ion beams are not well understood
- *In situ* ion irradiation TEM allows real time observation of these effects at nanometer length scales

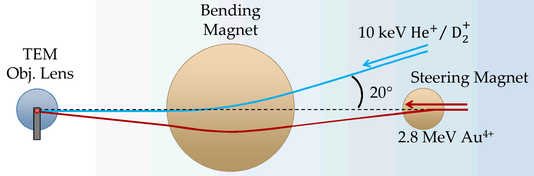
We must design a system capable of combining beams within the TEM, and must characterize the effects on samples.

Governing Equations

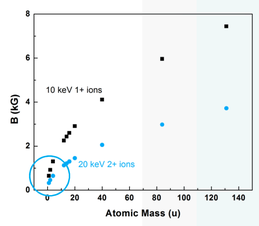
Beam Mixing and Steering

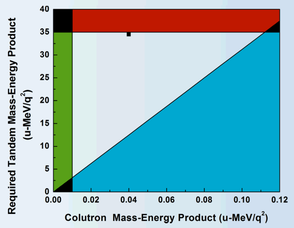
- Steering governed by mass-energy product: $^{mE}/_{q^2}$
- Radius of circular path taken by an ion in a mag. field, B:

$$r = \frac{\sqrt{2mE}}{Bq^2}$$



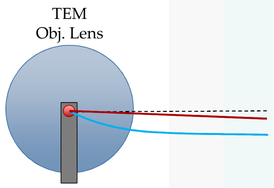
- Determined B fields needed to turn low energy beam
- Found resulting MEP bounds for high energy beam





Final Deflection Inside the TEM

The TEM's electron optics substantially affect trajectories of low MEP beams:



- Determined lower MEP bound of ~10 u-keV (e.g. 10 keV H^+)
- More rigid beams can be used at full OBJ lens strength
- Quantified effect of lens on angle of incidence

The Facility

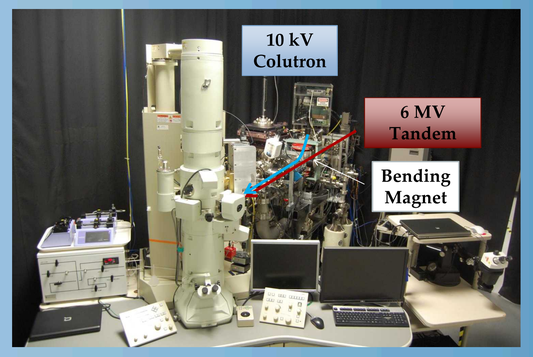
Two accelerators joined to a TEM optimized for *in situ* experiments

- Two source gases of equal mass (He/D_2) produce mixed beam from the Colutron

200 keV JEOL-2100

Stages:
- Hummingbird Heating
- Mechanical loading
  1. Hysitron PI 95
  2. Gatan Straining
- Protochips gas/liquid flow
- Tomography
  1. Hummingbird Single Tilt
  2. Gatan Double Tilt/Rotate

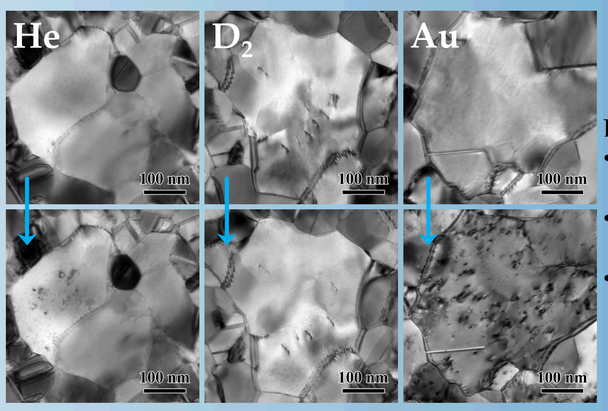
Other capabilities:
- Precession diffraction and orientation mapping



Effects on Material

Individual Ion Species into Gold

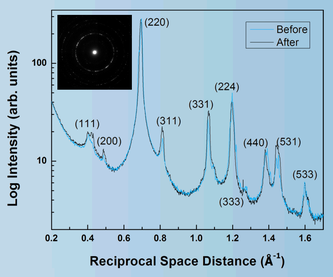
10 keV He^+, 10 keV D_2^+, and 2.8 MeV Au^{3+}, applied separately

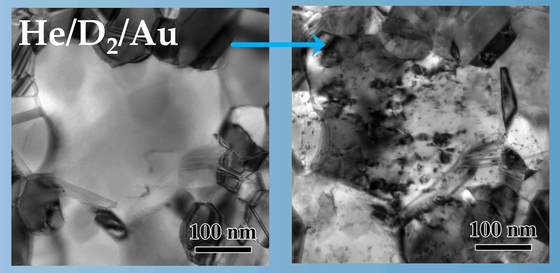


Left-to-right:
- He produced small dislocations and cavities
- D_2: Gradual dislocation motion and some damage
- Au produced far larger defect clusters

Concurrent Beams into Gold

The same three beams applied at once



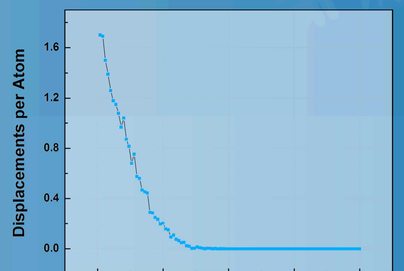


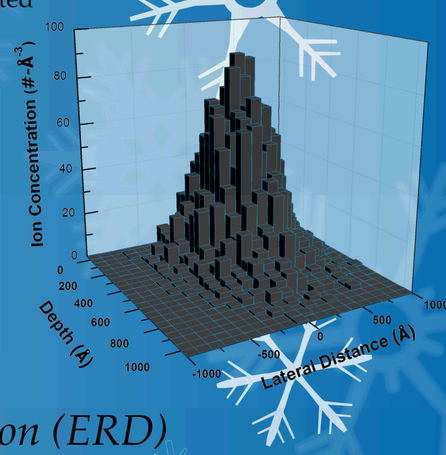
(left) Peak intensity changes in averaged selected area electron diffraction patterns, (right) accumulated damage as a result of the implanted components

Analysis of Implanted Ions

Simulated Ion Behavior

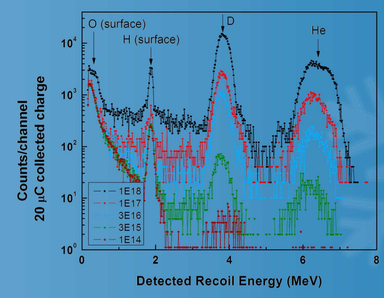
Prediction by TRIM (Transport of Ions in Matter), a Monte Carlo code
- 10 keV He ions into 100 nm Au at 60° from normal incidence
- ~55% backscattered/transmitted
- ~45% remain in Au layer
- D_2 behaves similarly

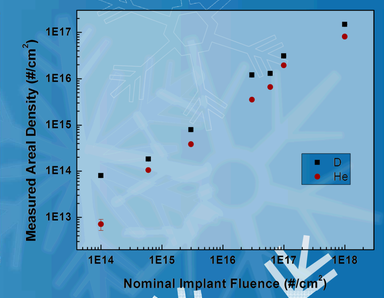




Elastic Recoil Detection (ERD)

Ion beam analysis technique ideal for depth profiling of light elements





(left) Recoiled ions sorted by energy and counted. (right) Quantified areal densities.

- Confirms the presence of implanted D and He
- Quantitatively determines the D/He ratio to be ~1.8

Conclusions

- Necessary beam rigidity requirements computed
- Three-component concurrent irradiation demonstrated in the TEM

Further work is needed to develop a quantitative understanding of the microstructural changes caused by these irradiations.

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