

Microstructural development in model ErT₂ films

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Approach

- Study microstructural evolution in ErT_2 films
 - as a function of time
 - as a function therefore of He concentration
- Use model films
- TEM investigations performed in parallel with neutron scattering

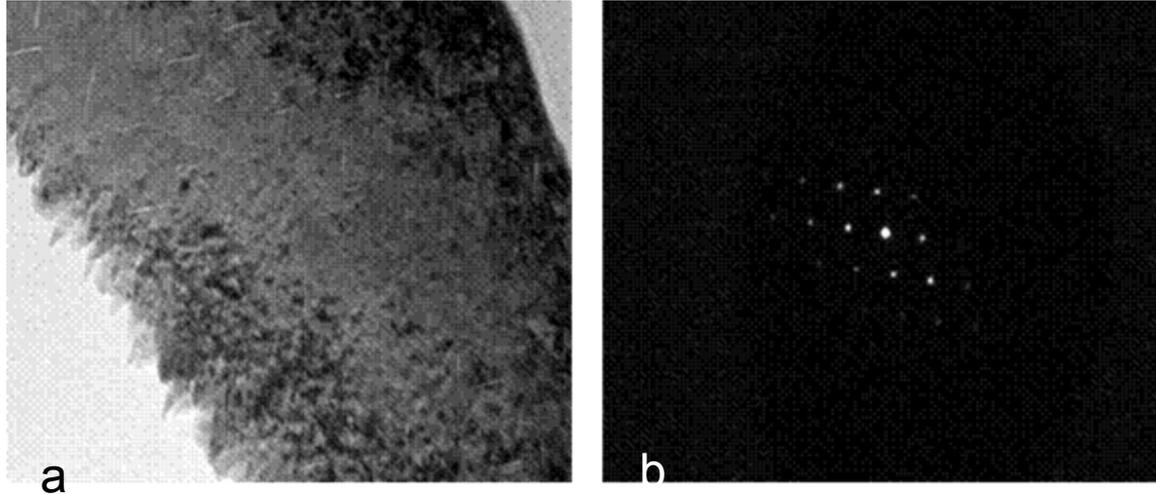
Samples

- {100} Si wafer as substrate
- 1000 Å Mo
- 5000 Å Er
- Samples prepared by Loren Espada (SNL)
- Er loaded with tritium by Tom Venhaus (LANL)
- No subsequent thermal treatment

Cross-sectional TEM samples

- Wafer with films cleaved into strips
- Strips mounted in sandwich configuration
- Cross-section cut, ground and polished
- Sample dimpled until film thickness $\sim 10 \mu\text{m}$
- Ion milled at $\sim 3.5 - 4^\circ$ and 5kV until perforation
- Examined in JEOL JEM-2000FX TEM at 200 kV

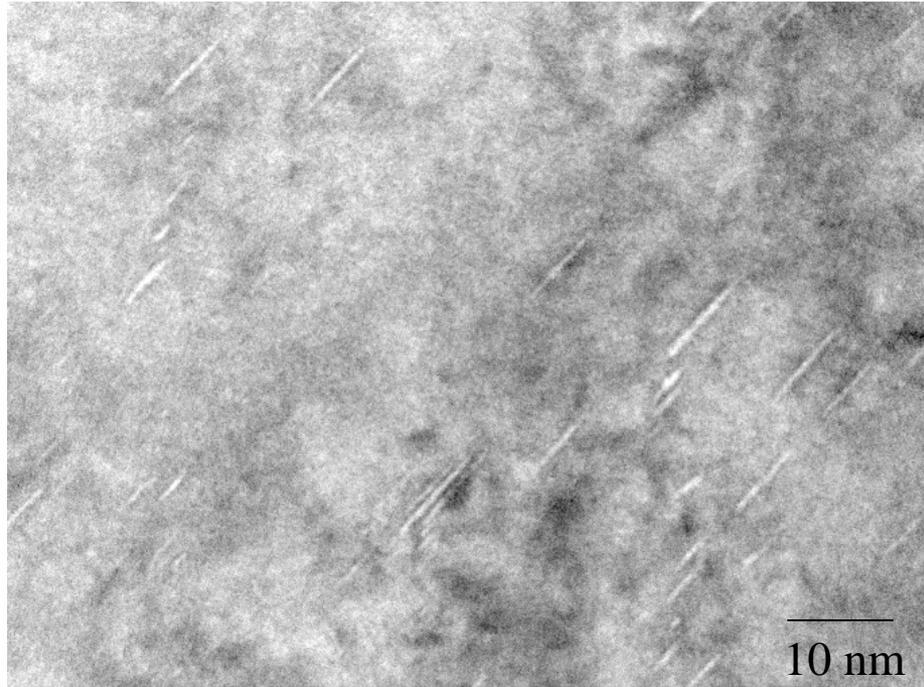
Helium bubbles on $\{111\}$ planes



a) Bright-field transmission electron micrograph (827 days)
b) Selected-area diffraction pattern close to $\langle 110 \rangle$ zone axis

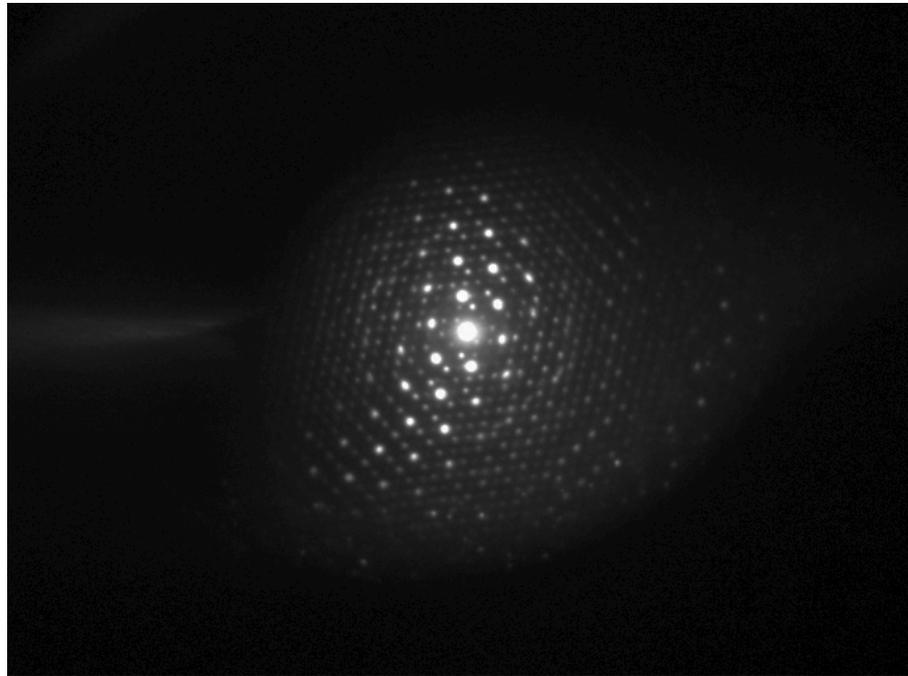
- Two sets of plate-like helium bubbles are visible (at an angle of $\sim 72^\circ$)
- Helium bubbles lie on $\{111\}$ planes
- No indication of denuded layer in this model system

Helium bubbles 62 days after tritium loading

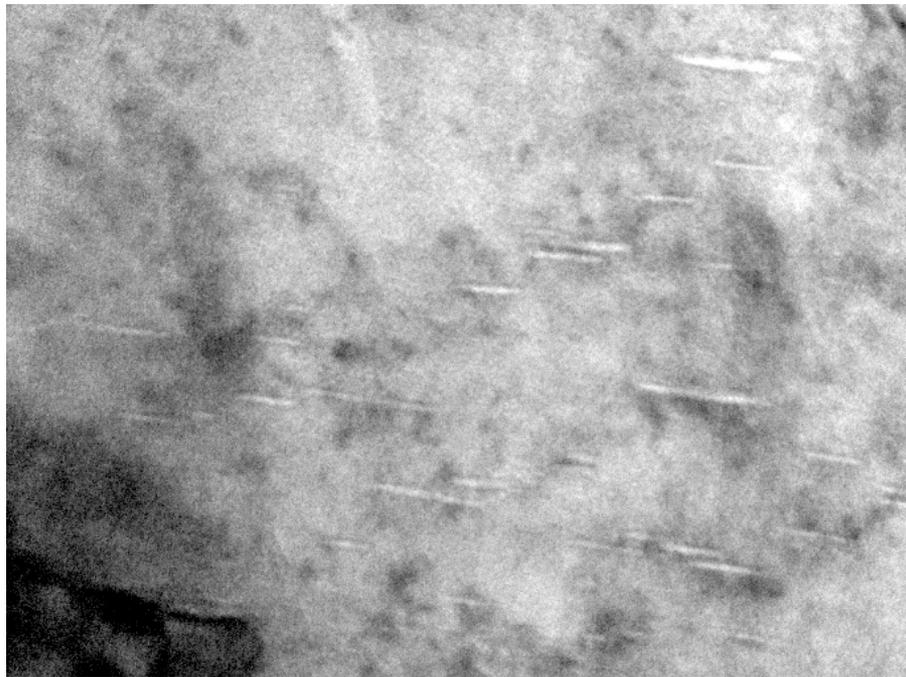


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Additional diffraction spots (62 days)

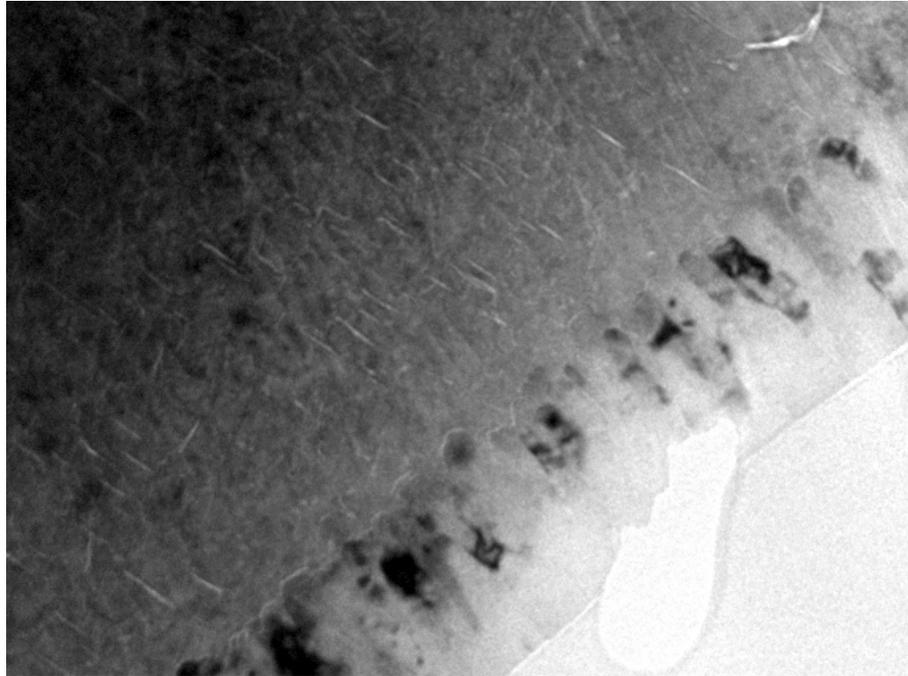


Helium bubbles 62 days after tritium loading



10 nm

ErT₂ and Mo films (820 days)

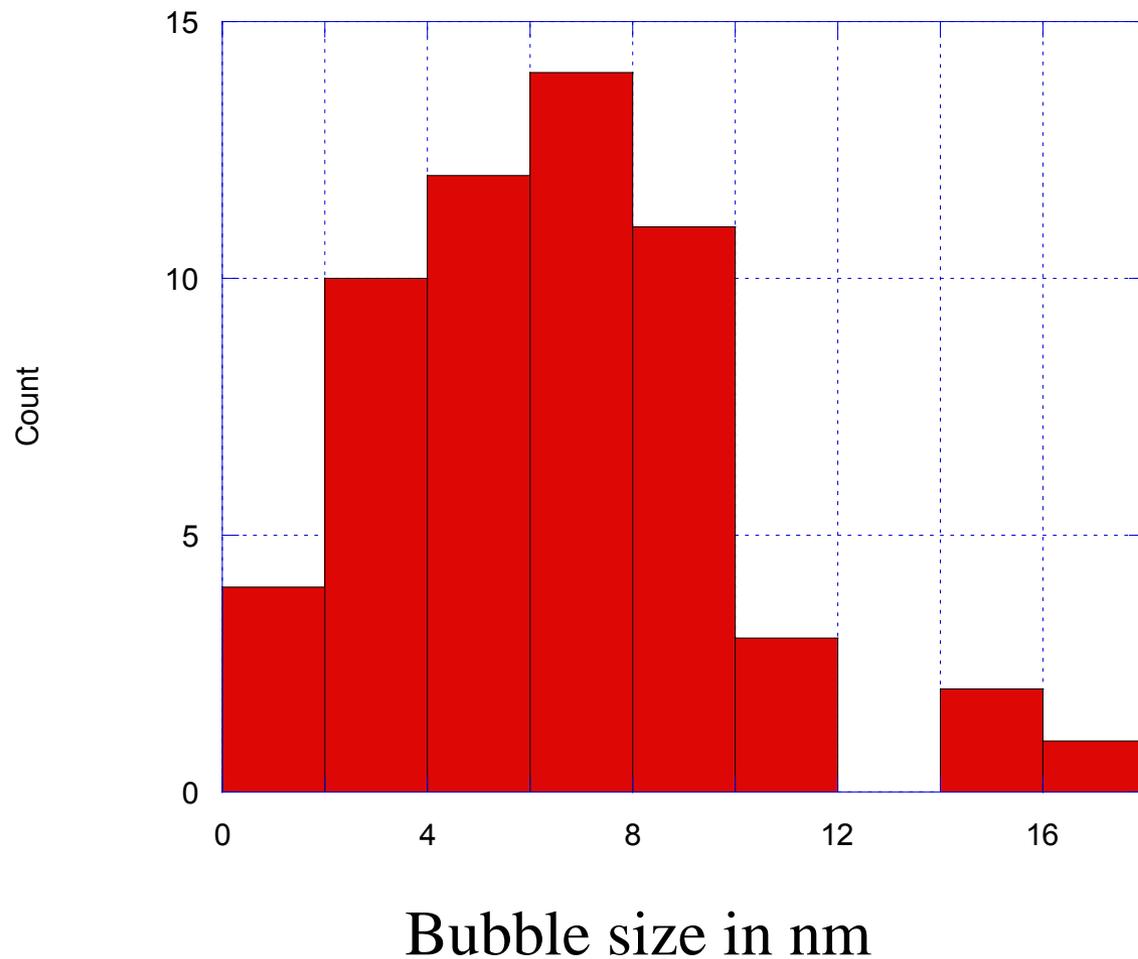


Note much smaller grain size of Mo, waviness of Er/Mo interface, and presence of particle of another phase

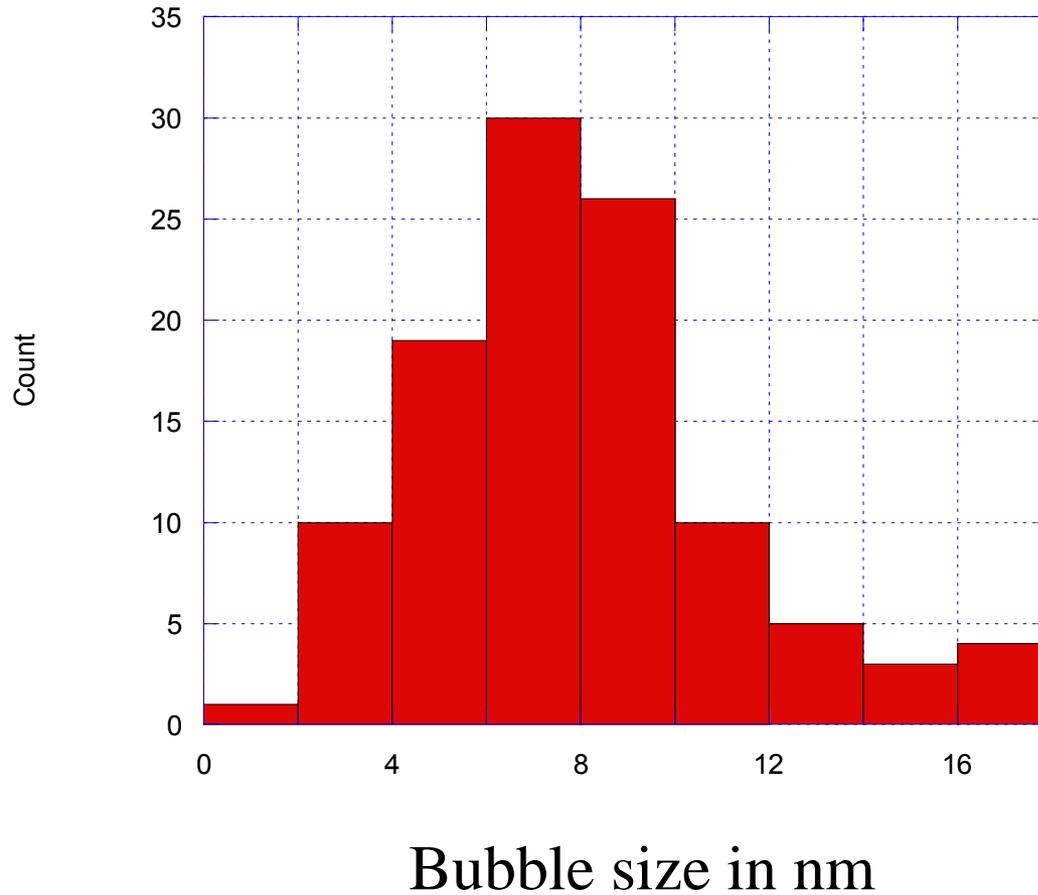
ErT₂ film (820 days)



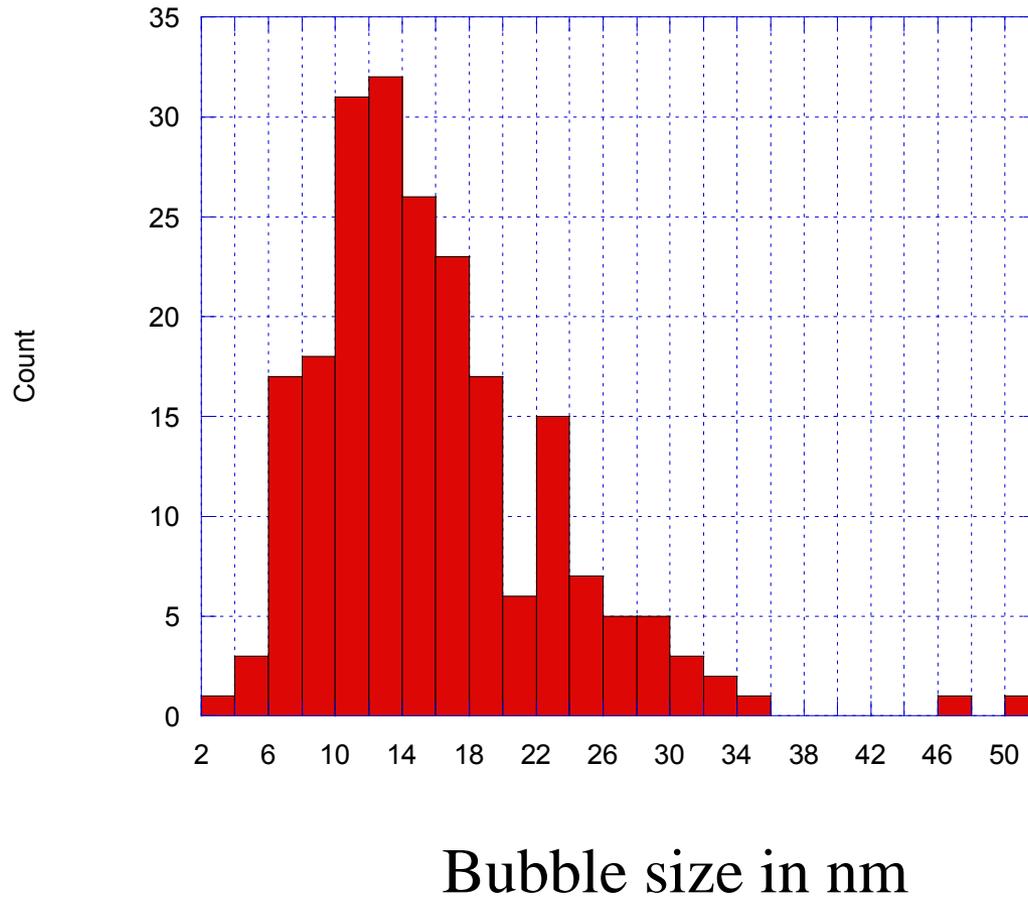
Note milling artifacts, and presence of particle of another phase with associated cavitation



Histogram of bubble sizes (62 – 64 days after loading)
[n = 56]

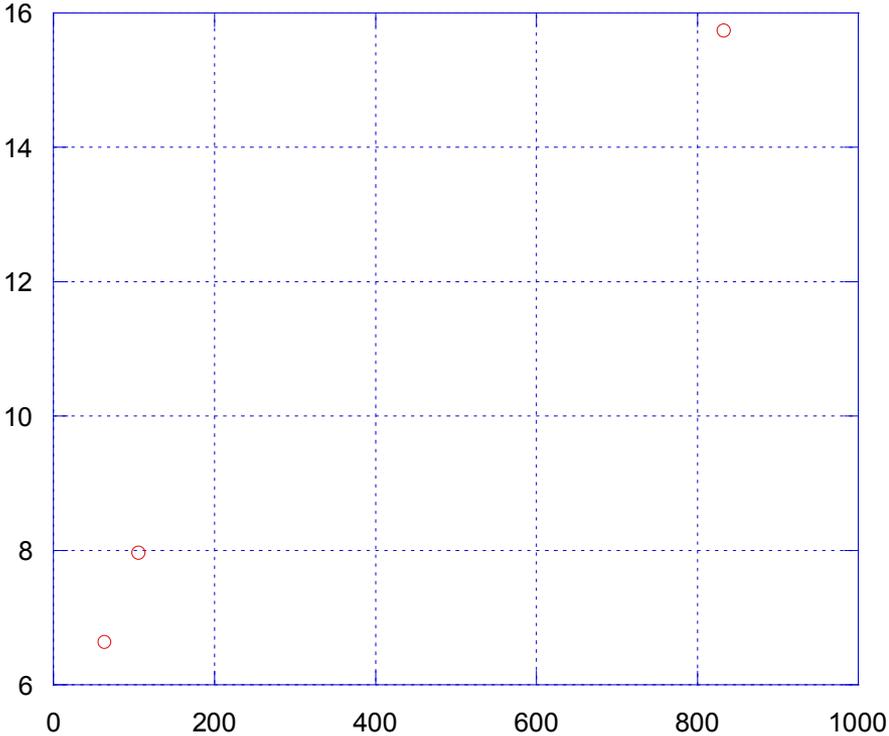


Histogram of bubble sizes (106 days after loading)
[n = 108]



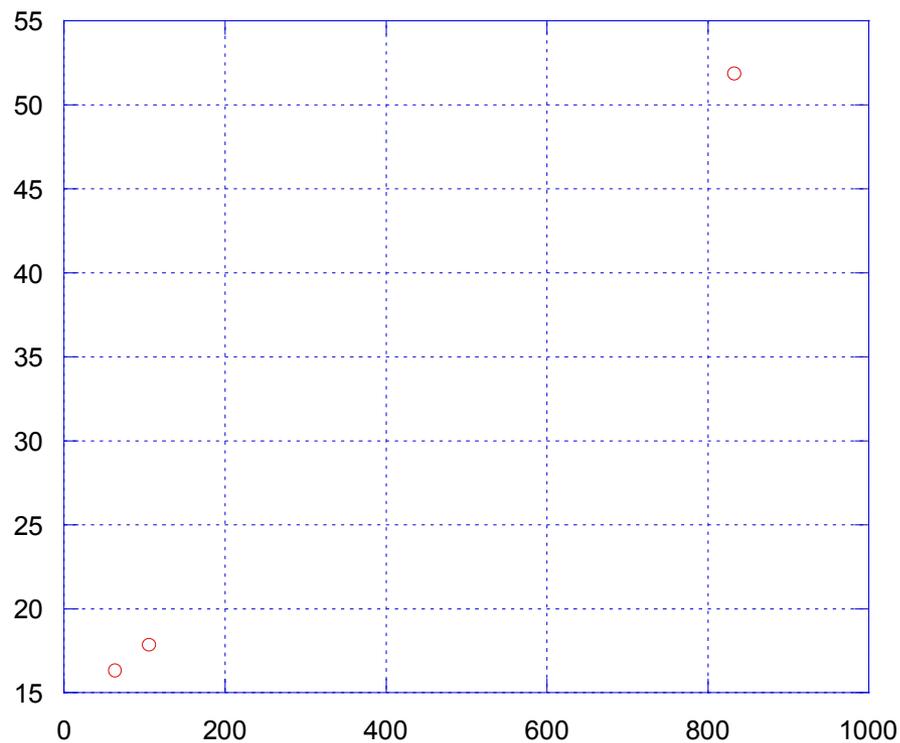
Histogram of bubble sizes (820 – 846 days after loading)
[n = 214]

Mean
bubble
size (nm)



Time after loading (days)

Maximum
bubble
size (nm)



Time after loading (days)

Summary

- Er grain size is much larger than Mo grain size, and Er “smooths over” undulations in Mo surface (confirmed by AFM)
- Plate-like He bubbles are already present at 62 days after tritium loading, lying on $\{111\}$ planes
- Mean bubble size increases with time
 - from 6.64 nm to 15.74 nm between 62 days and 286 days
- Some larger second-phase particles are seen
- Indication that fine precipitation may be occurring

Future work

- Characterization of films at shorter times after loading, to study initial development of He bubbles
- Characterization of films over longer times, to follow microstructural development up to helium release

Future work

- Study possibility of fine precipitation
- Look for other microstructural damage
- Look for influence of single-crystal Si substrate

Future work

- Comparison of TEM and SANS data
- SANS on series of samples stacked $45^\circ/45^\circ$ angles
- Thick films ($\sim 50,000$ Å Er)
- Films on {111} Si wafers

Acknowledgement

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.