Physics and Chemistry of Metal Tritides
Working Group
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Helium Bubbles and Microstructure in Aged Erbium Tritide Films

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- **Objective**

To provide quantitative microstructural information on helium bubble development in aged, tritided erbium films

- **Specimen conditions of interest**

T5

loaded 9/1998 examined 2/2002,100% T-loaded, 0.18 release fraction

3143006

loaded 9/2000 examined 6/2004, 40% nominal thickness

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- **Specimen preparation, etc**

Cut piece off dome with low-speed saw (under ethylene glycol with no adverse effects)

Glue to other Mo backing pieces using thermal setting epoxy (~150°C), place in brass tube and slice for cross section disks

Polish one side, dimple to less than 10 μm

Mill in Gatan PIPS using (5keV) Ar ions

Examine in JEOL 2000EX

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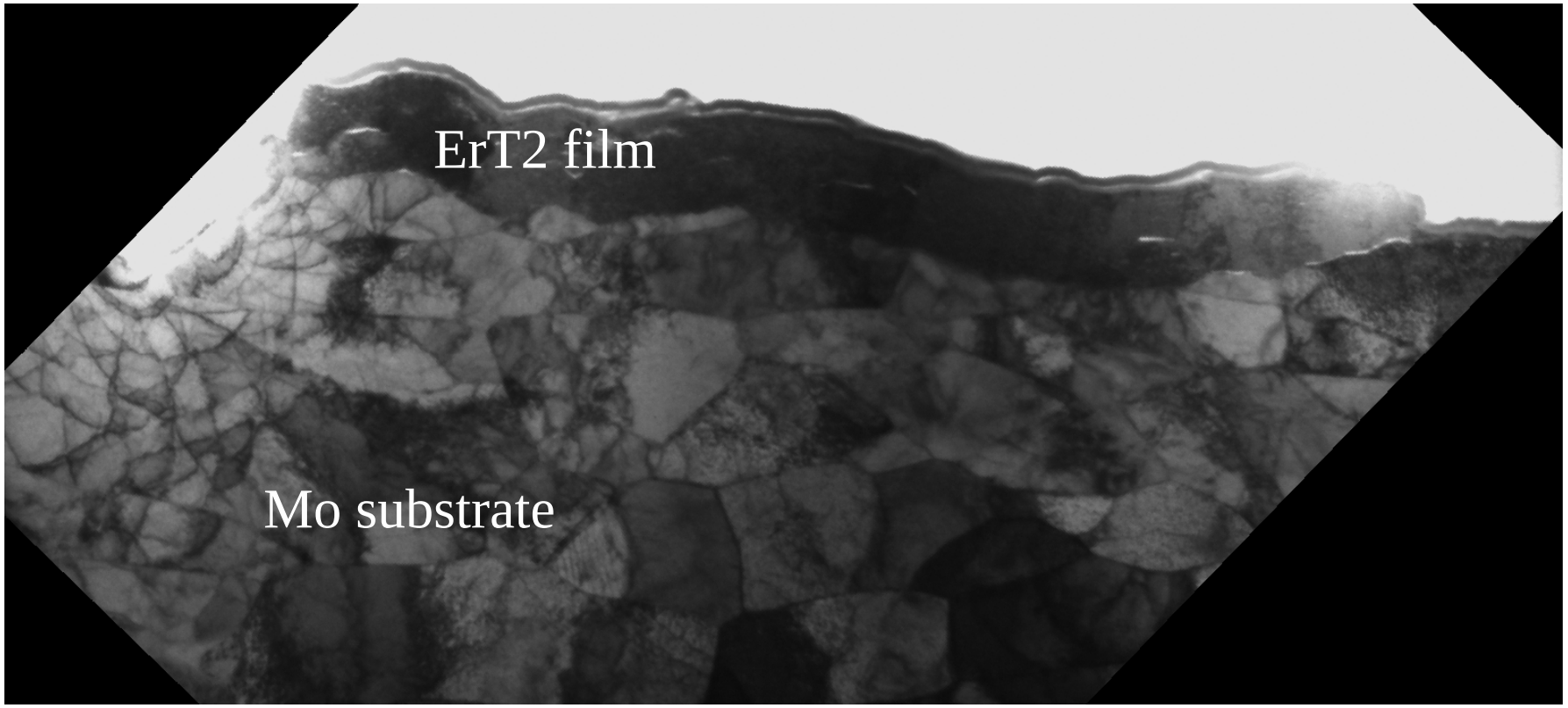
T5 – Low Magnification Overview



Grain size for Mo in this region 0.5 – 1 μm

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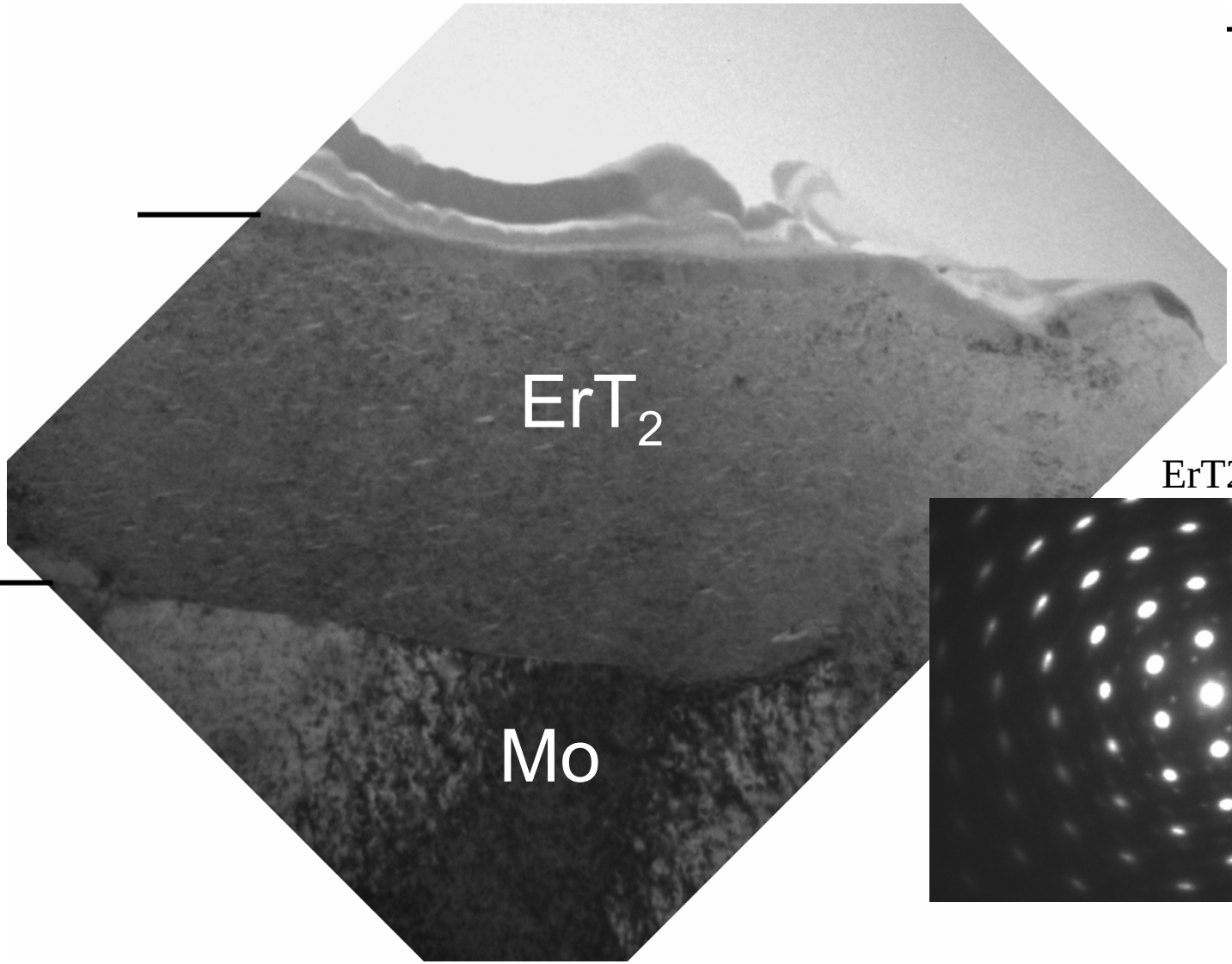
T5 – Tritide layer



ErT2 SAD Pattern

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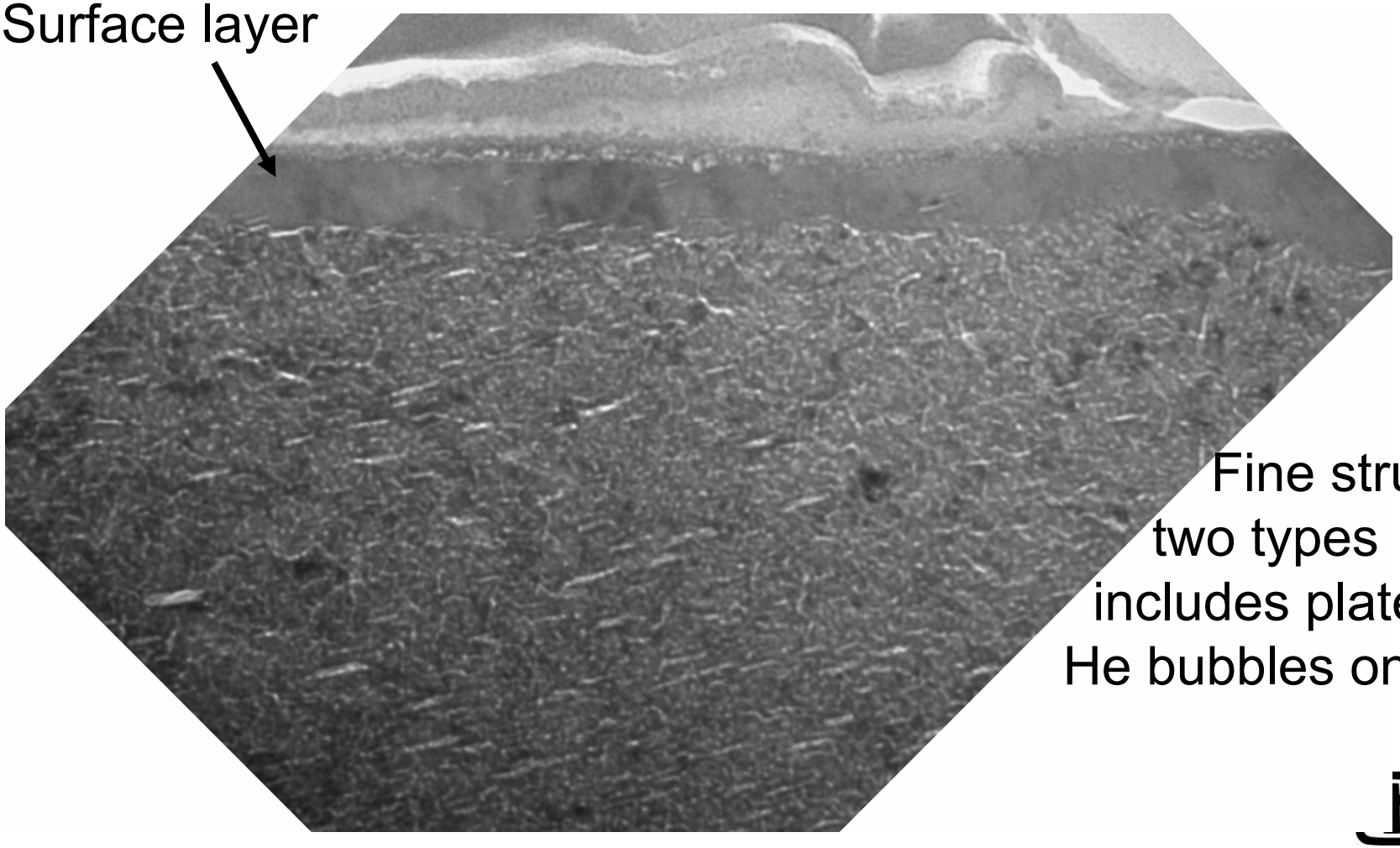
T5 – Tritide layer details

~40 nm
Surface layer



Fine structure
two types
includes plate-like
He bubbles on {111}

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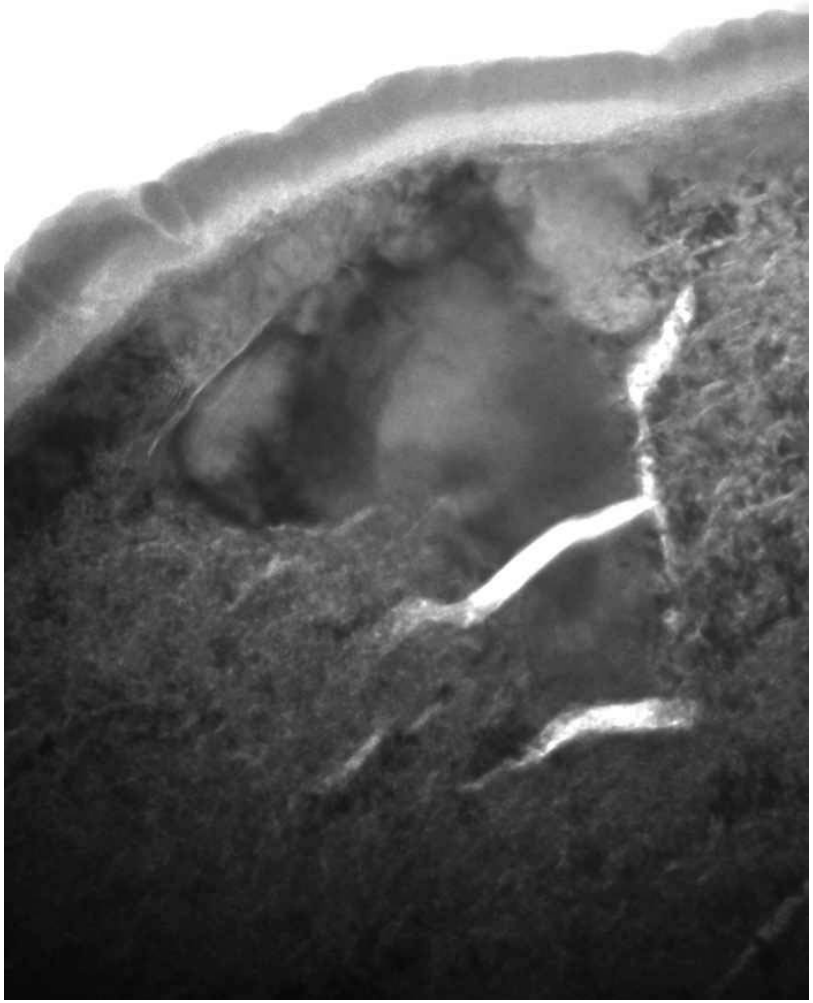
T5- Anomalous denuded region



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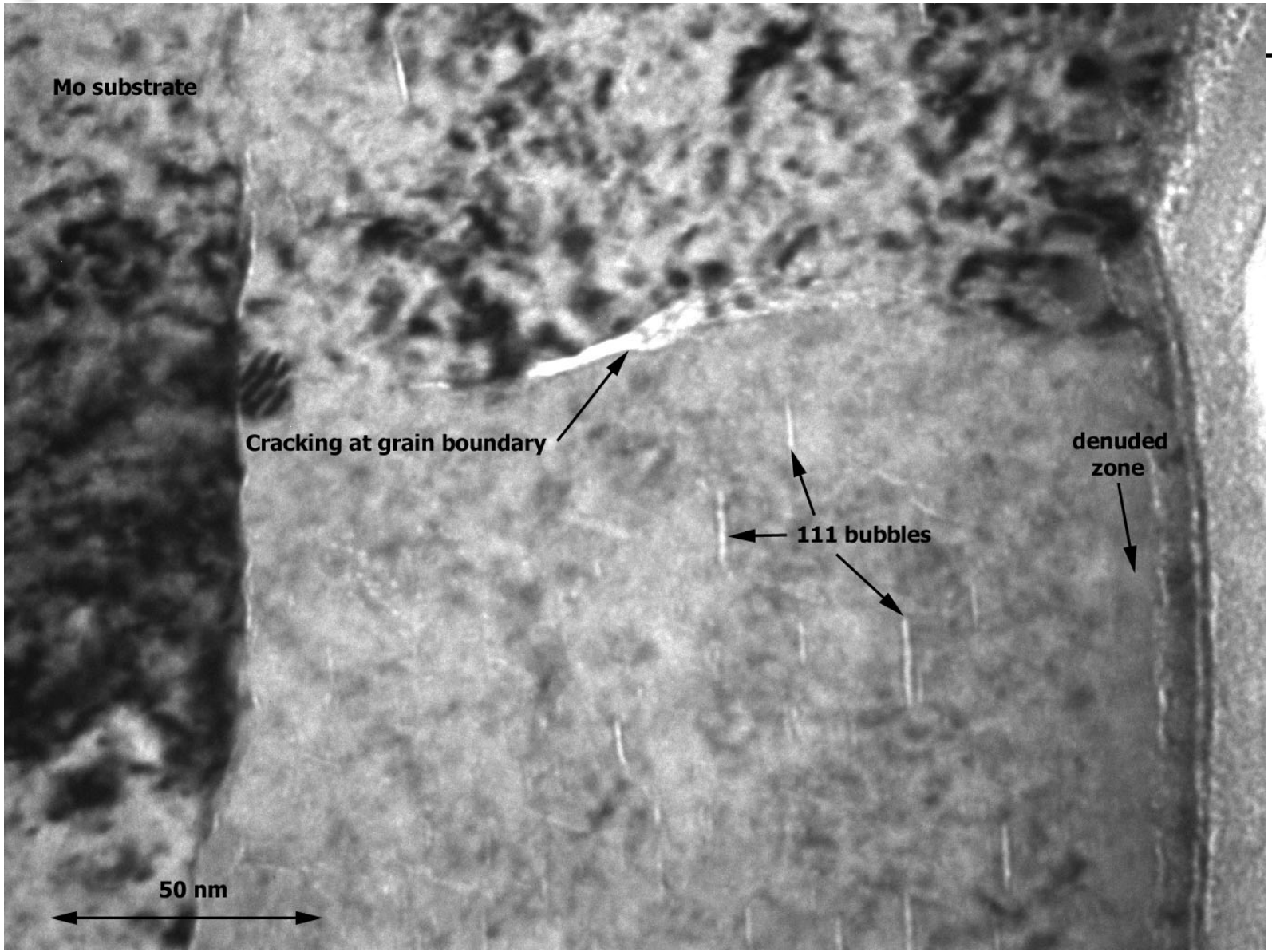
3143006-40% nominal thickness – Tritide layer



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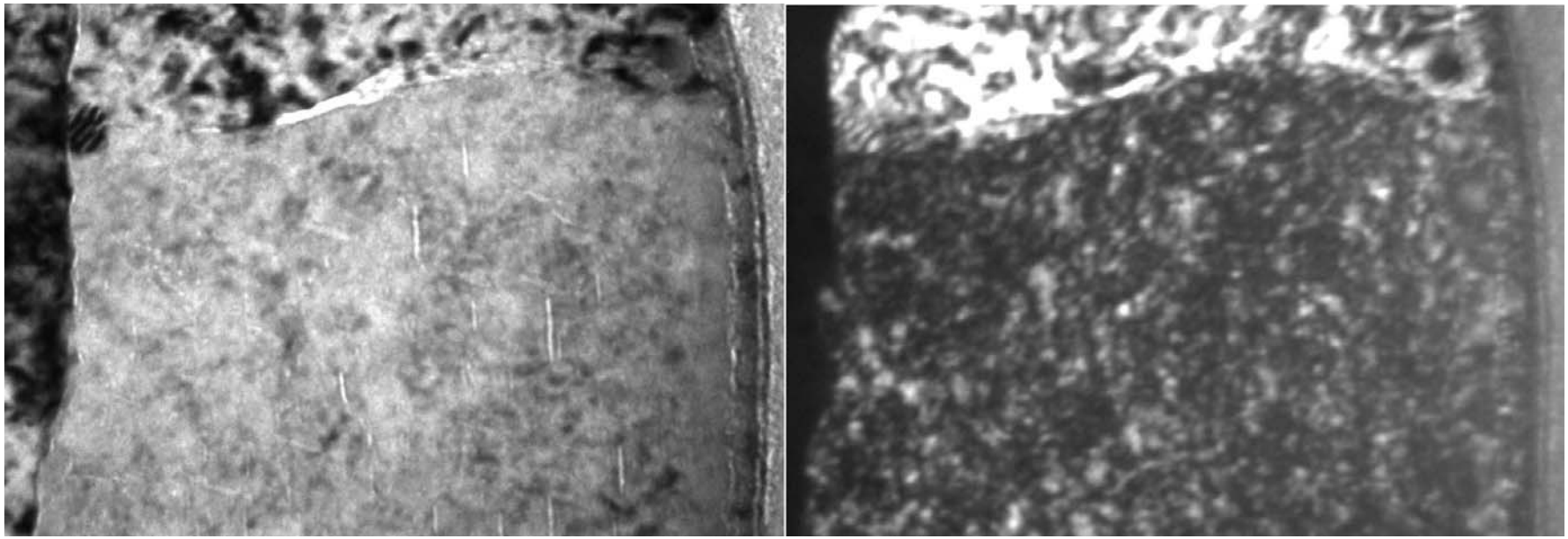
3143006 - Bright and Dark Field Images



The second type of fine structure is probably precipitates ~5 nm in diameter

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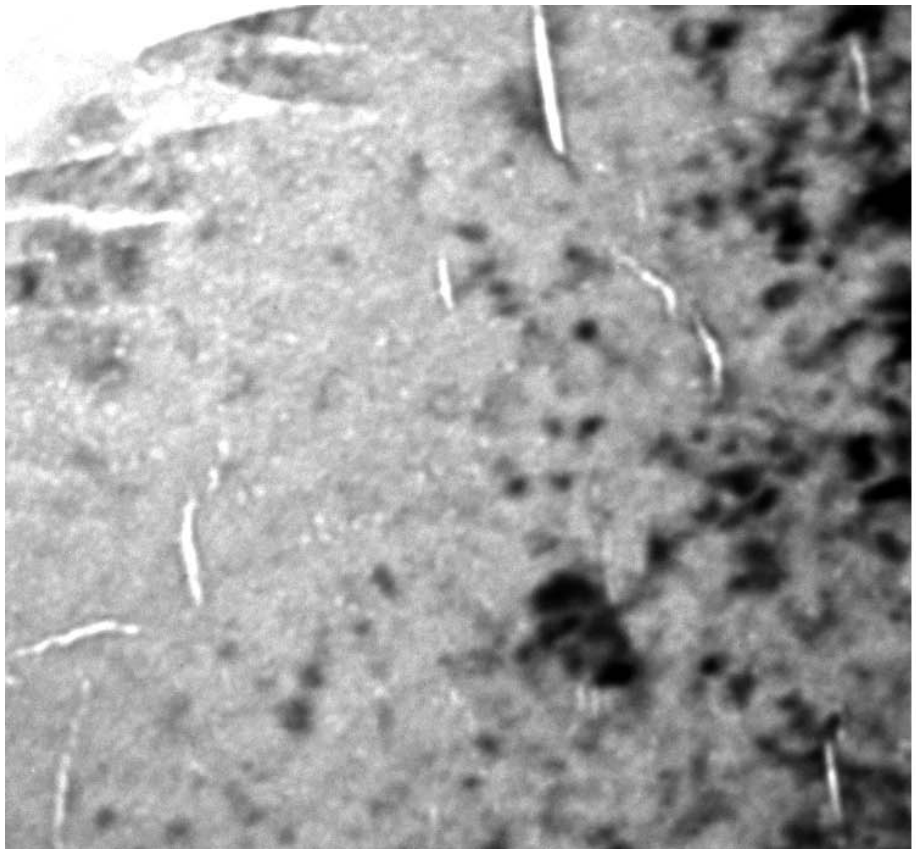
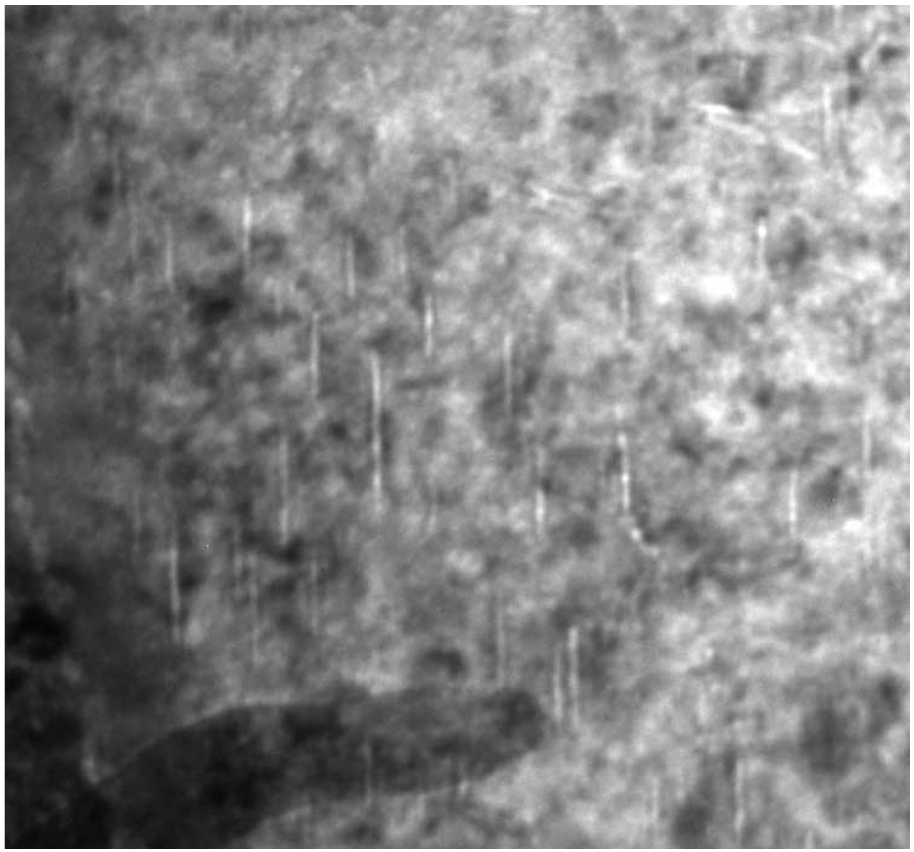
3143006 – Ion milling artifacts



Thin section



Thick section

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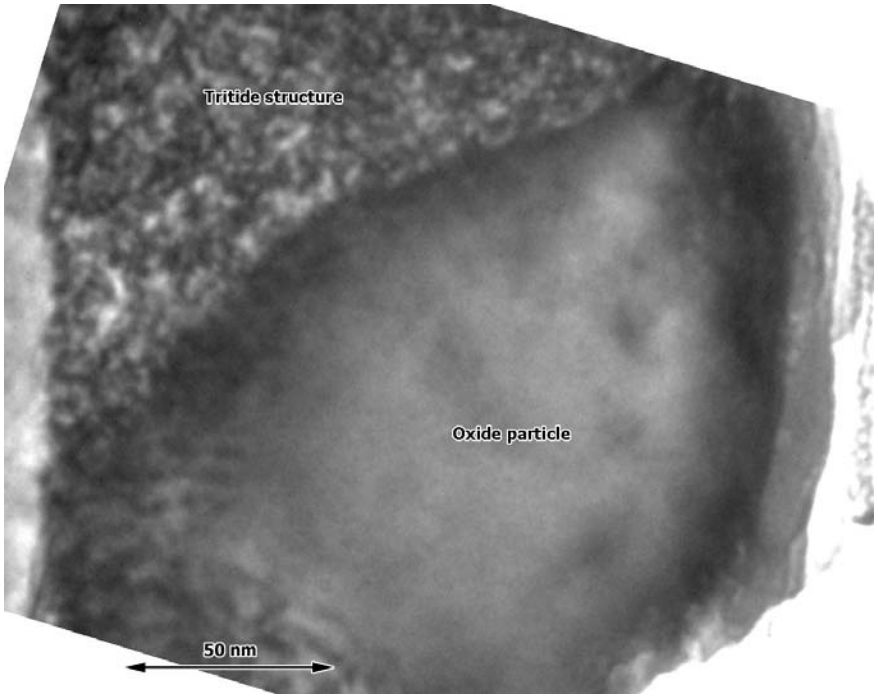
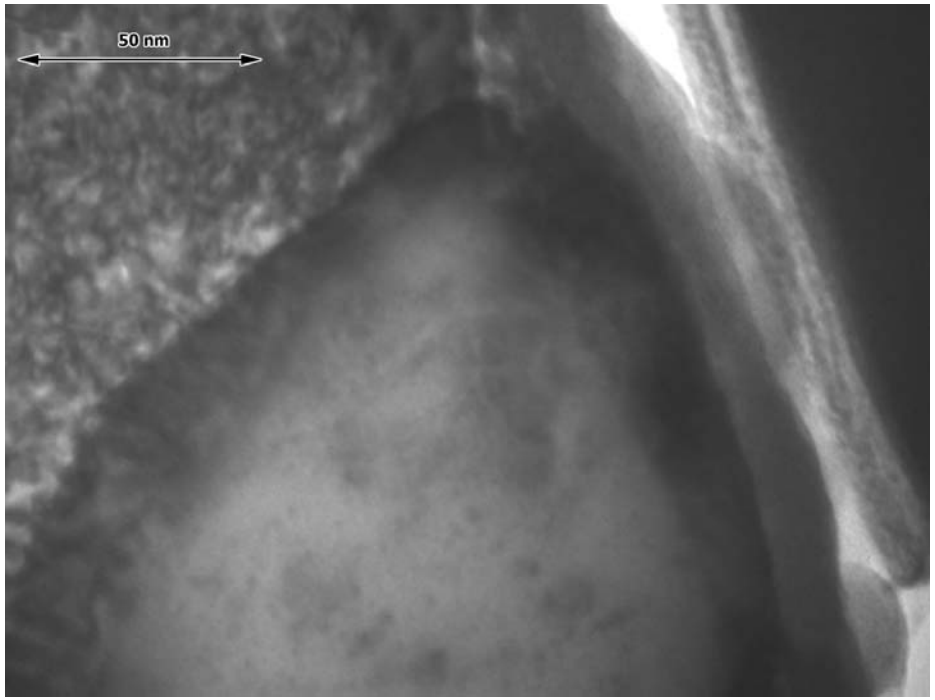
3143006 - Large oxide particle





Particle is almost the size of the tritide layer and contains fine structure.

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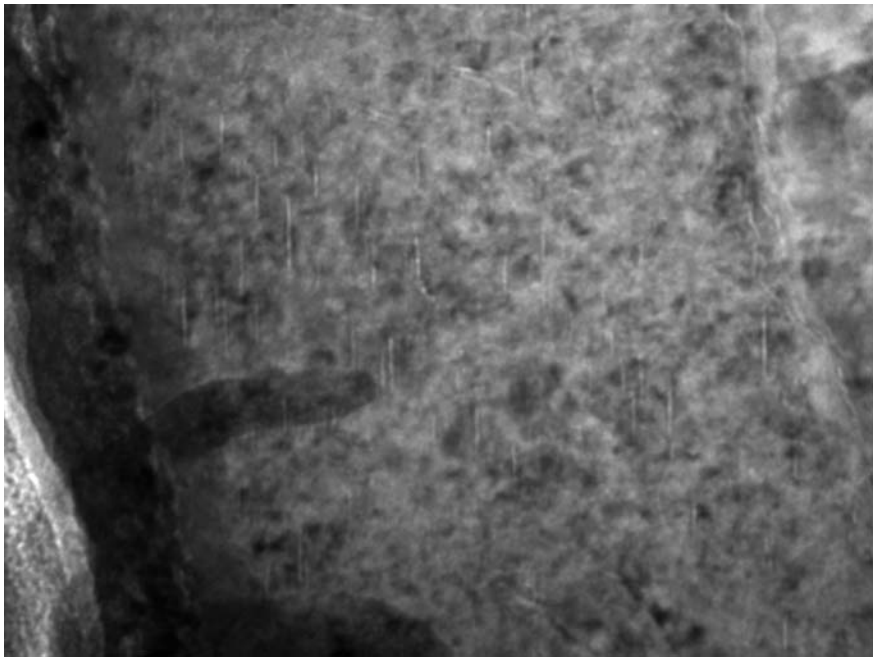
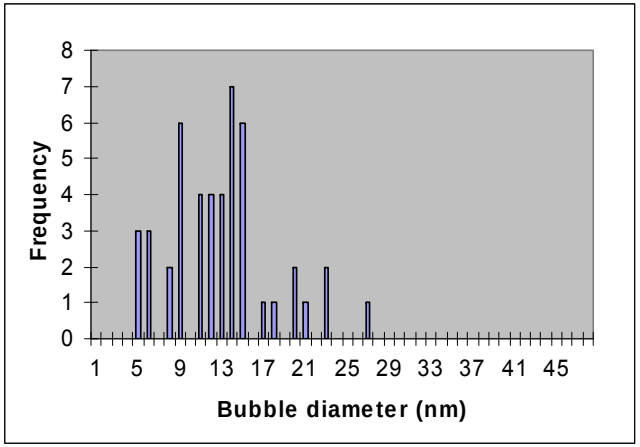
3143006 - Bubble Quantification





Average bubble diameter = 12.3 nm
and bubble thickness = 1 nm

If we estimate the foil thickness at 100 nm, probably a low estimate, the bubble density is 6 x $10^{22}m^{-3}$ (allowing for four sets on {111}) and the bubble volume fraction is ~0.9%.

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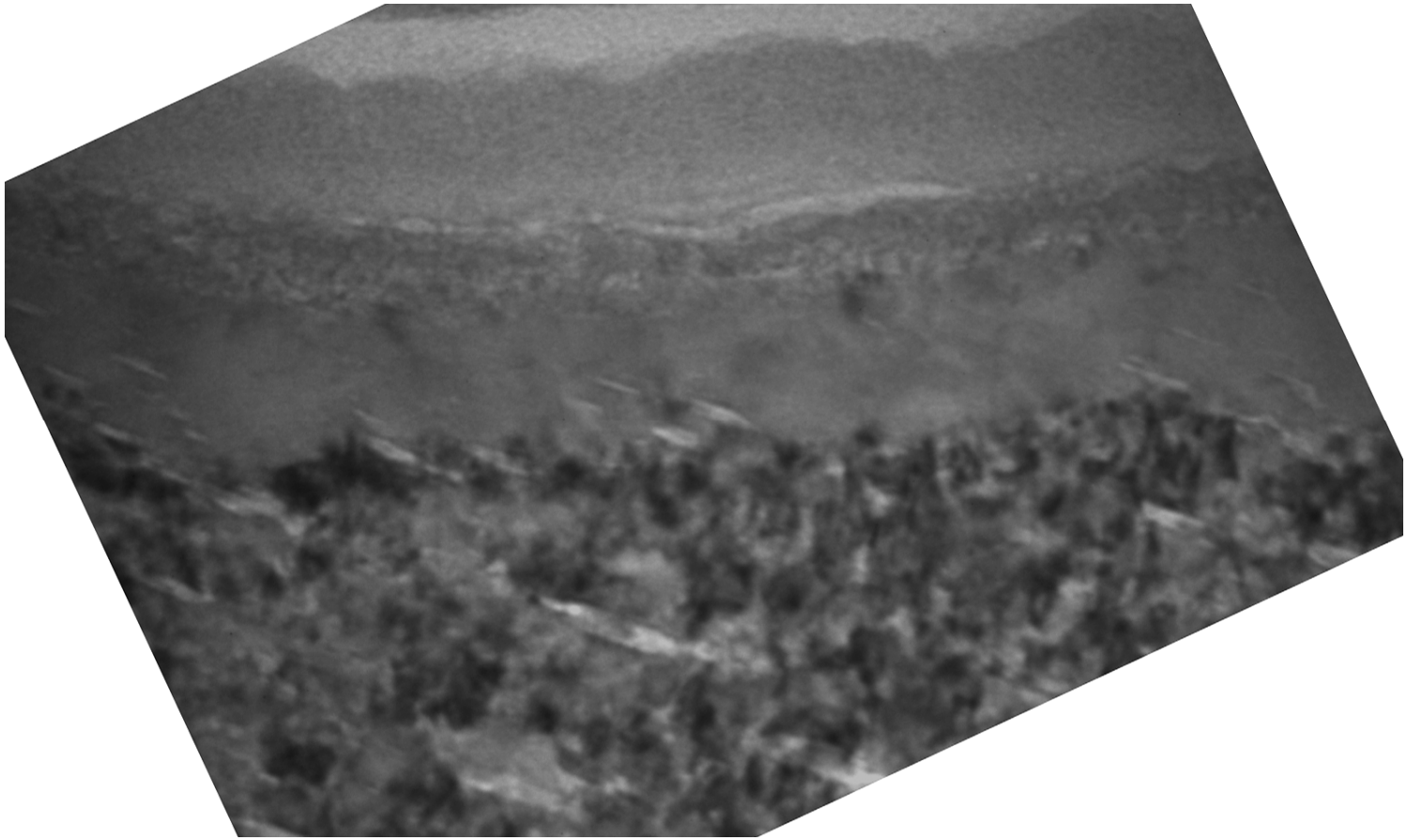
T5 another view of denuded surface region



Bubble are present in the surface denuded layer,
suggesting that the layer grows into bubble-bearing structure.

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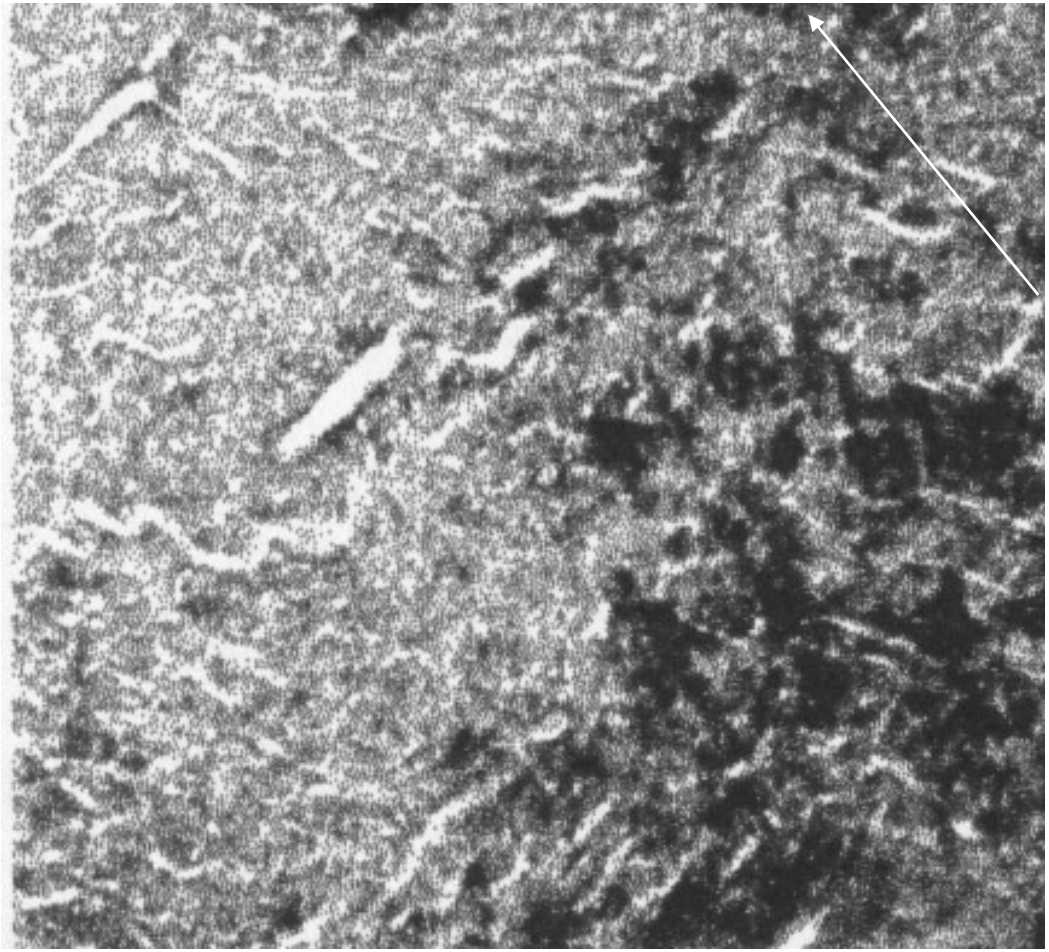
T5 – A possible cracking mechanism

Stress concentrations at bubble corners can lead to cracking between bubbles, and eventually provide a path for tritium desorption



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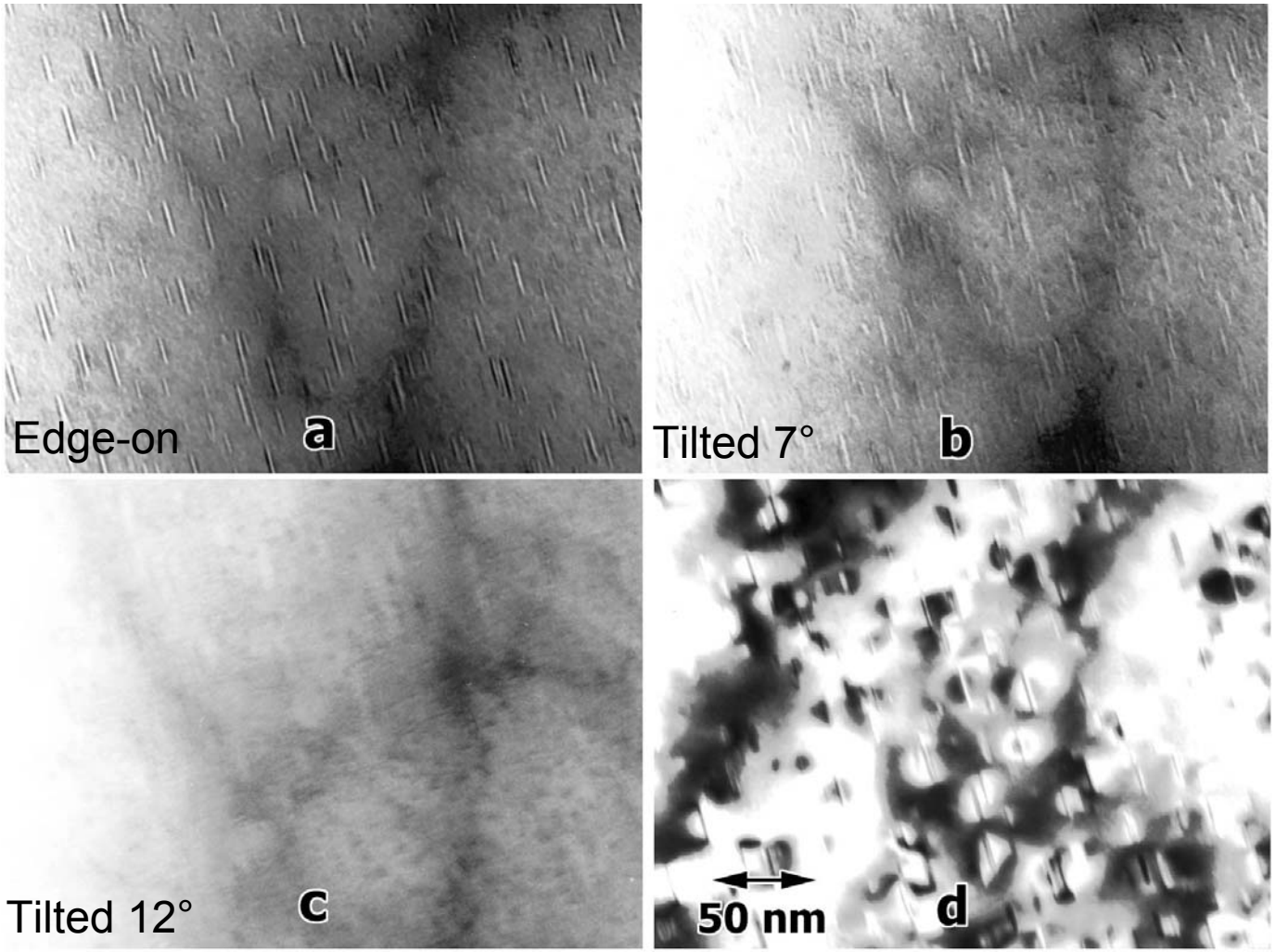
Summary

- Aged erbium tritide foil specimens were found to contain four distinctly different features.
- The general structure was of large columnar grains of tritided erbium that showed a distinct mottled appearance on a scale of ~5 nm due to precipitation, as indicated by extra spots in diffraction patterns and dark field imaging.
- However, the external edge of the foil showed a band of material about 20 nm thick in 3146006 and 40 nm thick in T5 which did not have this mottled appearance that can be describe as a denuded zone.
- When the specimen could be suitably oriented, unusual cavities (bubbles) were observed. The cavities ranged in diameter from 5 to 25 nm, but with a uniform thickness of ~1.0 nm lying on {111} planes.
- Occasionally, grains were found within the foil that did not show mottling. One such grain that extended almost all the way through the foil was analyzed to provide lattice spacing and composition and was found to be an oxide particle.
- Therefore, the four distinct features are 1) the base erbium tritide structure with fine precipitation, 2) a surface denuded zone without fine precipitation, 3) pancake shaped cavities presumably pressurized with helium, and 4) large particles of erbium oxide that can extend through the foil.

Discussion

- **Similar thin He bubbles have been seen in neutron irradiated beryllium (4.3×10^{22} n/cm^2, E>0.1MeV, at 380°C)***



* ASTM STP 1366, pp1051-61.

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Discussion continued

Issues to be resolved:

- **Nature of surface denuded layer**
  layer thickness dependent on T-load level? Same crystal structure? What are those precipitates?
- **Formation mechanism for large erbium oxide particles**
  formed during film formation?
- **Mechanism for tritium desorption**
  cracking? GB diffusion? Bulk diffusion?

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Conclusions

- **Erbium tritide coatings contain large columnar grains of erbium tritide and smaller large grains of erbium oxide. Following aging of ~3.5 years, the erbium tritide grains generally contain two types of fine structure; highly flattened helium bubbles on {111} planes and fine precipitation, but an outer surface layer is found to be relatively structure-free.**
- **It is proposed that tritium desorption may be controlled by cracking at columnar grain interfaces and between helium bubbles**

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