Initial observations of He Bubbles in Palladium

Hydrogen and Helium Isotopes in Materials Working Group
Albuquerque, NM

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Outline: Initial Observations

- **Specimen Prep/Microscopy**
  - ***In situ* FIB lift out (He Implanted Pd)**
  - **"Powder in tube" method (Tritided Pd)**
  - **RAD: JEOL 2000FX, TEM/STEM, EDS, digital camera**
  - **Non RAD: Tecnai F30ST, TEM/STEM, HAADF, EDS, digital cameras, EFTEM**
- **He-Implanted Pd foil**
- **Tritided Pd powders**
- **Conclusions**

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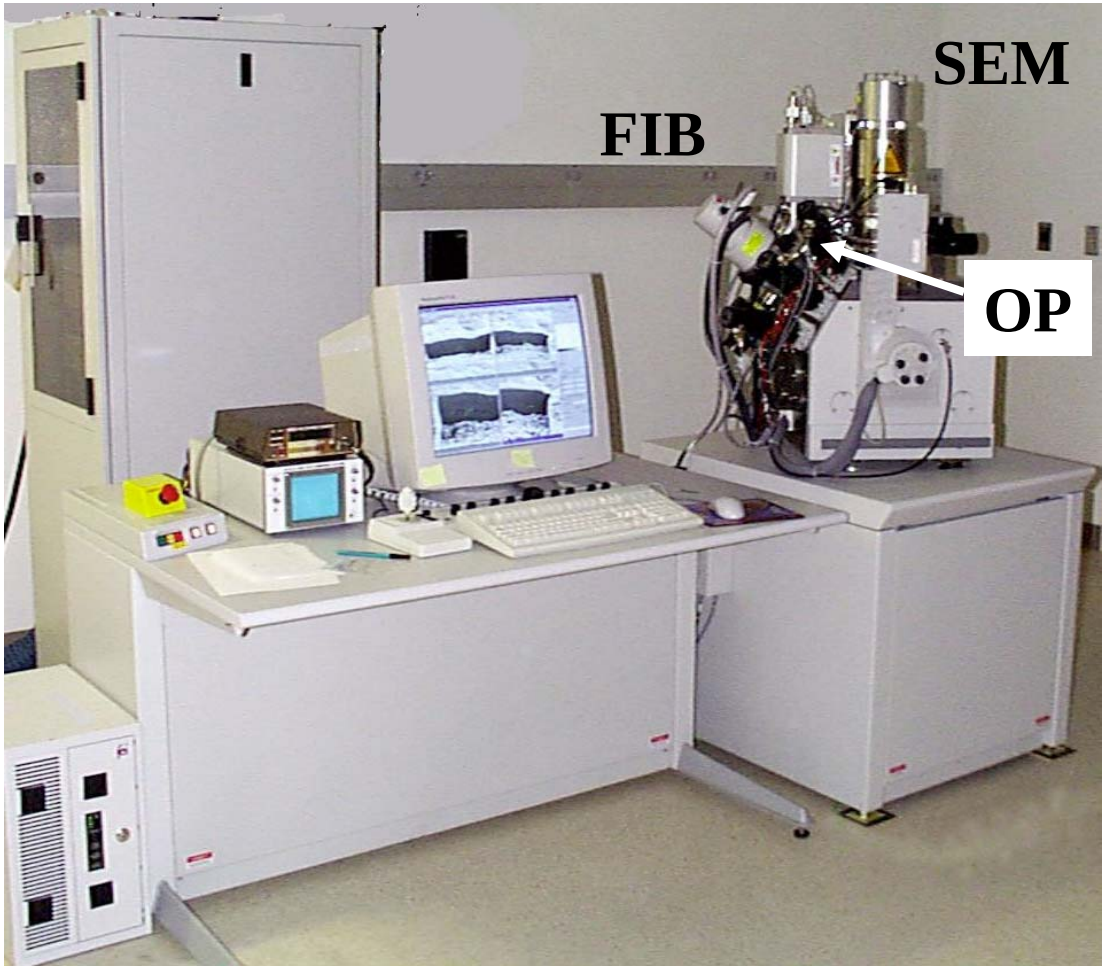
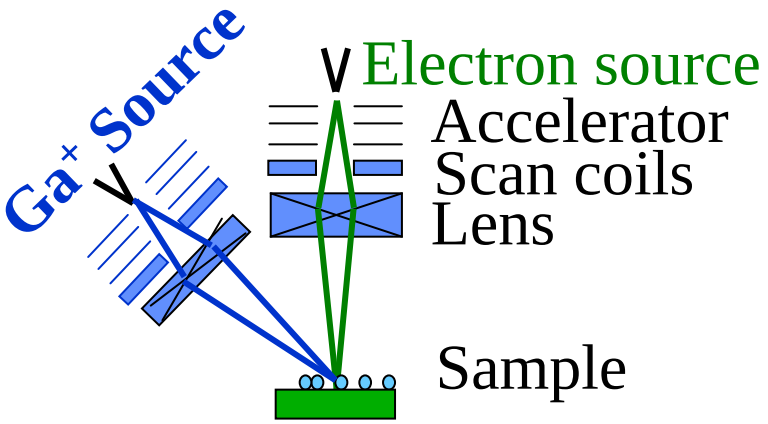
Dual-Beam FIB / SEM / Micromanipulator





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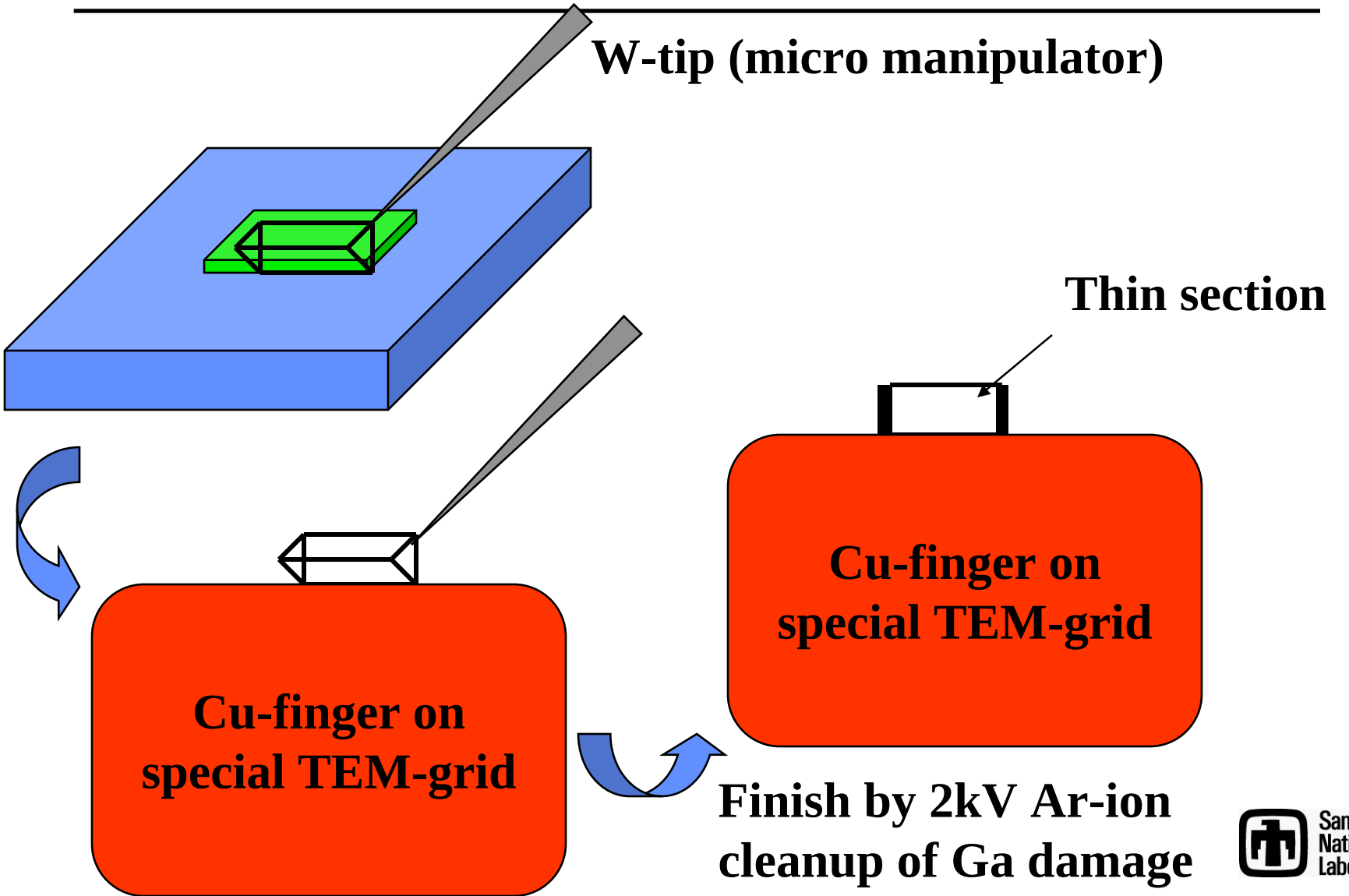
In-situ lift out method



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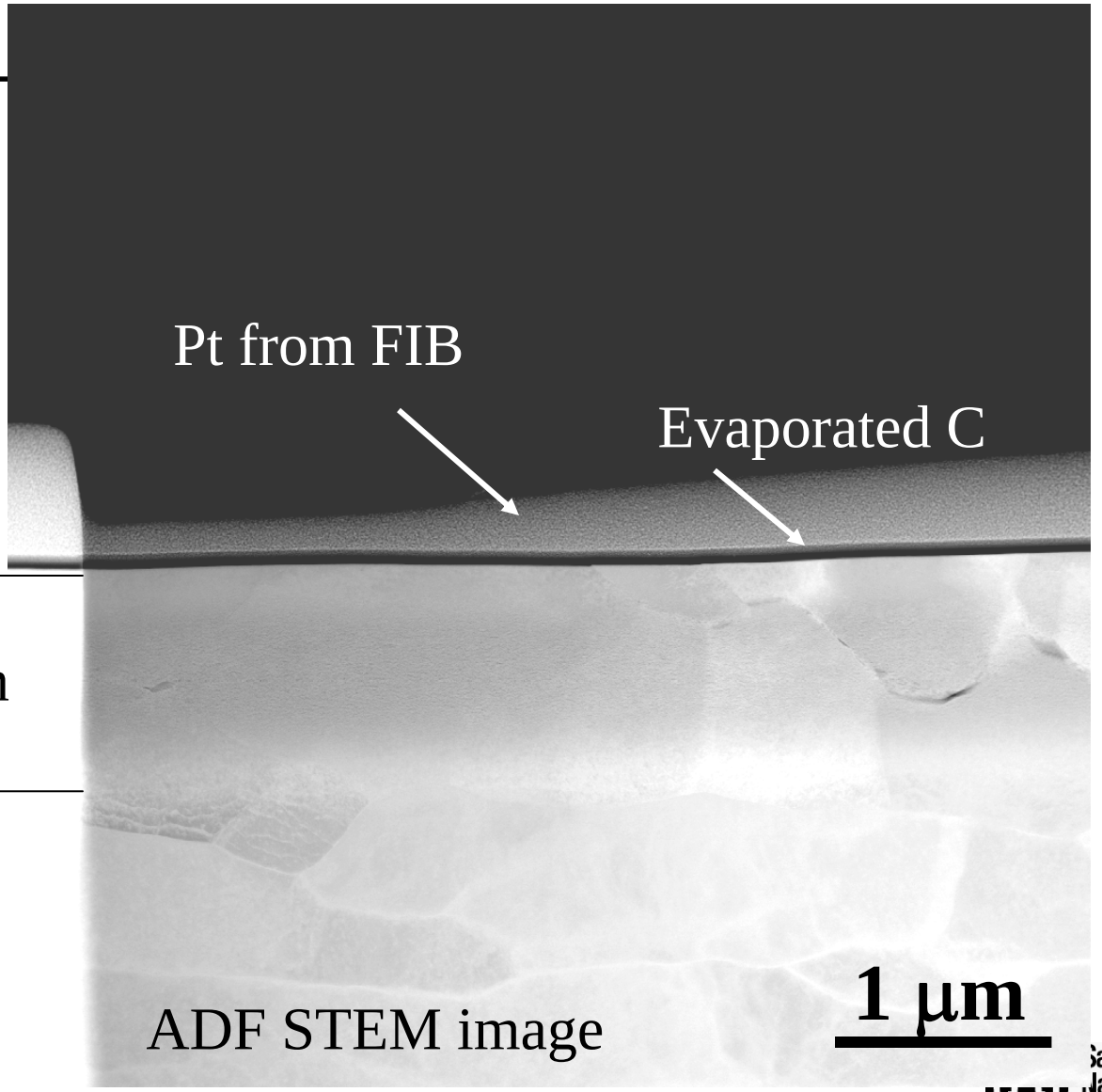
Pd powder prep procedure

- Entire procedure done in a Tritium envelope
- Powder placed in brass tube
- Tube filled with epoxy (Gatan G1, Epotec 353ND)
- Epoxy vacuum infiltrated
- Cured at 50°C overnight or at 100°C for a few minutes
- Sliced/lapped/polished to 100μm thickness
- Dimple polished to 10-20μm
- Argon ion milled to perforation
  - Low angle (4 degrees) ion-milling from both sides until perforation in the center. Finish off with <2kV ions.
- It is unlikely that the electron transparent powder saw any mechanical deformation from the sample prep process.

He-implanted Pd

Pd foil annealed at 1025°C and implanted with 200 KeV He to a He/metal ratio of 0.15



Tecnai F30-ST

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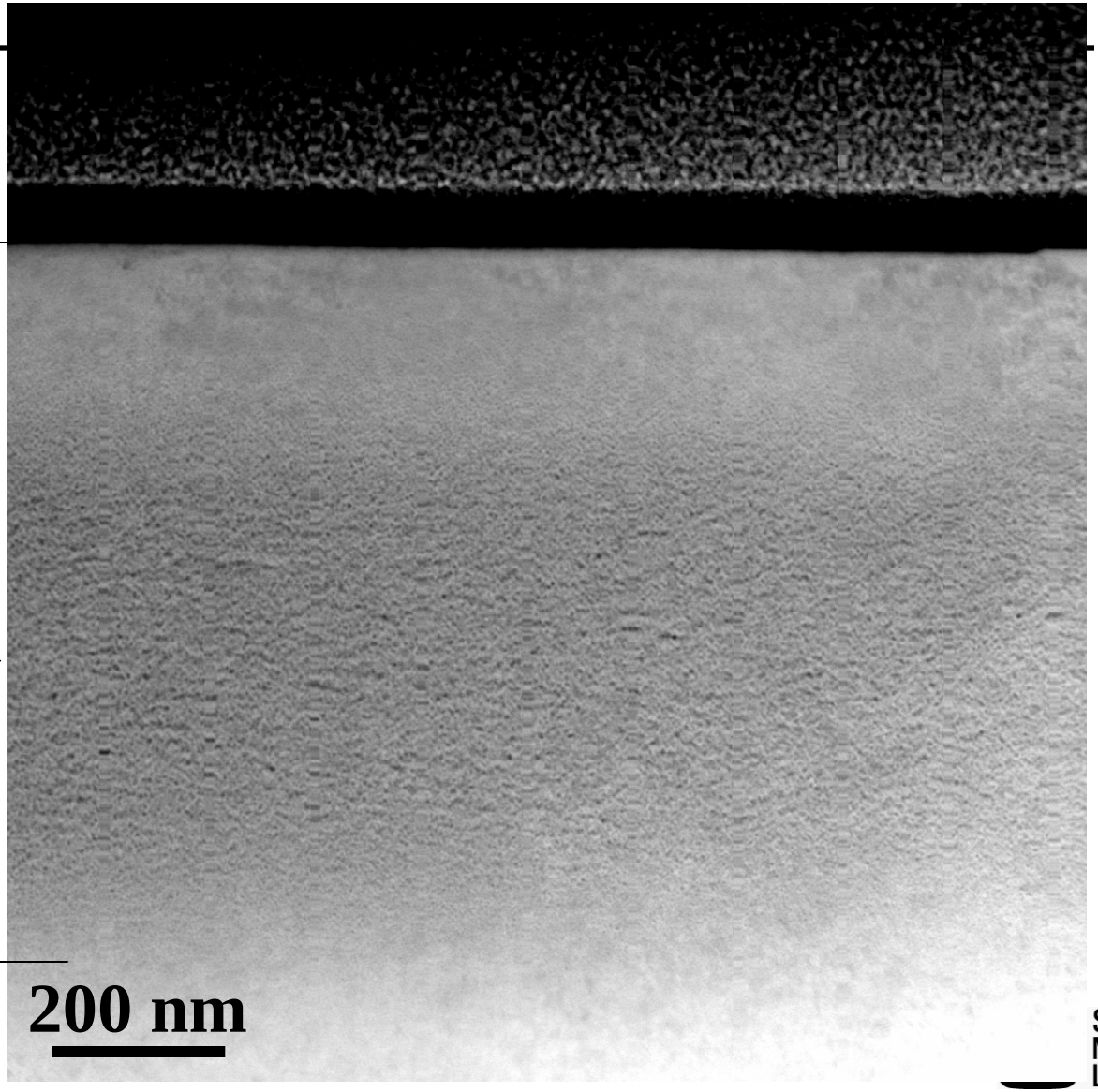
He-implanted Pd

ADF STEM image



Implanted region

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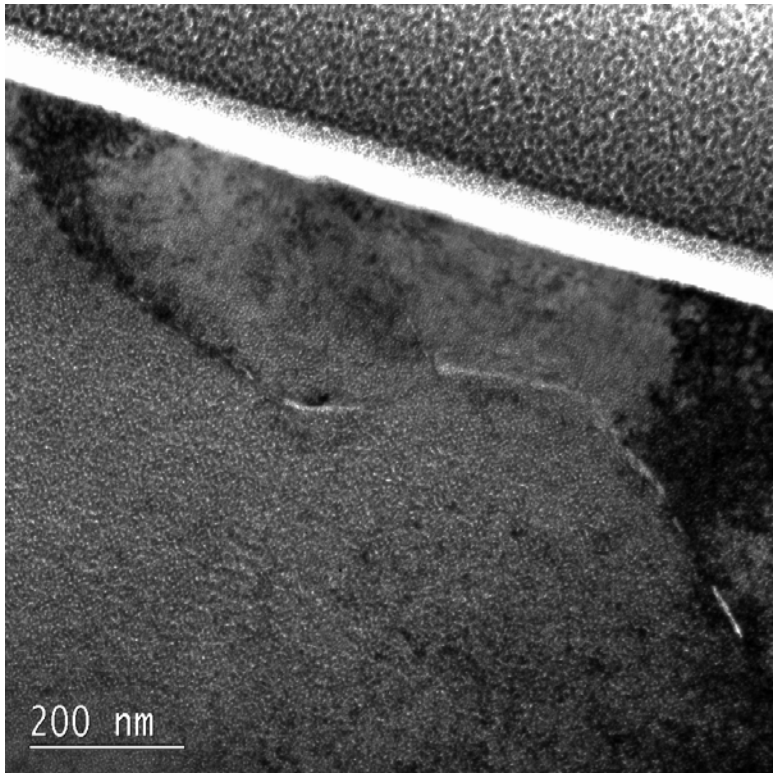
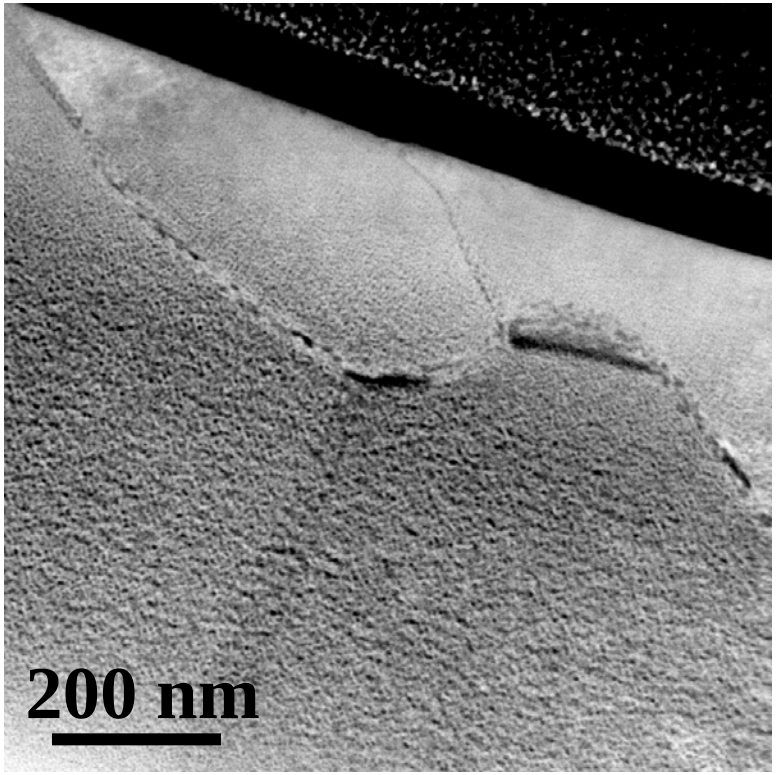
He-implanted Pd

Underfocused BF Image



ADF STEM Image



Note: Coalescence of bubbles on grain boundaries

Tecnai F30-ST

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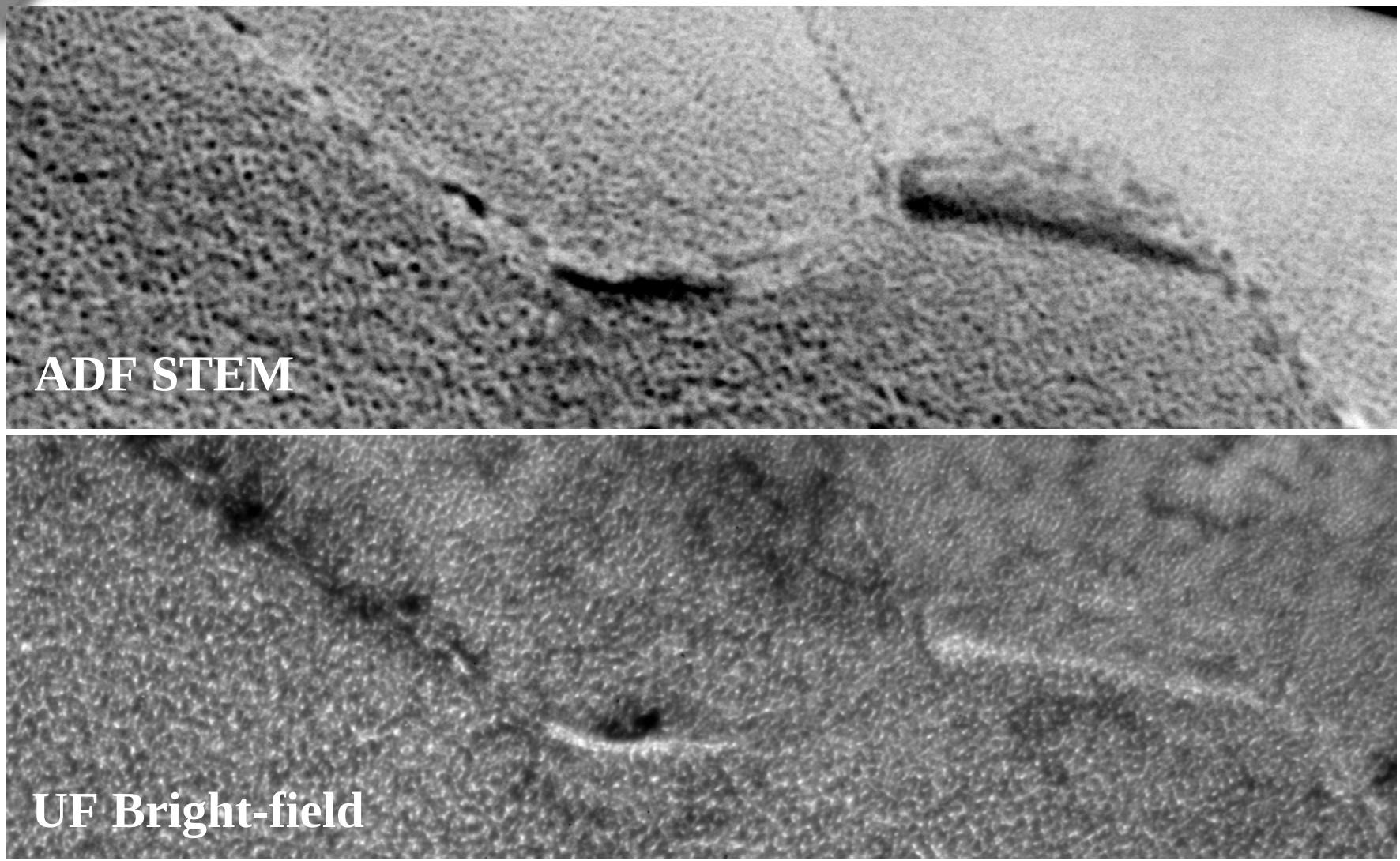
Comparison of ADF-STEM and Underfocus BF Images



Tecnai F30-ST

100 nm

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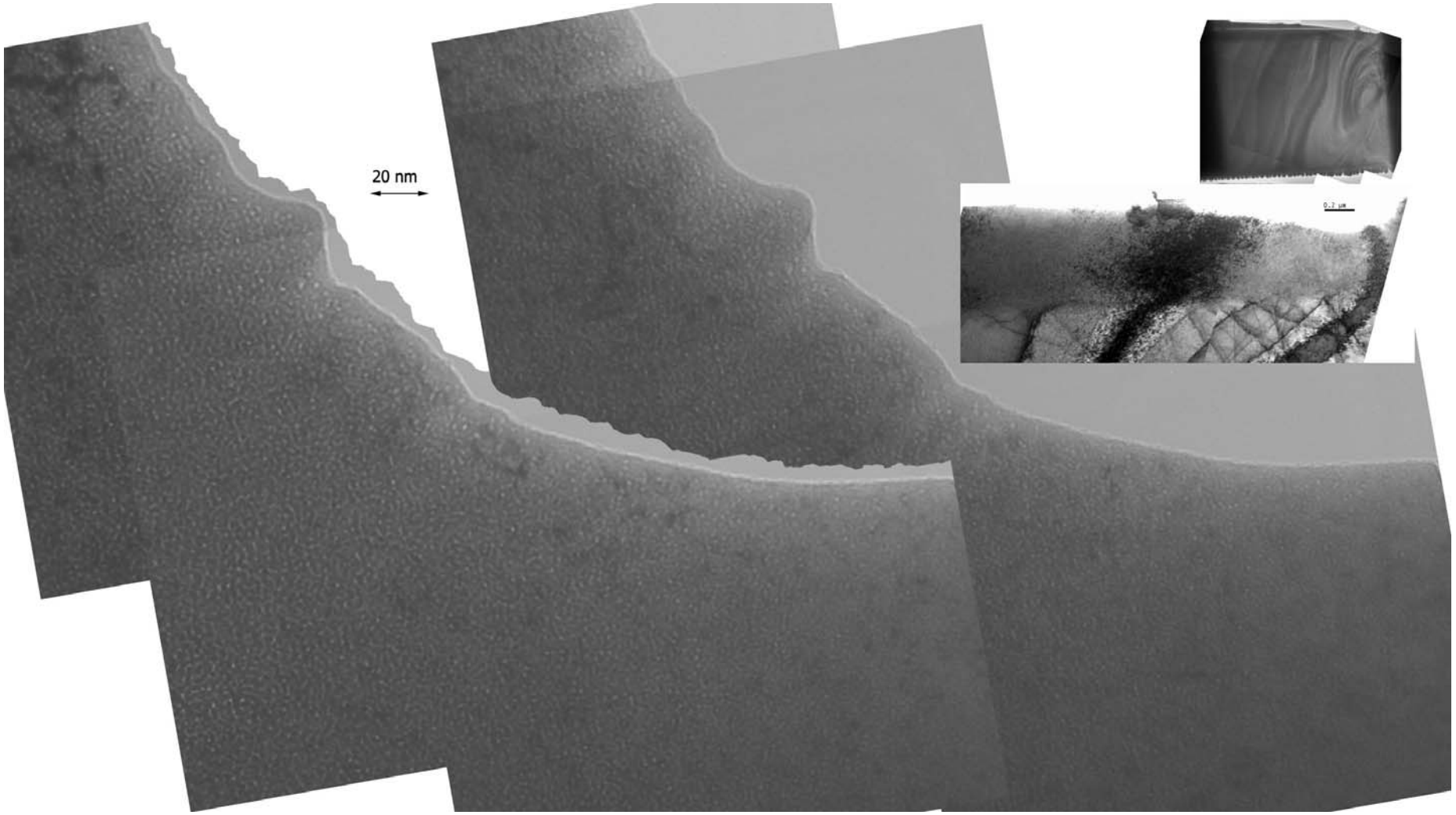
Bubble density measurement in helium injected Pd foils



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Bubble density/swelling in helium injected Pd foils

- Quantification of bubble density/swelling

Area	Depth (nm)	Mean diameter (nm)	Swelling (%)	Density (cm^{-3})
1	130	1.12	0.28	3.65×10^{18}
2	180	1.15	0.23	2.73×10^{18}
3	250	1.17	0.34	3.74×10^{18}
4	310	1.29	0.46	3.94×10^{18}

Therefore, TEM results are in agreement with previous measurements.

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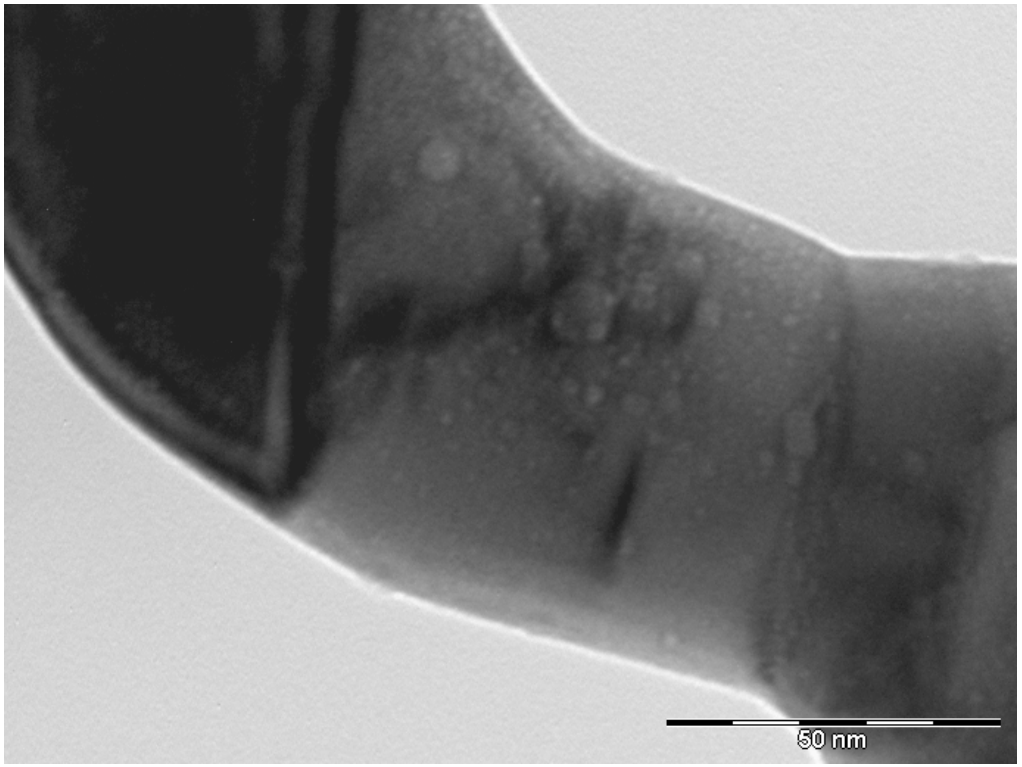
First Observations of Tritided Pd powder

~1-10nm bubbles



Distribution of bubble sizes observed

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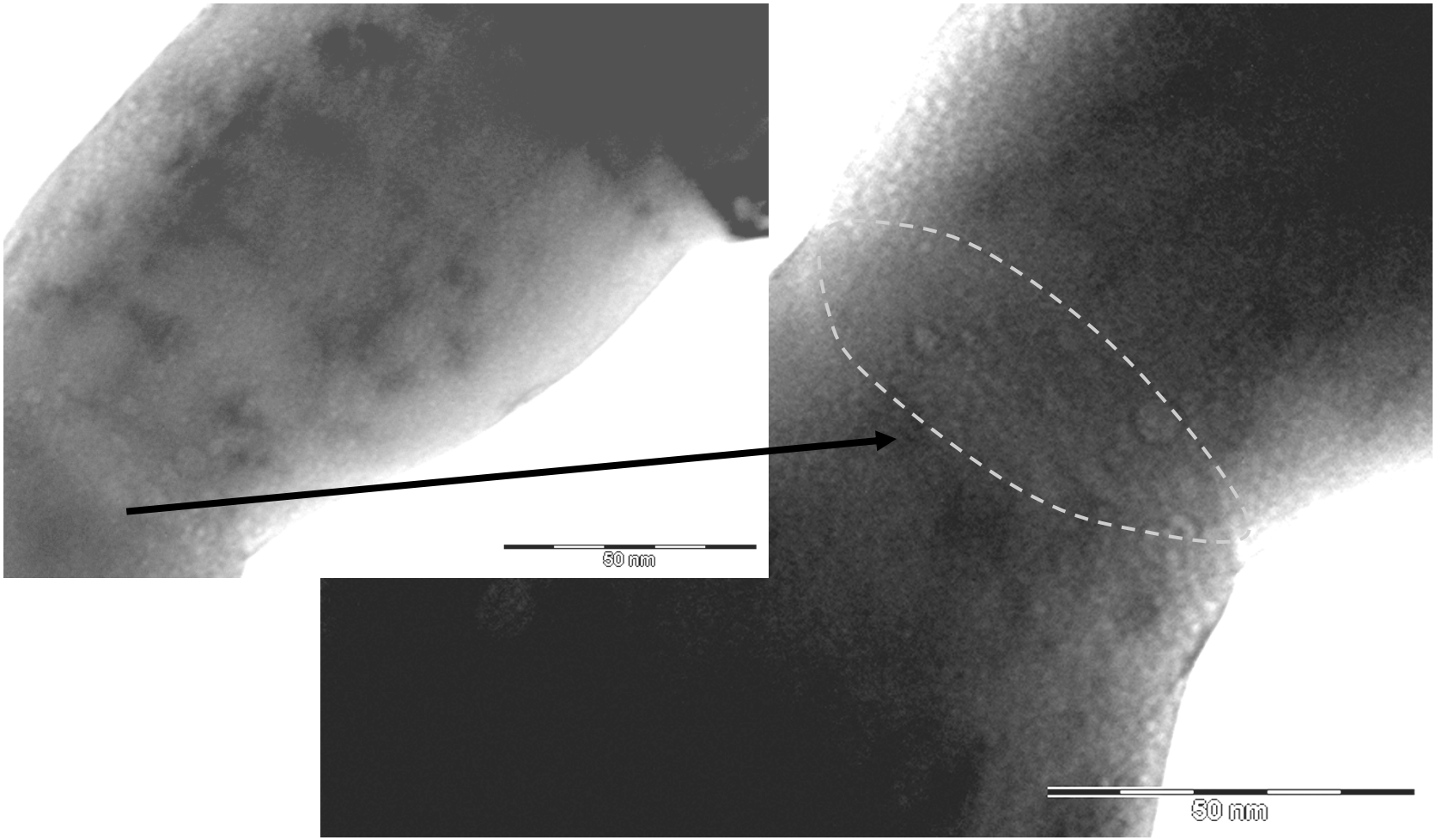
Larger bubbles observed on grain boundaries



Distribution of bubble sizes
Larger bubbles on grain boundary

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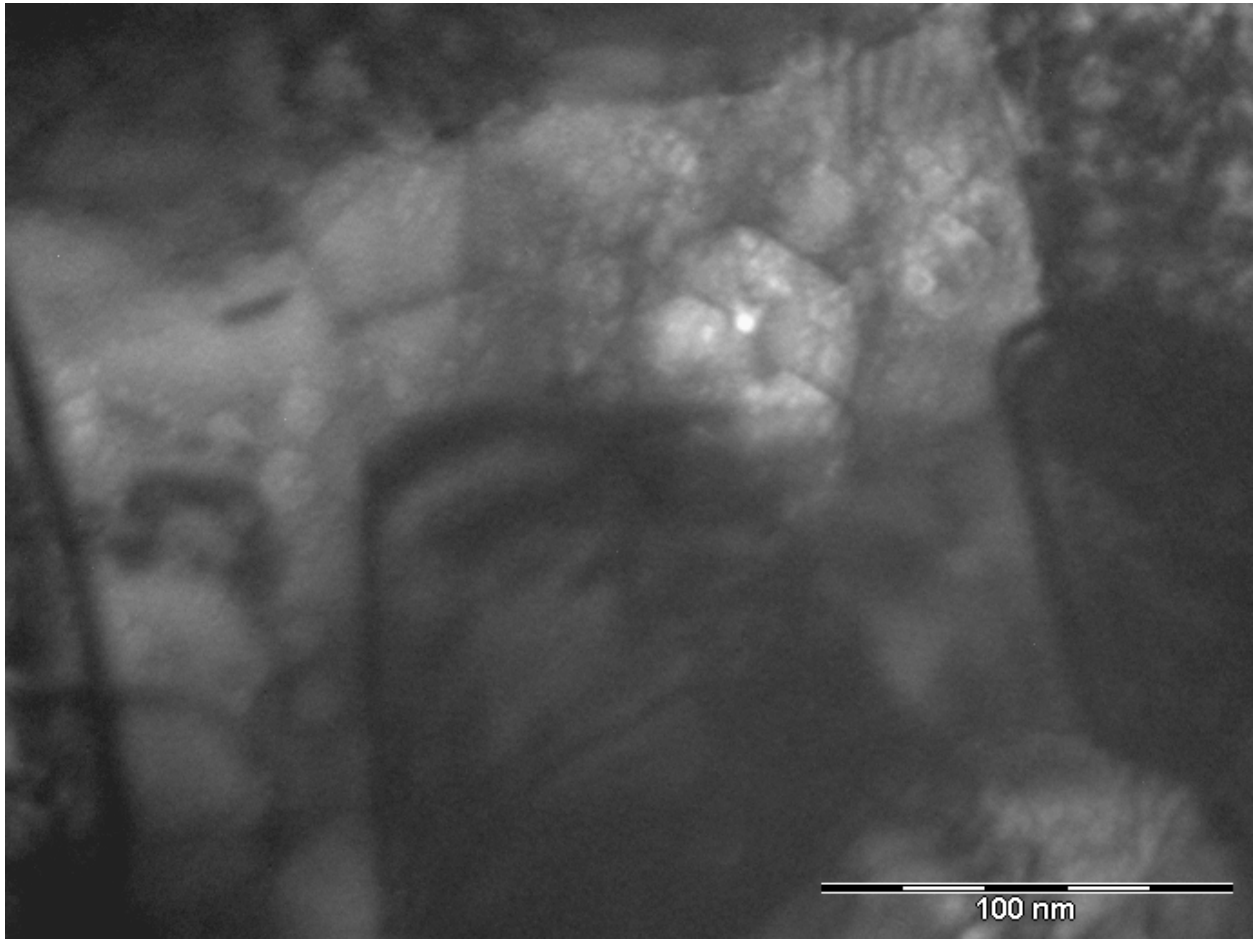
Apparent faceting of bubbles in thicker regions



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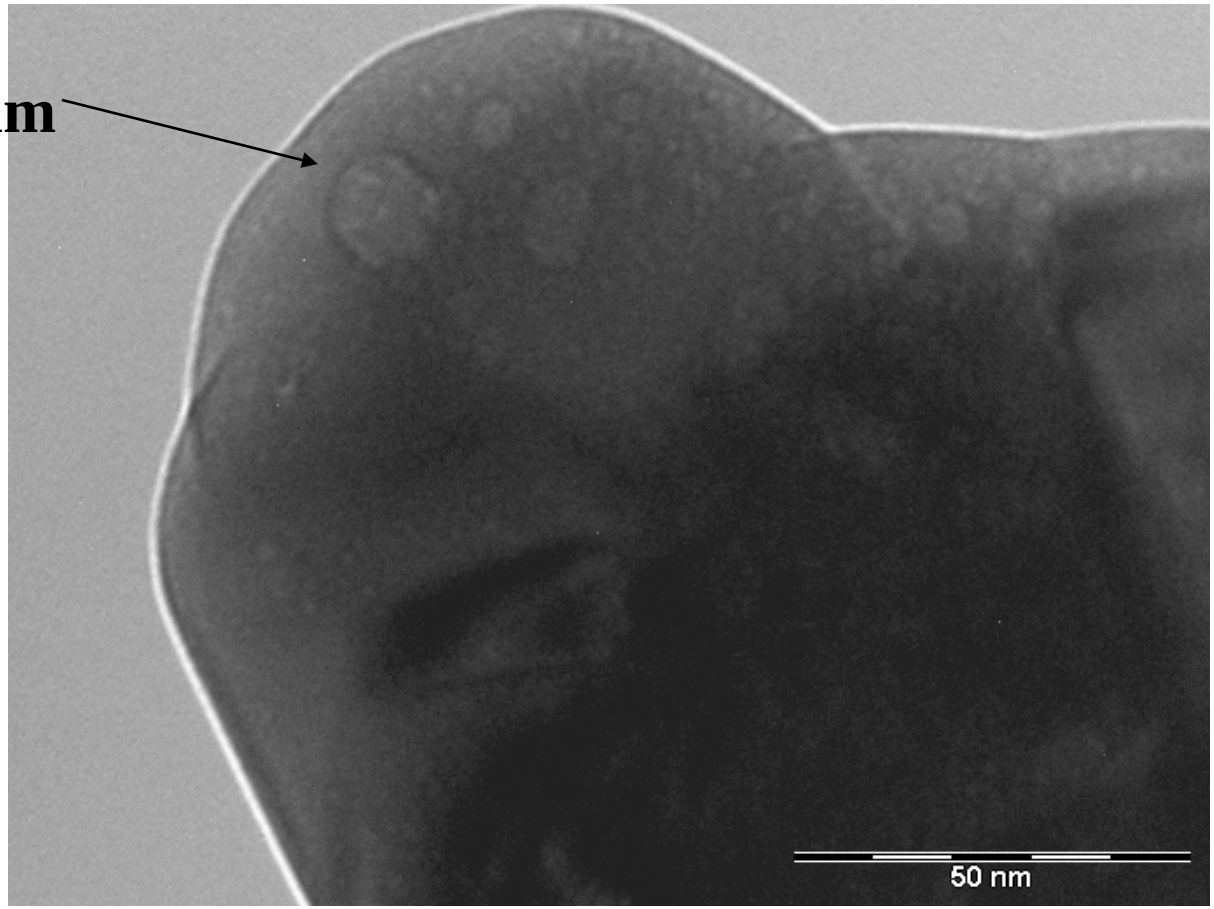
Bubble size distribution



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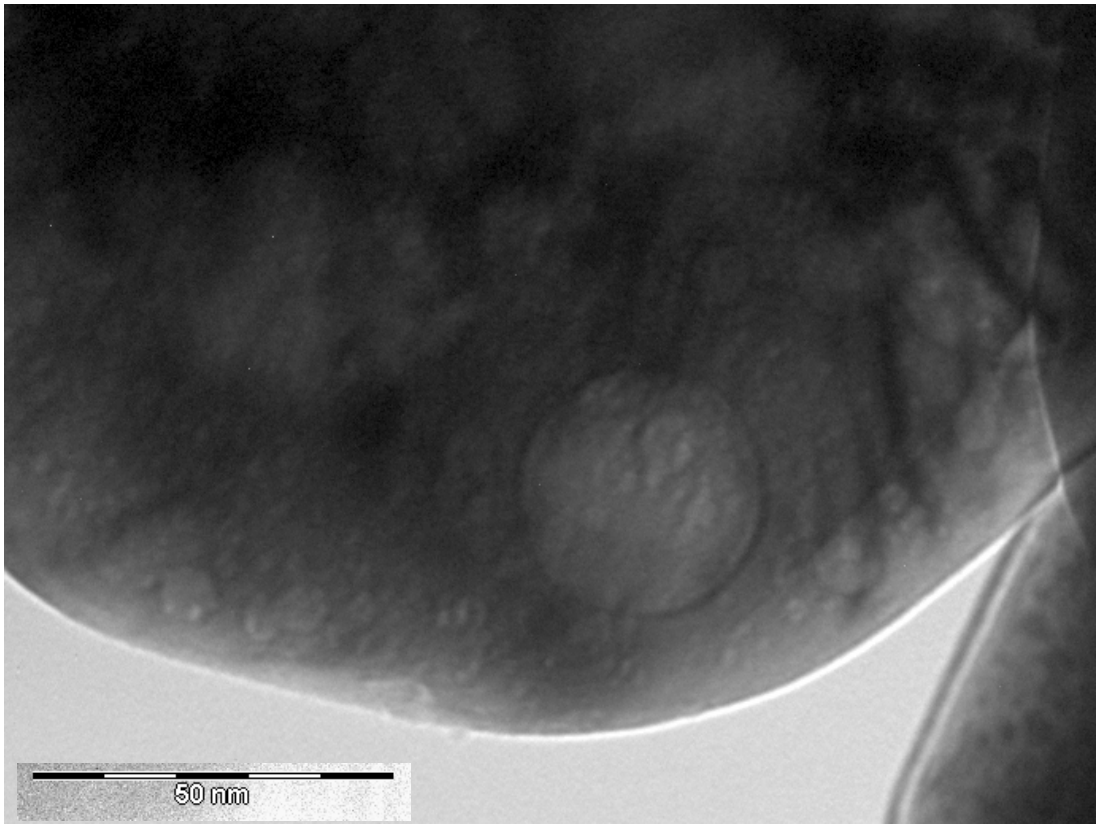
Very large bubble



30nm bubble

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Conclusions

- FIB (in-situ lift out, low kV Ar-ion milling) can be used effectively to prepare high-quality TEM sections of FCC metals with nm-sized bubbles
  - Less chance of having contamination issues
  - Much faster specimen preparation
  - Bubble density measurements
- “Powder in tube” also an effective method for preparation of tritided powders
- Tritided Pd
  - Low kV Ar-ion final thinning essential
  - Distribution of bubble sizes 1-30nm in a limited sampling with some faceting and large bubbles on some grain boundaries
  - Larger bubbles due to coalescence?