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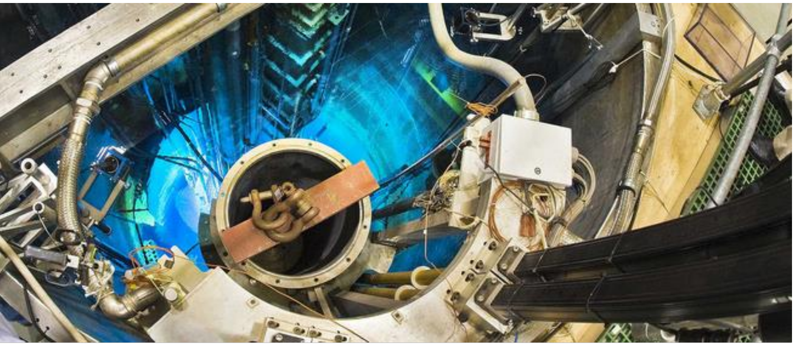
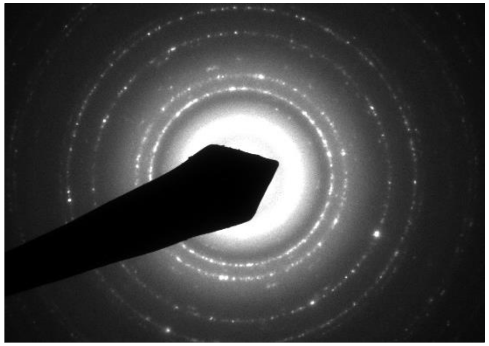
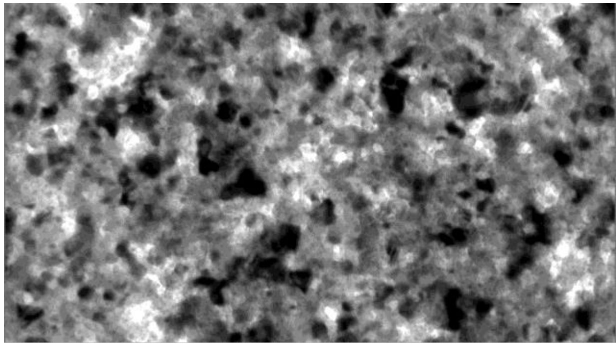

Nanodispersed Cu-Nb for enhanced radiation tolerance

Presented by:

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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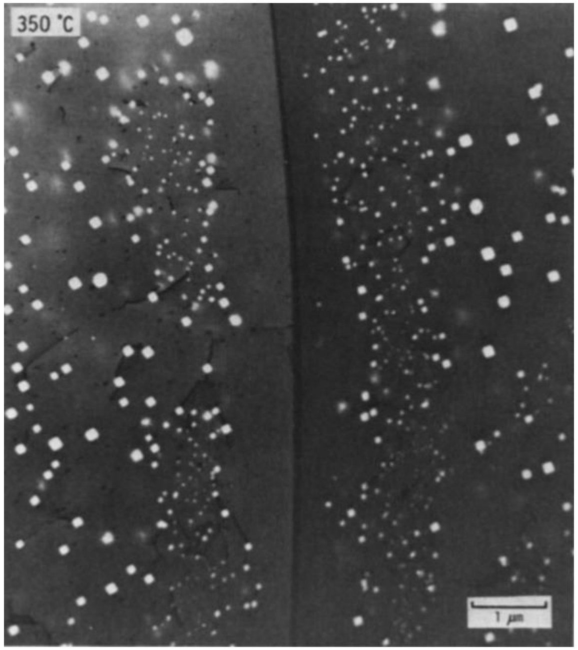
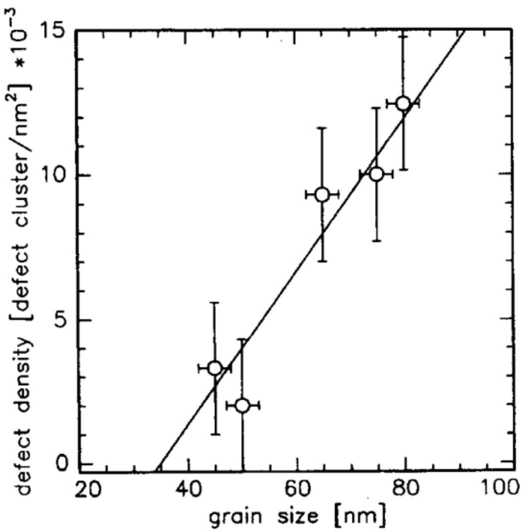
Motivation

- Nanostructured materials are of general interest to the nuclear materials community for improving radiation tolerance
  - High densities of features and interfaces serve as sinks for point defects resulting from radiation damage
- Cu-Nb serves as an idealized system for studying these systems
  - Cu (fcc) & Nb (bcc) are immiscible, allowing for study of ideal interfaces without potentially complex intermediate phases or chemical effects
- Most work to date has been performed on nanolayered Cu-Nb
  - Nanodispersed or nanograined Cu-Nb geometries are more relevant to real systems (ODS, UFG)



Void-denuded zone in Cu
(Neutron-irradiated, 350 °C)

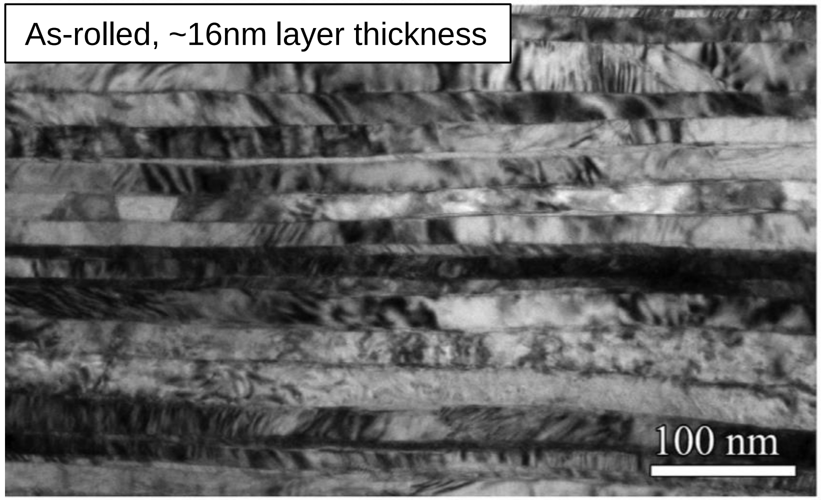


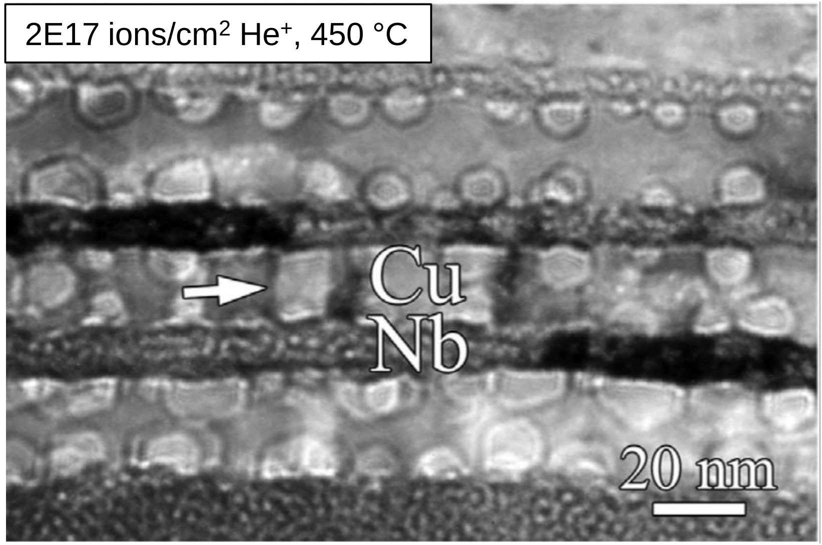
Defect densities vs. grain size in irradiated Pd
(240 keV Kr, 2E16 ions/cm^2)

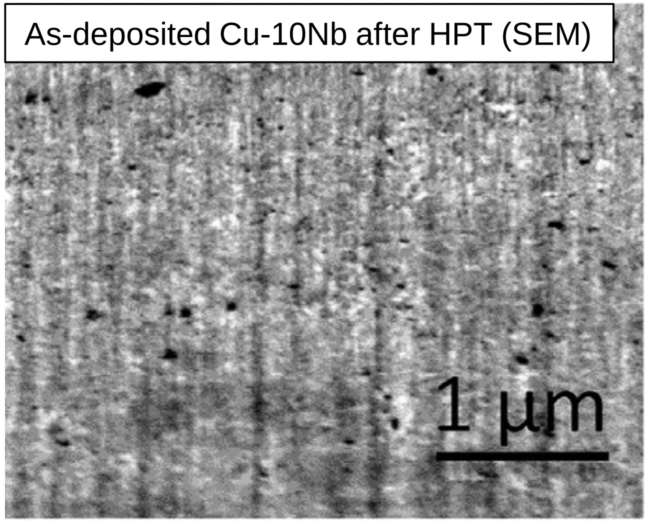
[1] Zinkle, S.J., et al. Void swelling and defect cluster formation in reactor-irradiated copper. *Journal of Nuclear Materials.* **168** (1989) 262-267.
[2] Rose, M., et al. Instability of irradiation induced defects in nanostructured materials. *Nuclear Instruments and Methods in Physics Research B.* **127/128** (1997) 119-122.

Previous studies of radiation tolerance in Cu-Nb

- Many studies of radiation damage and He implantation in Cu-Nb nanolaminates
  - Usually produced by accumulated roll-bonding (bulk) or alternating deposition (thin films)
  - Have demonstrated He bubble growth is arrested by these interfaces
- Averback group @ UIUC has had success preparing nanodispersed Cu-Nb specimens via HPT and has shown phase separation under irradiation, but little is reported on the resulting radiation tolerance







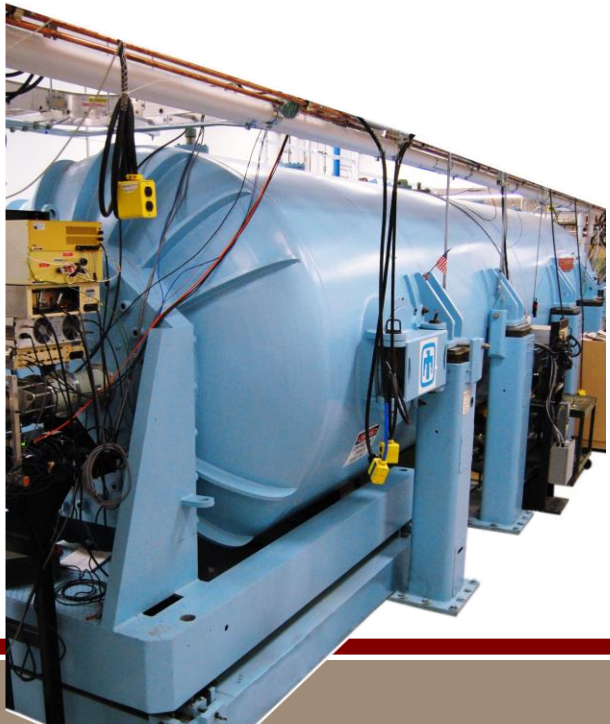
[1] Yang, L.X., et al. Effects of He radiation on cavity distribution and hardness of bulk nanolayered Cu-Nb composites. *Journal of Nuclear Materials.* **487** (2017) 311-316.
[2] Beach, J.A., et al. Self-organized, size-selection of precipitates during severe plastic deformation of dilute Cu-Nb alloys at low temperatures. *Acta Materialia.* **140** (2017) 217-223.

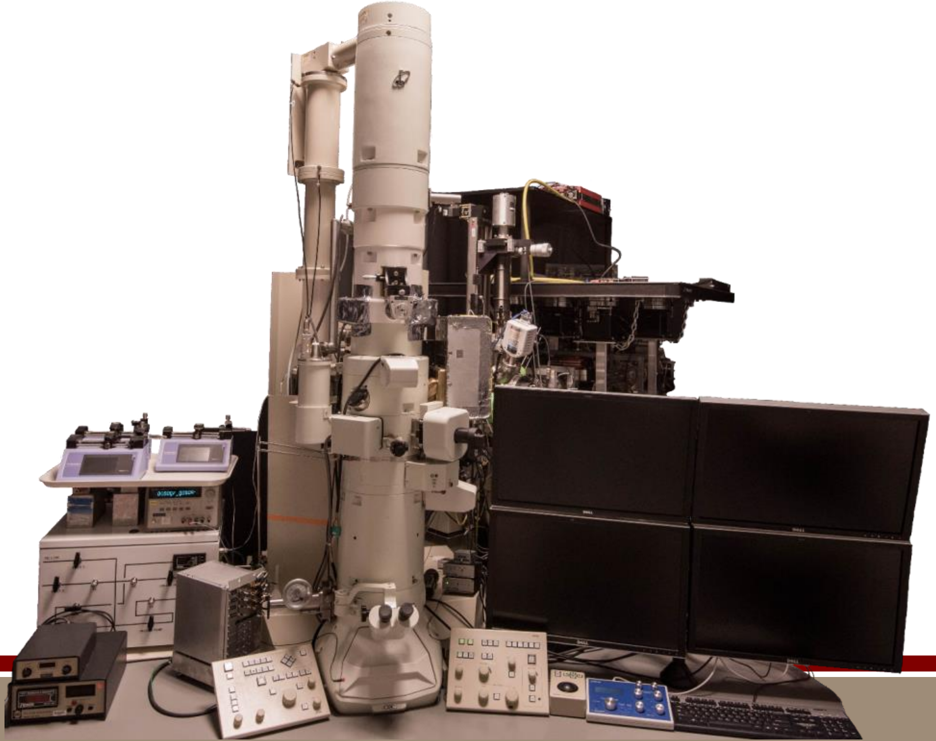
Experimental Goals

- Generate nanodispersed Cu-Nb thin films with varied compositions
  - Characterize and optimize microstructures for a given condition
- Irradiate films with heavy ions and characterize damage microstructures
- Implant films with He and characterize resulting bubble microstructures

Experimental

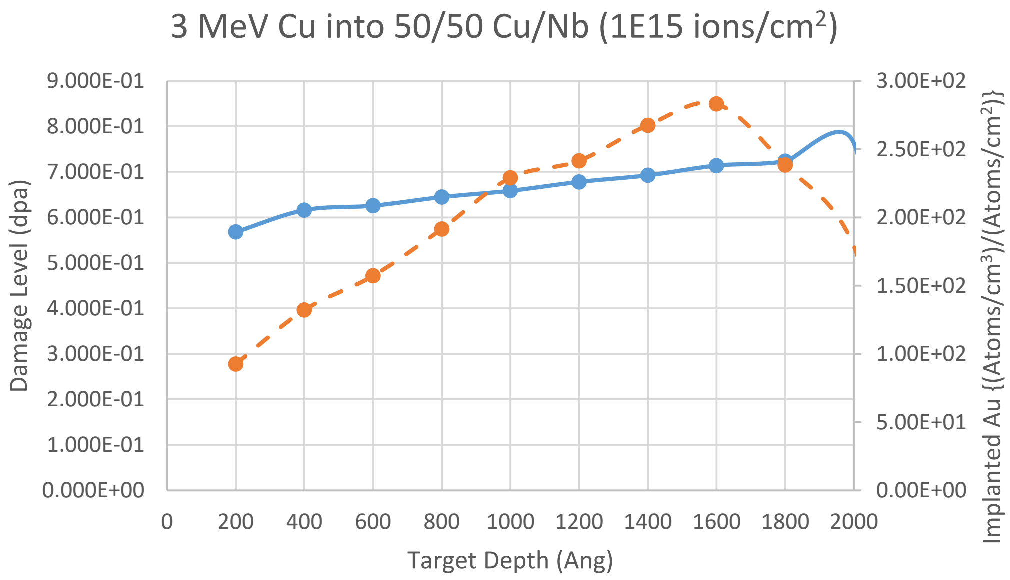
- Cu-Nb thin films with various atomic ratios have been created through magnetron sputtering at RT
  - Either single crystal NaCl or Si_3N_4 window grid substrates
- As-deposited films were either vacuum annealed or irradiated at temperature to encourage recrystallization
  - 500 °C anneal, or 3 MeV Cu^{++} to 1 dpa @ 350 or 500 °C

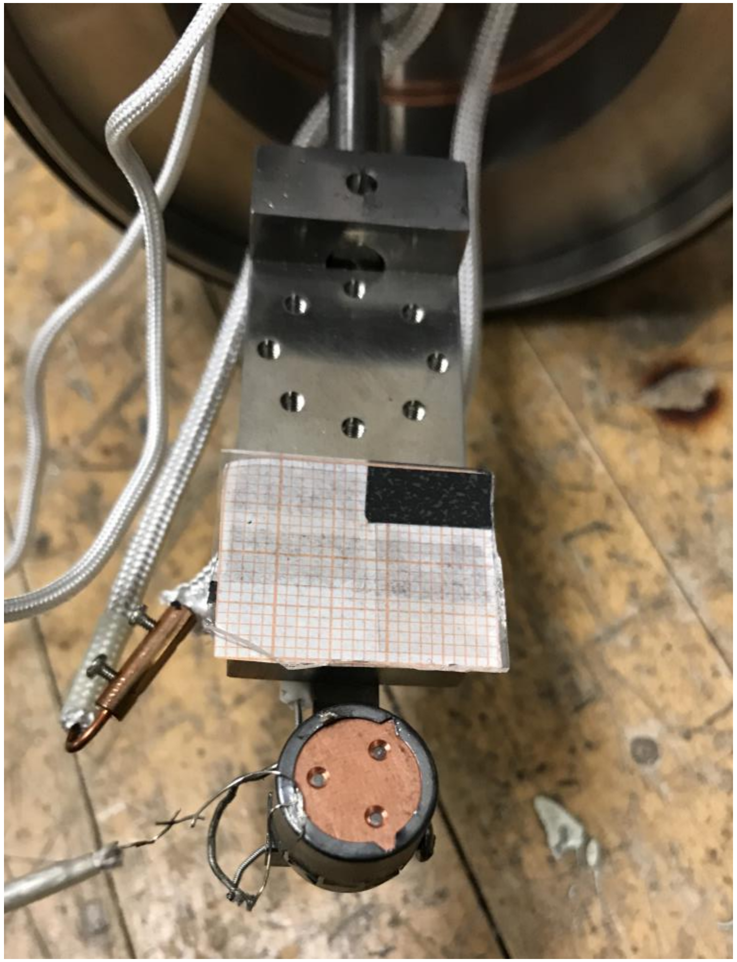




Ion Irradiation Calculation

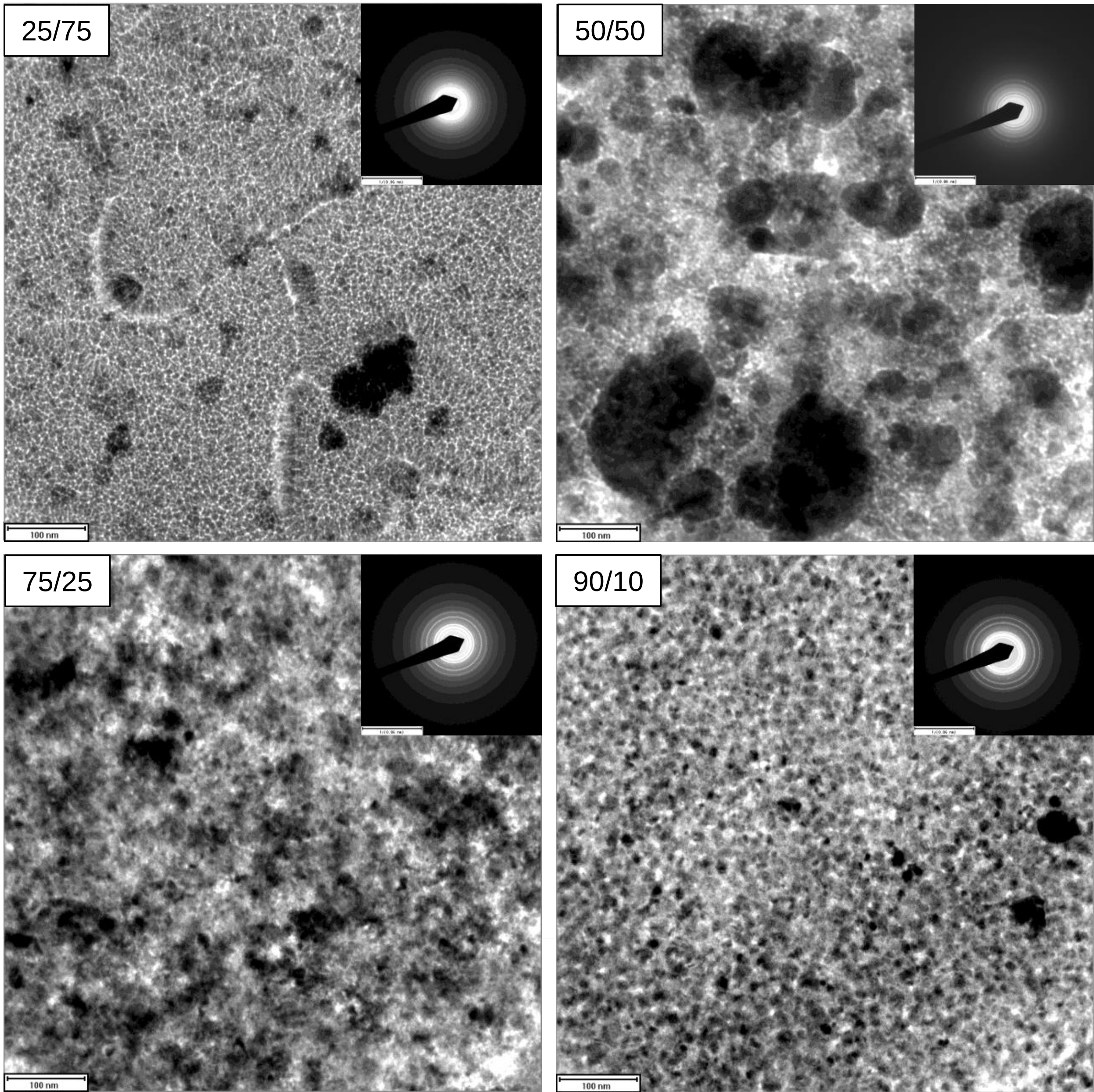
- SRIM was used to calculate damage in specimens
  - 1.61E15 ions/cm^2 per dpa
- Specimen temperature was maintained using LabView-controlled button heater



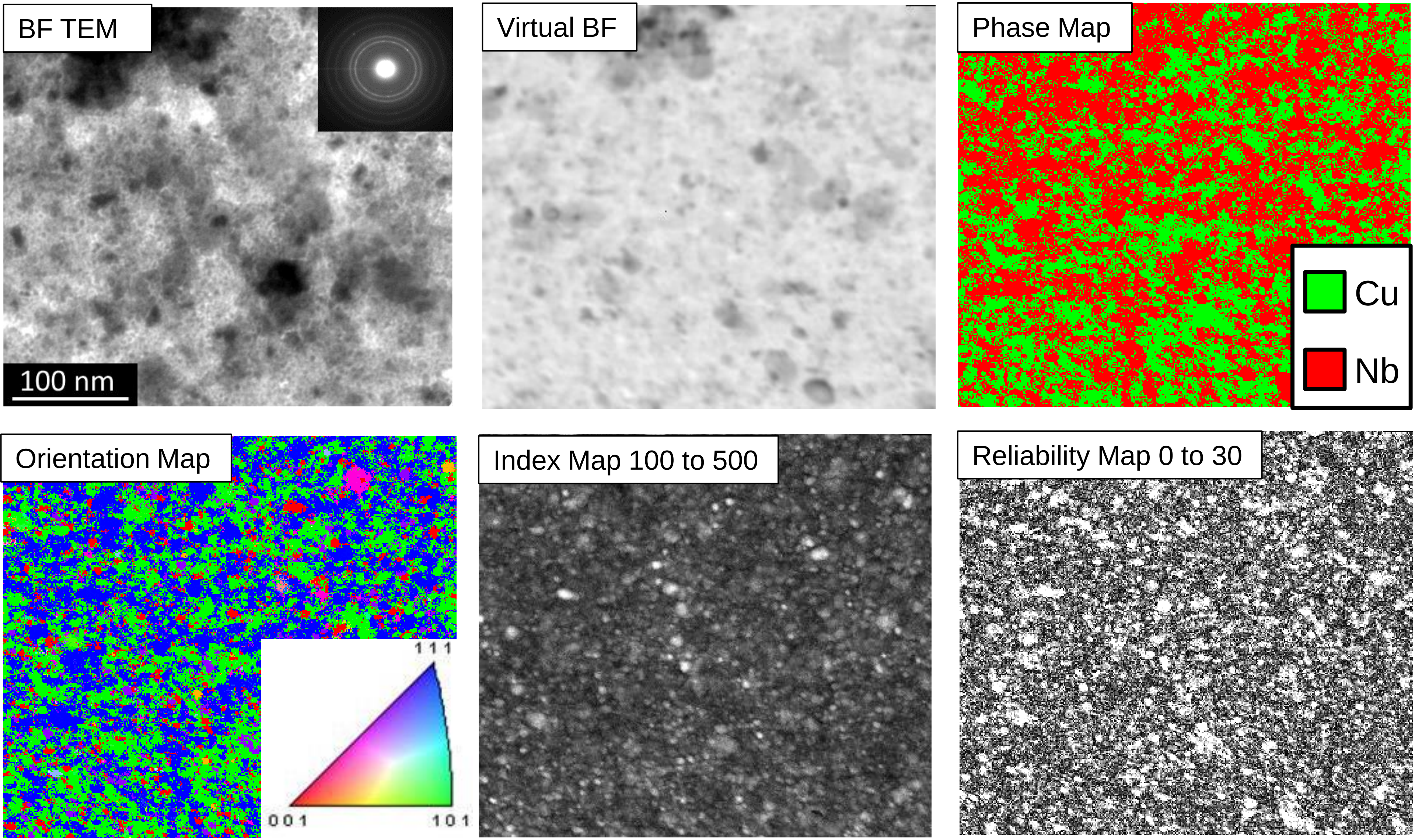


As-deposited Microstructures

- Microstructures appear porous
- Increasing crystallinity with increasing Cu content

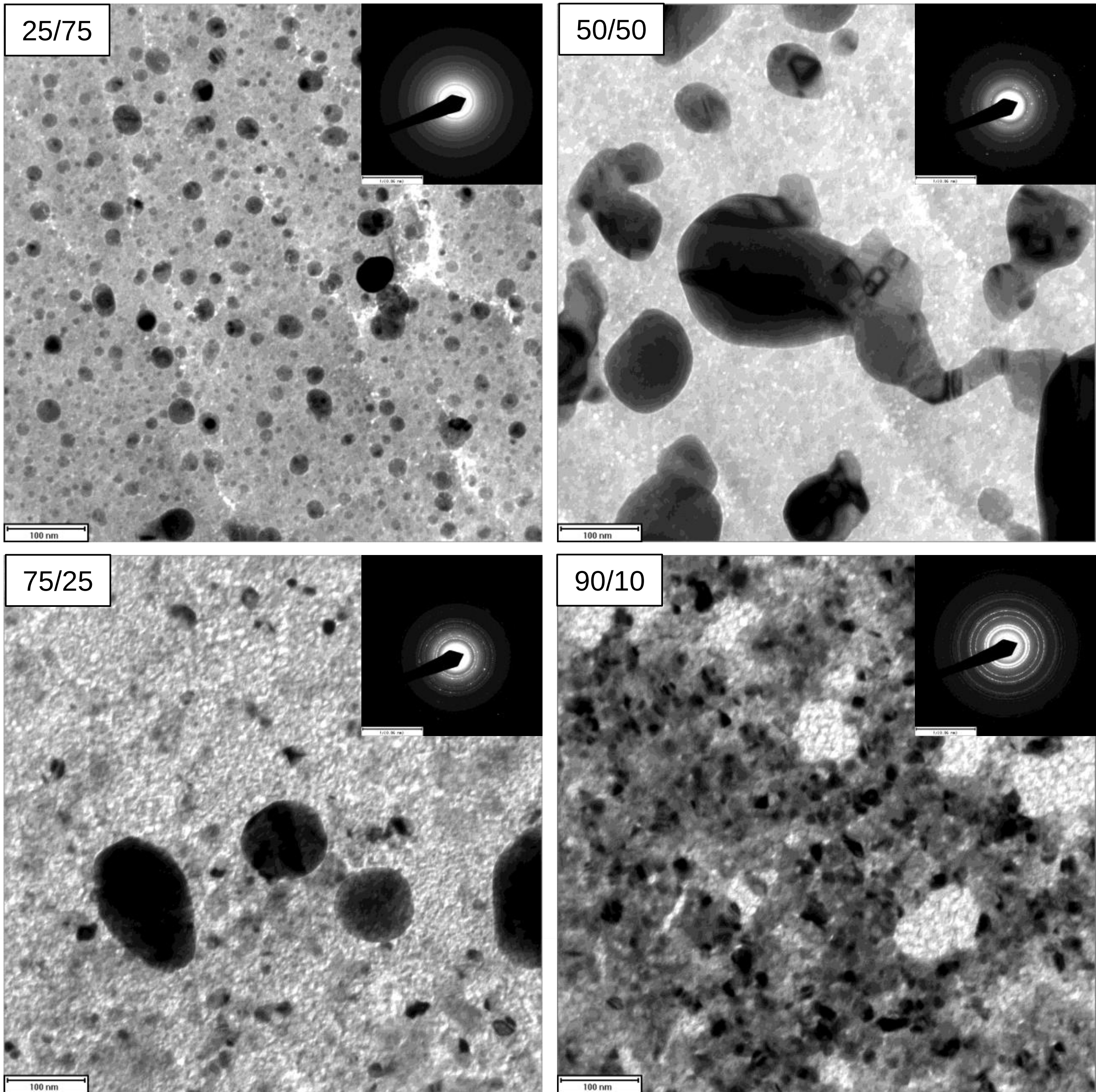


PED of As-Deposited Cu-10Nb



Annealed Microstructures (1hr, 500 °C)

- Annealing results in Cu islands on a mostly amorphous Nb substrate
- Size of islands varies dramatically with composition
- Porosity is prevalent on Nb "substrates"

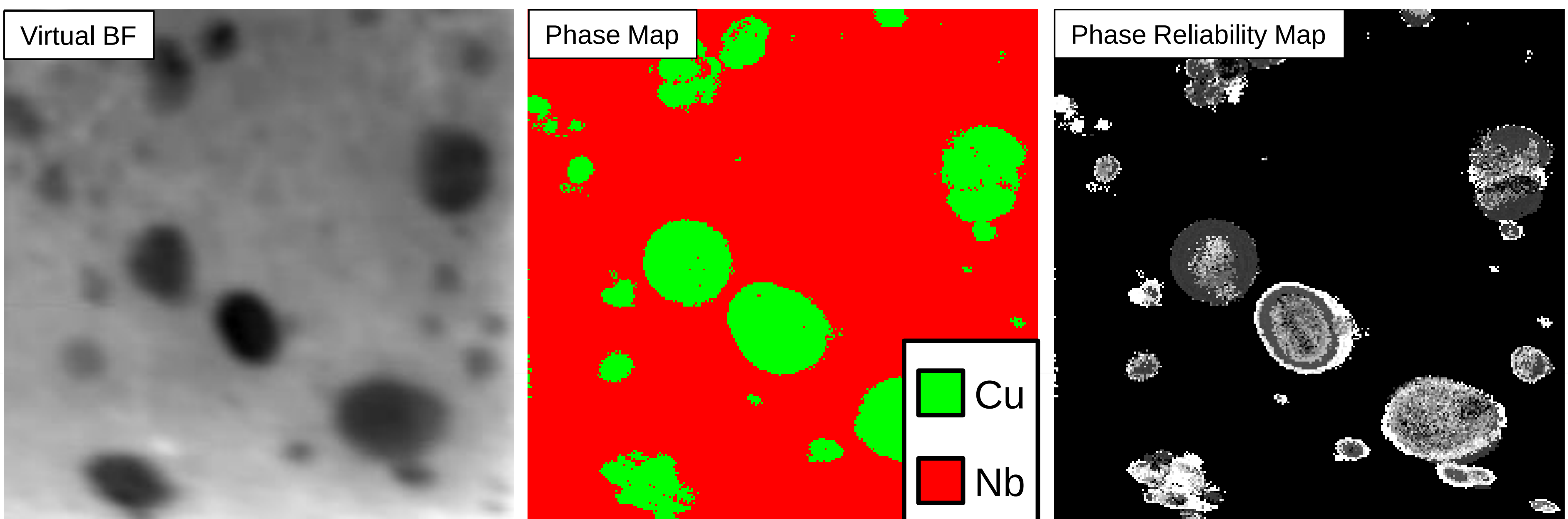


PED of Annealed Microstructures

- PED of annealed specimen confirms that dark contrast is a Cu phase.
- Most patterns corresponding to Nb phase actually appear amorphous.

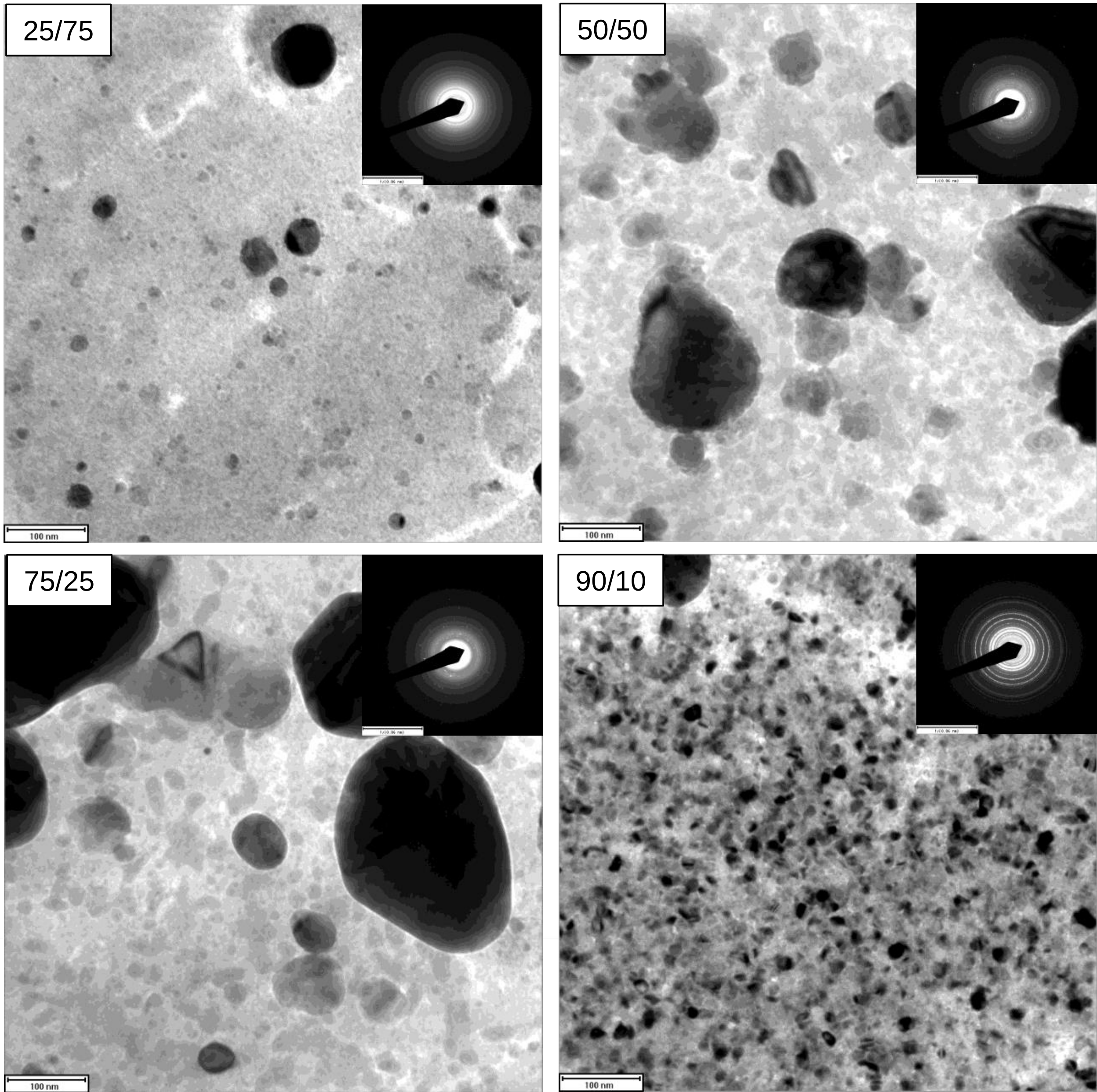
25/75 Cu/Nb on NaCl

Annealed, 500 °C, 1 hr



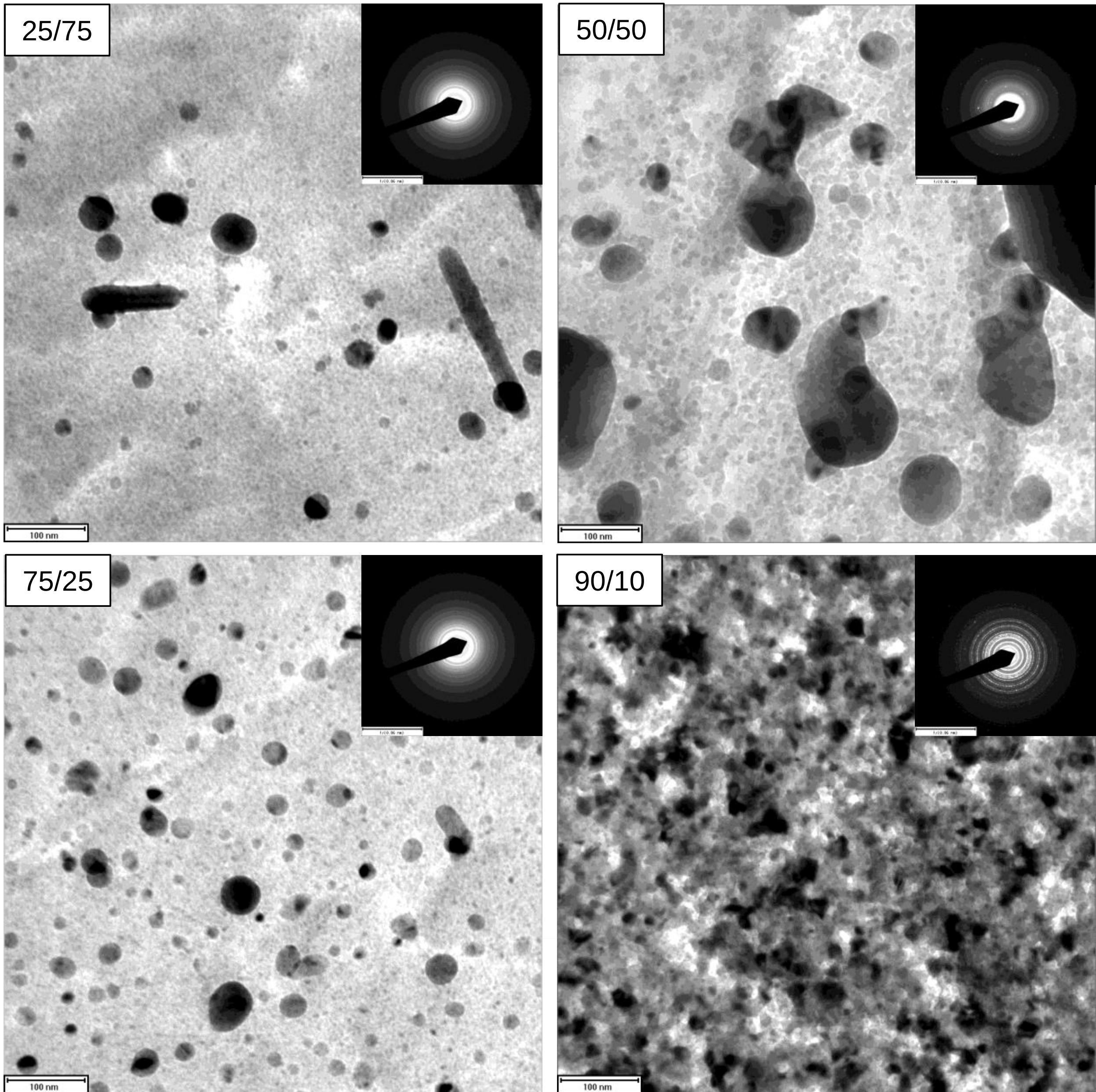
Irradiated Microstructures (1 dpa, 350 °C)

- Irradiation at a lower temperature yields similar structures to higher temperature anneal



Irradiated Microstructures (1 dpa, 500 °C)

- Irradiation at a higher temperature results in more uniformly sized Cu islands (90/10 exception)
- Still not enough to recrystallize Nb



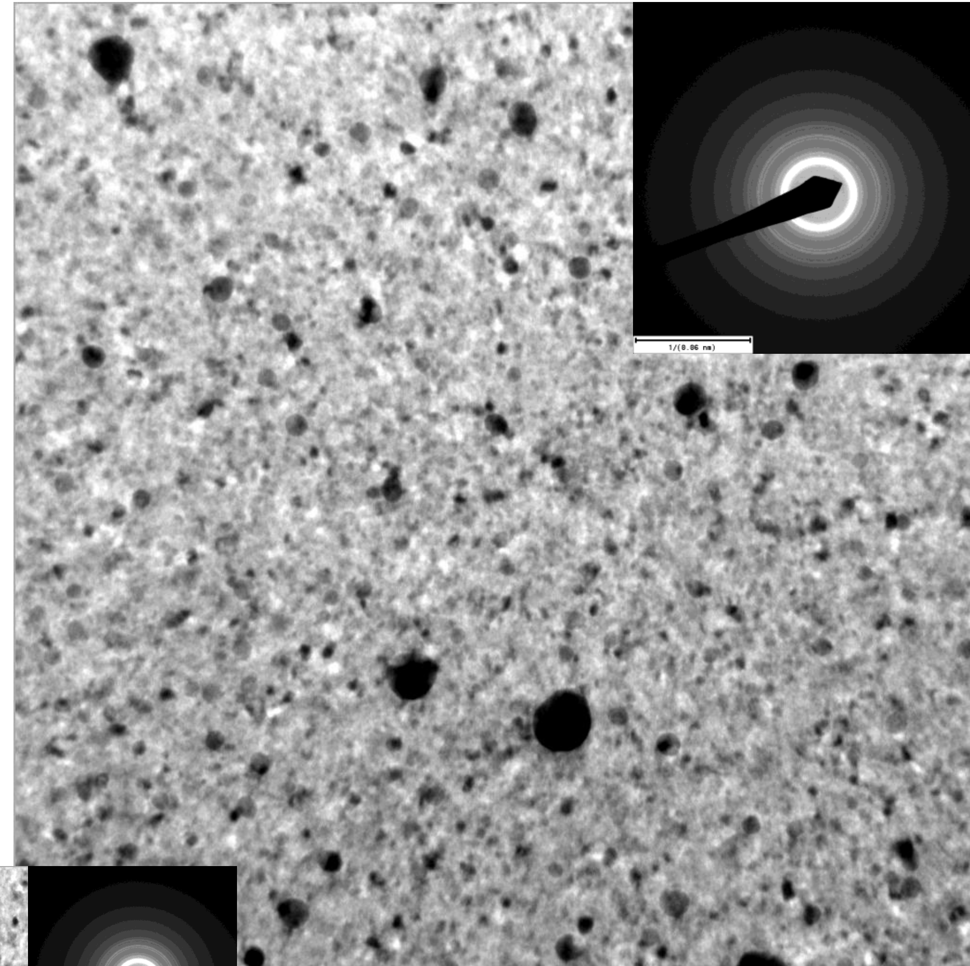
Discussion

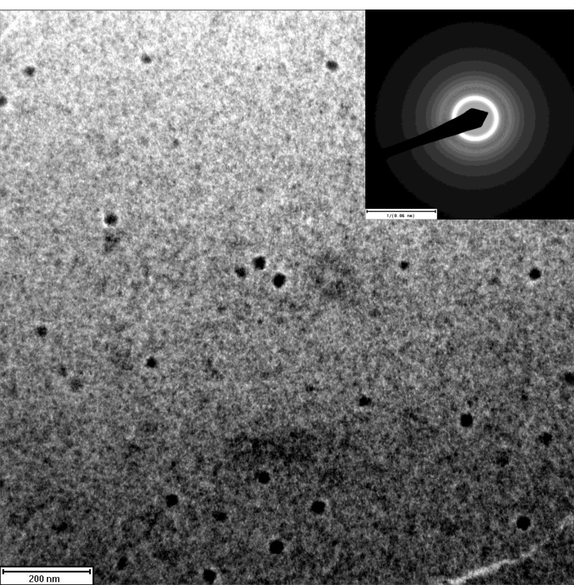
- Due to drastically different melting temperatures and thin-film geometries, Cu appears to be "wetting" to the surface and organizing into individual precipitates/crystals
- Nb substrate remains immobile and amorphous, but contains nanometer-sized pores
  - Smaller than those seen using de-alloying, may still be of use to study nanofeatured materials for radiation tolerance
- This effect not previously observed in nanolayered materials – may be due to higher stresses in co-deposited films
- Open to other hypotheses…

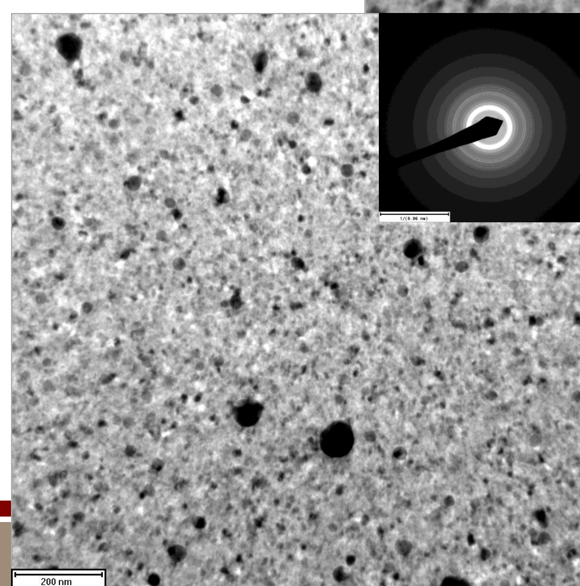
In-Situ Anneal

- 50/50 Cu/Nb on Si_3N_4 grid
- Ramp rate of 30 °C/min (14 mins)
- Held at 450 °C for 10 mins

- Smaller Cu phase precipitates observed to start growth at ~120 °C
- Handful of precipitates begin to coarsen at ~200 °C
- Small grains begin to recrystallize with continued annealing at 450 °C







Next Steps

- Further in-situ annealing & irradiation may help elucidate mechanism of microstructural evolution
- Pursue bulk sample preparation via cryo-ball milling
  - May be able to avoid surface wetting effect by using bulk materials
- He bubble distribution following implantation still of interest
- Eventual neutron irradiations in ACRR planned

Summary & Conclusions

- Microstructural optimization of various co-deposited Cu/Nb films was attempted using annealing and irradiation treatments (up to 500 °C)
- Cu was observed to wet and coalesce on the film surface while Nb remained immobile became porous
- Observed behavior, previously unobserved in nanolayered materials, is likely due to high stresses inherent in co-deposited materials
- Future efforts are pursuing cryo-ball milling to avoid these surface effects

Acknowledgements

- Special thanks to Guild Copeland, Dan Buller, and Remi Dingreville.

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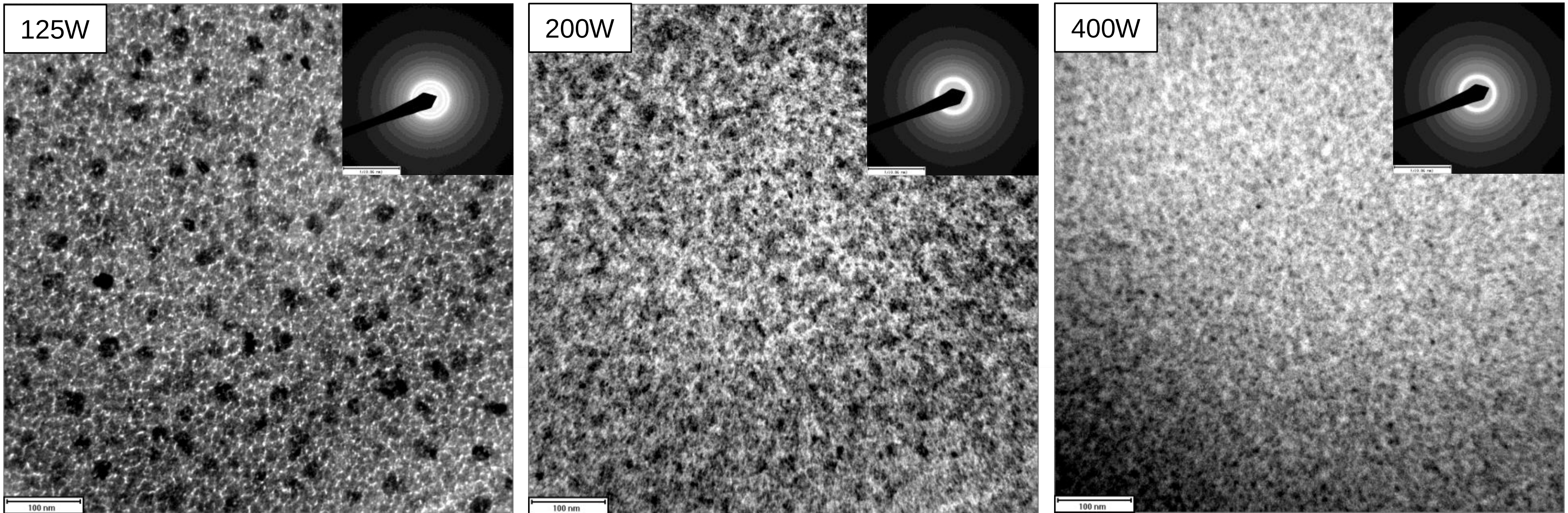
Thank you for your attention.
Questions?

As-Deposited Microstructures

- Deposited on silicon nitride TEM windows
- Cu/Nb contents listed



Irradiated Microstructures (1 dpa, 500 °C)

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