Inhomogeneous Cu Diffusion in NIF Beryllium Ablator Capsules

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Unlike GDP, beryllium is heterogeneous at micron scale

- **Process/structure interactions critical to performance**

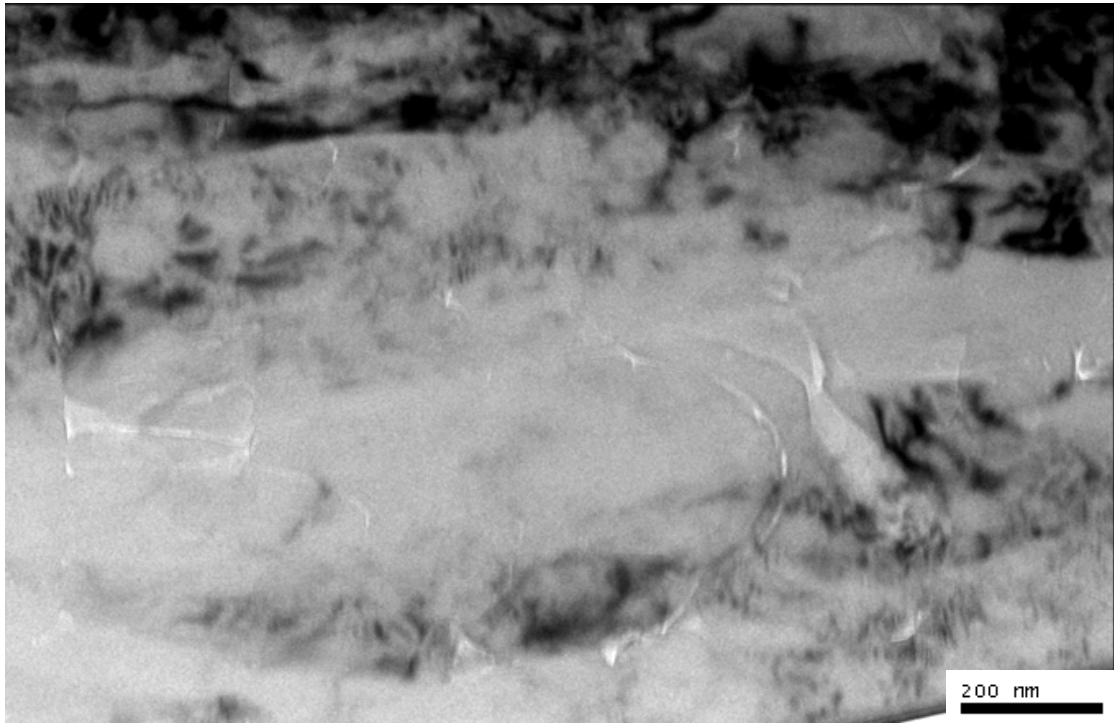
Fabrication process

↓

Micro-structure

↓

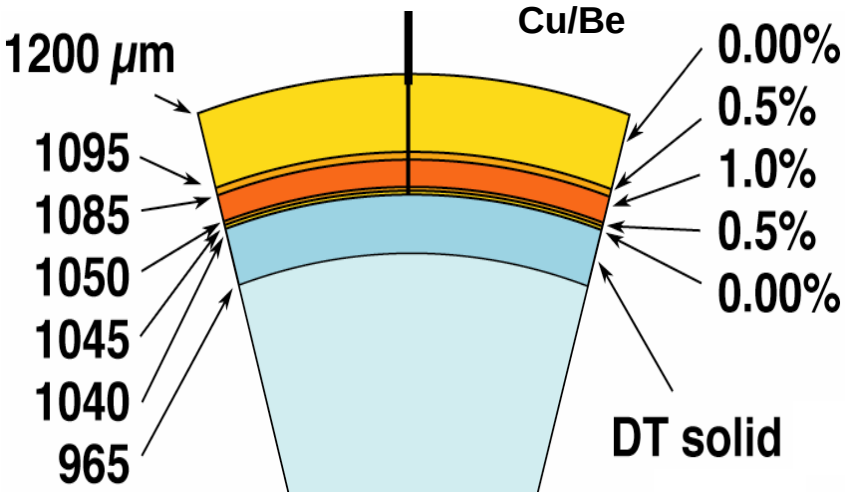
Target performance

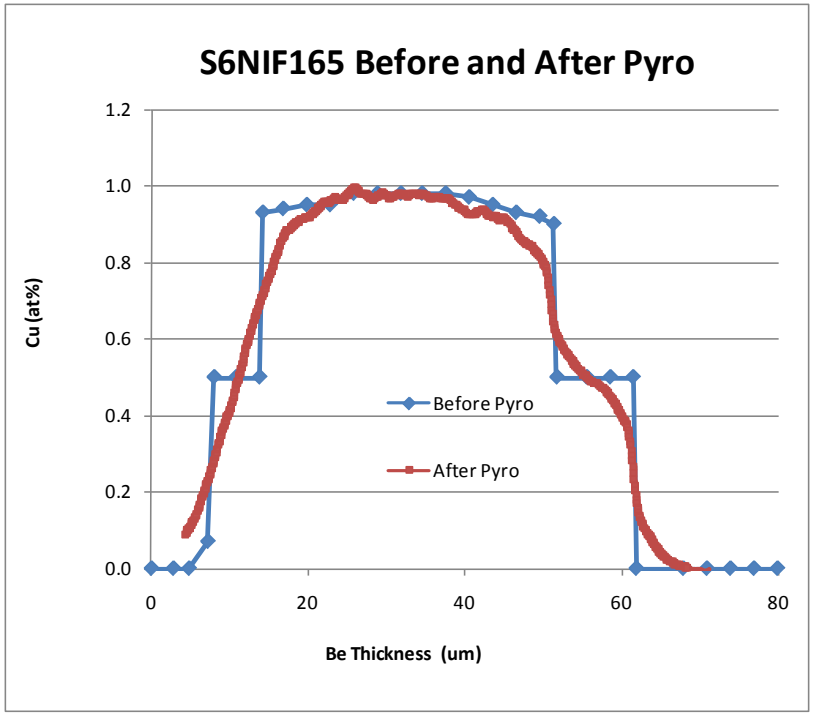


- **This talk presents a Cu-Be case study**
- **Same methodology applicable to other NIF dopants**

NIF Be capsules have step-wise dopant profiles; diffusion during pyrolysis blurs the steps

- **NIF design uses 5 distinctive layers to optimize shock timing**



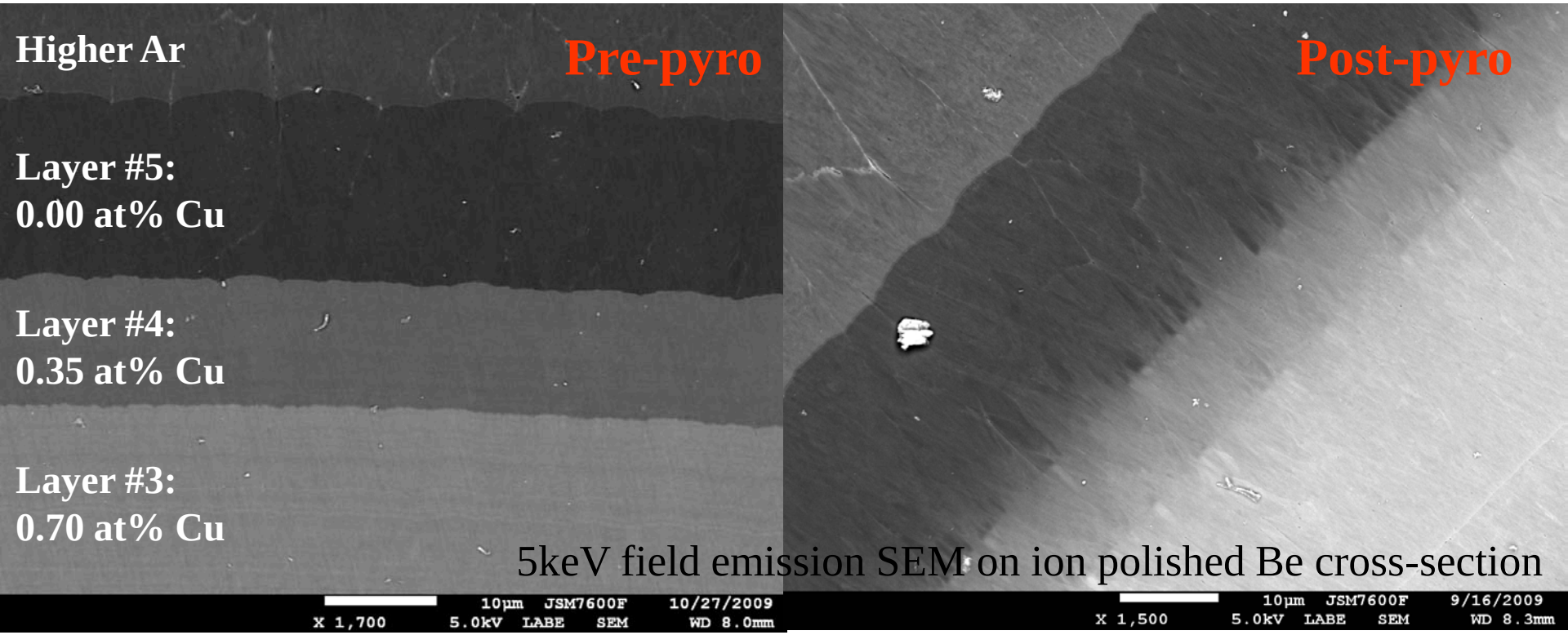


- **Interfaces become diffused after mandrel removal**
  - Measured non-destructively by contact radiography

Need quantification to estimate the impact on implosion

SEM Backscattered Electron (BSE) contrast reveals dramatic difference in pre- and post-pyro samples

- **Post-pyro interfaces become diffused and heterogeneous**

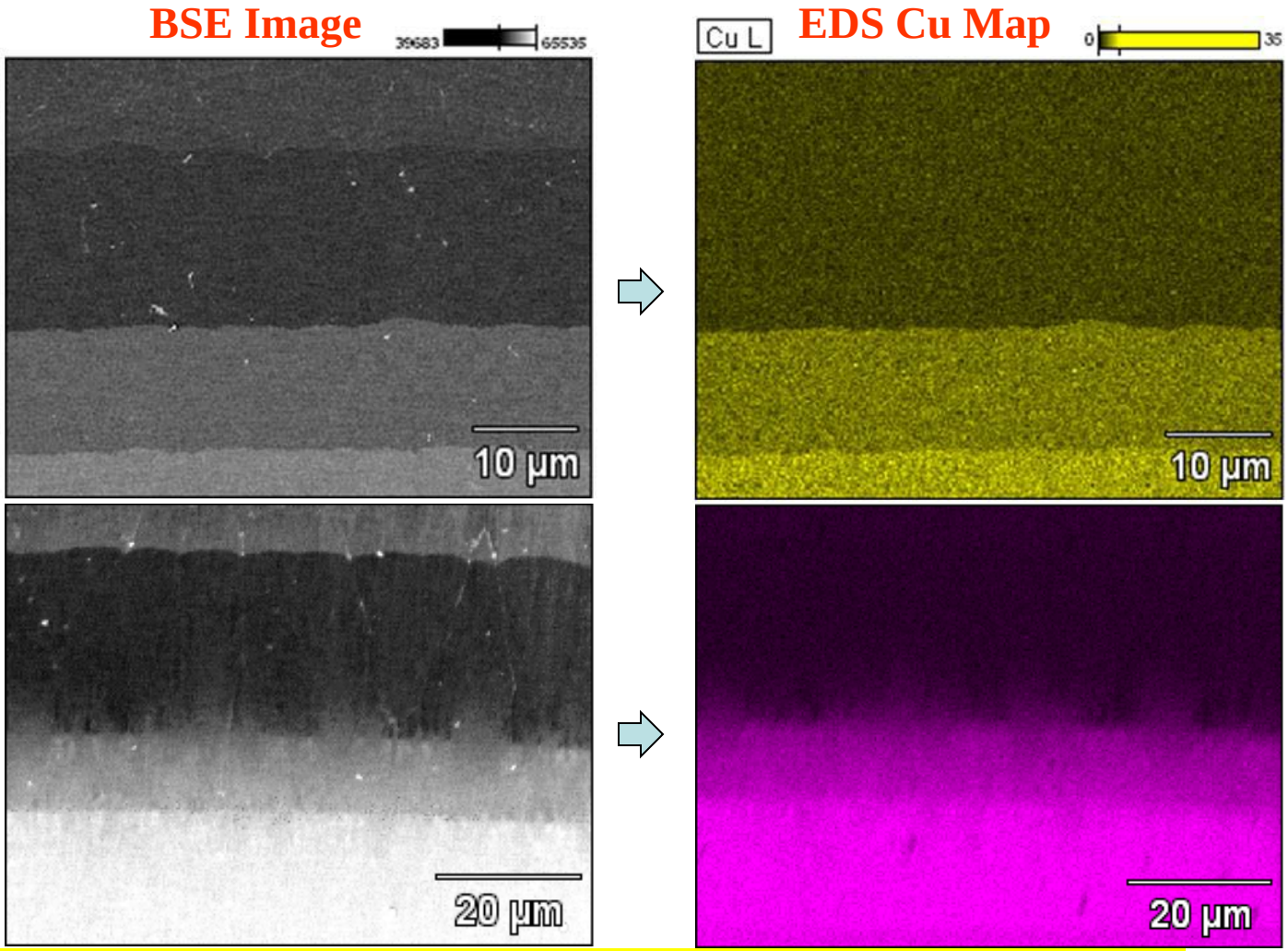


- **Heterogeneity length scale: 0.5-5 µm radial, 0.5-10 µm lateral**
  - >10x larger than bulk diffusion behavior (Butrymowicz 1975)

Energy-Dispersive Spectroscopy (EDS) unambiguously identified Cu variations as the cause of BSE contrast

Pre-pyro:
Sharp Interface

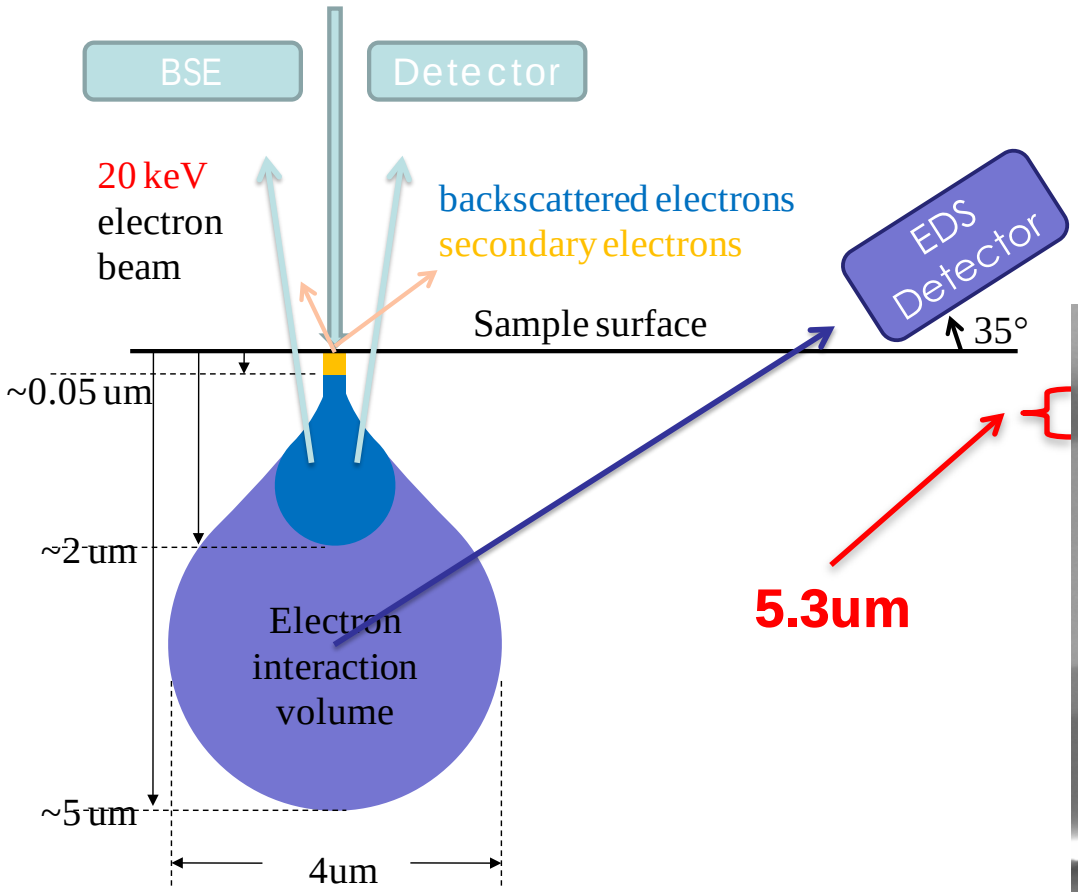
Post-pyro:
Diffuse Interface



Need to distinguish two scenarios: (1) polishing smear (2) Cu diffusion

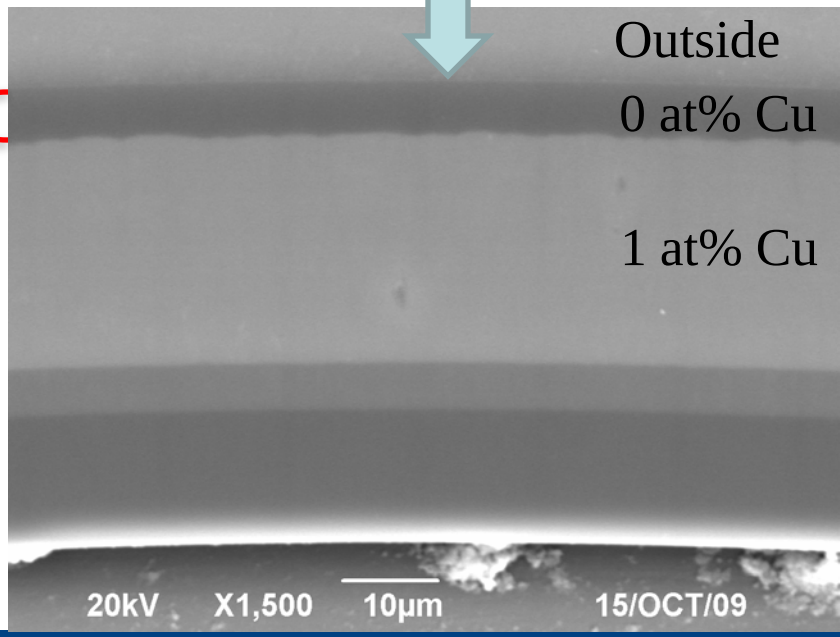
Measuring Cu from outside, through undoped Be layer, eliminates the possibility of polishing smear

- **20keV e-beam probes <5 μm depth into bulk beryllium**
  - Can't see through 5.3 μm undoped buffer layer unless Cu diffuse



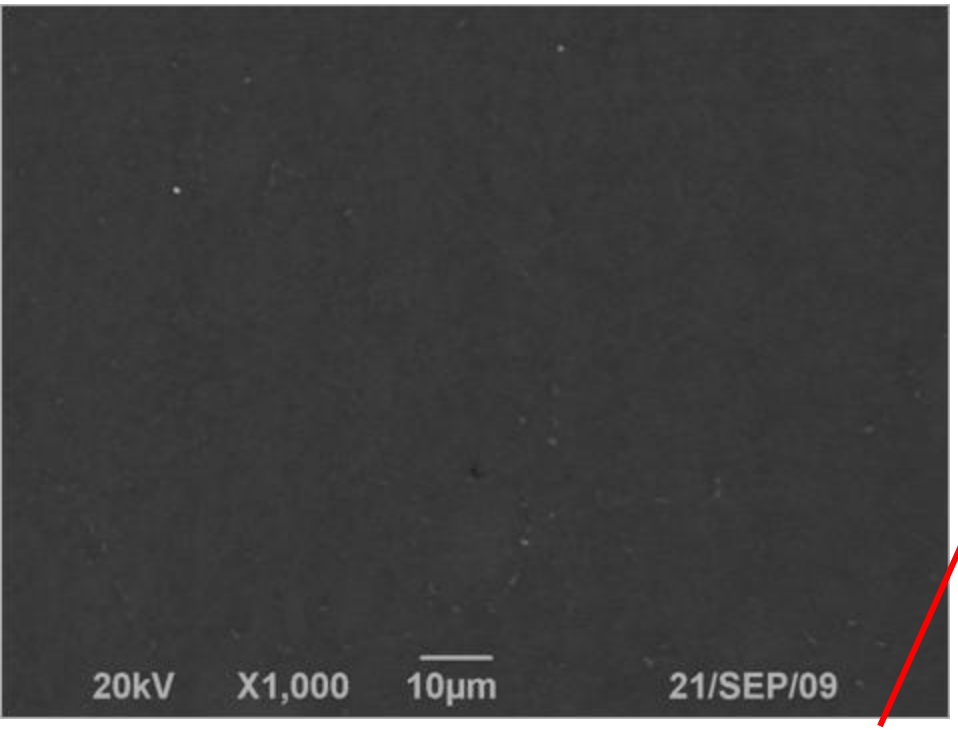
Test suggested by J. Edwards @ LLNL

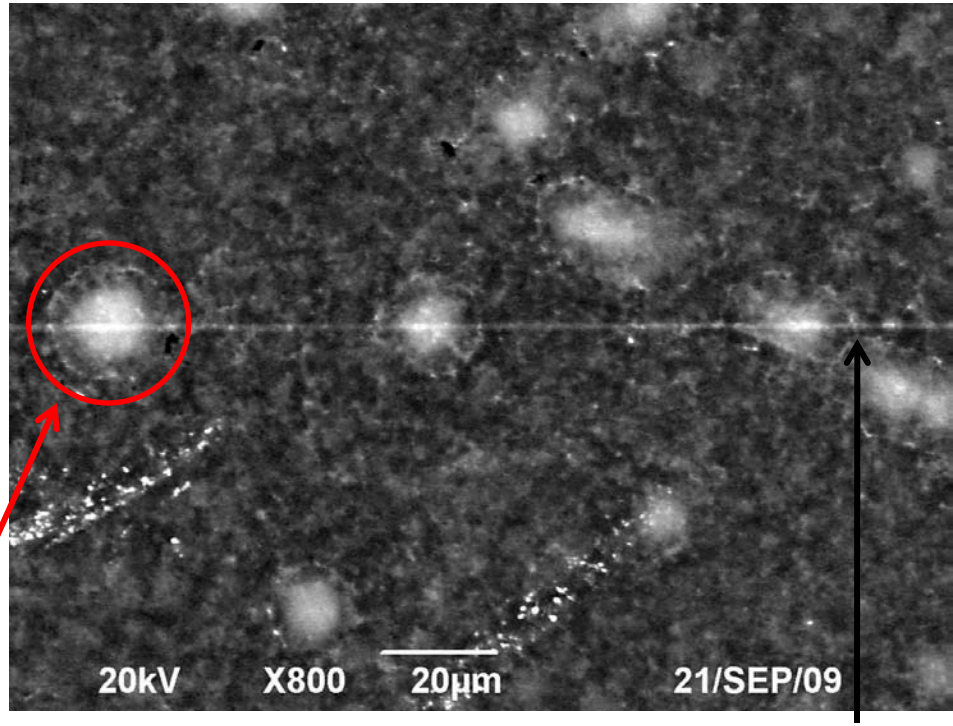
e-beam probes outside surface



Copper diffused into the undoped buffer layer in highly heterogeneous manner during pyrolysis

- BSE contrast invisible on pre-pyro surface
- BSE contrast conspicuous on post-pyro surface





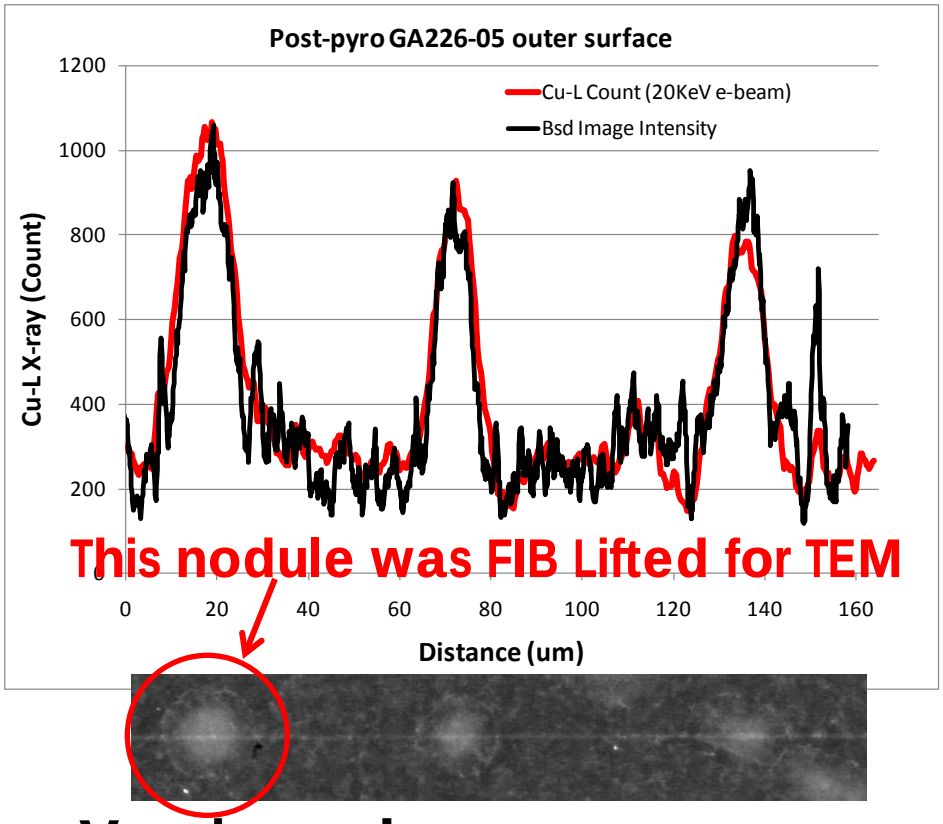
This nodule was FIB Lifted for TEM

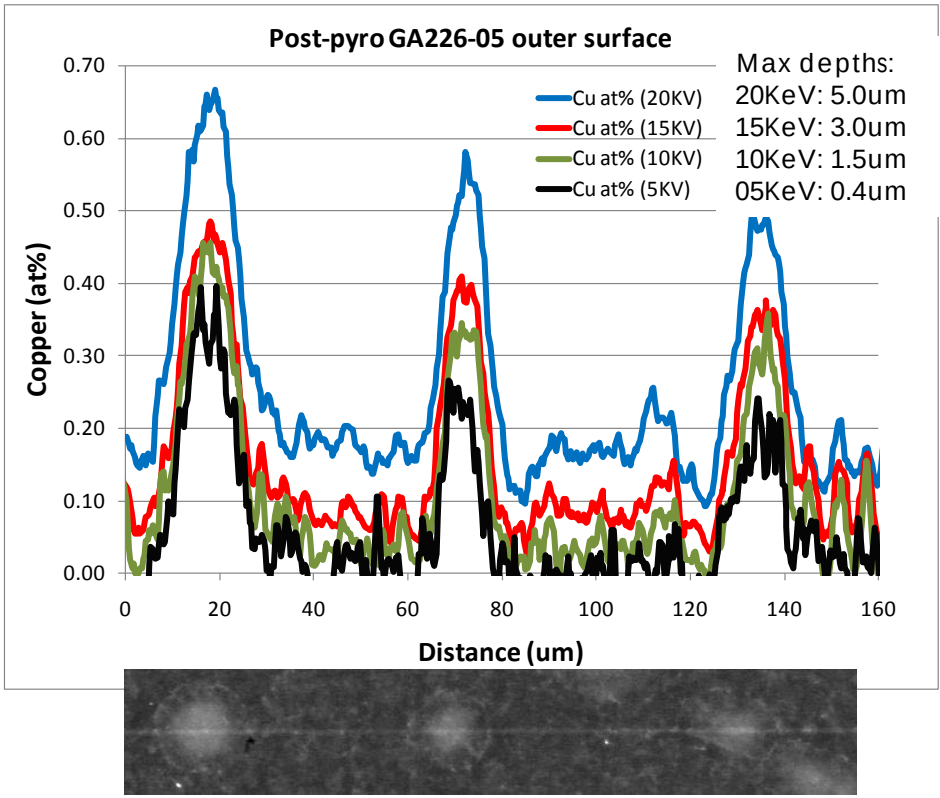
Line is where EDS scan was performed

- Post-pyro contrast: “super nodules” on textured background

EDS element line-scans provide further validation

- **BSE contrast agrees with Cu profile measured by EDS linescan**
  - Establish BSE as quick semi-quantitative Cu mapping technique

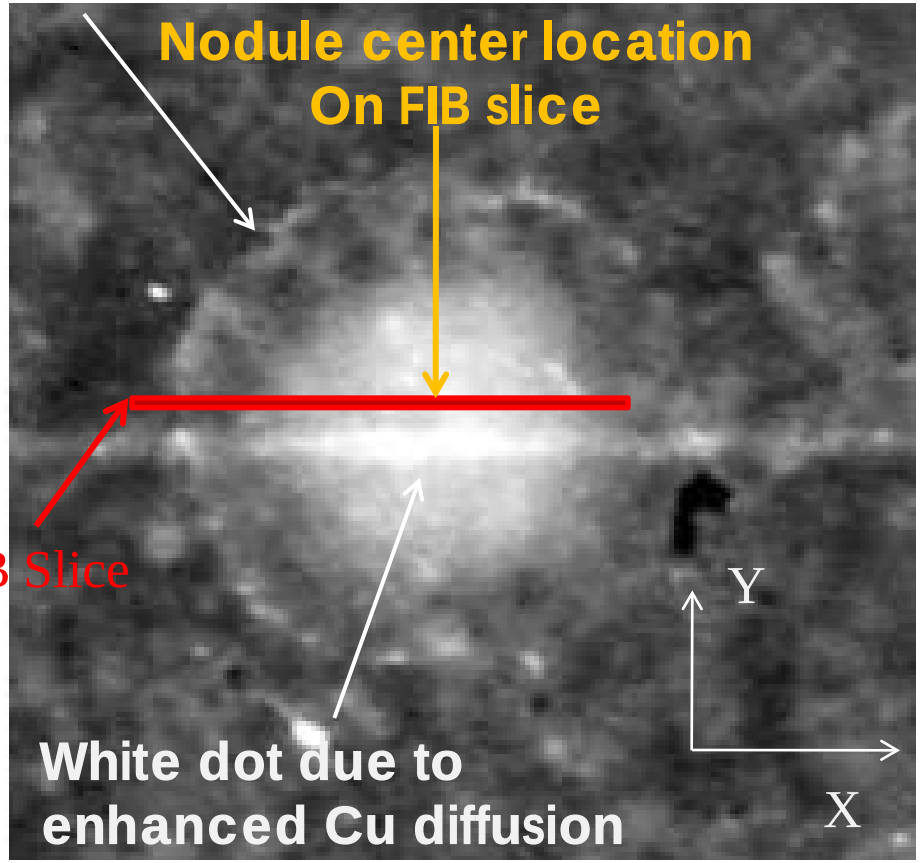




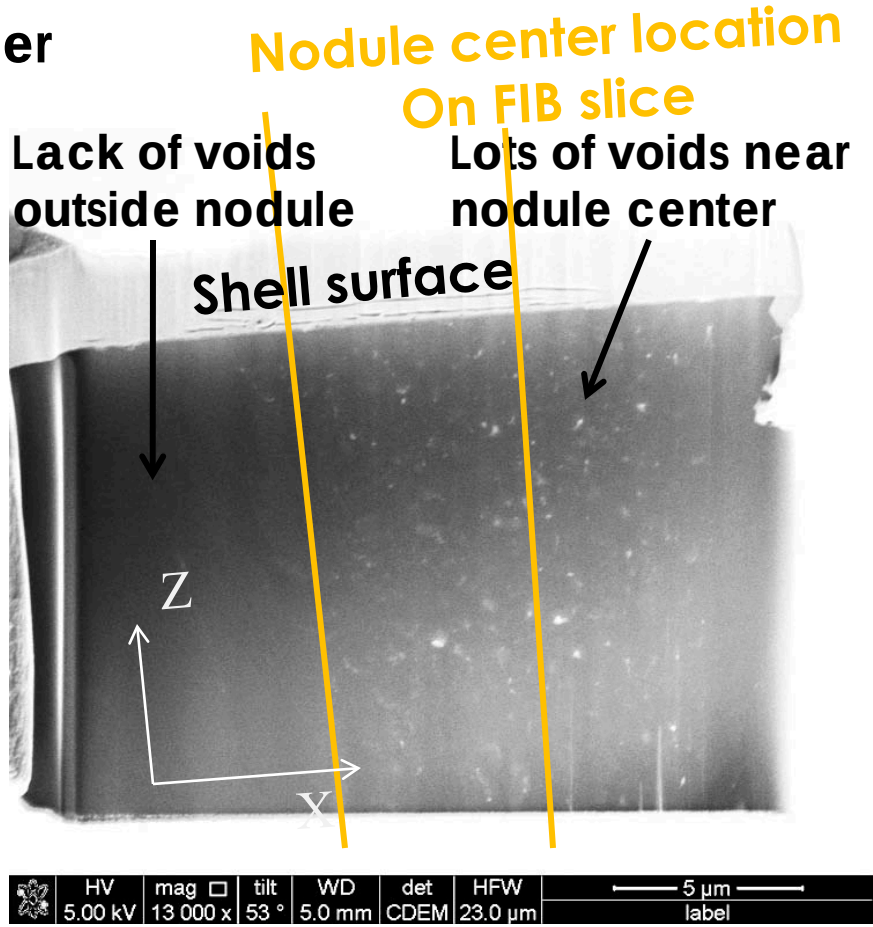
- **Varying e-beam energy proves Cu from an internal source**
  - Higher energy probes deeper and measures higher Cu at%

FIB slice was lifted-out from the "super nodule" on post-pyro sample to study structural-composition correlation

White ring, due to oxide, marks the "super nodule" growth column boundary



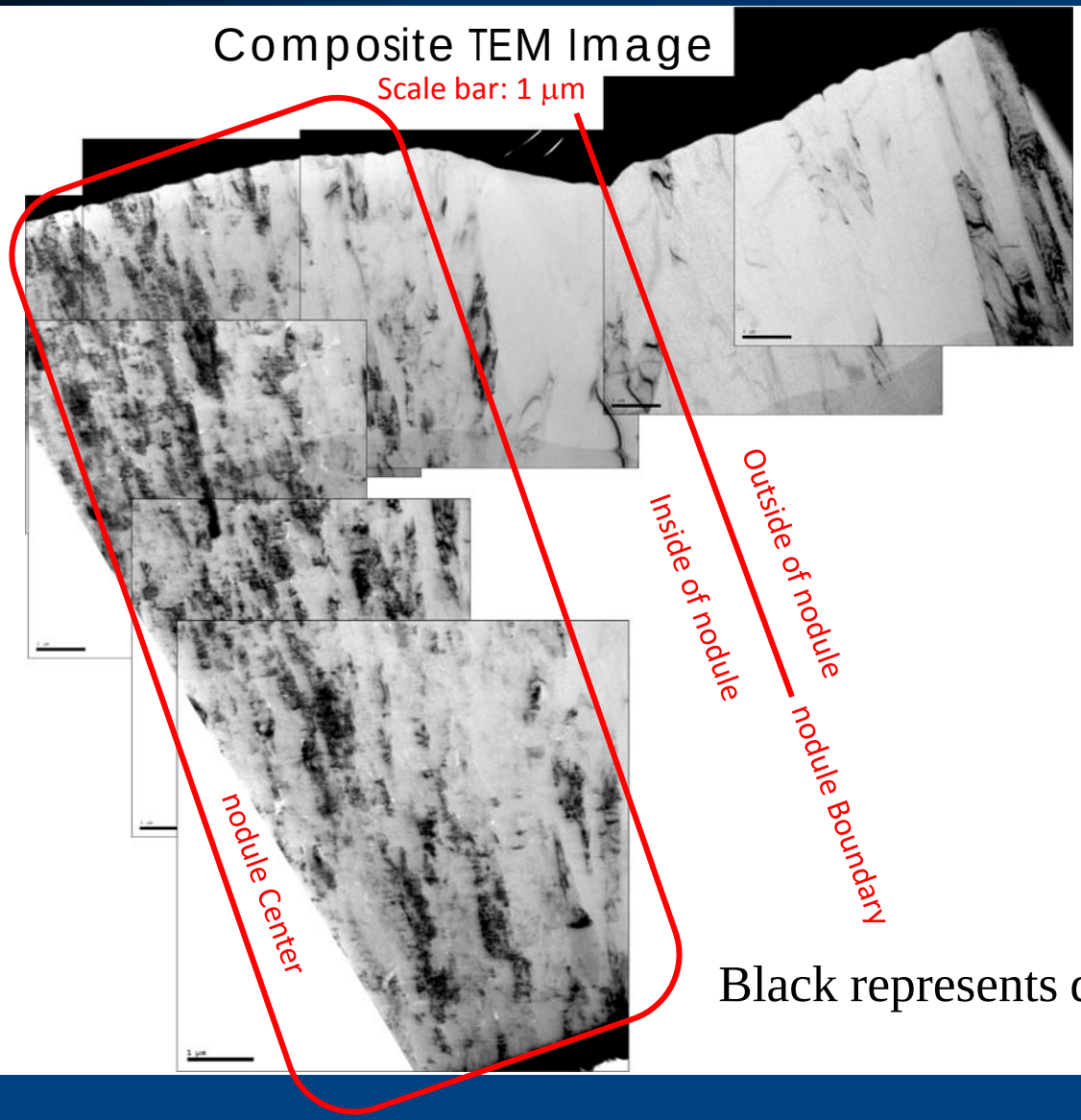
Top-down SEM image (BSE contrast)



~130 nm thin FIB slice shows porous structures near nodule center

TEM shows nodule center having finer, heterogeneous, and porous structures, which can enhance Cu diffusion

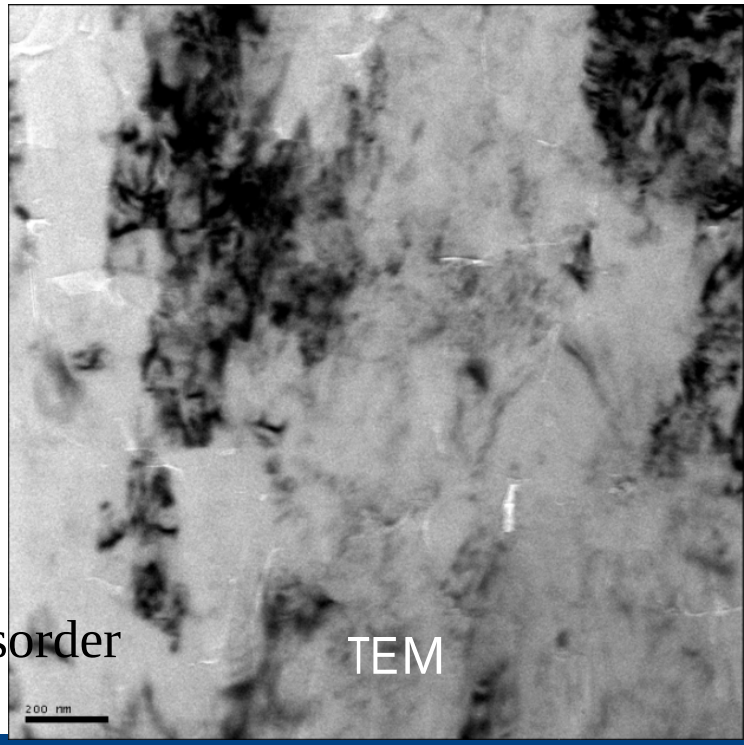
Composite TEM Image



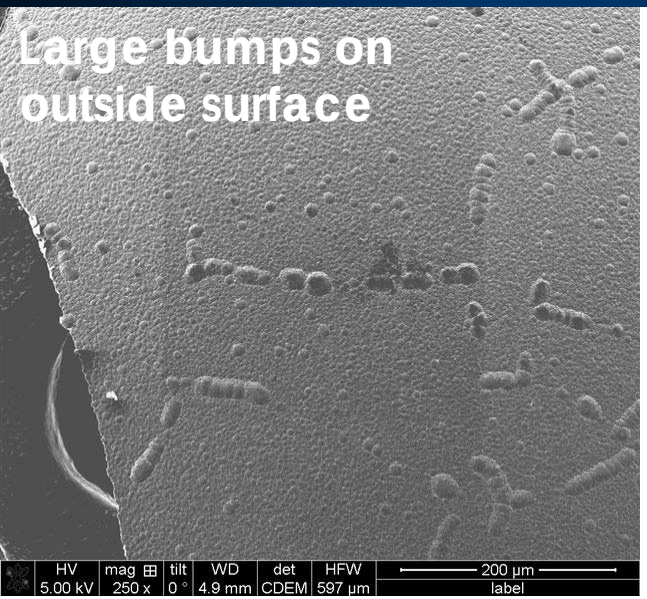
Black represents disorder

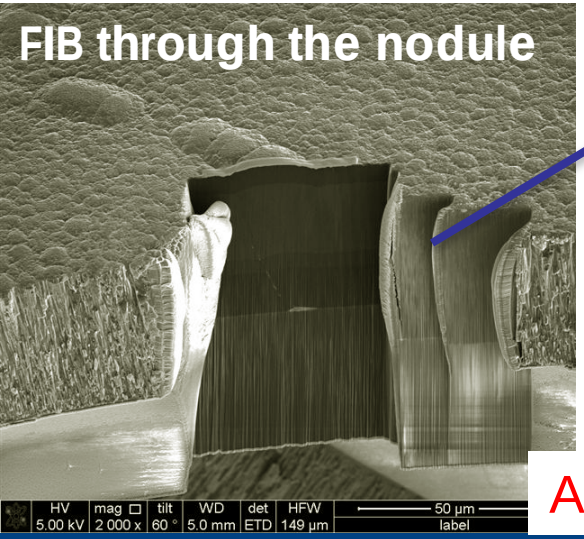
Grain sizes vary greatly:
50-200nm near nodule center
500-1000nm outside nodule

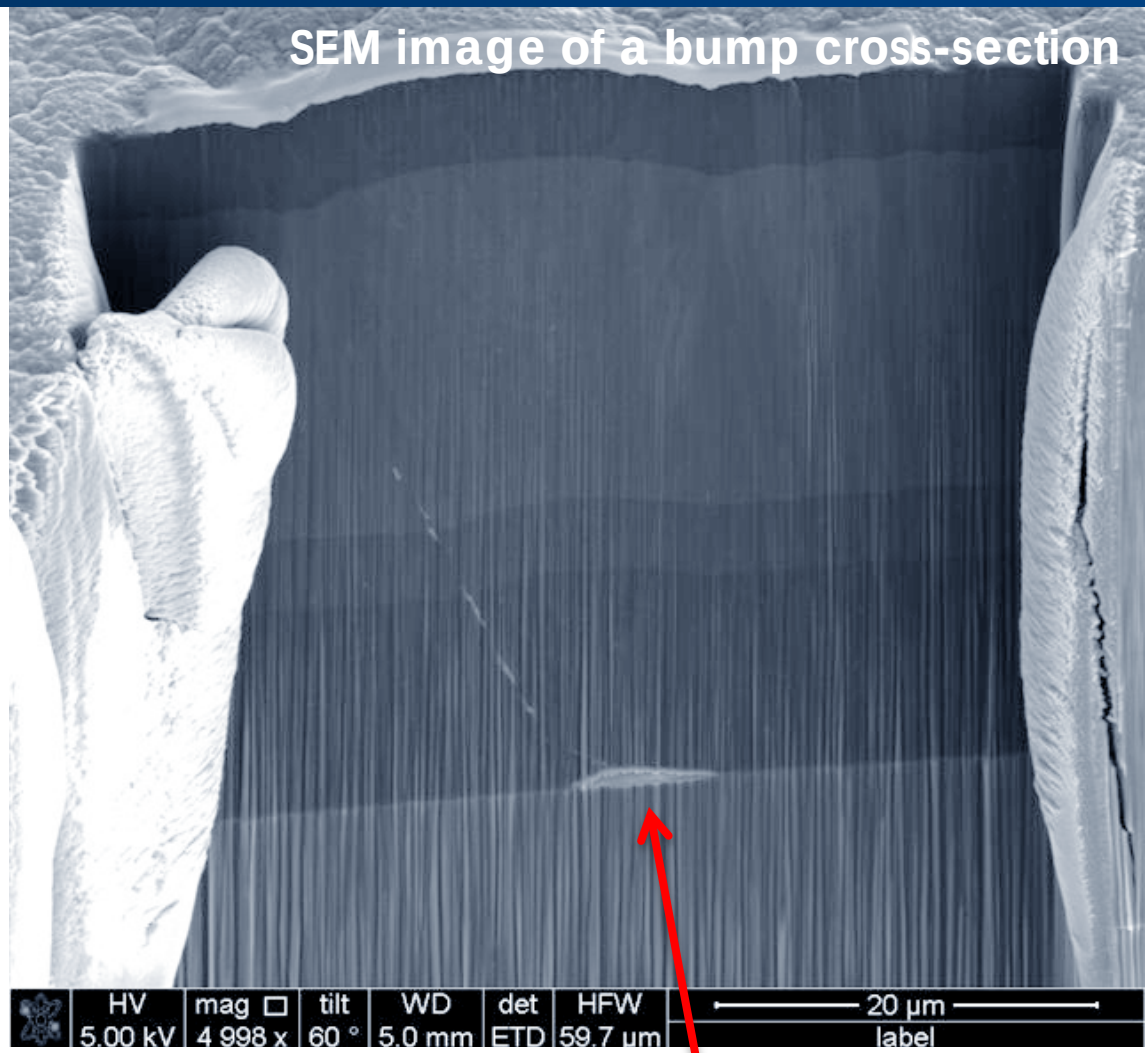
Nano-voids present mostly near nodule center



~20 μm "super nodules" are growth columns induced by mandrel defects, terminating as surface bumps

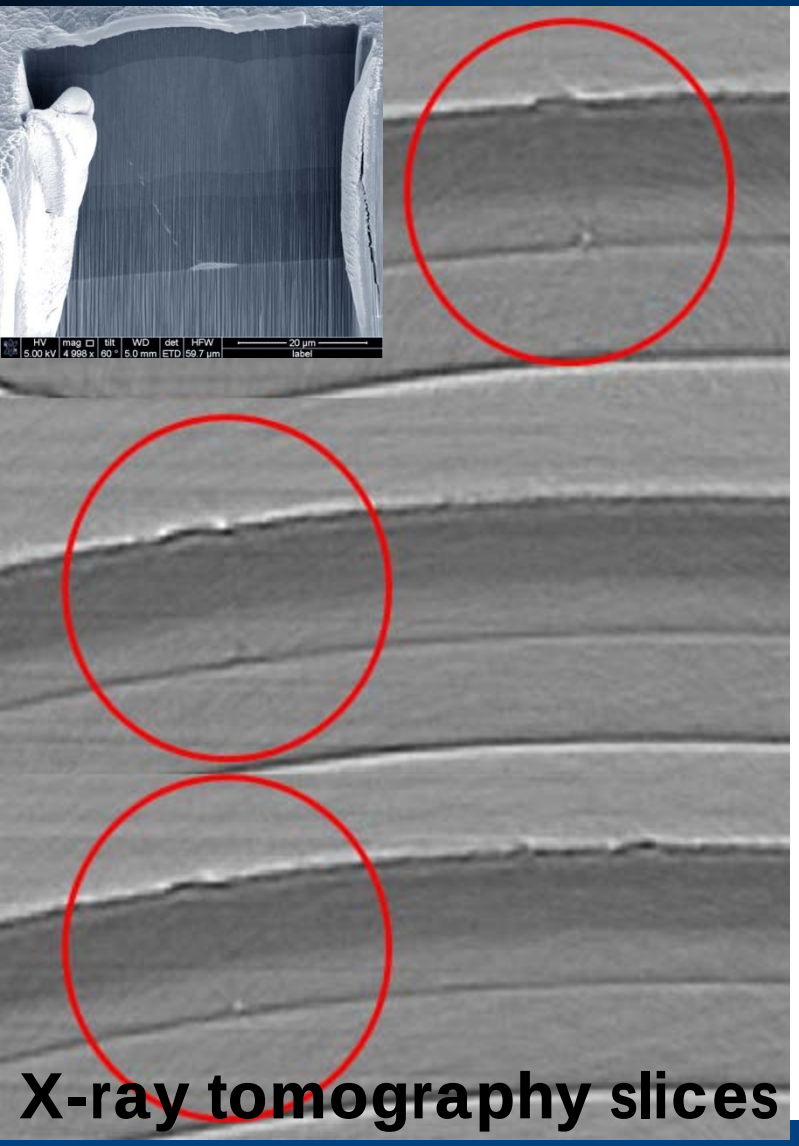




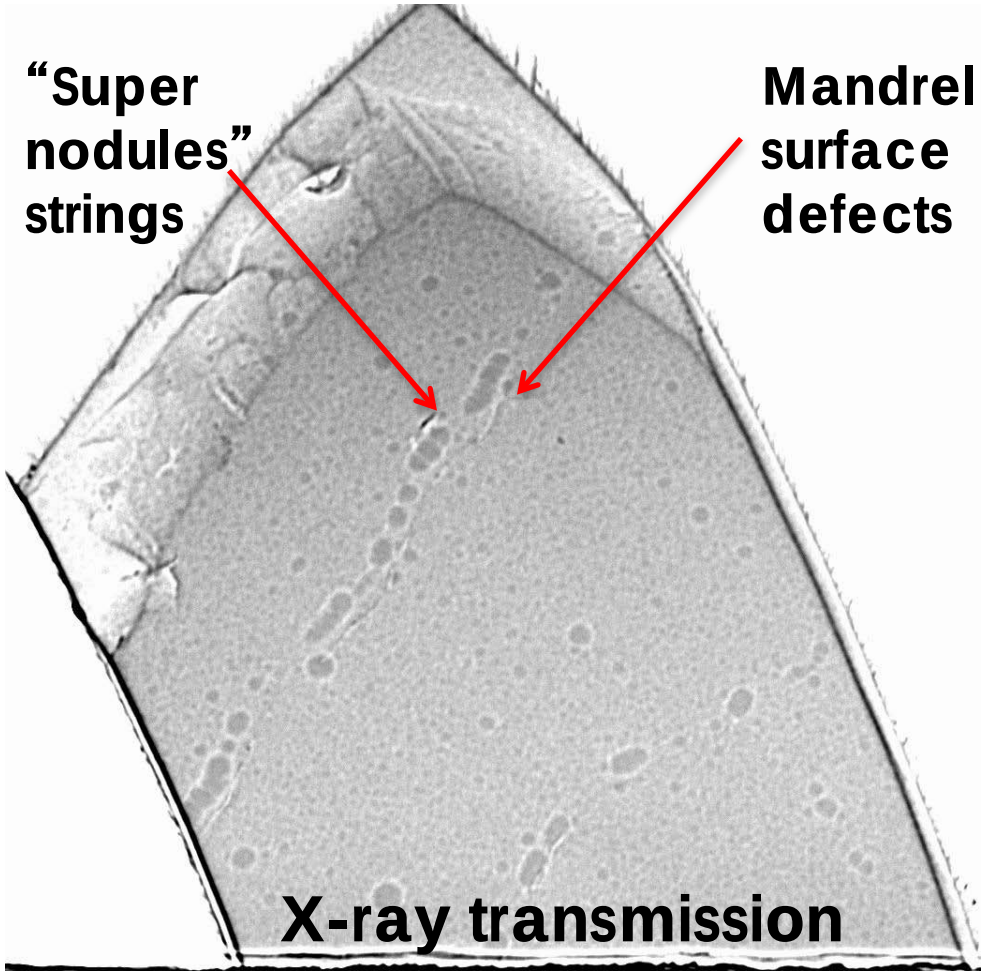


Abnormal growth initiated by mandrel contaminant

X-ray imaging confirms strong correlation between "super nodules" and mandrel surface contaminants

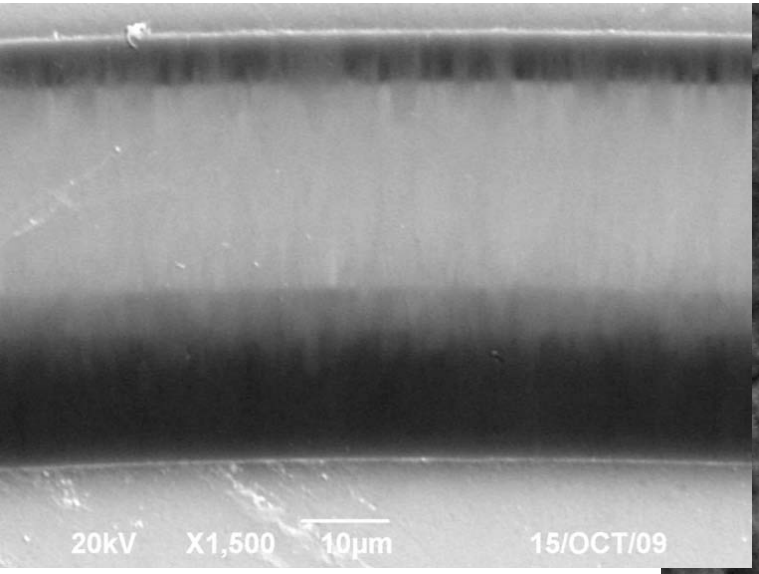


Two features parallel to each other



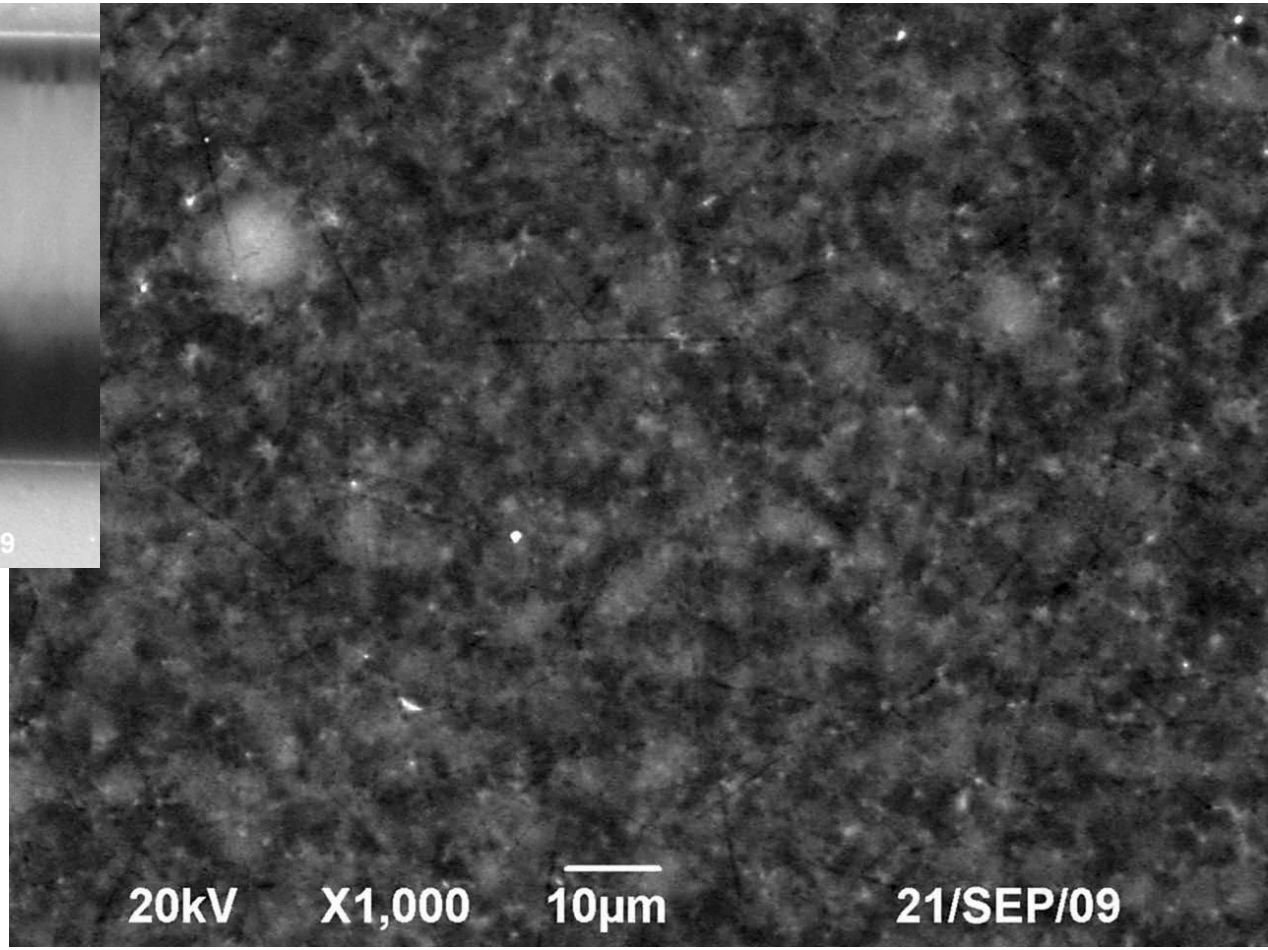
Inhomogeneous background Cu diffusion produces atomic Z contrast on both cross-section and OD

- **Pattern length scales match between two BSE views of the same post-pyro sample**



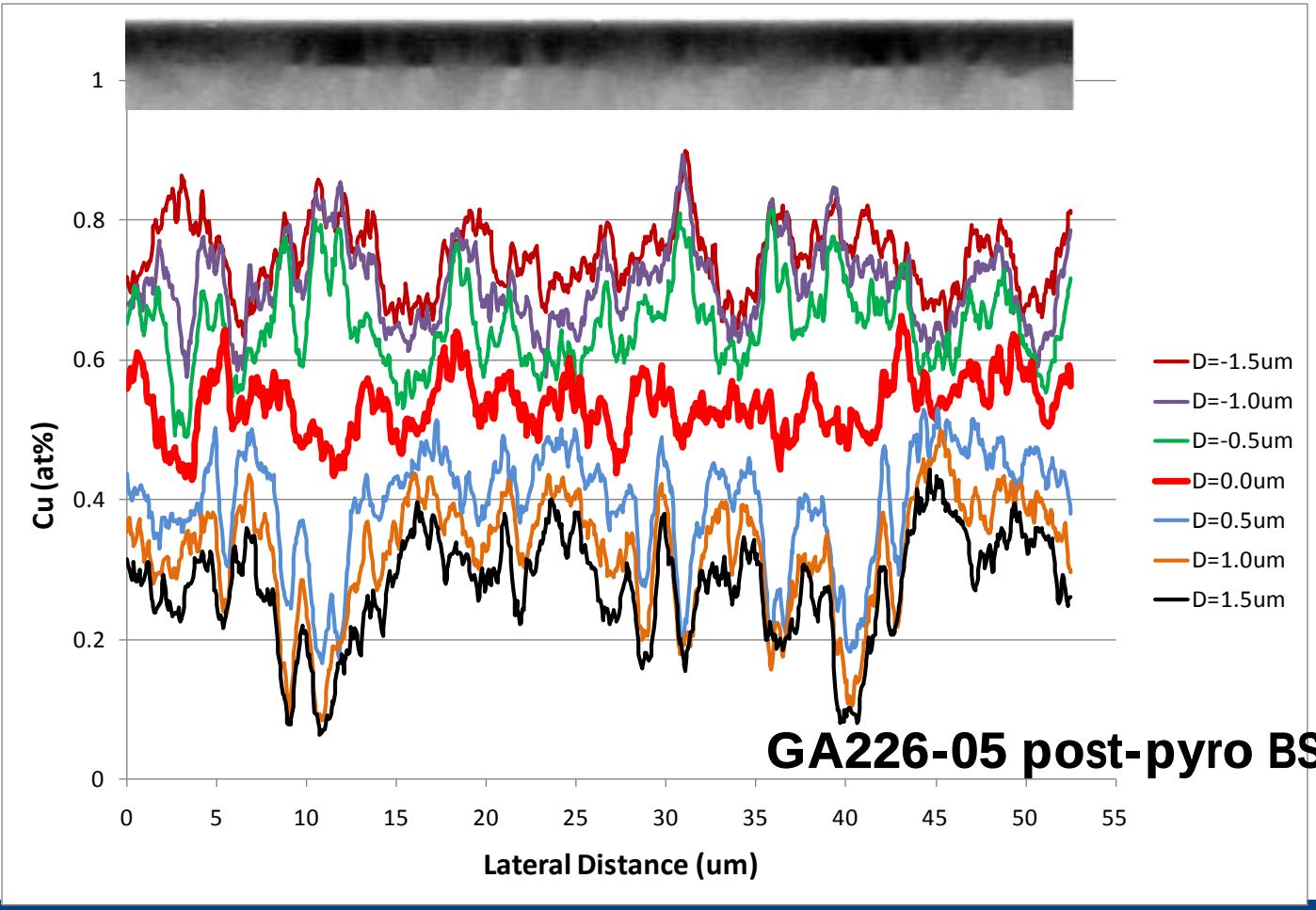
(1) Cross-section view

(2) Top surface view



Background Cu diffusion produced ±0.06 at% 1-σ variations, which is unwanted by target designers

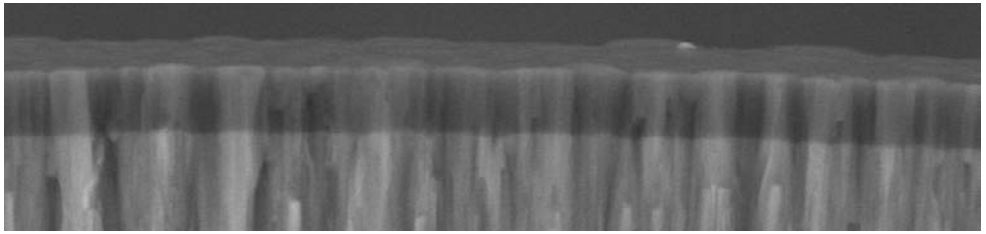
- **Copper profiles near interface are anti-phased, expected if a conserved total quantity is redistributed**



"D" indicates distance to the original Cu interface

GA226-05 post-pyro BSE contrast line-outs

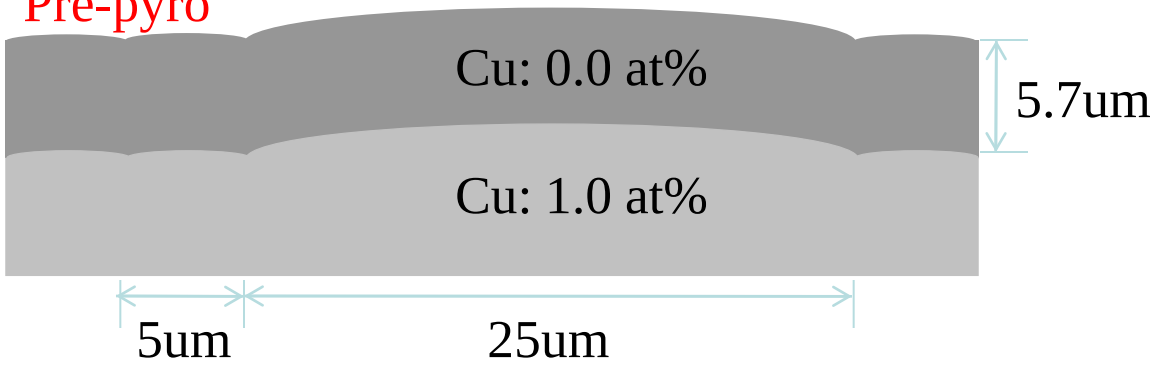
A phenomenological description of Cu diffusion in beryllium



85um

Sputtered beryllium grow in vertical columns, capped at the surface as nodules. Each column is a cluster of submicron columnar grains

Pre-pyro



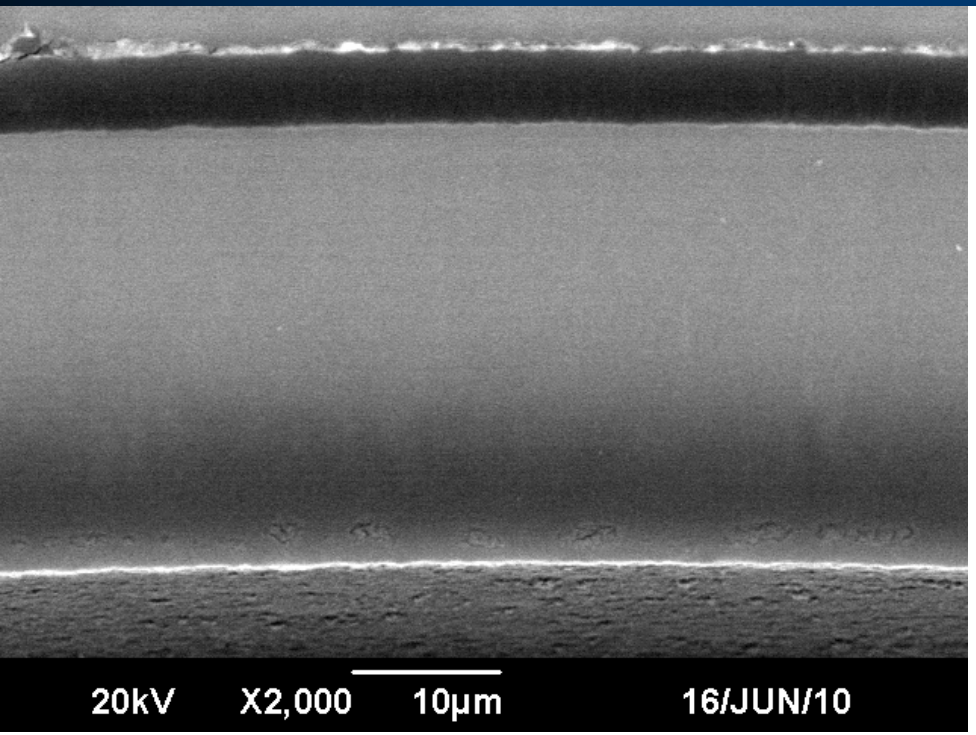
There are ~25um "super" nodules embedded inside a sea of 5um regular nodules.

Post-pyro

Cu diffuses faster toward center of each nodule, even faster for "super" Nodules

See Poster P-28 by H. Huang for metrology technique details

Oxide provides an effective diffusion barrier



Thermal oxide stopped Cu diffusion

Strong Cu diffusion where no oxide was introduced

- **Oxide idea evolved**
  - ALD Al2O3
  - Thermal oxide
  - In-situ oxide

See Oral Tue. AM1-4 by K. Youngblood

Cu diffuses inhomogeneously in sputtered beryllium

- **Cu structure length scale varies:**
  - 0.5 to 5 μm radially,
  - Minimum 10x larger than bulk prediction
- **Two types of Cu structures:**
  - background variations & "super nodules"
- **Background Cu varies by ~+/-0.06 at% (1-σ)**
- **"Super nodules" correlates to mandrel contamination**
  - Nodule centers are porous, with smaller grains which enhanced diffusion
- **Cu diffusion, uniform or not, can be stopped by oxide barriers**
  - Beryllium can remain to be an ablator choice

GENERAL ATOMICS

Same methodology can be applied to other dopants, Work in progress on W, Zr, Al, Si

- **Dopant behaviors vary greatly**
  - Uniform and stays uniform: W and Zr
  - Uniform and becomes non-uniform: Cu
  - Non-uniform but doesn't further evolve: Si
  - Non-uniform and worsens upon pyro: Al
- **Different physical processes in the play**
  - Solubility => phase segregation
  - Mobility => diffusion
- **Same measurement strategy apples**

GENERAL ATOMICS