ILLINOIS INSTITUTE
OF TECHNOLOGY

Preliminary Transmission Electron Microscopy imaging results on hot/cold cutouts.

Yulia Trenikhina
Alexander Romanenko

7th SRF Material Workshop
07/17/2012

Fermilab

Purpose of this work

IMPROVEMENT OF QUALITY FACTOR: $Q_0 = \frac{\omega_0 U}{P_c}$

real material performance → $Q_0 \sim 1/R_{surf}$, $R_{surf} = R_{BCS} + R_{res}$, $R_{BCS} \sim 1/T, f^2$ → *Quality factor* *limitations*: *HFQS, local quench*

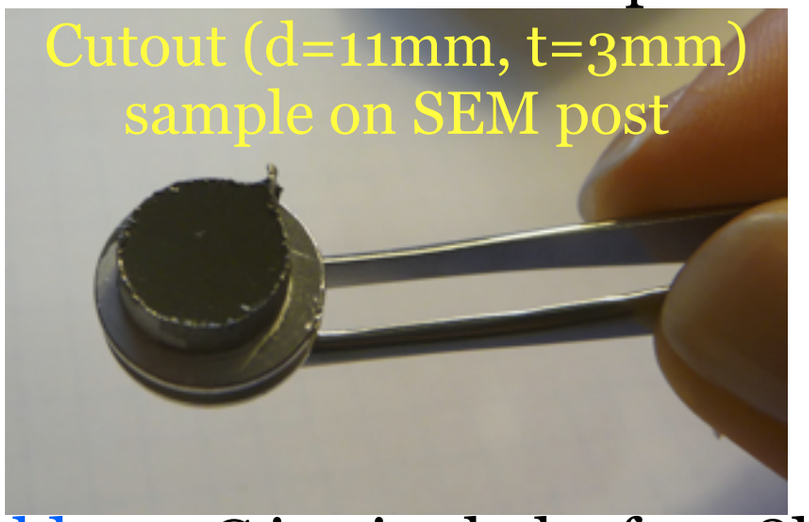
Questions:

1. material features of Nb that cause anomalous RF losses?
2. what happens to Nb during 120C *in situ* vacuum bake?

Material Science problem for TEM

Scale of investigation: several tens of nm from the surface

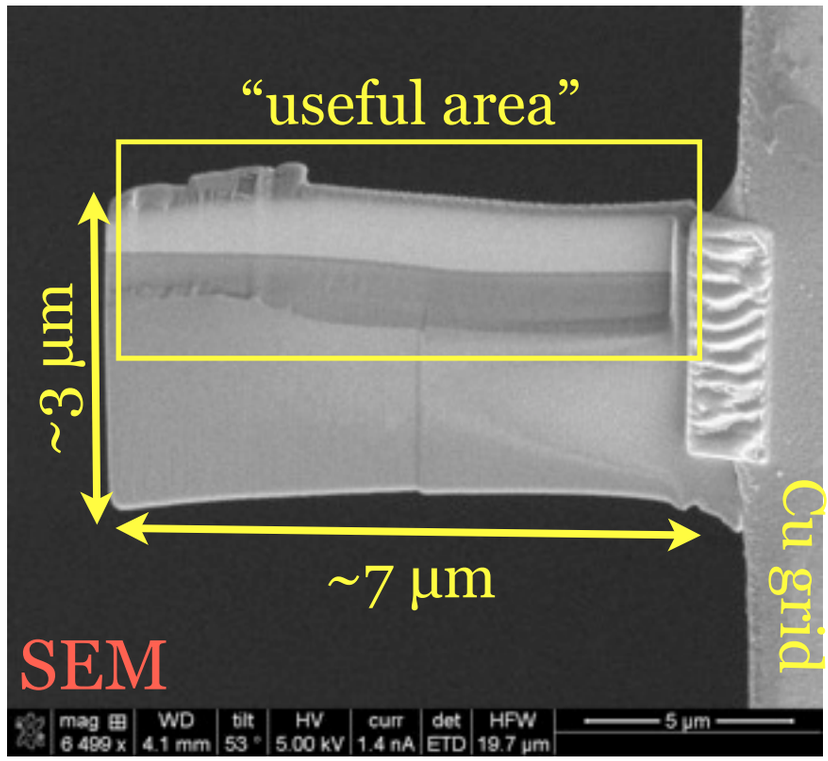
Heating: comparison of "cold" and "hot" spots



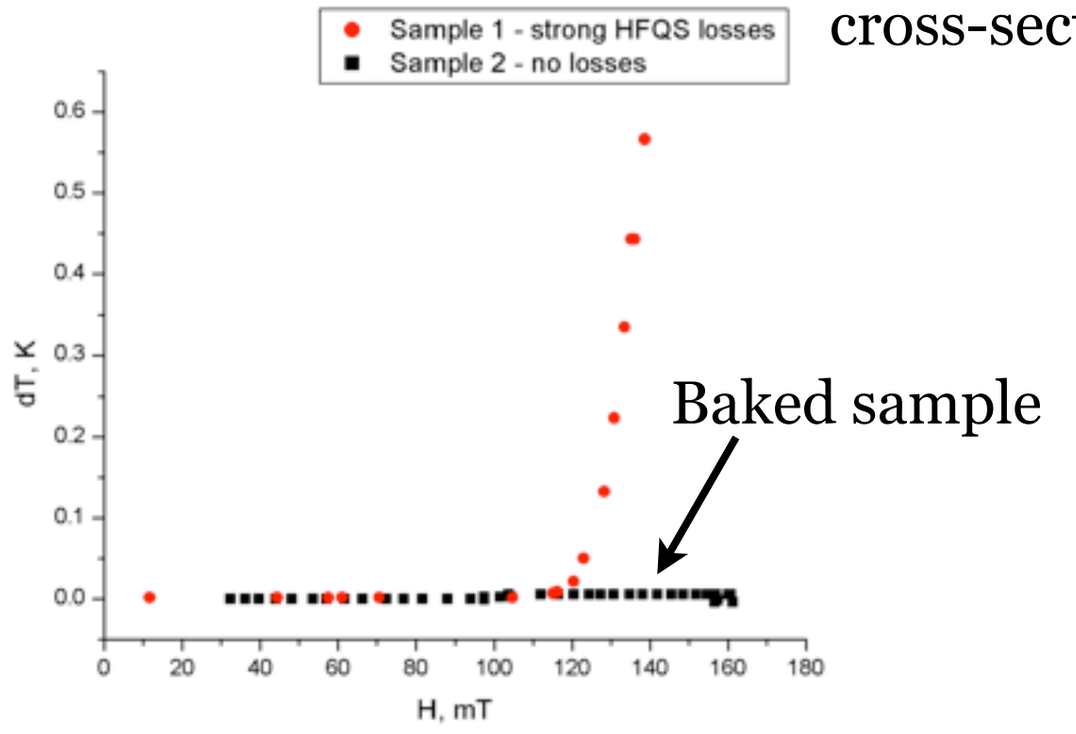
Cold: 120C in situ bake for 48hours

Hot: no such bake

FIB prep sample for TEM



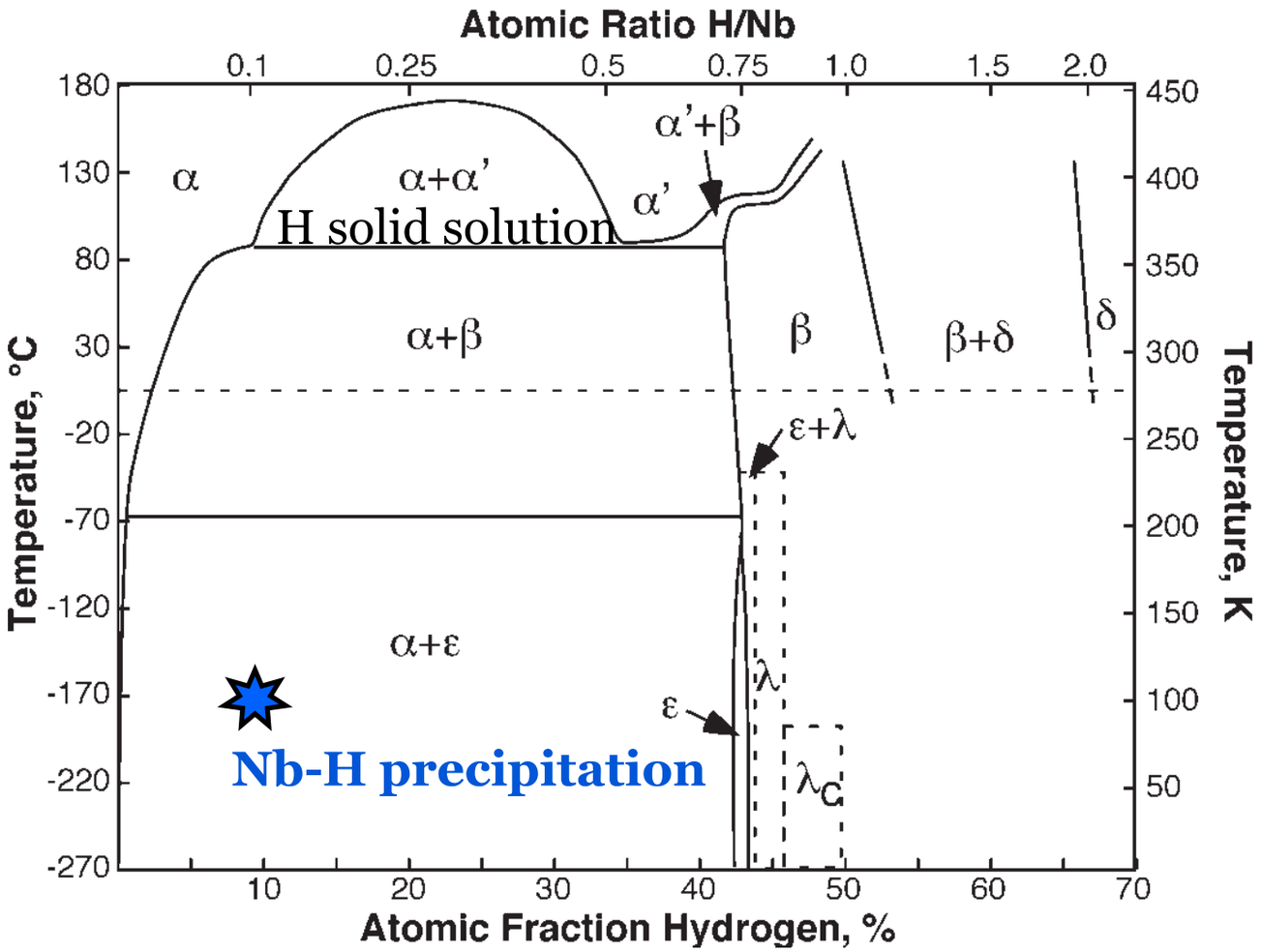
cross-sectional cut from the bulk



Baked sample

What do we expect to see?

DIFFERENCE between Hot and Cold Spot



Niobium-Hydrogen phase diagram reported by Manchester and Pitre.

H concentration in near-surface layer > 10 at.%

↓

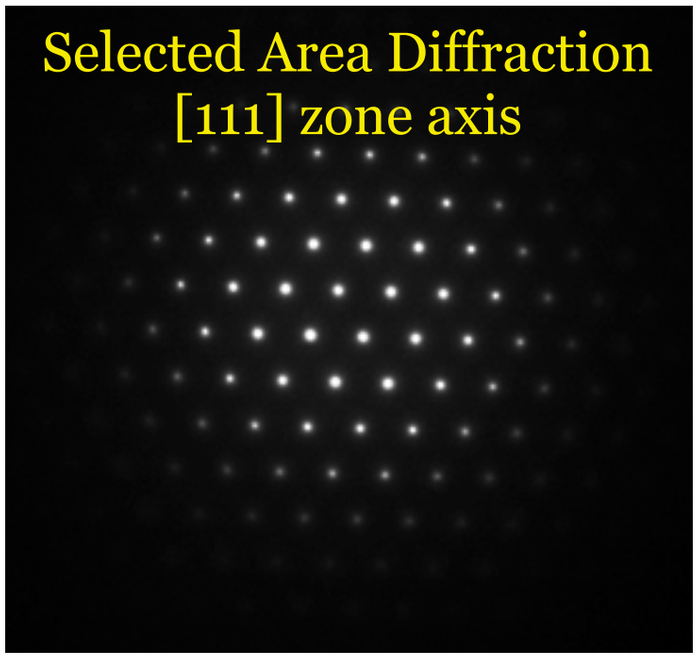
H precipitation at T~100K
ε-phase: Nb_4H_3

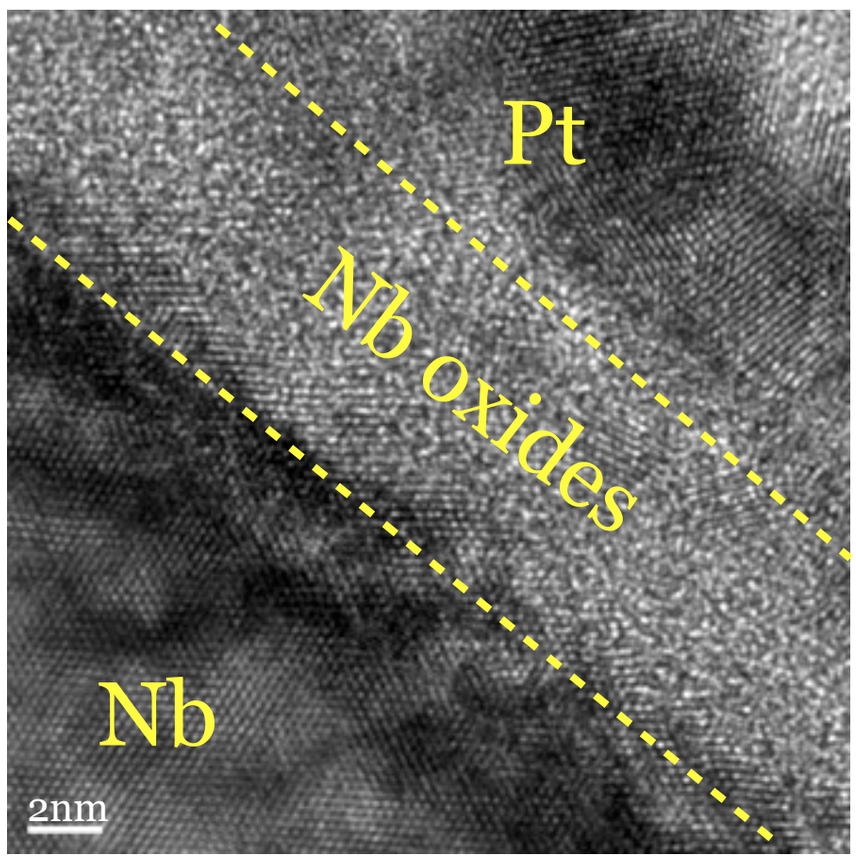
↓

change of Nb local structure

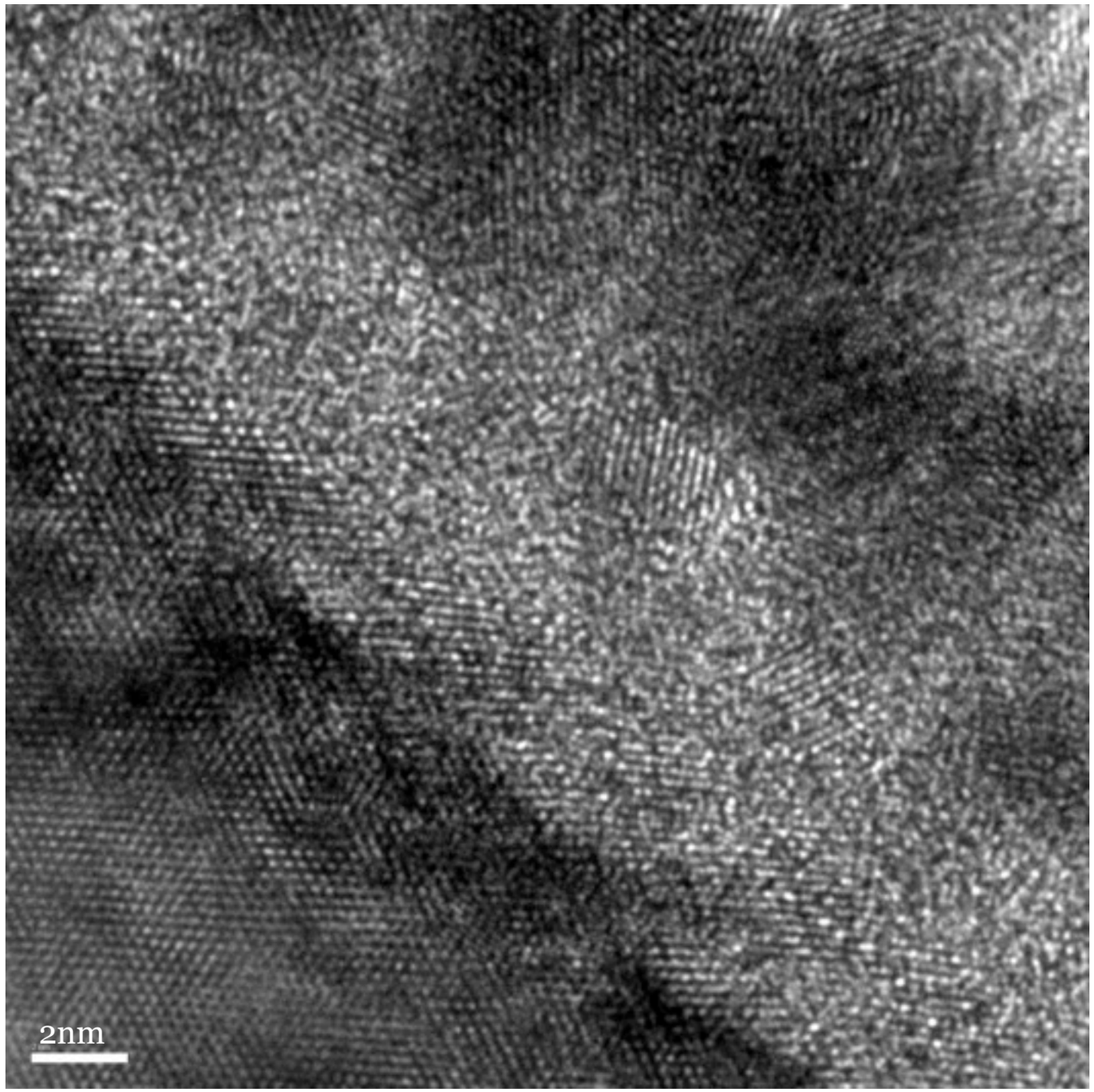
"Baked" vs. "Unbaked":
different precipitation state

HRTEM imaging of Cold Spot at Room T

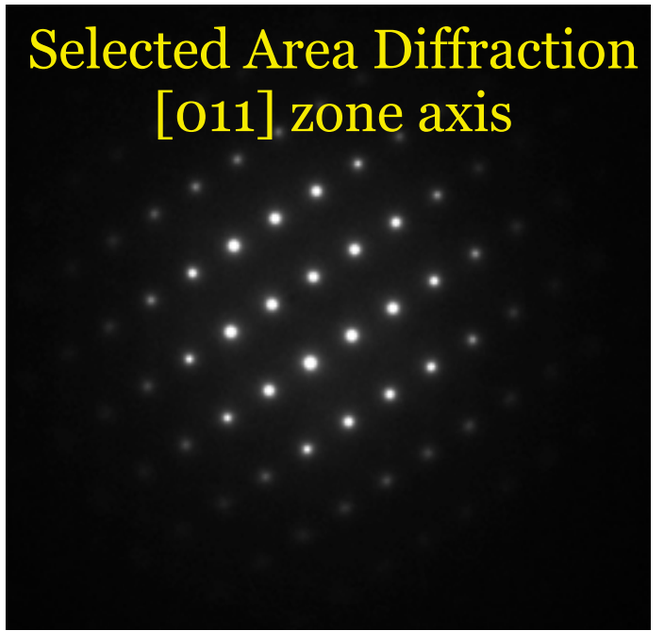


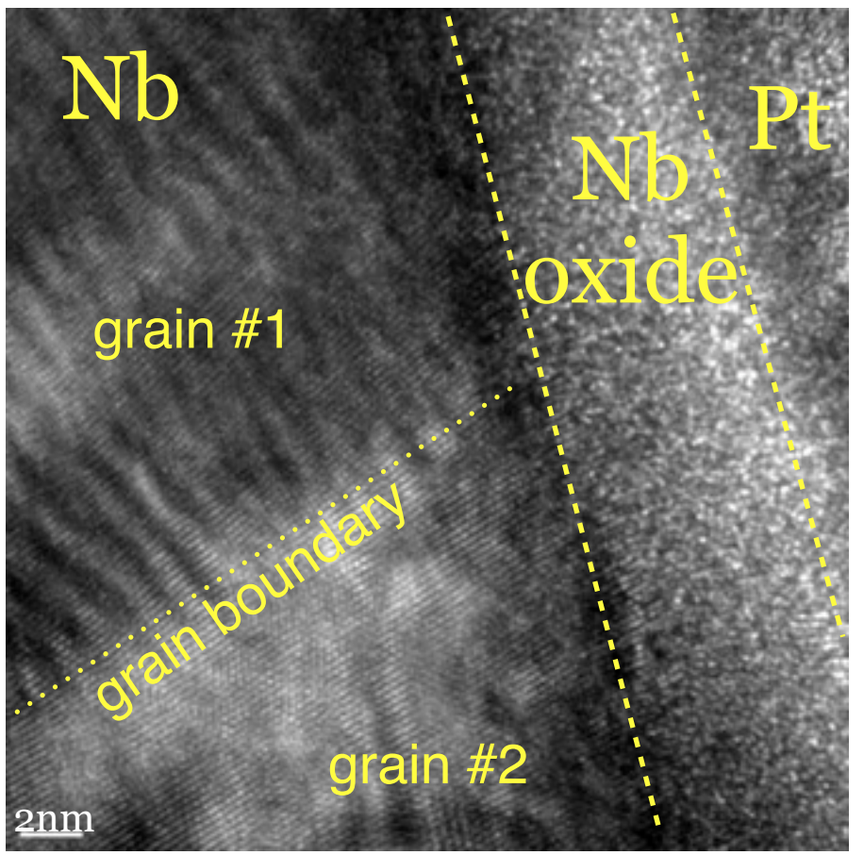


Phase Contrast of the interface Nb oxide - Nb

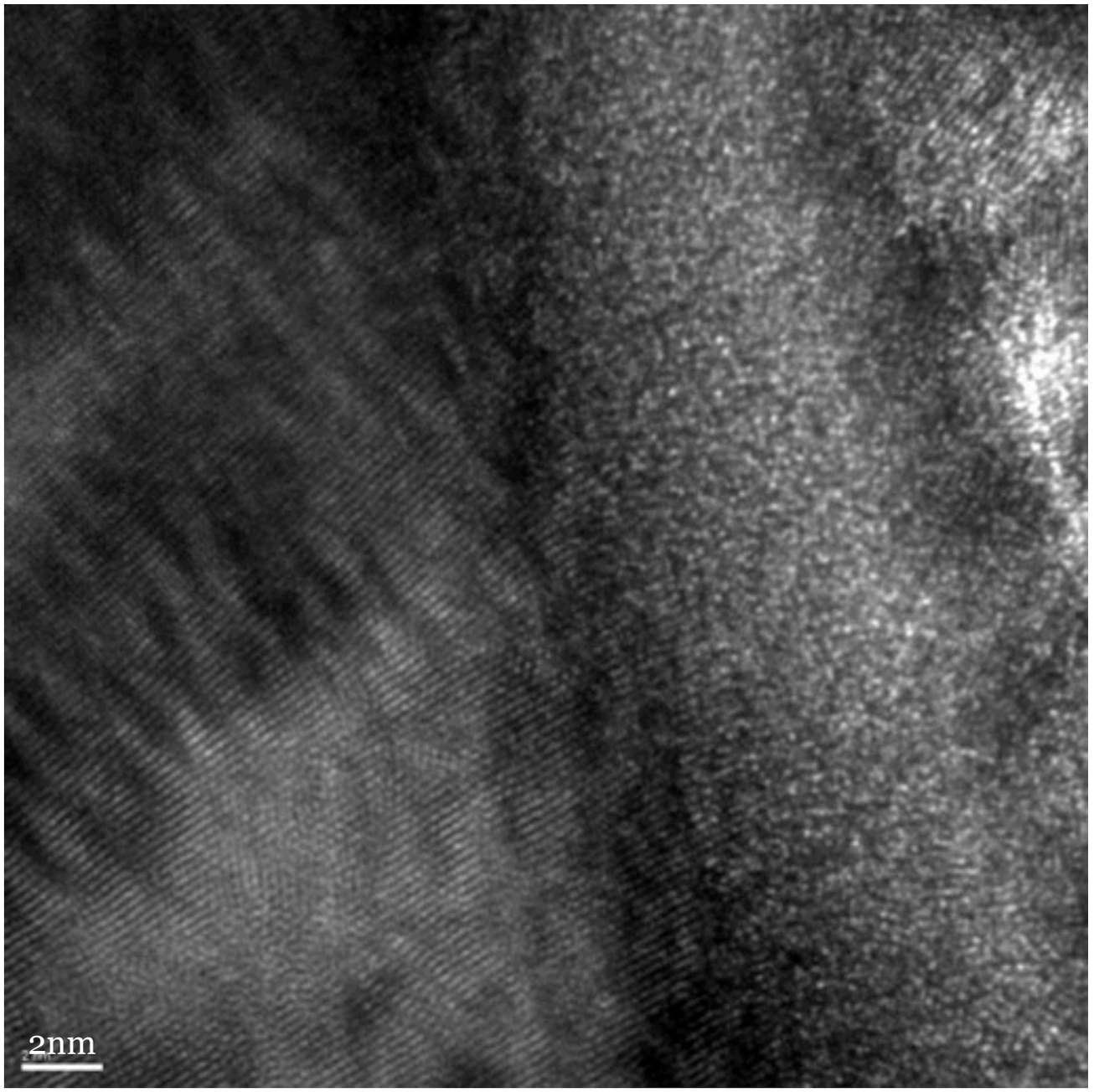


HRTEM imaging of Cold Spot at Room T continue



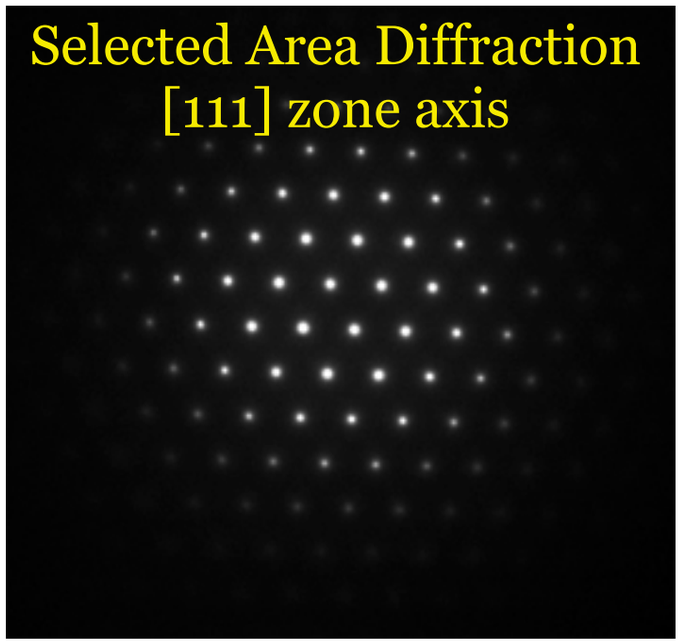


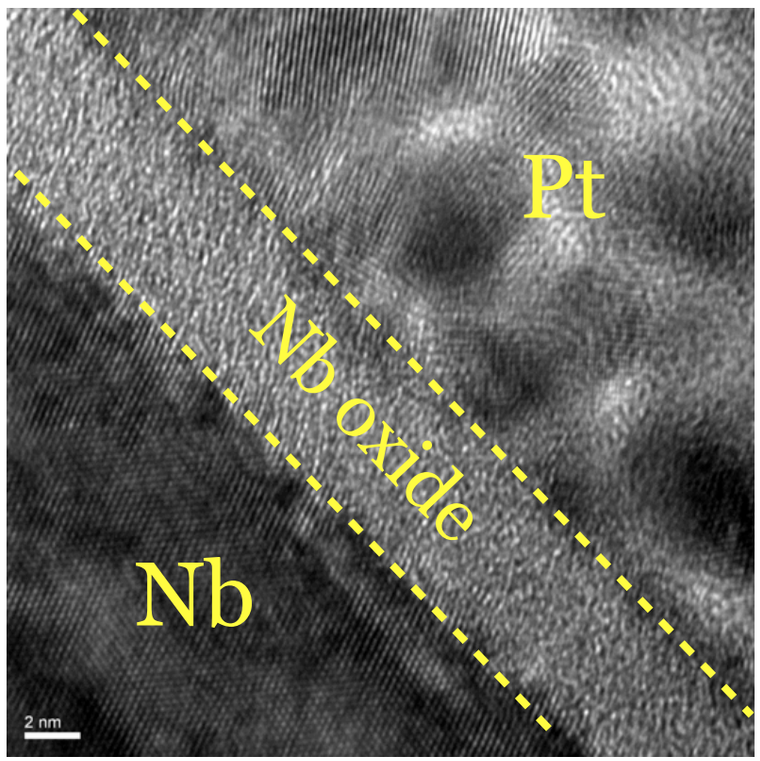
Phase Contrast of the Grain Boundary



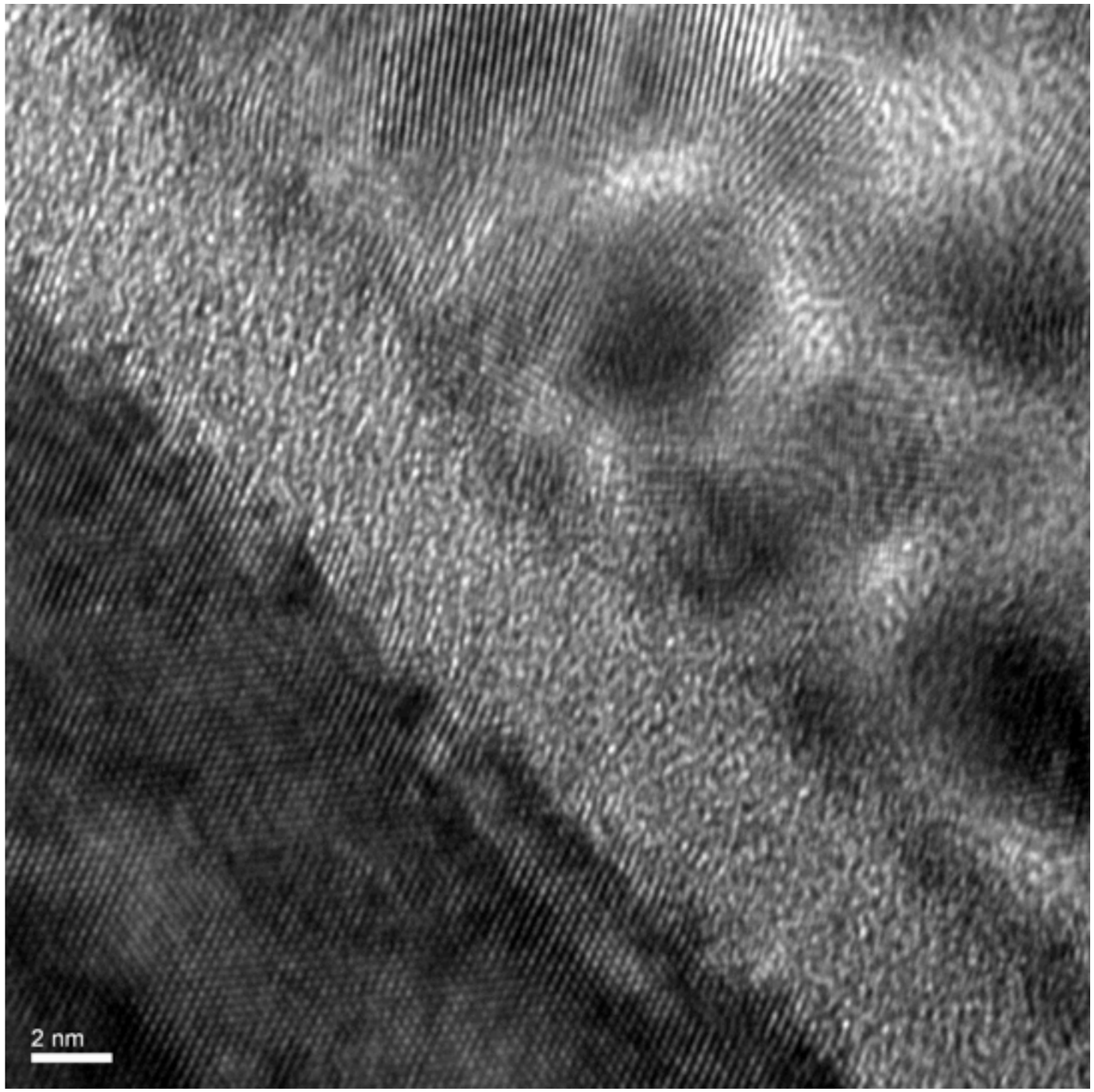
No significant oxidation along Grain Boundary in contradiction to J.Halbritter (2001)

HRTEM imaging of Hot Spot at Room T



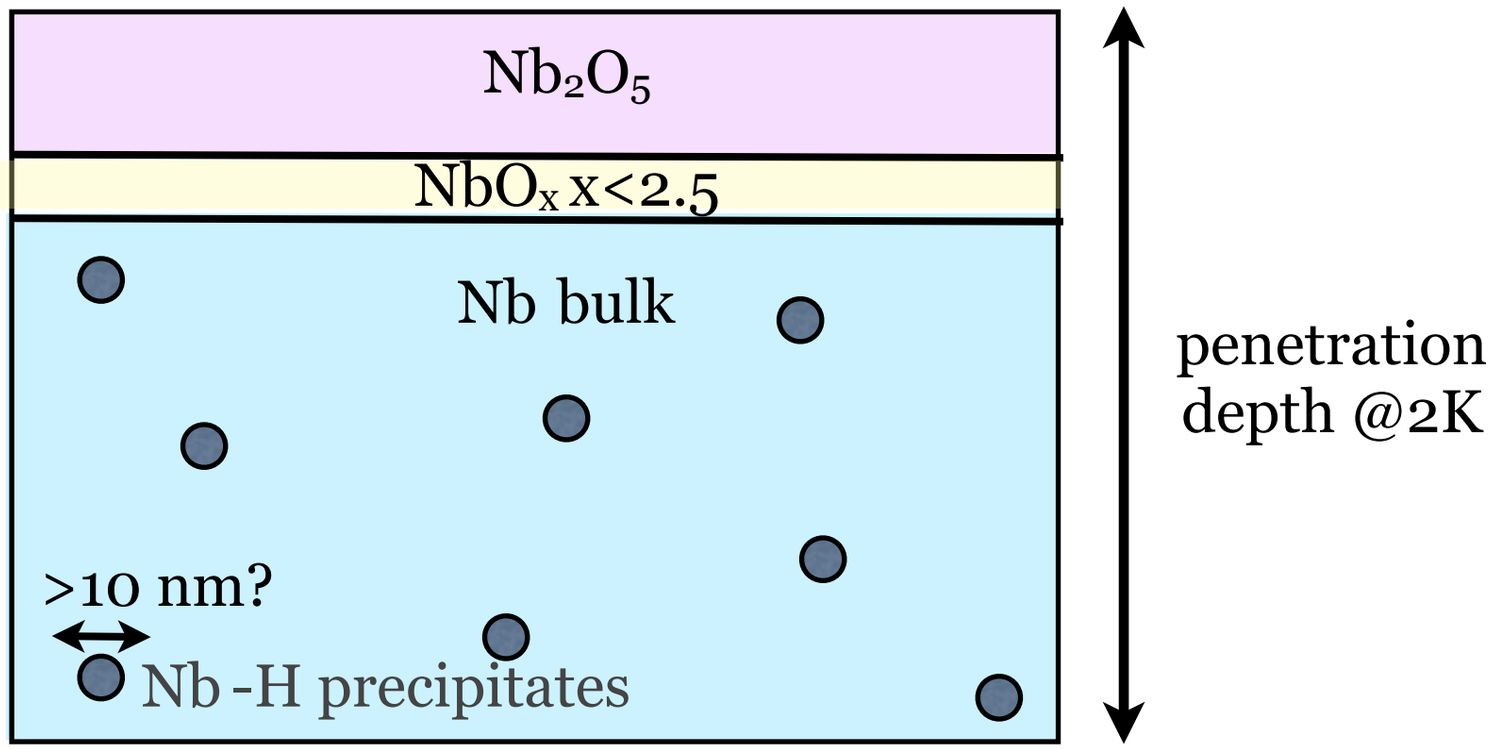


Phase Contrast of the interface Nb oxide - Nb



At Room T: No significant difference between Cold and Hot Spots.

What we expect to see at ~100K on Hot Spot:
Future Plans



ε-phase: NORMAL CONDUCTOR
Nb_4H_3 (orthorhombic, a~b=9.72Å, c~3.42Å)

Dissipations?
Work is in progress...

Thank you!

Acknowledgments:

Alexander Romanenko, Fedor Barkov (Fermilab)
Shu-You Li, Jinsong Wu (Northwestern University)

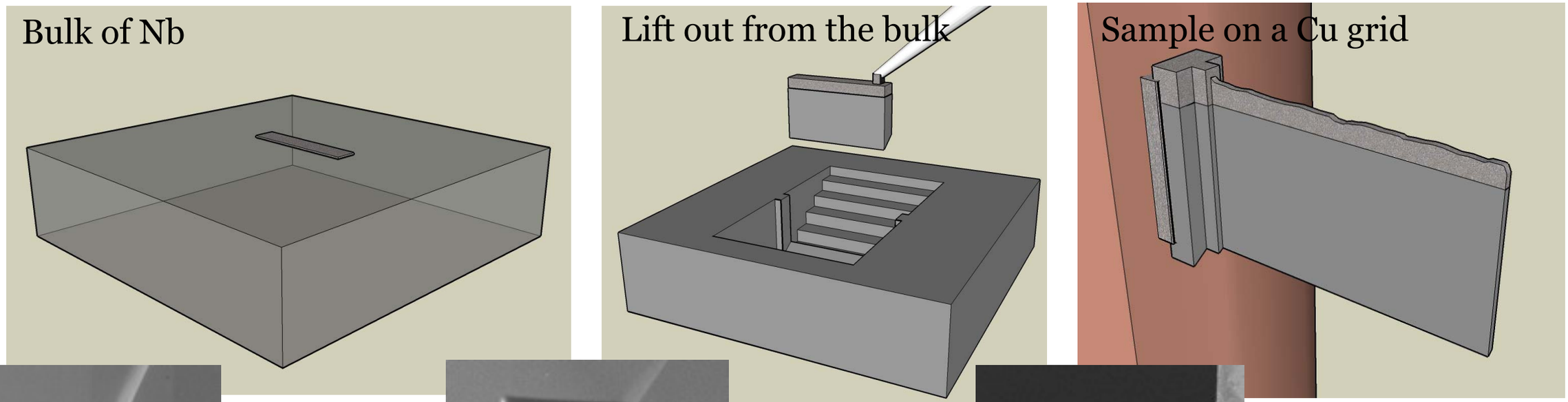
Sample Prep for TEM/STEM studies

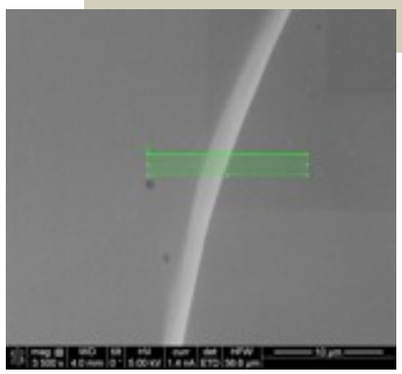
TEM: high spatial resolution but sample has to be thin *for HRTEM thickness < 50nm*

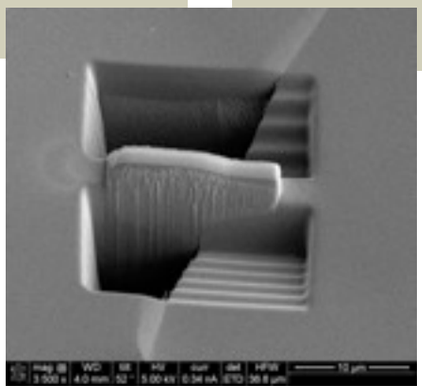
Focused Ion Beam:

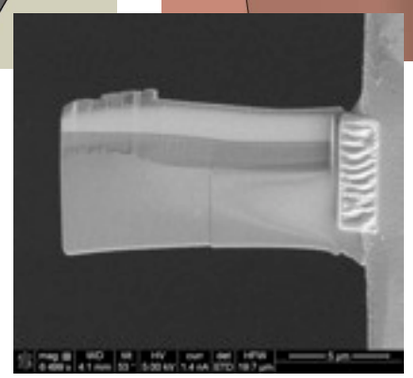
vertical SEM column + Ga ion column + micromanipulator
+ gas injection system + detectors

Schematic of the method [B.Myers]



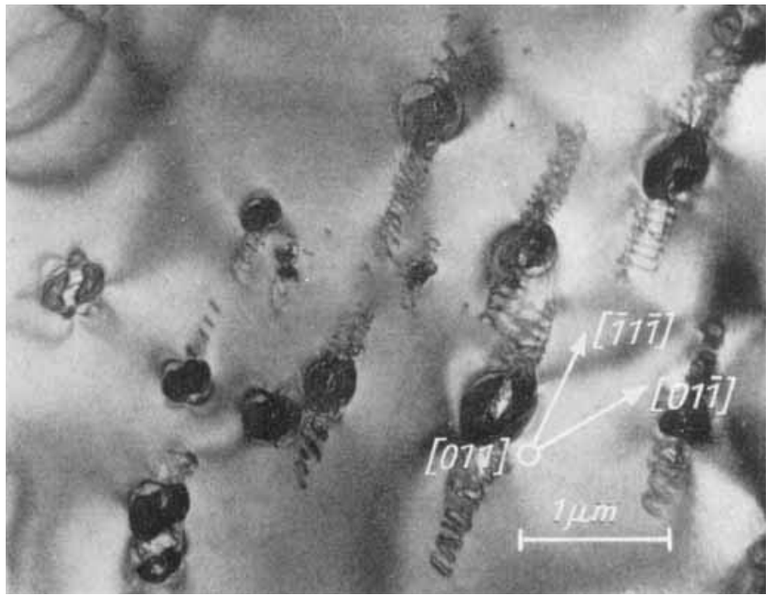




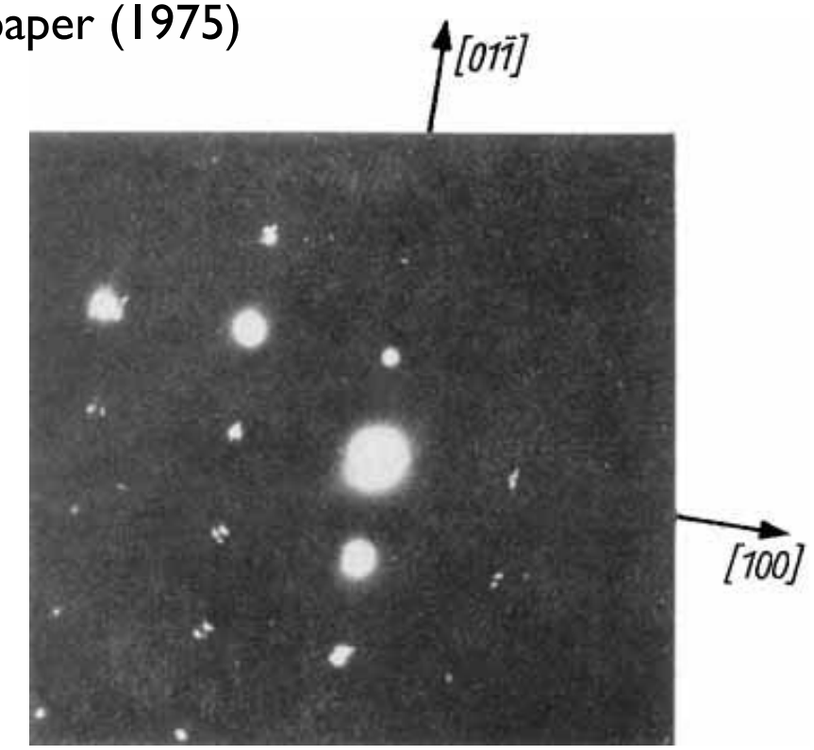


Result: cross-sectional cut from the bulk

Precipitation of H into epsilon phase at similar concentrations was observed by Schober TEM paper (1975)



TEM kinematical image along [111].T=-160C



SAD pattern[110]. T=-160C