Review of High Field Q-slope, Surface Measurements

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Motivation

- Key properties of the HF Q-slope
  - Mild baking removes completely (in EP) or improves it (in BCP)
    - Why the difference between EP and BCP after baking?
  - Air exposure has no effect on baking benefit
  - Observed distribution of losses in the high magnetic field region is not uniform – "hot" and "cold" spots
    - But – both "hot" and "cold" regions exhibit HF Q-slope, only amount of heating is different
- Surface studies – looking for:
  - What changes during mild baking and is preserved after air exposure?
    - Fine, large and single grain Nb BCP and EP samples
  - What is different between "hot" and "cold" regions?
    - Samples dissected from a not baked cavity with the HF Q-slope

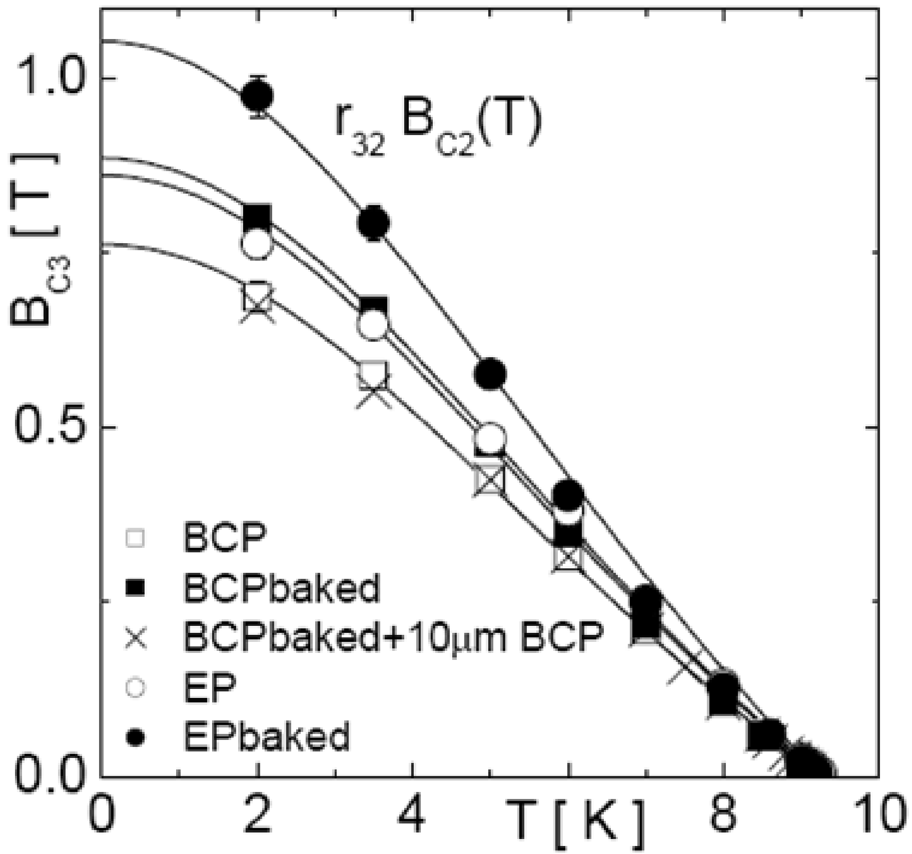
Tools Used

- Roughness
  - AFM, OP
- Grain Boundaries
  - Magneto-optical imaging
- Oxide structure
  - XPS
- Interstitial impurities (O, H, N, …)
  - SIMS, ToF-SIMS, 3DAP
- Crystalline microstructure
  - EBSD

What changes during mild baking and is preserved after air exposure?

Baking Effect: B_{c3}

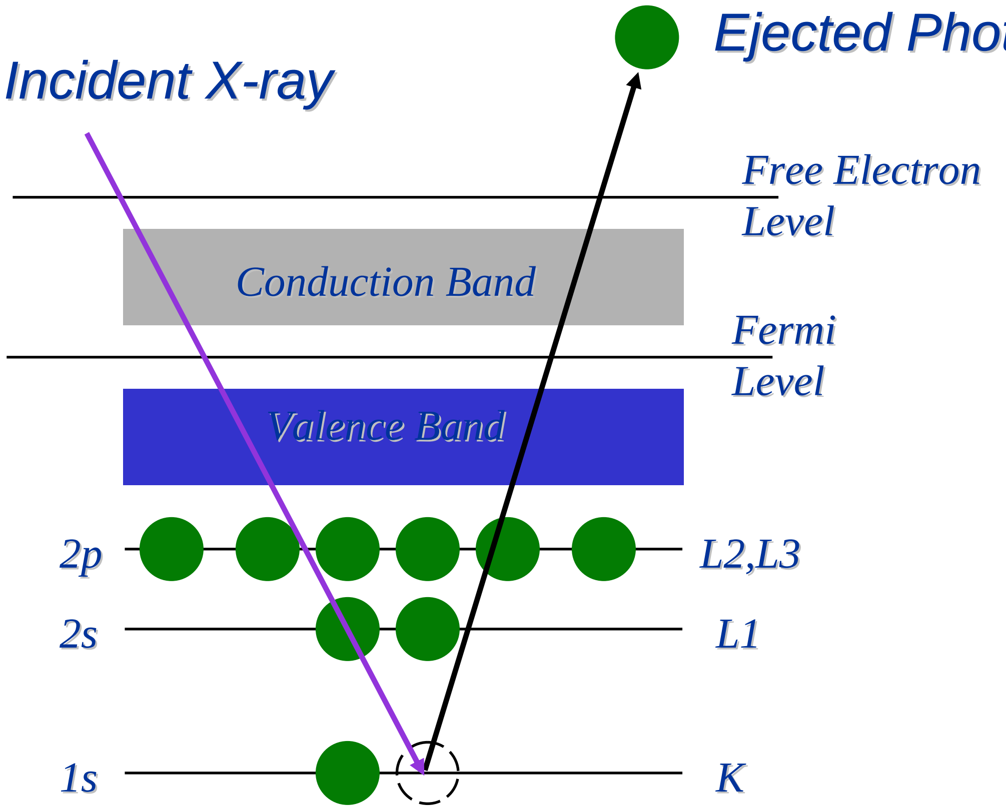
[S. Casalbuoni et al, Hamburg]



- Baking effect clearly observed
- EP superior to BCP after baking – higher B_{c3}?

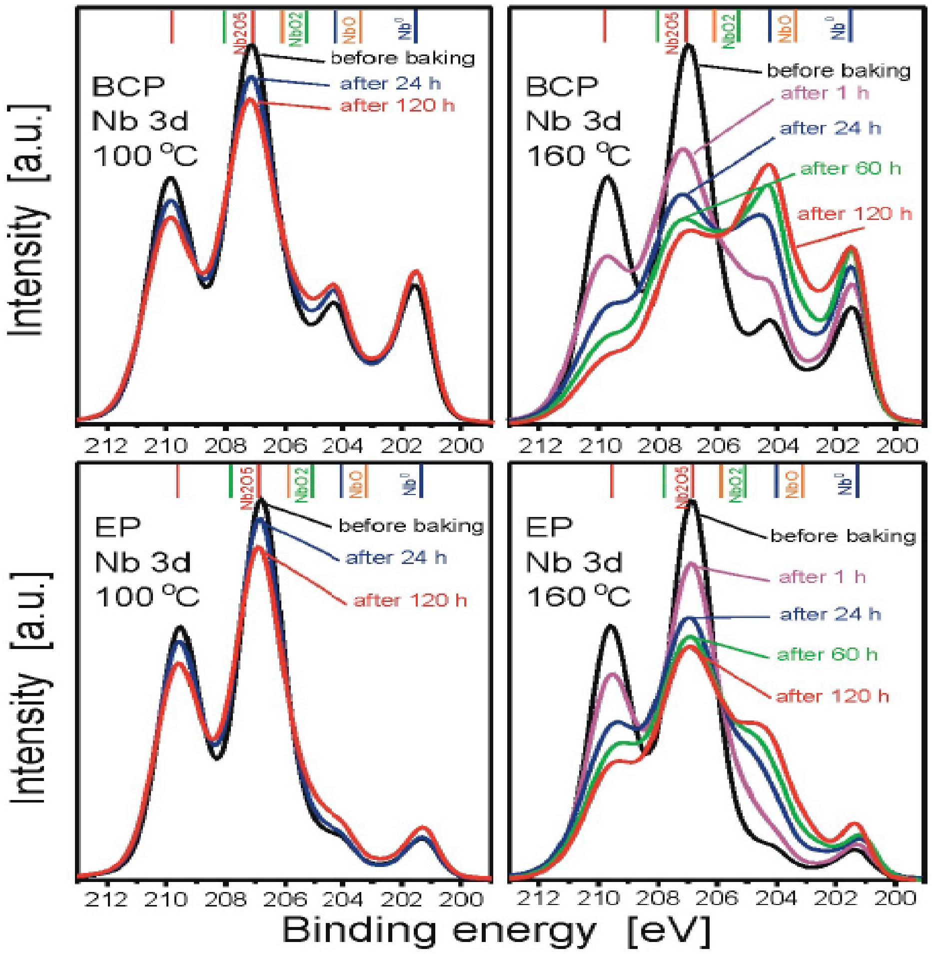
Is it oxide structure?

X-Ray Photoelectron Spectroscopy



- Elemental composition within first few nm (except for H and He)
- Chemical state information
- Sensitivity limited to about 0.1-1 at.%
- Non-destructive depth profiling possible with variable X-ray energy or angle-resolved XPS
- But - mostly oxide signal

Oxide structure (XPS)

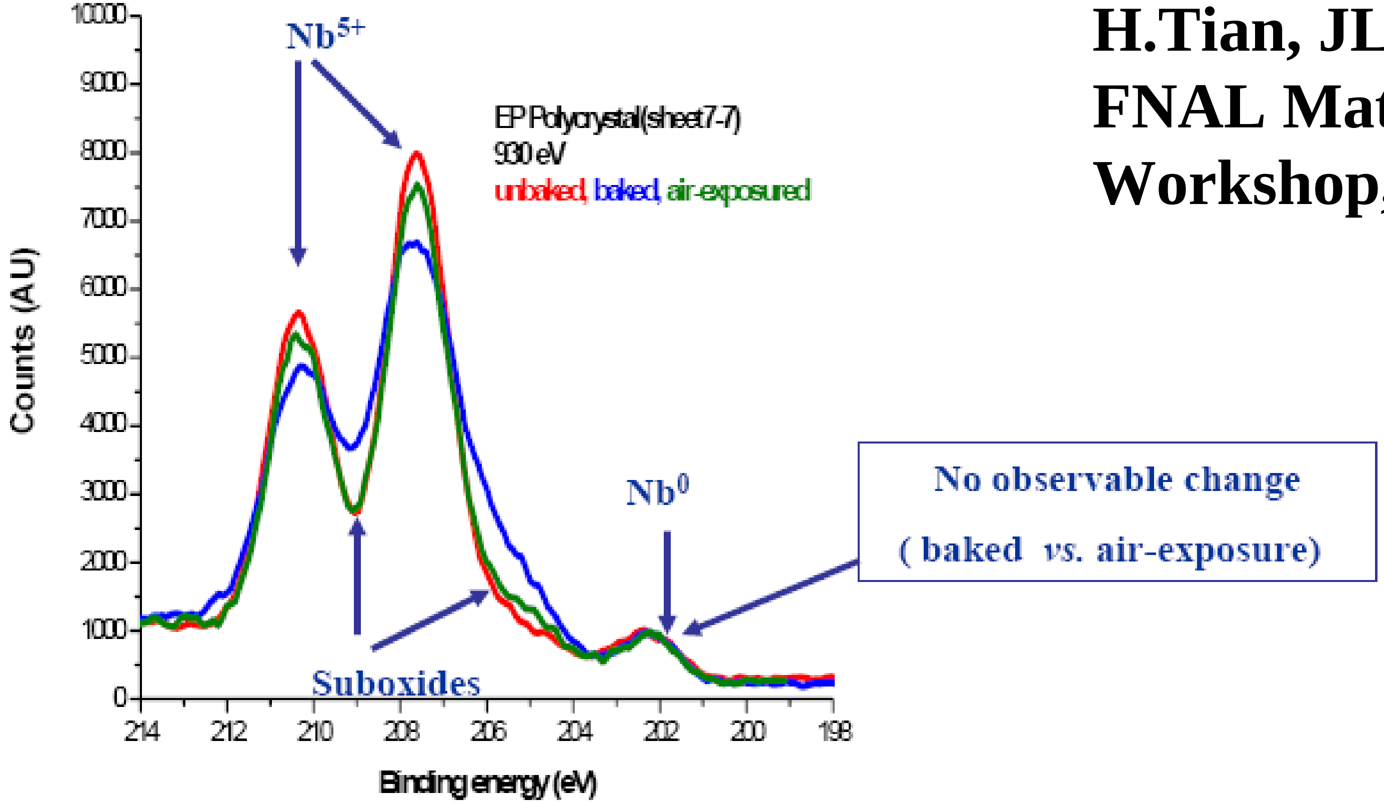


Kowalski et al., SRF'01

- Al Kα hν=1486.6 eV
- Systematic study of mild baking effect on niobium oxide structure
- BUT only "in situ" – no air exposure
- Nb_2O_5 gets partially converted to suboxides – possible cause of residual losses increase (up a few nOhms)

Baking + Air exposure (XPS)

Classic recipe : 120 º C , 48 hrs, UHV



H.Tian, JLab, FNAL Material Workshop, 2007

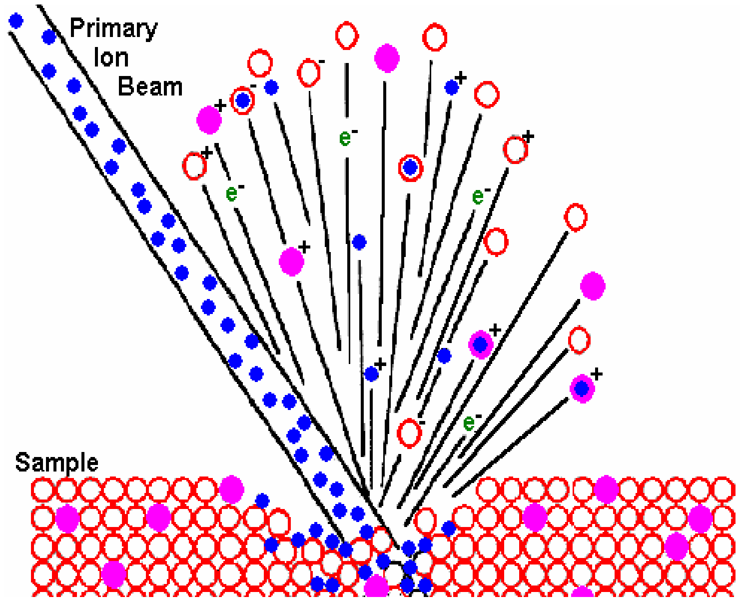
- Air exposure = back to unbaked – oxide and interface are not responsible

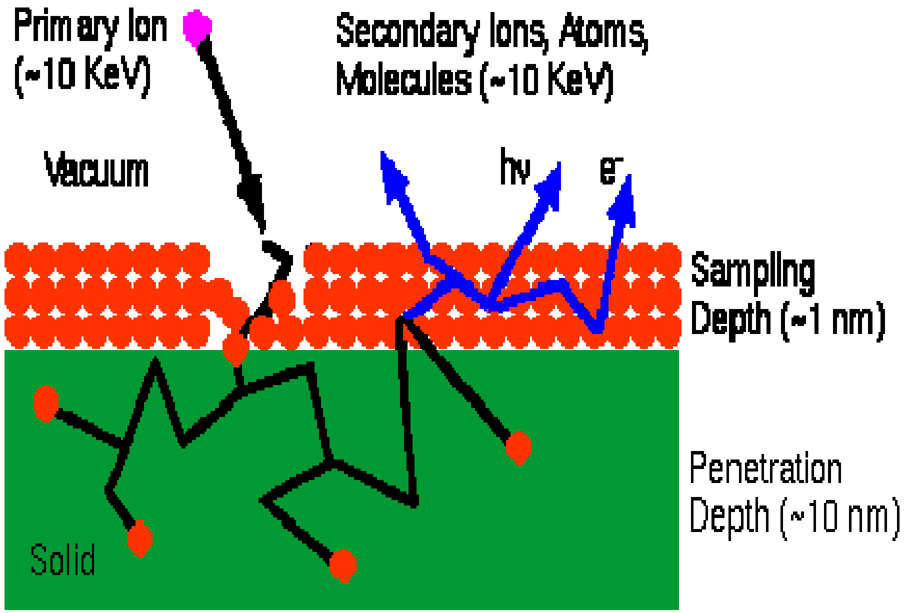
What XPS tells us?

- Oxide structure = mostly Nb_2O_5 + small amount of suboxides
- Mild baking = Nb_2O_5 -> suboxides + slight breakup
- Air exposure after baking = oxide structure back to before baking
- Oxide modification is not responsible for HF Q-slope

Is it interstitial impurities (O, H,…)?

Secondary Ion Mass Spectrometry



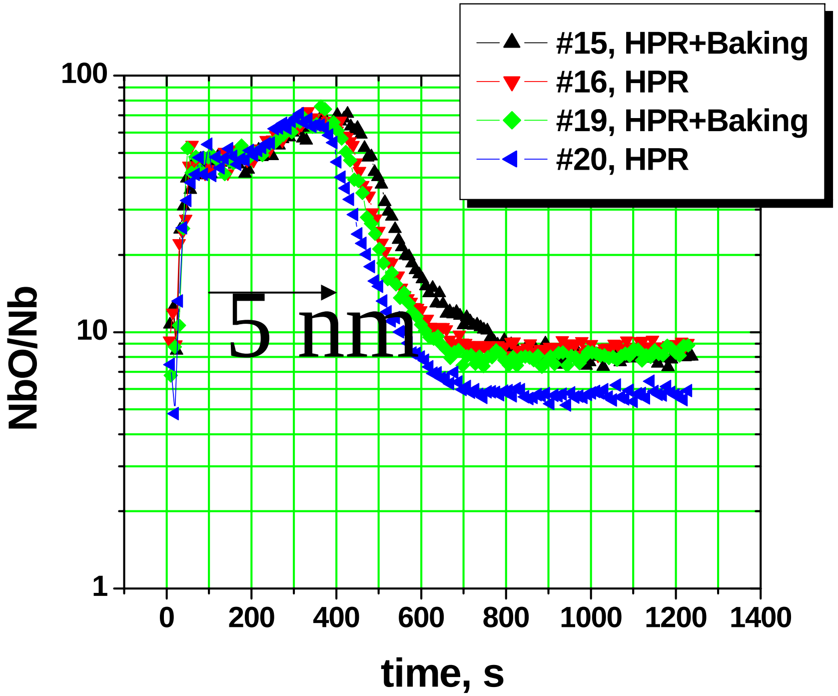


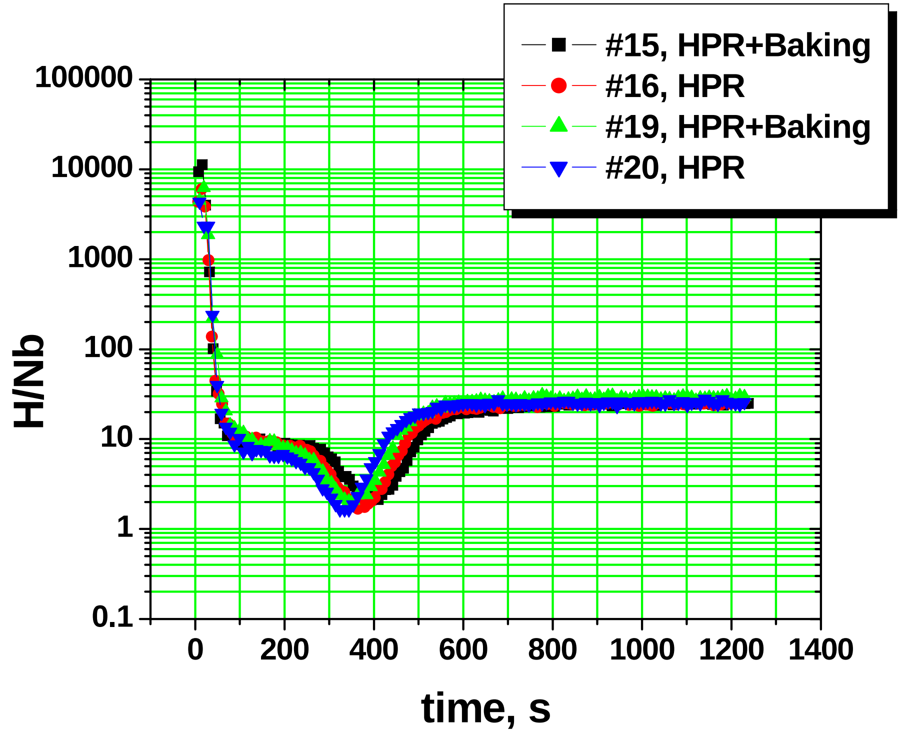
- Very sensitive – ppb detection possible
- Destructive depth profiling

BUT

- Instrumental effects – preferential sputtering of oxygen, roughness effect on signal, chemical information not reliable due to sputtering-induced ion production

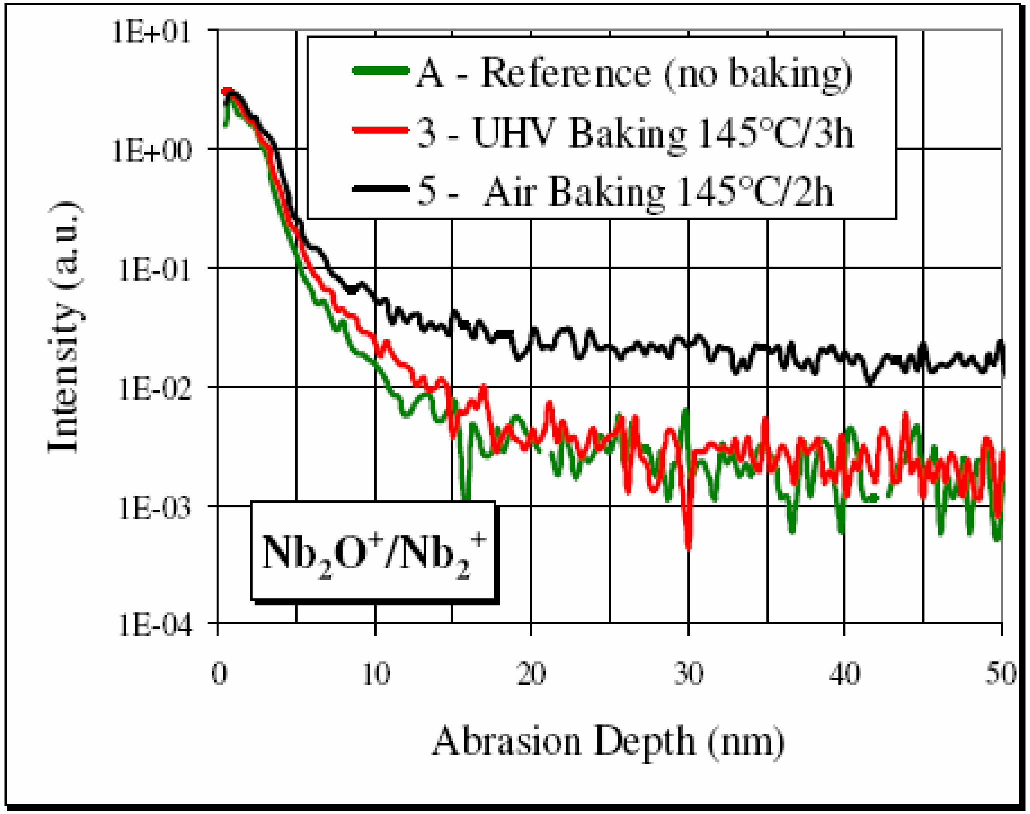
ToF-SIMS (G.Eremeev, Cornell, J.Francis, UWO)





- Single grain BCP samples
- HPR + 110C 48hrs UHV baking + air exposure
- Spot to spot variation comparable to bake/unbake
- O and H profiles unchanged due to bake

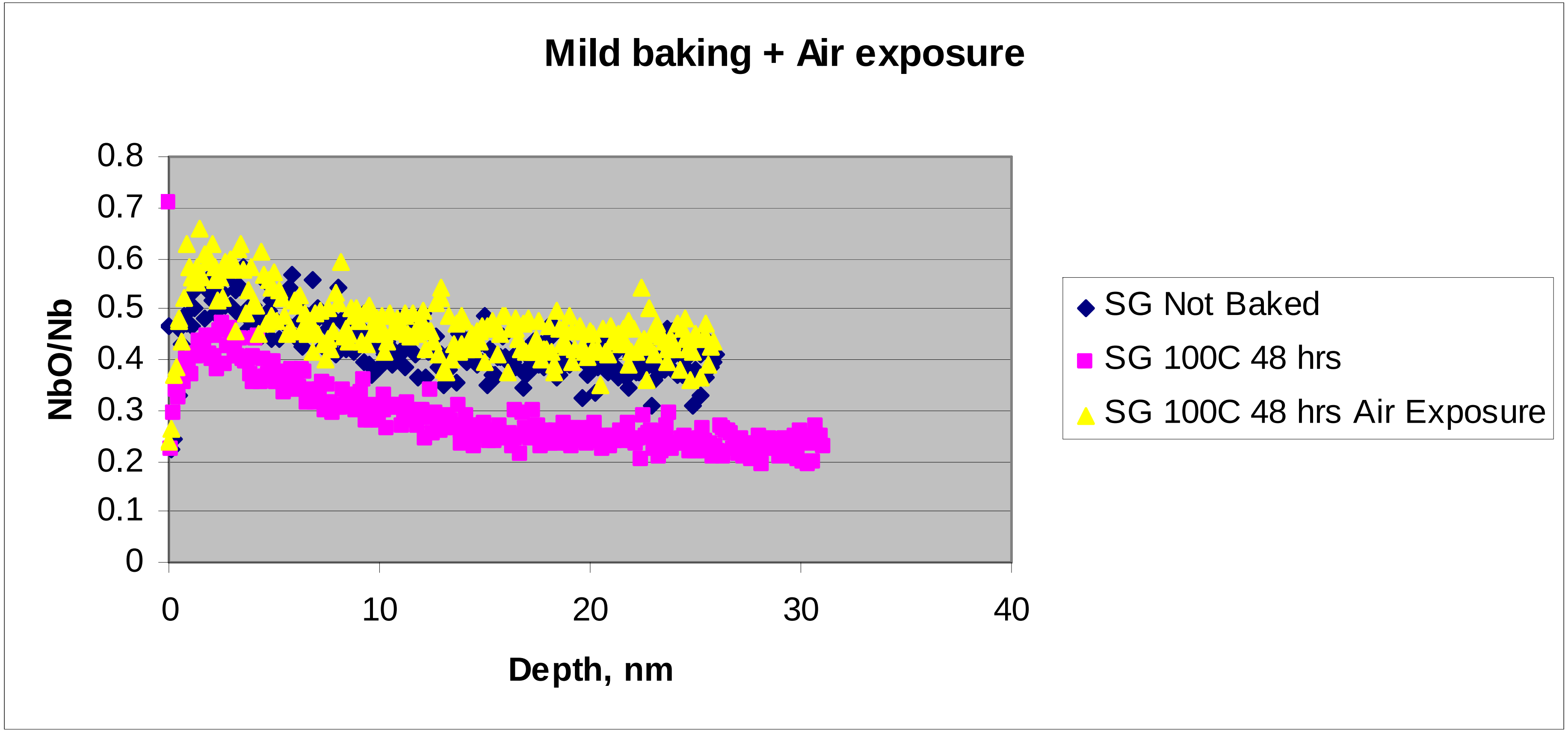
Baking Effect (SIMS)



B.Visentin, Thin Film Workshop, 2006

- Oxygen diffusion is not responsible for mild baking improvement
- But responsible for degradation after higher T baking

Baking + Air Exposure (SIMS)



Single grain BCP

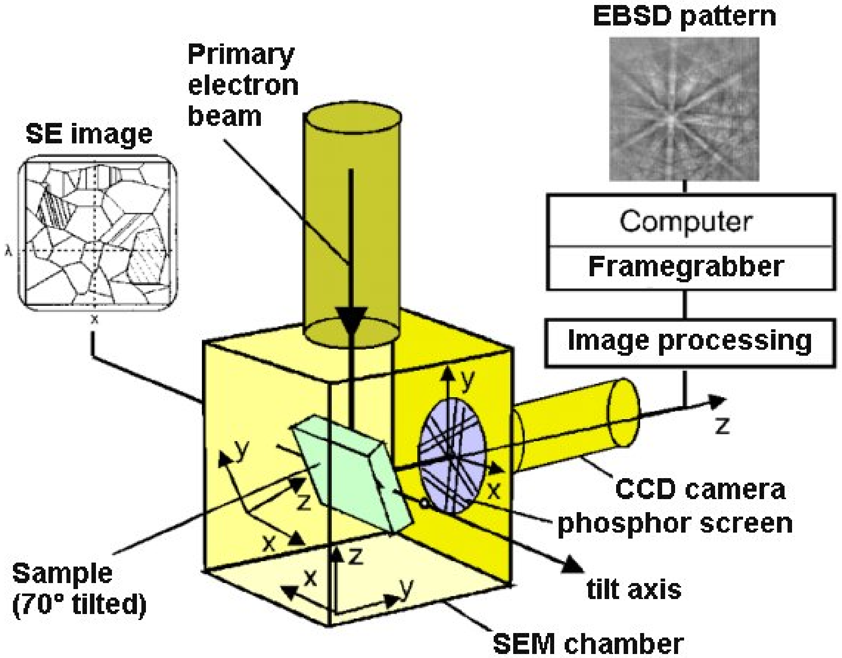
J.Kaufman, Cornell

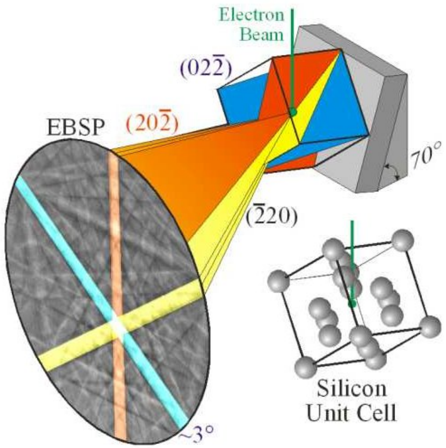
SIMS Conclusions

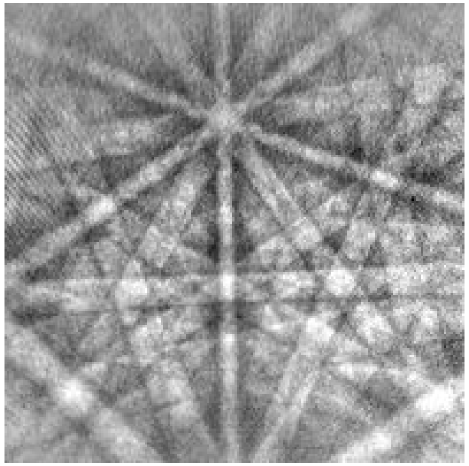
- No conclusive evidence that oxygen-(impurity-) enriched layer underneath the oxide is responsible for HF Q-slope
- Insignificant change in O related signals after mild baking
  - As opposed to significant effect on cavity performance
- Changes in surface O, NbO, … due to baking eliminated by air exposure
  - As opposed to cavity performance, which stays same after air exposure

Is it crystalline microstructure?

Electron Backscattered Diffraction

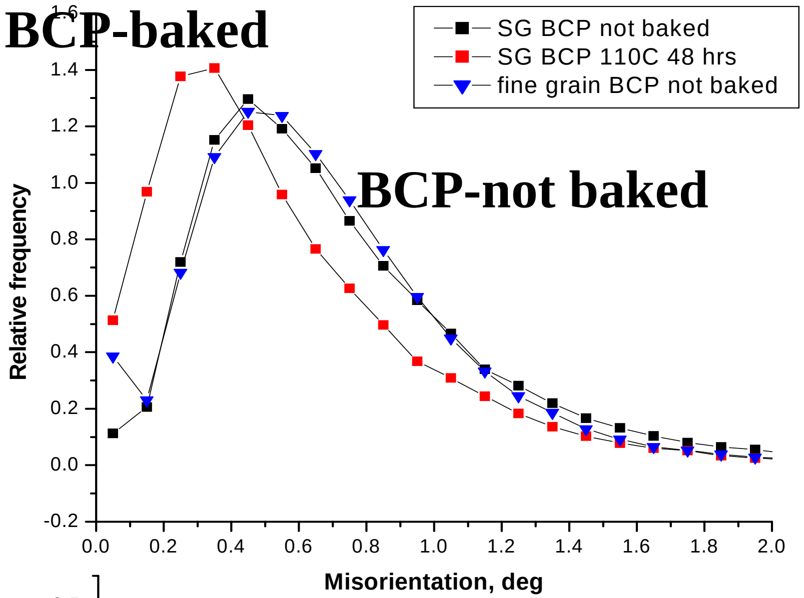


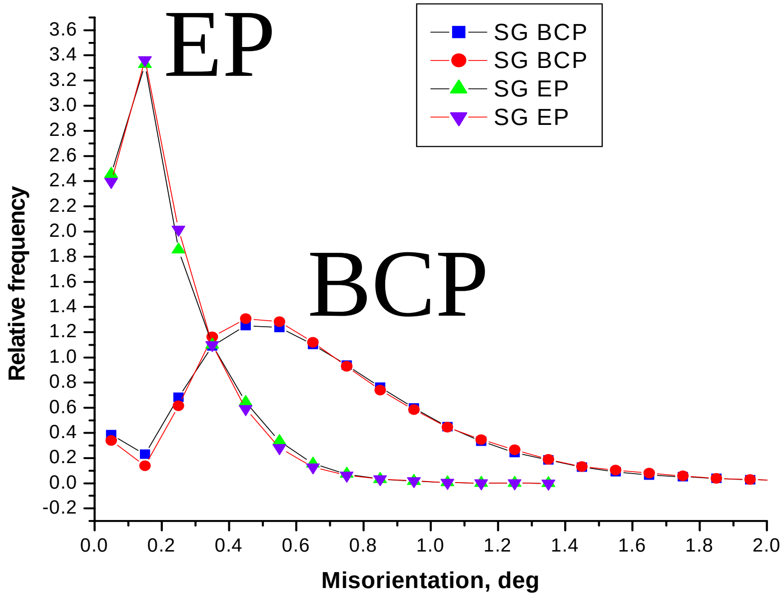


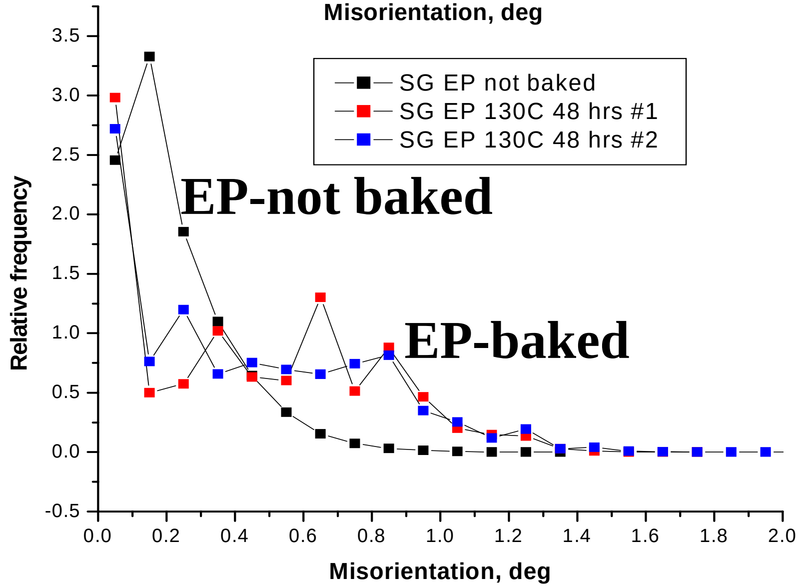


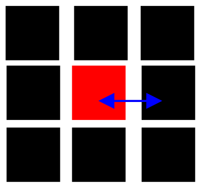
- Based on diffraction of backscattered electrons
- Information depth – 20-100 nm
- Crystallographic orientation mapping
- Information on crystal defects distribution

EBSD: Local Misorientation









$\Delta\alpha_i$ – angle to rotate the unit cell to make orientations equal

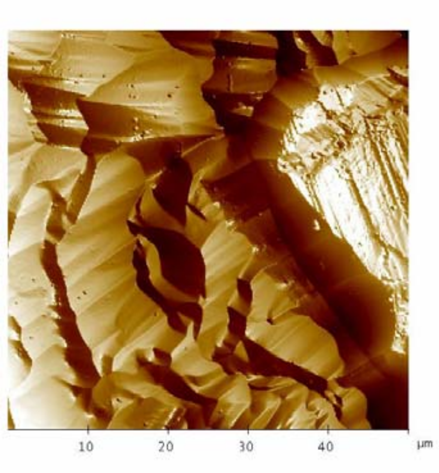
Local misorientation = 1/8 * $\Sigma(\Delta\alpha_i)$

Baking effect is seen

Roughness role?

AFM: Roughness is the major difference between EP and BCP

BCP



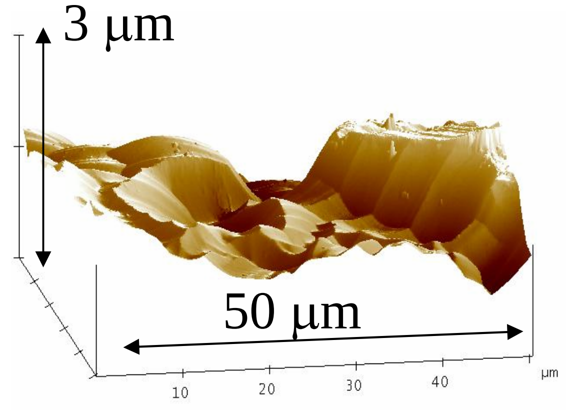
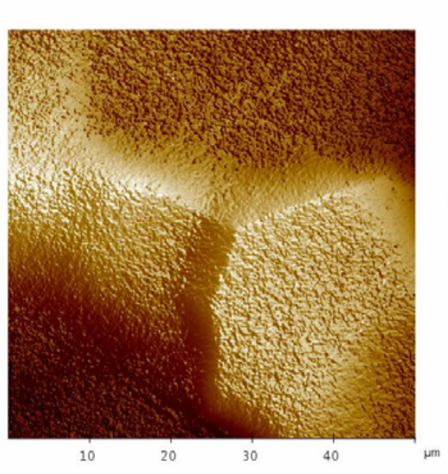


Image Statistics

Img. Z range	2.681 µm
Img. Mean	0.077 nm
Img. Raw mean	-243.23 nm
Img. Rms (Rq)	447.55 nm
Img. Ra	371.30 nm
Img. Rmax	2.608 µm
Img. Srf. area	2526.5 µm²
Img. Prj. Srf. area	2500.0 µm²
Img. Srf. area diff	1.061 %
Img. SAE	1.005

EP



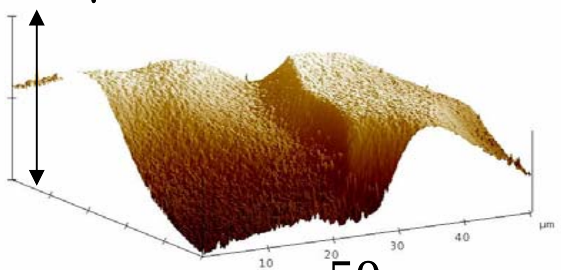


Image Statistics

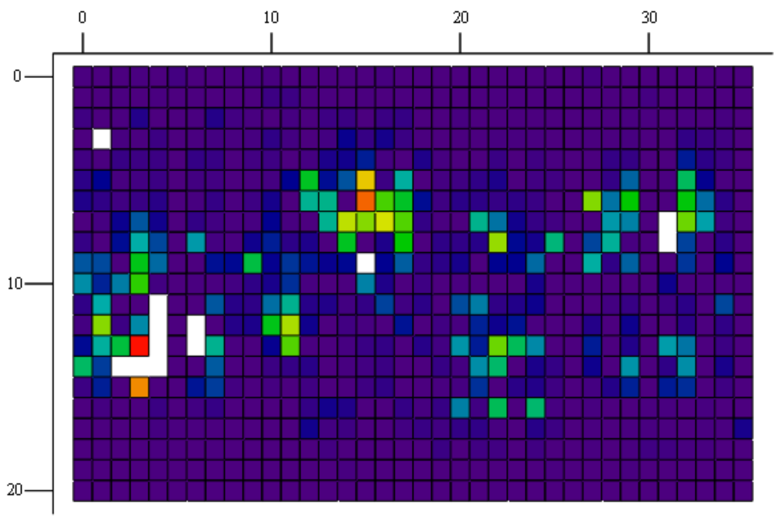
Img. Z range	349.61 nm
Img. Mean	0.121 nm
Img. Raw mean	-344.43 nm
Img. Rms (Rq)	78.660 nm
Img. Ra	67.523 nm
Img. Rmax	345.21 nm
Img. Srf. area	2501.3 µm²
Img. Prj. Srf. area	2500.0 µm²
Img. Srf. area diff	0.051 %
Img. SAE	1.000

EP - “smoother”, rounded GB steps

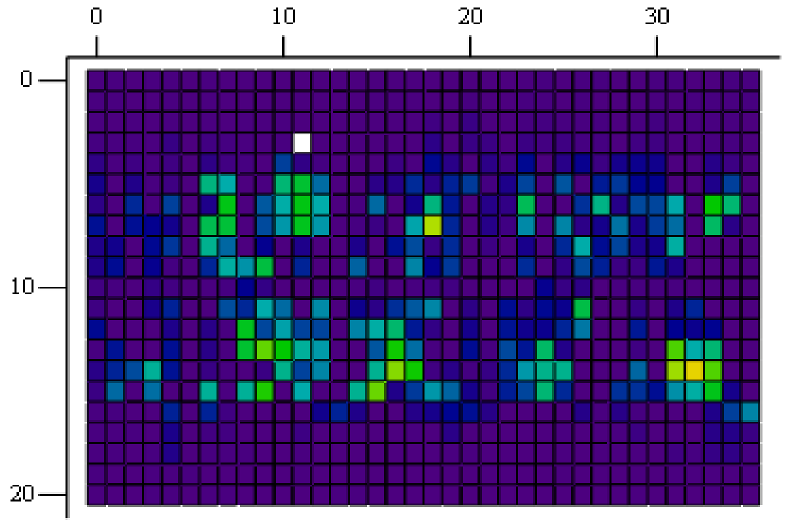
Mild baking conclusions

- Oxide not responsible
- Interstitial O not responsible
- Roughness is not a cause of the Q-slope
  - Because EP and BCP have similar Q-slopes before baking
  - But it might contribute to full explanation of why EP is better than BCP after baking
- B_{c3} changes due to mild baking
- Crystalline defect structure may play a role

What causes non-uniformity of heating in the high field Q-slope regime?



Large Grain



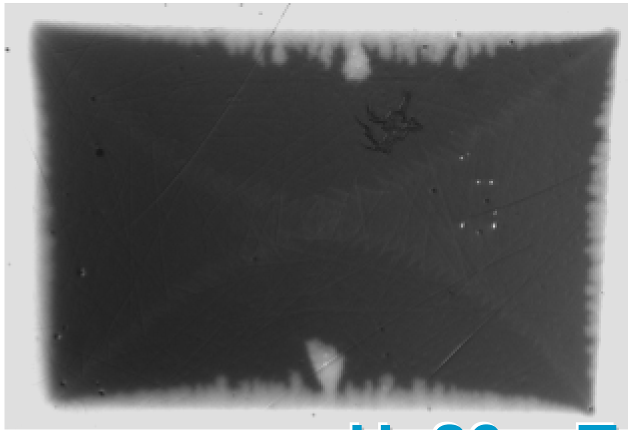
Small Grain

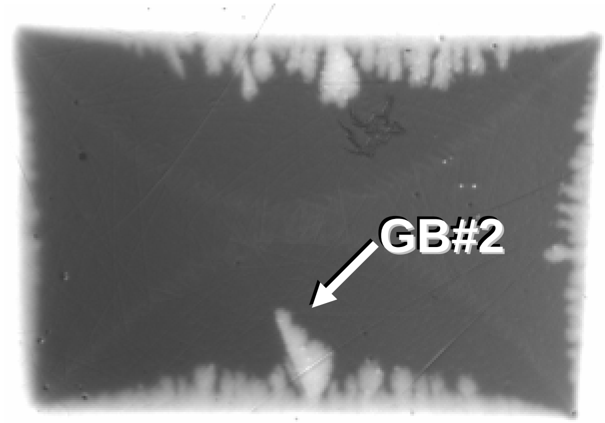
T_map

a

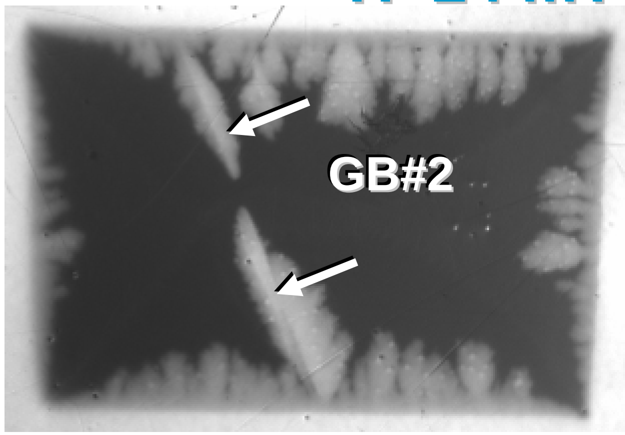
Magneto-Optical Imaging (FSU/ASU – Polianskiy et al.)


H=24 mT

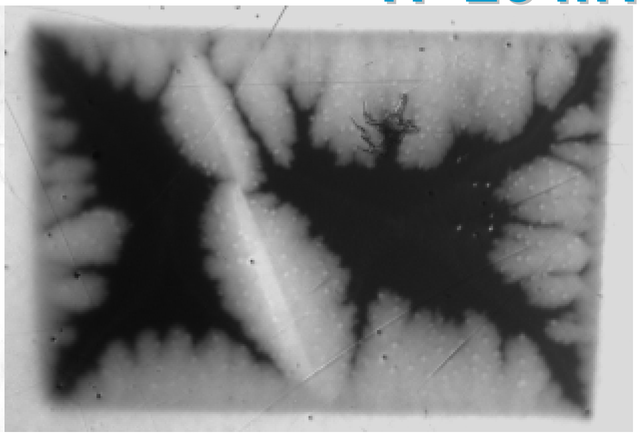

H=26 mT



H=28 mT



H=32 mT


H=40 mT

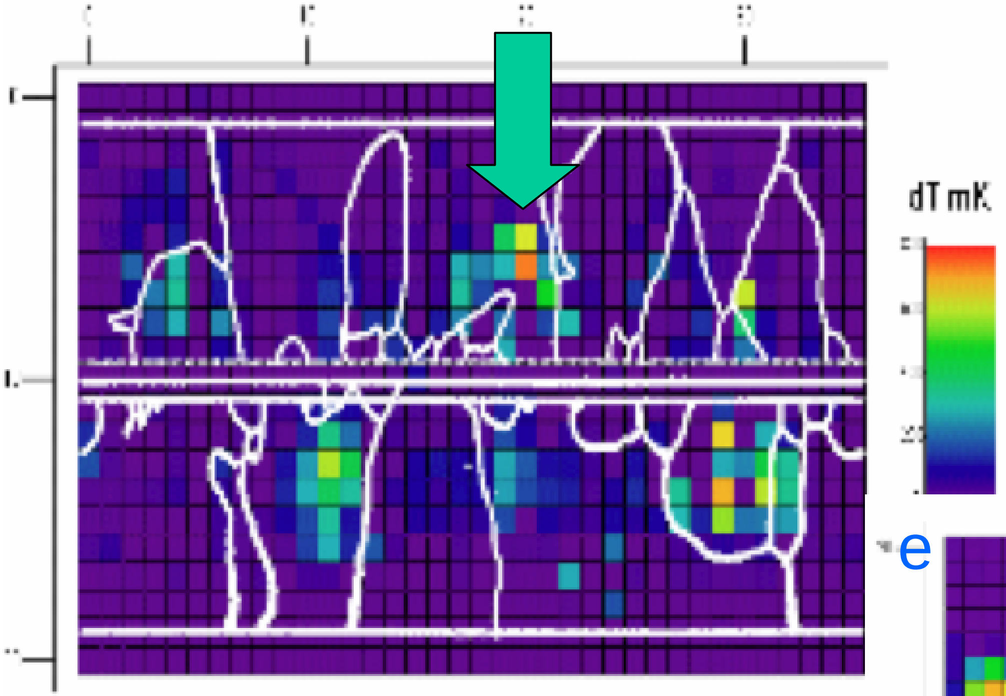
1 mm

Is it GBs?
DC - yes

T=6 K

DC Magnetic flux penetrates
when magnetic field is parallel to plane of GB).

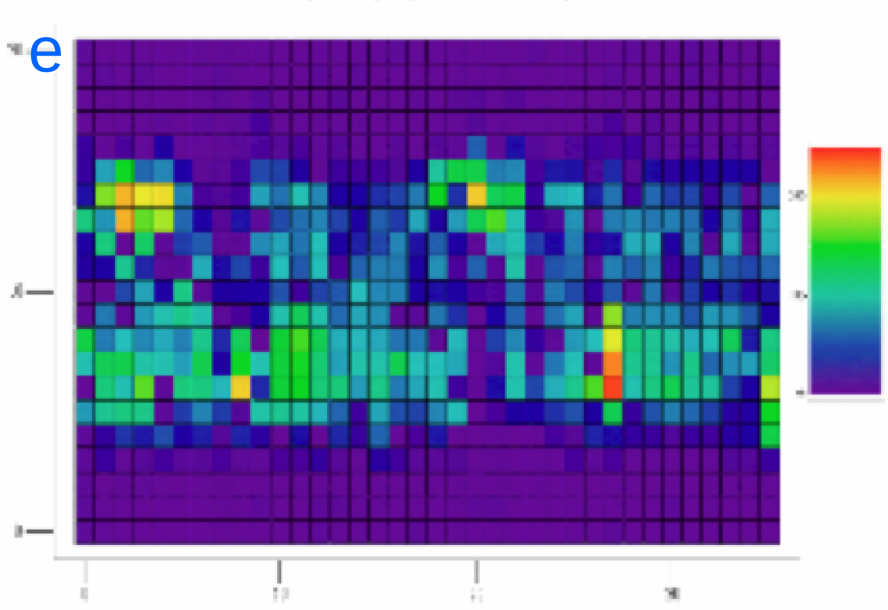
No heating at GBs (G.Eremeev, Cornell)



RF - No!

Fine-Grain Cavity

Red = 250 mk

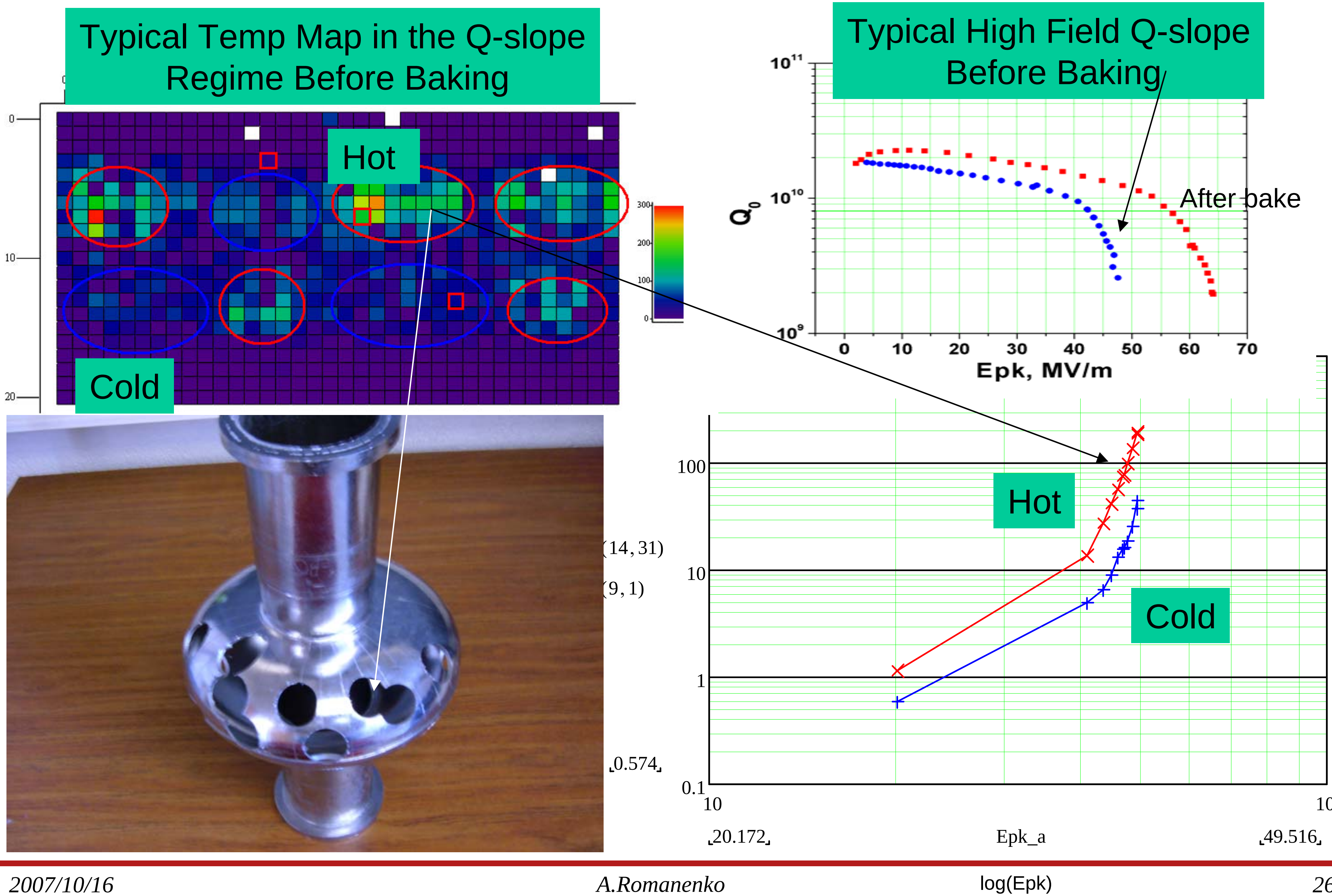


Large Grain Cavity

Red = 800 mK

- Grain boundaries are not the regions where stronger heating takes place

Cornell: Cavity Dissection



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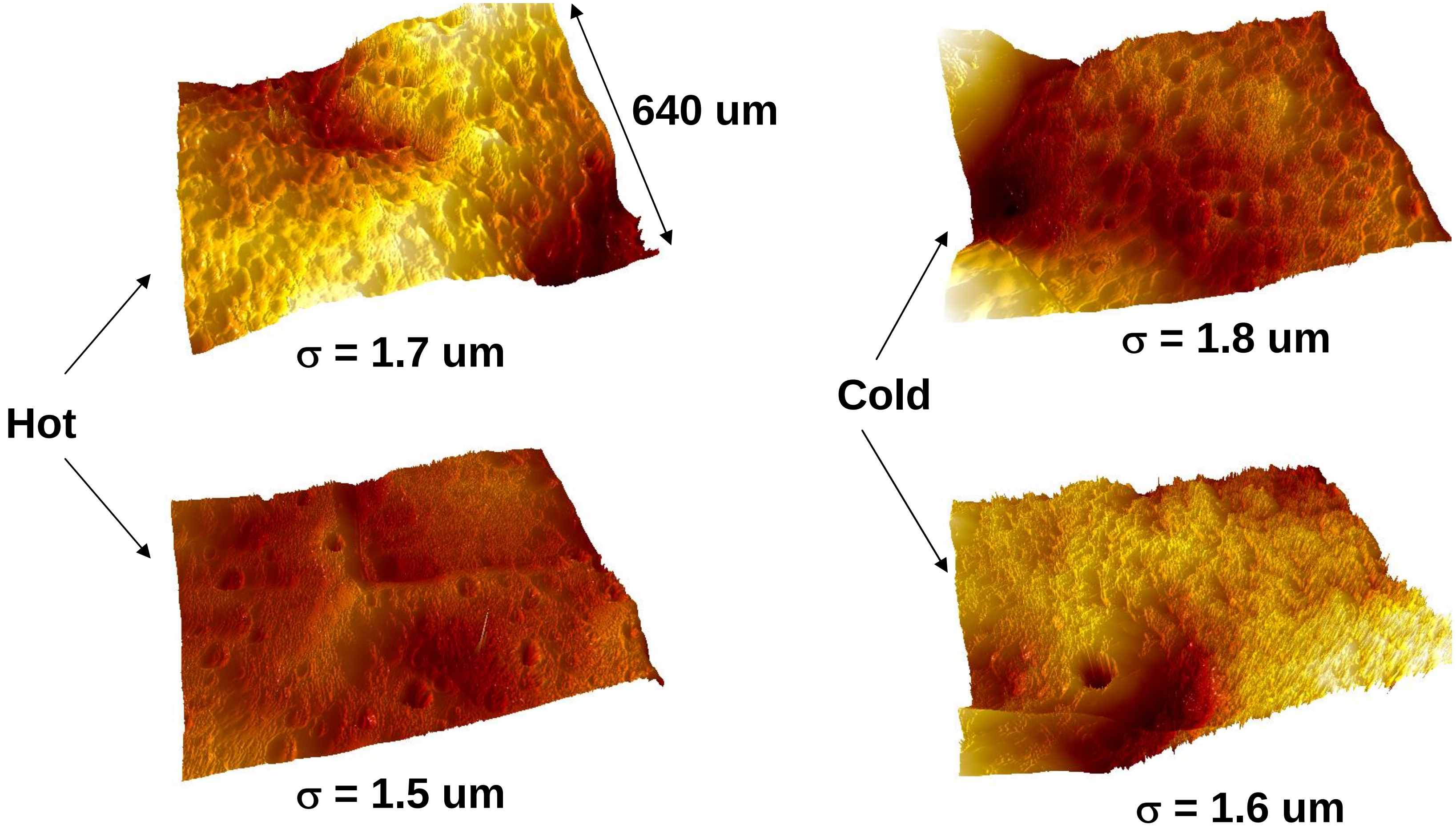
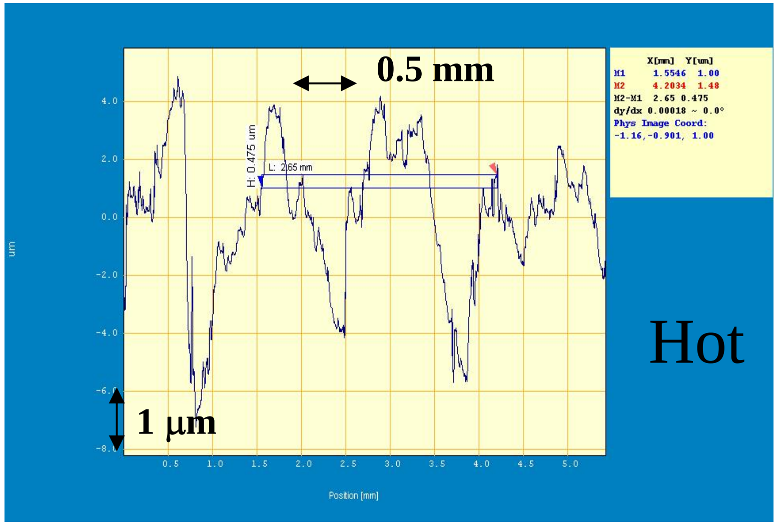
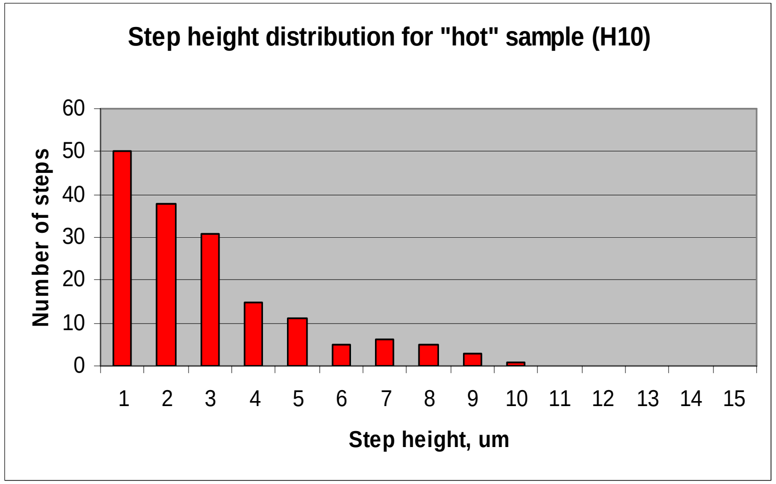
Is non-uniformity caused by roughness variations? – No!

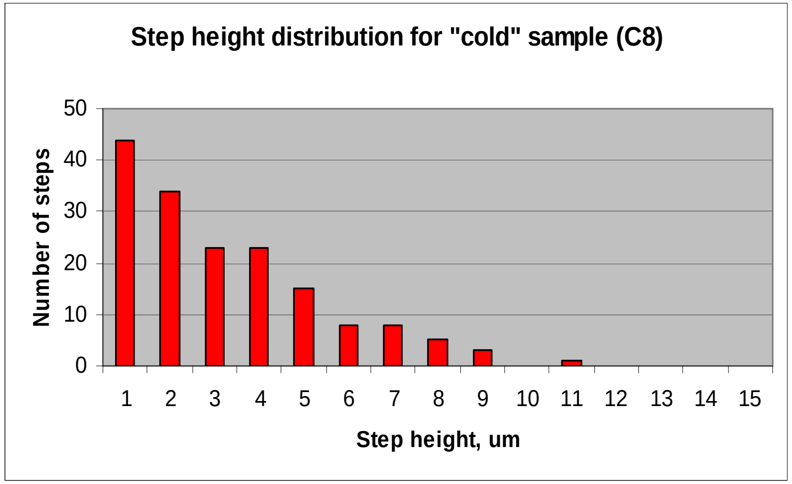


- Average roughness – σ = 1.5-1.8 μm is the same

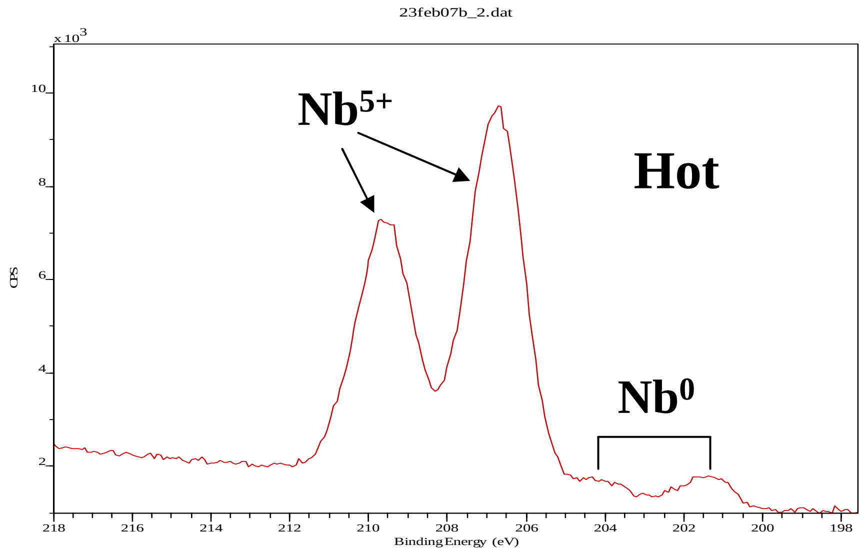
OP: No difference in step heights

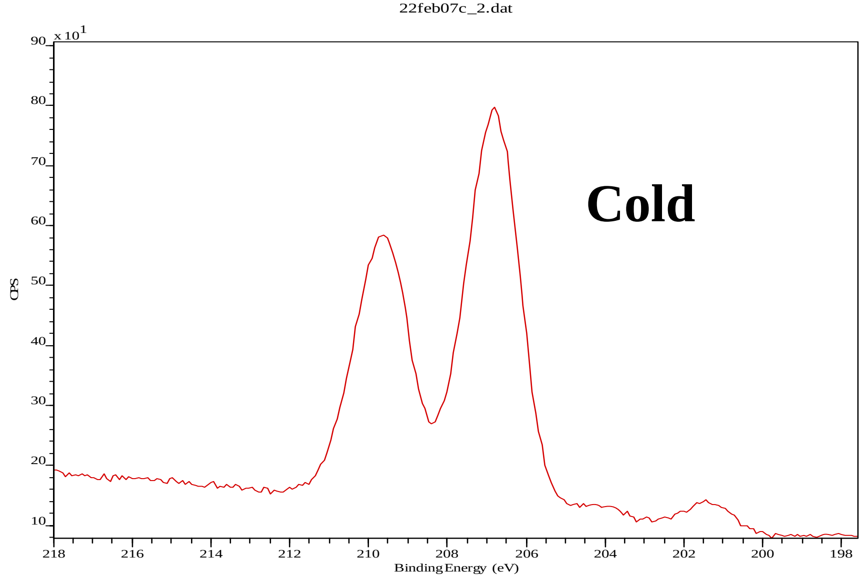






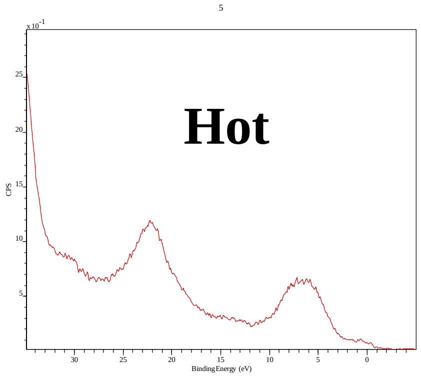
XPS: Oxide structure

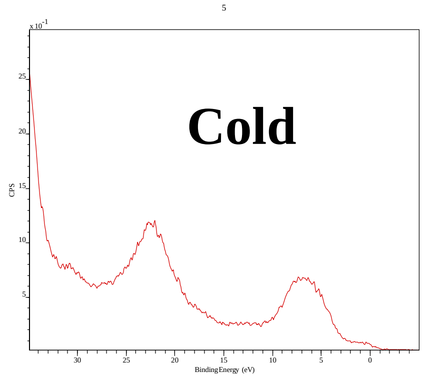




- Is patchiness caused by oxide structure differences? – No!

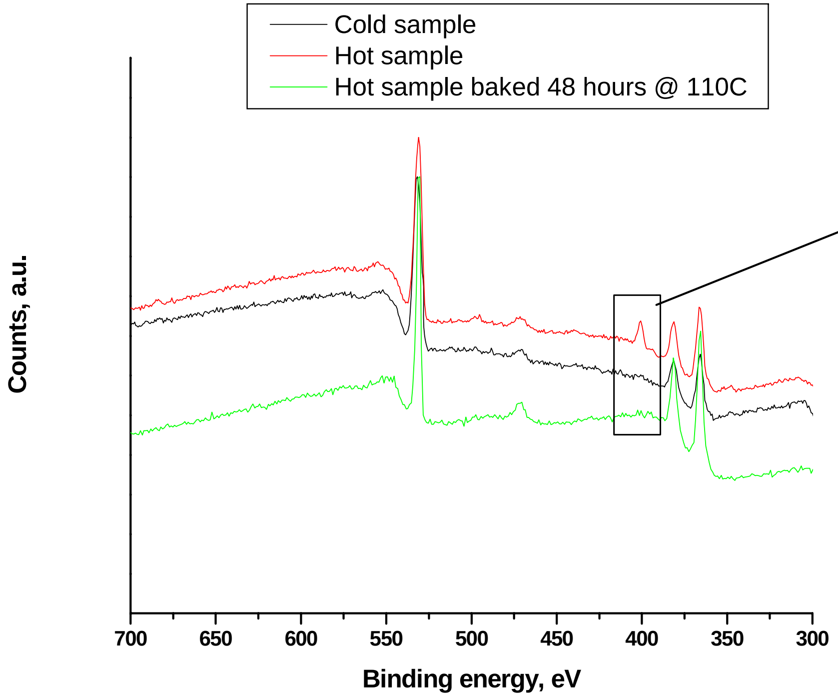
Valence band

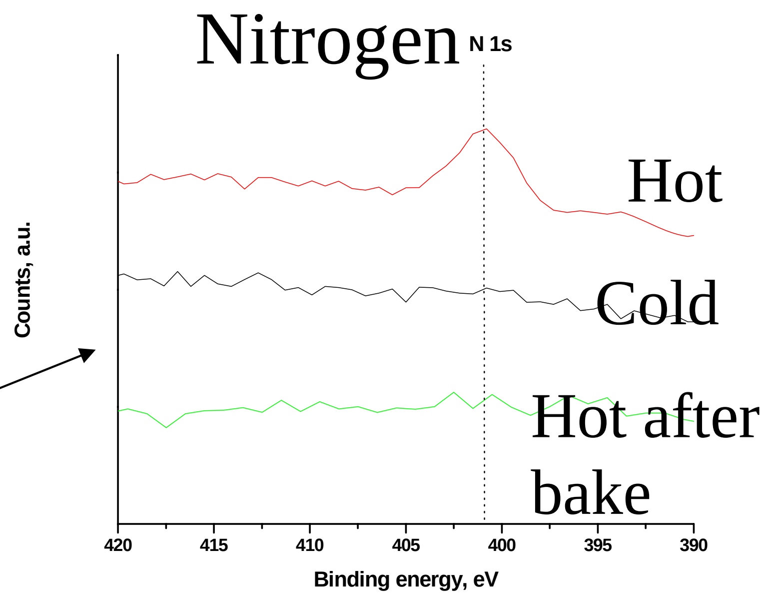




Is non-uniformity caused by contaminants other than O?

- Al Kα 1486.6 eV X-ray source
- Information depth ~7 nm
- 0.5-1 mm grain size

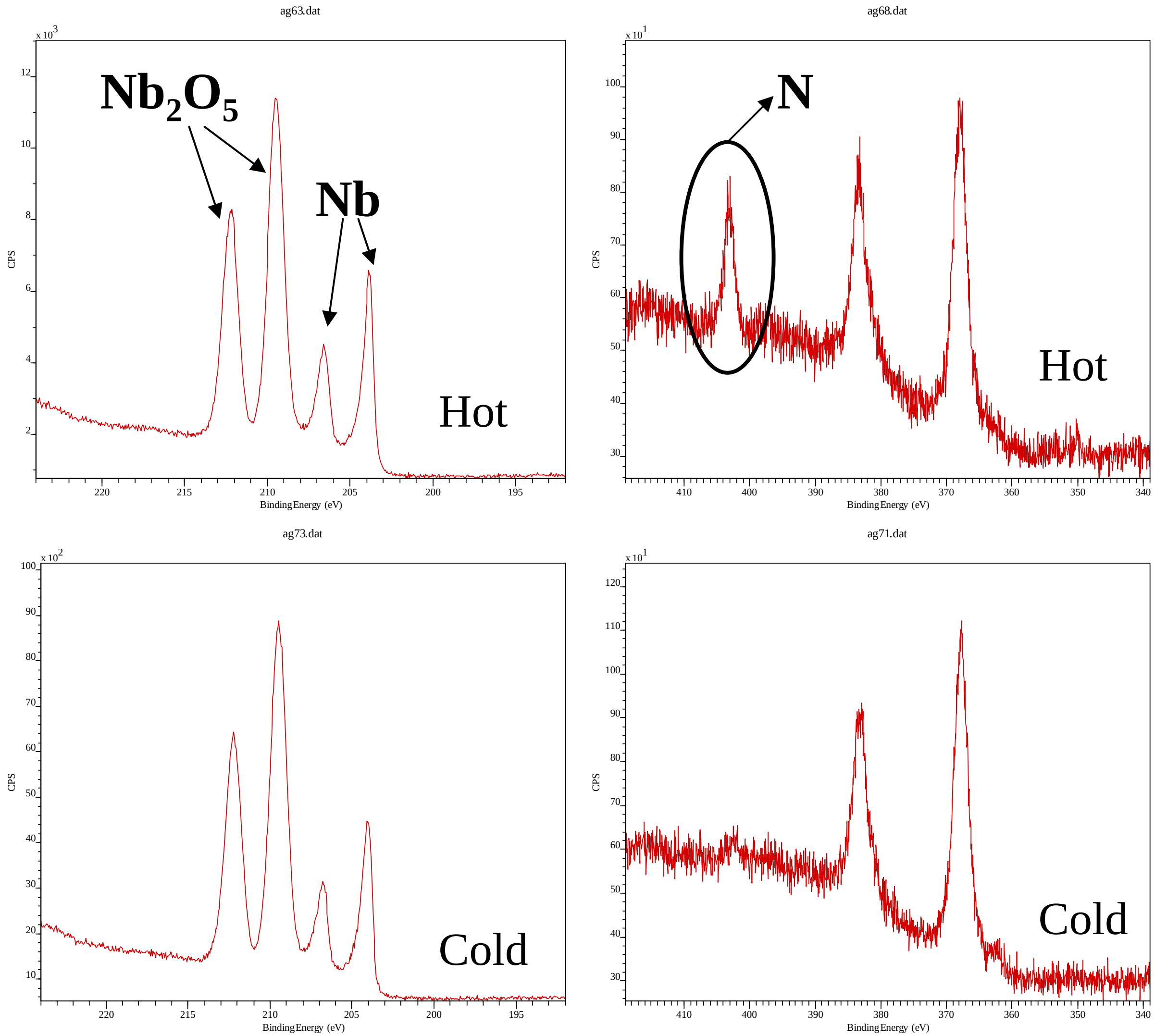




May be:

- Three hottest samples showed nitrogen
- Baking reduced nitrogen

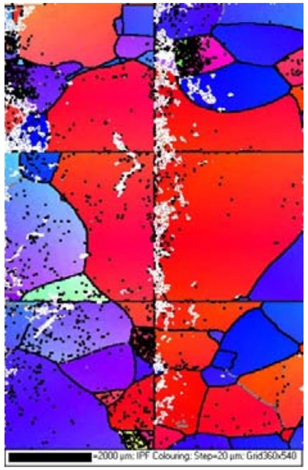
XPS: “Hot” and “Cold” Spots

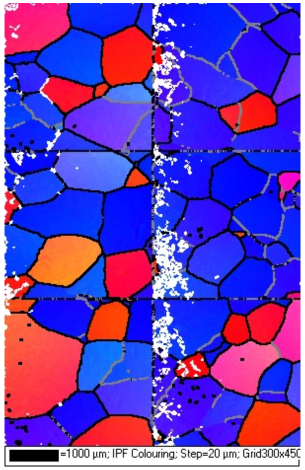


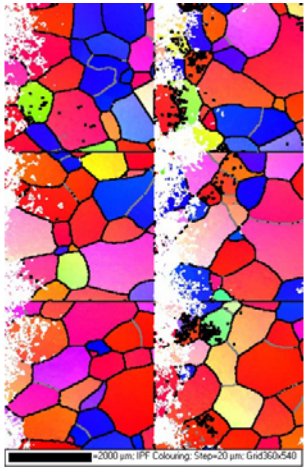
- Synchrotron X-ray source - NSLS
- hν = 2139 eV
- Information depth ~10 nm

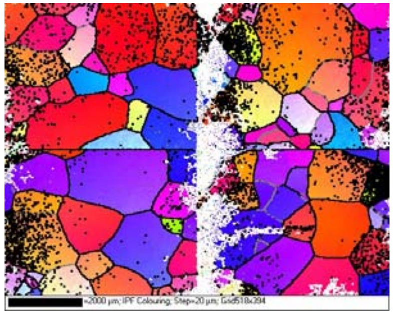
A.Romanenko (Cornell), J.Woicik (NSLS)

EBSD: Is grain orientation responsible? (Small grain BCP cavity)

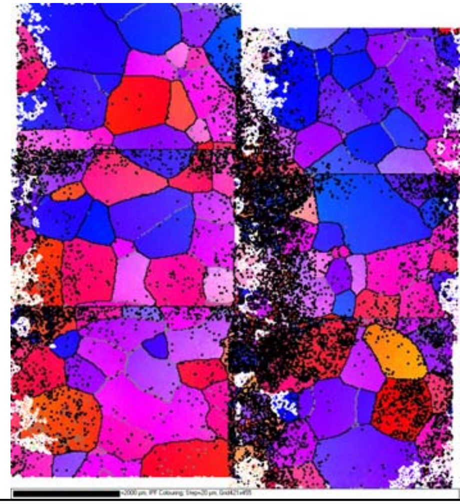


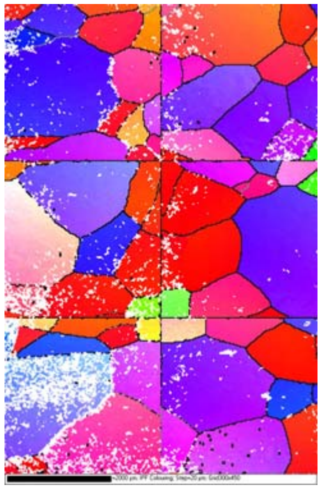
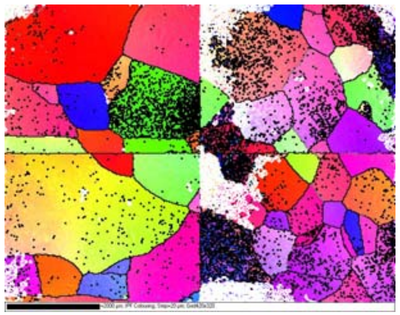
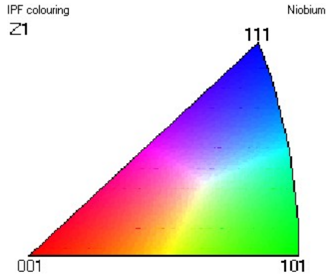




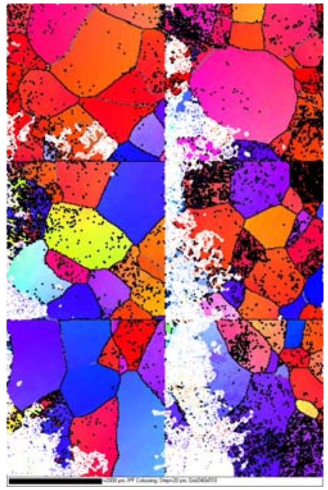
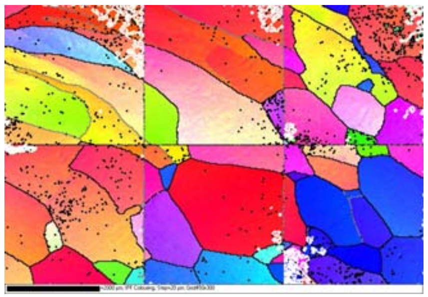
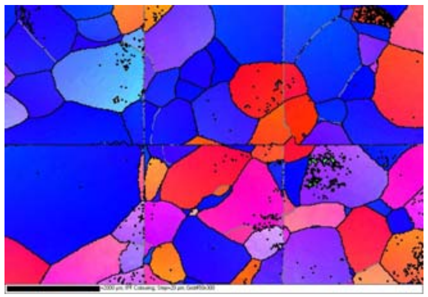


Hot

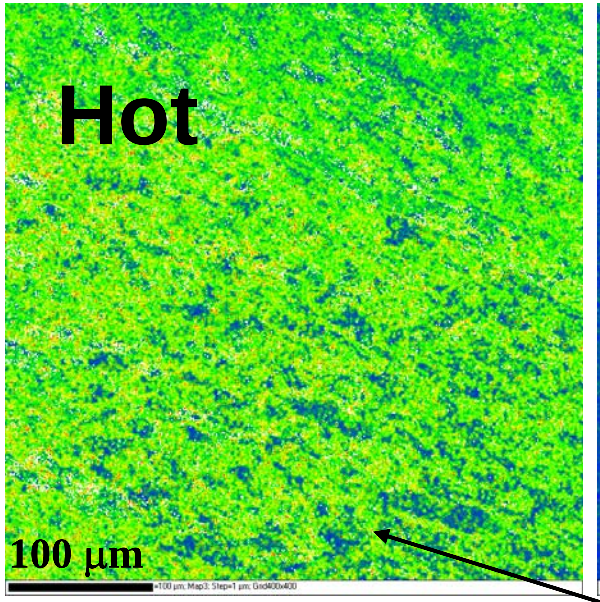


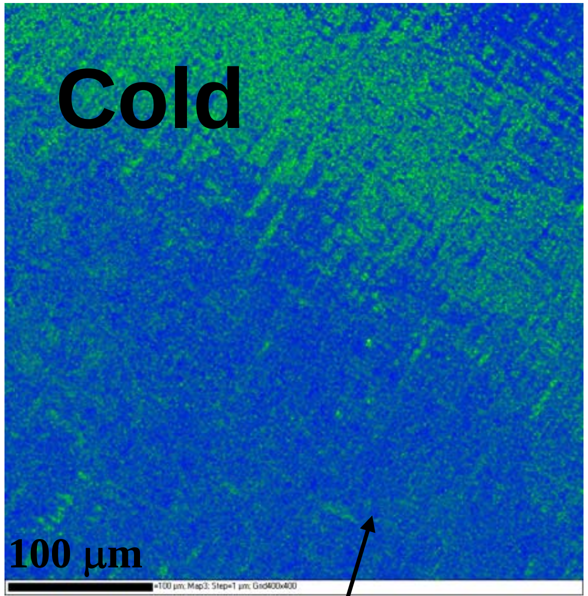




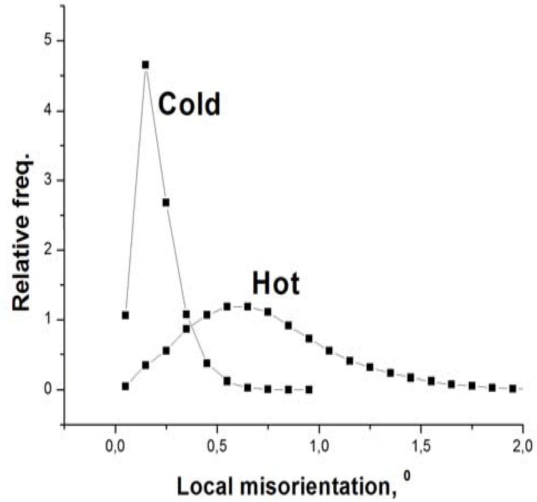
Cold

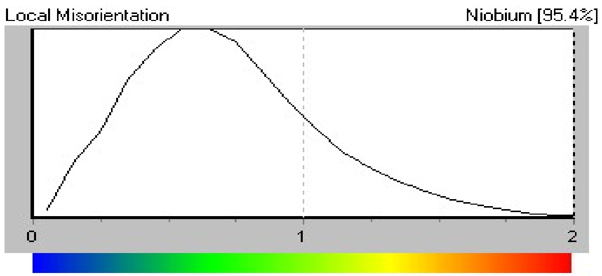




EBSD: Is it crystalline microstructure?









Significant difference!

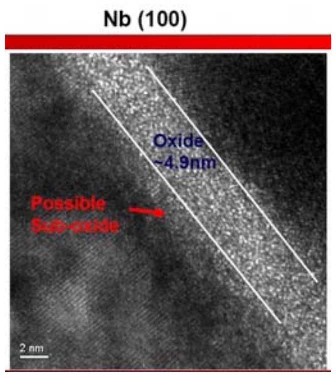
- Crystalline microstructure is different
  - Different defect (vacancies, dislocations) density

Cause of heating non-uniformity?

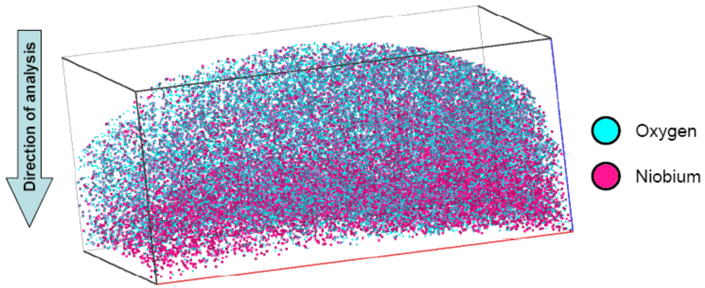
- Contaminants other than O?
  - Nitrogen found in small grain BCP cavity “hot” regions
- Crystalline defects, strain?
  - Difference observed between hot and cold regions in large grain BCP cavity

New Info Coming…

- TEM (Russell, …)



- 3DAP (Yoon, …)



- X-ray techniques (Delheusy, Antoine)
  - Diffuse diffraction, crystal truncation rod, reflectometry

Summary

- SIMS, XPS do not support O related pollution layer or its change due to baking
- Grain boundaries – no contribution observed
- Preferred crystalline orientation – not a cause
- Roughness is not playing a primary role but may be subsidiary
- Crystalline defect structure within penetration depth might play a role
  - Different in BCP and EP
  - Sensitive to mild baking
  - Preliminary - different in “hot” and “cold” spots
  - More studies needed