Fermilab

U.S. DEPARTMENT OF ENERGY

Recent Developments in Understanding the Mild Baking Effect

Alexander Romanenko

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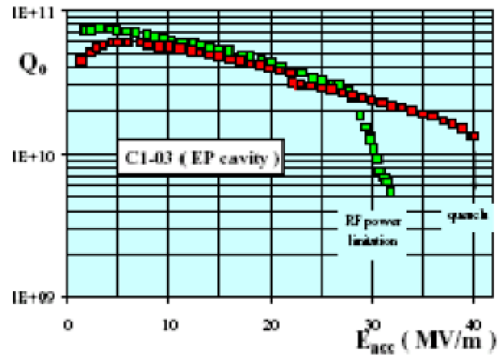
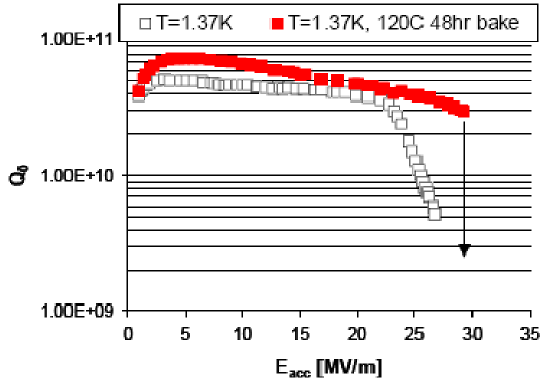
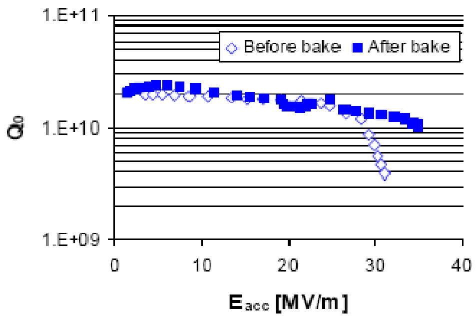
Mild baking – main focus

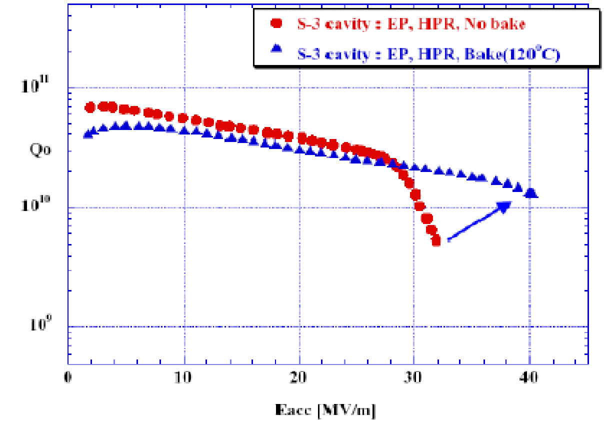
- UHV annealing at 100-120C, up to 48 hours depending on the grain size
  - Eliminates HFQS in all EP cavities - 100% efficient
  - Improves HFQS in fine grain BCP ones (not consistently)
- Most of the models for the effect considered were oxygen-related (interstitial or oxide)





G. Ciovati-*J. Appl. Phys.* 96, p. 1591 (2004)





Empirically found – mechanism not clear!

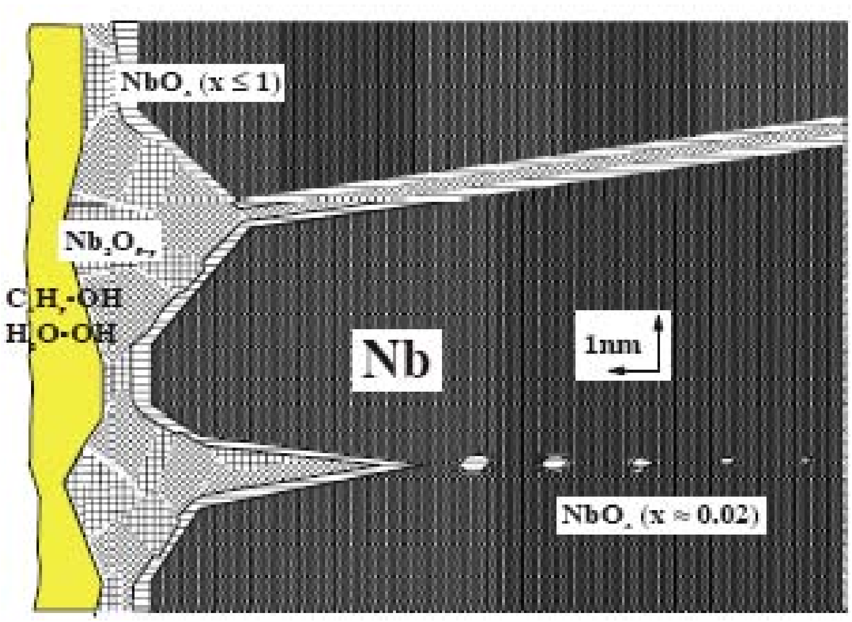
Why is it important?

- Single known thermal treatment having a drastic and predictable effect on HIGH field performance of superconducting Nb
- Mechanism is unclear
  - If known could shed light at what is causing all high field phenomena
    - High field Q-drop
    - High field quench
- Personal point of view – one of the most important SRF physics problems at the moment

Mild baking – key facts

- 100% efficient on EP cavities, and large/single grain BCP cavities
- Not consistent effect on fine grain BCP cavities
- Duration of baking required strongly depends on the grain size -> larger grains = shorter baking
- Depth of surface layer modified by baking ~20 nm (Eremeev, Ciovati)
- Baking benefit is not destroyed by
  - HF rinsing = removing the oxide layer and reoxidizing again in water
  - High pressure rinsing
  - Air exposure of baked cavities

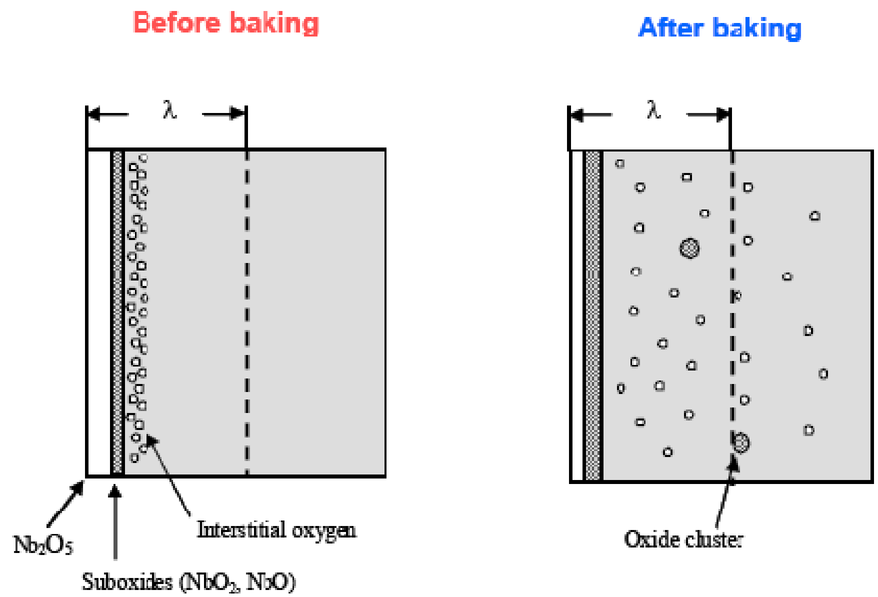
Previous models of Nb surface and its change with mild baking



Courtesy of J. Halbritter

Oxygen pollution model

G. Ciovati et al



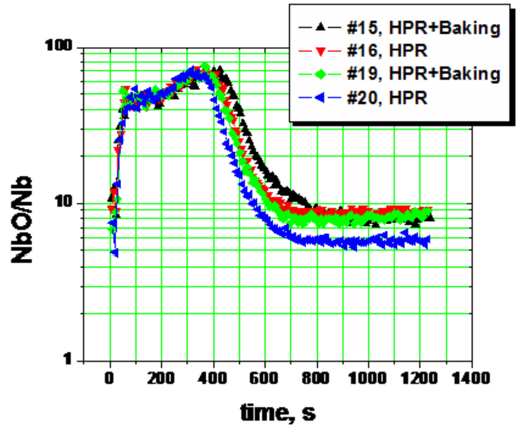
Change in properties attributed to diffusion of interstitial impurities.
O was considered the main suspect.

Interstitial oxygen role

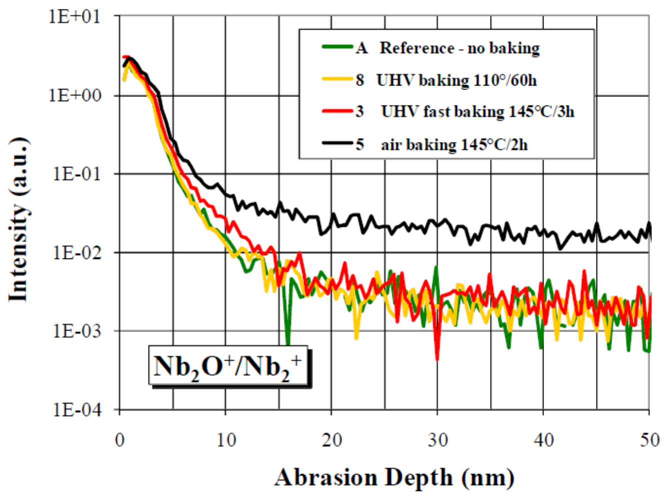
- Oxygen is known to have a strong effect on superconducting properties of Nb
  - Critical fields depend on interstitial oxygen content
- Oxygen is one of the primary interstitial impurities
- Diffusion length of oxygen in niobium corresponds well to the ~20 nm depth of baking modified layer
  - But there is an implicit assumption of anodizing/oxypolishing just consuming some Nb and not changing anything else

Oxygen diffusion not observed

- Oxygen diffusion length at 100-120C – consistent with baking modified layer (~20 nm)
- Problems
  - No significant oxygen diffusion observed – no O gradient to diffuse away? all goes back after air exposure?
  - Cavity experiments
    - More O (Ciovati) in the surface layer – no deterioration
    - 300-400C in situ baking (Eremeev)



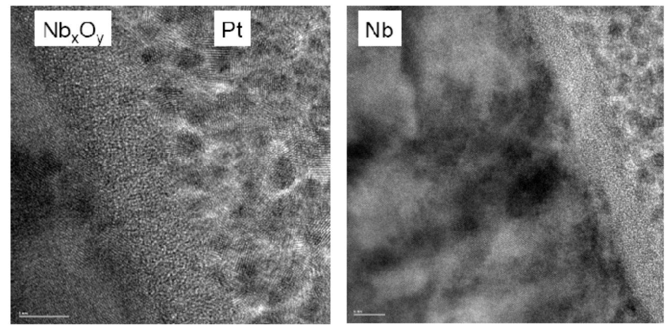
G. Eremeev et al. (Cornell)

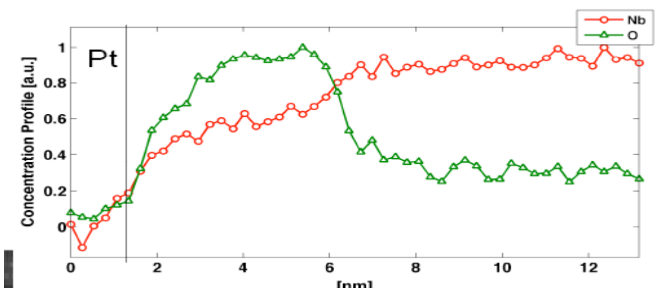


B. Visentin et al. (CEA/Saclay)

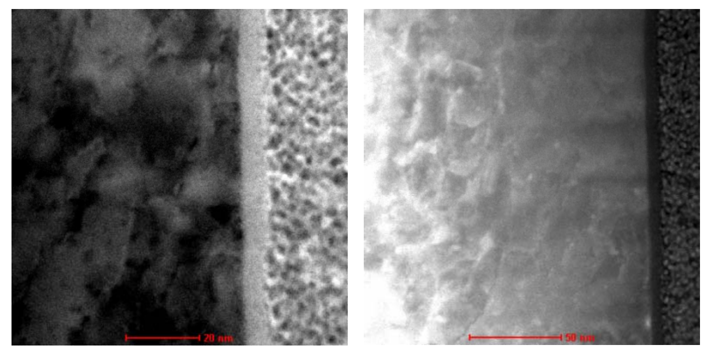
A. Romanenko, J. Mundy, P. Ercius, J. Grazul – details elsewhere

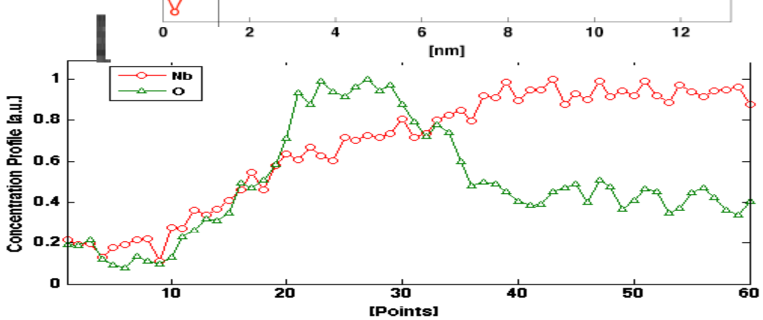
- FIB prepared samples from “hot” spot before and after 120C baking
- EELS elemental analysis with atomic scale resolution
- **No oxygen-enriched layer**
- **No oxygen diffusion**
- **No oxide modification**
- **No cracks or suboxide clusters – very nice and uniform oxide**





Not baked





Baked

Oxide modification models

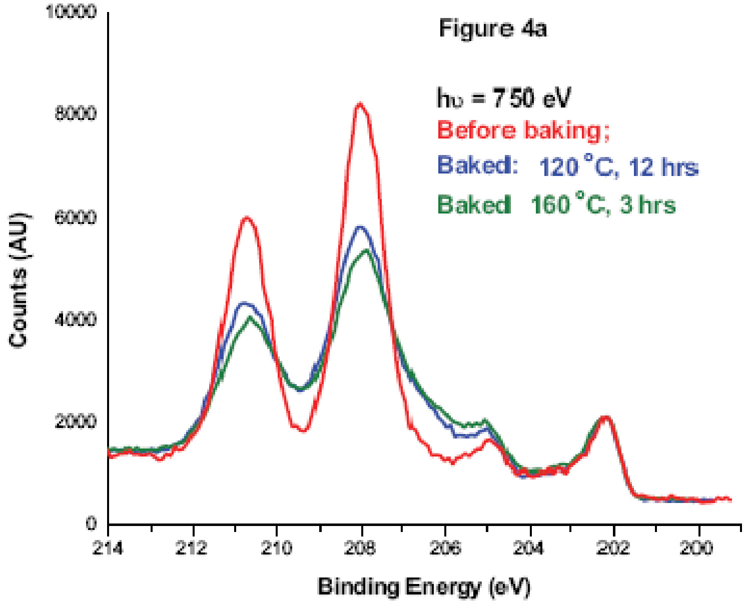
- Few models for the HFQS, which require the oxide modification via:
  - Decrease in density of magnetic impurities (non-stoichiometric Nb2O5) – T. Prolier et al (ANL)
  - Change from “wet” oxide with many electron localized states to “dry” oxide – ITE model by J. Halbritter

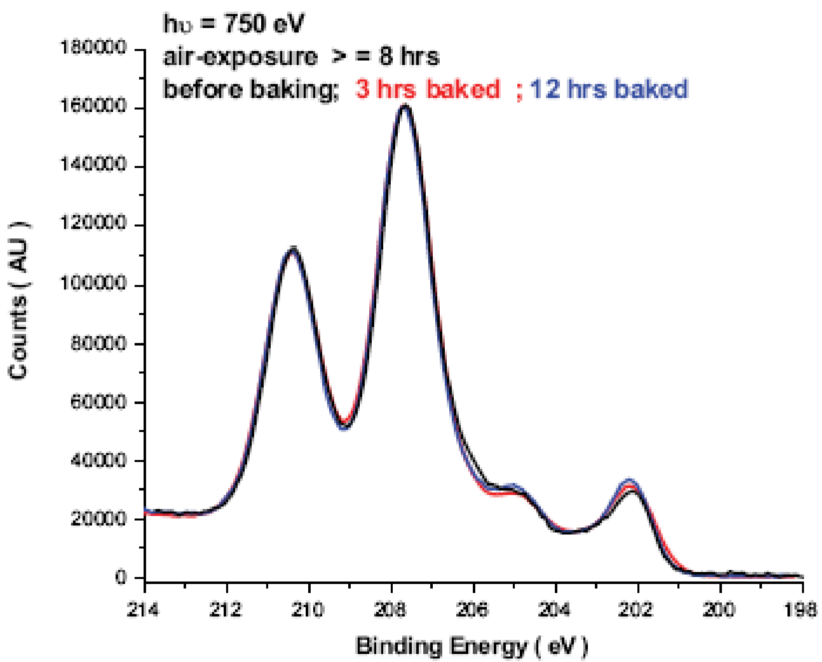
- Problems (evidence against)
  - Cavities - regrowing the oxide does NOT bring the high field Q-slope back
    - HF rinsing
  - Oxide structure after baking + air exposure = *exactly same* as before baking
    - XPS data (H. Tian et al/JLab)

H. Tian (JLab) et al, Proceedings of SRF'07 Workshop

Baked in situ, **no air exposure**

+ **air exposure** = ALL back to original

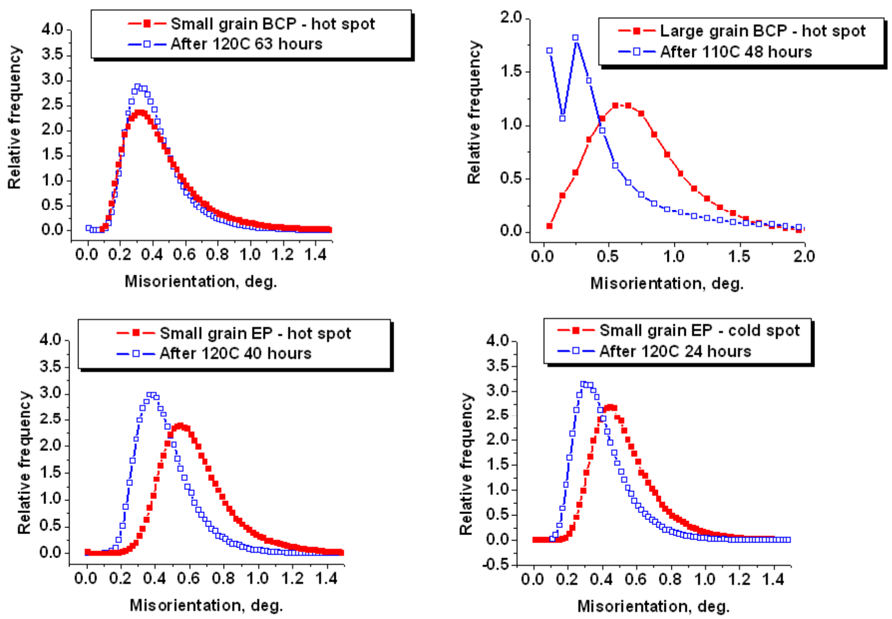




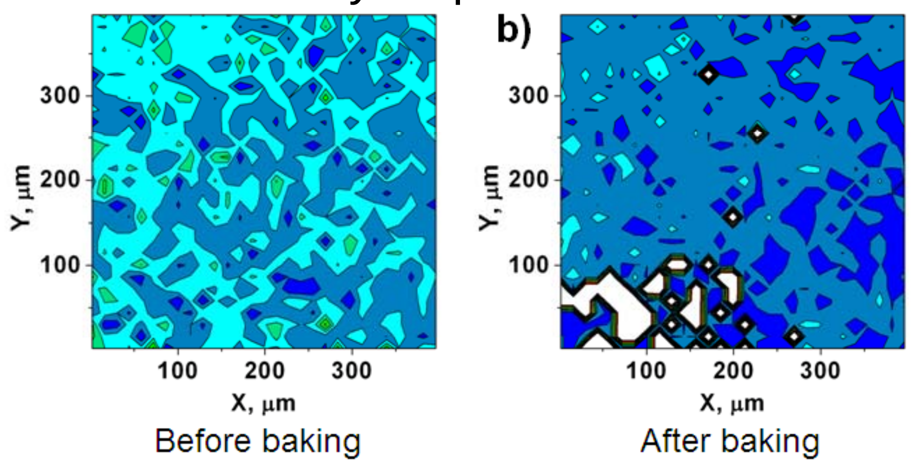
Current status

- Oxygen-related models for mild baking contradict surface data
- Model of Nb surface should be updated
  - No crack corrosion
  - Oxide clusters not observed
  - etc
- New mechanisms for mild baking under consideration (more later)
  - Change in surface crystalline defect structure?
    - Dislocations, vacancies
  - Hydrogen?
- Key - we need to observe something to change with surface analytical tools during 100-120C annealing

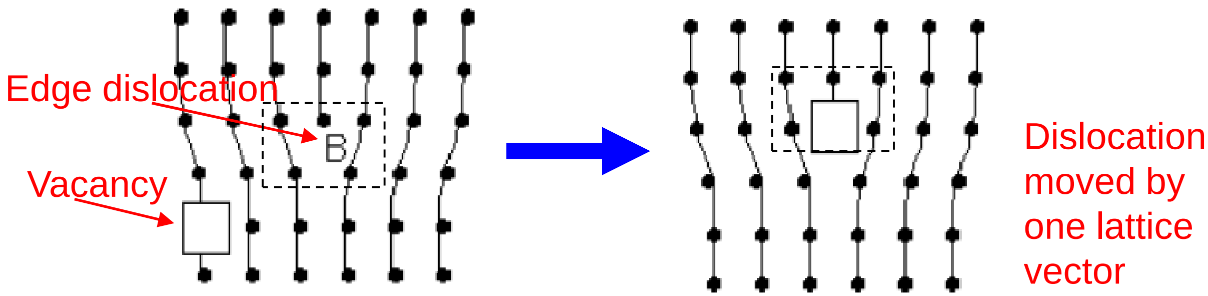
Change in dislocation density (Cornell)



Average edge dislocation density maps

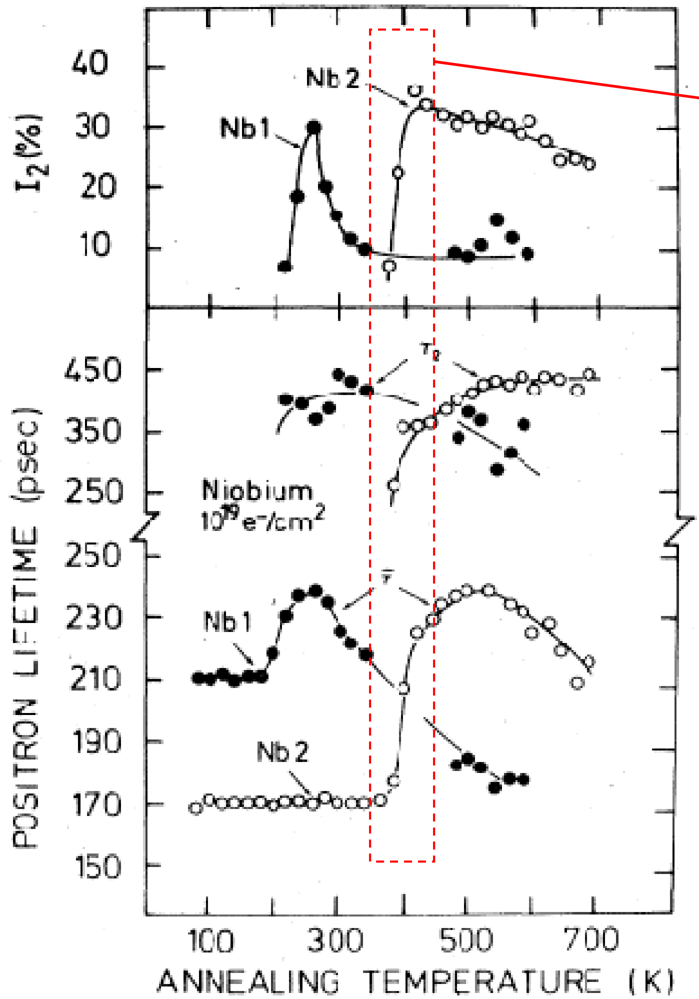


Apparent change in average dislocation density caused by mild baking in HFQS-limited cavity samples



Dislocation climb?

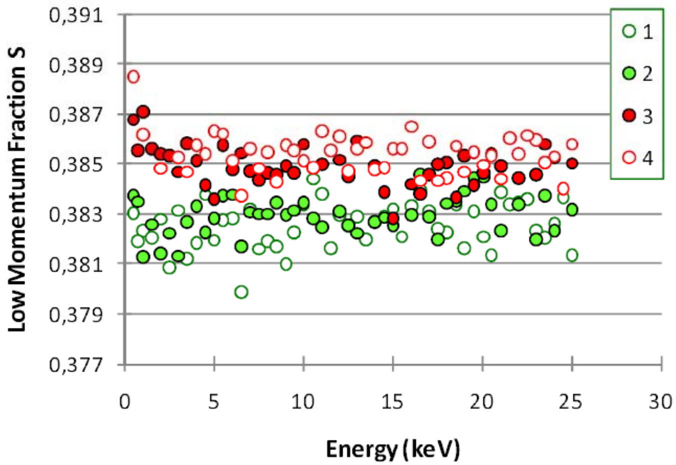
Possible key mechanism

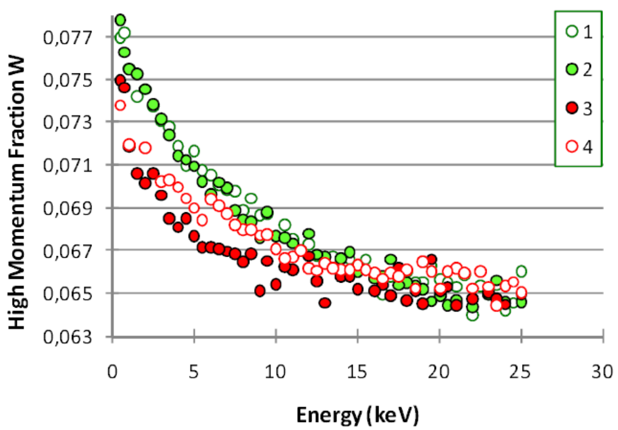


- Vac-H complexes in niobium containing some H (Nb2 in plots) dissociate at ~380K (107C) – compare to baking temperatures
- Mobile vacancies => dislocation climb becomes possible

[P. Hautojarvi et al., Phys. Rev. B., Vol.35, Num.7, 1985]

Vacancies – positron annihilation data





- Increase in S-parameter = increase in lattice open volume (positron trapping sites) – higher vacancy concentration

- Increase in number of free vacancies after baking can be interpreted as a result of Vac-H dissociation and
  - Release of H => more free vacancies
  - Mobile vacancies provide mechanism for dislocation climb => dislocation annihilation => trace of vacancies left => more vacancies

B. Visentin et al., Proceedings of SRF'09

Hydrogen data

- 800C baking without any chemical treatment afterwards
  - Improvement in the high field Q-slope
- G. Ciovati's talk earlier

Conclusions

- Oxygen diffusion/oxide modification probably not relevant to mild baking effect
- New mechanisms of 100-120C baking are being investigated (dislocations, vacancies, H)
  - Which is at work? Further investigations needed
- High T baking is not under much of investigation – more studies needed
- Toolset was tailored for O studies
  - Should change it: TEM/EELS etc – look at microcrystalline structure and H!