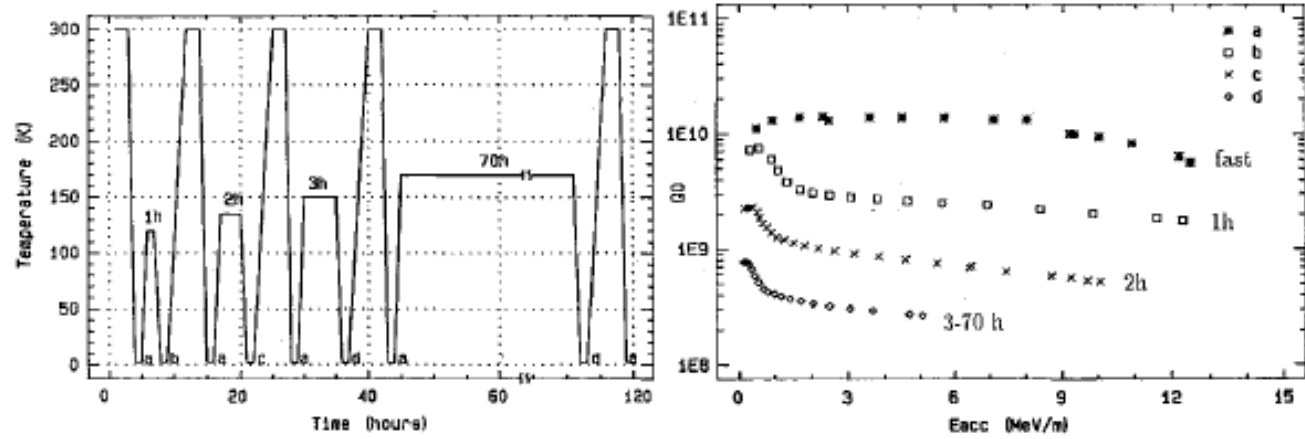


Direct Observation of Hydrides in Cavity-Grade Niobium

A. Romanenko, F. Barkov
Fermilab



- S. Isagawa, J. Appl. Phys. 51 (1980)
- B. Bonin, R. Roth, SRF'91
- R. Roth et al, SRF'91
- J. Halbritter et al, SRF'93
- ...

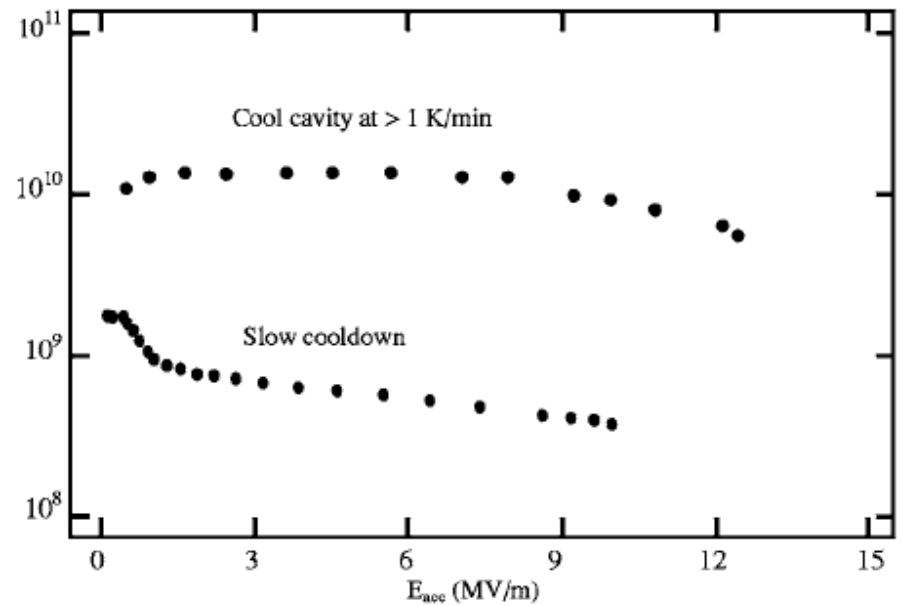
What exactly are these hydrides?

- Phase, islands, layer, GB precipitation?
- Size/density as a function of treatments and cooldown



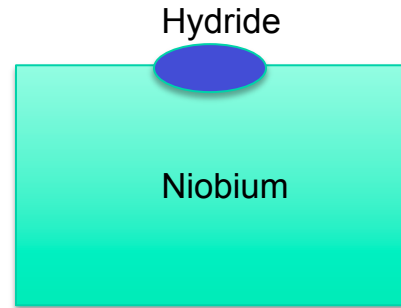
Quantitative understanding

Aune et al, LINAC'90

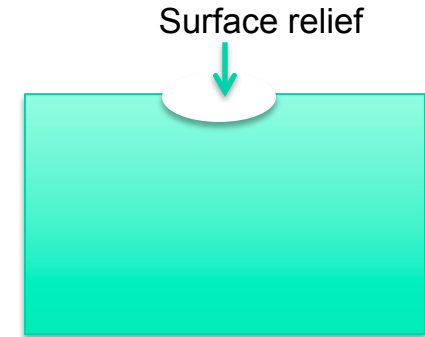


- The easiest technique – optical microscopy after sample hold in the temperature range of interest (77-200 K)
- Looking for the surface relief left behind by hydrides
 - Laser confocal imaging frequently helpful
- Sample has to be very smooth to see the relief of <100 nm deep
 - Requires special sample preparation techniques – i.e. vibromet polishing

$T < \sim 150\text{K}$



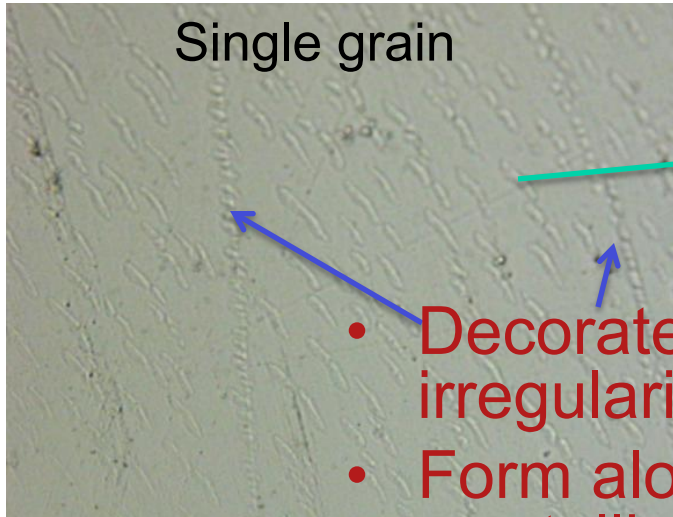
Room T



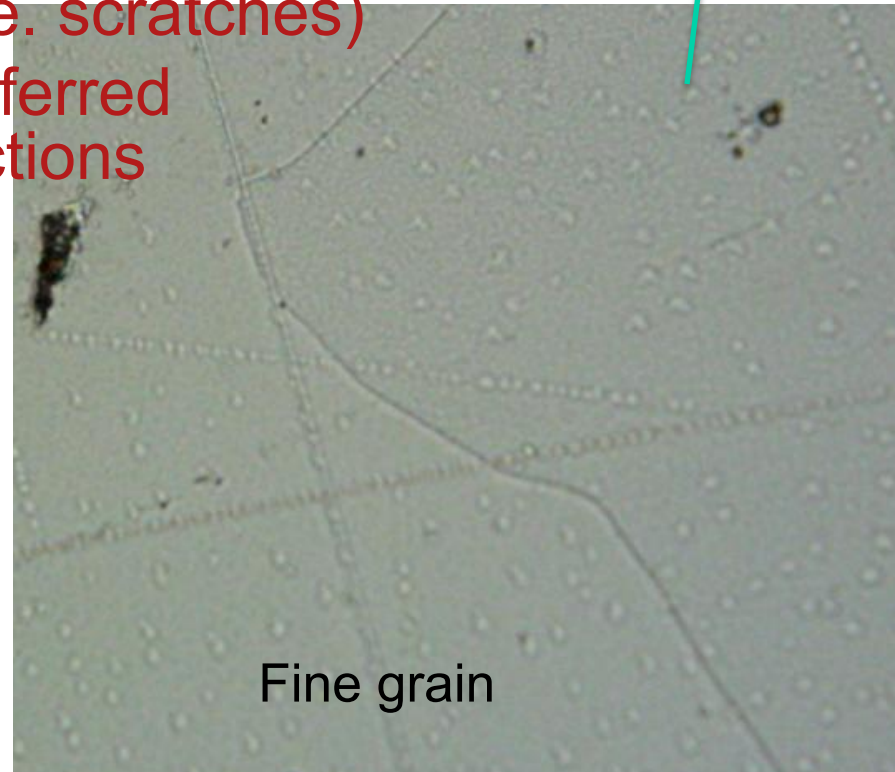
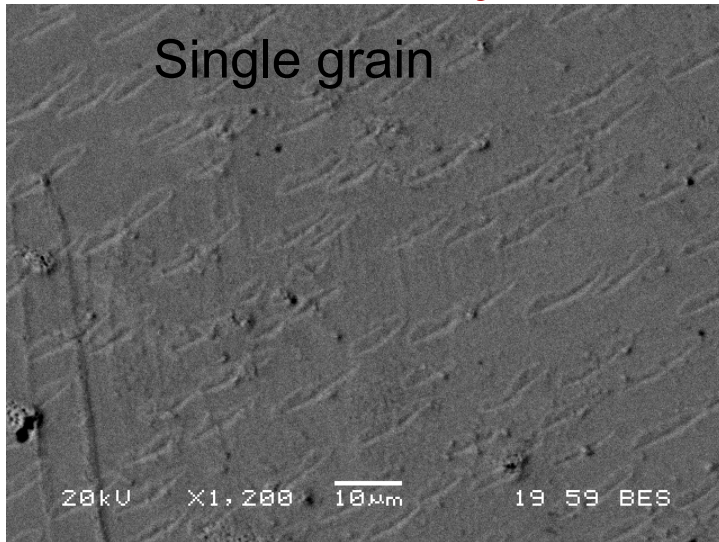
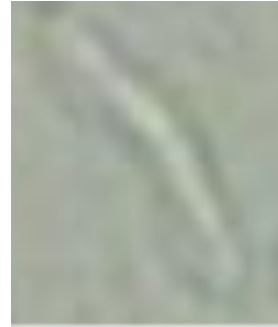
Example – single grain BCP/mechanical polish

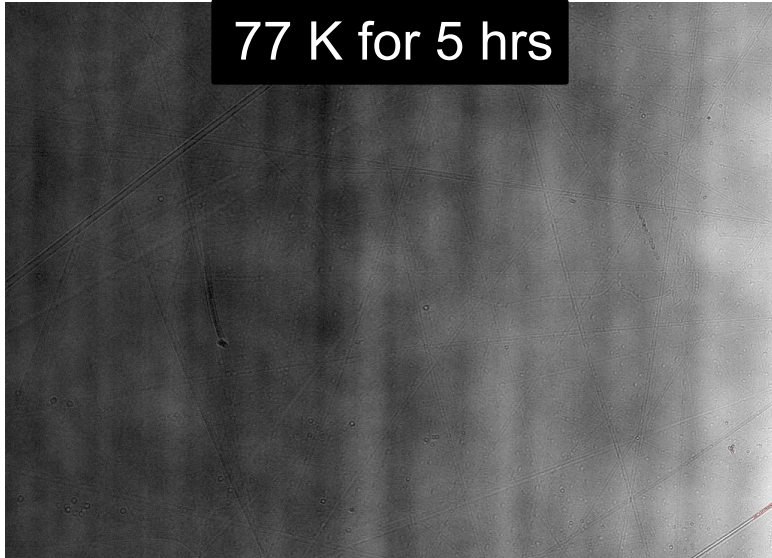


Lens-like hydrides produced surface relief after 77K hold as seen in optical/laser confocal microscope

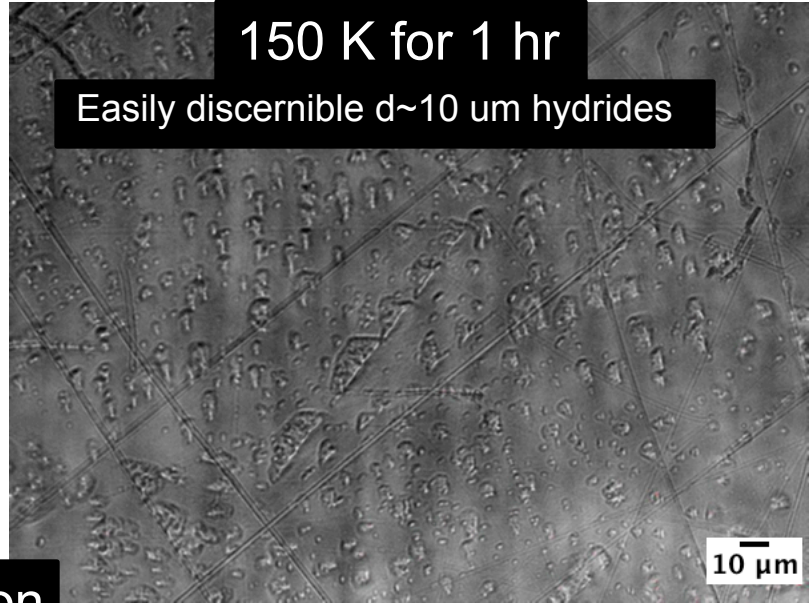


- Decorate surface irregularities (i.e. scratches)
- Form along preferred crystalline directions





77 K for 5 hrs

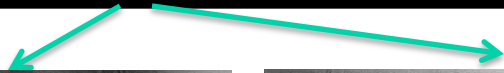


150 K for 1 hr

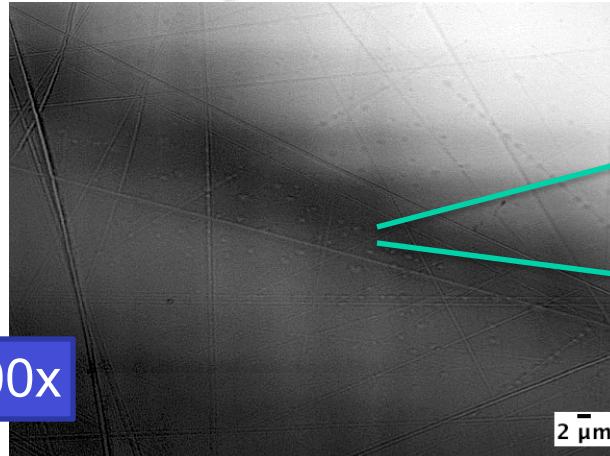
Easily discernible d~10 um hydrides

10 μm

Can only be found at highest magnification



3000x

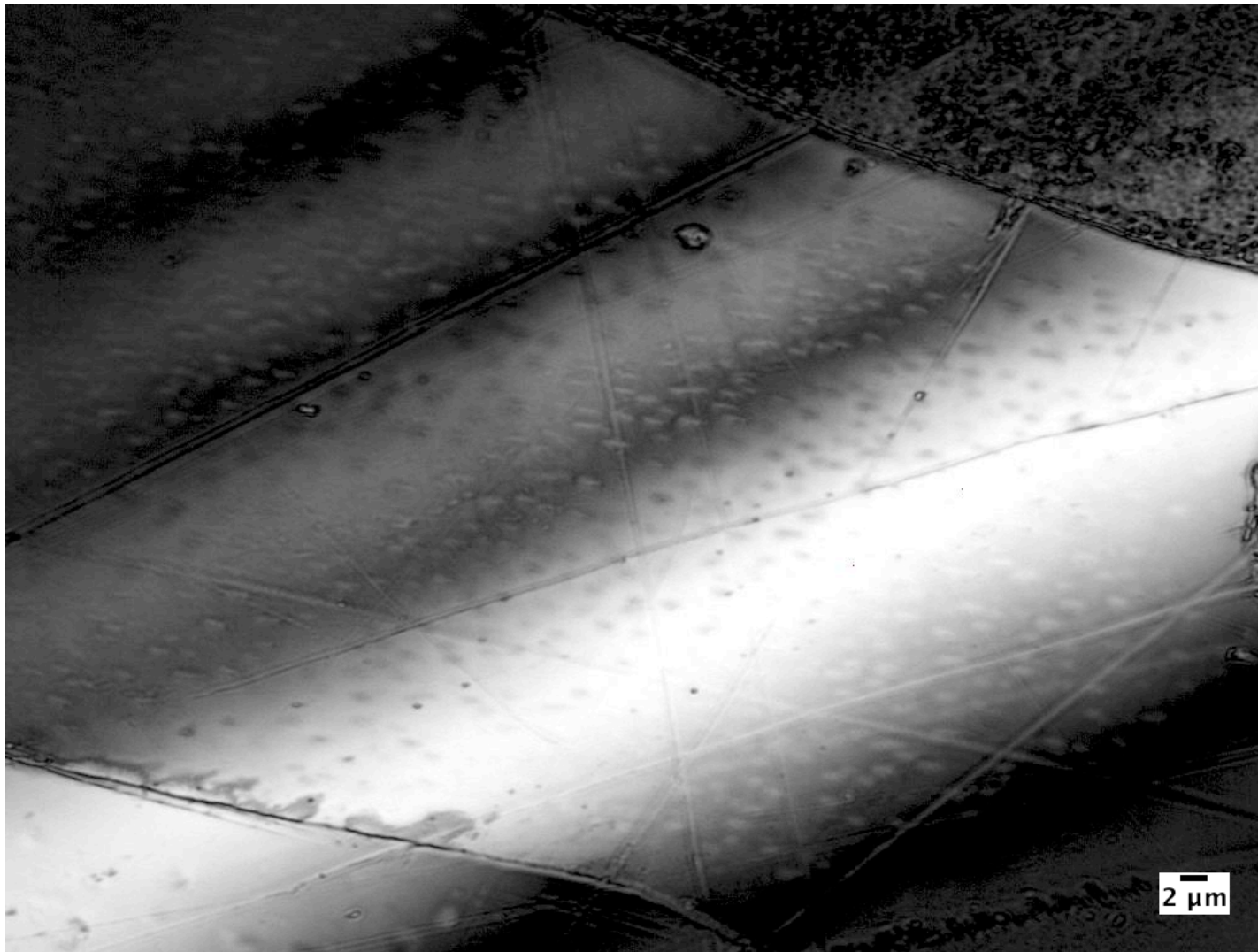


2 μm

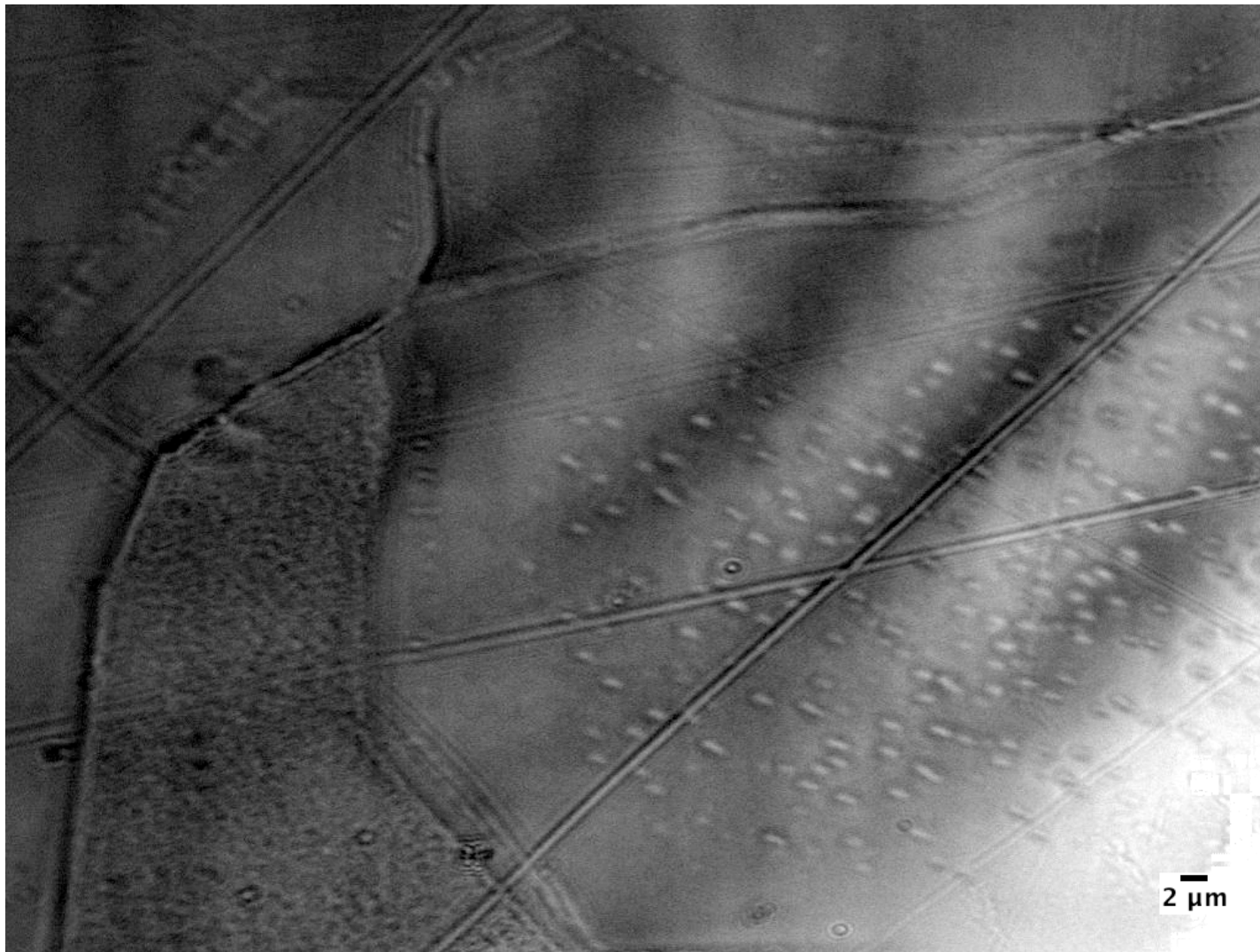


d~1 um hydrides

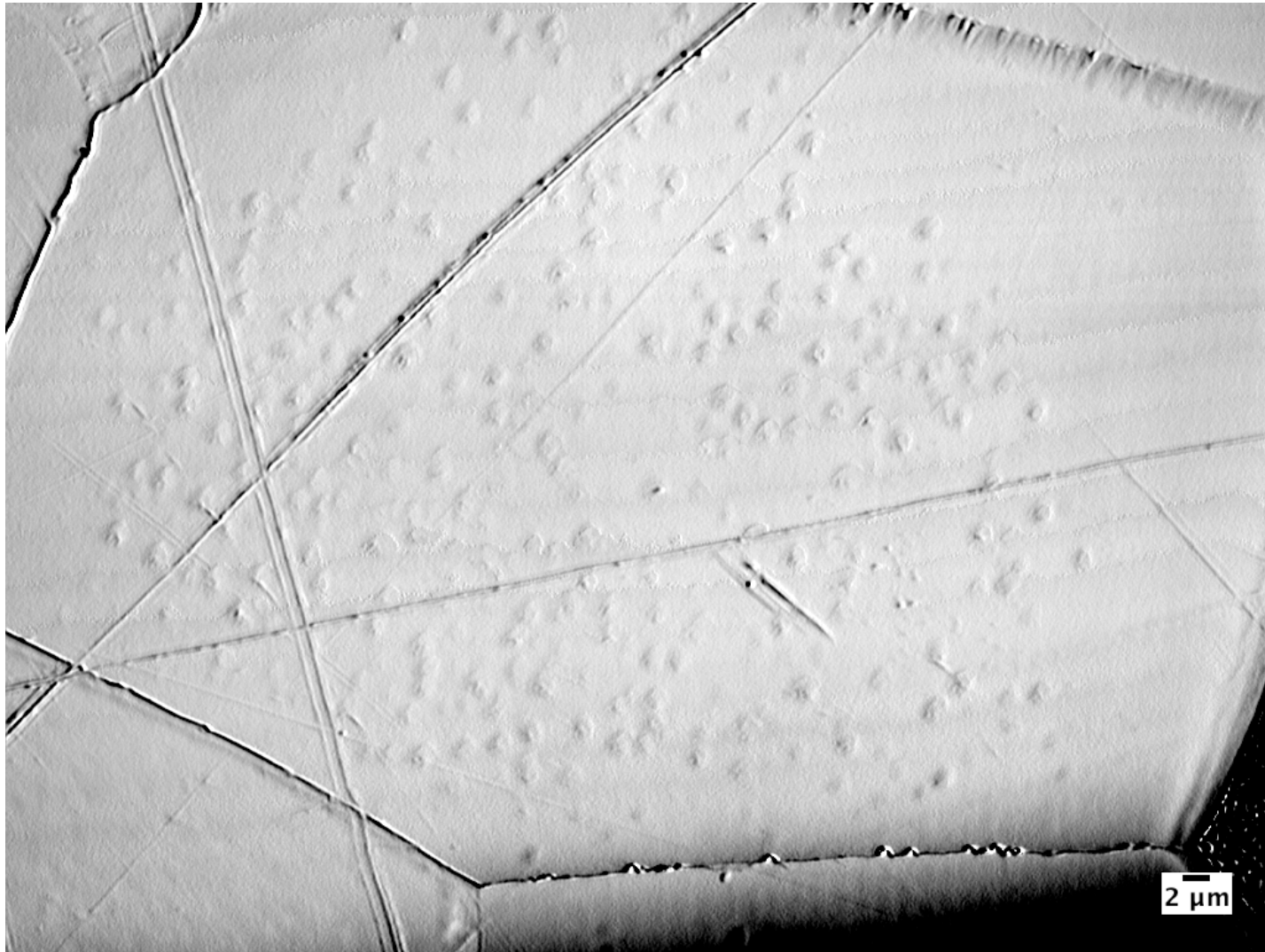
Hold at 150 K for 1 hour



Hold at 150 K for 2 hours



Hold at 150 K for 4 hours



Treatment	77-150K hold outcome
Mechanical polish (MP)	Large hydrides
+800C 3 hrs	No hydrides seen
+BCP 5 min	No hydrides seen
+120C 48 hrs	No hydrides seen
MP + 800C 3hrs + EP 40 min	Small hydrides after 150K

- Hydrides leading to Q-disease are lens-like and star-like precipitates -> “Islands” to answer the question posed in [*R. W. Roth, SRF’93, “Islands or Films? - New Aspects of the NbHx-puzzle”*]
 - 1-10 um in diameter
 - 50-100 nm thick (based on the depth of surface relief pits)
- Diffusion/concentration controlled formation
- We can directly see how many/where/what size hydrides form
 - Limited by the observable surface relief
 - Is it why we don’t see any after 800C and no chemistry?
- Will a particular combination of surface/heat treatment produce a Q-disease?
 - We can check on small samples
 - Now in real time! – cryostage for LCSM installed
- Model for the HFQS -> please come to the TFSRF’12 talk on Thursday