Niobium Hydride Studies using Cryo-AFM

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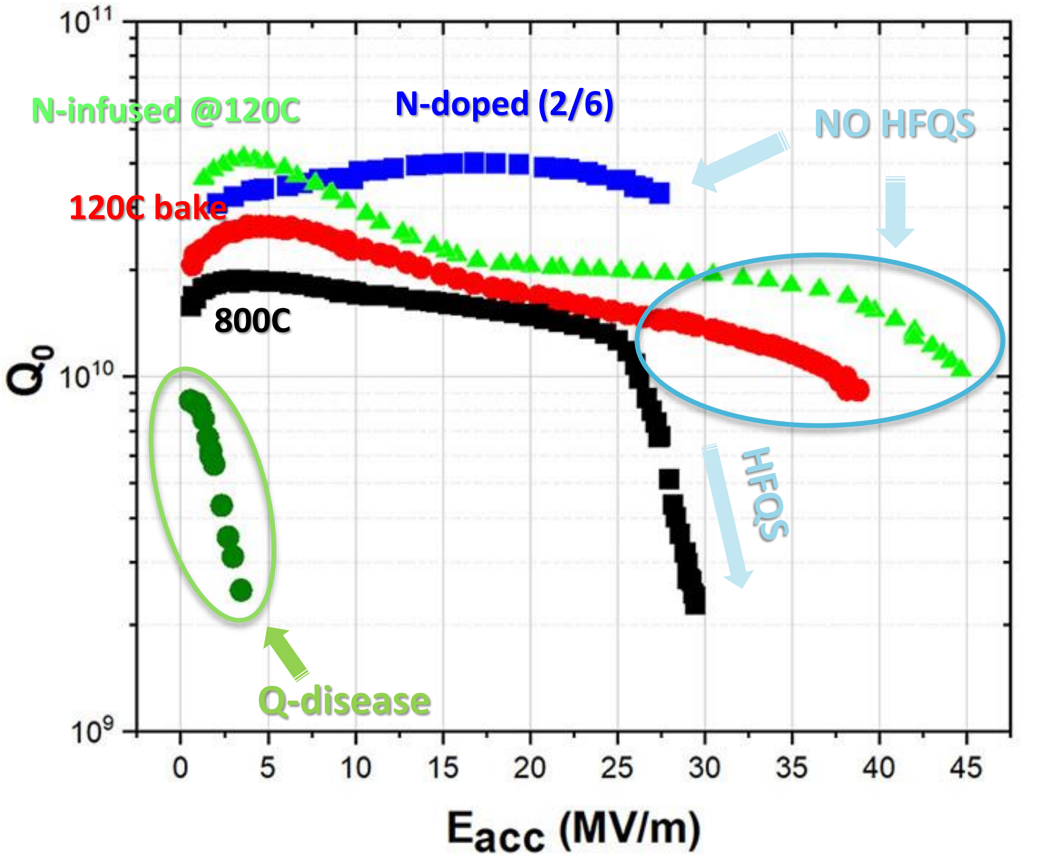
Outline

- Performance degradation of SRF Nb cavities (HFQS), which can be correlated to niobium hydride precipitation.
- Lessons from the previous Nb-H studies.
- First direct identification of nm-size NbH precipitates.
- Morphological and statistical comparison of NbH formation on the *state-of-art* SRF Nb cavities.
- Promising model of NbH segregation mechanism.
- Summary.

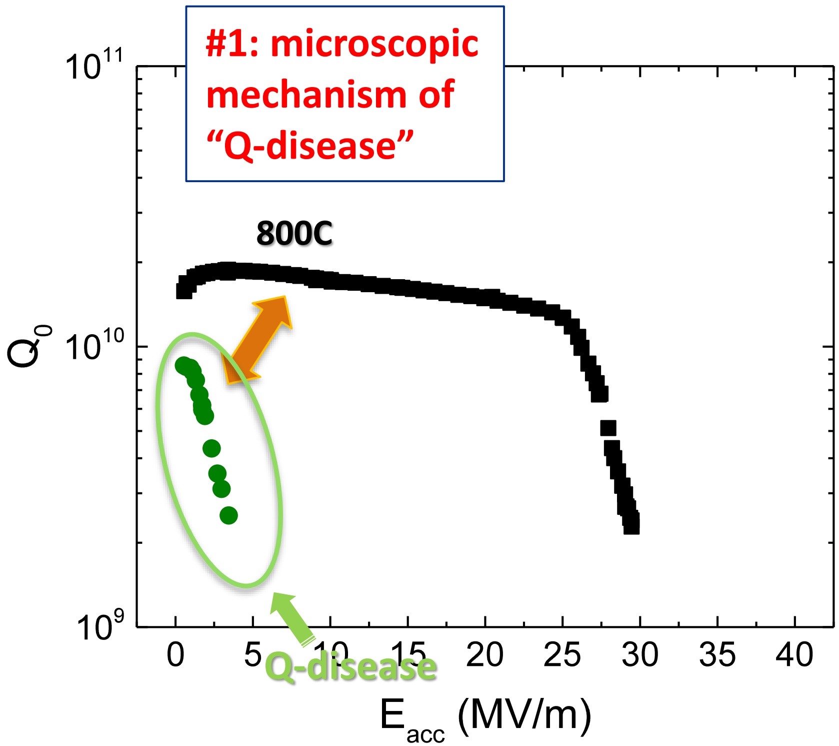
Hydride formation related SRF Nb cavity performance degradation (HFQS)



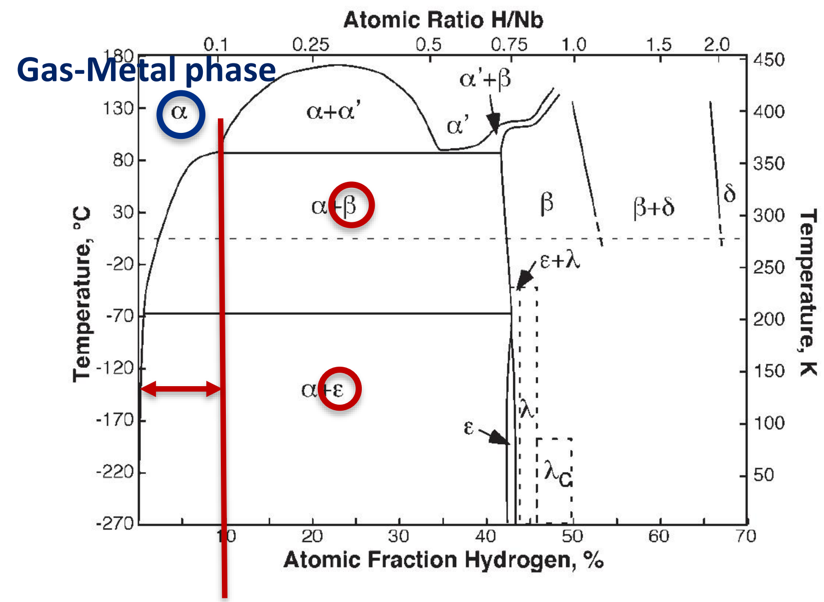
A. Grassellino et al., Supercond. Sci. Technol. 26 102001, (2013) & Supercond. Sci. Technol 30, 094004 (2017)

- ✓ **800°C HT reset "H" in SRF Nb bulk, but 800°C HT'ed cavity still shows HFQS**
- ✓ **120°C bake is an empirical remedy for HFQS**
- ✓ **Nitrogen plays a pivotal role in "NbH" formation**
- ✓ **Further require understanding of NbH formation for achieving ultimate SRF performance**

Clear understanding of excess “H” for onset of Q-disease



A. Romaeneko, TTC Topical Meeting, 2017

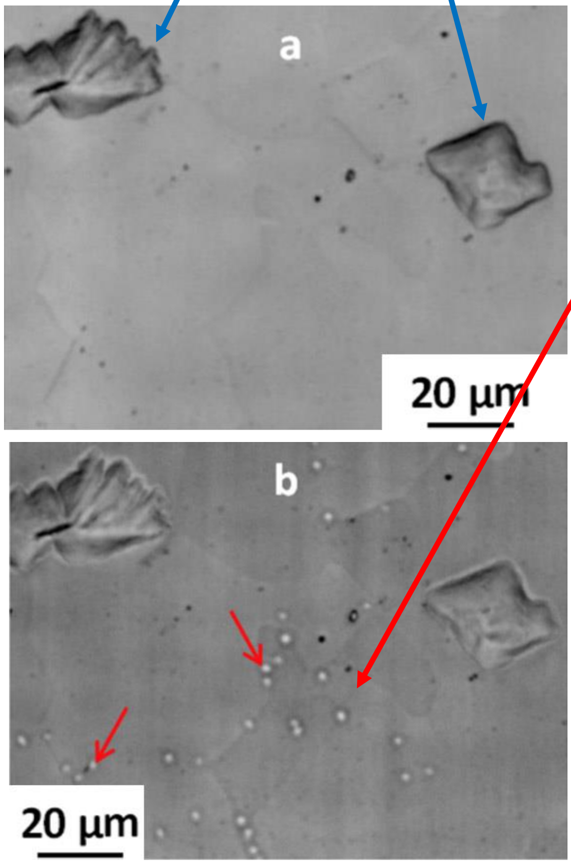


Manchester F D and Pitre J M 2000 *Phase Diagrams of Binary Hydrogen Alloys* ed F D Manchester (Materials Park, OH: ASM International) pp 115–37

- Q-disease was proposed in the past to be caused by excess hydrogen, which forms non-superconducting niobium hydrides (β, ε-phases) upon cooldown
  - Remedy found 600–800°C vacuum anneal to degas hydrogen

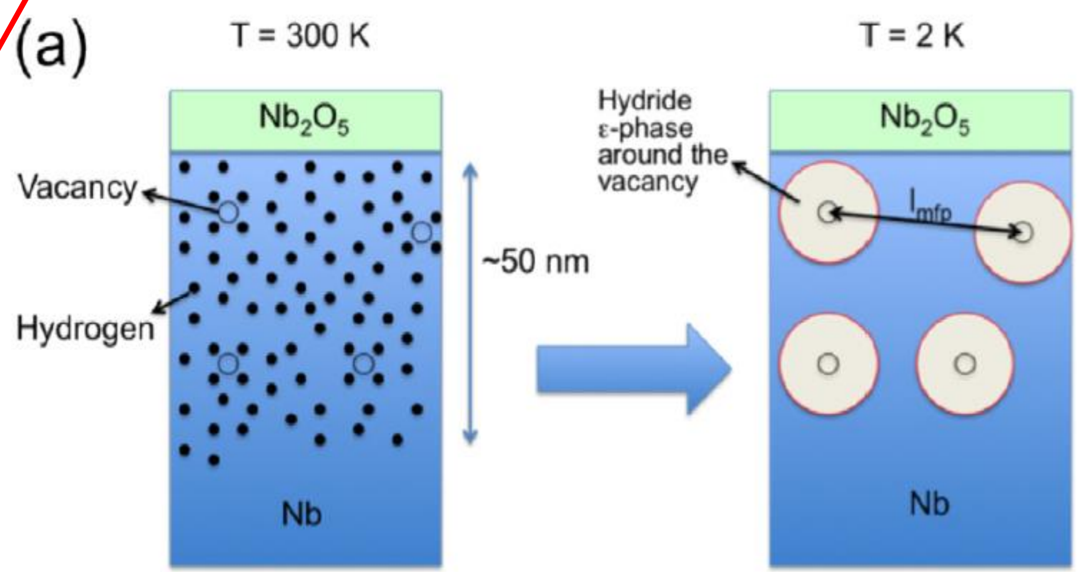
Different NbH morphology between Q-disease and HFQS

μm-size NbH precipitates due to excess H, observed by Cryo-Laser Optical Microscope



F. Barkov et al., J. Appl. Phys. 114, 164904, (2013)

Furthermore, nm-scale NbH phases also segregates on SRF Nb surface



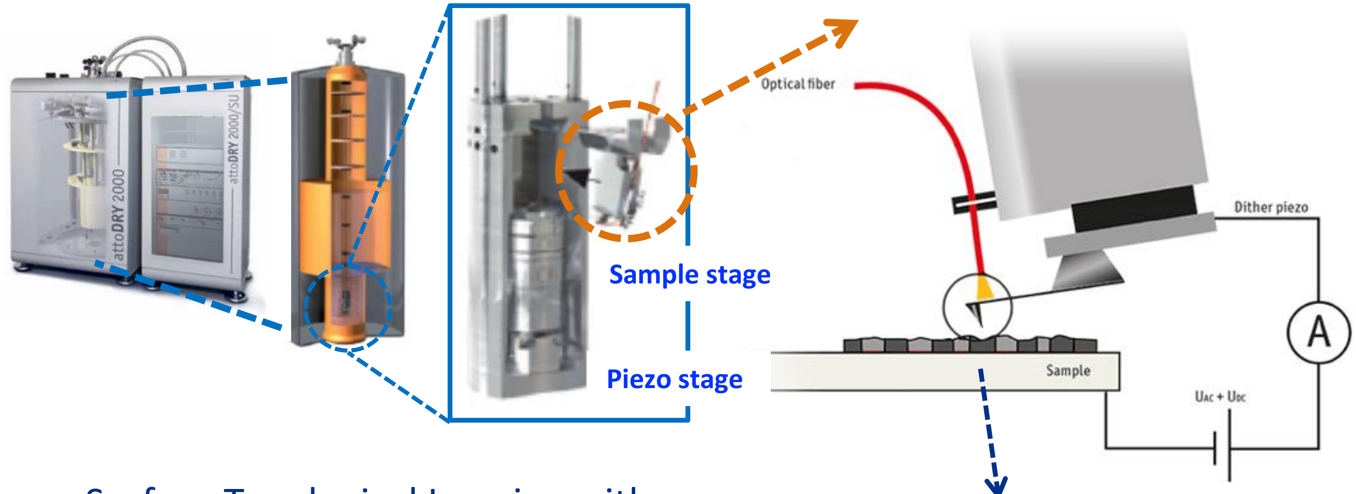
Strong possible way of influence of pretty small Nb-H proximity effect on HFQS

A. Romanenko et al, Supercond. Sci. Technol. 26, 035003, (2013) and Appl. Phys. Lett. 102, 232601 (2013)

Cryogenic AFM/MFM (attocube) + 9T PPMS System (QD Design) @SRF Materials Science Lab at APS-TD, FNAL

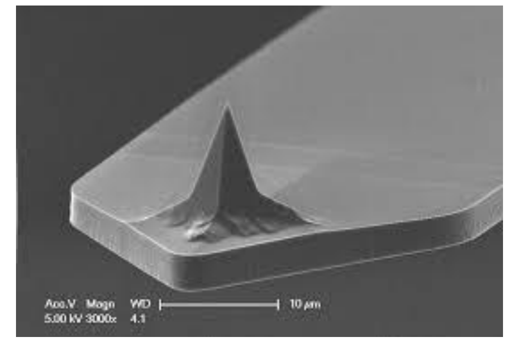
Cryogenic System with 9 Tesla Magnet

AFM Head Part

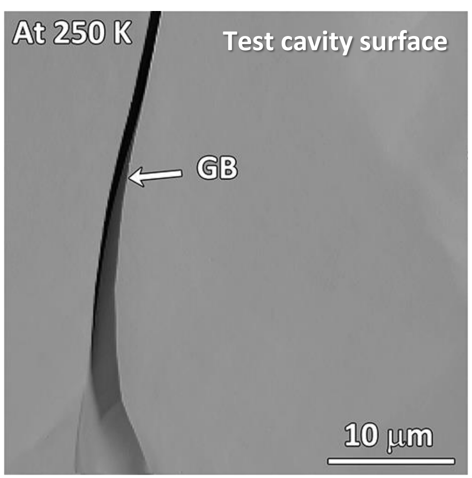


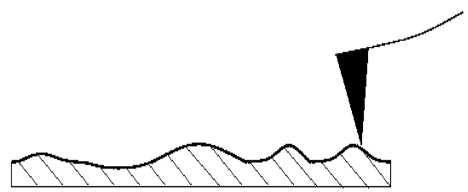
AFM: Atomic Force Microscope

AFM tip

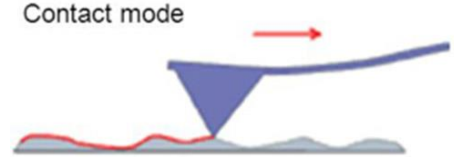


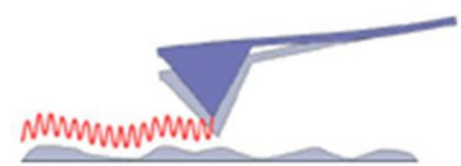
Surface Topological Imaging with nm-scale resolution in x, y, z





AFM Operation Mode

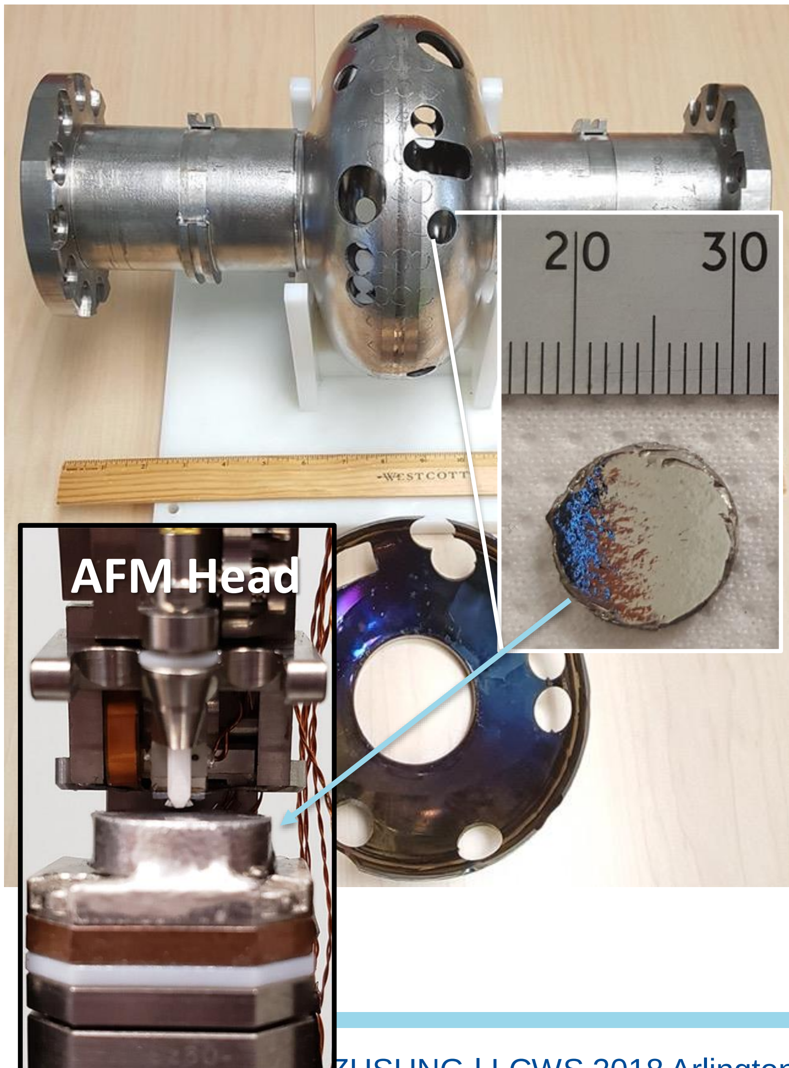




Fermilab

Samples for Nb hydride study using cryogenic-AFM

Tested cavity cut-out sample

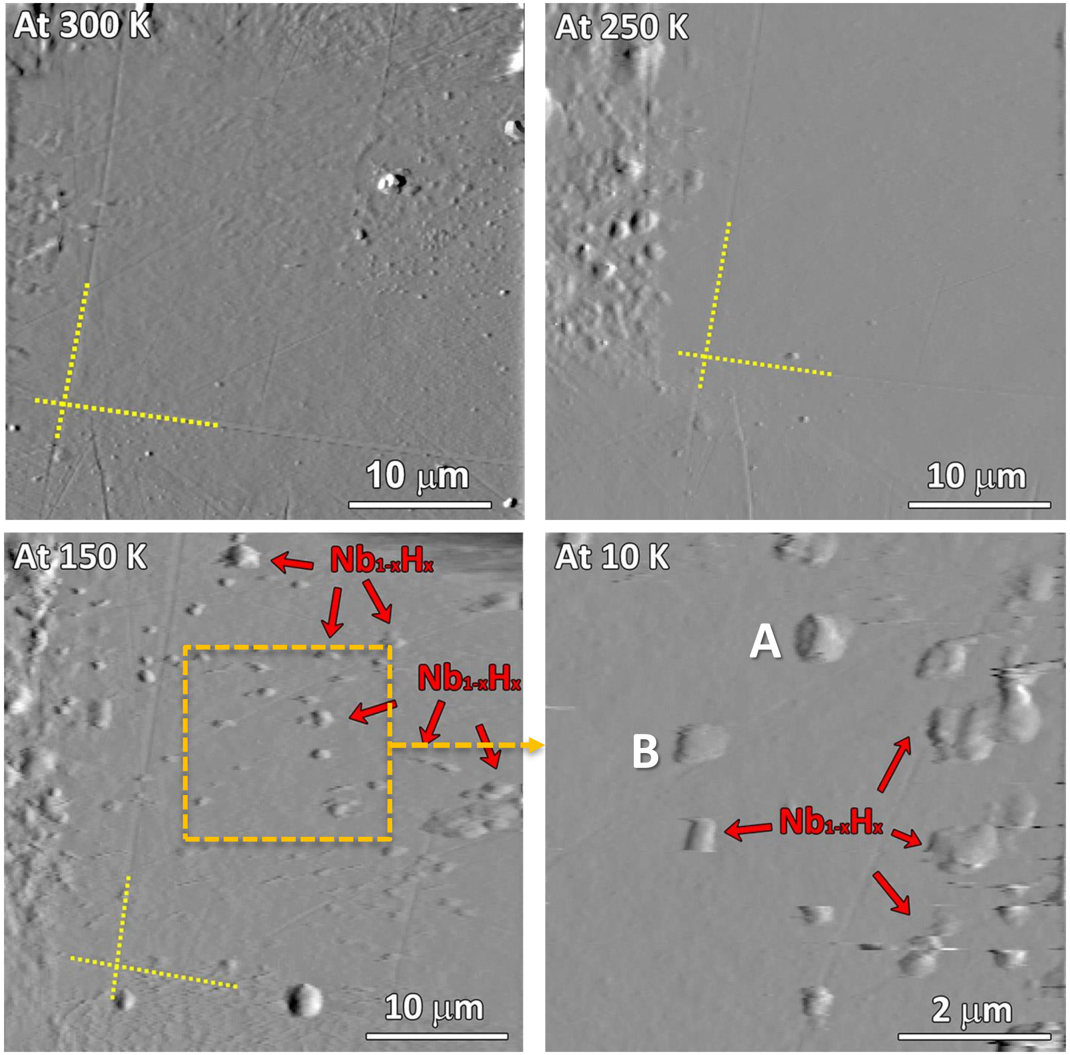


Cut-out samples from the *state of art* cavities

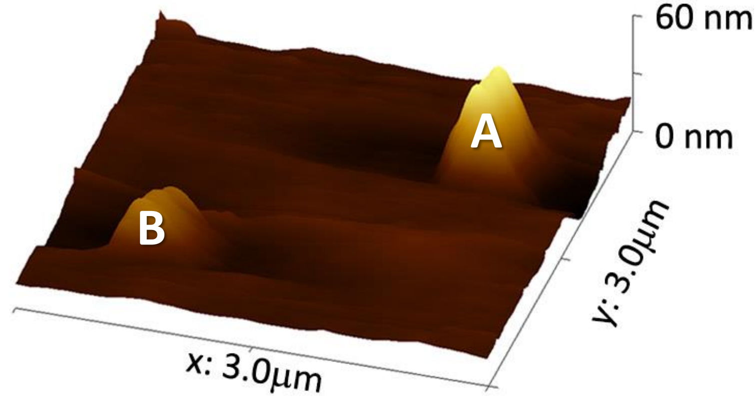
No.	Sample ID	Features	Cavity ID
1	Hot Spot	EP'ed Cavity (non- degassed)	TE1ACC003
2	800°C HT'ed hot spot	800°C HT + 20 µm BCP applied on hot spot cut out (#1)	
3	120°C Baked	EP + 120°C baked	TE1AES004
4	N-Doped	N-doped @800°C 25mTorr (2/6)	TE1ACC002
5	N-Infused	N infused at 120°C	TE1PAV012

First direction observation of nm-size NbH

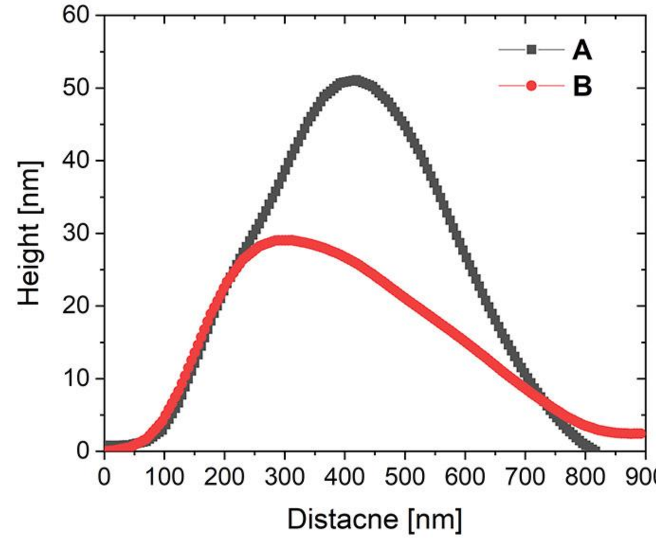
Hot spot cut-out



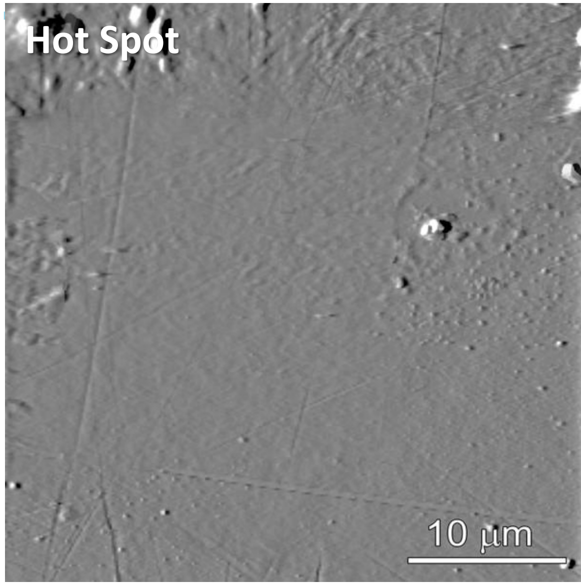
3D surface topology

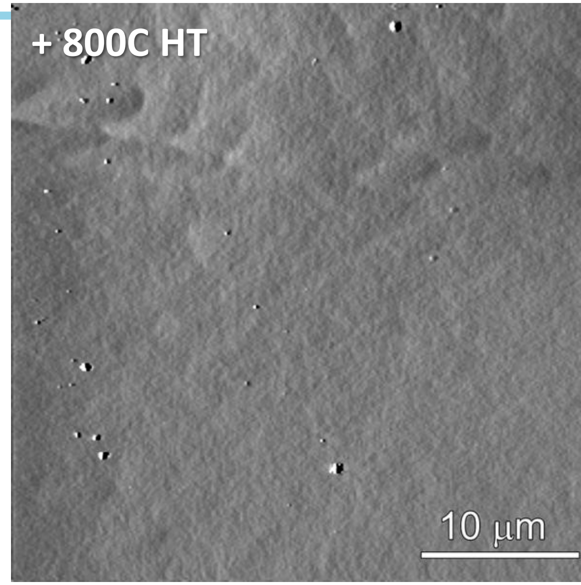


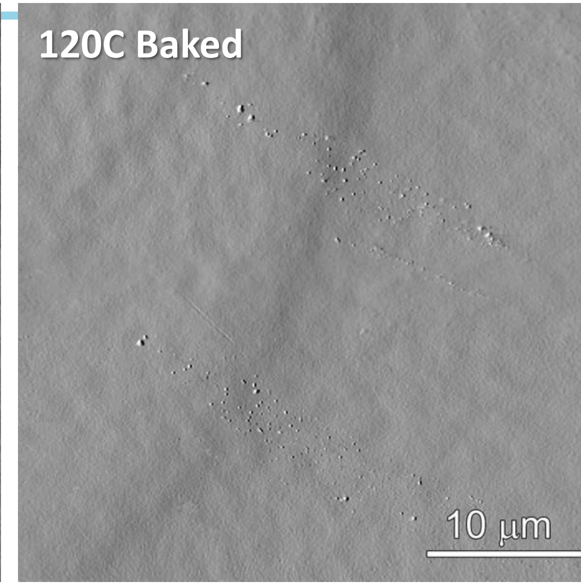
Height Profile across NbH

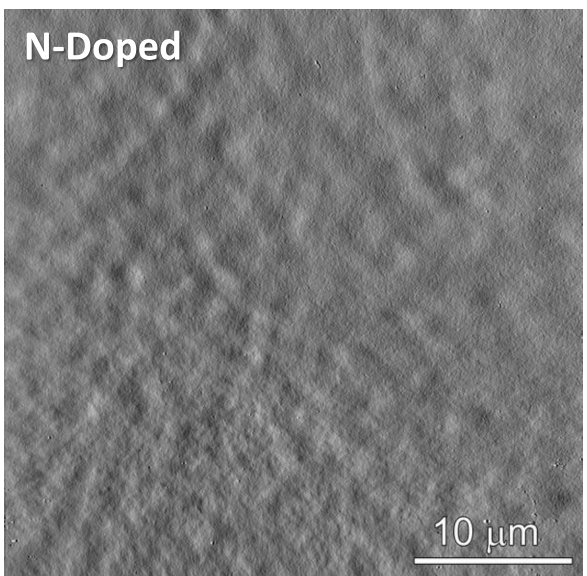


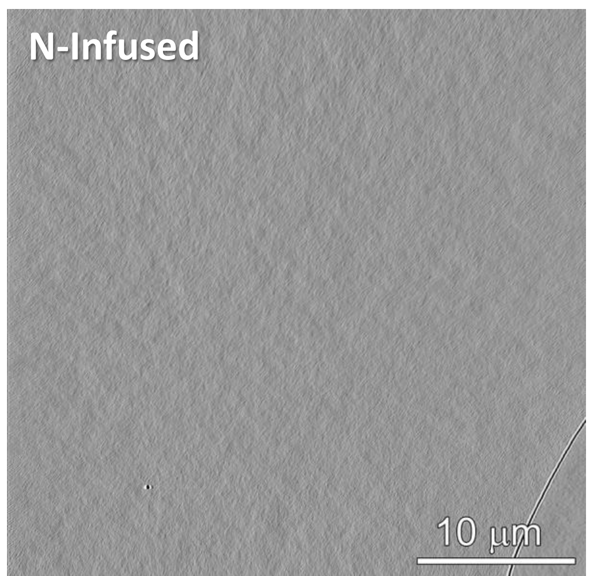
300K Surface features on cavity cut-out samples

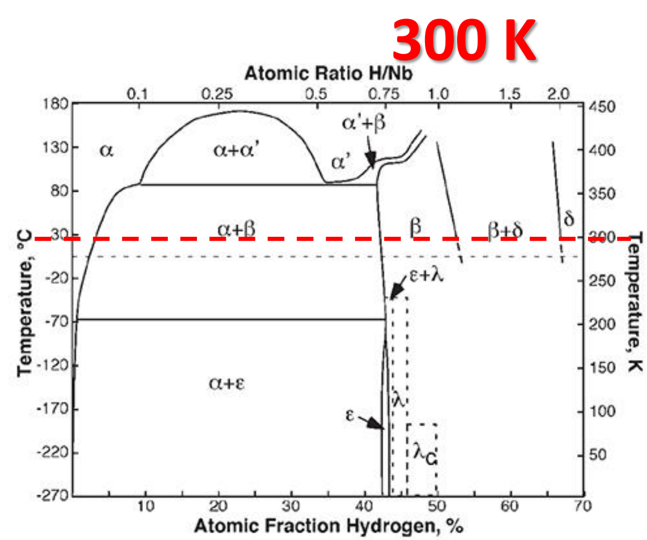












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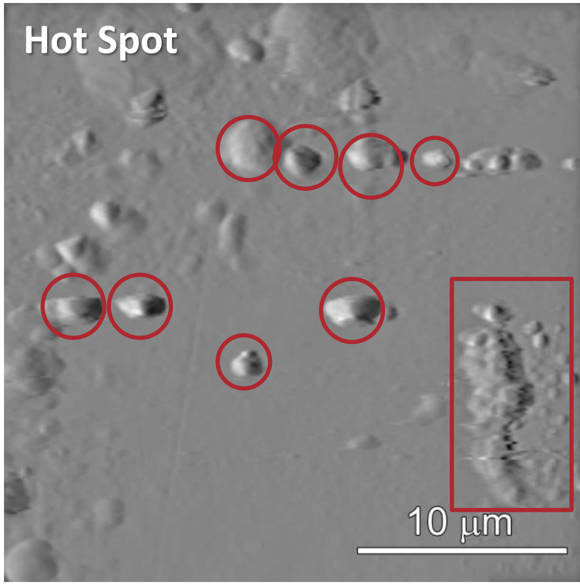
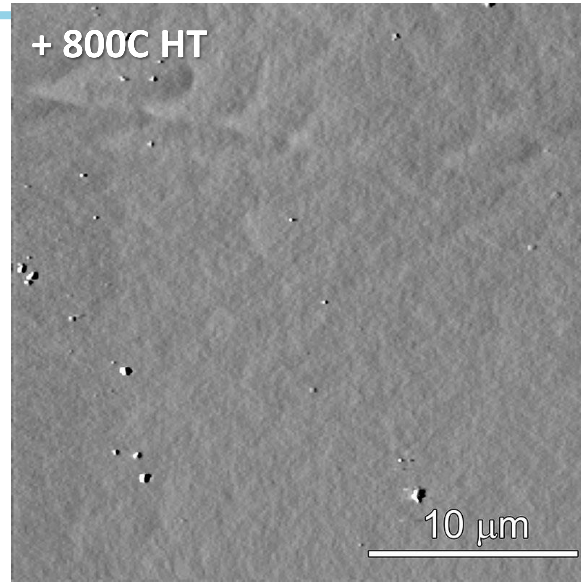
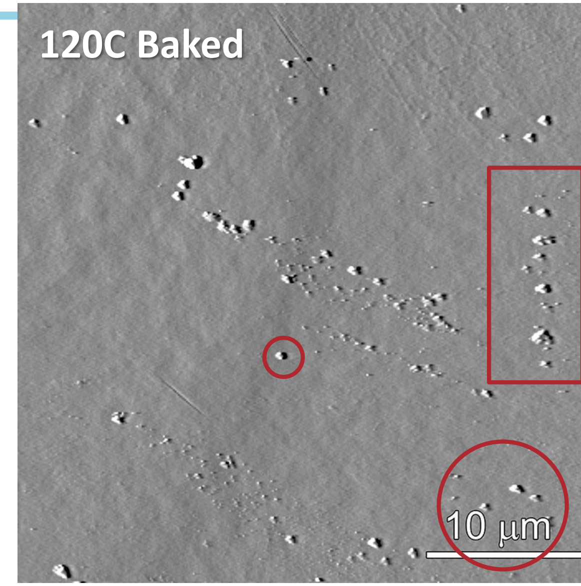
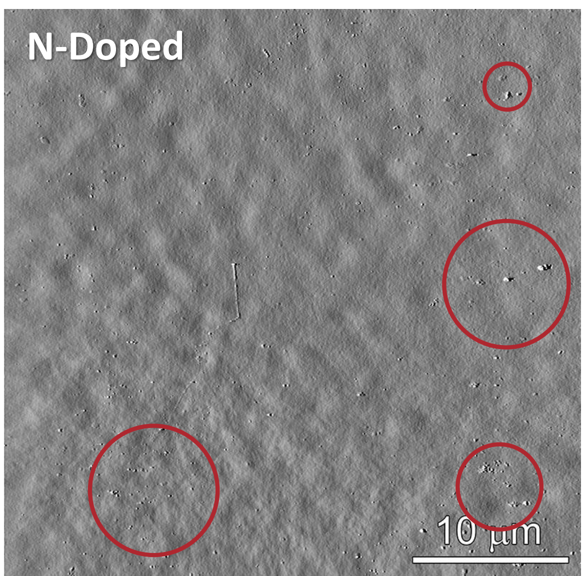
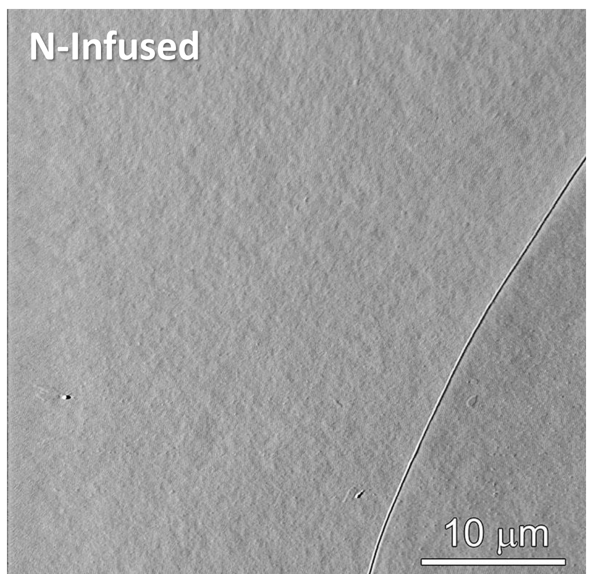
300-200K Surface features on cavity cut-out samples



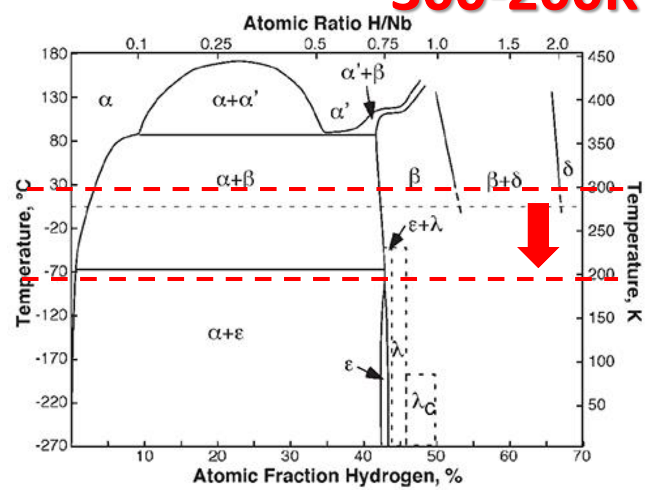








300-200K



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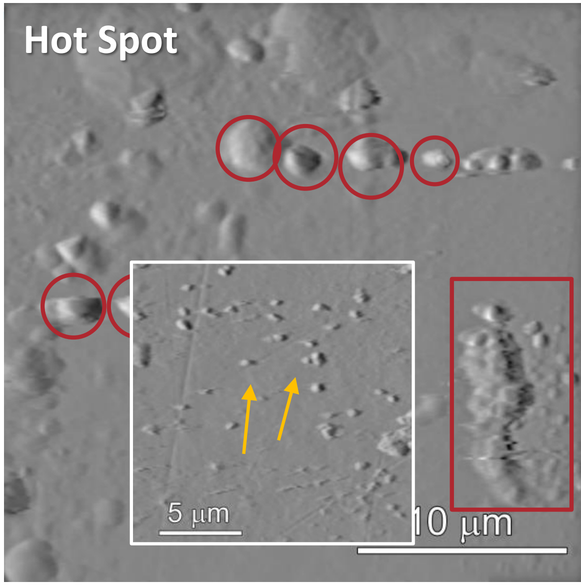
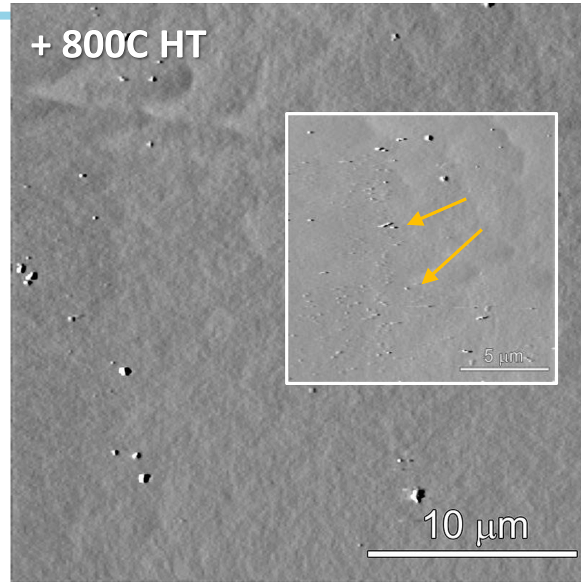
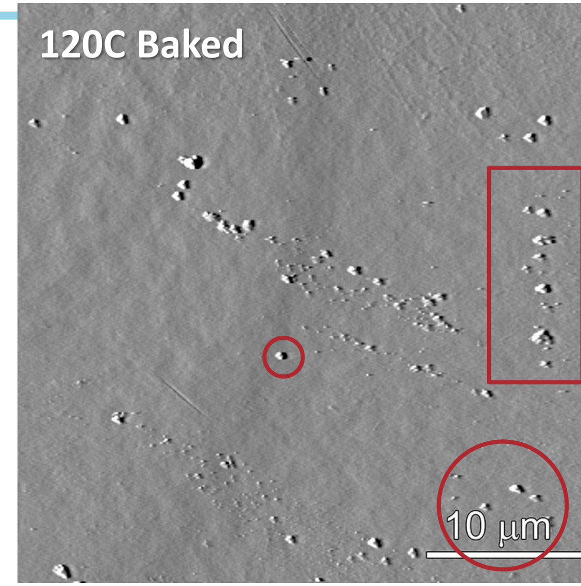
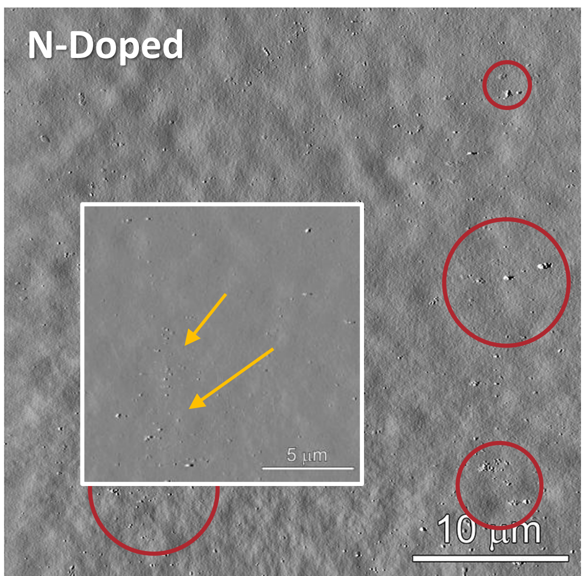
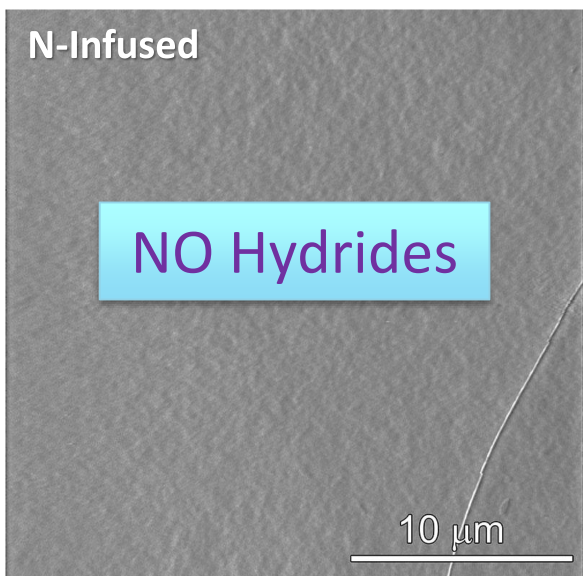
200-100K Surface features on cavity cut-out samples

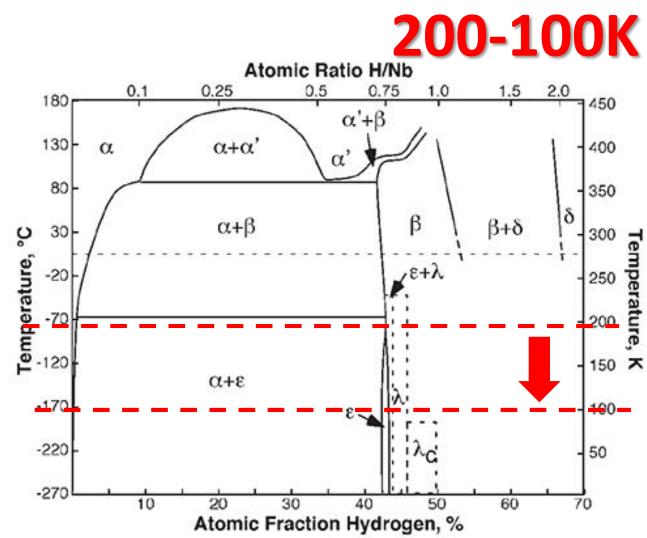












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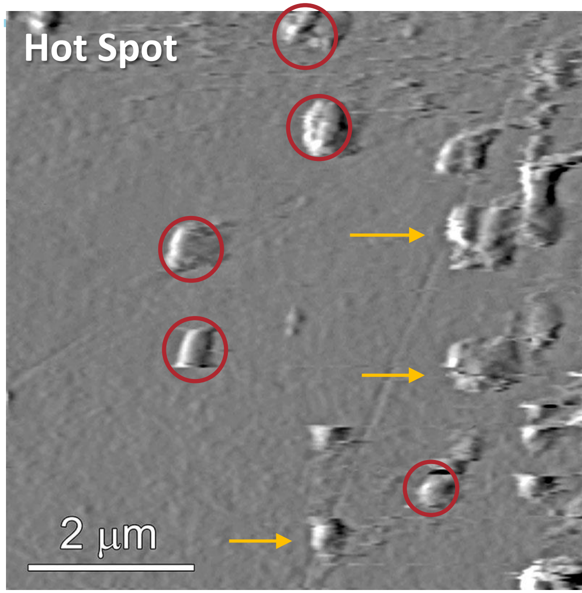
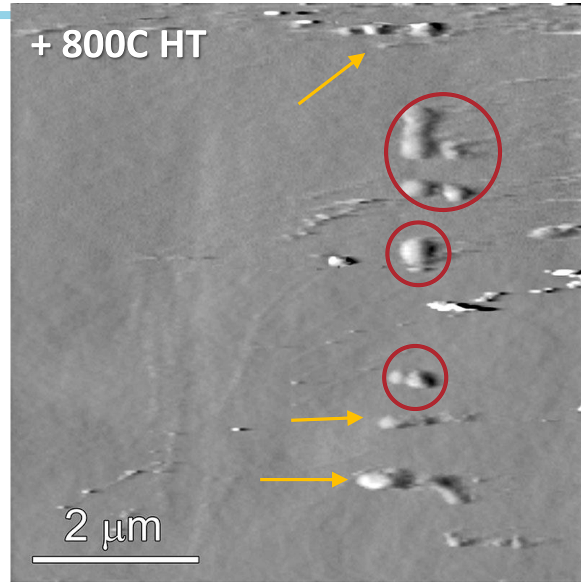
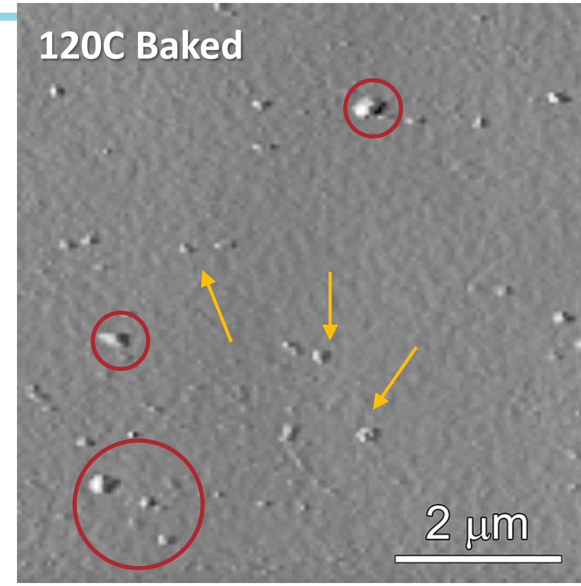
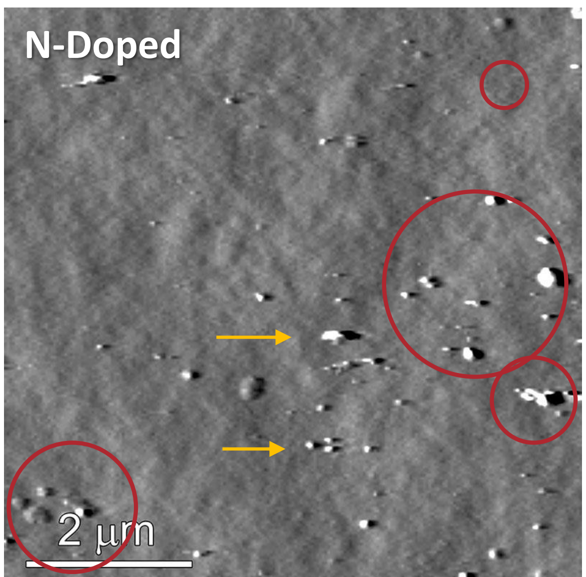
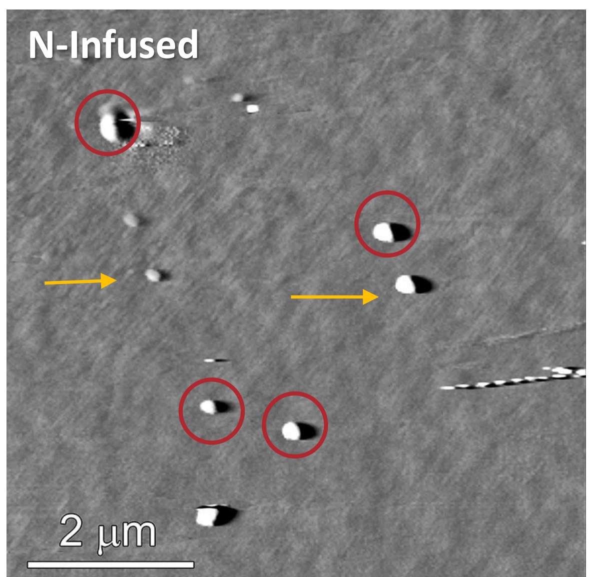
100-10K Surface features on cavity cut-out samples



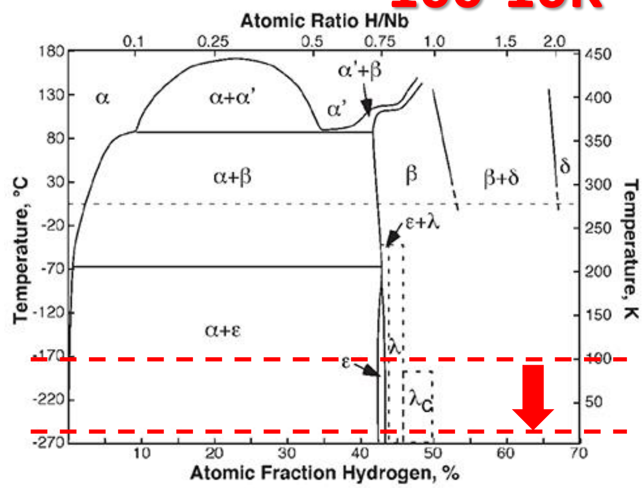






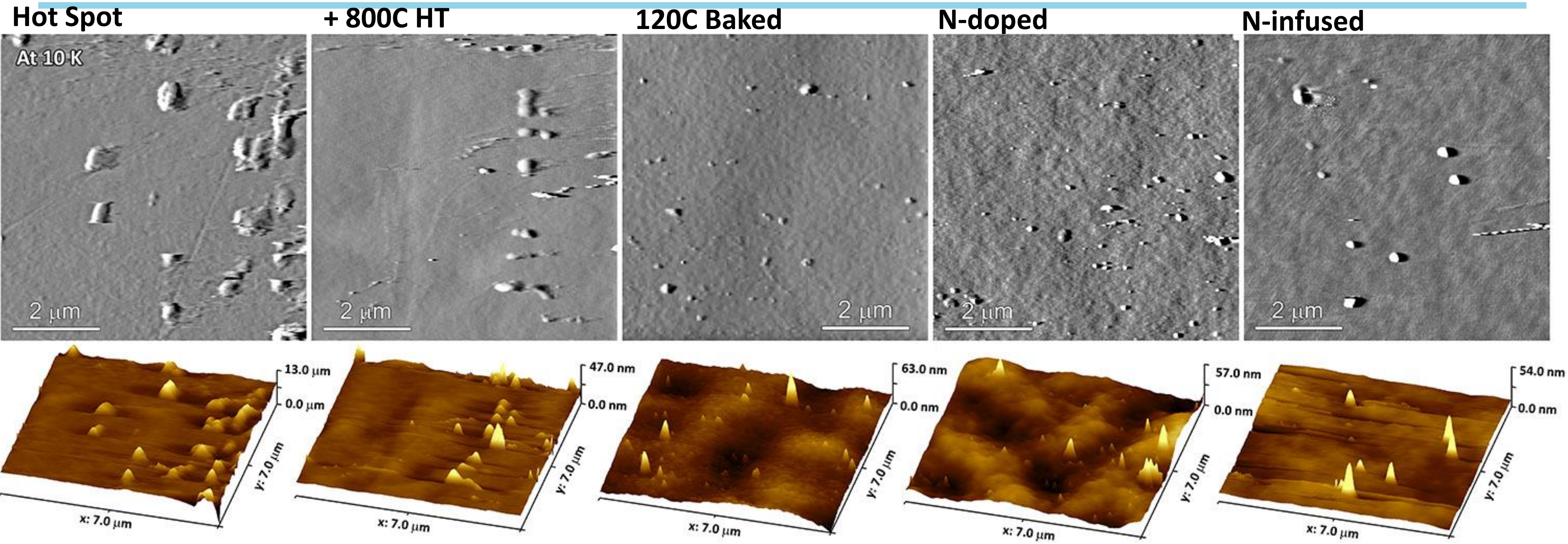


100-10K



At 10K

Surface features on cavity cut-out samples



[7 by 7 µm^2]	No. of Nb hydrides	Total projected area (abs. [µm^2])	Total projected area (rel. [%])	Total NbH volume [10^{-3} × µm^3]
Hot Spot	59	3.579	7.33	337.7
800C HT	36	0.990	2.03	22.29
120C Baked	24	0.462	0.93	14.22
N-Doped	40	0.564	1.14	13.27
N-Infused	16	0.579	1.17	12.82

H trapping by N (?)

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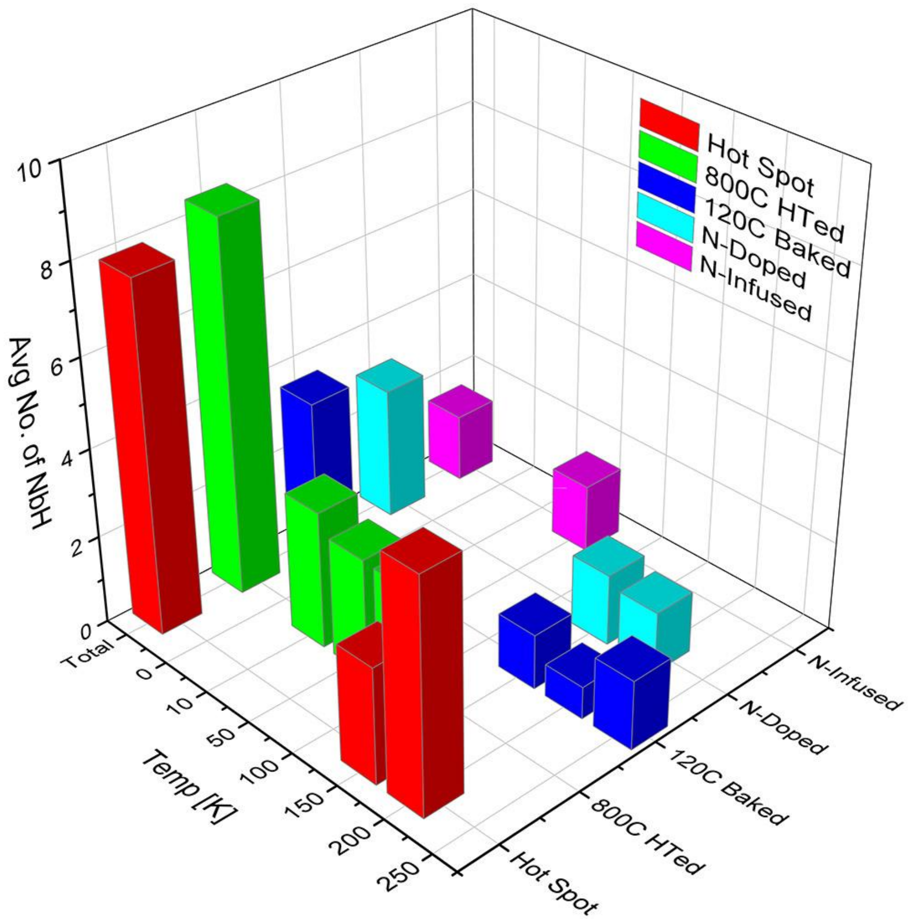
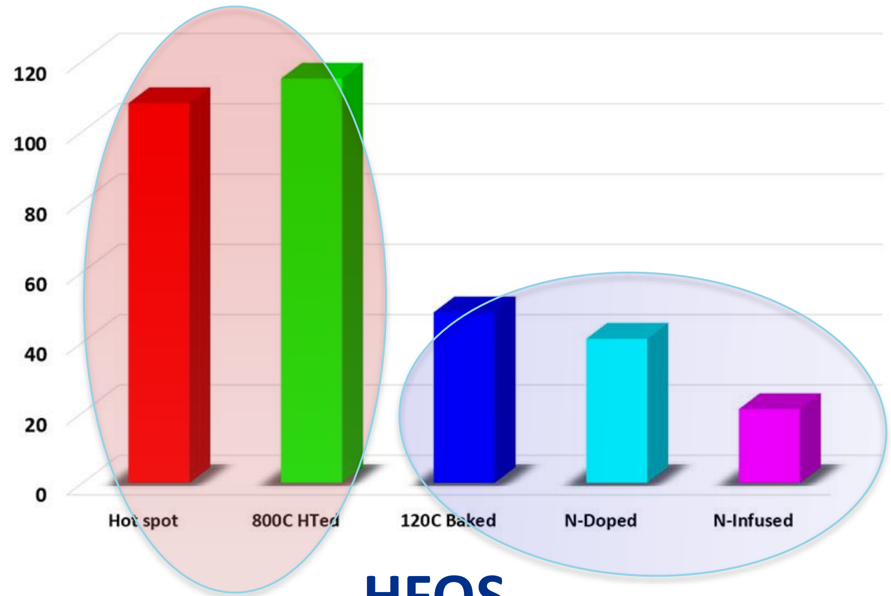
Statistical comparison of NbH precipitation morphology I

Avg. No. of NbH appearance within 10 x 10 μm² unit area during cooling



Total NbH phases during cooling to 10K



HFQS

Yes	No
Hot Spot	120C Baked
800C HT'ed	N-Infused
	N-Doped

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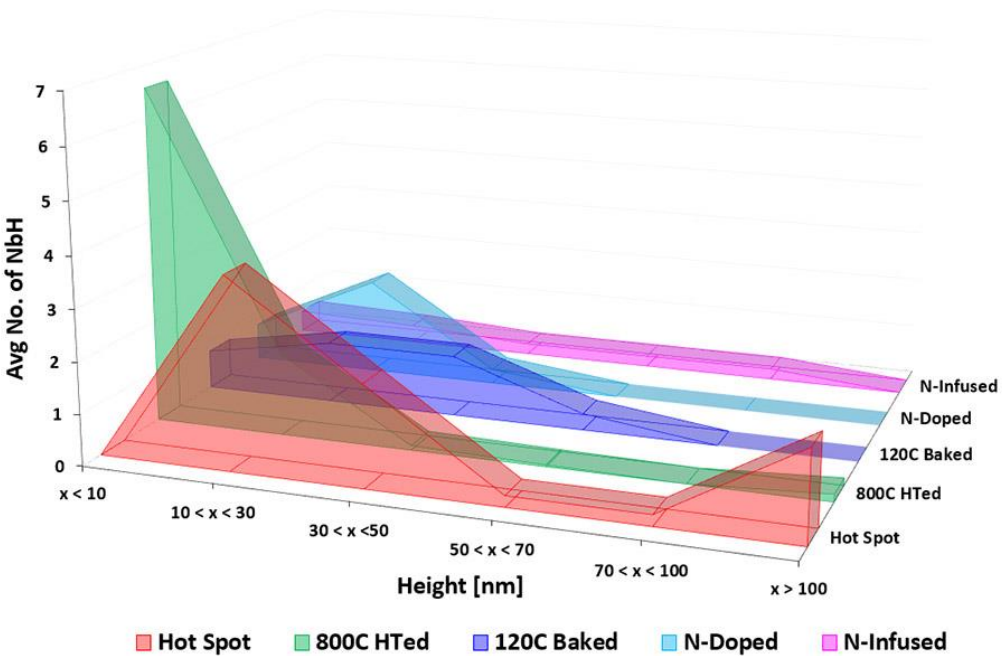
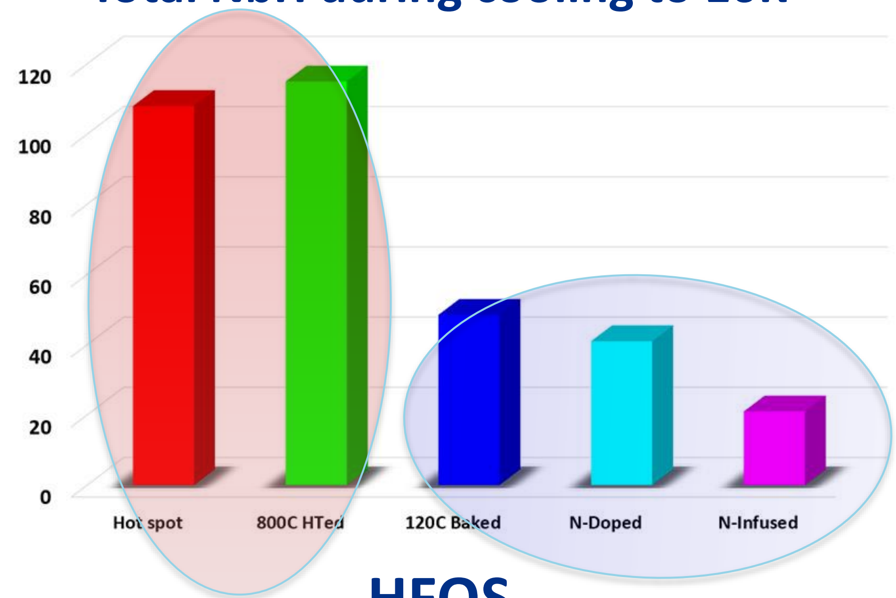
Statistical comparison of NbH precipitation morphology II

NbH size distribution within 10 x 10 μm^2 unit area when cooled to 10K



Total NbH during cooling to 10K



HFQS

Yes	No
Hot Spot	120C Baked
800C HT'ed	N-Infused
	N-Doped

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Complexity on the SRF Nb cavity surface cavity: various NbH phases, vacancy-H superabundance, N-H trapping, etc…

TEM image **Electron Diffraction**

Nb

Oxide

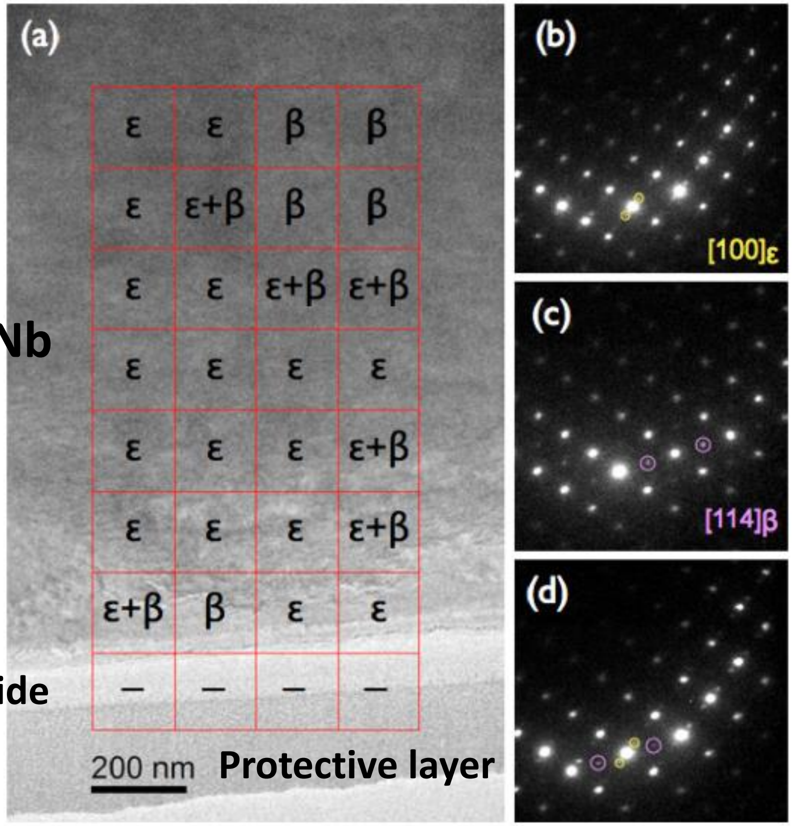
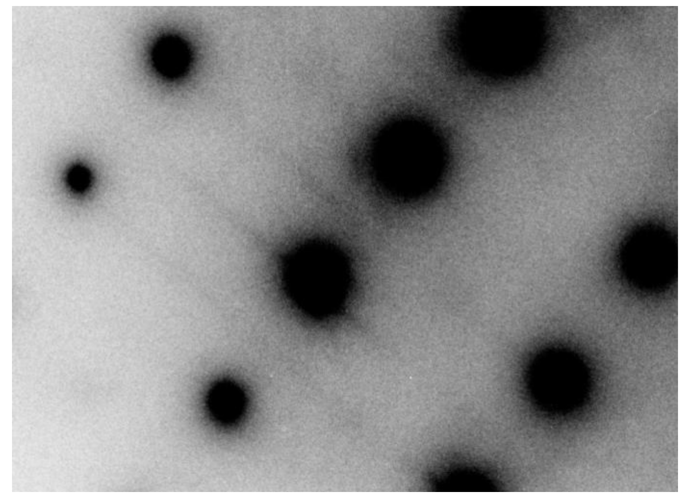


FIG. 6. (a) SEND map of EP sample at 94 K using LaB_6 TEM, (b) ϵ-phase Nb_4H_3 overlapped with Nb, (c) β-phase NbH overlapped with Nb, and (d) ϵ- and β-phases overlapped with Nb.

Y. Trenikhina et al, J. Appl. Phys. 117, 154507 (2015)

Electron Diffraction on N-doped cut out by Cryo-TEM : No Hydrides at 94K



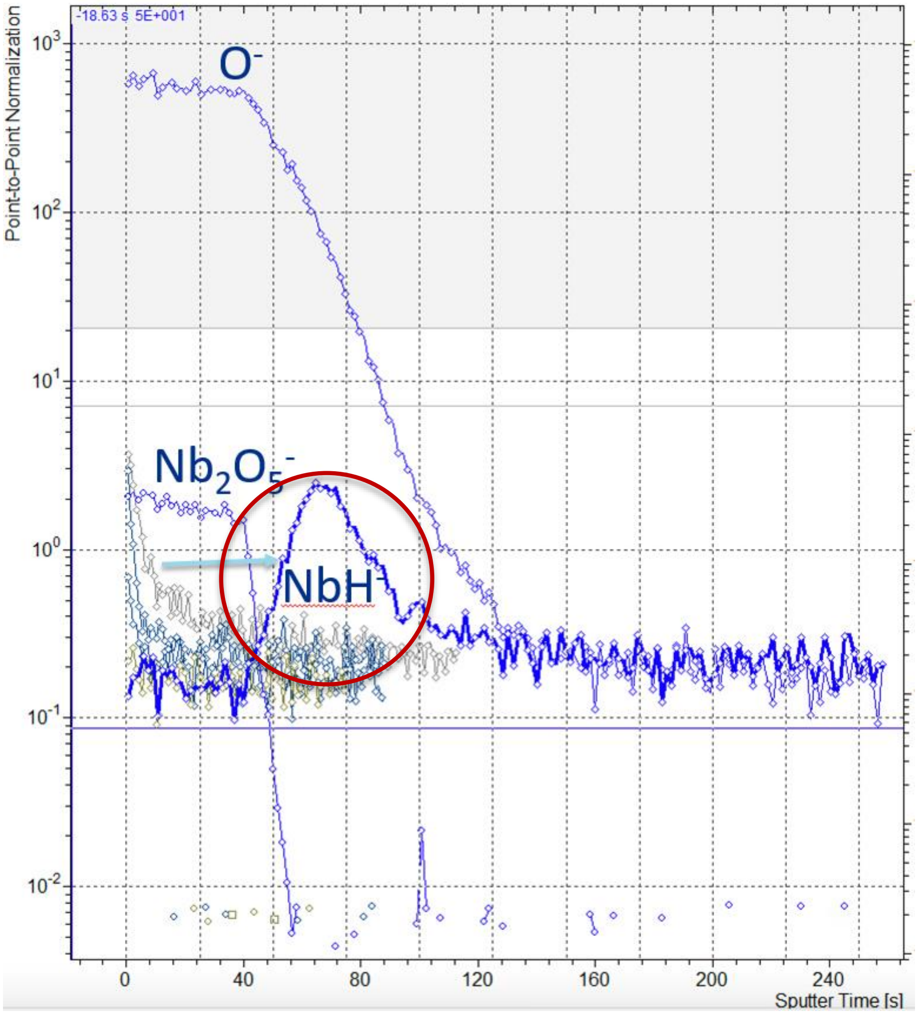
Courtesy of Y. Trenikhina

→ Nitrogen favorably trap "Hydrogen"

G. Pfeiffer and H. Wipf, J. Phys. F: Metal Phys. Vol 6. No. 2, (1976)

Intaken hydrogens remain at the interface between Nb matrix and Nb_2O_5 oxide layer

SIMS (Secondary Ion Mass Spectroscopy) Profile on SRF Nb Surface



Performed by A. Romanenko
to be published

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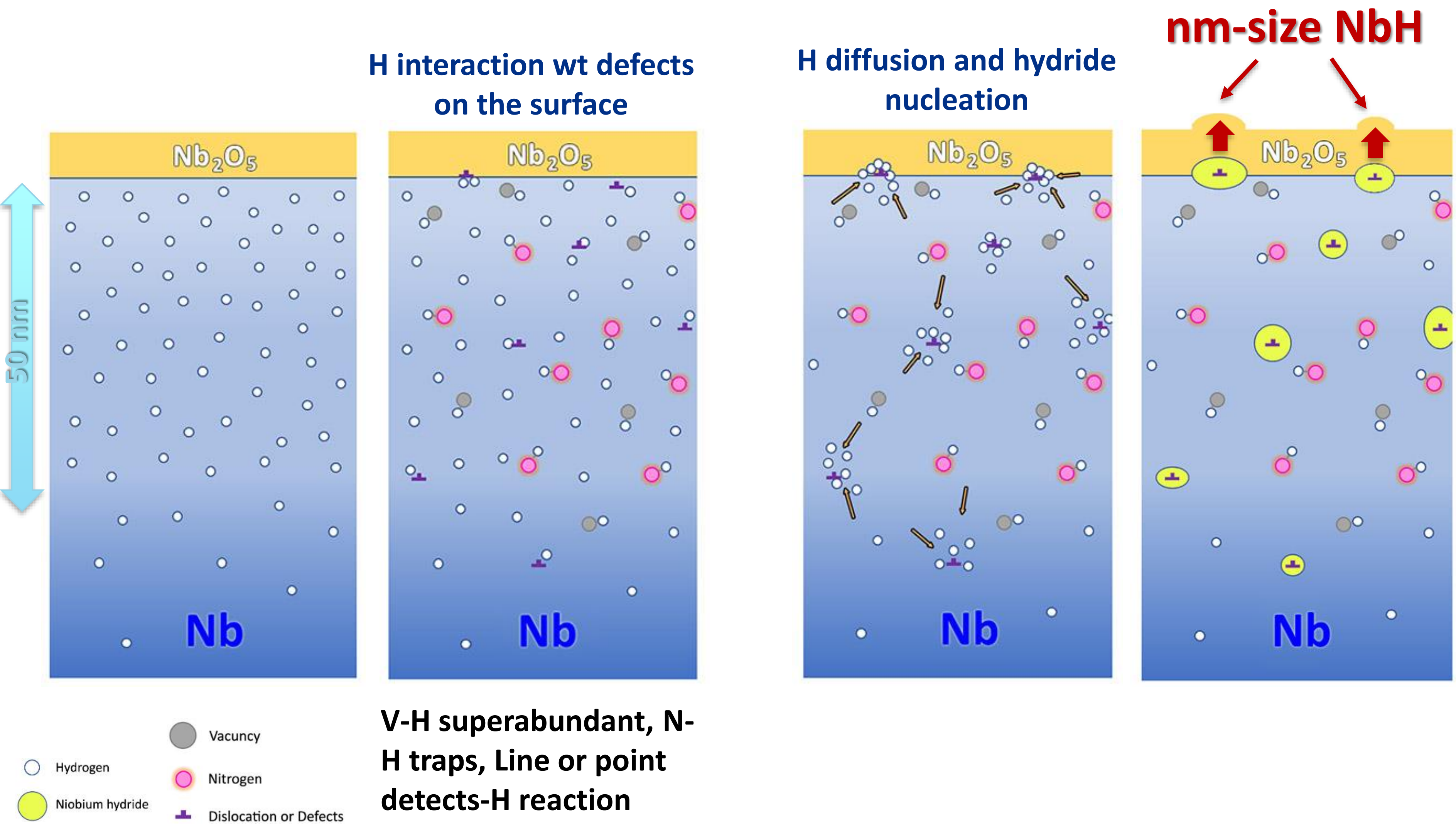
Proposed nm-size NbH precipitation mechanism on SRF Nb

300K

<< 300K: Cryogenic Cooling



V-H superabundant, N-H traps, Line or point detects-H reaction

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Summary

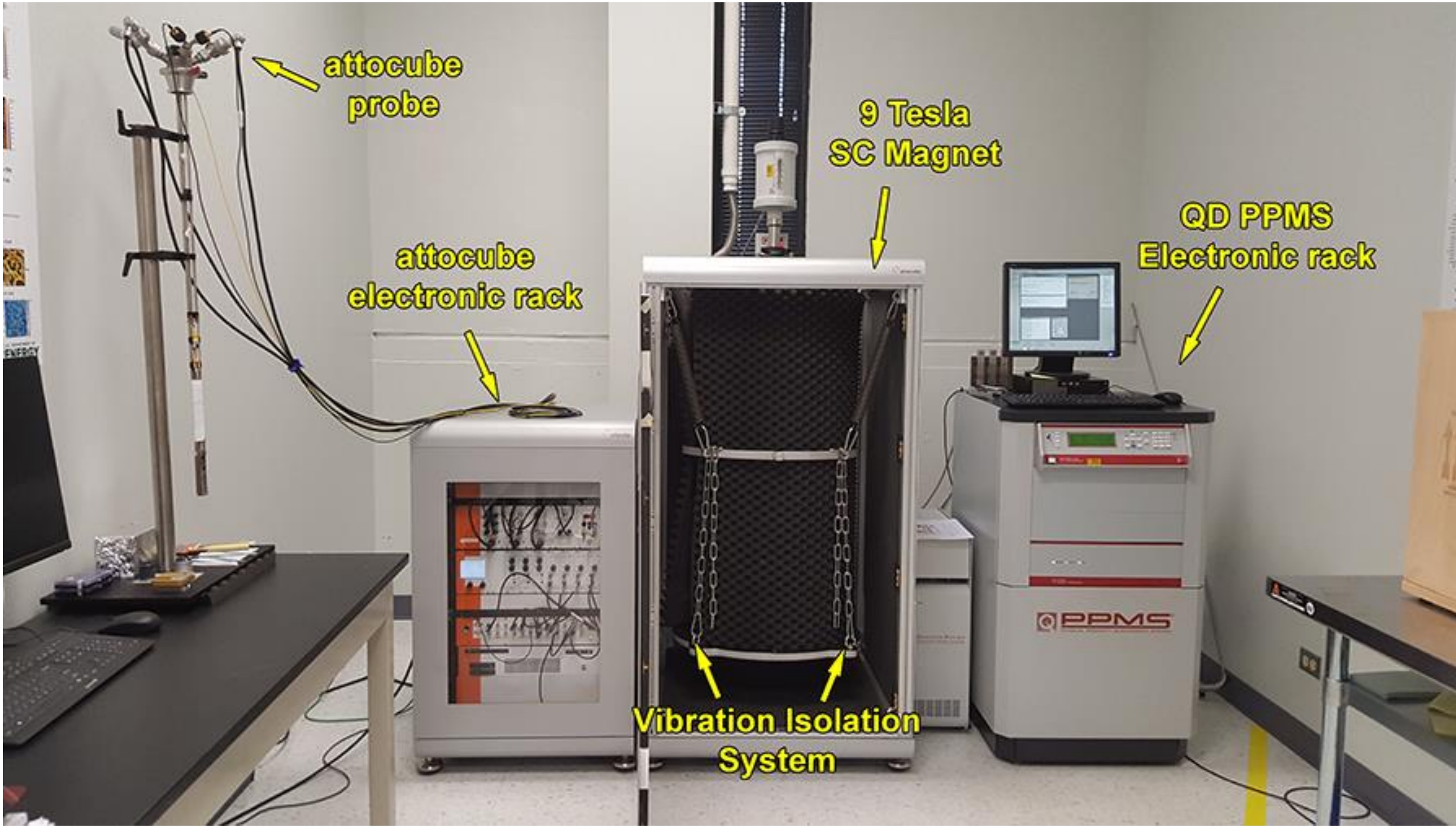
- nm-size NbH phase favorably precipitates on SRF Nb cavities.
- Using cryo-AFM, we successfully observed nm-size NbH precipitation, which can be strongly related to HFQS on SRF Nb cavities.
- We confirmed the scenario of the previous studies that nitrogen and vacancy are favorable to trap “Hydrogen”, leading to suppression of nm-NbH segregation.
- In contrast to “heavily deformed surface structure by µm-size NbH phase” for Q-disease, nm-size NbH phase likely segregates at the interface between Nb_2O_5 oxide layer and Nb matrix, and then re-dissolve into the Nb matrix after warming up above 300K without lattice destruction.

Acknowledgements

- A. Romanenko, A. Grassellino, Y. Trenikhina (now at Sila Nano tech), S. Posen, M. Checchin, M. Martinello, T. Spina, D. Bafia, B. Giaccone, C. Crawford, R. Schuessler.

THANK YOU!

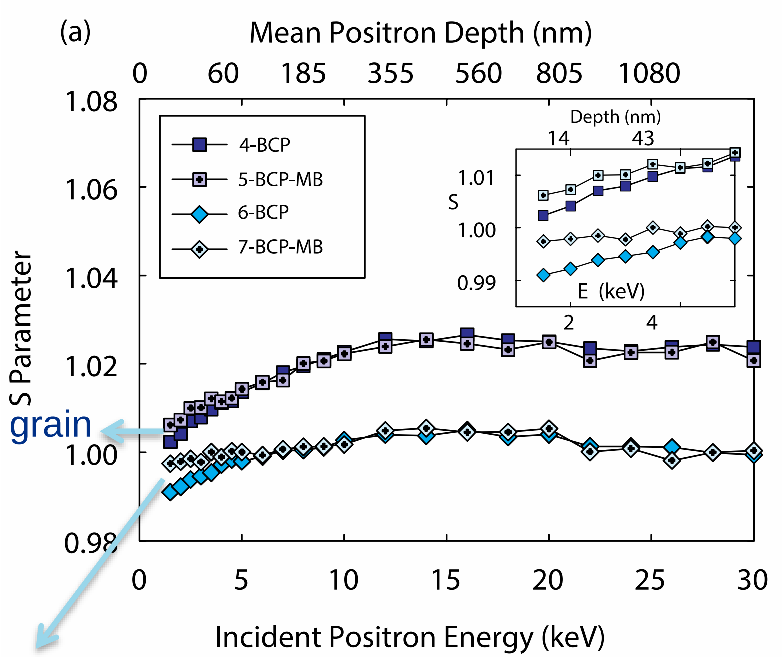
Cryogenic AFM/MFM (attocube) + 9T PPMS System (QD Design) @SRF Materials Science Lab at APS-TD, FNAL



Positron annihilation studies on cavity cutouts

Collaboration with Bath University (UK) and Western University (Canada)

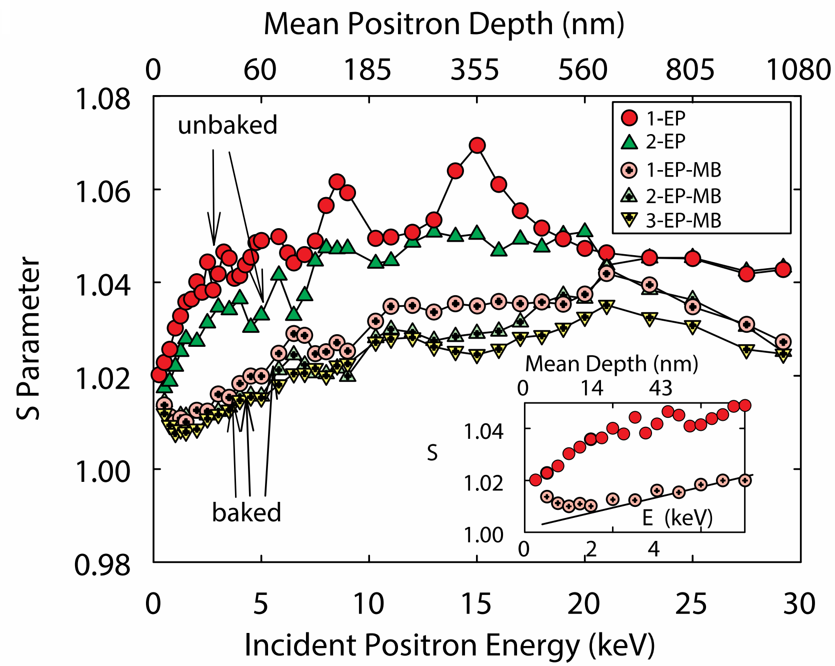
A. Romanenko, C. J. Edwardson, P. G. Coleman, P. J. Simpson, Appl. Phys. Lett. **102**, 232601 (2013)



Fine grain

Large grain

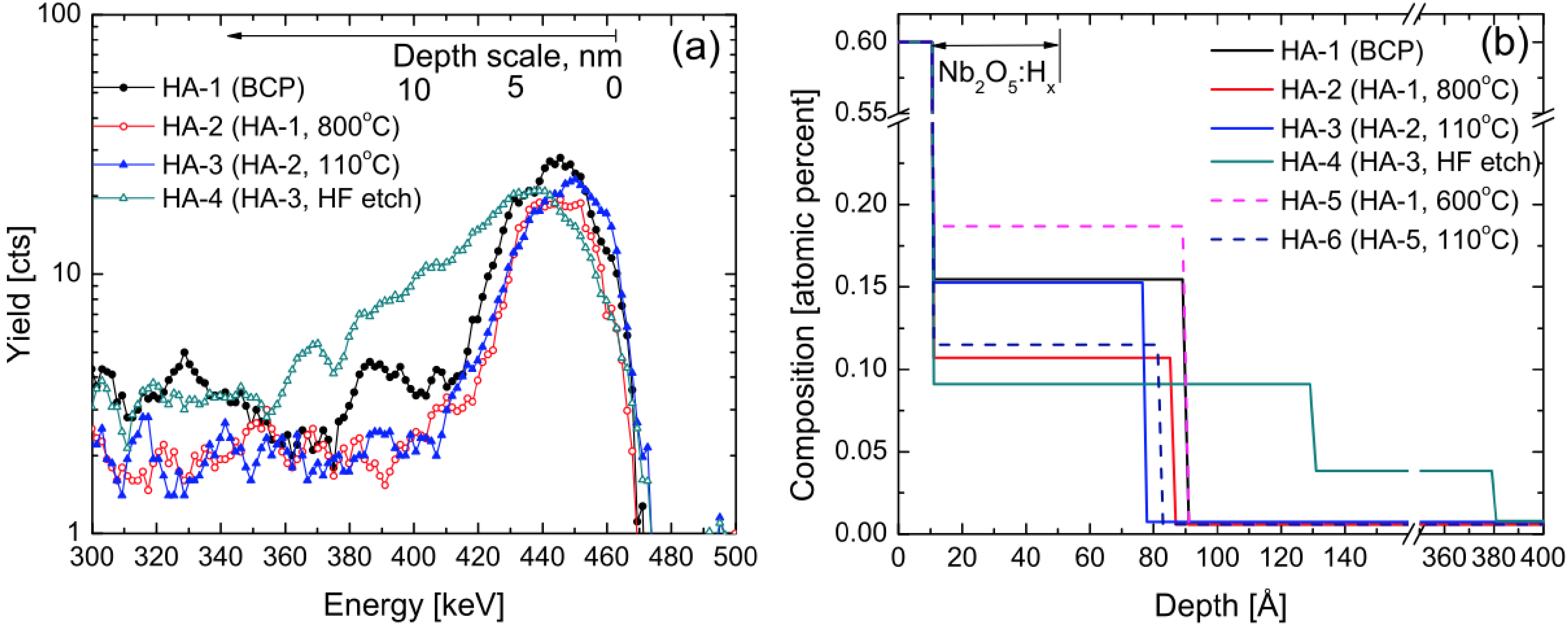
BCP



EP

- Positron annihilation spectroscopy: 120C baking results in “doping” of the first ~50 nm from the surface with vacancies
  - So-called superabundant vacancy formation mechanism manifested in niobium [Y. Fukai and N. Okuma, Phys. Rev. Lett. **73**, 1640 (1994)]

Near-surface H present even after 600-800C degasing



A. Romanenko and L. V. Goncharova, 2011 Supercond. Sci. Tech. **24**, 105017

Q-disease is eliminated by the 600-800C H degassing (bulk H content drastically reduced), but the near-surface H-rich layer remains