Direct correlation of state of the art cavity performance with surface Nb nano-hydrides cutouts observed via cryogenic-AFM

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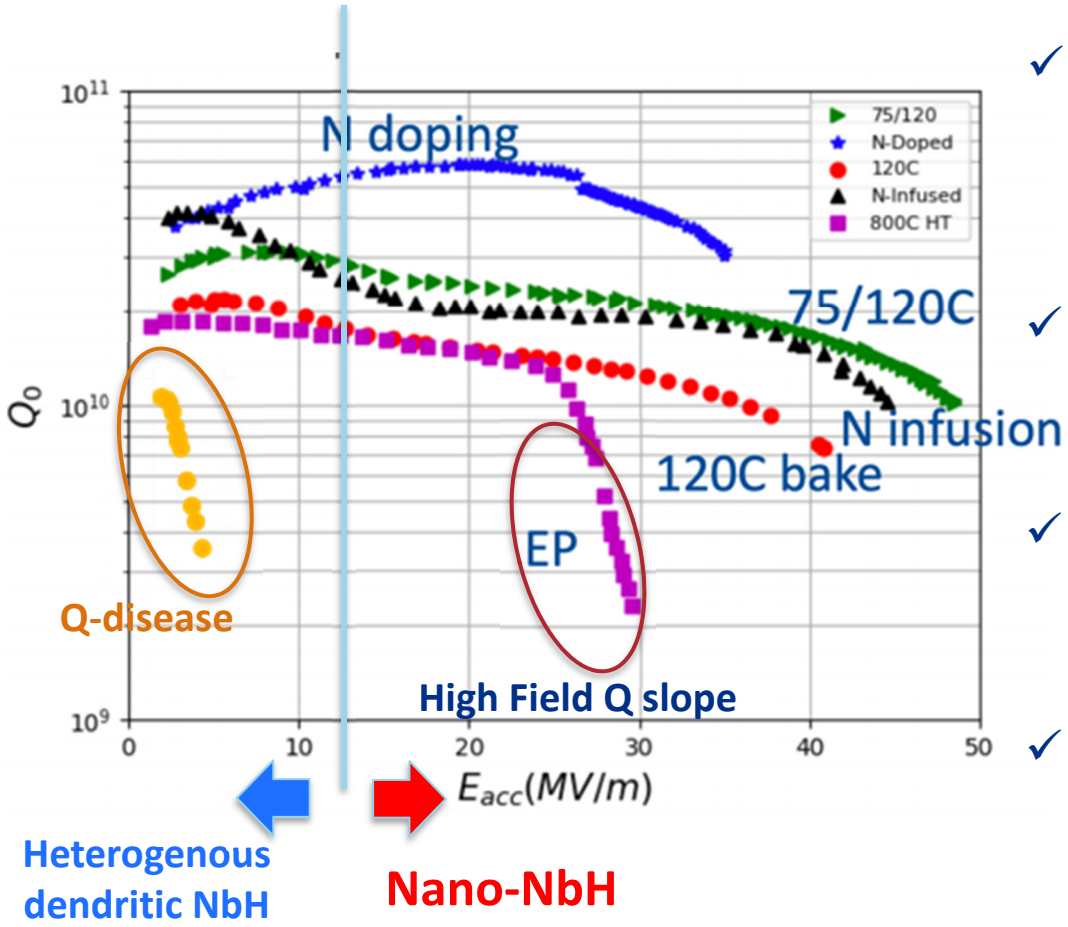
Outline

- Q) What is the best scenario of SRF Nb cavity performance regarding to hydride segregation morphology?
- First direct identification of nm-size NbH using cryo-AFM.
- Comparison of NbH formation on the *state-of-art* SRF Nb cavities.
- First observation of NbH phase segregation on SRF cavity surface at or above room temp.
- Summary

Cavity performance relative to Nb hydride precipitates, beyond Q-disease

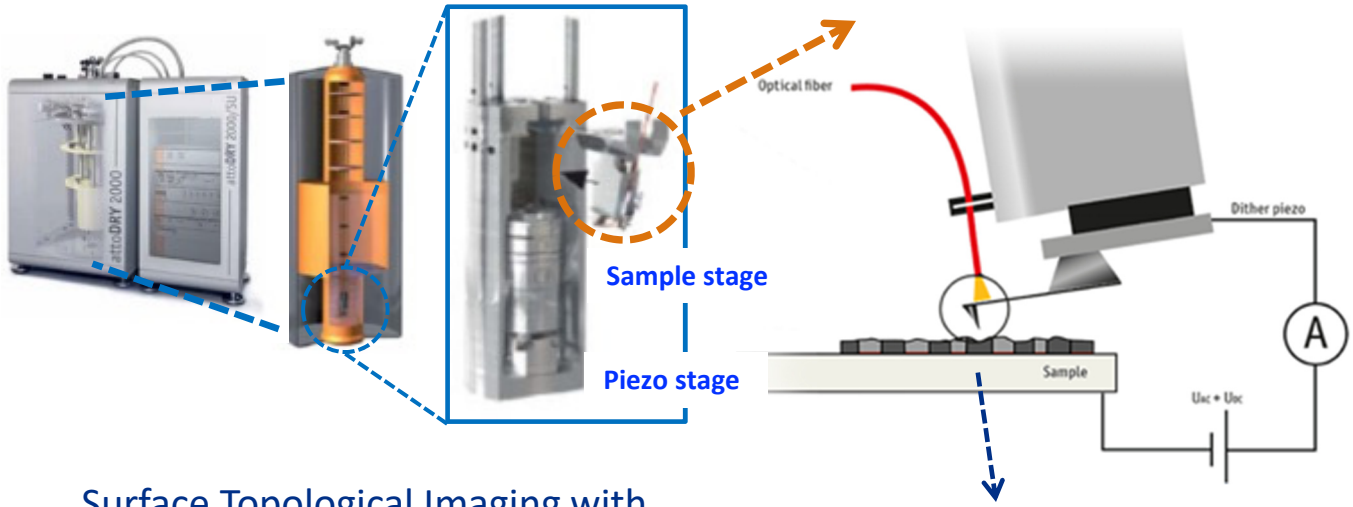


- ✓ 800°C HT reset "H" in SRF Nb bulk for Q-disease, but 800°C HT'ed cavity still shows HFQS.
- ✓ 120°C bake is an empirical remedy for HFQS: Vacancy-Hydrogen complexes.
- ✓ Nitrogen's H-trapping is stronger than V-H complex formation energy.
- ✓ Recent 75/120C baked cavity requires detailed NbH study in comparation to other cavities.

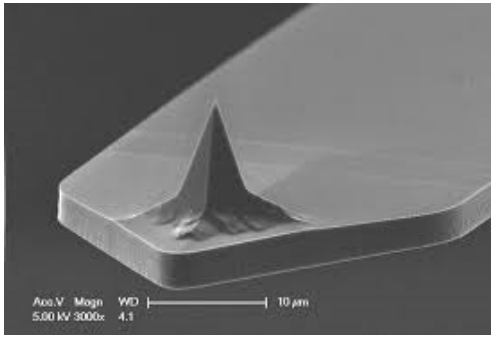
Courtesy of A Grassellino, TTC, Vancouver, 2019

Fermilab

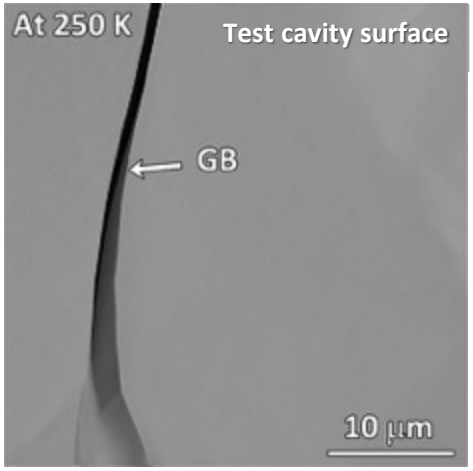
Cryogenic AFM (attocube) + 9T PPMS System (QD Design) @SRF Materials Science Lab at APS-TD, FNAL

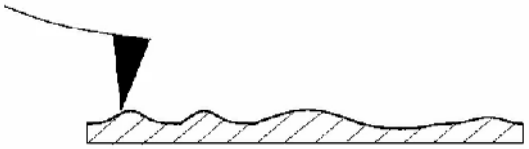


Atomic Force Microscopy tip



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

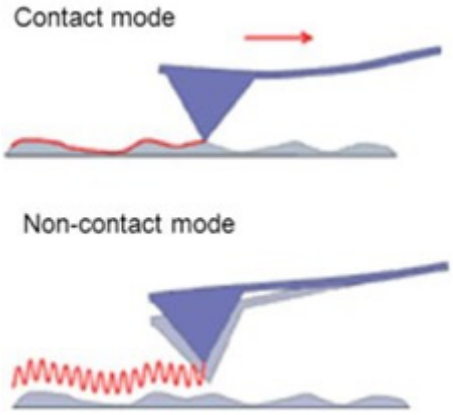




1-5 nm X-Y-Z resolution

H_{ext}= 0T-9T,
T=1.8K-400K

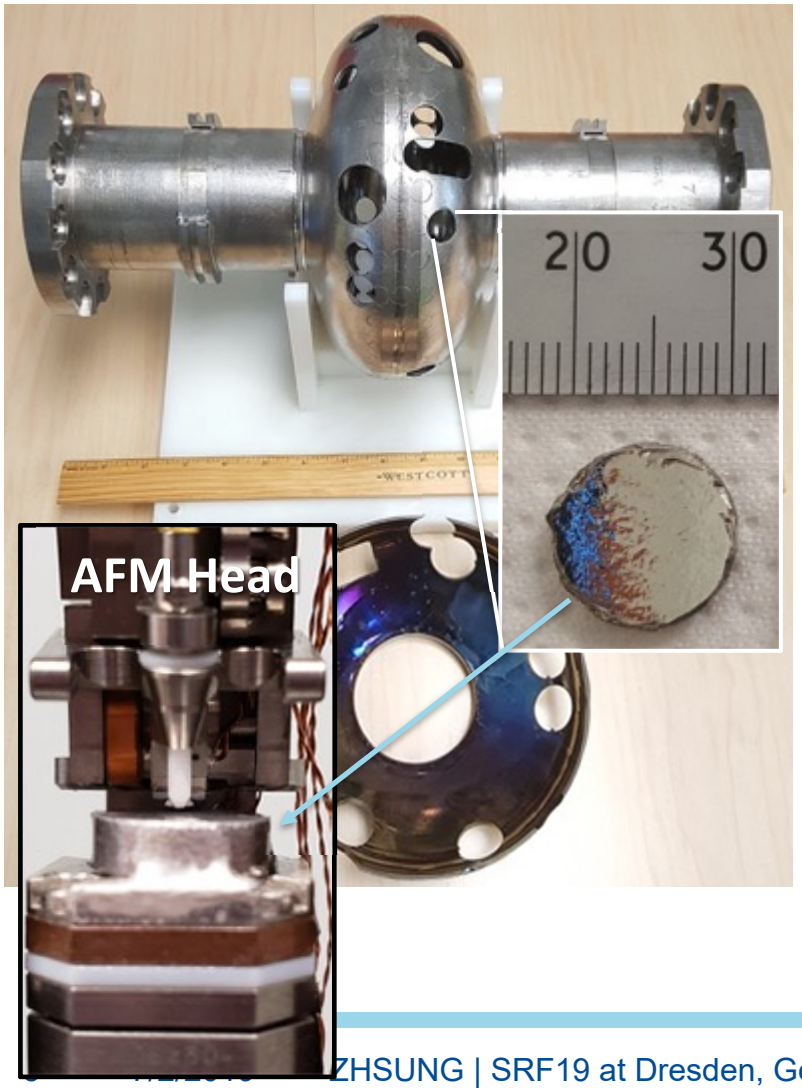
AFM Operation Mode



Fermilab

Sample for direct study on nano-Nb hydride precipitation

Tested cavity cut-outs

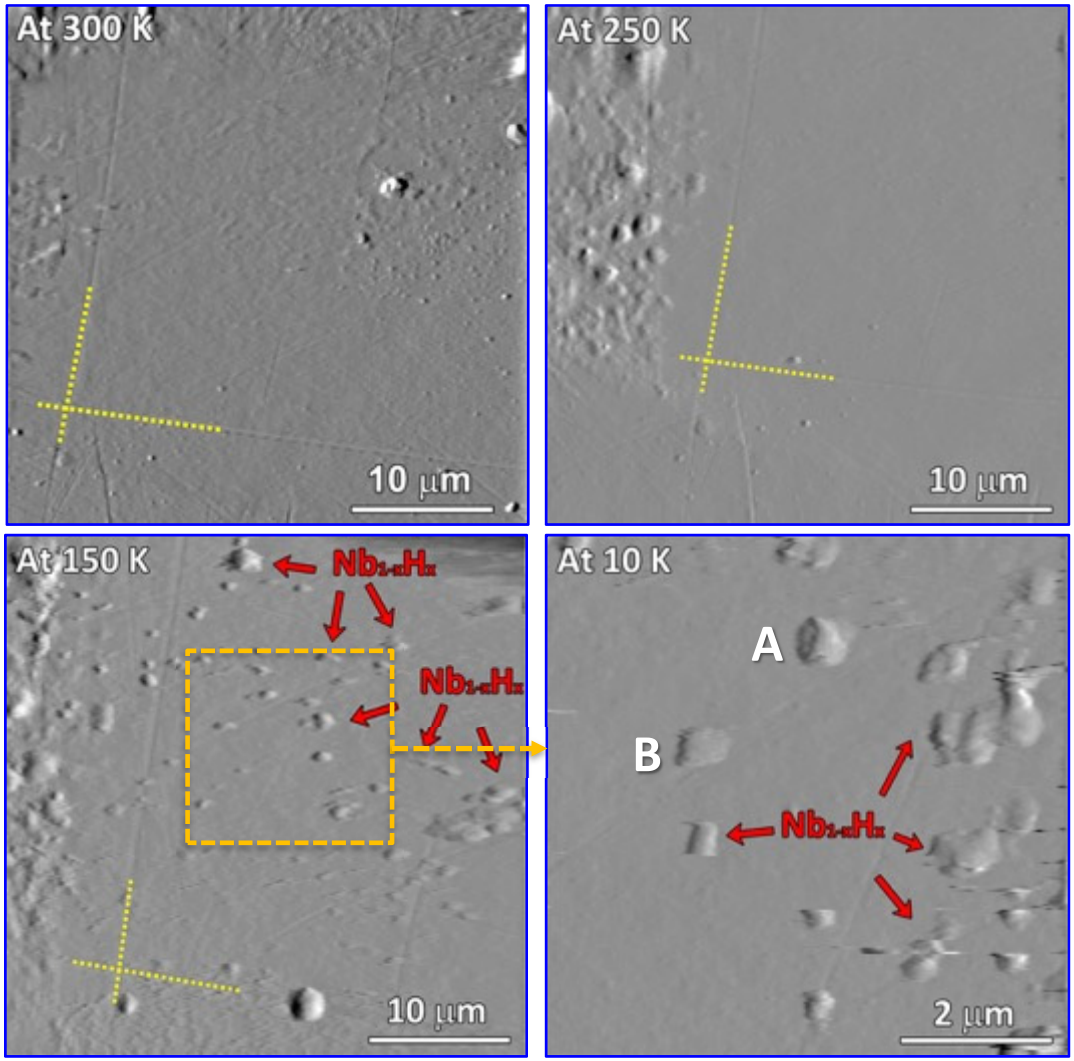


Cut-out samples from the *state of art* cavities

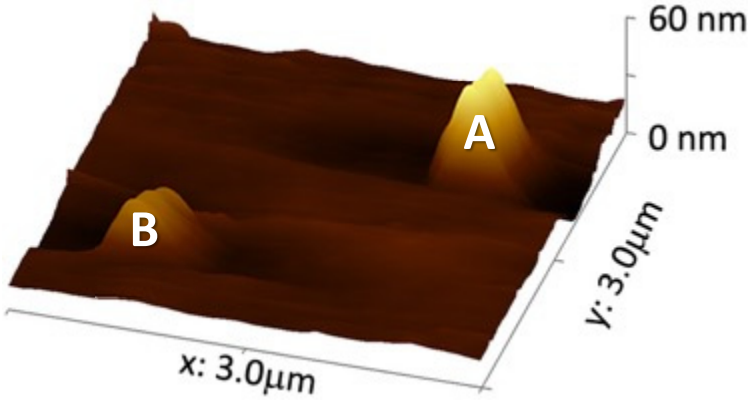
No.	Sample ID	Features	Cavity ID
1	Hot Spot	EP'ed Cavity (non-degassed)	TE1ACC003
2	800°C HT'ed hot spot	800°C HT + 20 µm BCP applied on hot spot cut out (#1)	
3	120°C Baked	EP + 120°C baked	TE1AES004
4	N-Doped	N-doped @800°C 25mTorr (2/6)	TE1ACC002
5	N-Infused	N infused at 120°C	TE1PAV012

First direct observation of nm-size NbH precipitates

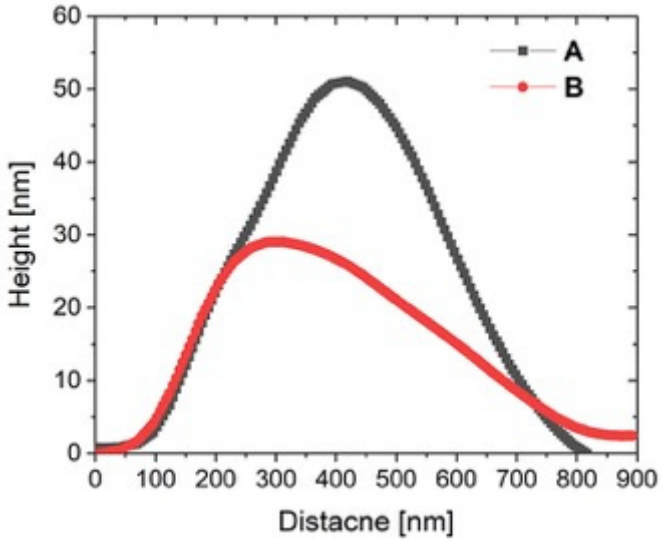
Cryo AFM on hotspot (EP'ed non-degassed cavity)



3D surface topology



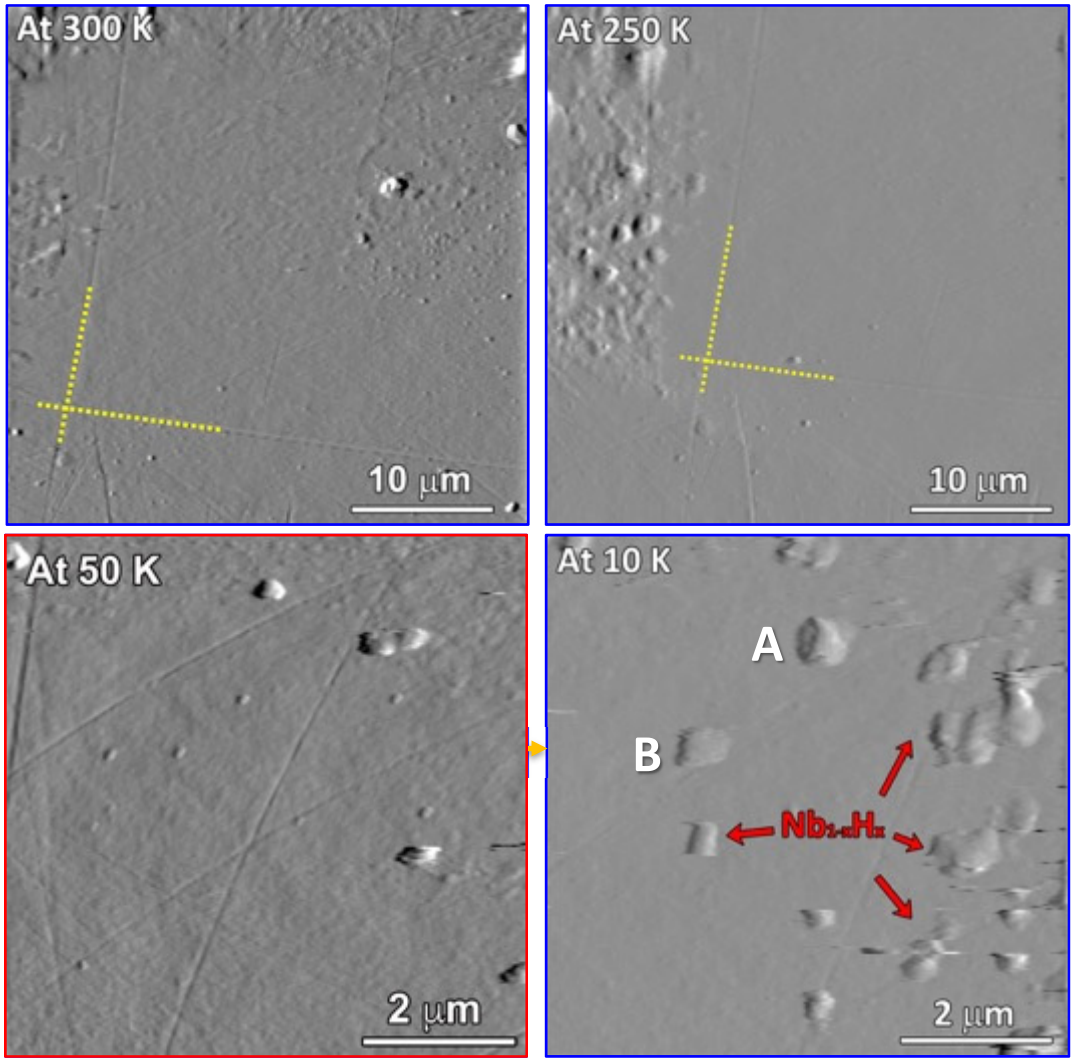
Height Profile across NbH Phase



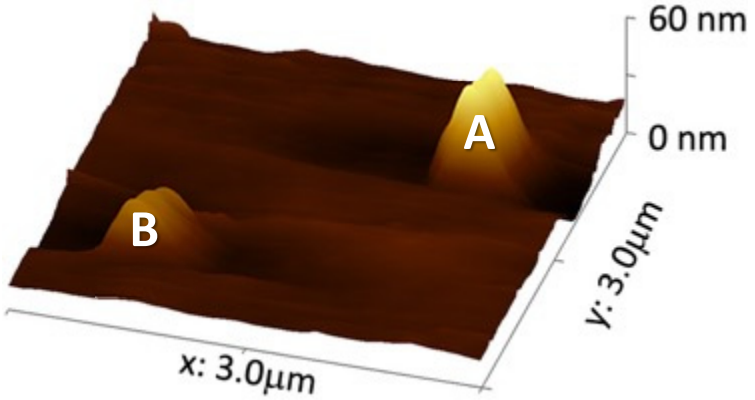
Fermilab

First direct observation of nm-size NbH precipitates

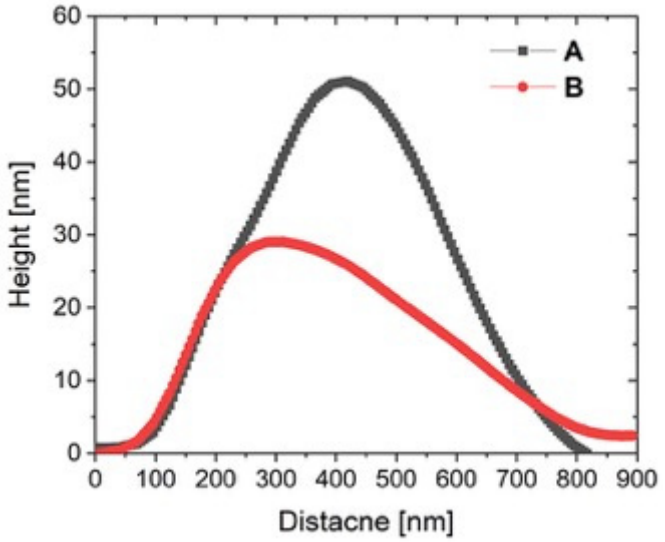
Cryo AFM on hotspot (EP'ed non-degassed cavity)



3D surface topology



Height Profile across NbH Phase



Fermilab

First direct observation of nm-size NbH precipitates

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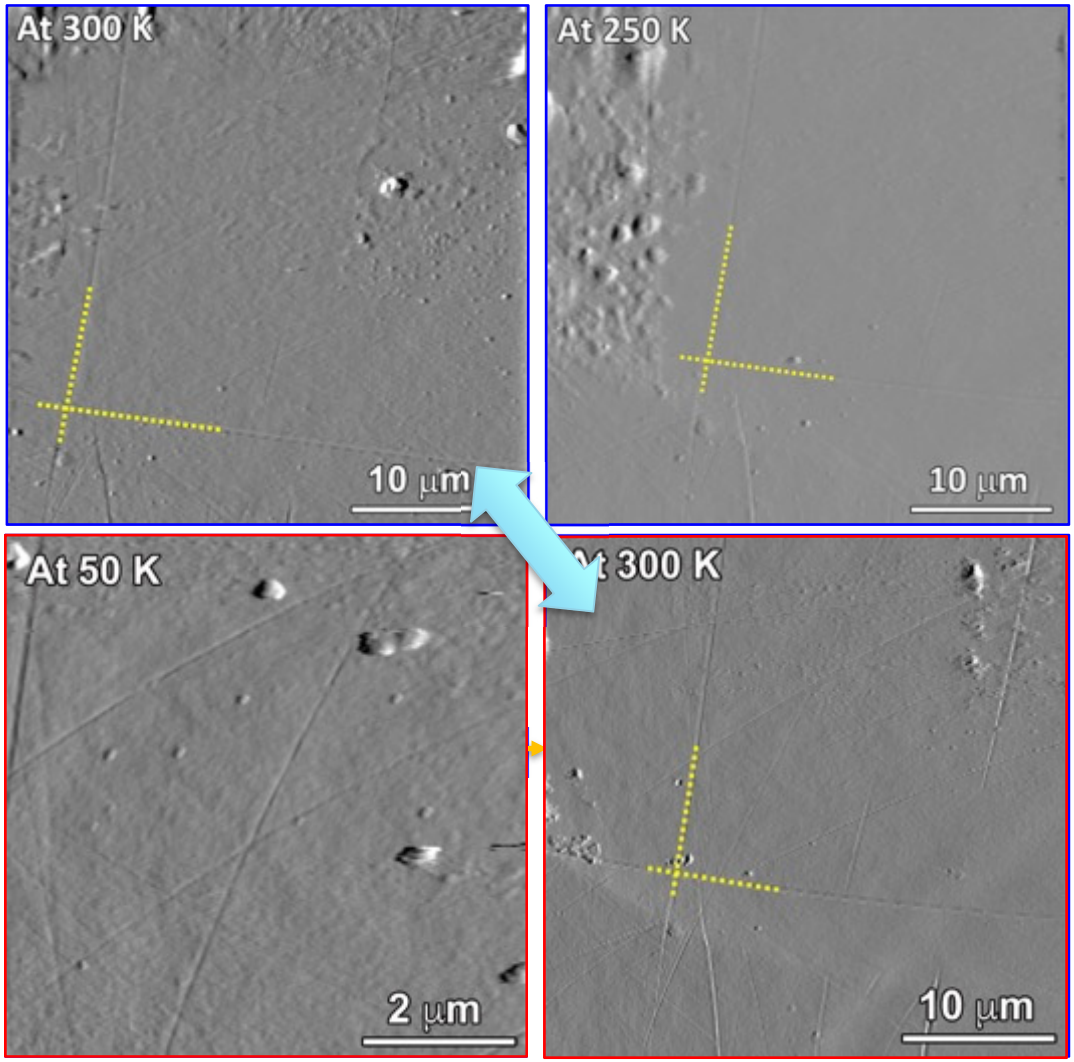
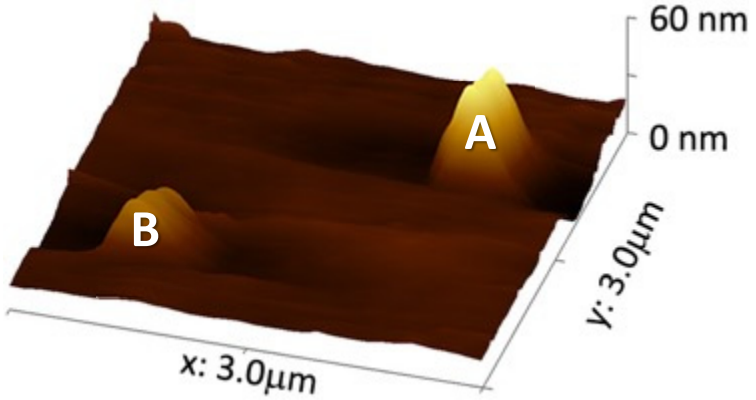
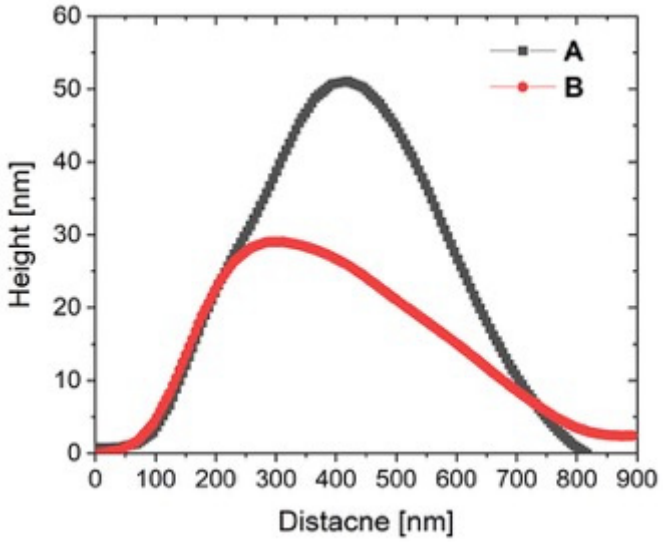


3D surface topology

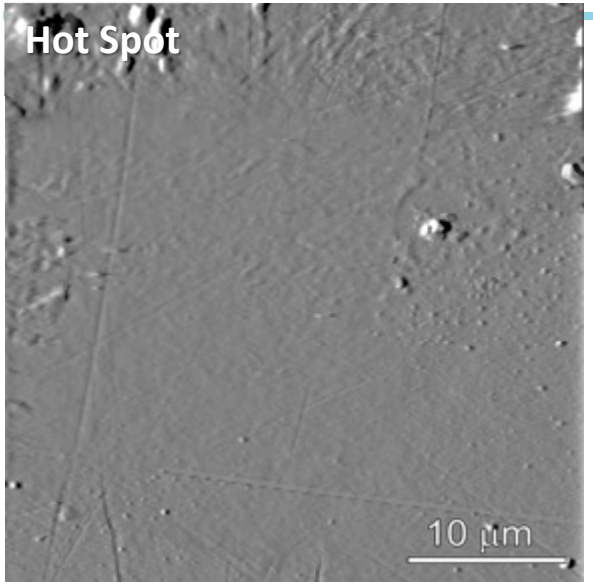


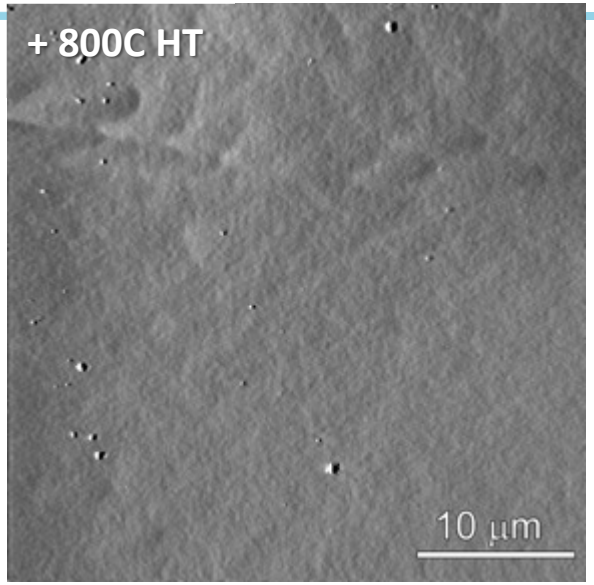
Height Profile across NbH Phase

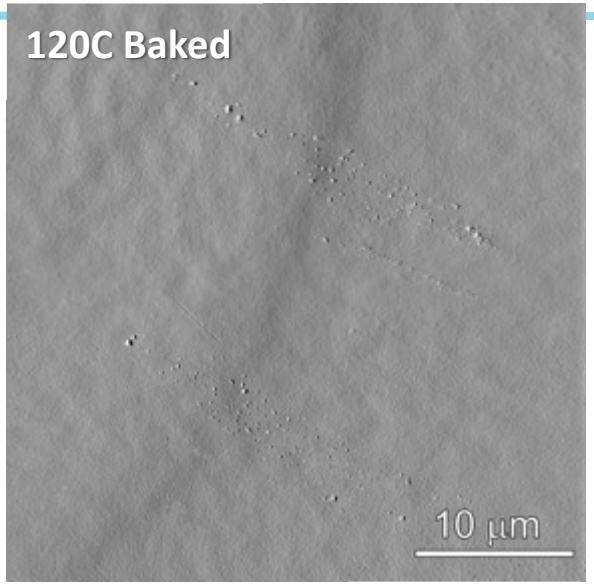


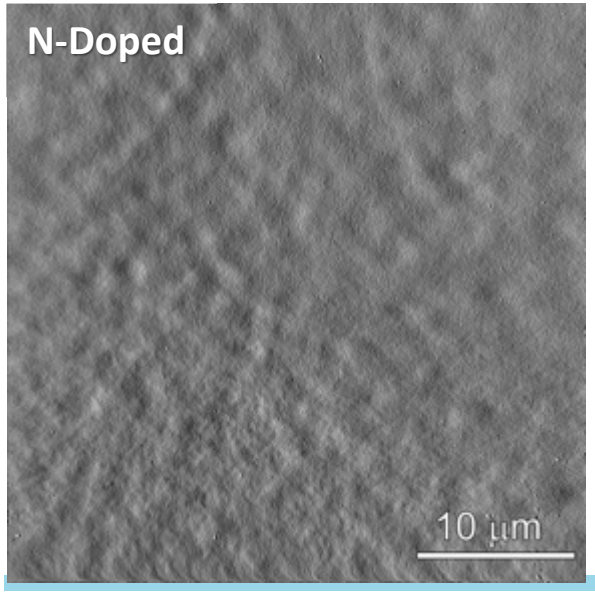
Fermilab

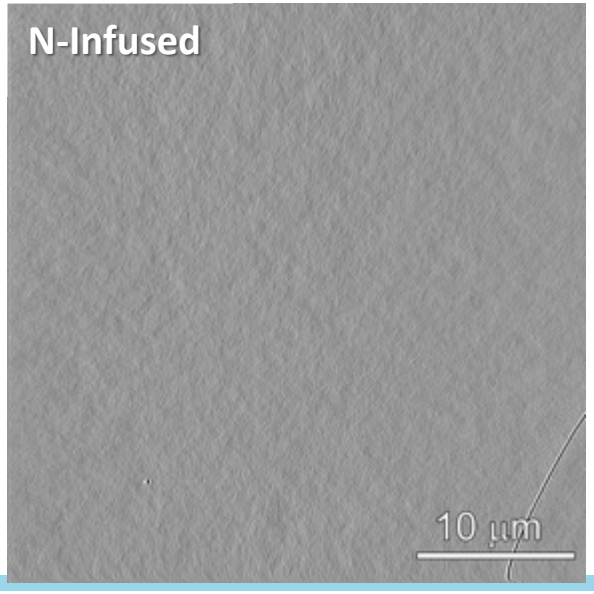
300K Surface features on cavity cut-out samples

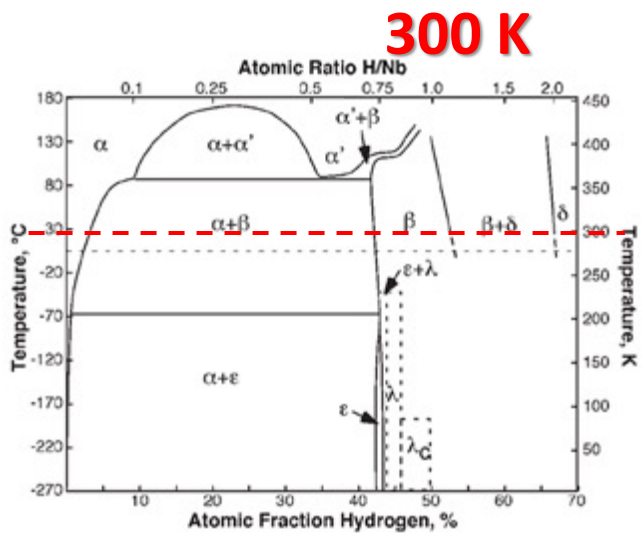






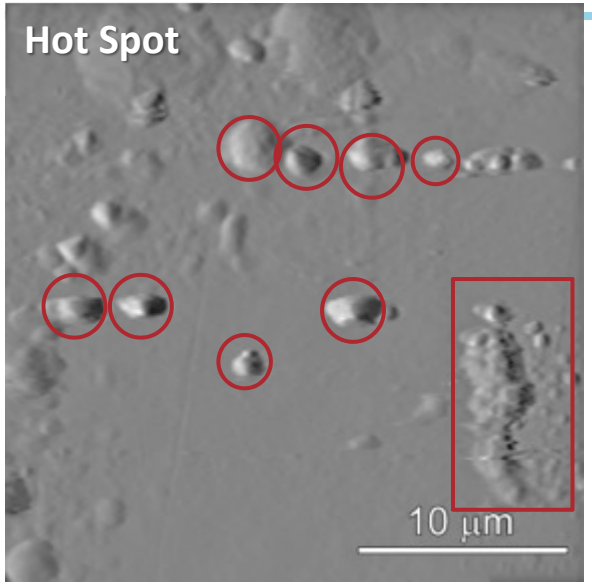


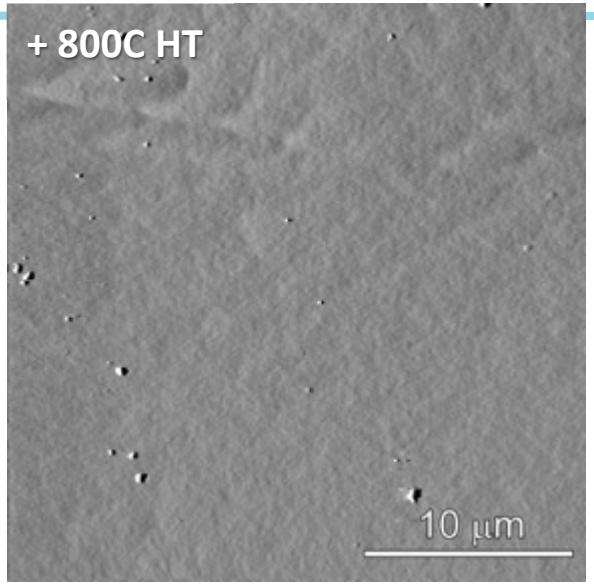


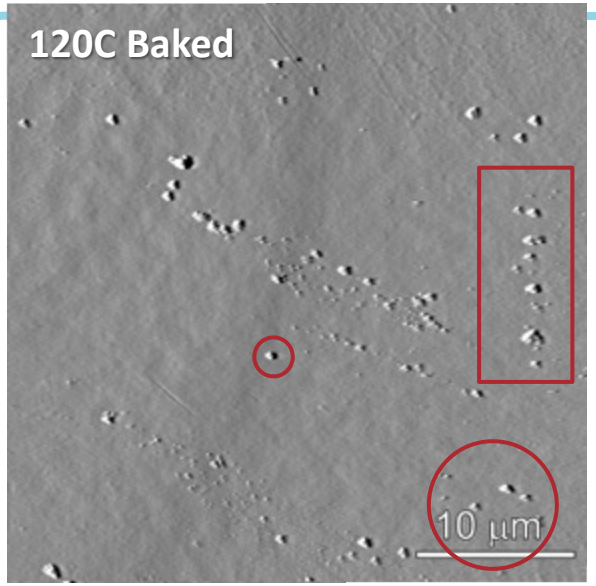


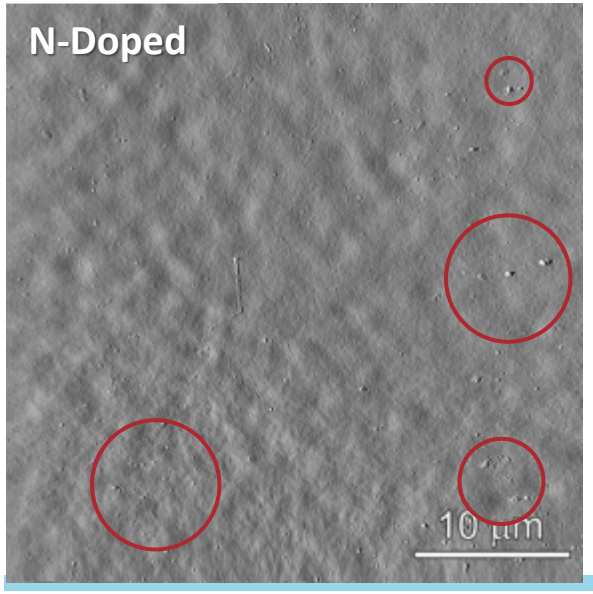
Fermilab

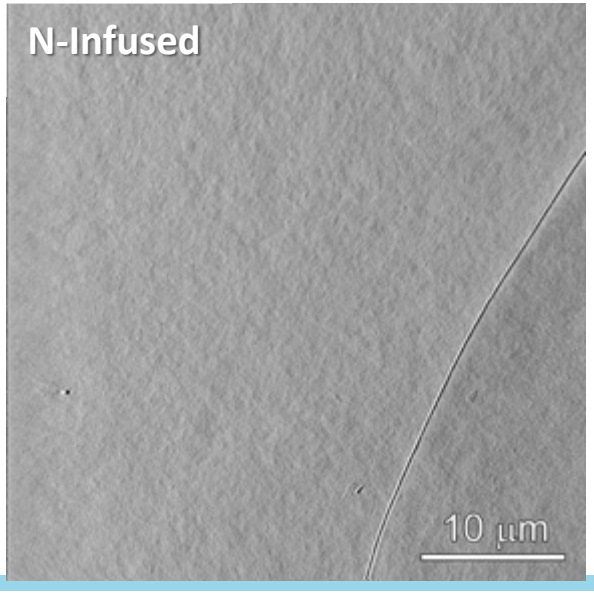
300-200K Surface features on cavity cut-out samples



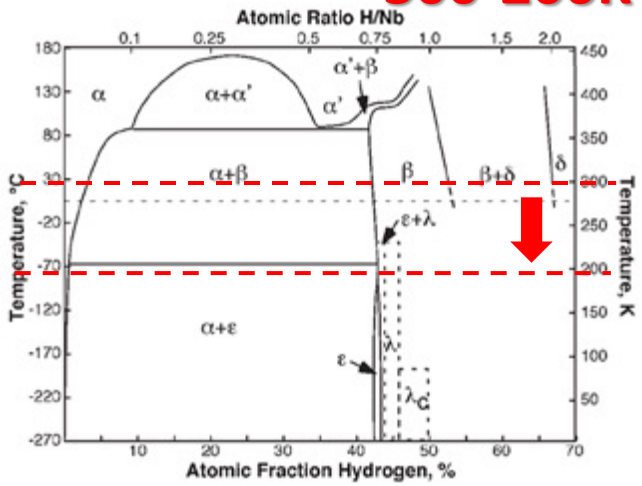






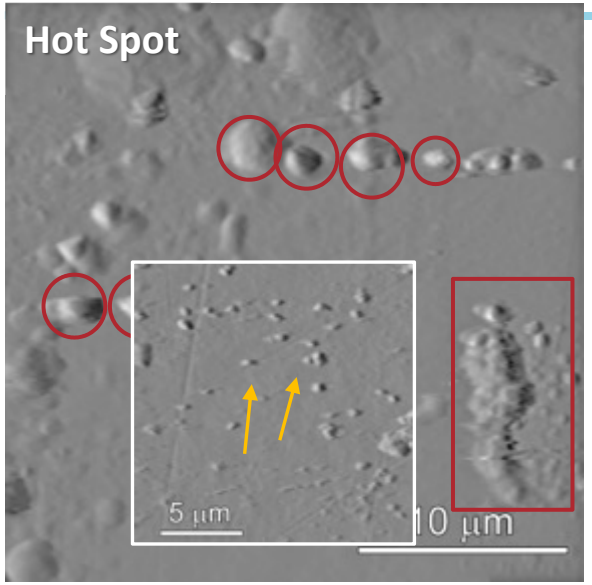


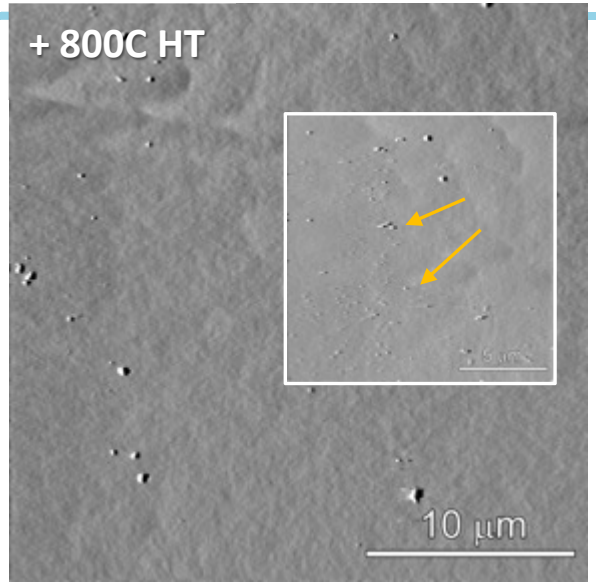
300-200K

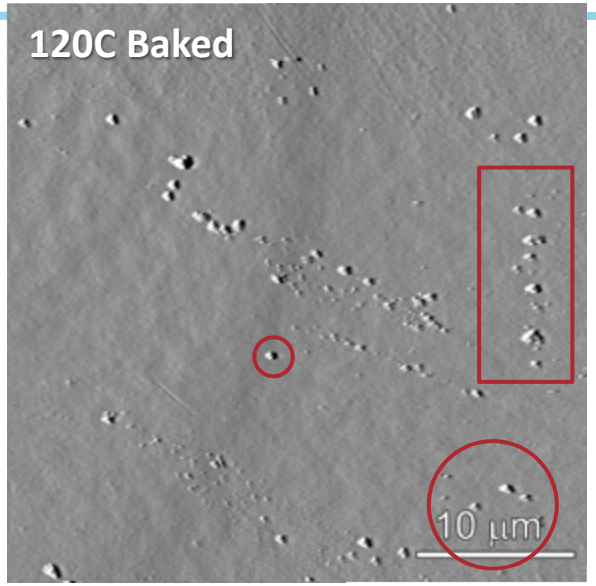


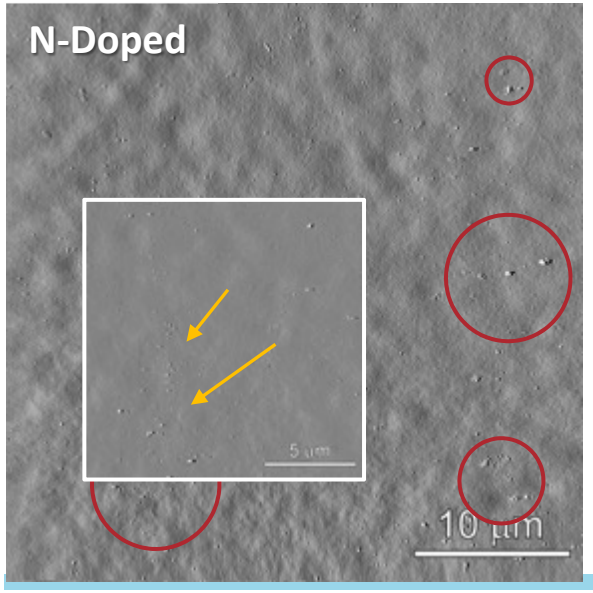
Fermilab

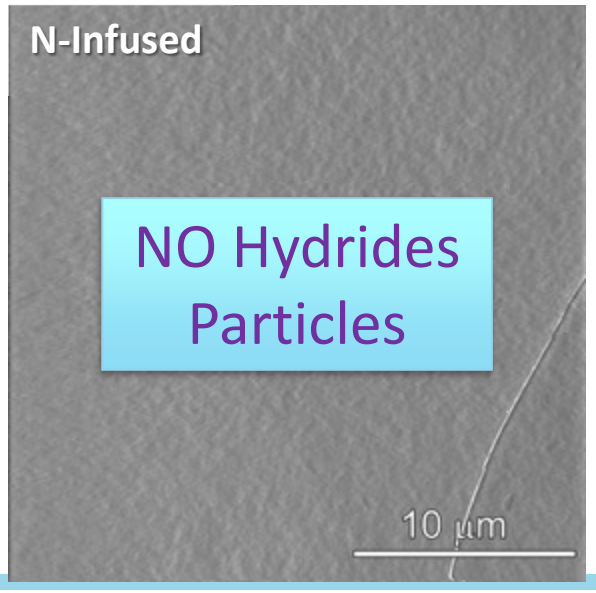
200-100K Surface features on cavity cut-out samples



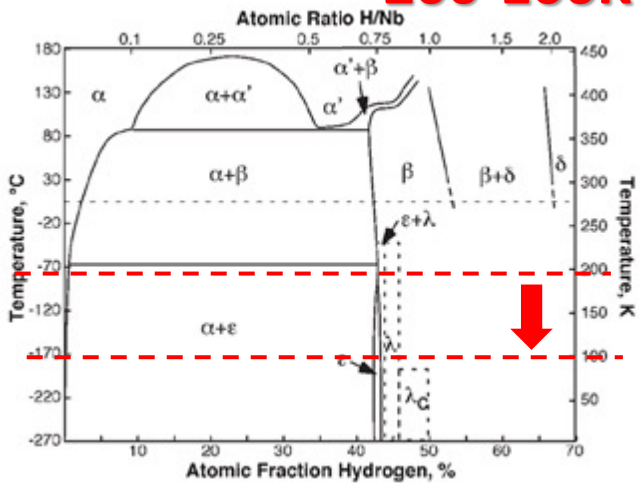








200-100K



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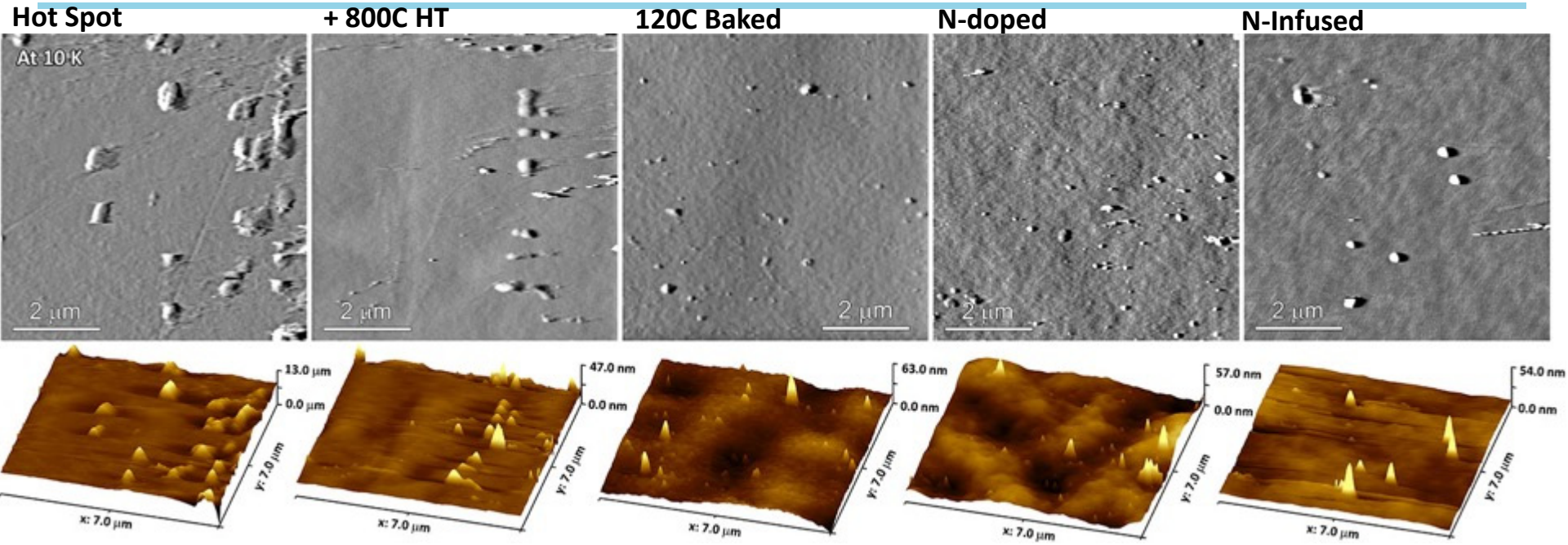
At 10K

Surface features on cavity cut-out samples



	No. of NbH precipitations	Total projected area* (abs. [µm²])	Avg. Height [nm]	Avg. Diameter [nm]	Total NbH volume [10^{-3} × µm³])†
Hot Spot	59	3.58	30.0	687.3	337.7
800C HTed	36	0.99	6.12	397.3	22.3
120C Baked	24	0.46	20.1	305.1	14.2
N-Doped	40	0.56	17.9	272.1	**13.3**
N-Infused	16	0.58	32.1	496.4	**12.8**

H trapping by N

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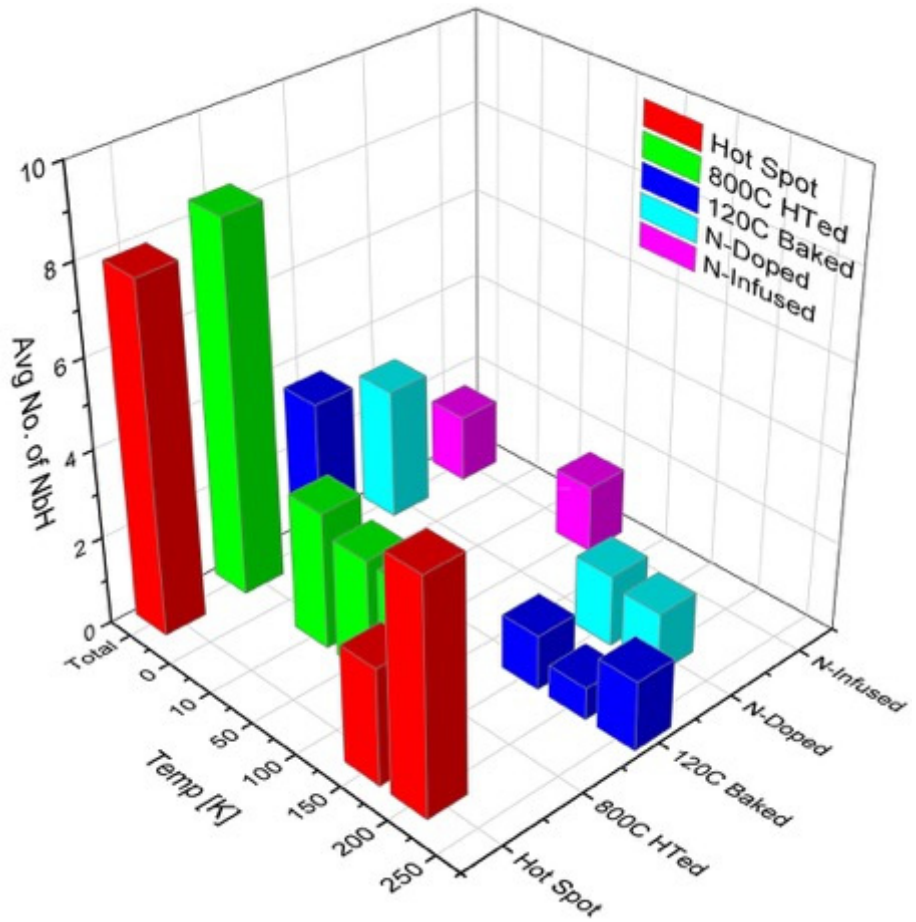
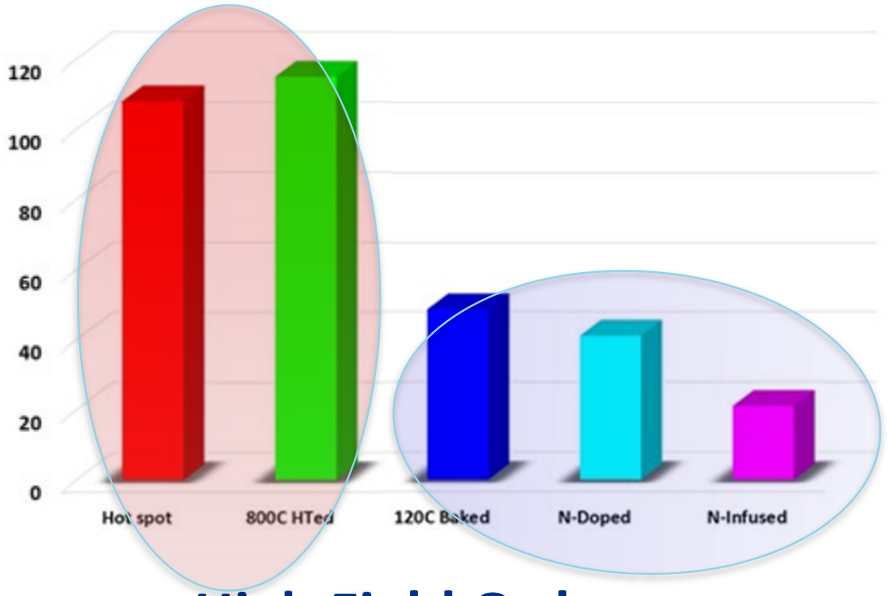
Statistical comparison of NbH precipitation morphology

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



Total NbH phases during cooling to 10K



High Field Q slope

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

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First observation of NbH phase segregation on SRF cavity surface at or above room temp

on TEAES004 120°C baked cut-out + post-HPR

1. 300K →380K, then 380K → 250K
   - At 380K: “chickenpox-shape” appeared
   - Below 280K: “worm-shape” appeared
2. 250K → at room temp (300K): stayed 9 days 17 hours on the bench of PPMS room with double-wrapped with micro-cloth and Al foil
   - “worm-shape” from 280K still exited
3. 300K → 380K → 5K
   - At 320K: “worm-shape” disappeared

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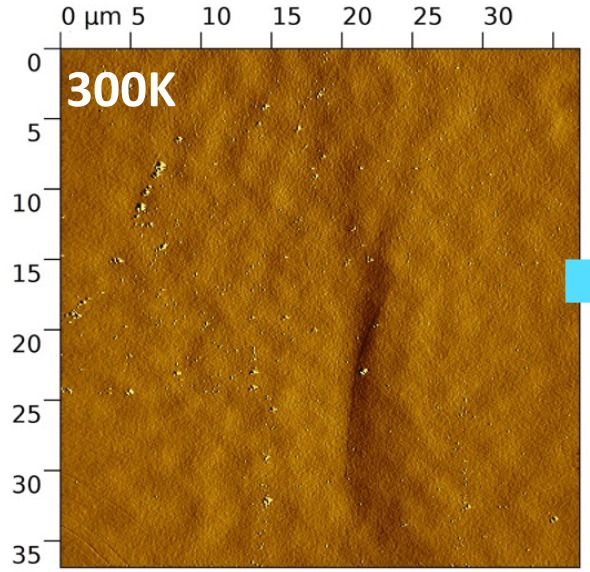
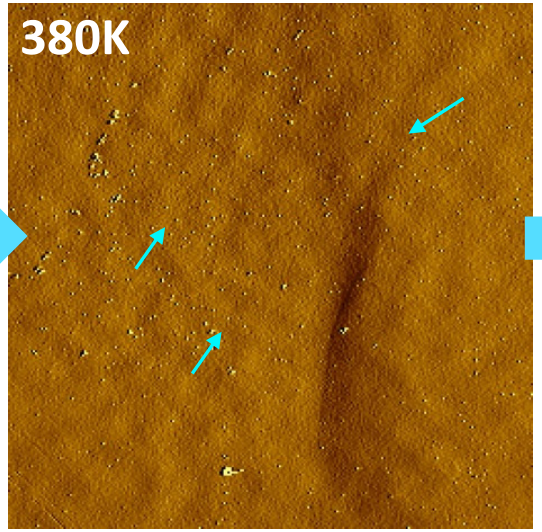
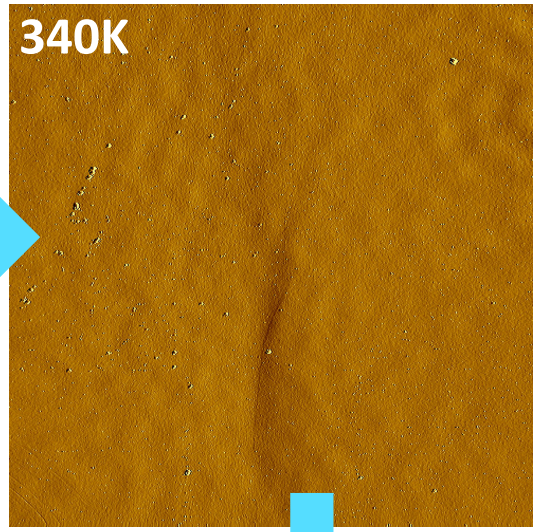
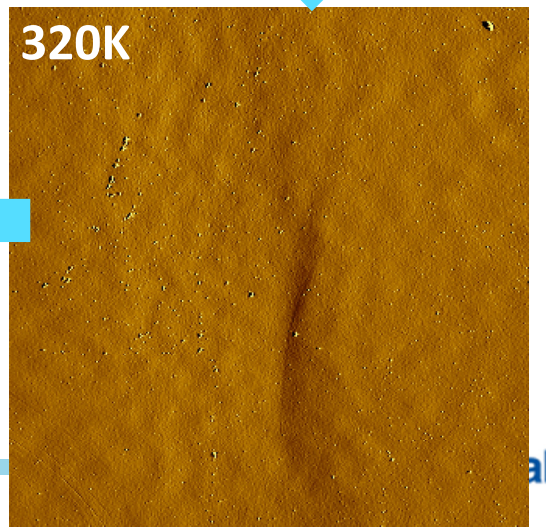
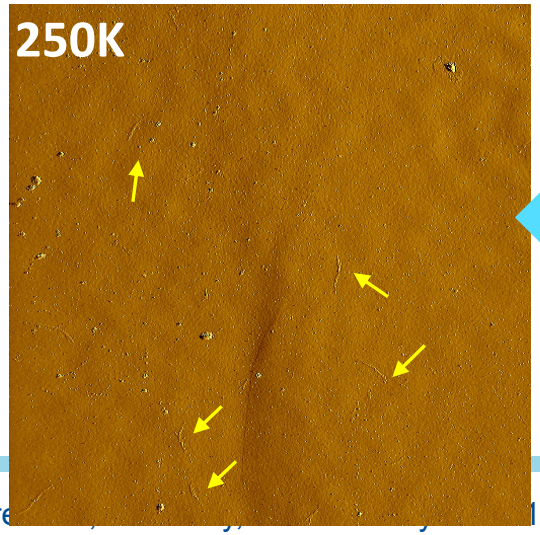
Series of AFM images on warming and cooling from 300K, to 380K and 5K

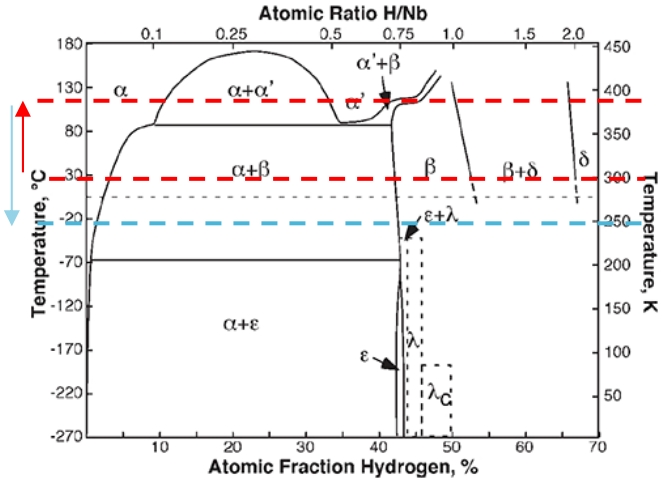


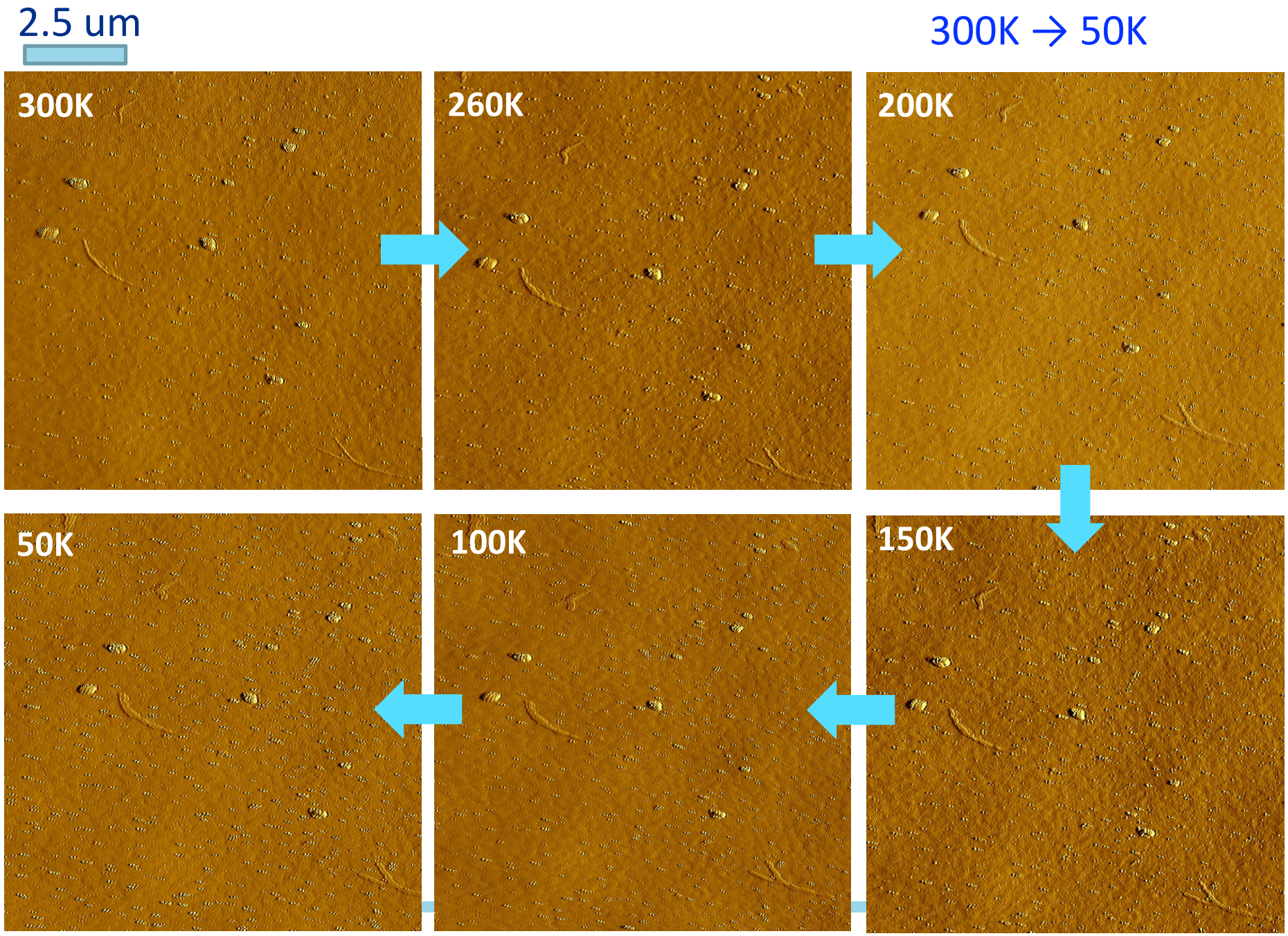


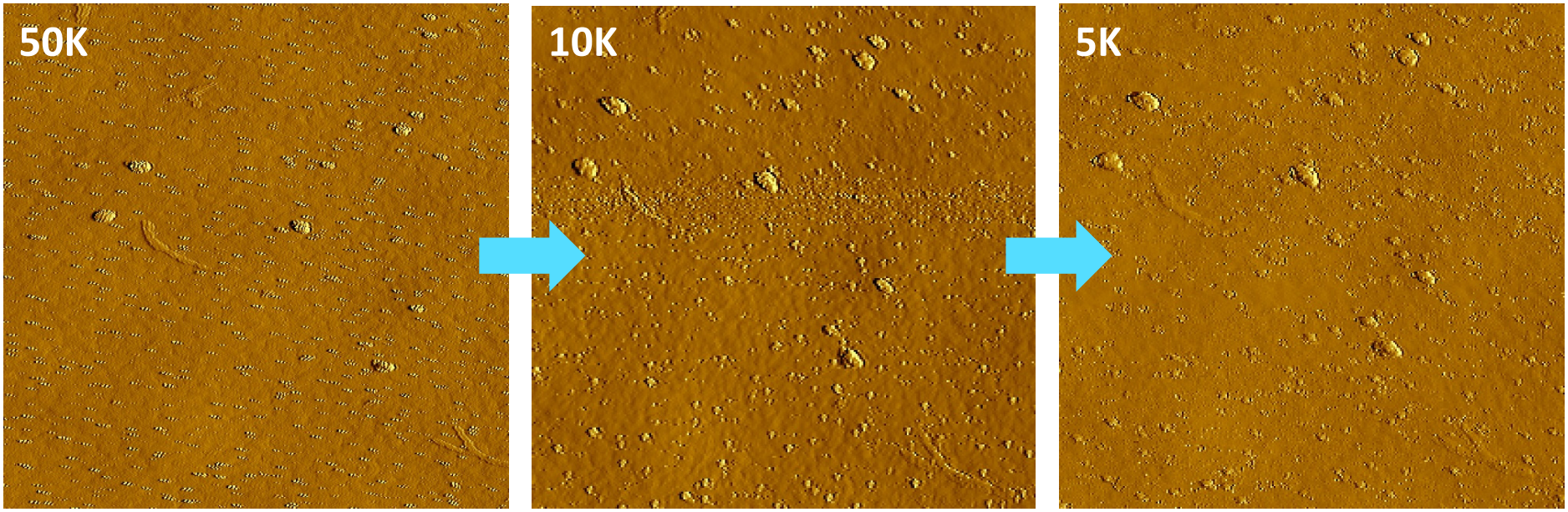








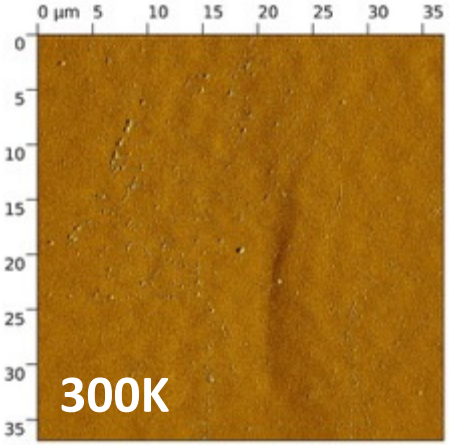

2.5 um
300K → 50K
300K
260K
200K
150K
100K
50K



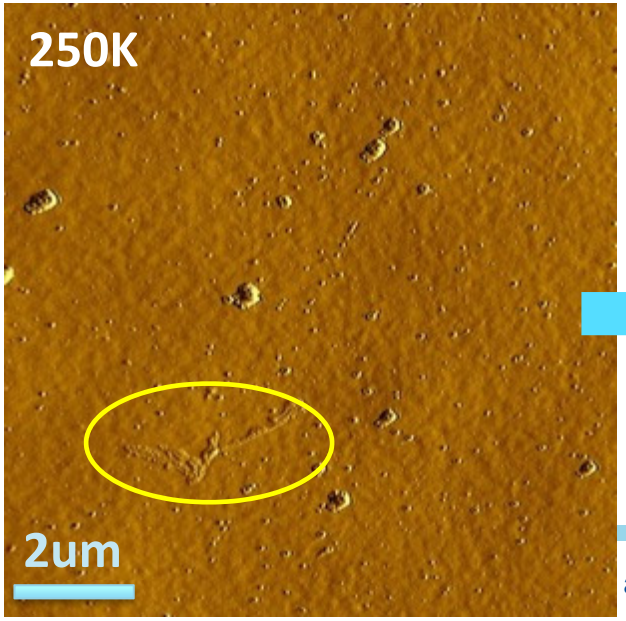
Surface features appeared above 200K are still sitting on the surface even at 5K

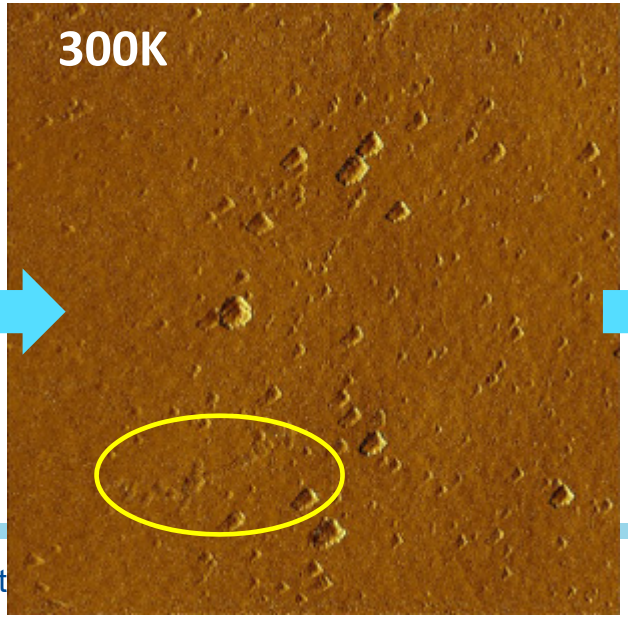
First observation of NbH phase (α, α', β) segregation at 300K

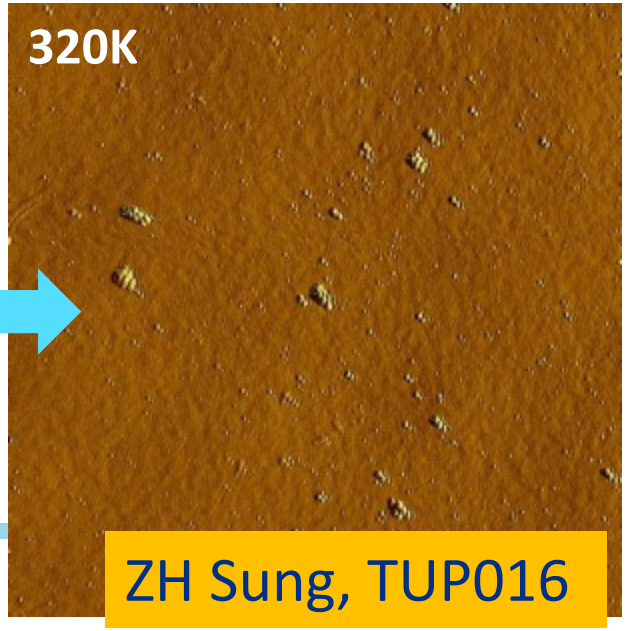
TEAES004 120°C baked cut-out + post-HPR



Cooling/Warming sequence: 300K -> 250K -> 300K -> 320K







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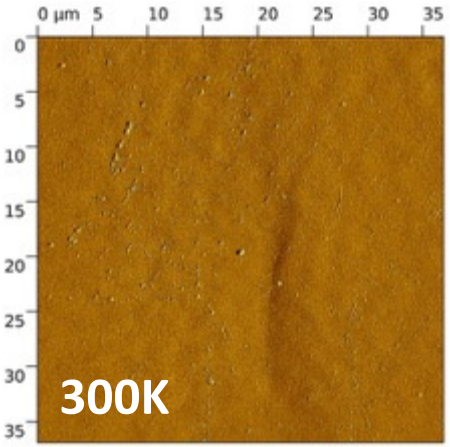
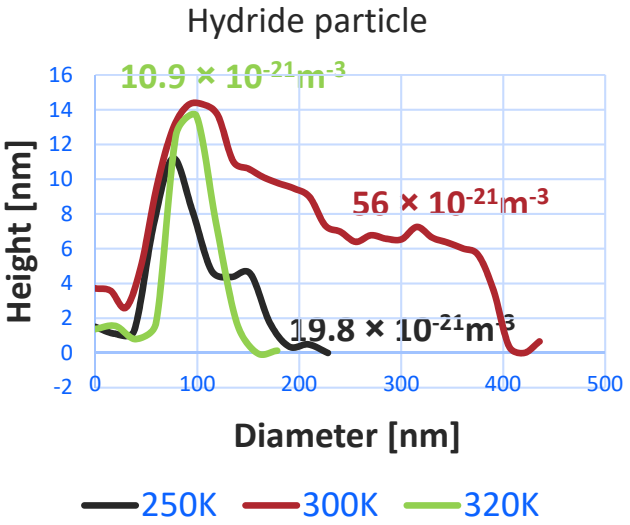
First observation of NbH phase (α, α', β) segregation at 300K

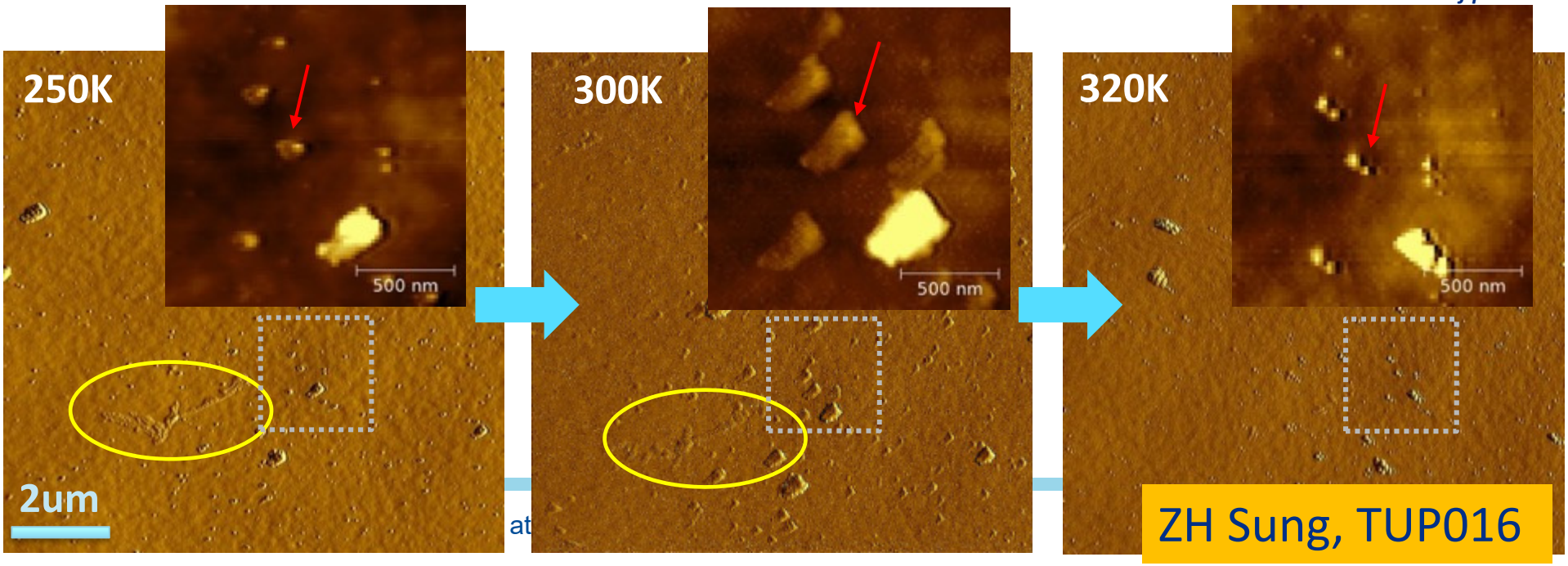
TEAES004 120°C baked cut-out + post-HPR



Cooling/Warming sequence: 300K -> 250K -> 300K -> 320K

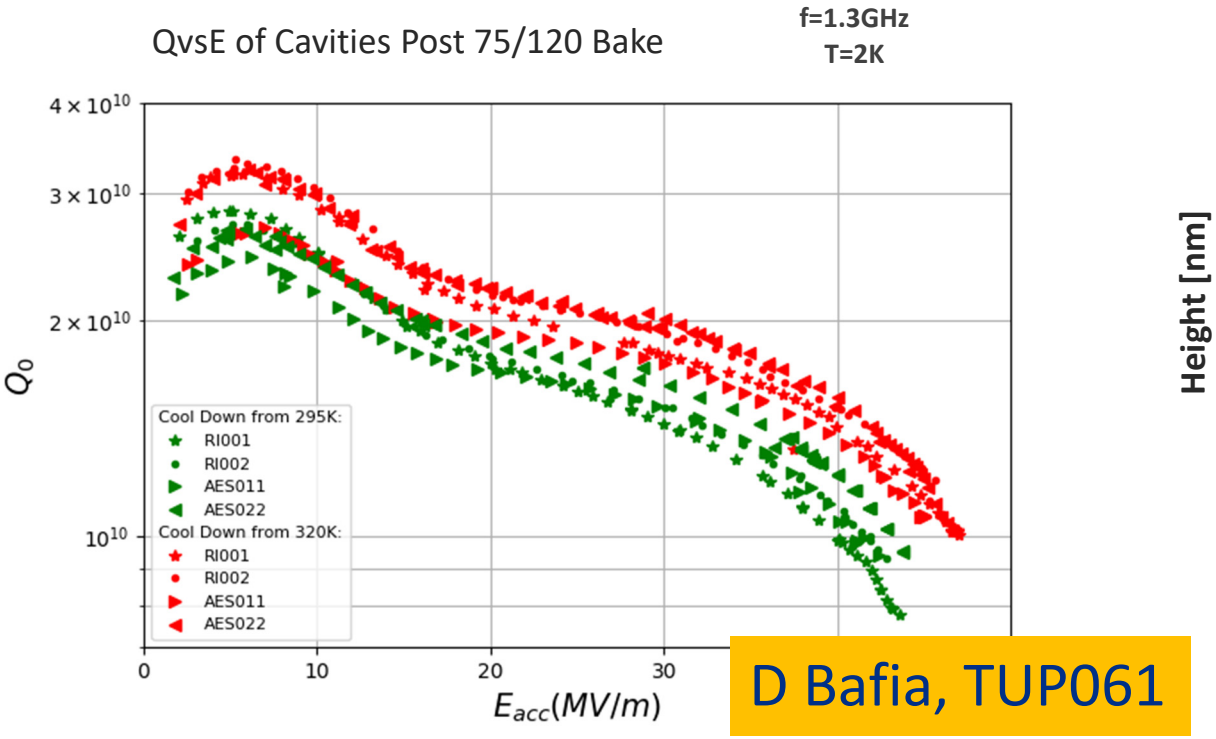


** Number = Volume of particle*

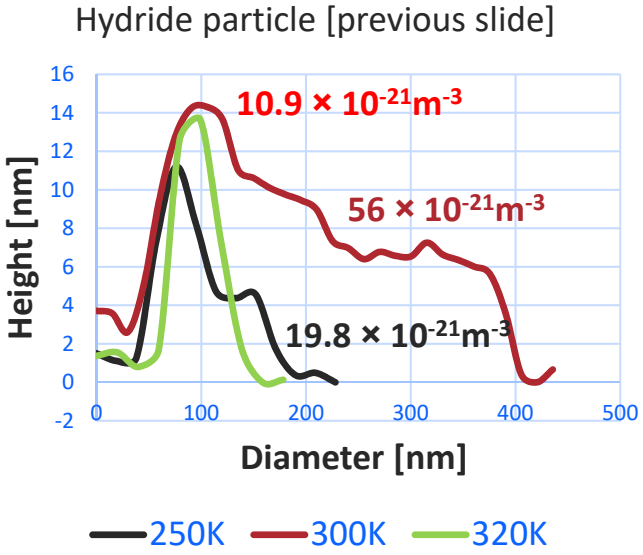


ZH Sung, TUP016

Comparison of 75/120 Bake Cavities Cooled From 295K vs 320K



D Bafia, TUP061



**** Number = Volume of particle***

Courtesy of D Bafia, TTC, Vancouver, 2019

- Cooling cavities from 320K shows consistently higher quench field and Q_0 at high fields for cavities post 75/120 bake when compared to cooling from 295K.
- In-situ 320K may be dissociating non-superconducting niobium hydrides

Summary

- Using cryo-AFM, we have successfully observed nm-size NbH precipitates, which are highly related to HFQS on SRF Nb cavities.
- nm-size Nb hydride precipitates on SRF Nb cavity surface underneath the oxide layer. Its size, population, and saturation temperature are the key parameters for HFQS
- Confirmed that nitrogen are favorable for trapping “hydrogen”, leading to suppression of nm-NbH segregation.
- Nb hydride could be also saturated at 300K, same as the scenario over the cryogenic range. But detailed spectroscopy is required to clarify this issue.
- We learned nano-NbH related cavity performance degradation, but the solution to annihilate nano-NbH phase requires further works.

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