



---

Managed by Fermi Research Alliance, LLC for the U.S. Department of Energy Office of Science

---

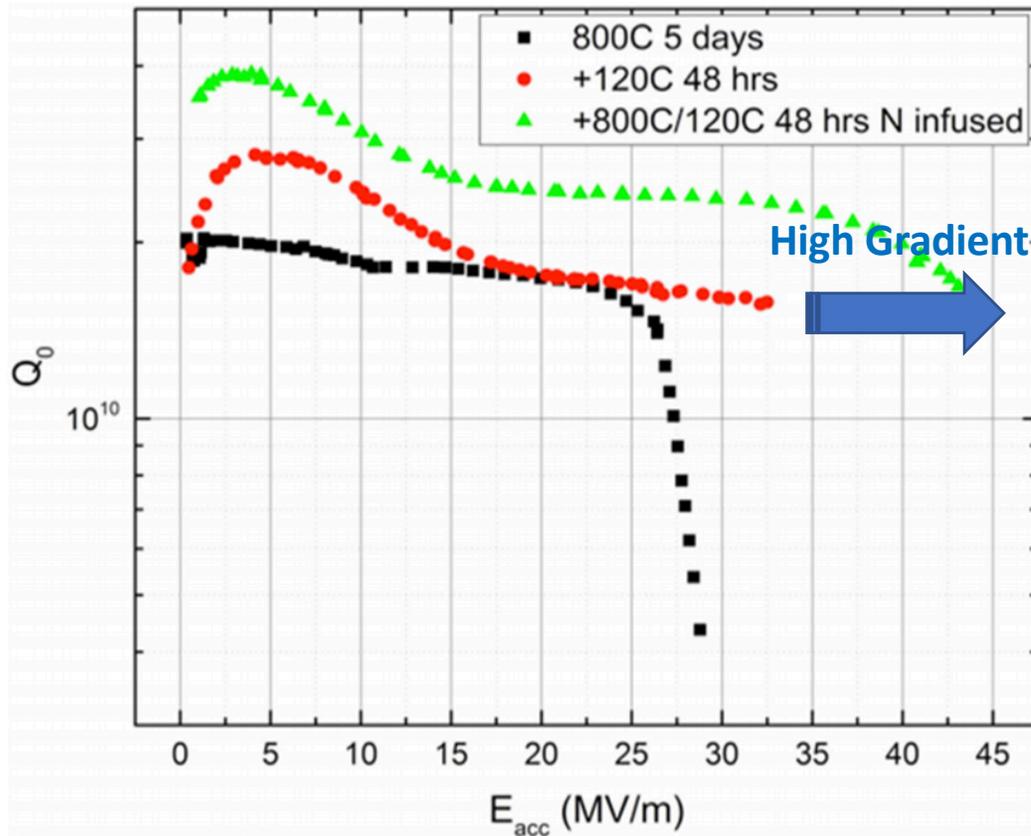
## **Latest material analysis on state of the art treatments (including doping and infusion cutouts)**

Zuhawn Sung, Alex Romanenko, Yulia Trenikhina, and Anna Grassellino

TESLA Technology Collaboration Meeting

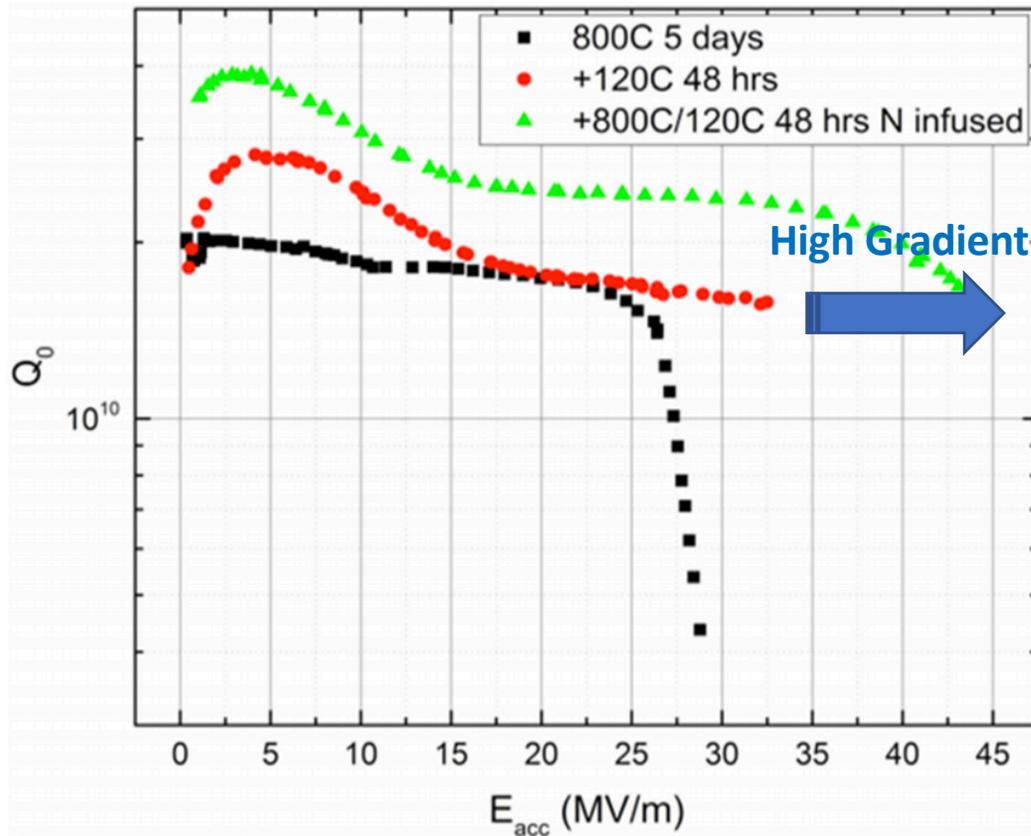
6-9 Feb 2018

# Fundamental material studies to investigate origin of high Q and high gradient



A Grassellino, et al., Technology, "Unprecedented quality factor at accelerating gradients up to 45MV/M in niobium" Supercond. Sci. Technol. 30, 094004, (2017)

# Fundamental material studies to investigate origin of high Q and high gradient



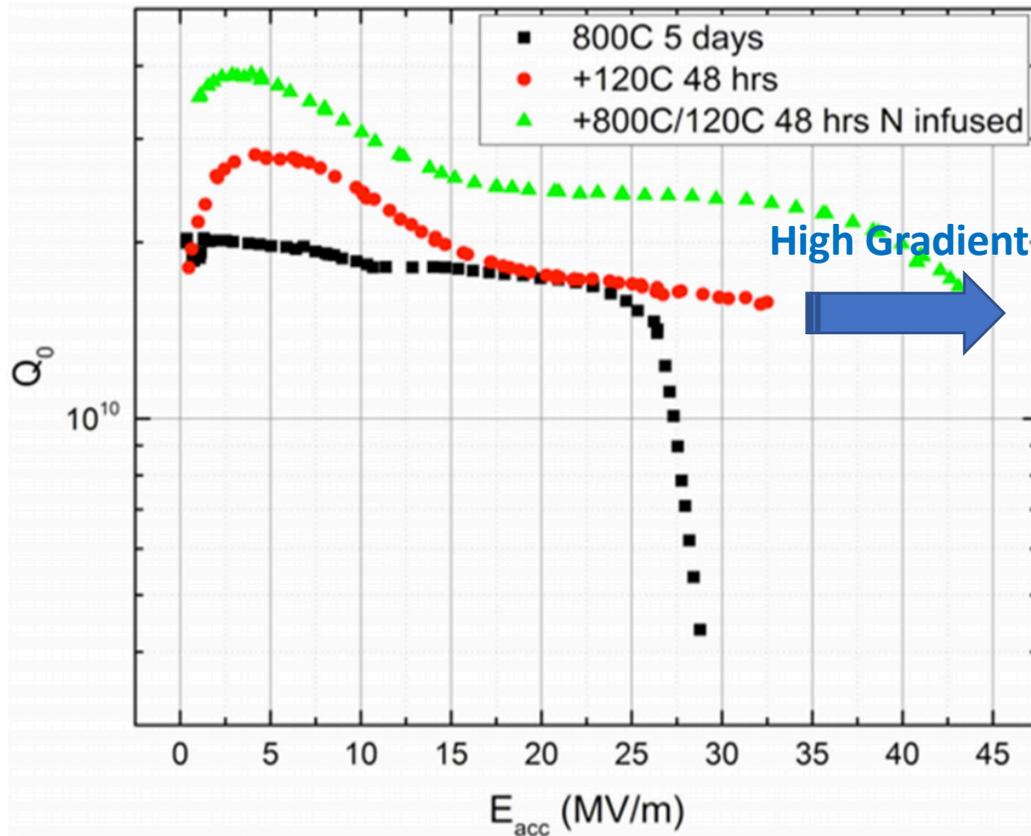
✓ Cryo-Atomic Force Microscopy for  $Nb_{1-x}H_x$  (hydride phase) proximity effect

✓ Cryo-MFM (Magnetic Force Microscopy) for trapped vortices

✓ AC Susceptibility characterization for penetration dynamics of surface magnetic vortices

A Grassellino, et al., Technology, "Unprecedented quality factor at accelerating gradients up to 45MV/M in niobium" Supercond. Sci. Technol. 30, 094004, (2017)

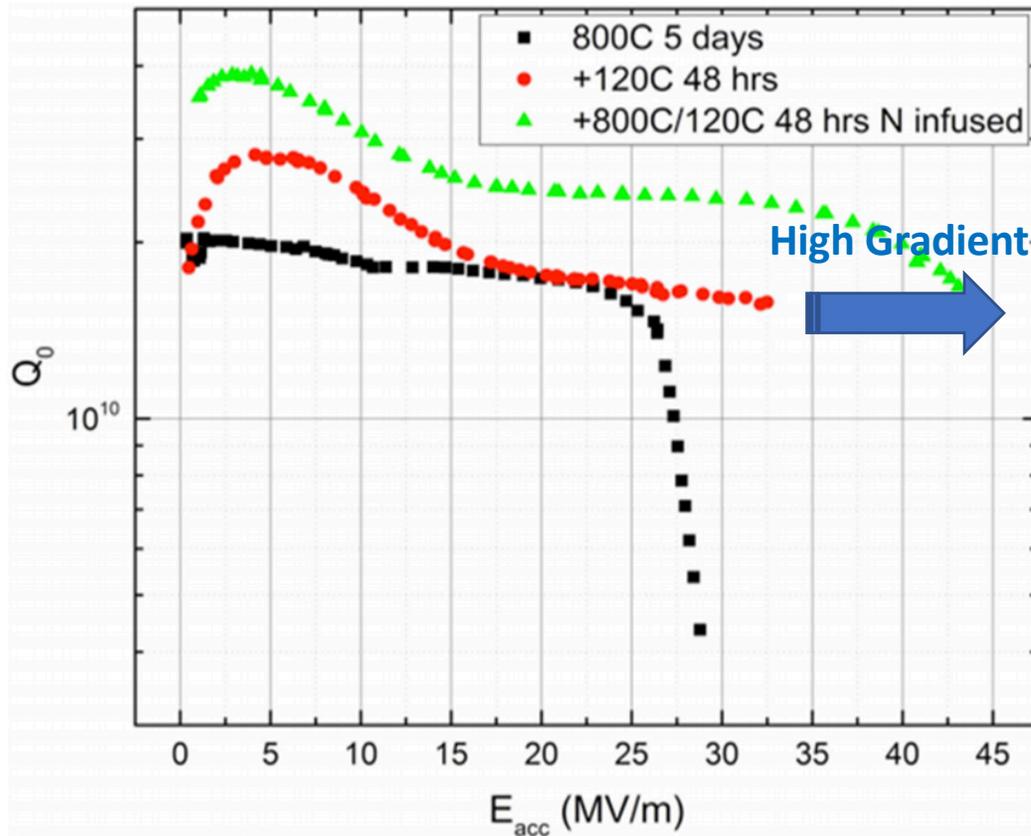
# Fundamental material studies to investigate origin of high Q and high gradient



- ✓ Cryo-Atomic Force Microscopy for  $\text{Nb}_{1-x}\text{H}_x$  (hydride phase) proximity effect
- ✓ Cryo-MFM (Magnetic Force Microscopy) for trapped vortices
- ✓ AC Susceptibility characterization for penetration dynamics of surface magnetic vortices

A Grassellino, et al., *Technology*, "Unprecedented quality factor at accelerating gradients up to 45MV/M in niobium" *Supercond. Sci. Technol.* 30, 094004, (2017)

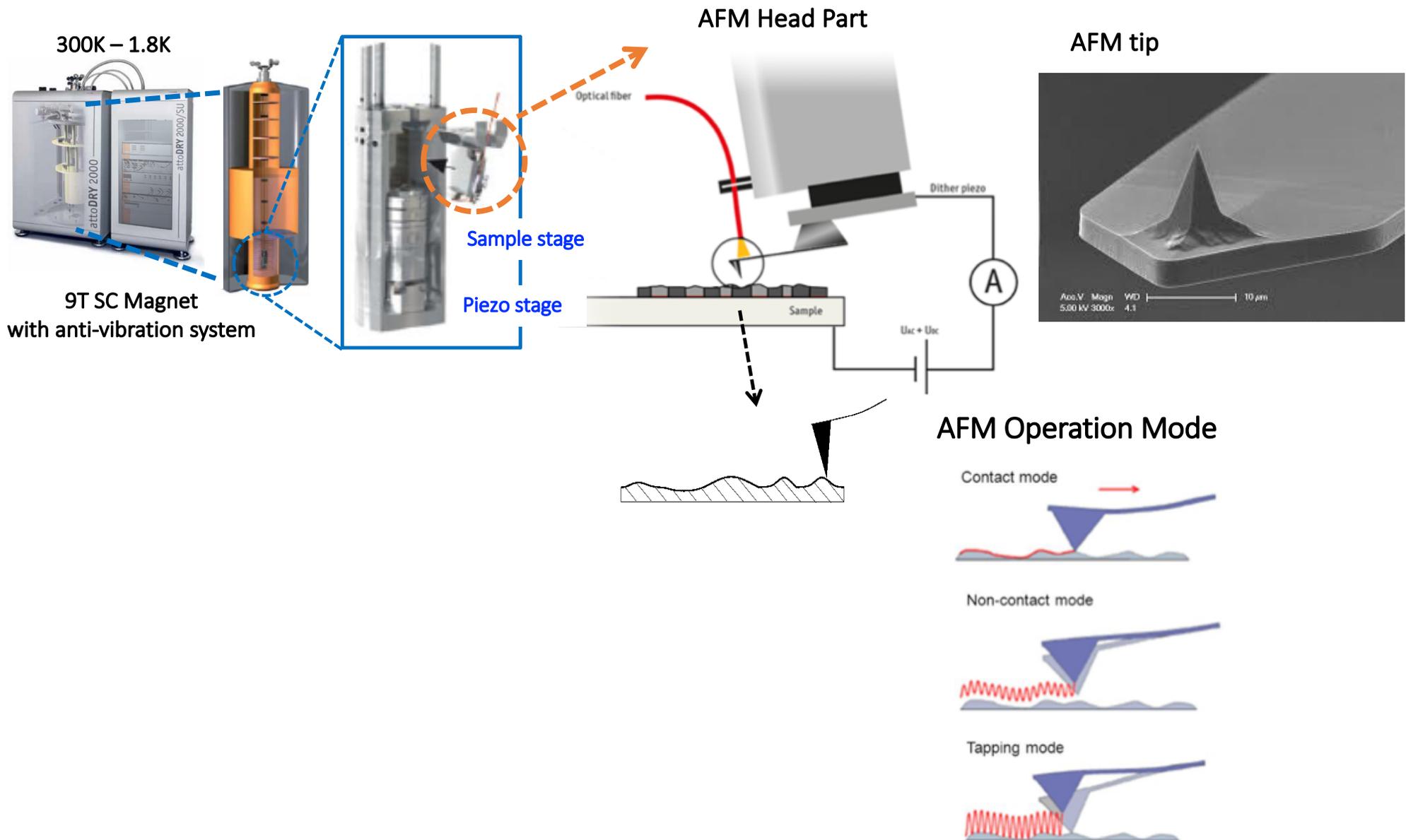
# Fundamental material studies to investigate origin of high Q and high gradient



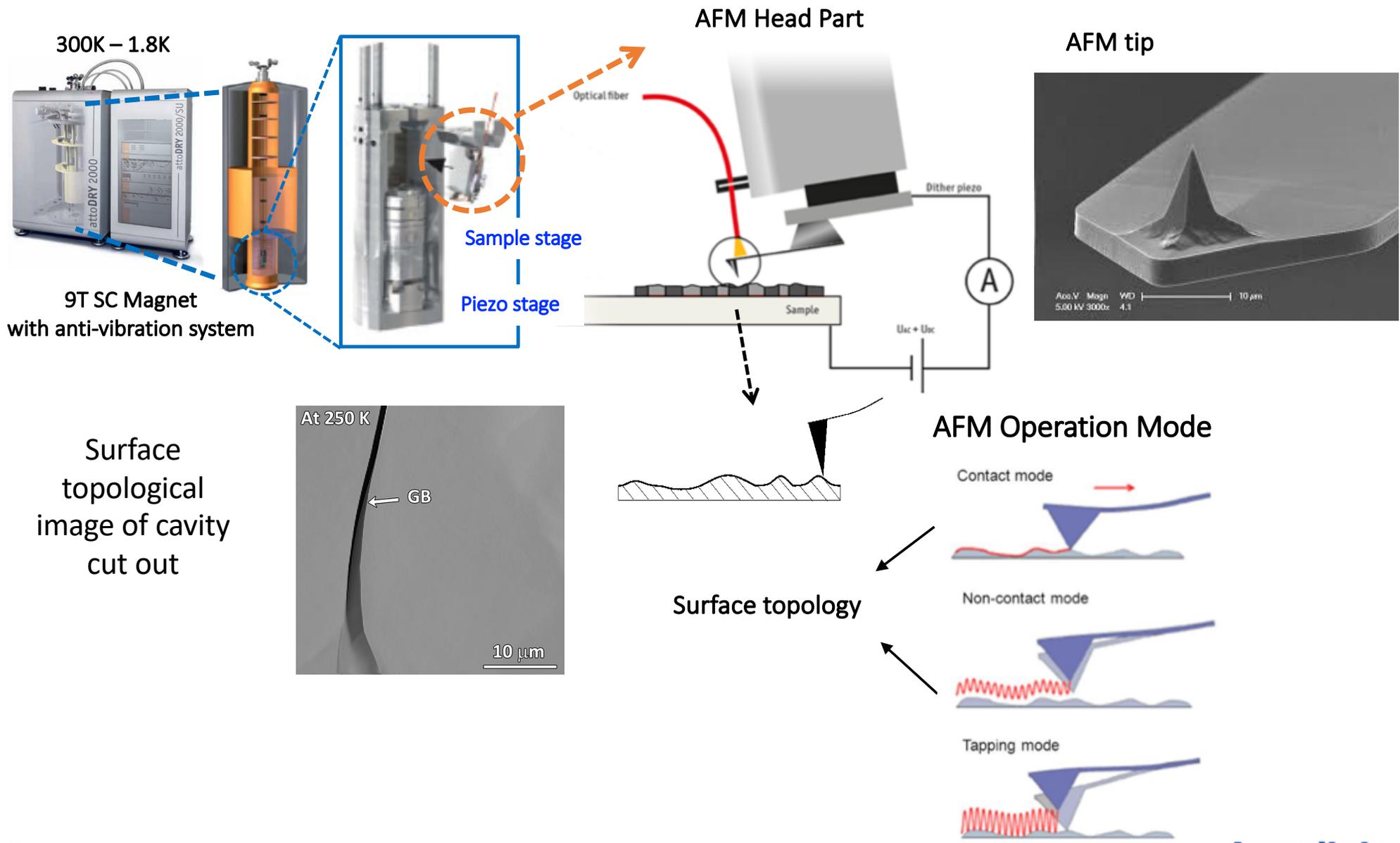
- ✓ Cryo-Atomic Force Microscopy for  $Nb_{1-x}H_x$  (hydride phase) proximity effect
- ✓ Cryo-MFM (Magnetic Force Microscopy) for trapped vortices
- ✓ AC Susceptibility characterization for penetration dynamics of surface magnetic vortices

A Grassellino, et al., Technology, "Unprecedented quality factor at accelerating gradients up to 45MV/M in niobium" Supercond. Sci. Technol. 30, 094004, (2017)

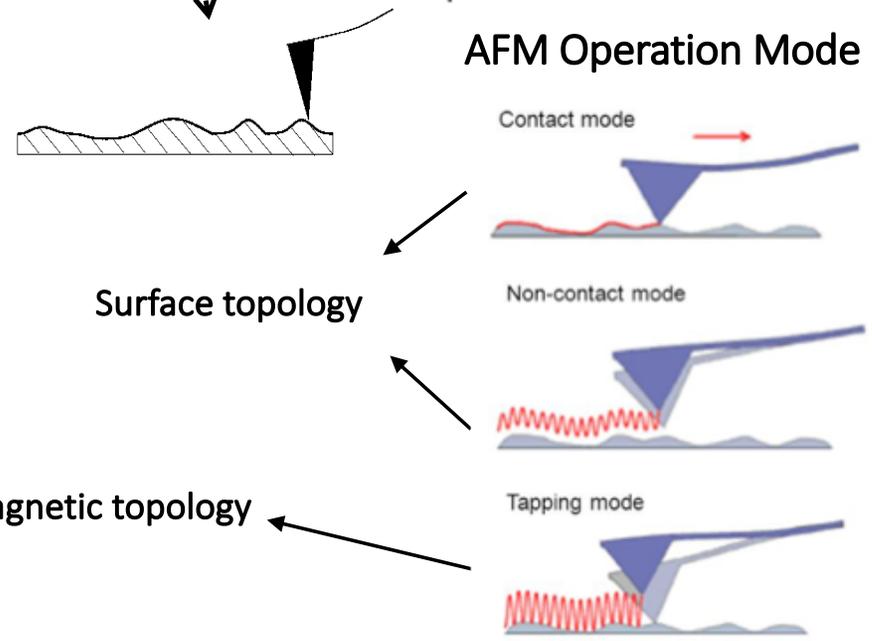
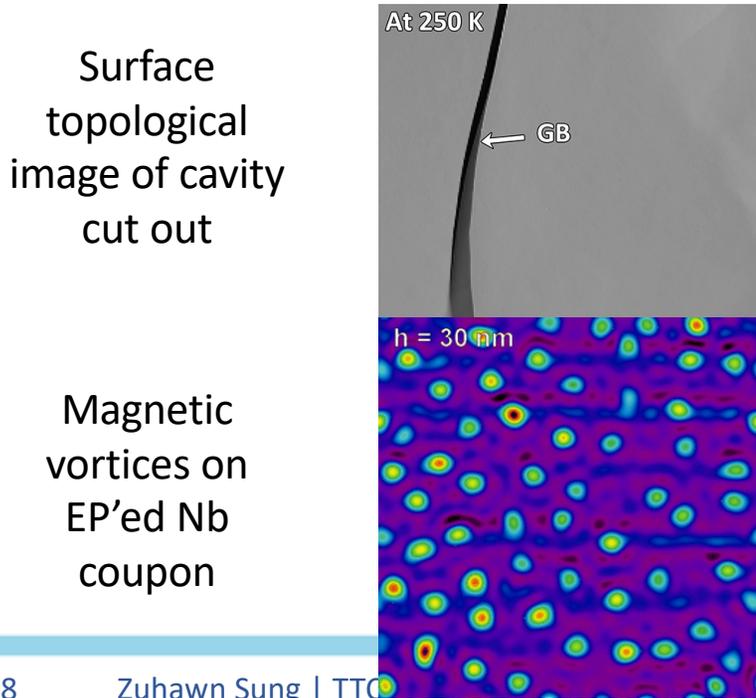
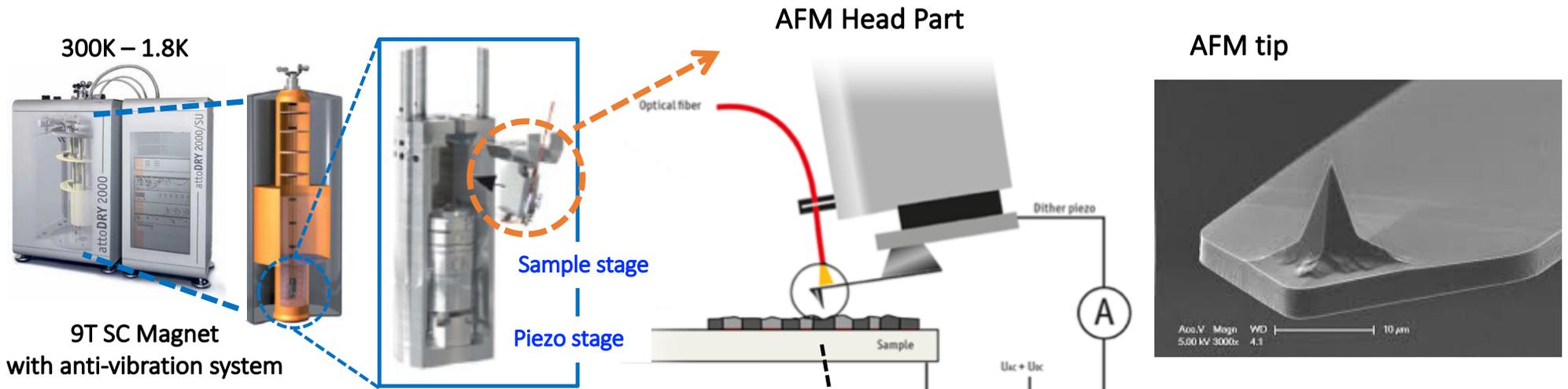
# Cryogenic Atomic Force Microscope (AFM) + Magnetic Force Microscope (MFM) in 9 Tesla Superconducting Magnet @FNAL\_MSL



# Cryogenic Atomic Force Microscope (AFM) + Magnetic Force Microscope (MFM) in 9 Tesla Superconducting Magnet @FNAL\_MSL

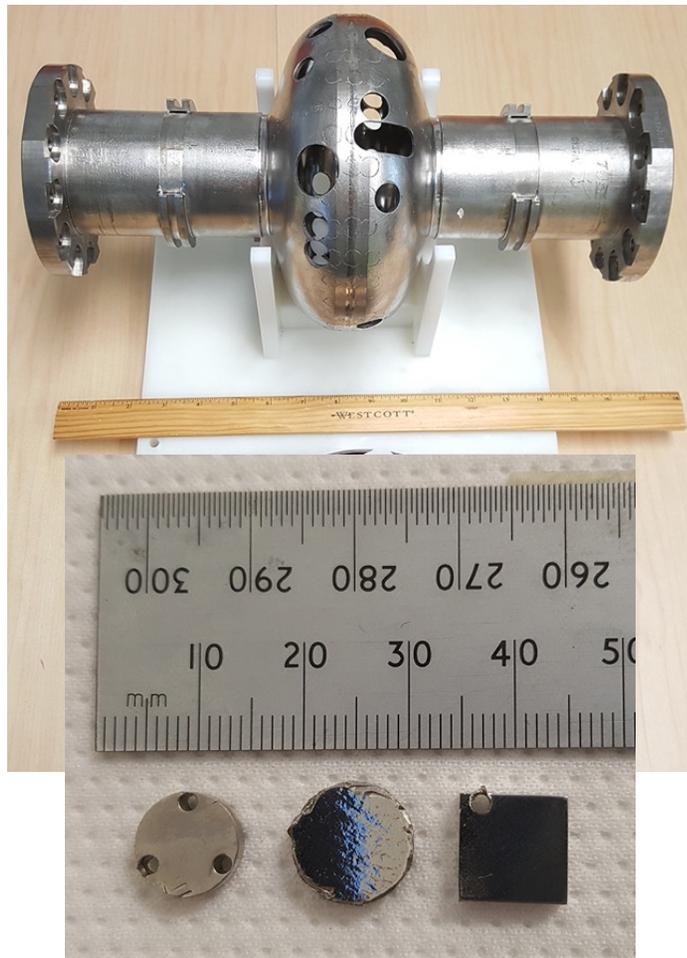


# Cryogenic Atomic Force Microscope (AFM) + Magnetic Force Microscope (MFM) in 9 Tesla Superconducting Magnet @FNAL\_MSL



# Samples for Cryo AFM and MFM Study

## 1.3 GHz SRF Nb cavity



## Descriptions of Samples studied

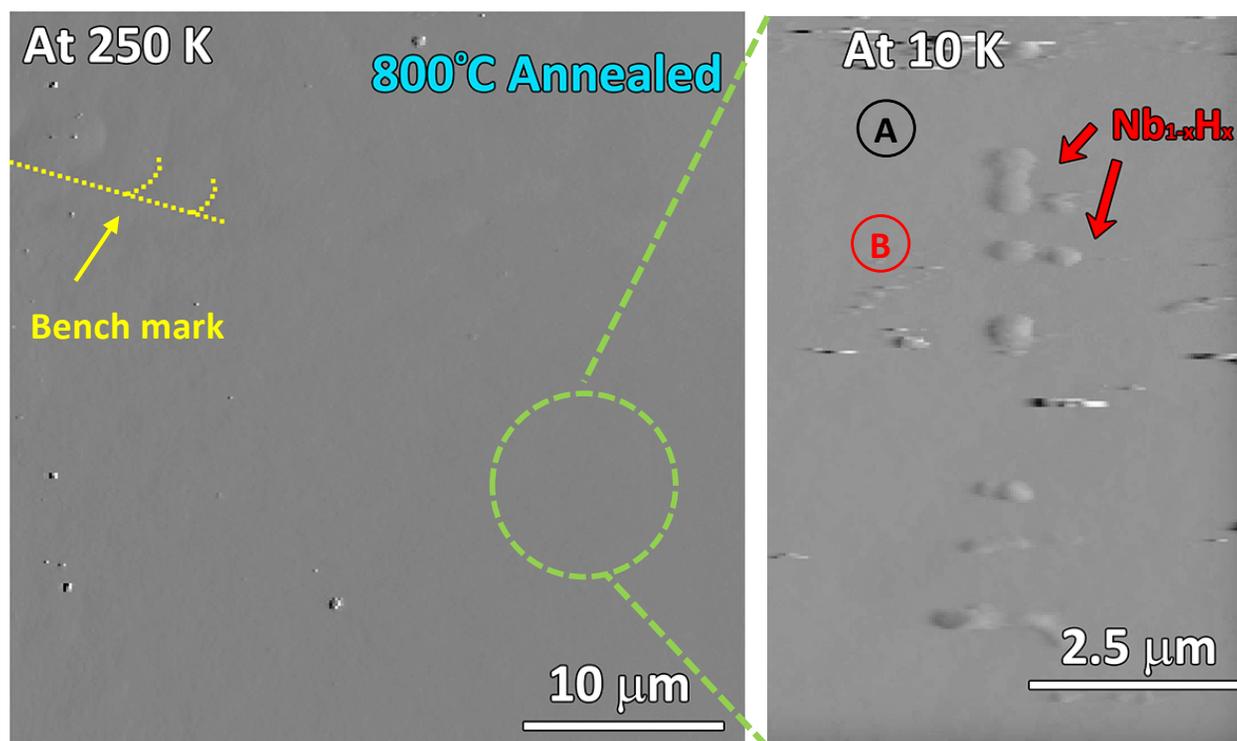
No.	Sample ID	Features
1	Hot Spot	EP'ed Cavity (non-degaussed cavity)
2	800°C baked hot spot	800°C HT + 20 $\mu\text{m}$ BCP applied on hot spot cut out (#1)
3	120°C baked	EP + 120°C baked cavity
4	N-doped	N-doped cavity @800°C 25mTorr (2/6)

---

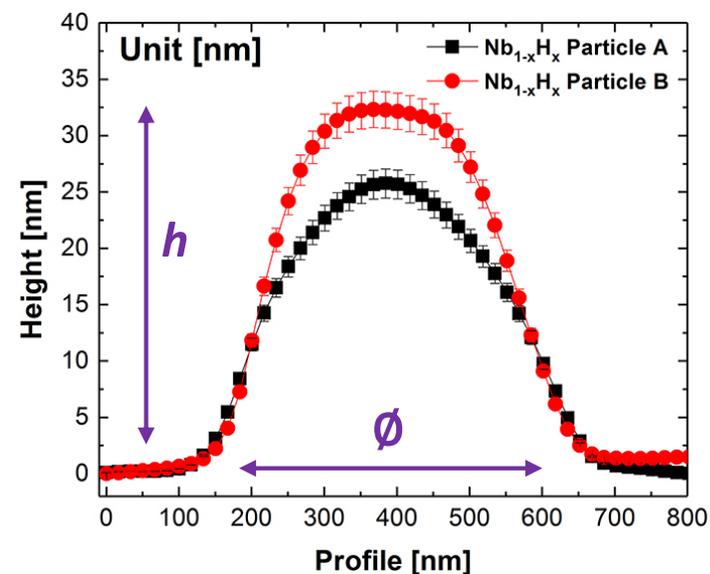
# *Atomic Force Microscopy*

# First observation of sub- $\mu\text{m}$ NbH phase at cryo-state

After applied 800°C + 20  $\mu\text{m}$  BCP to a hot spot cut-out from TE1ACC003 EP'ed cavity (non-degaussed), in order to clarify the effect of 800°C on H annihilation

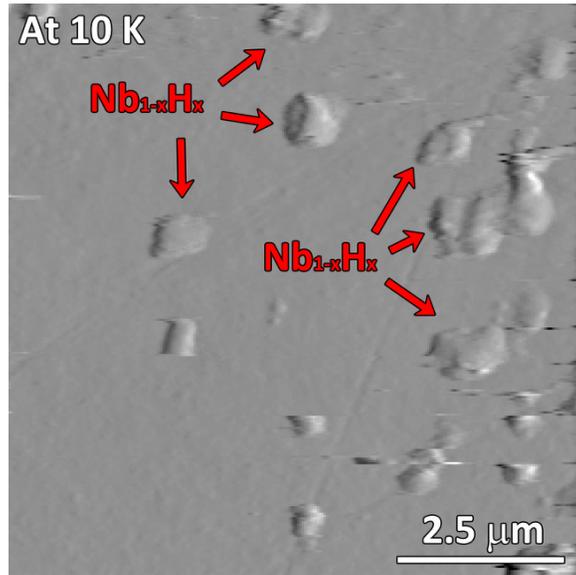


Height profile of two Nb hydrides

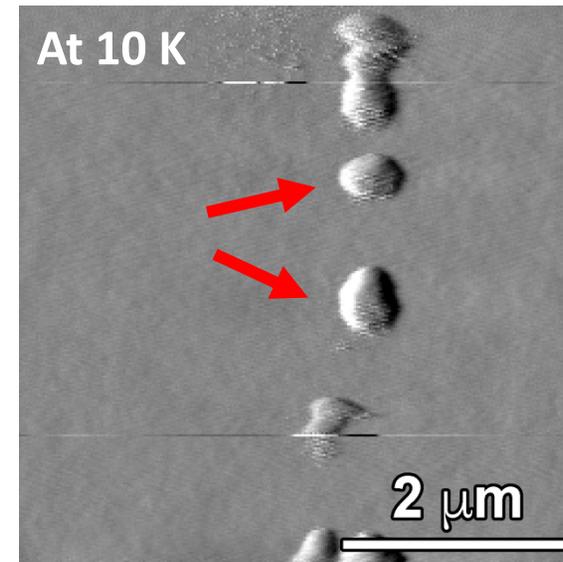


# Size and Distribution of NbH phases vary with cavity surface treatment condition

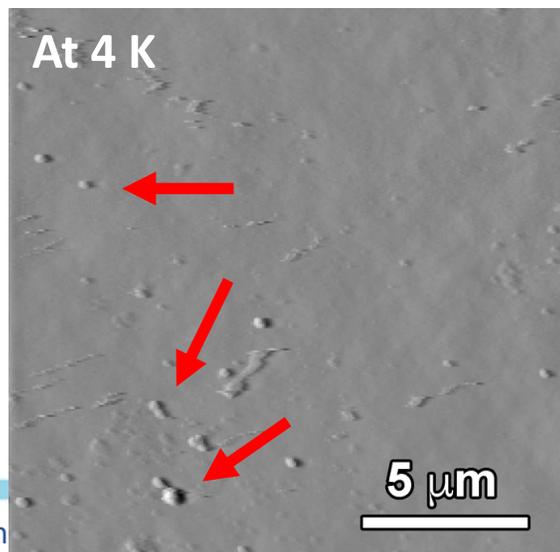
I. Hot Spot cut-out (EP'ed cavity, non-degaussed)



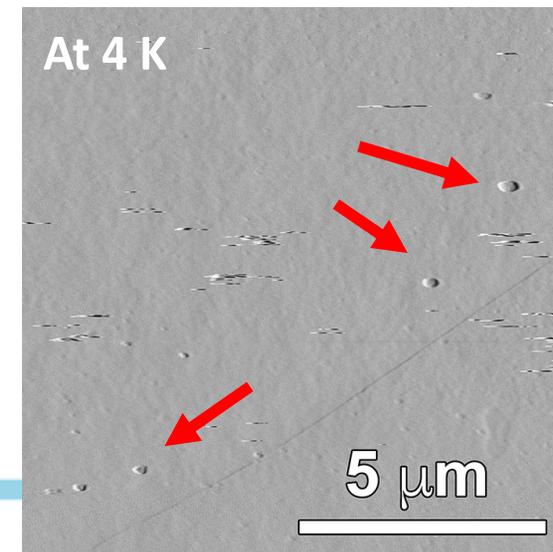
II. 800°C + BCP on hot spot cut-out



III. N-doped cavity cut-out (@800°C 25mT)

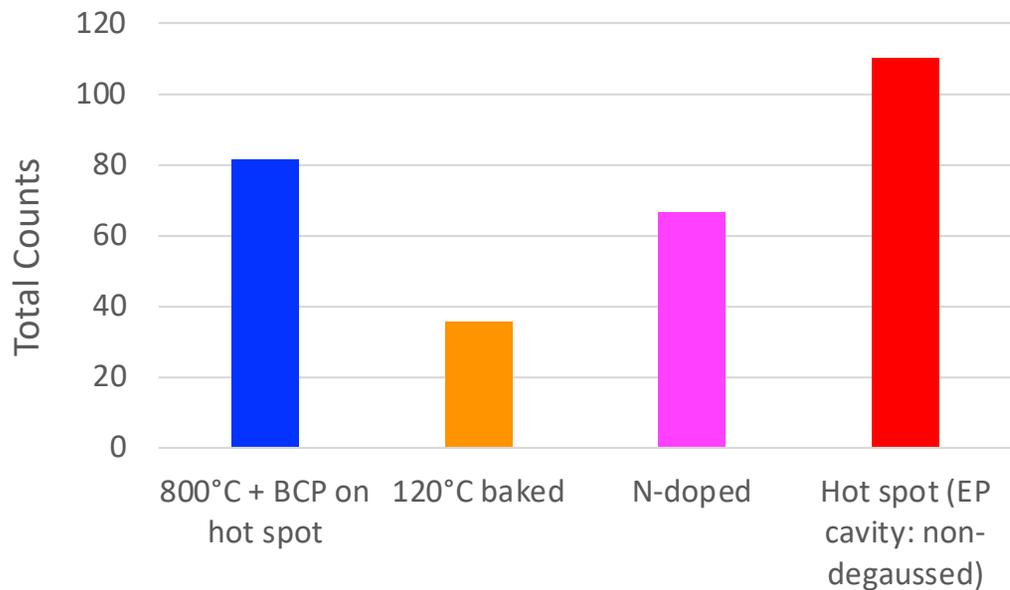


IV. 120°C baked cavity cut-out



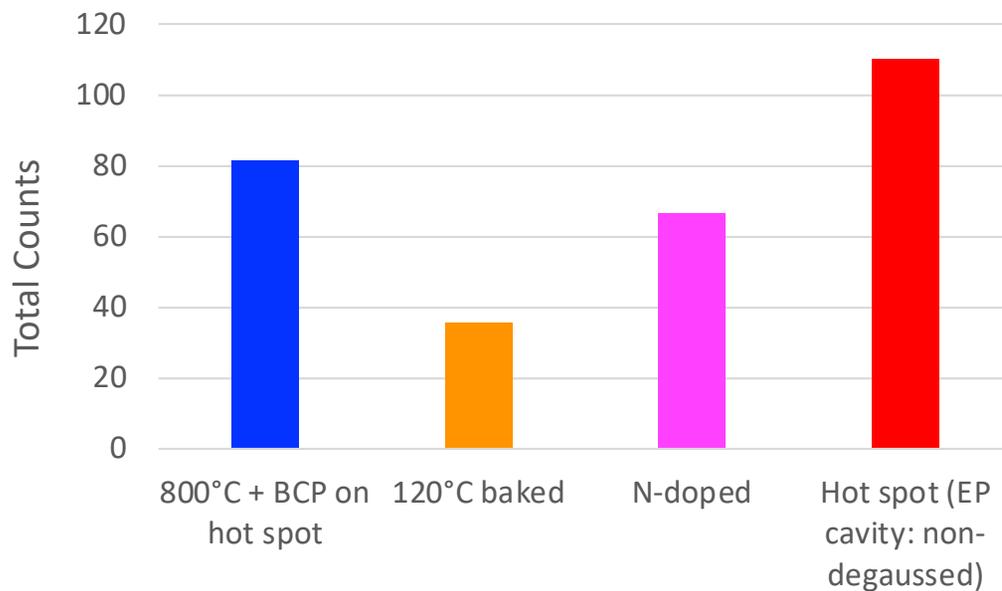
# NbH appearance frequency Vs its size distribution

**Total Avg # of NbH phase appearance  
within  $37 \times 37 \mu\text{m}^2$  scan area**

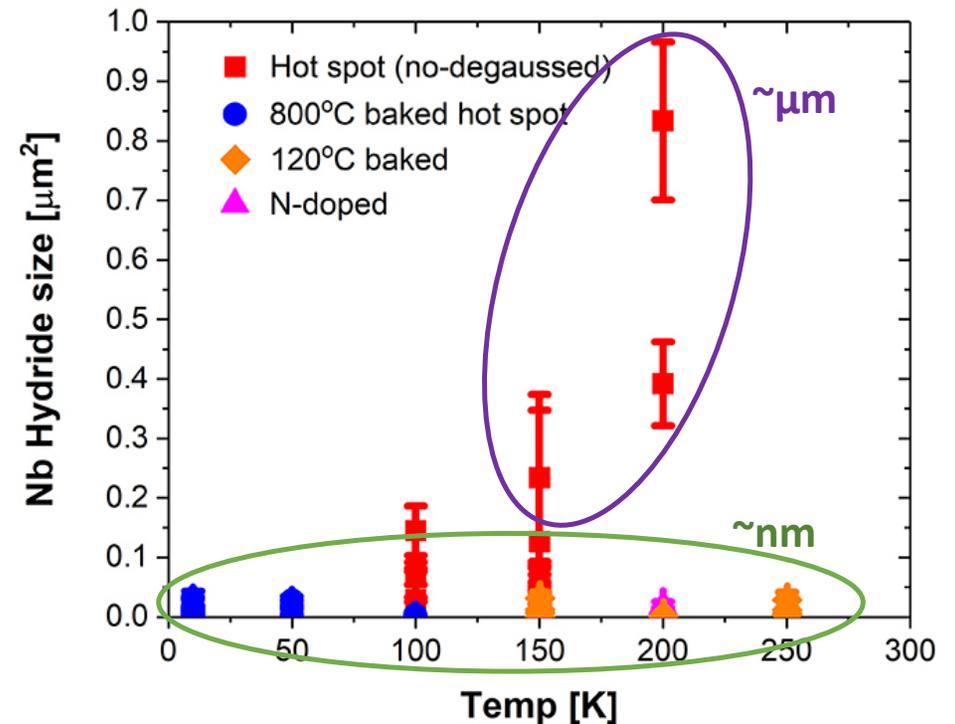


# NbH appearance frequency Vs its size distribution

Total Avg # of NbH phase appearance within  $37 \times 37 \mu\text{m}^2$  scan area



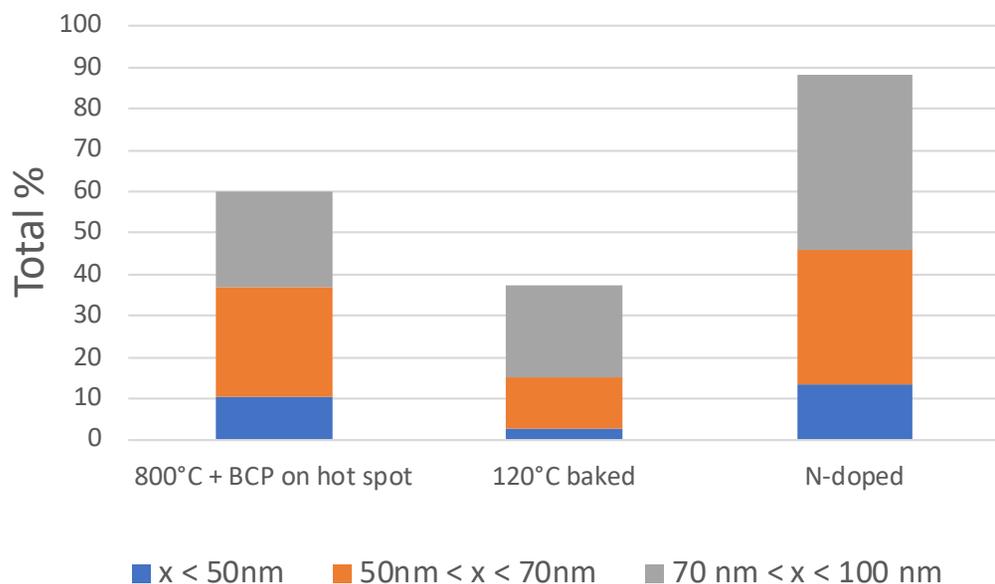
Size distribution as a f (T: 300K to 4K)



\* Size of NbH phase determined with its diameter ( $\phi$ ) and height (h) at cross section

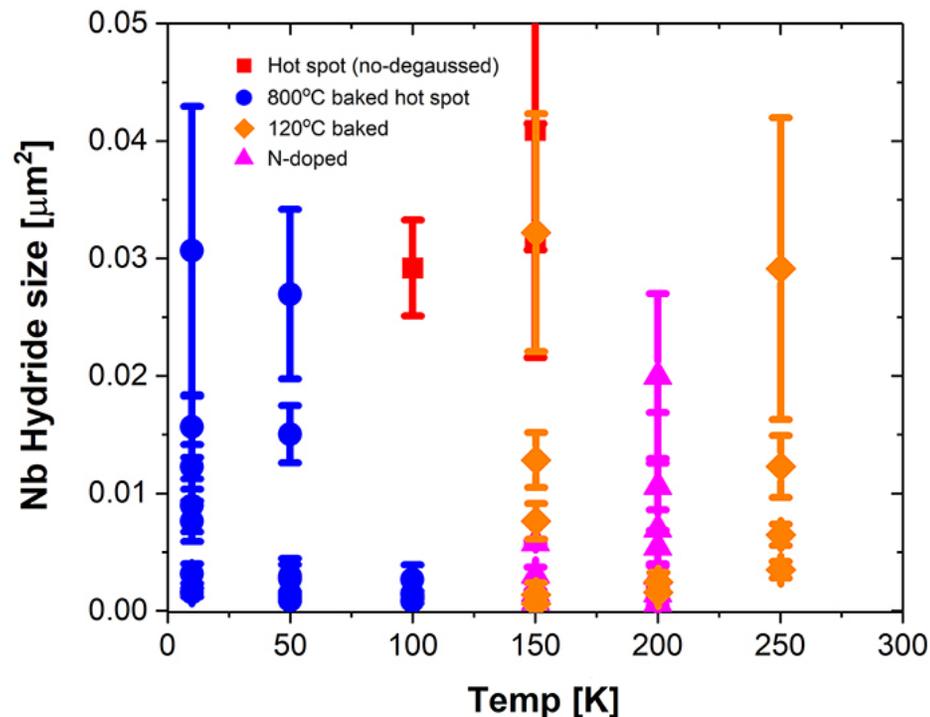
# 120°C baking likely suppressed NbH precipitations

Total Avg # of small NbH phase appearance within  $37 \times 37 \mu\text{m}^2$  scan area



X: height of NbH Phase

Size distribution as a f (T: 300K to 4K)



\* Size of NbH phase determined with its diameter ( $\phi$ ) and height (h) at cross section

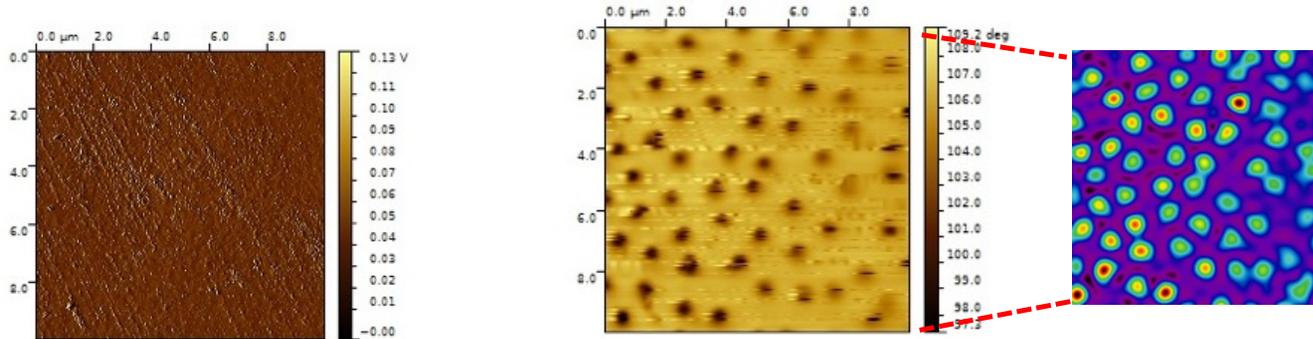
---

# *Magnetic Force Microscopy*

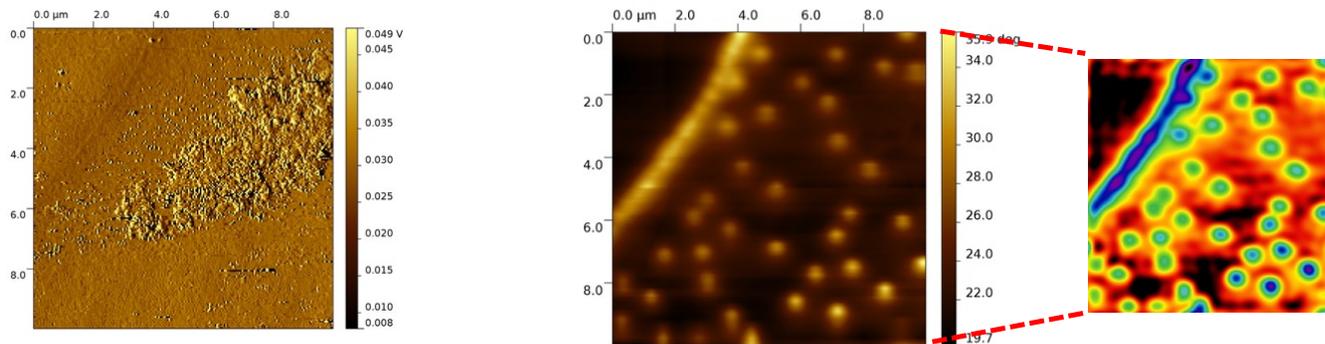
# Cryo-MFM analysis for magnetic vortices behavior on SRF Nb surface

- MFM Vortices Image

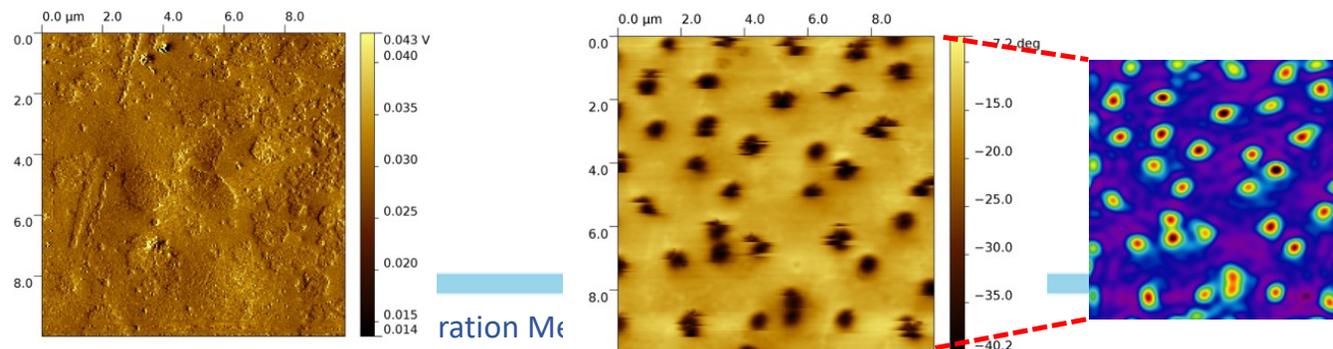
## I. 150 $\mu\text{m}$ EP'ed SRF grade Nb Coupon with 30 mT at 2 K



## II. EP + 120°C baked Nb Cavity cut-out with 5 mT at 2 K



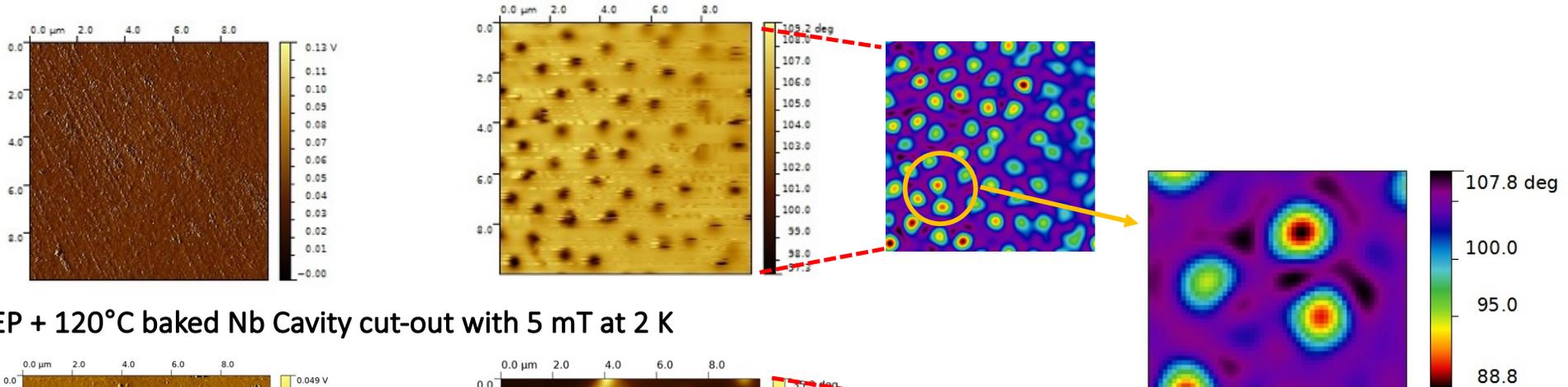
## III. Nitrogen-Doped at 800°C Nb Cavity cut-out with 5 mT at 2 K



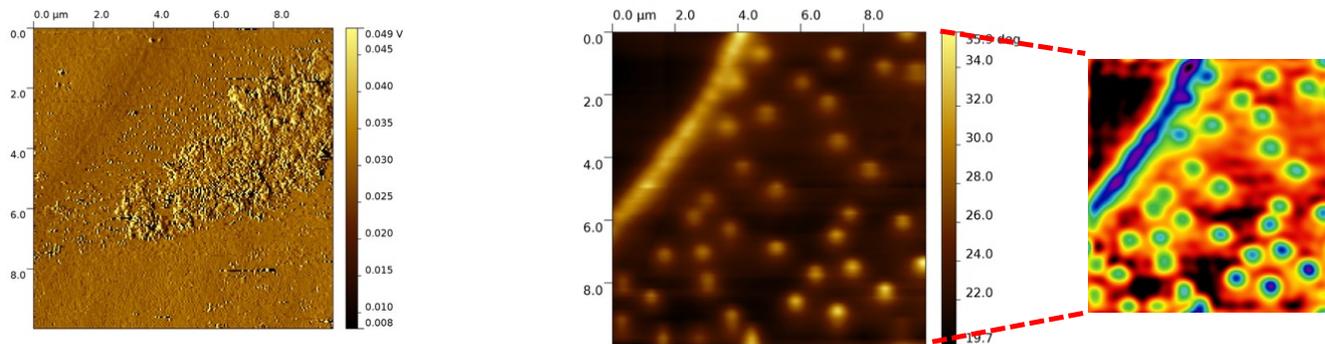
# Cryo-MFM analysis for magnetic vortices behavior on SRF Nb surface

- MFM Vortices Image

## I. 150 $\mu\text{m}$ EP'ed SRF grade Nb Coupon with 30 mT at 2 K

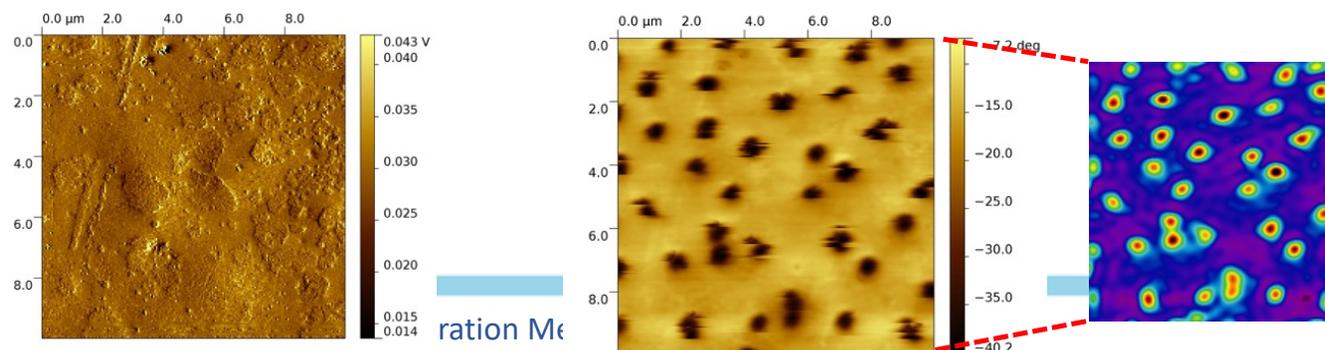


## II. EP + 120°C baked Nb Cavity cut-out with 5 mT at 2 K



*Individual vortex:  
 $\phi \sim n \cdot \lambda$   
 for SC surface  
 property*

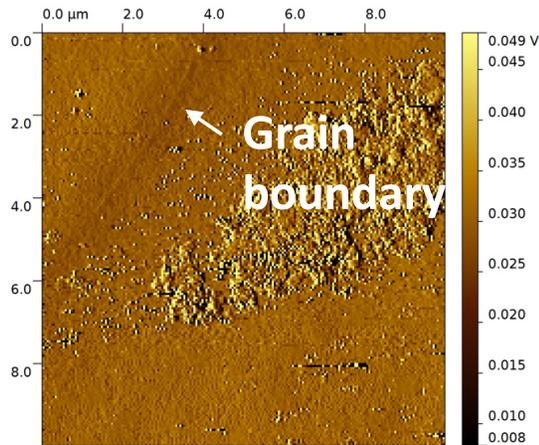
## III. Nitrogen-Doped at 800°C Nb Cavity cut-out with 5 mT at 2 K



*$\phi \sim$  diameter of a vortex  
 $\lambda$ : SC Penetration depth*

# Visualizing trapped magnetic vortices on GB and intra-grain of SRF Nb sample

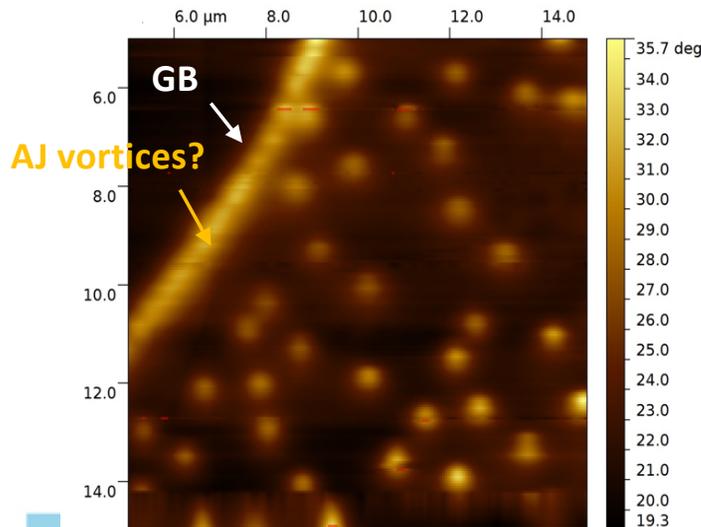
Surface topology of 120°C baked cavity cut-out



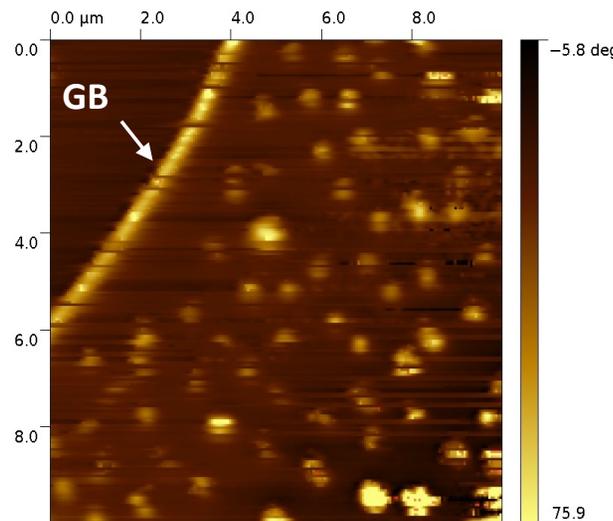
*Direct observation of flux trapping using different cooling mode wt DC field (currently only slow cool, plan to study flux trapping under thermogradients)*

- ✓ Vortex pinning at GB is not different between ZFC and FC mode
- ✓ Disorder pattern of magnetic vortices in grain

ZFC wt 5 mT, at 2K



FC wt 5 mT, at 2K



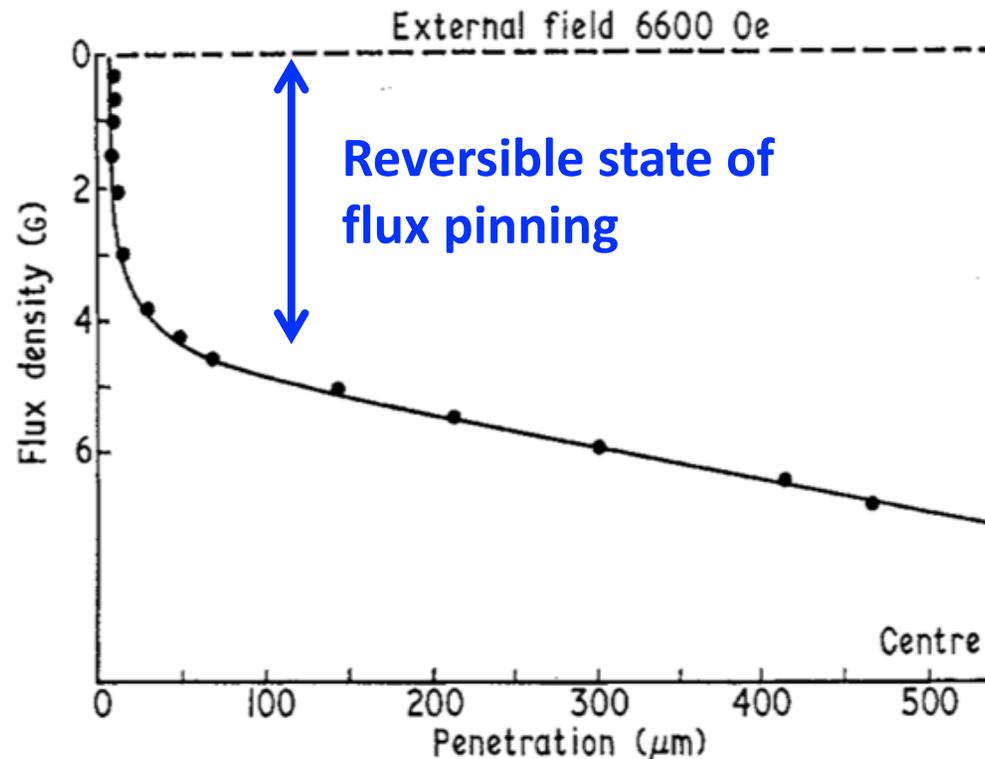
ZFC: Zero Field Cooling (cooling <  $T_c$  wo field)

FC: Zero Field Cooling (cooling <  $T_c$  wt field)

---

# *AC Susceptibility*

# AC susceptibility characterization for penetration dynamics of surface magnetic vortices in DC magnetic field



*“This displacement after linearity is due to the reversible movement of flux lines in their potential wells in the surface layer and indicates a large penetration depth which depends on the pinning”*

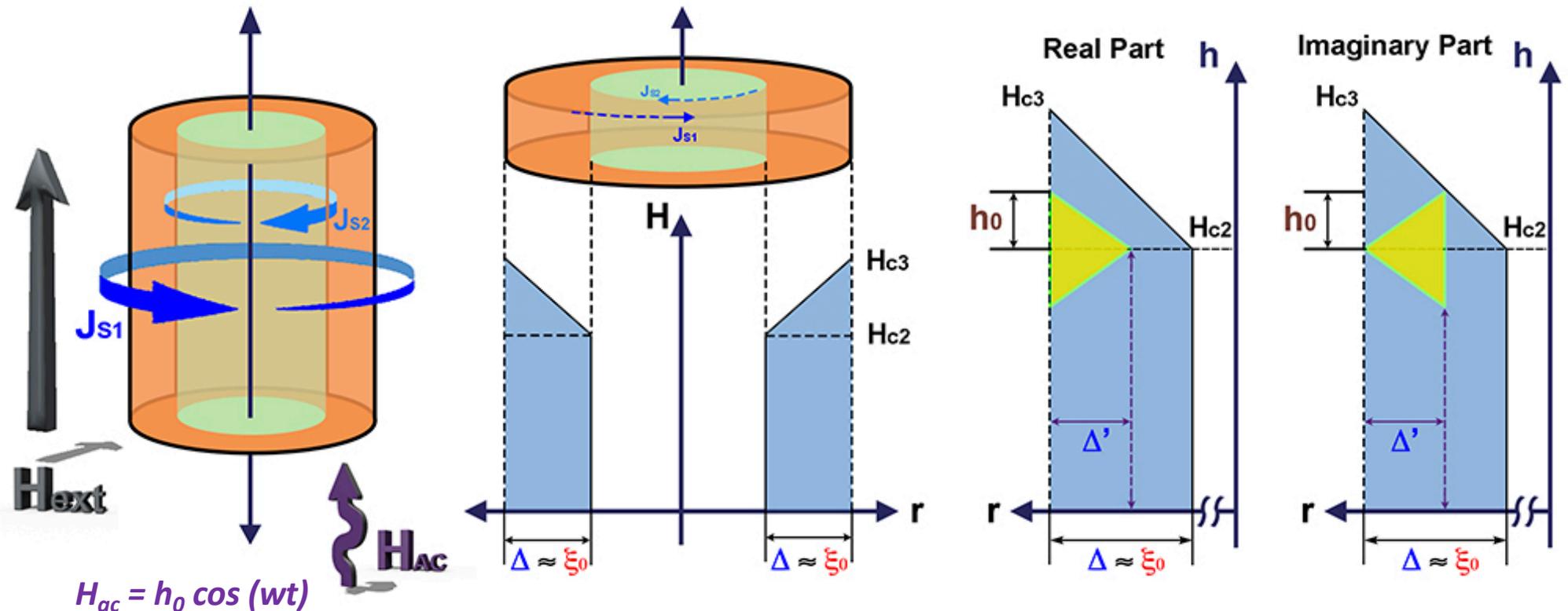
Figure 3. The flux distribution in a reversible specimen.

$$\lambda_{AC} \approx \frac{a}{2C} \cdot \frac{d\phi}{dh_0}$$

- A. C. Campbell, *J. Phys. C (Solid St. Phys.)*, 2, 8, (1969)
- A. C. Campbell, *DC Magnetization and Flux Profile Techniques*, pp 129-155, *Magnetic Susceptibility of Superconductors and Other spin system*, Springer, (1991)

# AC Susceptibility with Surface Critical Model

## Reversible Depth of Penetrating Magnetic vortices ( $\lambda_{AC}$ )



- R. W. Rollins and J. Silcox, Phys. Lett. 155, 2, (1967)
- J. R. Clem and J Perez-Gonzalez, Phys. Rev. B, 33, 3, (1986)
- J. R. Clem, Phys. Rev. B, 14, 5, (1976)
- A. C. Campbell, J. Phys. C (Solid St. Phys.), 2, 8, (1969)

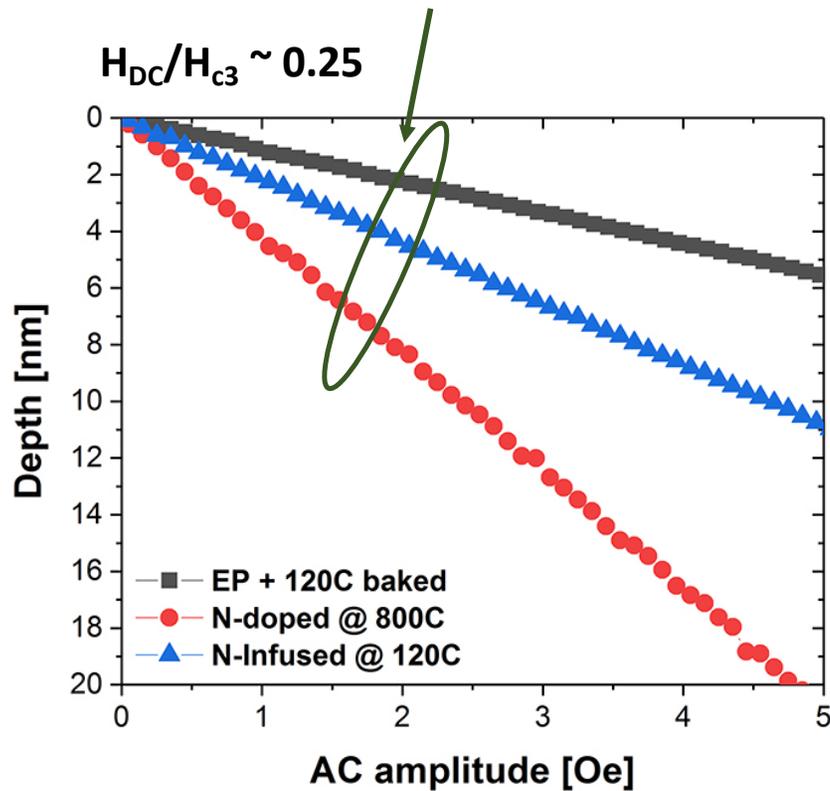
$h_0$  : AC amplitude

**YEL: Magnetization of AC Field**

**Blue: DC Magnetization**

# N-doping and Infusion show different mechanism of magnetic vortex behavior within 20 nm surface depth

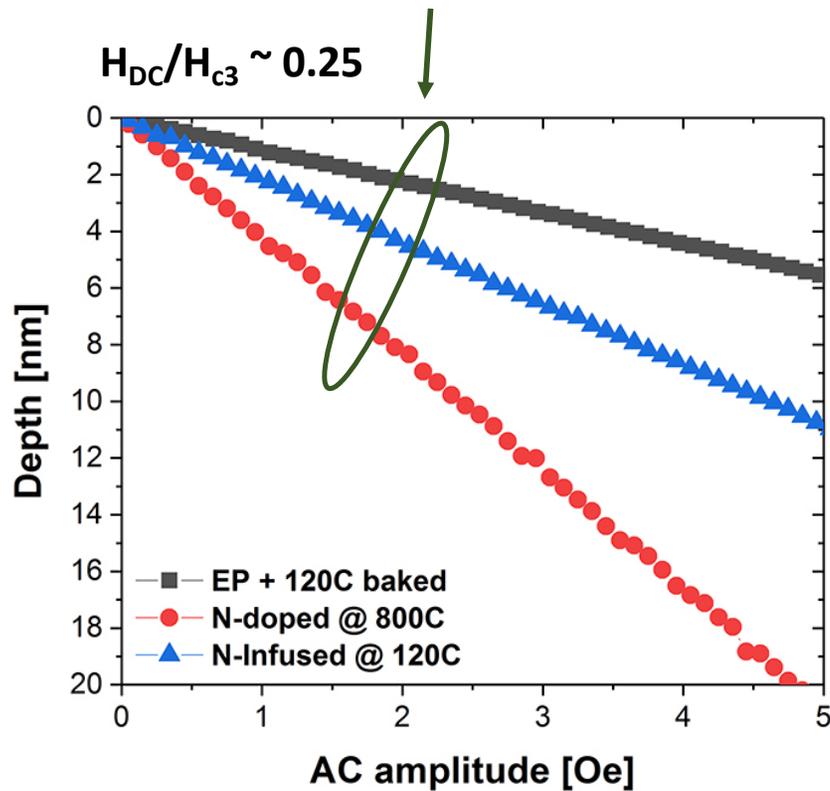
All Irreversible behavior,  
but different vortices penetrating  
into the surface



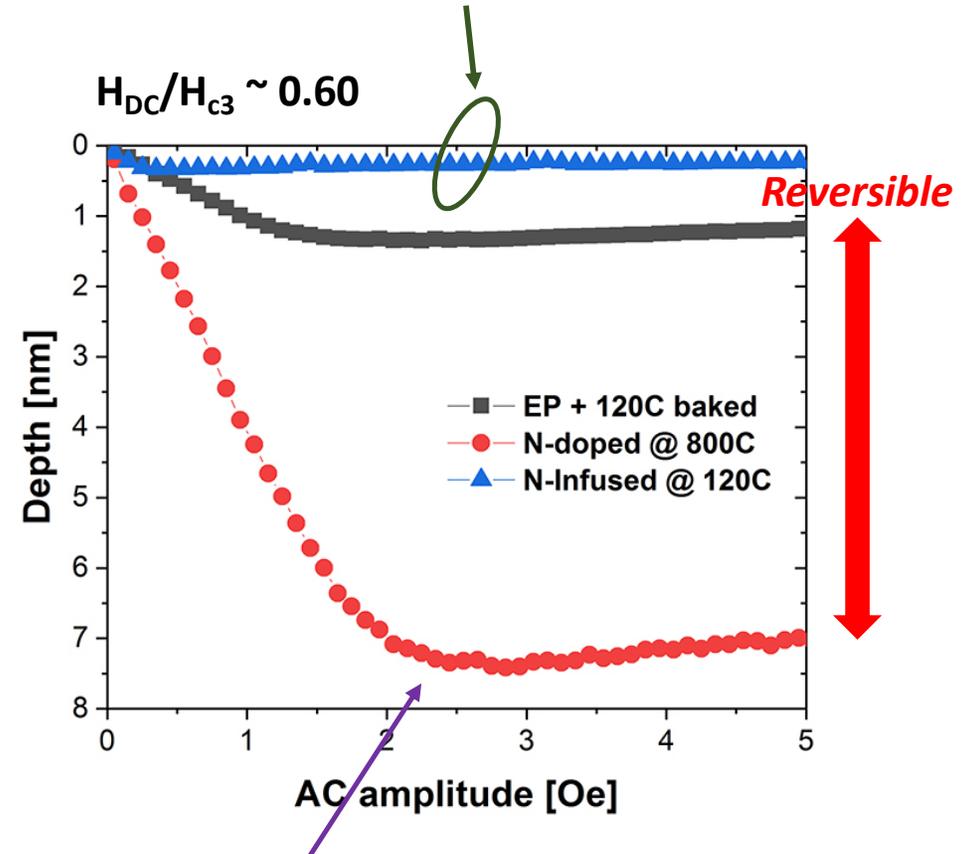
All tested samples are cylindrical shape (low  $F_{deg}$ ,  $\phi \sim 3\text{mm}$ , length  $\sim 7\text{mm}$ )

# N-doping and Infusion show different mechanism of magnetic vortex behavior within 20 nm surface depth

All Irreversible behavior, but different vortices penetrating into the surface



N-infused surface significantly expel magnetic vortices from the surface



N-doping surface has large reversible vortex penetration depth ( $\lambda_{ac}$ )

All tested samples are cylindrical shape (low  $F_{deg}$ ,  $\phi \sim 3mm$ , length  $\sim 7mm$ )

# Summary

---

- **First successful observation of nanometer size Nb hydride phase on SRF Nb cavity surface.**
- **120°C baking leads to earlier NbH formation, compared to 800°C HT, but likely suppressed # of its precipitation.**
- **Cryo-MFM is a promising tool that can directly visualize trapping of magnetic vortices on SRF Nb cavity.**
- **Unlike N-infusion, N-doped Nb surface likely has much deeper reversible vortices penetration depth.**

# Variation of the thickness ( $\Delta$ ) of superconducting surface with $\kappa$ and $H_{\text{app}}$

$$\Psi = e^{ik\varphi} \cdot F(\rho)$$

$$\rho = (x/\xi) \cdot (H_0/H_{c2})^{1/2}$$

$$\Delta = \frac{1}{F^2(0)} \int_0^\infty F^2(\rho) dx$$

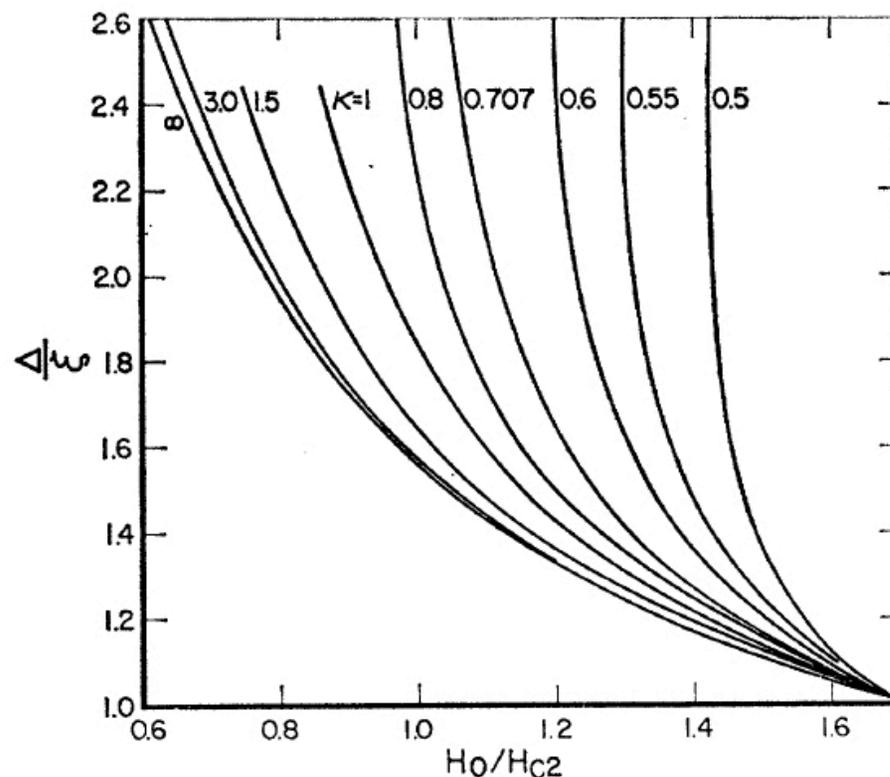
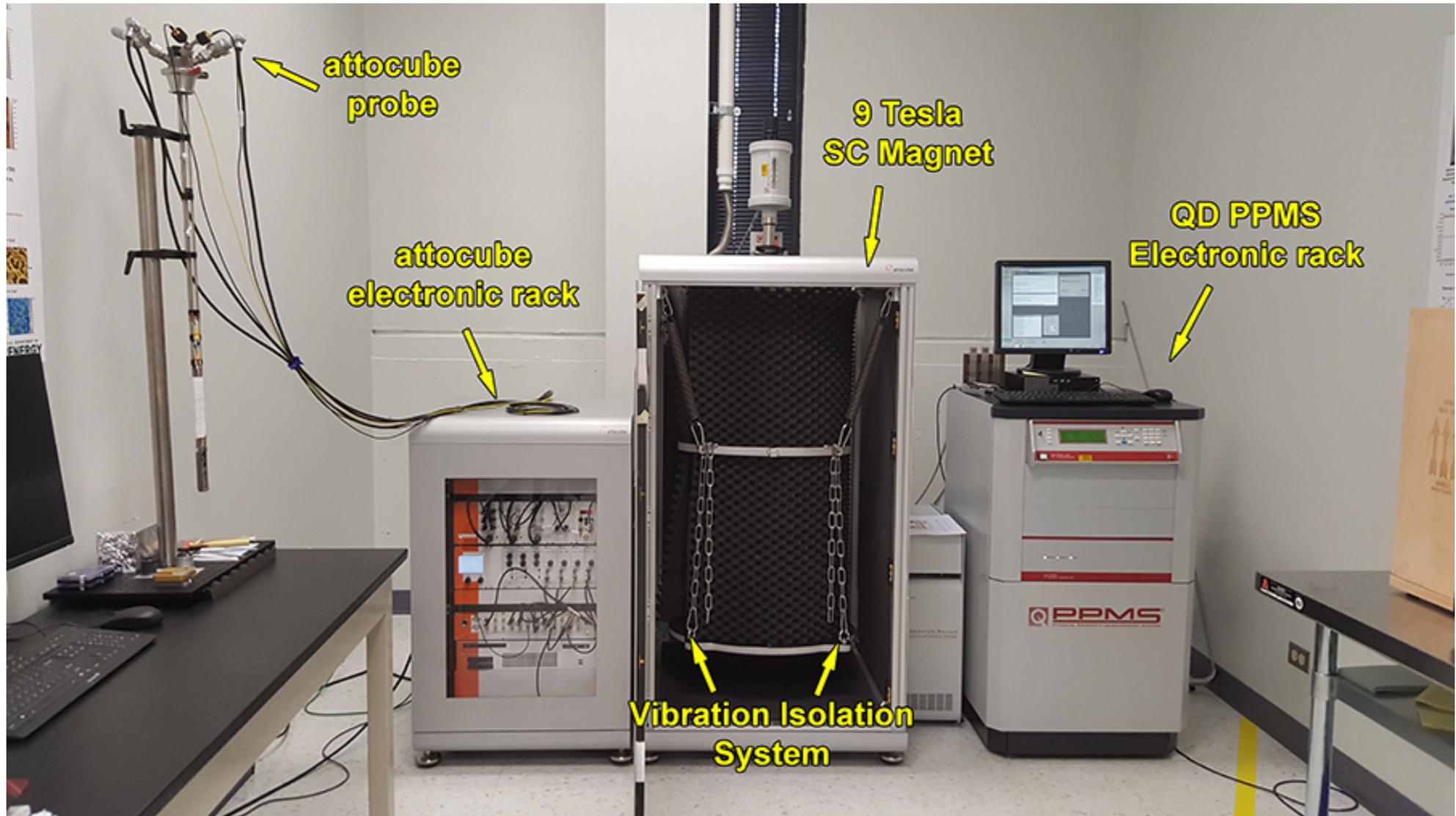


FIG. 5. The thickness of the superconducting surface sheath  $\Delta$  in units of the coherence length  $\xi$  as defined by Eq. (26) is shown as a function of  $H_0/H_{c2}$  and the Ginzburg-Landau parameter  $\kappa$ . Near the critical fields  $H_c$  (for  $0.707 > \kappa > 0.417$ ) and near  $H_{c1}$  (for  $\kappa > 0.707$ ) the thickness of the sheath becomes very large. At  $H_{c3}$  the value of  $\Delta/\xi$  is 1.00764.

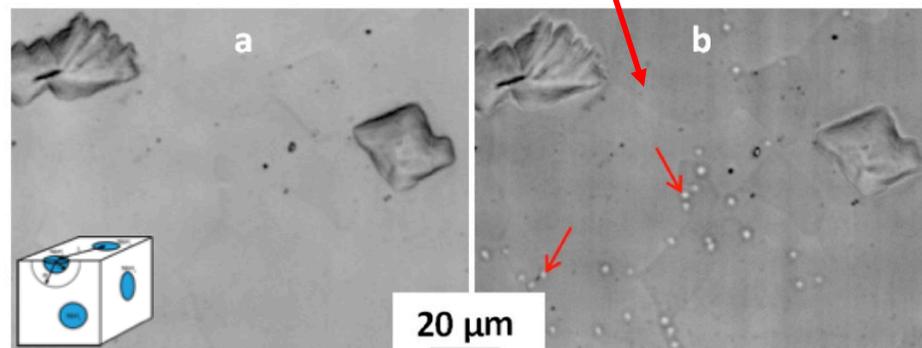
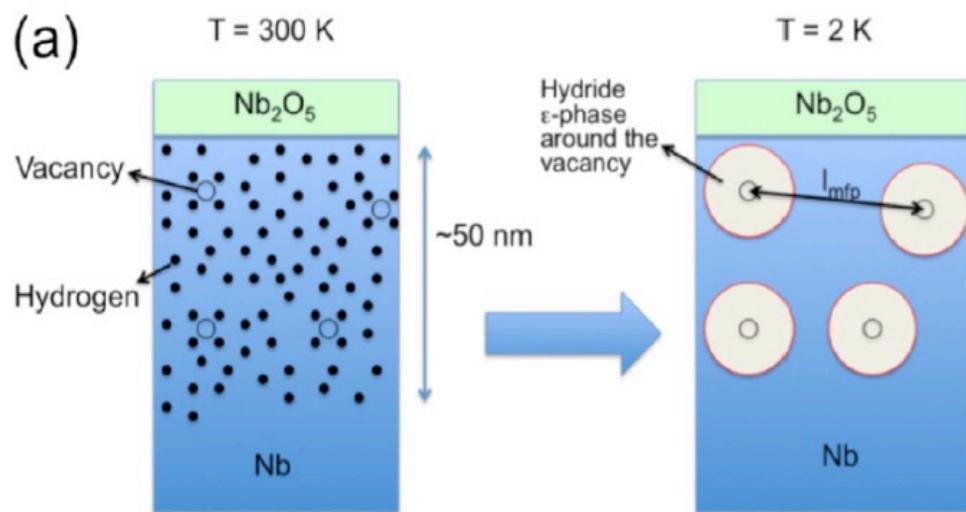
H. J. Fink and R. R. Kessinger, Phys. Rev. Lett. 140. 6A. 1937, (1965)

# Cryogenic AFM/MFM (attocube) + 9T PPMS System (QD Design) @FNAL SRF MSL



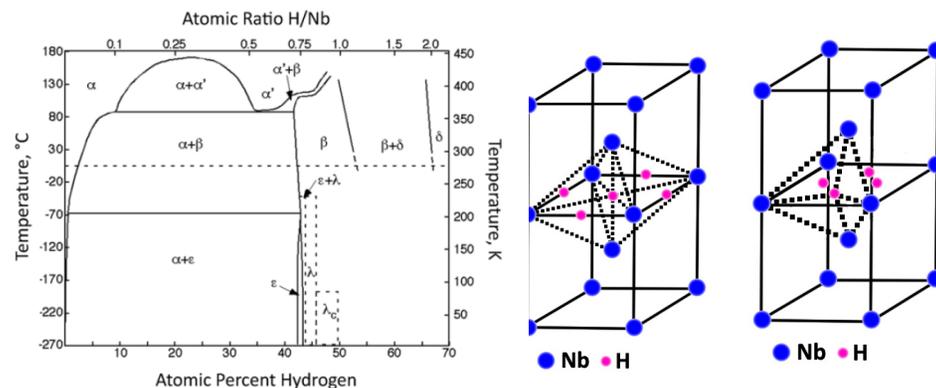
# Breakdown of superconductivity by NbH proximity effect

Nano size NbH phases are detectable ONLY by Cryo-AFM



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

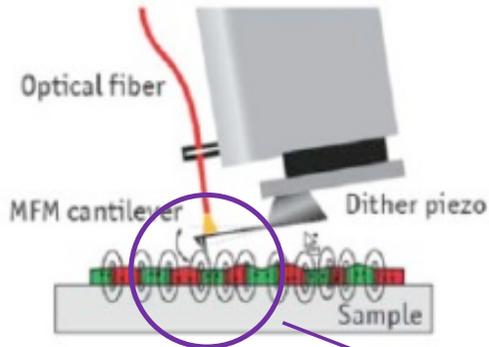
Nb-H Proximity Effect : A. Romanenko, *Supercond. Sci. Technol.* 26, 035003, (2013)



Hydrogen atoms interstitially entered in niobium bcc cells (a) Octahedral position; (b) tetrahedral positions [S. Isagawa, *J. Appl Phys.* 51(8), 1980]

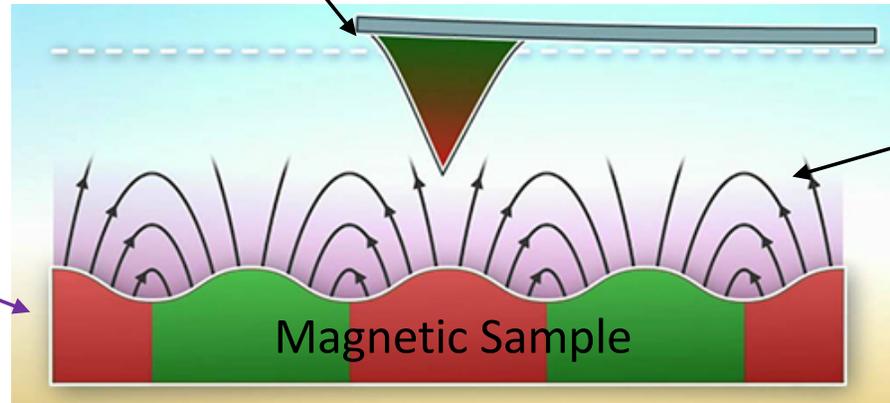
# Cryogenic Magnetic Force Microscope (MFM)

## AFM Head Part



Sketch of the MFM setup. An AFM tip (here: magnetically coated), is brought into close vicinity of the sample. The deflection of the cantilever due to forces on the tip can be read for magnetic field variations using a build-in fiber interferometer

## Co Coated – MFM tip

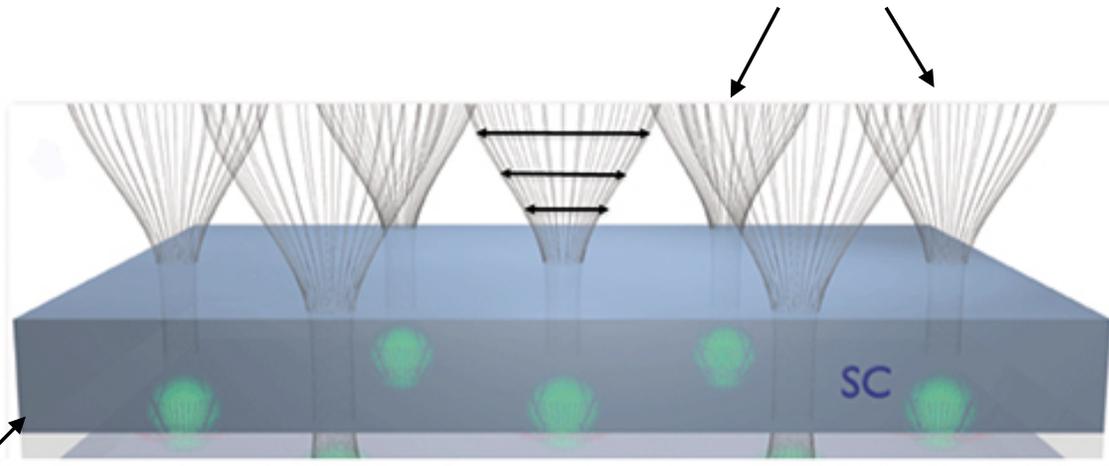


Magnetic field lines

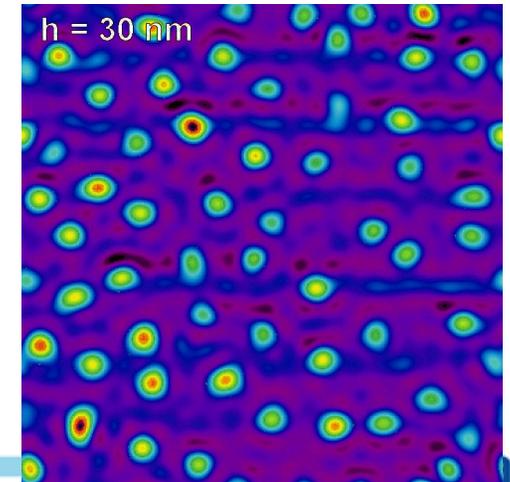
Magnetic Sample

Size variation of vortices as a  $f(h)$  on EP'ed Nb

## Strays of Magnetic Vortices



Superconductor | TTC Collaboration Meeting Feb 6-9 2018



2/8/2018