Fermilab U.S. DEPARTMENT OF ENERGY Office of Science

Synthesis and characterization of $\gamma/\delta-\mathrm{NbN}$ for SRF cavity application and vortices study in superconductors

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Final review

27/09/2017

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PPMS-MFM Mentor: Zuhawn Sung

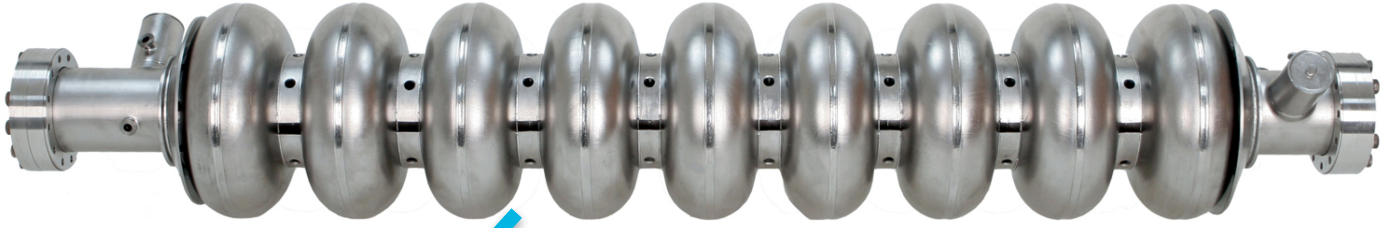
Overview

1. Synthesis and characterization of $\gamma/\delta-\mathrm{NbN}$

2. Study of vortices in superconductors.

1) Synthesis and characterization of $\gamma/\delta-\mathrm{NbN}$

Task and purpose overview



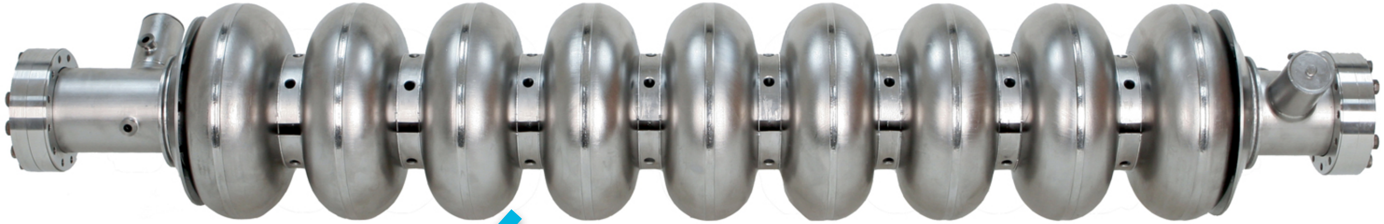
$$Q \propto \frac{1}{R_s}$$

Quality Factor

$$R_s \propto e^{-T_c/T}$$

Surface resistance

Task and purpose overview



$$Q \propto \frac{1}{R_s}$$

Quality Factor

$$R_s \propto e^{-T_c/T}$$

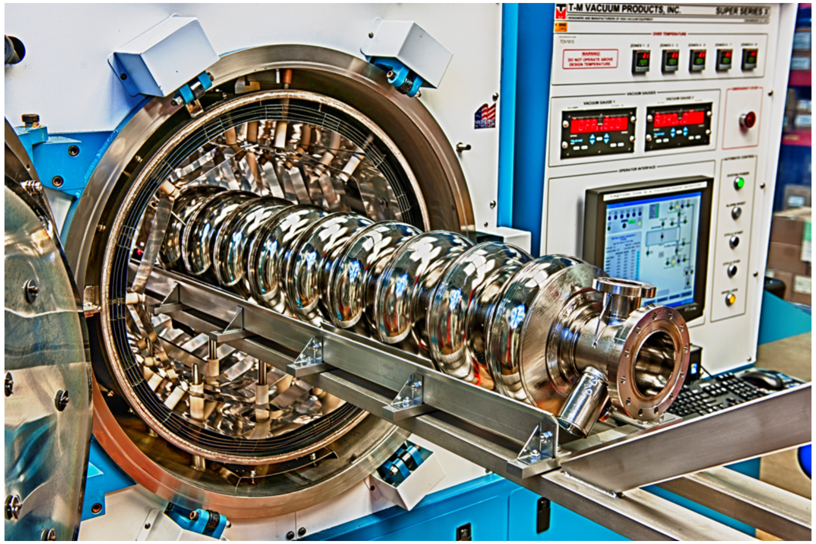
Surface resistance

Nb EP (Electro polished)
T_c=9.25 K

→

2-5 μm film of:
$\delta - NbN \quad T_c = 15\text{-}17\ K$
$\gamma - NbN \quad T_c = 12\text{-}15\ K$

Overview of the method



Synthesis in furnace:

$$p_{N_2}, T, time$$

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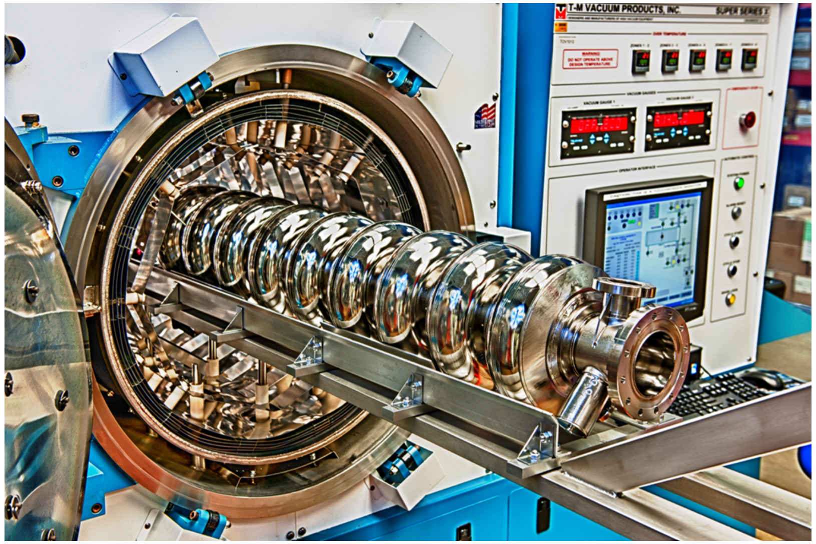
Overview of the method



Synthesis in furnace:

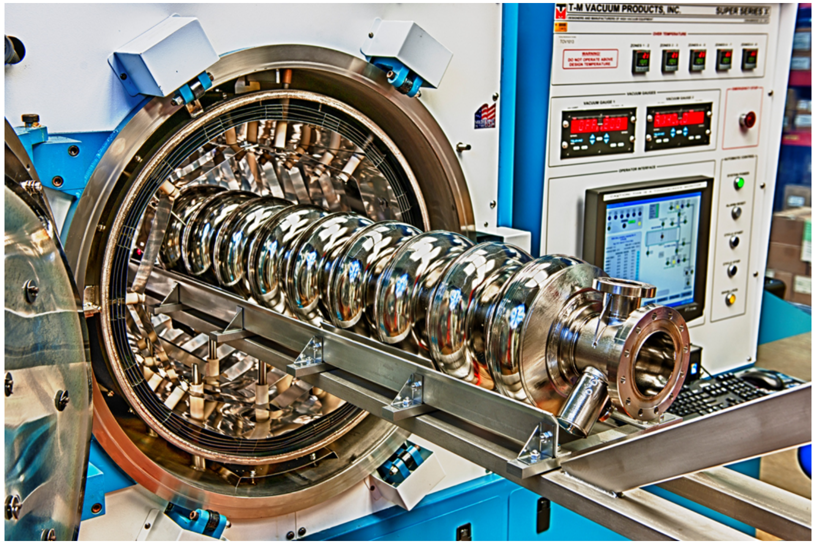
$p_{N_2}, T, time$

"not good"

Characterization:

SEM/EDS, AFM, MFM, PPMS, SIMS

Overview of the method



Synthesis in furnace:
$p_{N_2}, T, time$

"not good"

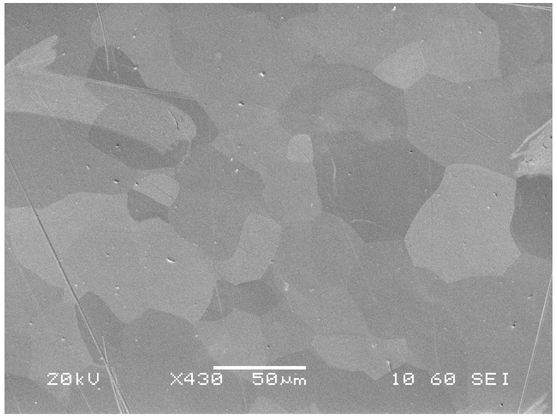
Characterization:
SEM/EDS, AFM, MFM, PPMS, SIMS

If results are "good" the process will be applied to a 1.3 GHz SRF cavity.

Reference sample Electro Polished (EP) Nb

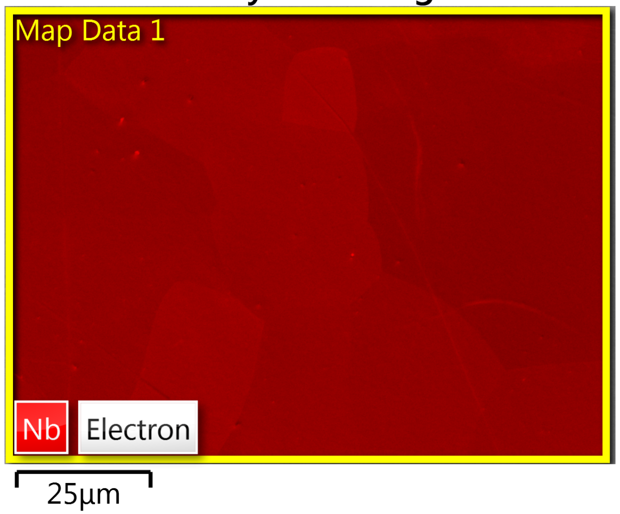
Morphology EP Niobium (SEM-AFM)

SEM (Scanning Electron Microscopy):

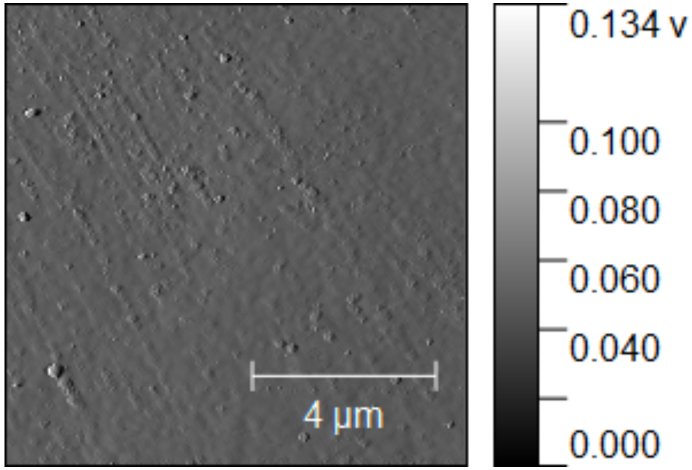


EDS (Energy Dispersive X-ray Spectrometry)

EDS Layered Image 1

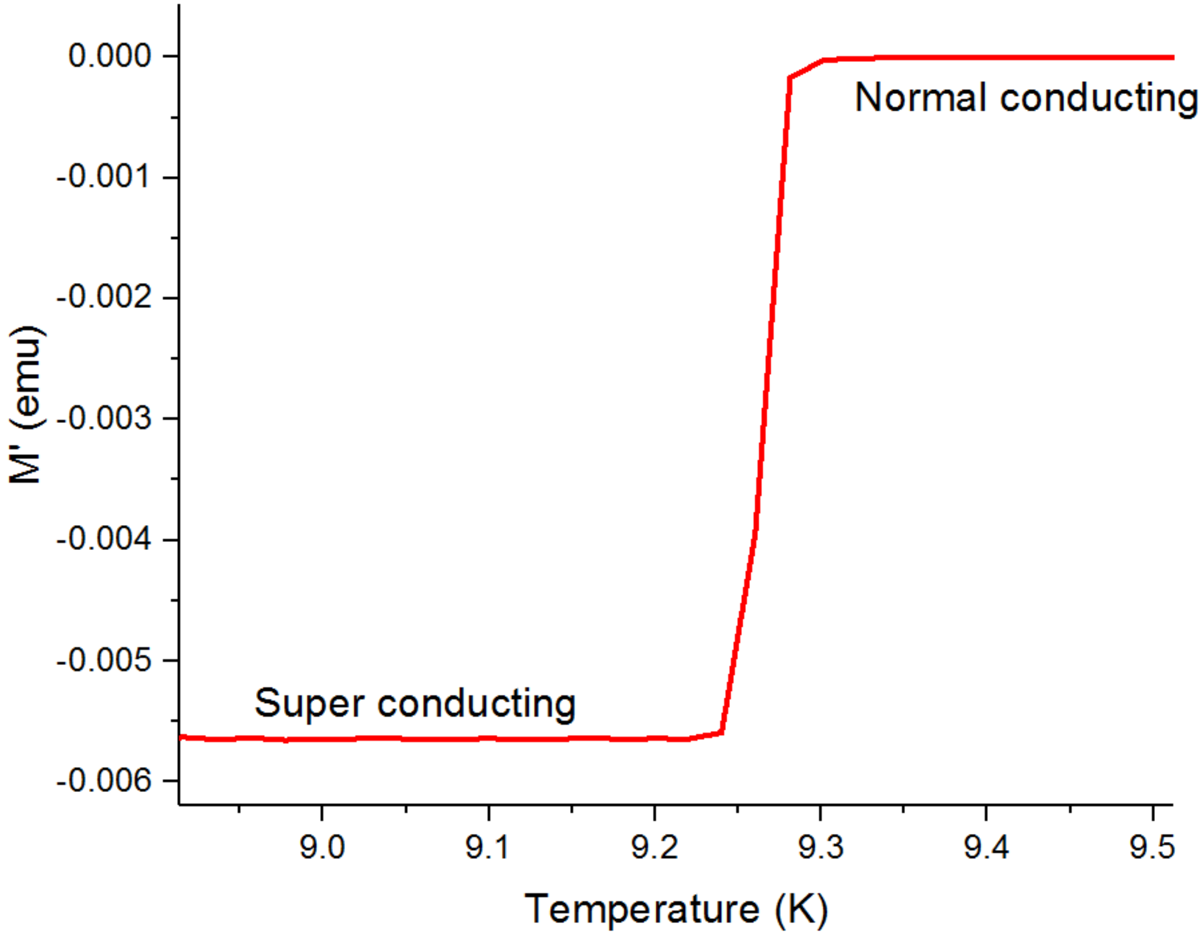


AFM (Atomic Force Microscopy):

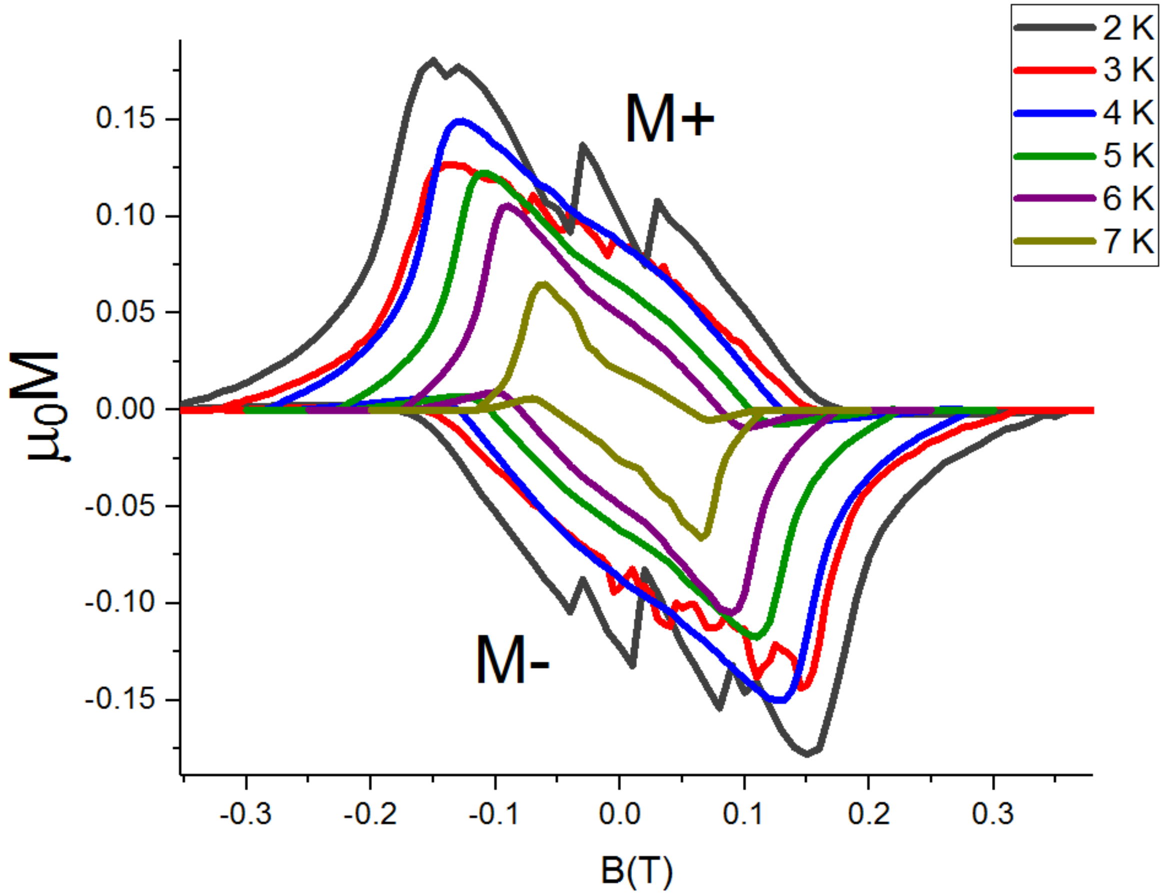


Reference Nb EP, AC Susceptibility (PPMS)

One transition:
T_c =9.26 K



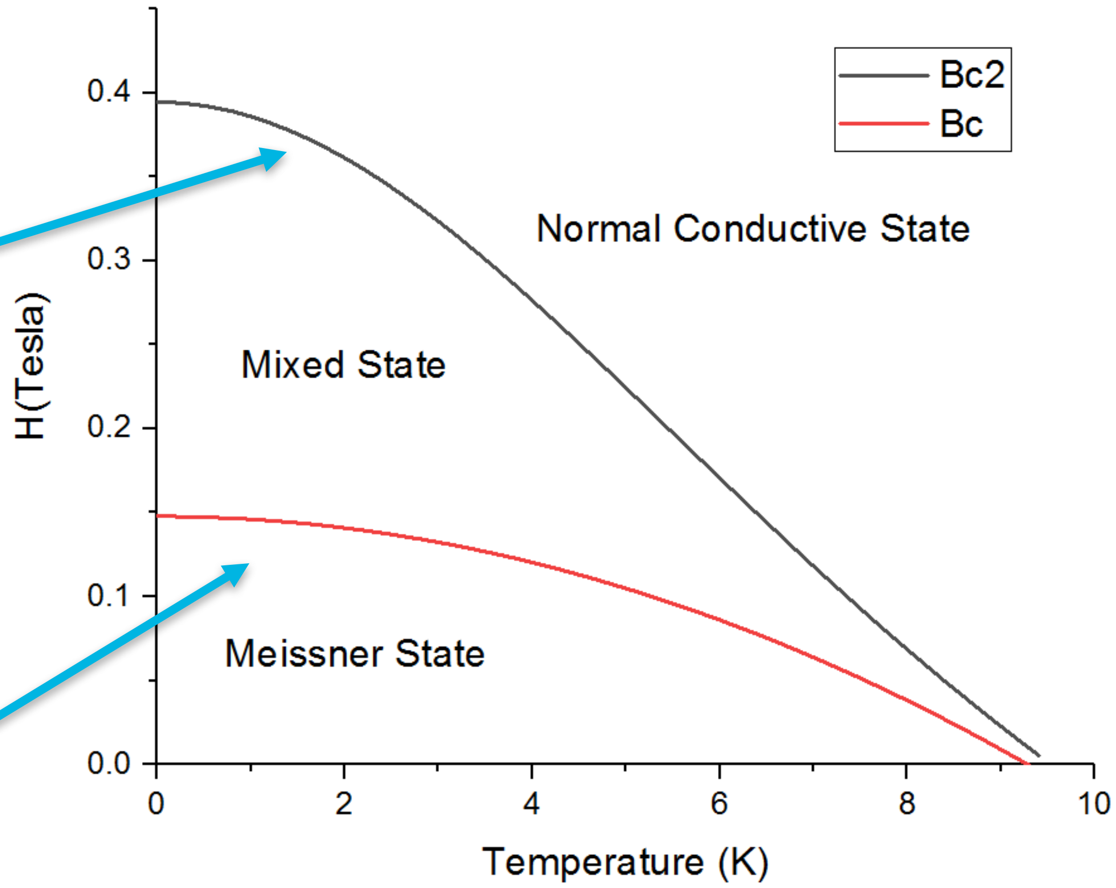
Nb EP, Bulk Magnetization loops (PPMS)



Nb EP, H(T) phase diagram

$$H_{c2}(T) = H_{c2}(0)\frac{1-(T/T_c)^2}{1+(T/T_c)^2}$$

$$H_c(T) = H_c(0)[1-(T/T_c)^2]$$



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Reference Nb EP, superconductor parameters

- Within the **Ginzburg-Landau** (GL) theory is possible to determine: the GL parameter (k), the coherence length (ξ) and the London penetration depth (λ_L) (all at 0K).

$$k(0) = \frac{B_{c2}(0)}{\sqrt{2}B_c(0)}$$

$$\xi(0) = \sqrt{\frac{\hbar}{2eB_{c2}(0)}}$$

$$\lambda_L(0) = k(0)\xi(0)$$

$B_{c2}(0)$(Tesla)	0.394$\pm$0.005
$B_c(0)$(Tesla)	0.148$\pm$0.005
T_c(K)	9.41$\pm$0.42
k(0)	1.89$\pm$0.07
$\lambda_L(0)$(nm)	54$\pm$2
$\xi(0)$(nm)	28.9$\pm$0.2

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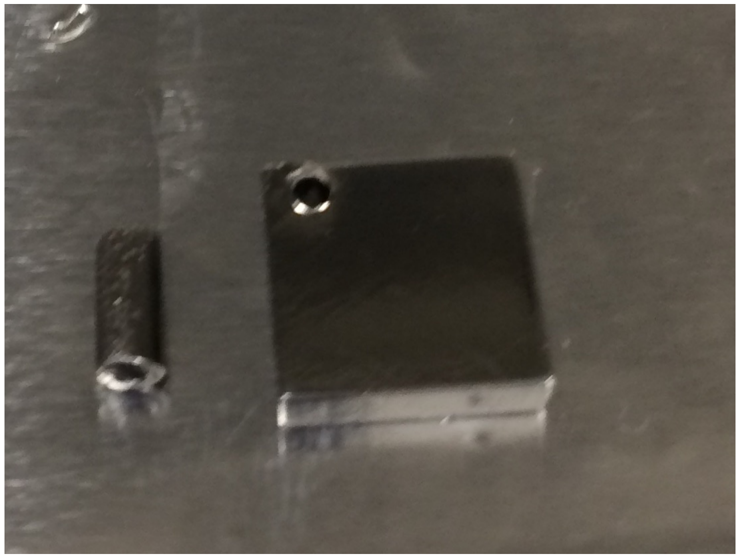
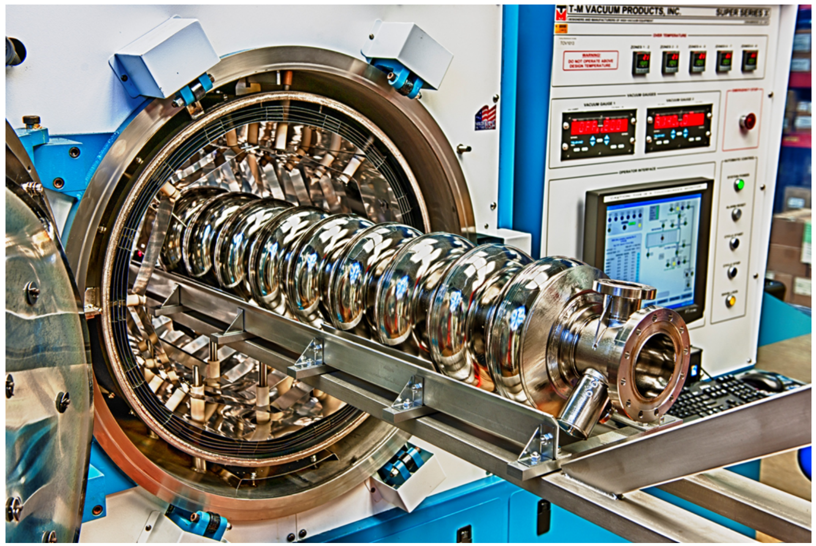
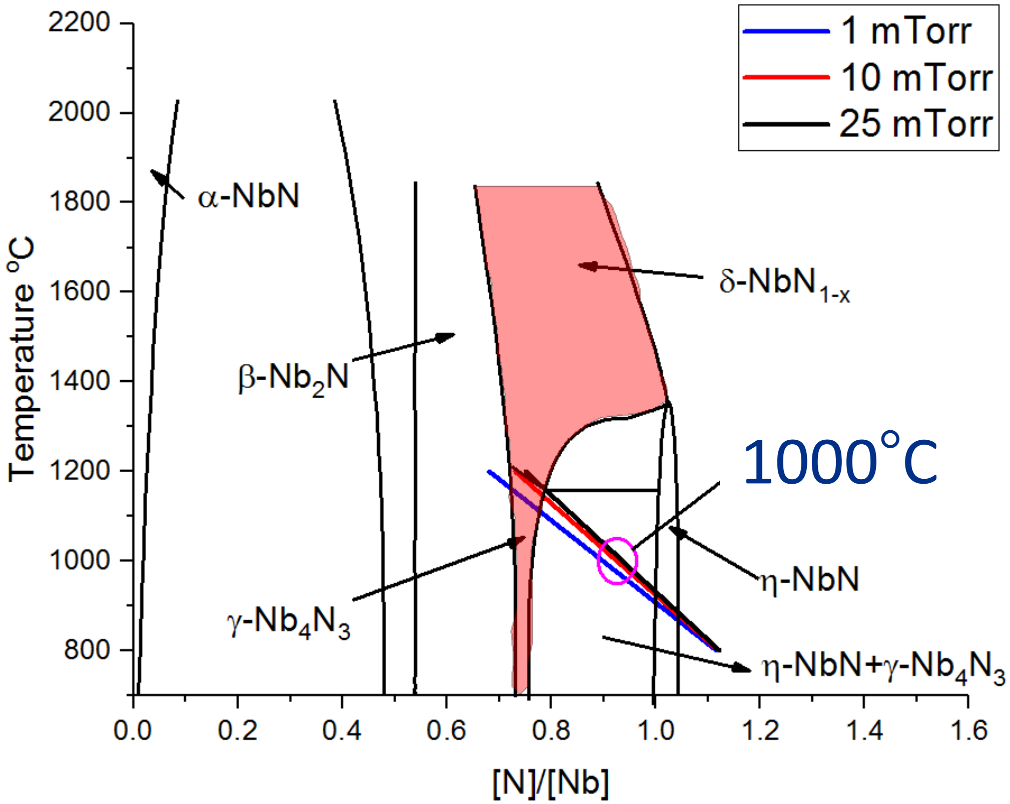
N_2 Treated samples





N_2 pressure dependence of Nb-N phase diagram



Phase of interest in this study:

- ***δ*-NbN T_c=15-17,3K**
- ***γ*-NbN T_c=12-15K**

$$\frac{[N]}{[Nb]} = 1.585 - 4.148 \cdot 10^{-4} \cdot T - 0.05176 \cdot ln(p_{N_2}) + 4.984 \cdot 10^{-5} \cdot T \cdot ln(p_{N_2})$$

M. Joguet, W. Lengauer, M. Bohn, J. Bauer, *J. of Alloys and Compounds 269 (1998) 233-237*

Samples

-Nb Electro Polished → Starting material

Pre-treatment: 800°C 3h no nitrogen

1) T=800°C p_{N2}=25 mTorr time=25min

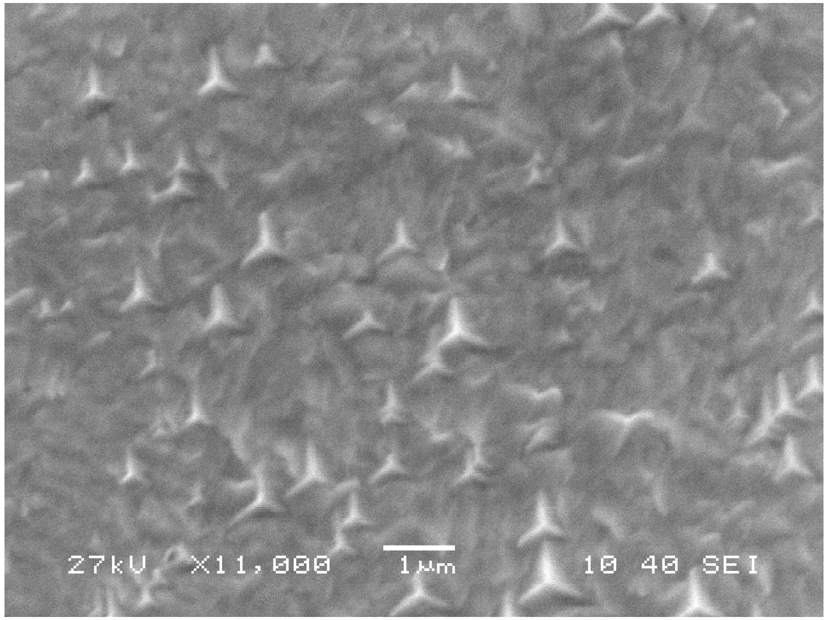
2) T=1000°C p_{N2}=10 mTorr time=2h

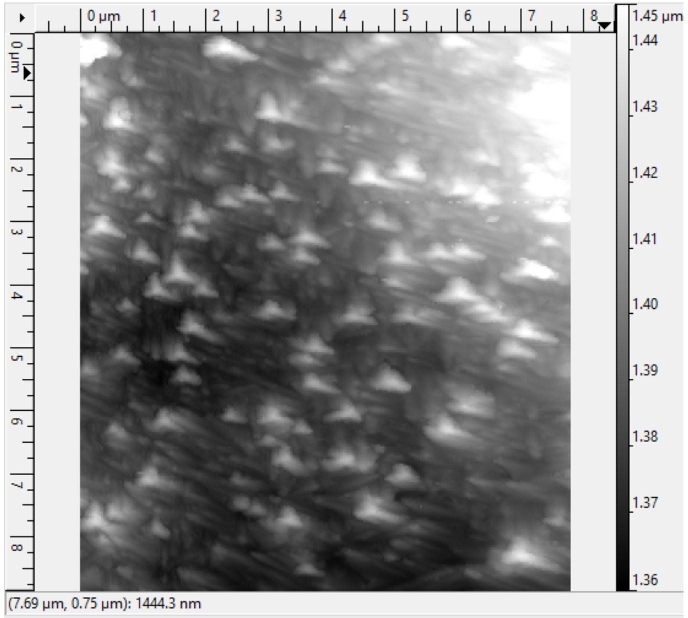
3) T=1000°C p_{N2}=50 mTorr time=2h

Surface morphology in top view

Surface morphology (1)

-T=800°C p_{N2}=25 mTorr time=25min



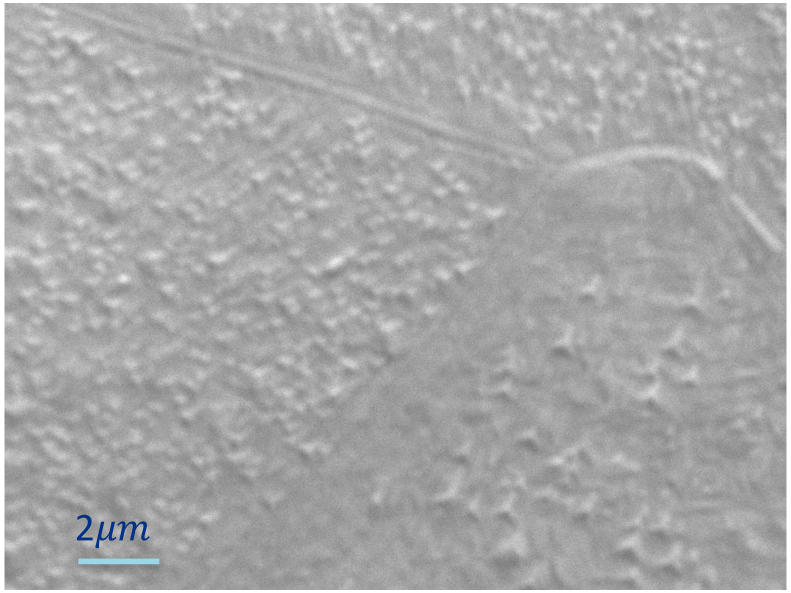


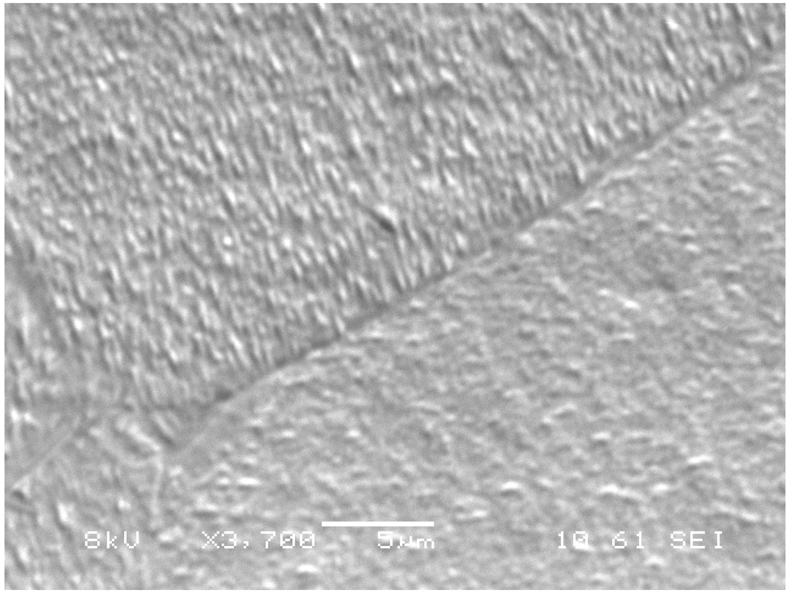
SEM trigonal nitrides

AFM nitrides height ~30-70 nm

Surface morphology (2)

-T=1000°C p_{N2}=10 mTorr time=2h



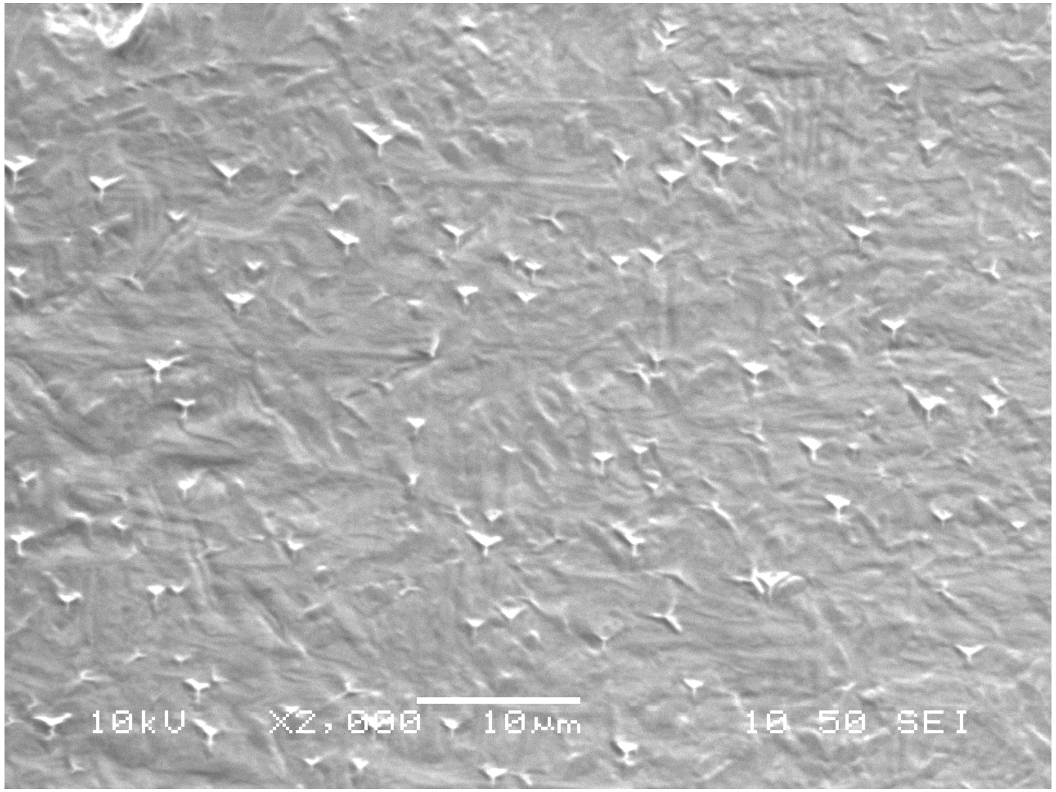


SEM -Trigonal nitrides observed

-Morphology dependent on the grain

Surface morphology (3)

-T=1000°C p_{N2}=50 mTorr time=2h



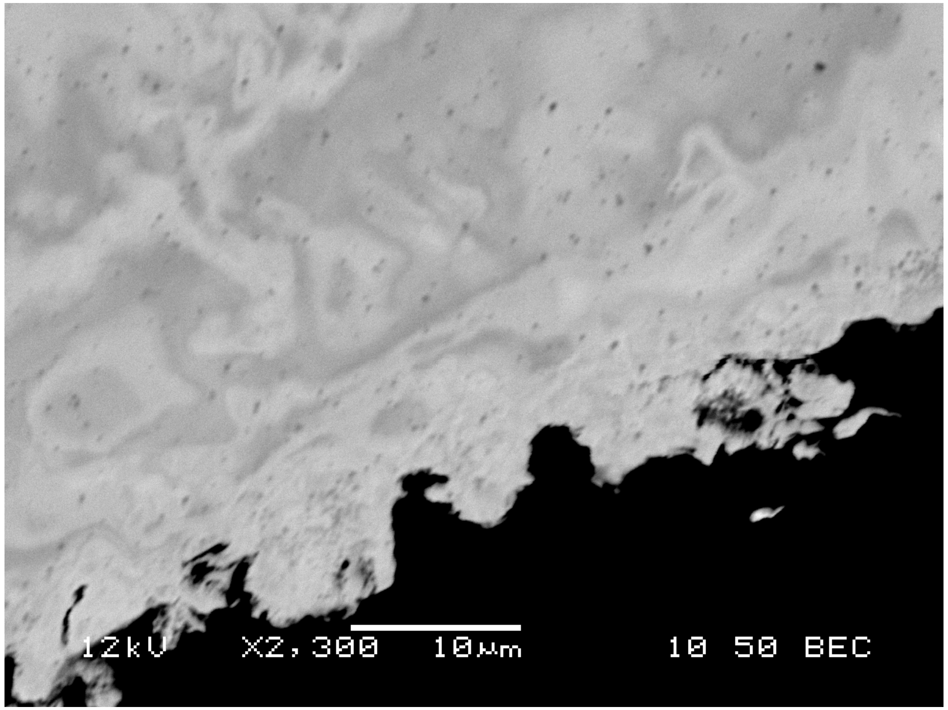
Surface morphology in cross section

SEM Cross section (1)

800°C 25 mTorr 30min

Contrast in the electronic image suggests the presence of an over-layer different from the bulk.

No EDS Nitrogen signal

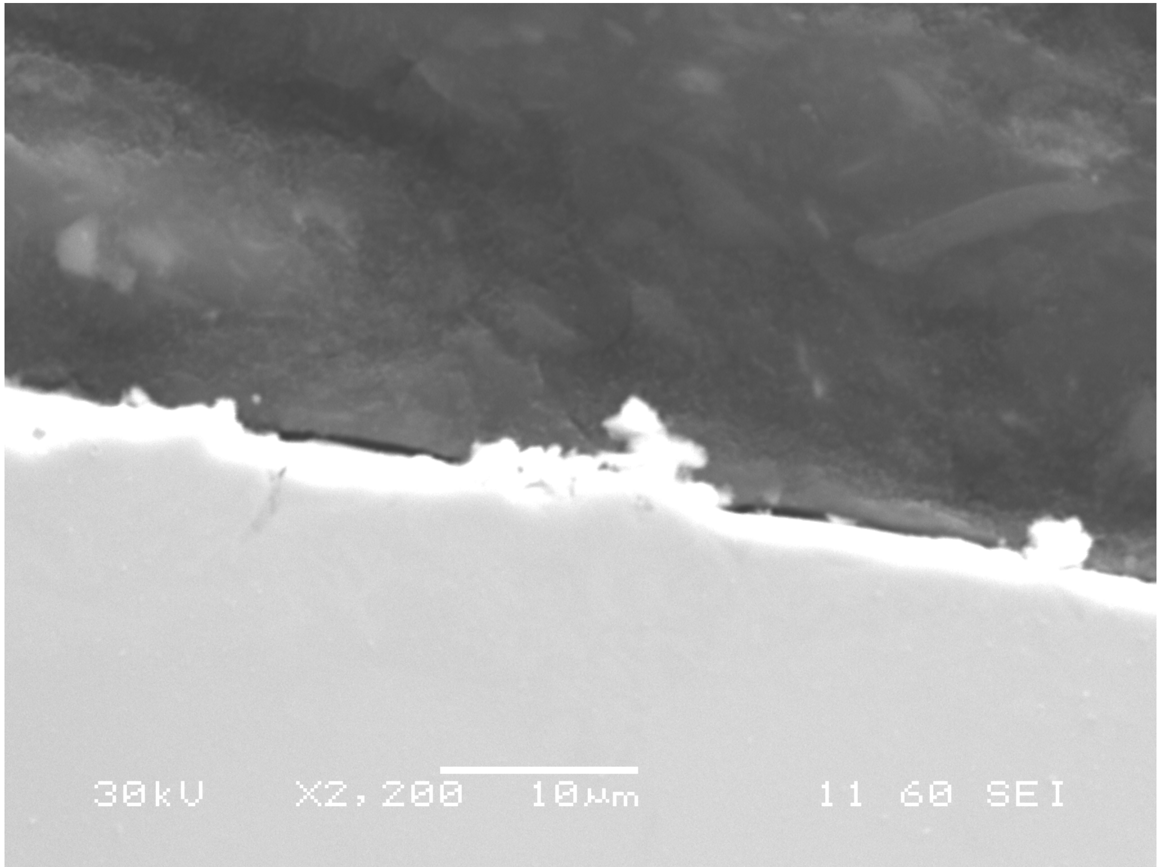


SEM Cross section (2)

1000°C 10 mTorr 2h

White layer of ~1μm only on one face of the sample.

No EDS Nitrogen signal



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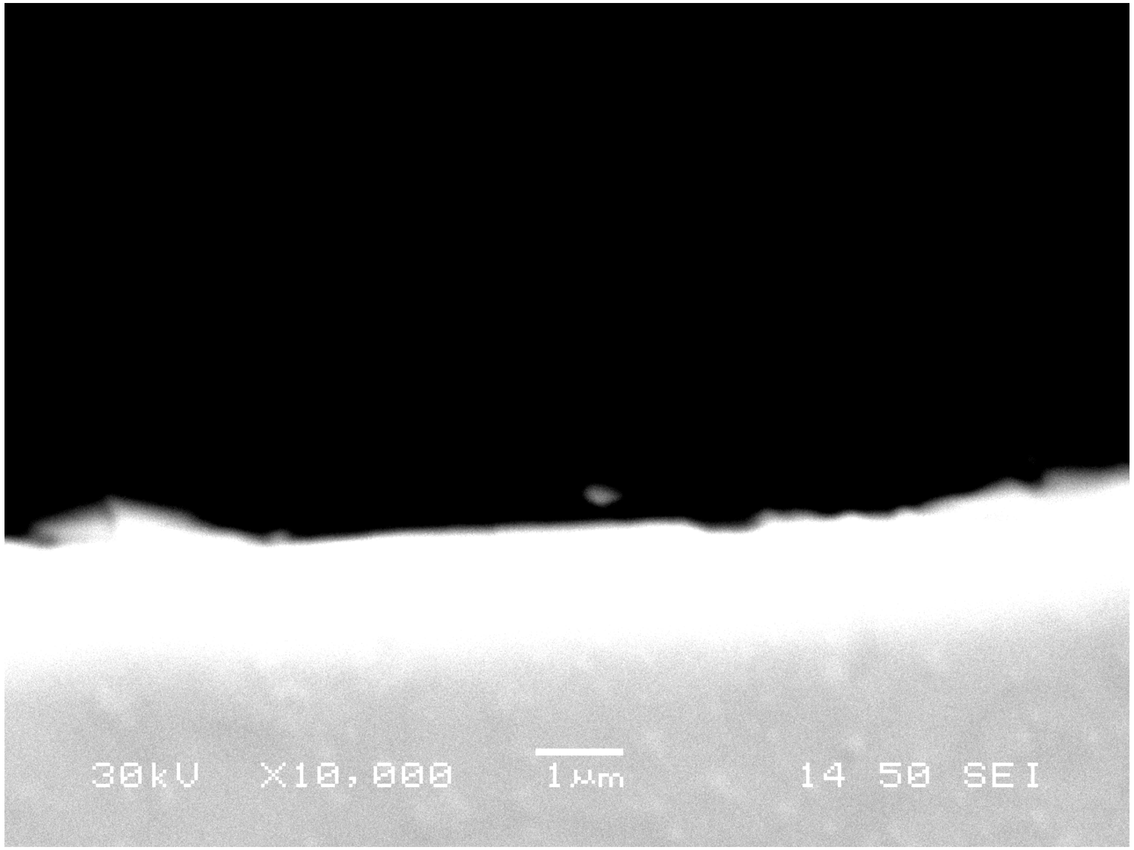
SEM Cross section (3)

1000°C 50 mTorr 2h

White layer of ~1μm only on one face of the sample.

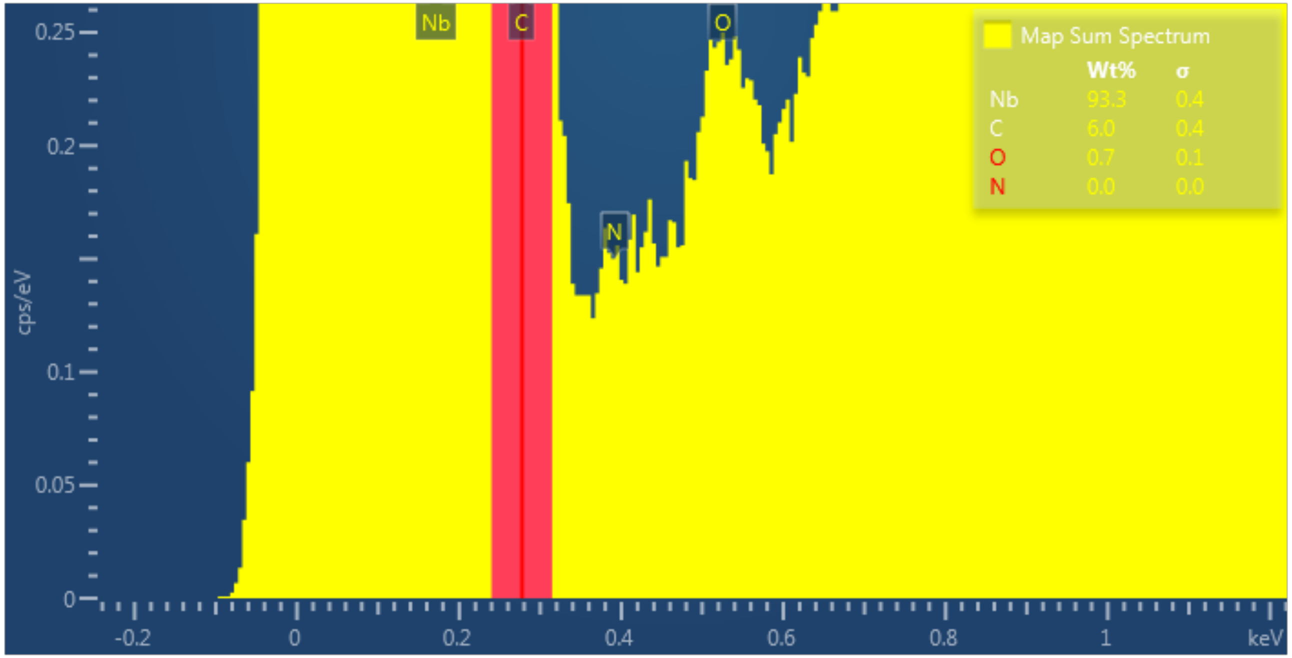
No EDS Nitrogen signal



Stoichiometry

Stoichometry (2)

Low sensitivity in all EDX measurements for nitrogen (both for top and cross-section measurements).



Nitrogen signal covered from carbon and oxygen's signals.

Superconducting properties

Superconductive properties (1)

1000°C 10 mTorr 2h

Comparison between:

-DC Magnetometry (bulk):

only one transition

$T_c \approx 9.2$ K

-AC Susceptibility (surface):

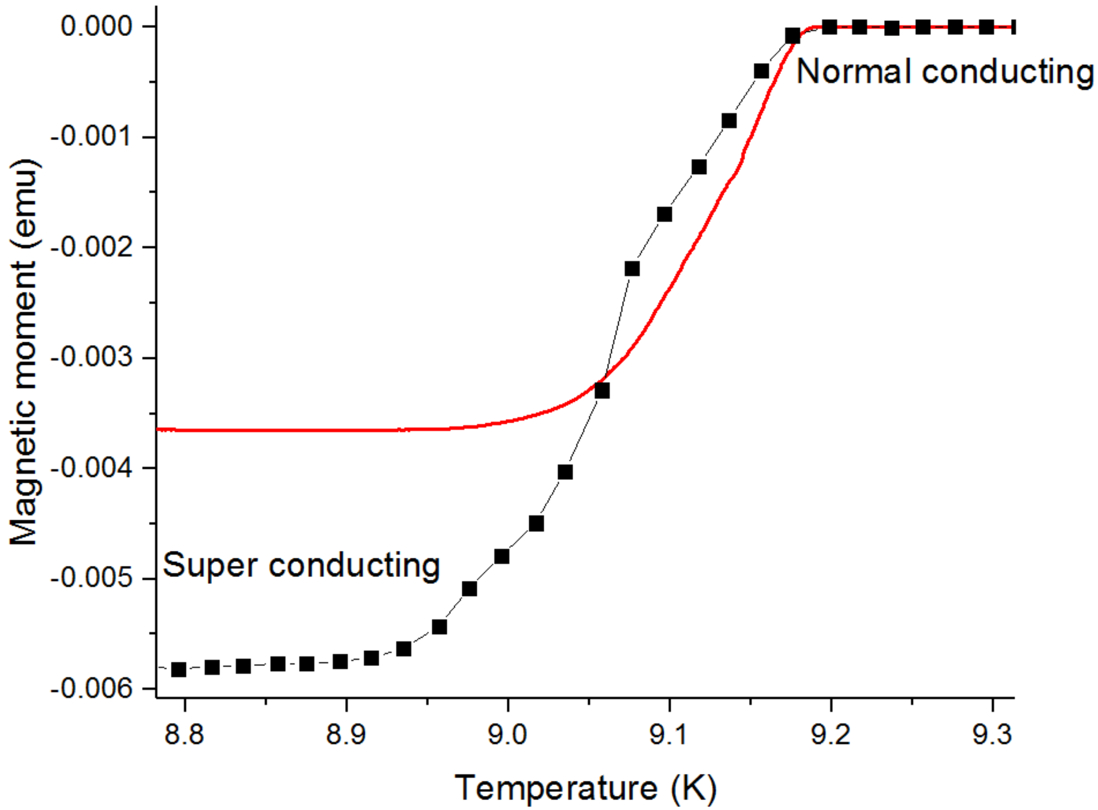
three transitions

$T_c \approx 9.0$ K

$T_c \approx 9.1$ K

$T_c \approx 9.2$ K

Poor SC surface phases.



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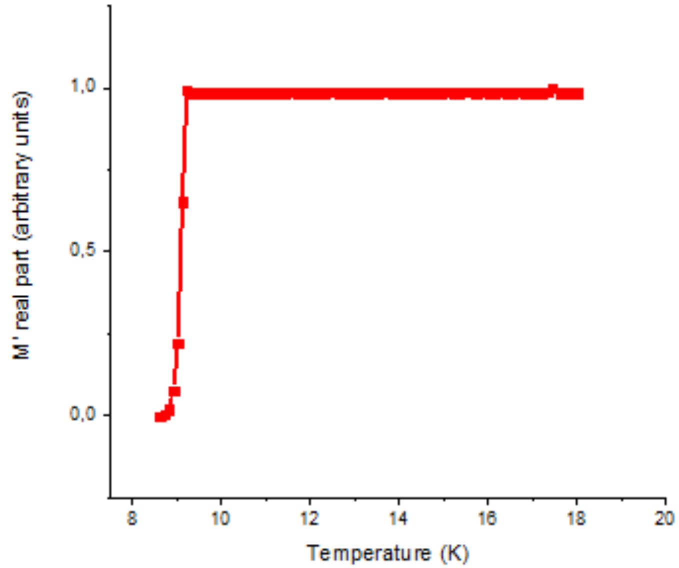
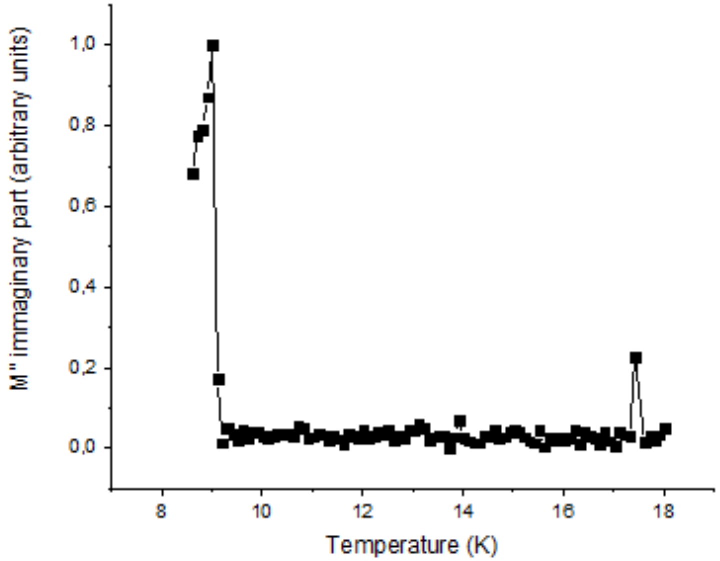
Superconductive properties (2)

1000°C 50 mTorr 2h



New signal at ~17 K



Could be systematic (1 pt)

More precise measurements:
8.5-10 K   15- 18.5 K

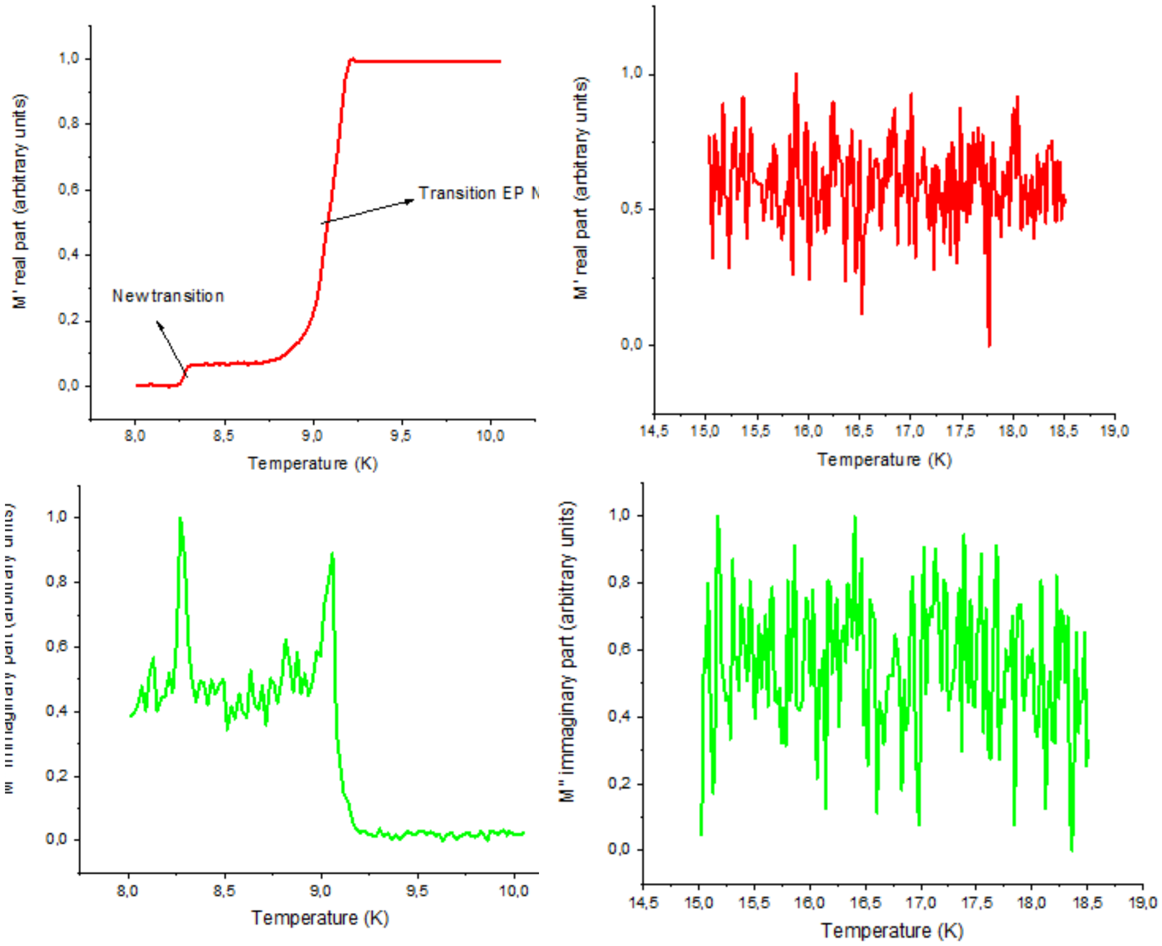
Superconductive properties (3)

1000°C 50 mTorr 2h

New transition:
Tc=8.33 K
Not observed before

No Nb_xN_y SC phases are known with this T_c.

New SC phase or
non stoichiometric NbN?



8.0-10 K **15-18.5 K**

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Summary NbN synthesis

1) Evidence of nitride layer formation, composition cannot confirmed with EDS (N signal too low)

2) Evident superconducting transition with Tc different from all the common nitride phases.

3) Deposition dependent on grain orientation.

Future:

- SIMS analysis to investigate stoichiometry.
- Study of the low T zone of the NbN phase diagram.

2) Direct observation of vortices in superconductors

Magnetic Force Microscopy (MFM) imaging

Purpose: measure the magnetic field near the surface.

It's fundamental to separate topological and magnetic effects.

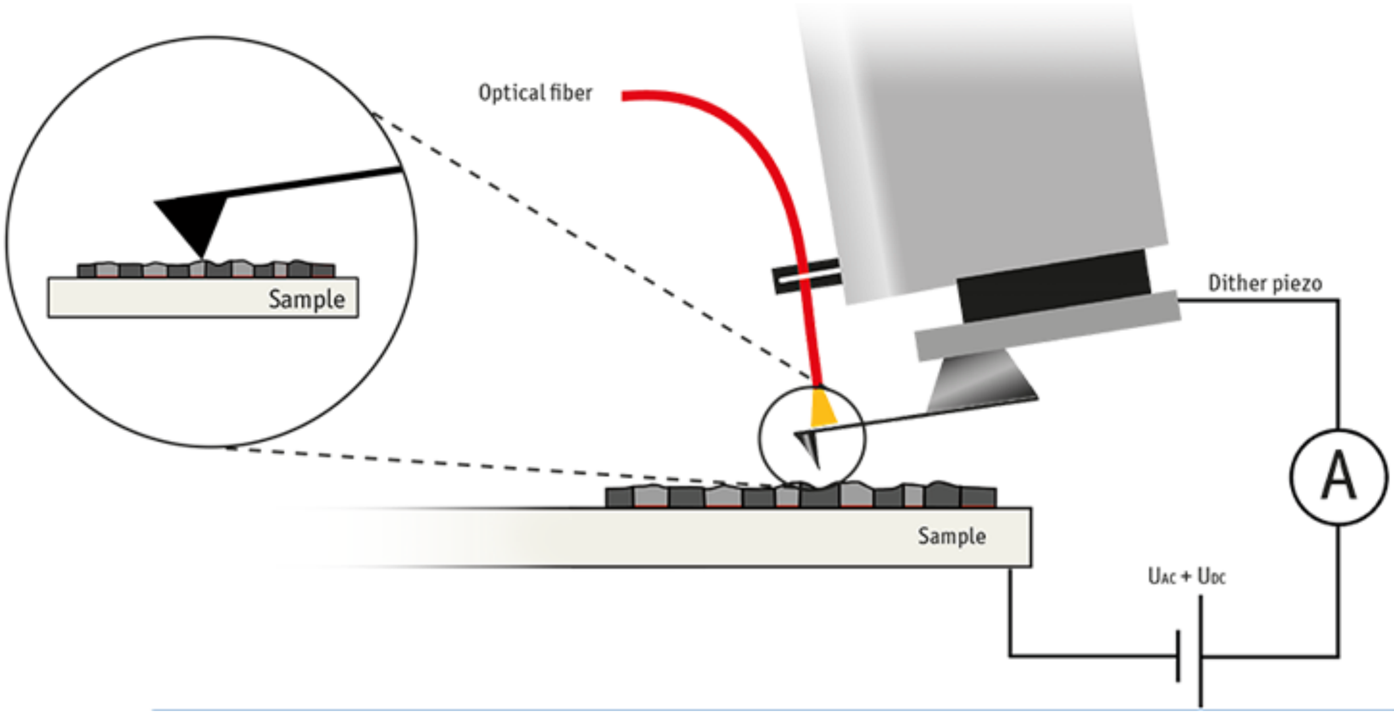
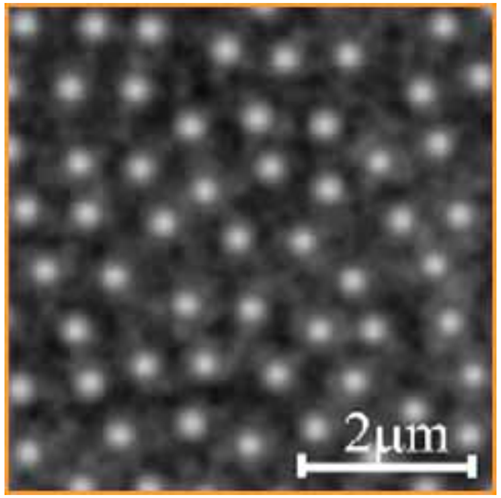


Figure of property of attocube systems

Clem's model in point dipole approximation

What we measure is the phase shift between the driving force and the oscillation of the tip in point dipole approximation:

$$\Delta\phi = -\frac{Q}{k}\frac{\partial F_z}{\partial z}$$



Europhys. Lett. 58, 582 (2002)

Than:

$$F_z = \frac{\partial E_{tip-sample}}{\partial z}$$

In MFM $E_{tip-sample}$ can be expressed as:

$$E_{tip-sample} = m_{tip}B_z$$

And so:

$$\Delta\phi = -\frac{Qm_{tip}}{k}\frac{\partial^2 B_z}{\partial z^2}$$

.

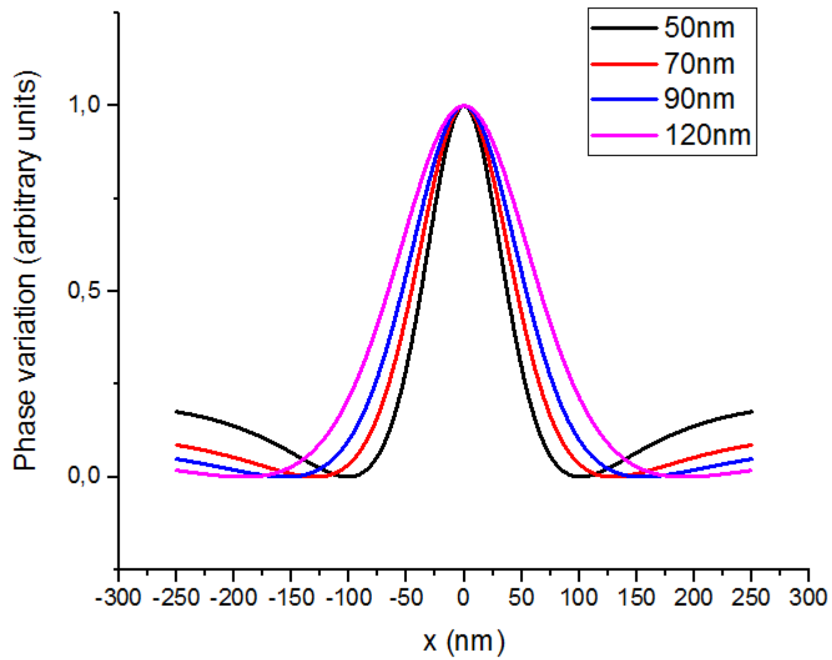
Clem's model in point dipole approximation

An expression for the B field is obtained from Clem Model:

$$\frac{\partial^2 B_z(z,r)}{\partial z^2} = \frac{\Phi_0 k^2}{2\pi\lambda^2}\int_0^\infty dk\,\frac{k\,J_0(kr)}{k^2+\lambda^{-2}}\,\frac{\sqrt{k^2+\lambda^{-2}}}{k+\sqrt{k^2+\lambda^{-2}}}\,e^{-kz}$$

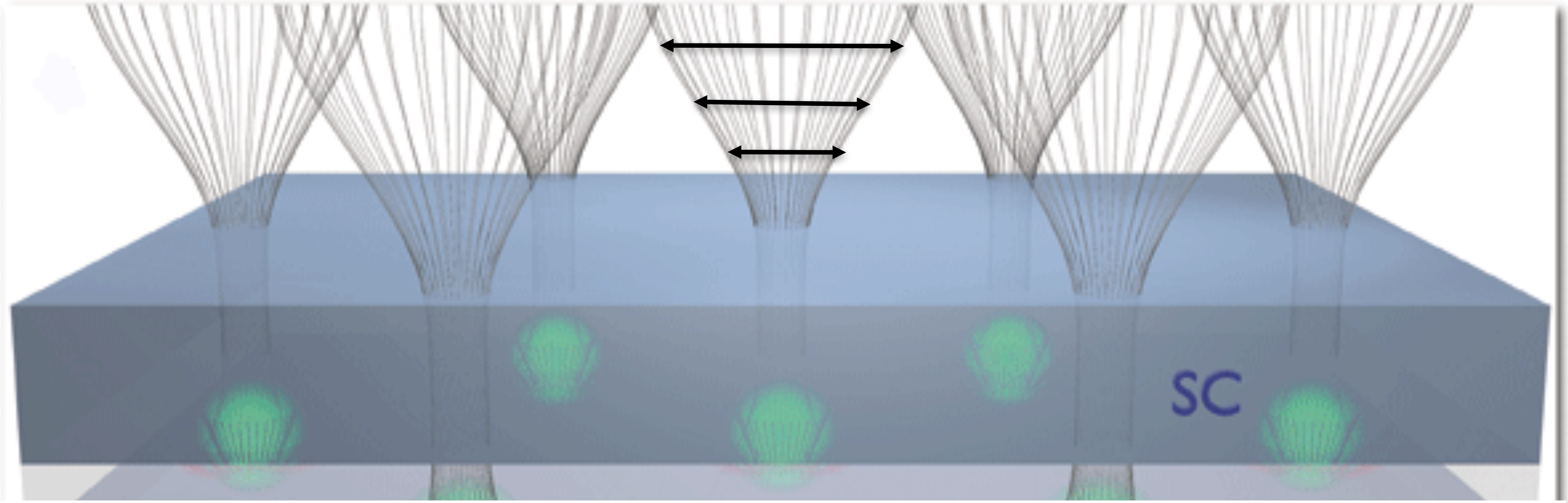
We look at relative values of $\frac{\partial^2 B_z(z,r)}{\partial z^2}$ so we can normalize the data.

In figure different profiles calculated for different values of z.



Carneiro, G., & Brandt, E. H. (2000). Vortex lines in films: Fields and interactions. *Physical Review B, 61*, 6370-6376.

Vortices size at different MFM's scan height



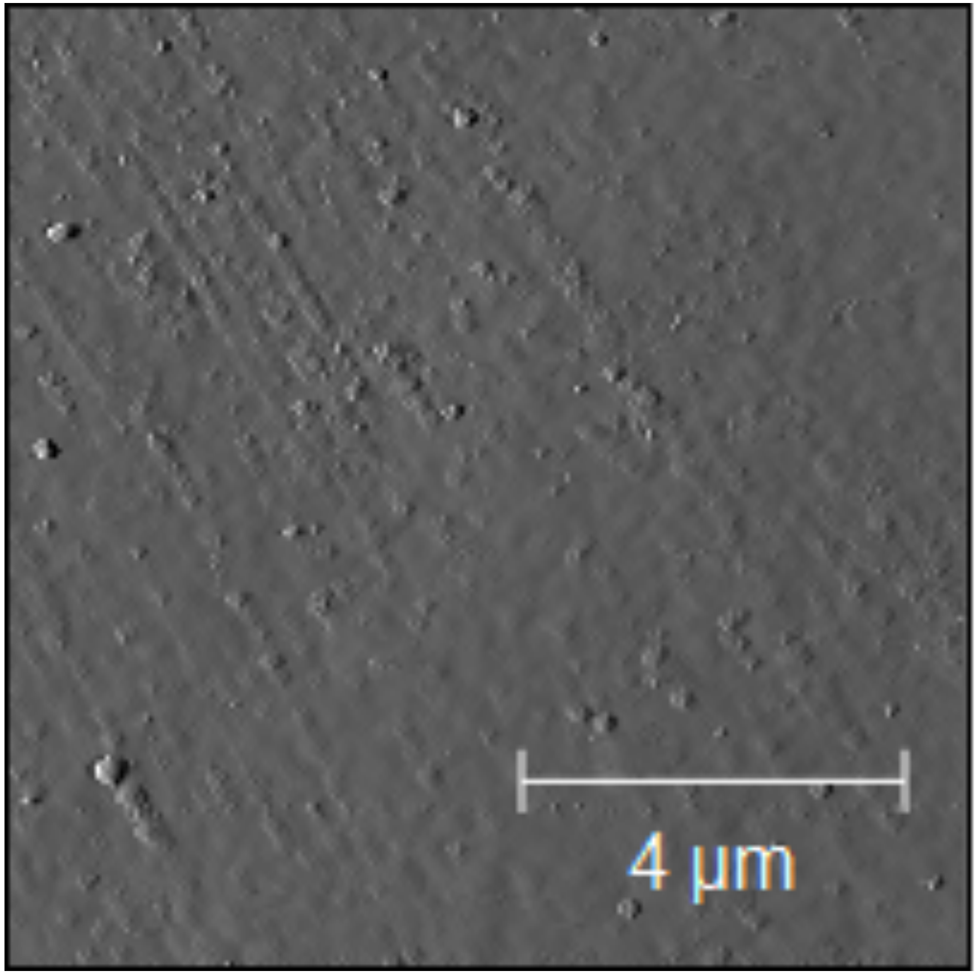
vortex profile width ∝ Scan height

Figure taken by T. G. Rappoport, L. Ghivelder, J. C. Fernandes, R. B. Guimaraes, M. A. Continentino, [Phys. Rev. B 75, 054422 (2007)]

Experimental data

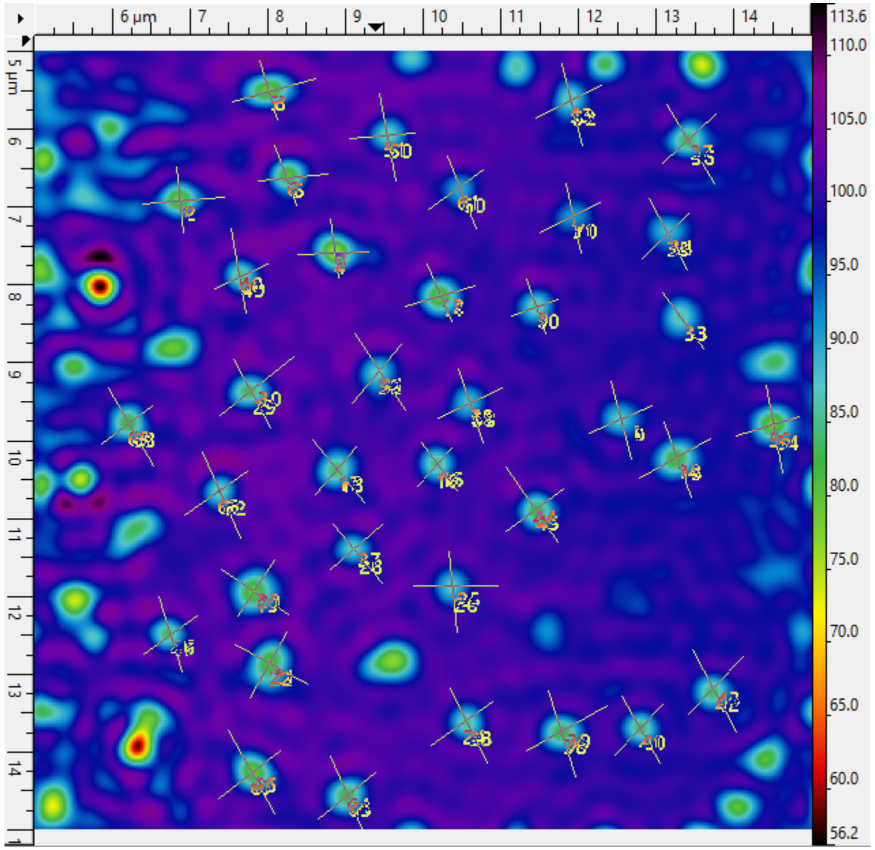
Sample: Nb Electro polished

Surface Nb EP AFM

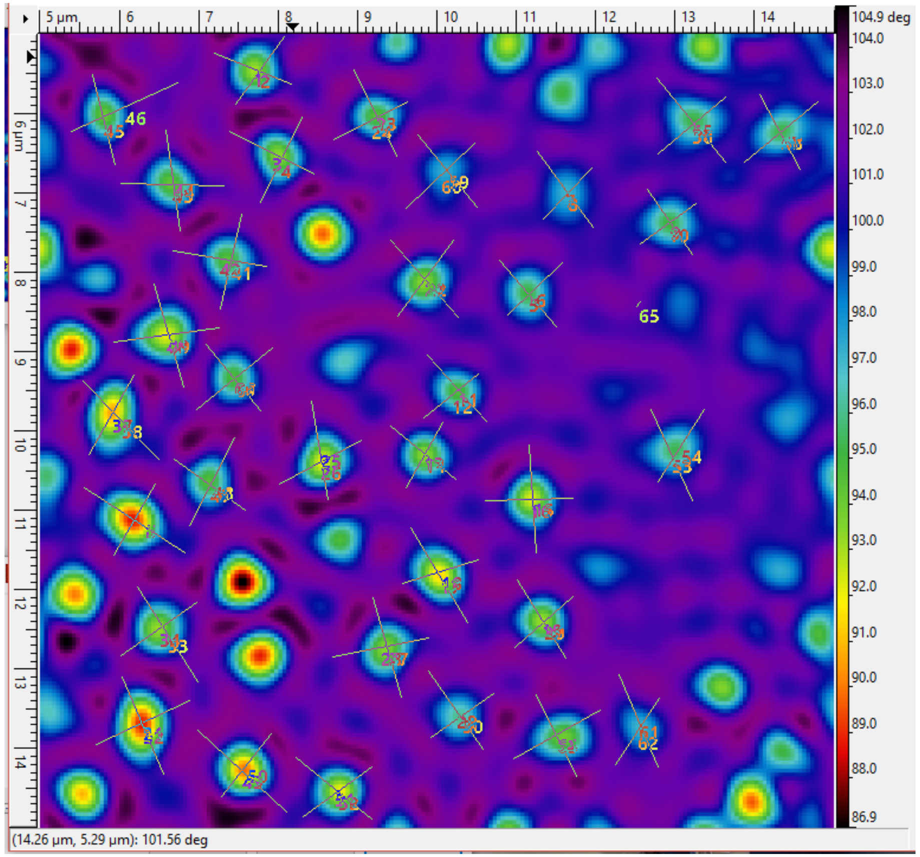


Vortices size at different MFM's scan height

T=4 K zero field cool mode B=30 mT (fixed)



50 nm MFM Scan Height



70 nm MFM Scan Height

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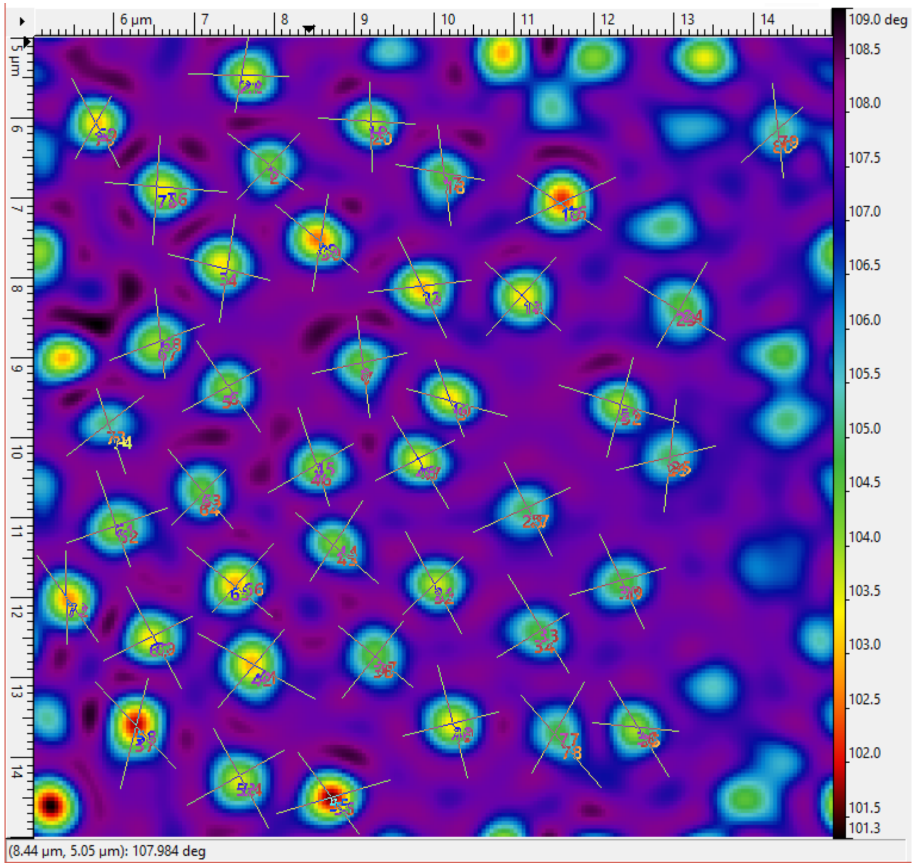
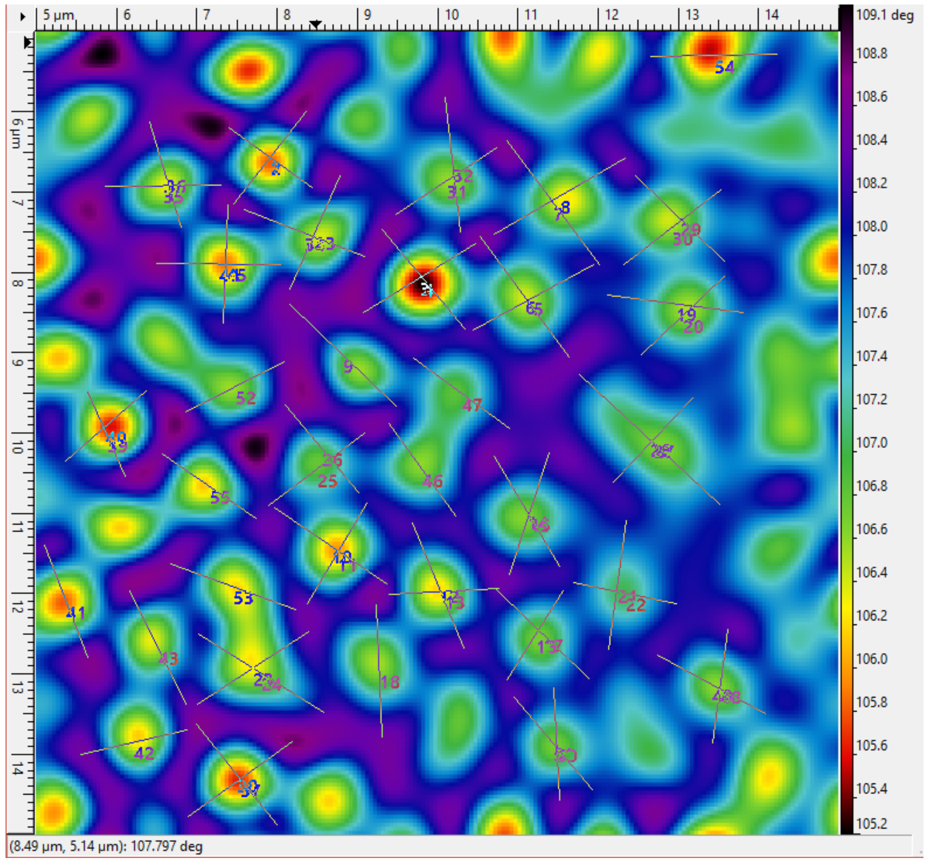
Vortices size at different MFM's scan height

T=4 K zero field cool mode B=30 mT (fixed)



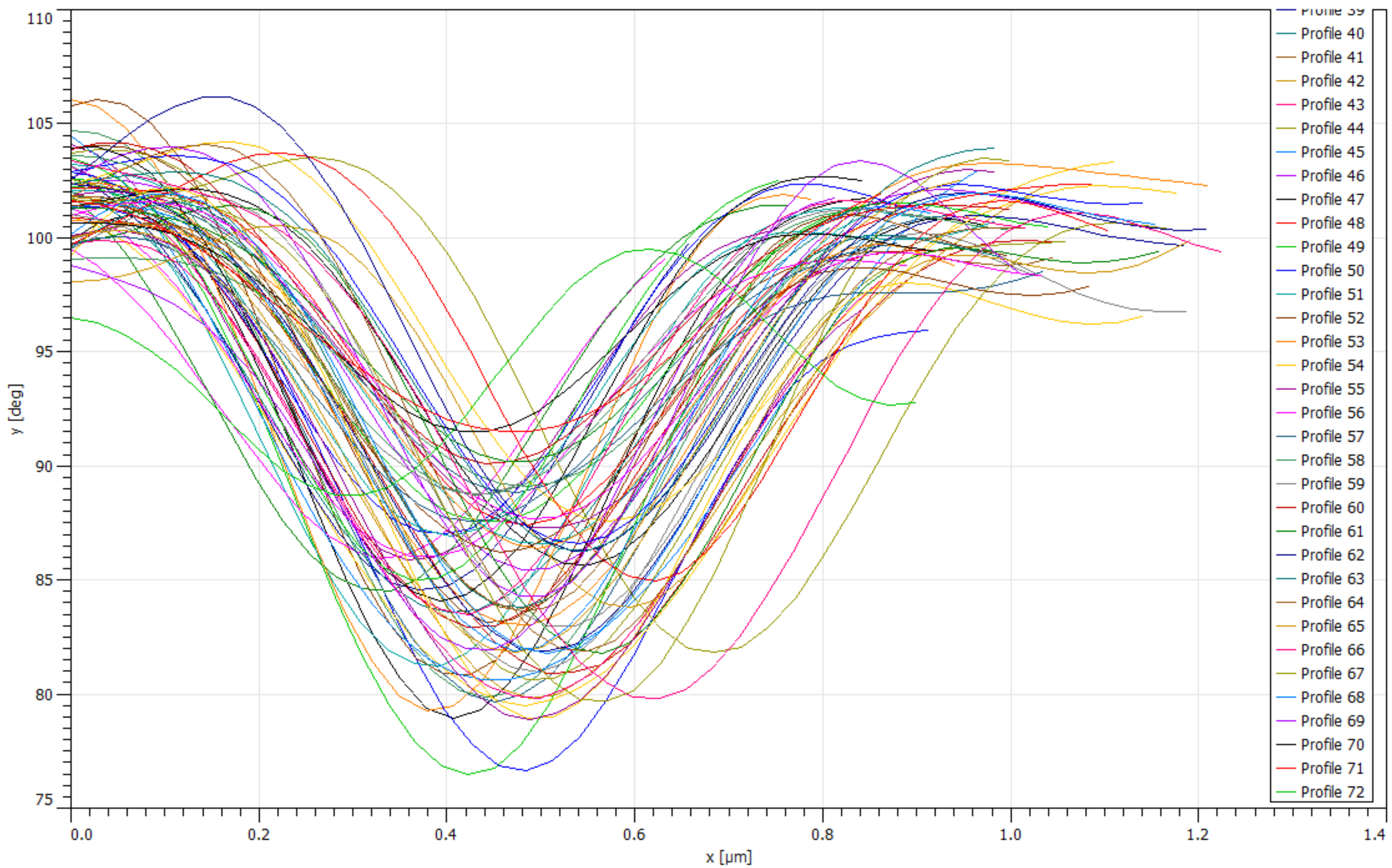
90 nm MFM Scan Height



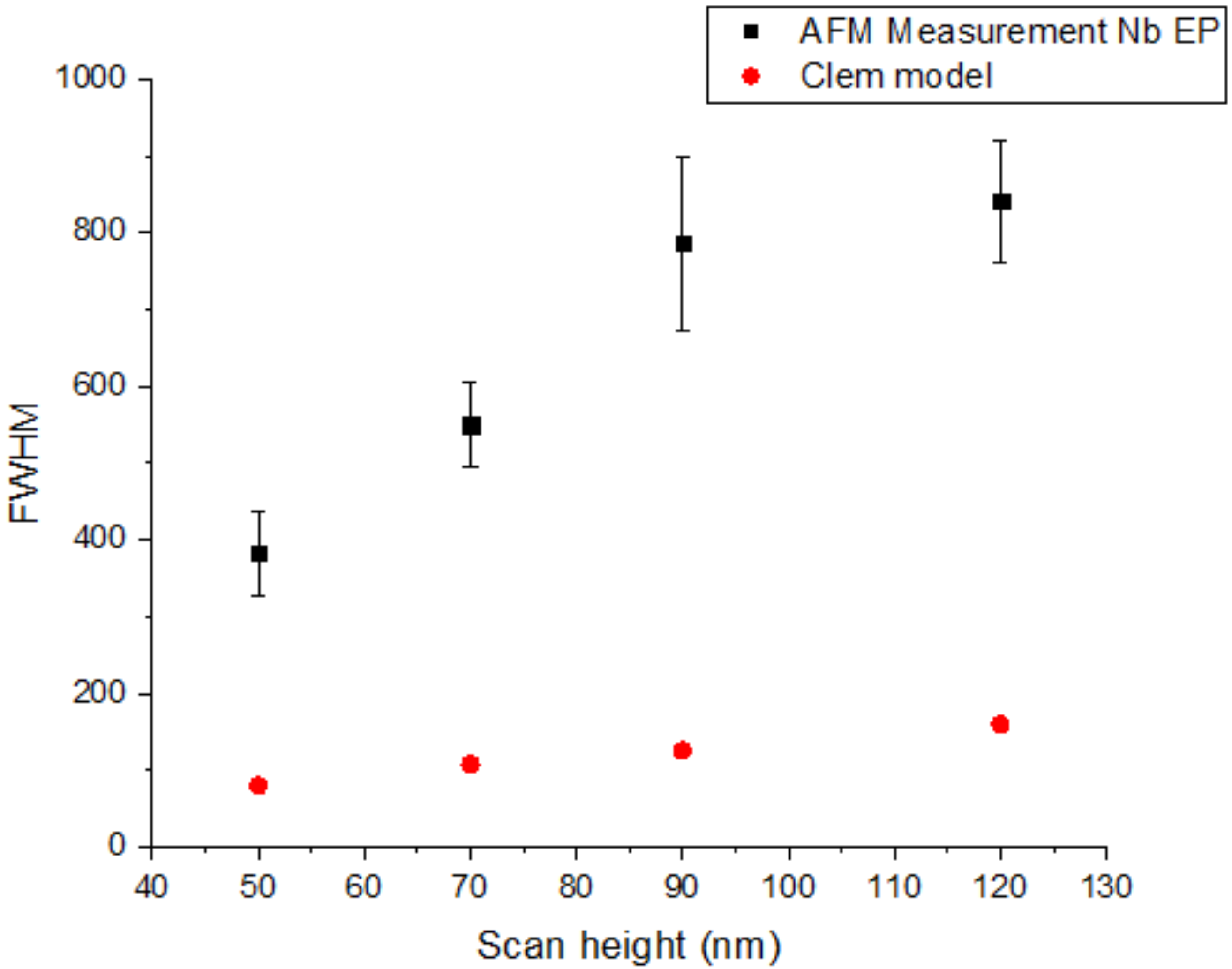
120 nm MFM Scan Height

Vortex profile fitting

FWHM values extracted with multi-gaussian fit.



Experimental data and simulated data

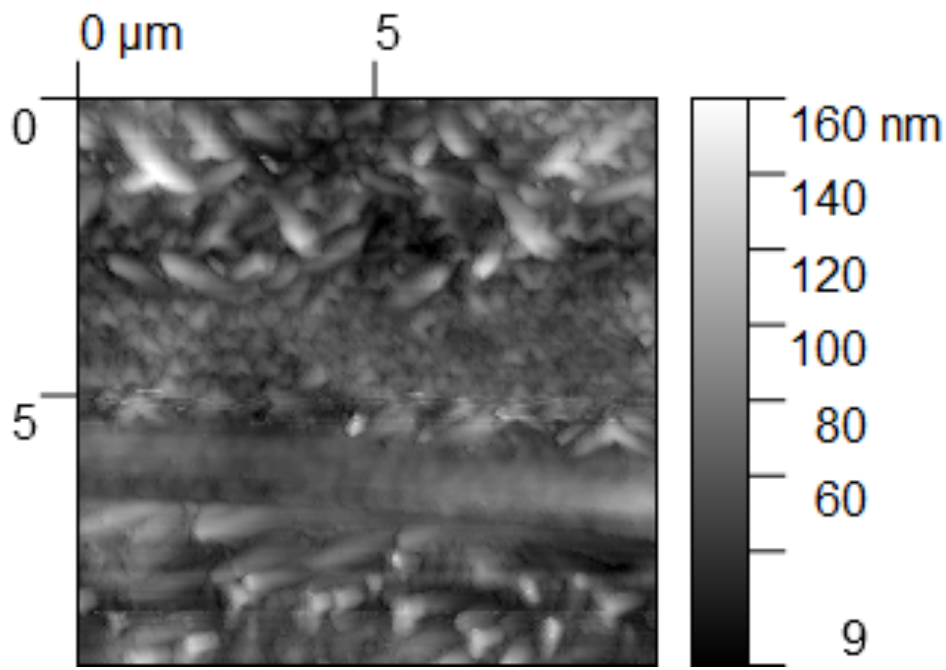


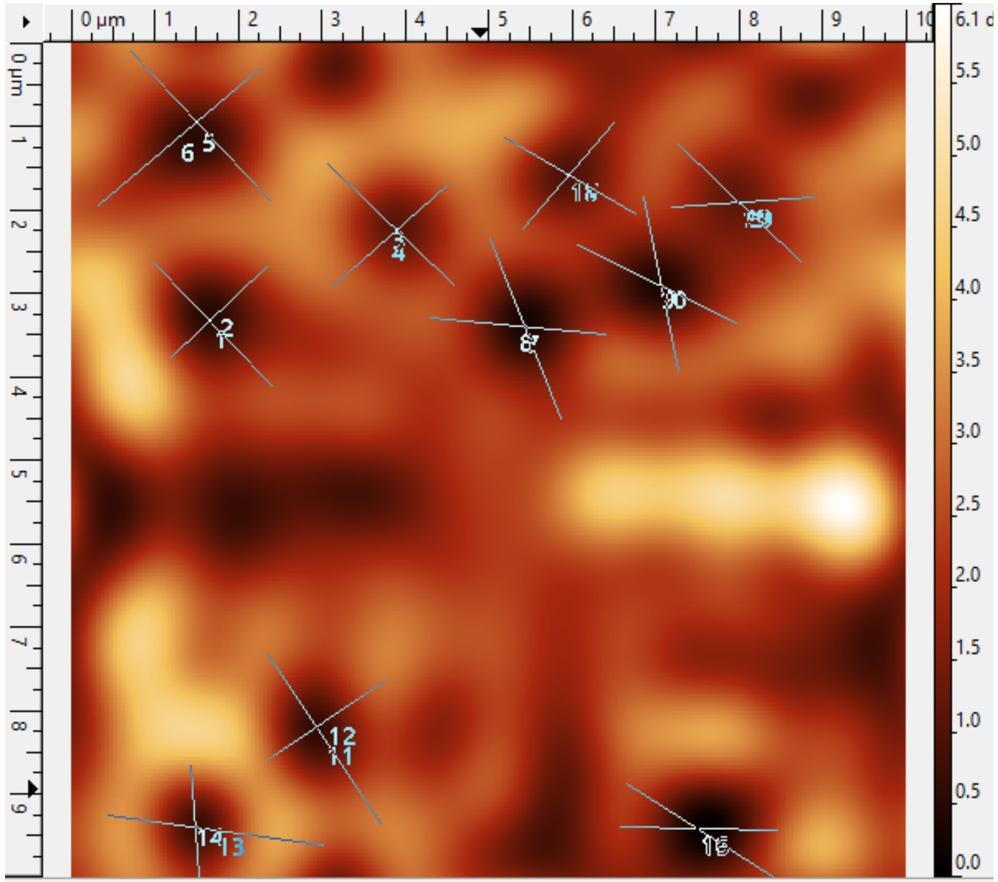
Similar trends

High disagreement of FWM values.

Point dipole tip is a rough model.

Dependence of vortex profile on surface





Average FWHM = 1,2 μm

Double than EP Nb!
Surface effects matters.

70 nm MFM Scan Height

Summary of vortices study

1) MFM is a good method to map the vortices.

2) FWHM depends strongly on **surface** London penetration depth. It's possible to measure surface properties separately from bulk properties.

Future: the point dipole tip-sample interaction model is too rough. A more sophisticated model has to be used:

MFM image → Convolution of Tip+sample

Thanks for the attention