

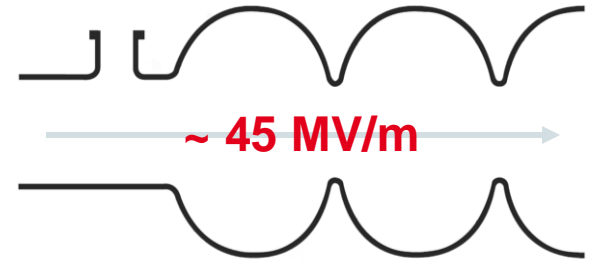
Characterization of PEALD Coated Thin Films for SRF Cavity Research

Lea Preece - on behalf of the SRF R&D Team Hamburg

11th International Workshop on Thin Films and and New Ideas for Pushing the Limits of RF Superconductivity – TFSRF2024 – Sept. 19, 2024

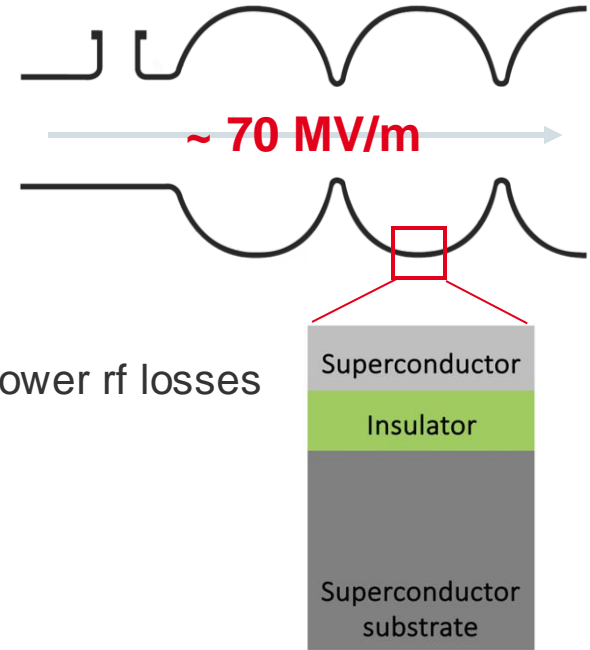
➤ Motivation

- SRF cavities approach thermodynamic limit of niobium
- **Superconductor-Insulator-Superconductor (SIS) multilayers**
 - Theory shows **potential to enhance E_{acc}** and achieve lower rf losses
- SIS coating of cavities creates new **challenges**
 - Experimental realization
 - Material properties of thin multilayer systems



➔ Motivation

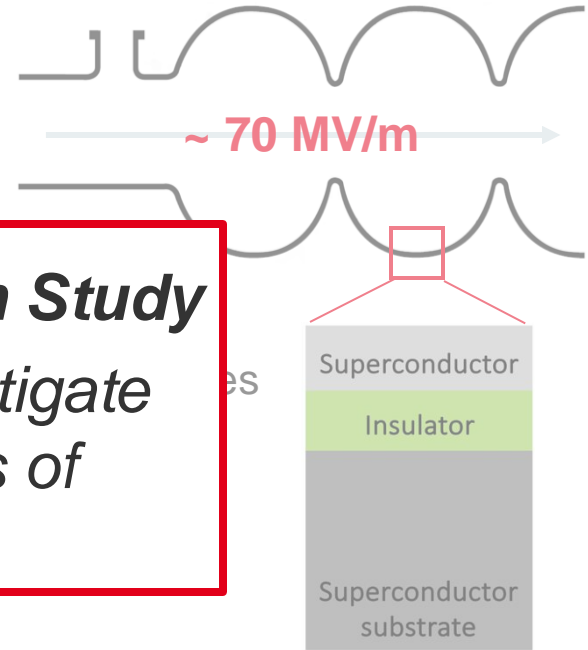
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- Superconductor multilayers
 - Theory shows
- SIS coating of cavities
 - Experimental
 - Material properties of thin multilayer systems

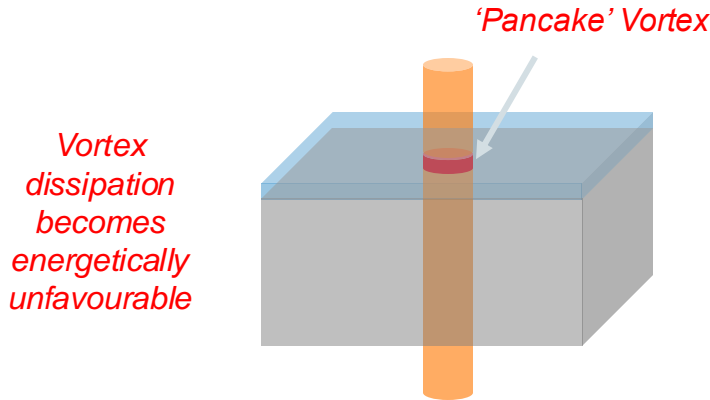
Magnetic Characterization Study
⇒ *comprehensively investigate the magnetic properties of SIS layers*



Thin Films and Screening Currents

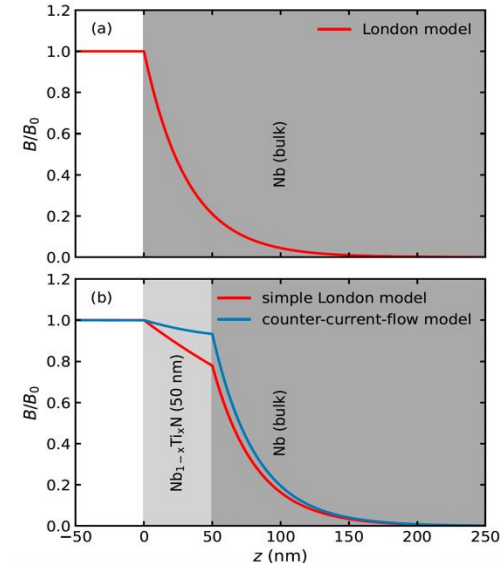
References:
 A. Gurevich, *APL* 88 (2006),
 T. Kubo, *SUST* 30 (2017)

- Gurevich: Vortices are energetically suppressed in SC thin films for $d < \lambda_L$



- Insulator as vortex barrier so that global vortex penetration is prevented

- Kubo: Vortex penetration shifts to $H_{sh} > H_{C1}$ through “**counter current**” at interfaces



[Asaduzzaman M. et al., Direct measurement of the Meissner screening profile in superconductor-superconductor bilayers using low-energy muon spin rotation (2023)]

SIS Multilayer Theory

- Top superconductor sees majority of rf field
→ **Surface resistance improves**
- Insulating layer creates more interfaces
→ **More counter currents**
- Layers must be thinner than $\lambda_{L,Top}$
→ **RF field is affected by counter currents**

→ Optimal layer thickness d_S depends on λ_L and H_{C1} of the used bulk and layer materials

↘ **Niobium**
(9.27 K)

↘ cubic high- T_C superconductors **NbN** (17.3 K) and **NbTiN** (17.8 K)
AlN as insulator

Reference:
T. Kubo, SUST 30 (2017)

Theoretical maximum surface field a SIS coated cavity can withstand:

$$H_{\max} = \min\{\tilde{\gamma}_1^{-1} H_{\text{sh},S}, \tilde{\gamma}_2^{-1} H_{\text{sh},\text{sub}}\}$$

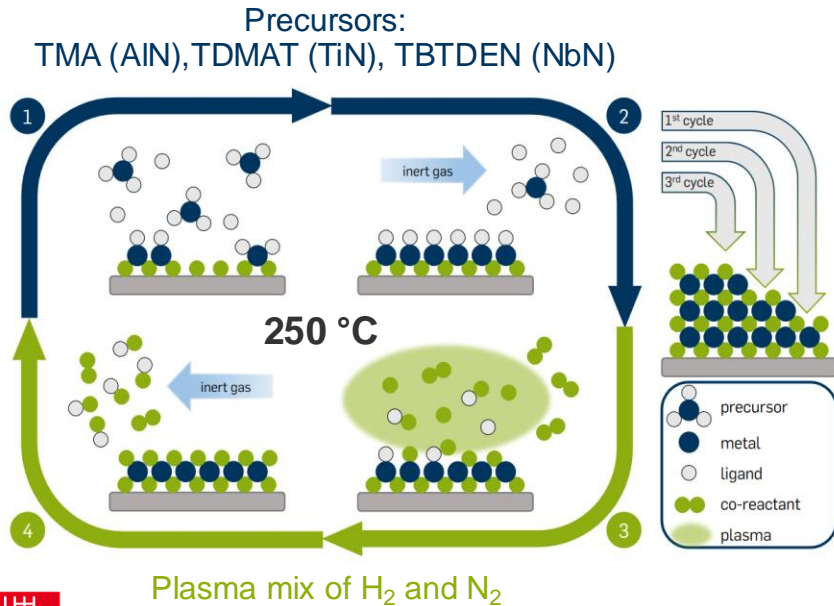
$$\tilde{\gamma}_1 = \frac{\sinh \frac{d_S}{\lambda_S} + \frac{\lambda_{\text{sub}} + d_I}{\lambda_S} \cosh \frac{d_S}{\lambda_S}}{\cosh \frac{d_S}{\lambda_S} + \frac{\lambda_{\text{sub}} + d_I}{\lambda_S} \sinh \frac{d_S}{\lambda_S}}$$

$$\tilde{\gamma}_2 = \frac{1}{\cosh \frac{d_S}{\lambda_S} + \frac{\lambda_{\text{sub}} + d_I}{\lambda_S} \sinh \frac{d_S}{\lambda_S}}$$

Literature values

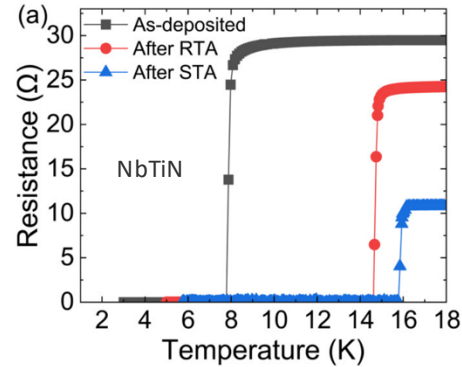
Sample Preparation

Plasma-Enhanced Atomic Layer Deposition (PEALD)



Post-Deposition Annealing to improve T_C and H_{C1}

- Recrystallisation and degas of impurities



[Gonzalez.I. et al., J. Appl. Phys. 134, 159902 (2023), 035301 (2023)]

$$\mu_0 H_{C1} = 15 \text{ mT}$$

$$\mu_0 H_{C1} = 81 \text{ mT}$$

$$\mu_0 H_{C1} = 98 \text{ mT}$$

$$\mu_0 H_{C1, \text{bulk}} \approx 33 \text{ mT}$$

900 °C annealing for 1 hour with controlled heating and cooling rates

Characterization Measurements

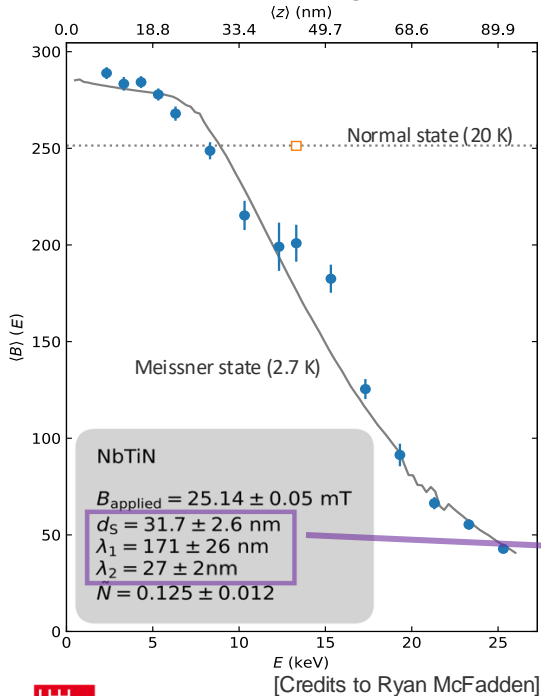
- **X-Ray Reflectivity (XRR) measurement**
 - Determine the PEALD growth per cycle (GPC)
- **Low Energy Muon Spin Rotation (LE- μ SR) measurement**
 - Determine the London penetration depth λ_L
- **T_C measurements**
 - Contactless Inductive T_C measurement on Nb
 - Physical properties measurements on Si (electrical transport, VSM)
- **Magnetic Flux Lens (MFL) measurement**
 - Measure flux expulsion (and T_C)

See also **Md Asaduzzaman's talk** on „Depth-resolved characterization of superconductor-superconductor bilayer properties beneficial for SRF applications“ ealier today

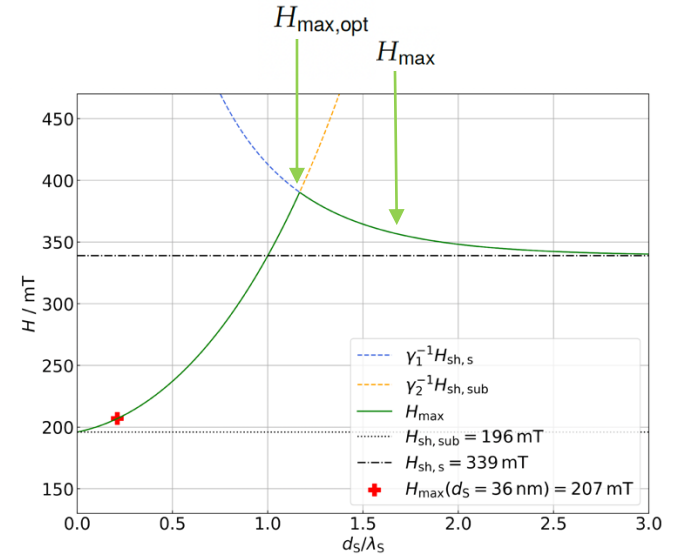
See also **Daniel Turner's talk** on „Enhancement of magnetic flux expulsion in multilayer structures“ ealier today

λ_L and the Estimation of H_{sh}

Meissner Screening Profile



- SS bi-layer Nb/NbTiN ($d \sim 36$ nm)
- Expected decrease in B with increasing E and z
- Kubo's counter current model used for fit
- H_{max} **estimated** for Nb/NbTiN SS bi-layer

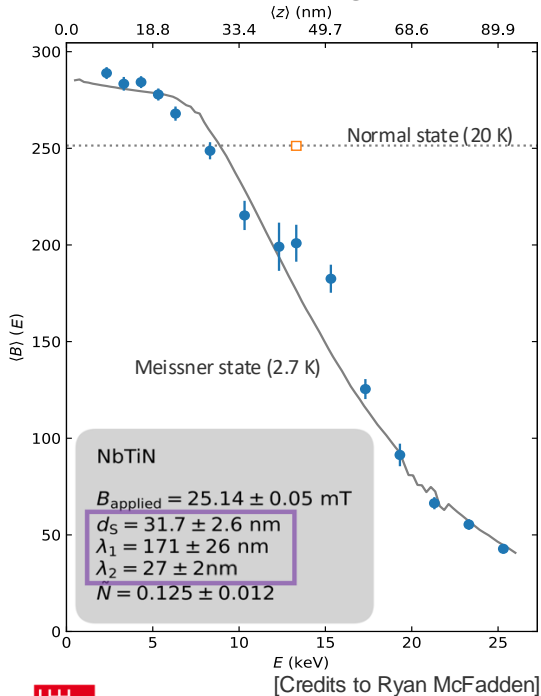


Material	Fit	Literature		Calculated			
	λ (nm)	λ_L (nm)	ξ_0 (nm)	κ (nm)	H_{c1} (mT)	H_c (mT)	H_{sh} (mT)
NbTiN	171	150 [65]	5 [65]	43	24	452	339
Nb	27	28 [56]	35 [65]	0.82	(no calc.)	200 [65]	196

Clean limit of λ_L

Discussion on Meissner Profile and H_{max}

Meissner Screening Profile

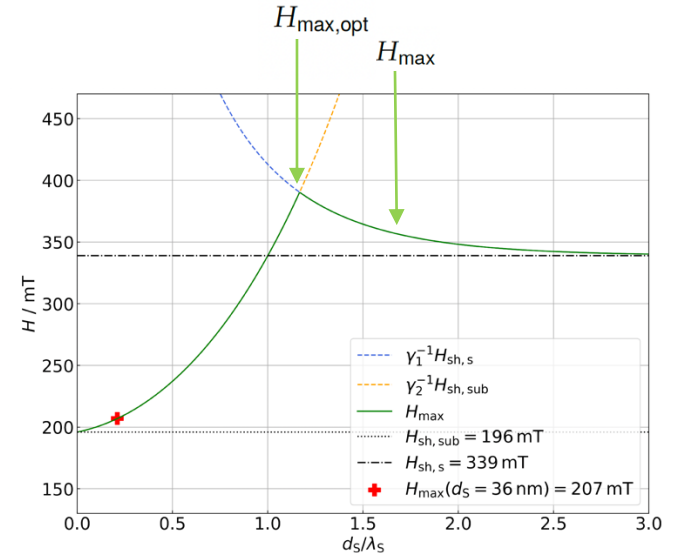


- H_{max} **estimate** visualizes Kubo's prediction

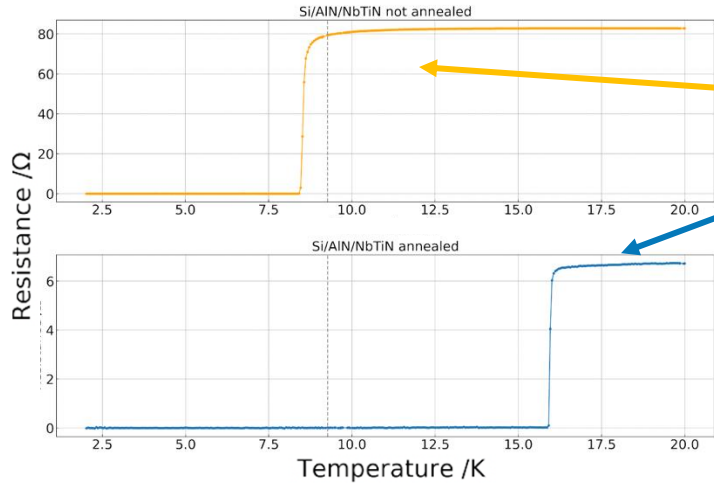
- $H_{max} > H_{sh,Nb}$
- SIS multilayers needed!

- Kubo's counter current model suitable to determine λ_L

- Estimate based on Meissner Fit → improvable!
- Illustrates principle of field enhancement in an SS bi-layer



T_C Measurements on Si Substrate



Sample	Ref. to	Measurement	T_C /K	ΔT_C /K
Si/AIN/NbTiN not annealed	DESY_3_1 DESY_5_1 Tc_5_1	Electr. Transport	8.402 ± 0.271	0.931
Si/AIN/NbTiN annealed	DESY_3_2 DESY_5_2 Tc_5_2	Electr. Transport	15.960 ± 1.952	0.106
		VSM	15.566 ± 0.242	0.666
Si/NbTiN annealed	DESY_2_2 DESY_6_2 Tc_6_2	VSM	14.948 ± 0.409	0.384
Si/AIN/NbN annealed	DESY_4_2	Electr. Transport	2.732 ± 0.070	0.880

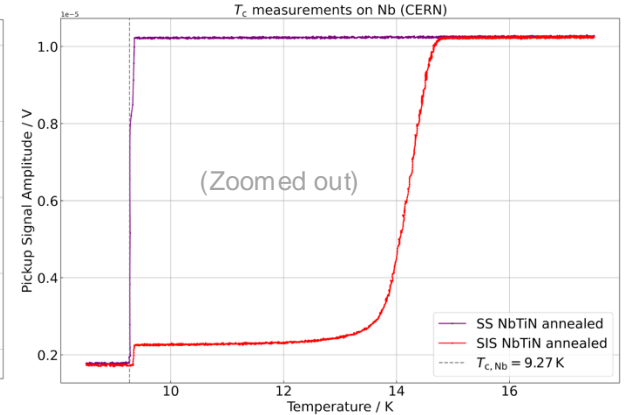
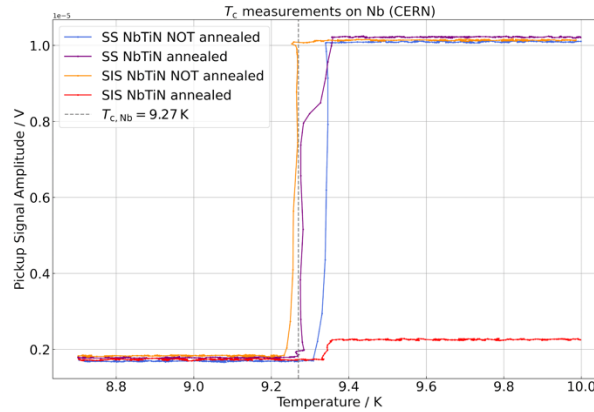
Highest
value
measured

Not the
same for
Nb
substrate!

- T_C can be clearly assigned to the SC thin film
- Highest T_C for **Si/AIN/NbTiN** in both measurement modes
- Measurement of an SIS Si/AIN/NbN sample \rightarrow very low T_C

T_C Measurements on Nb Substrate

- As-deposited: T_C not higher than 7 to 8 K
- Annealed SS with NbTiN behaves similar as unannealed samples
 - $T_C \sim T_{C,Nb}$
 - Two transitions?



[Measurement Courtesy of Erwan Rechtes, CERN cryolab]

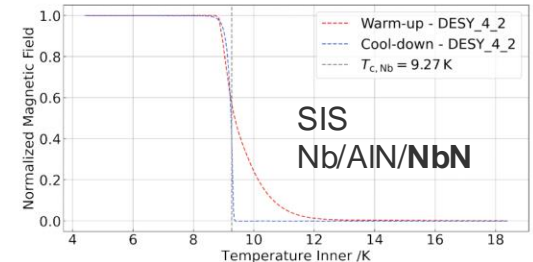
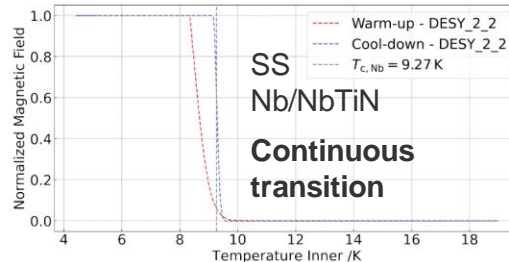
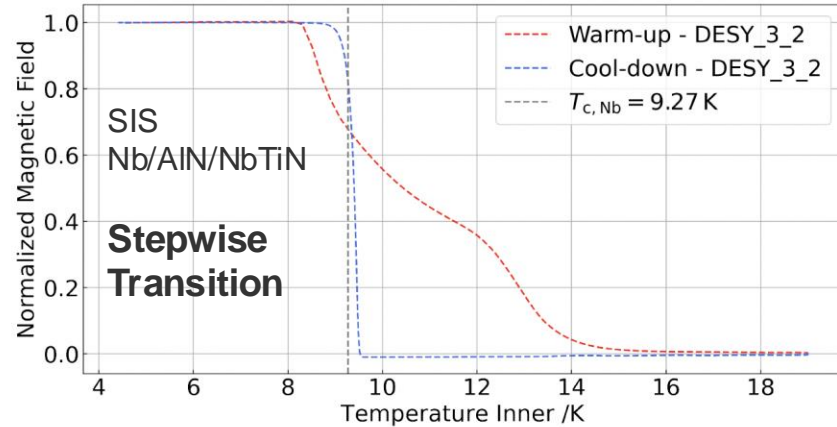
- Only **annealed SIS with NbTiN**
 - shows clear stepwise transition
 - reaches high T_C

Sample	Structure	$T_{c(1)}/K$	$\Delta T_{c(1)}/K$	$T_{c(2)}/K$	$\Delta T_{c(2)}/K$
Tc_6_1	Nb/NbTiN not annealed	9.339 ± 6.890	0.032	-	-
Tc_5_1	Nb/AlN/NbTiN not annealed	9.259 ± 6.998	0.023	-	-
Tc_6_2	Nb/NbTiN annealed	9.276 ± 10.663	0.044	9.340 ± 0.326	0.033
Tc_5_2	Nb/AlN/NbTiN annealed	9.343 ± 1.407	0.0144	14.196 ± 0.014	0.835

See also **Daniel Turner's talk** on „Enhancement of magnetic flux expulsion in multilayer structures“ ealier today

T_c Measurements with MFL

- Annealed Nb SIS and SS samples
- Only **SIS with NbTiN**
 - shows stepwise transition
 - reaches high T_c
- No double transition or significant increase in T_c for
 - SS Nb/NbTiN
 - SIS Nb/AlN/NbN
- All T_c measurements deliver matching results!



[Credits to Daniel Turner]

Magnetic Flux Expulsion

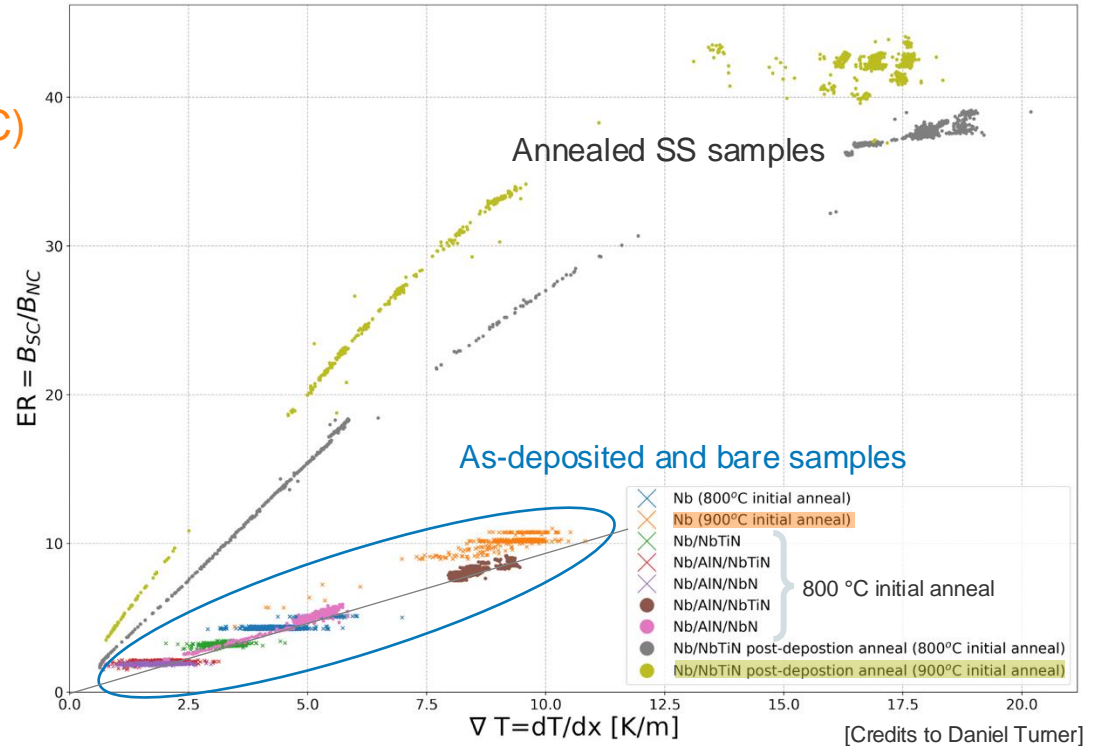
See also *Daniel Turner's talk* on „Enhancement of magnetic flux expulsion in multilayer structures“ ealier today

As-deposited SIS and SS thin films

- $ER(\text{Nb}@900\text{ °C}) > ER(\text{Nb}@800\text{ °C})$
- Limited spatial thermal gradient

Annealed SS thin films

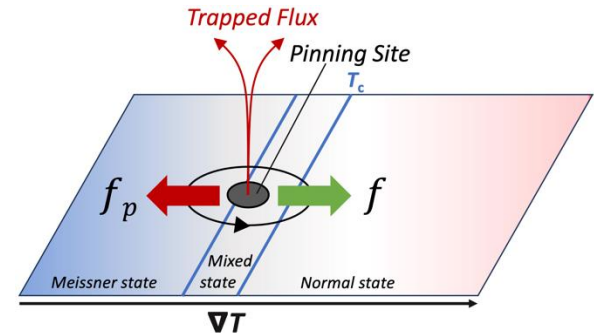
- Much higher flux expulsion
- Greater expulsion for 900 °C initial anneal



[Credits to Daniel Turner]

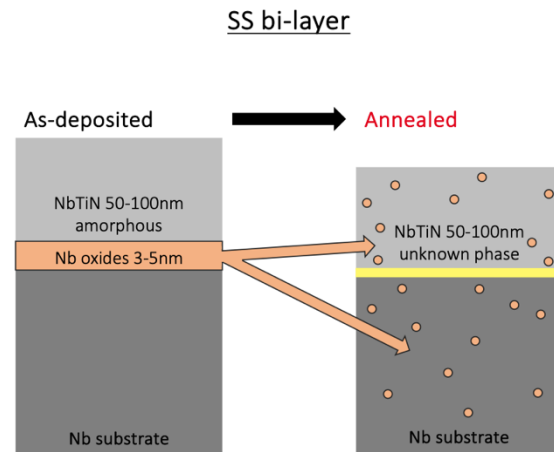
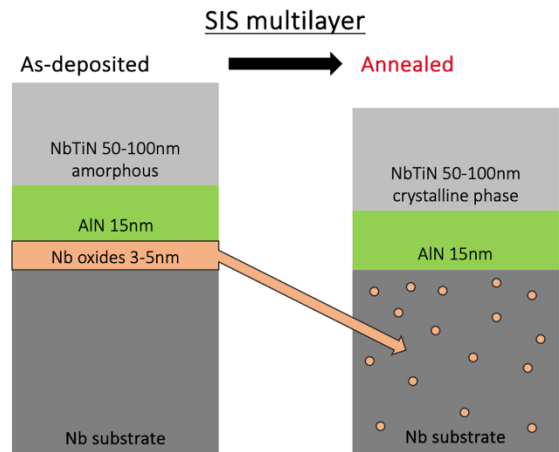
Discussion on T_C and Flux Expulsion

- Better flux expulsion for 900 °C initial anneal (*also observed in other labs*)
 - Increase in grain size with higher T_{anneal}
 - Reduction in pinning sites
- Post-deposition annealing enhances flux expulsion
 - Even for SS Nb/NbTiN if no high T_C is achieved
 - Assumption: Pancake effect adding additional force that counteracts pinning force



Discussion on T_C and Flux Expulsion

- High- T annealing required to *activate* the thin film and achieve high T_C
- Insulating layer on Nb required to ensure increase in T_C
 - Barrier layer prevents oxygen from diffusing into NbTiN layer
 - XRD of NbTiN on Si confirm high- T_C δ -phase formation for SS sample

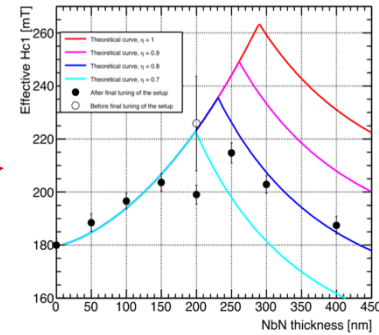


Intermediate oxygen layer

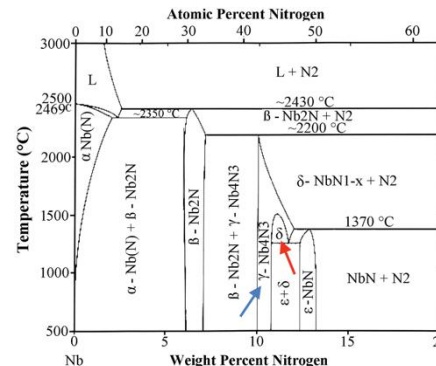
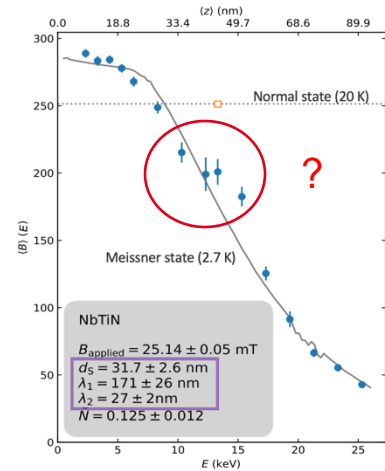
Conclusion

- Unclear if reduction of T_C implies reduction of H_C
 - Experimental determination required
 - Useful comparison with estimation of H_{max} following Kubo's model
- Findings on oxygen diffusion allow new considerations on μ SR data analysis
 - Intermediate layer in the SS bi-layer Nb/NbTiN?
 - Electron Microscopy probably shows oxygen-enriched phase with varying stoichiometry (*Ongoing TEM studies with EPFL*)
- 900 °C annealing of SIS Nb/AlN/NbN
 - Does **not** form intended high- T_C δ -phase
 - Ti as stabiliser of the cubic high- T_C δ -phase
 - NbN excluded from SIS studies**

Soon: NbTiN H_{C1} measurement @ KEK!



Ito, H. et al. [arXiv: 1907.03410] (2019)



Summary

- Further improvement of Nb SRF cavity performance necessary... **and possible with PEALD coated SIS multilayer!**
 - Coating thin high- T_C superconducting and insulating layers
 - Pushing the field of first flux penetration H_{ffp}
- Further characterization and experimental testing of the SIS theory required

SIS is a promising approach towards new technologies and improved future applications in SRF research!

Contact

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