Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

FAKULTÄT
FÜR MATHEMATIK, INFORMATIK
UND NATURWISSENSCHAFTEN

22.11.2023

Lea Preece

Magnetic Characterization of ALD coated thin films

Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

SIS Multilayer Studies

1 Status Update of PEALD System

2 Flux Expulsion Measurement at CERN

3 Determination of H_{C1} at KEK

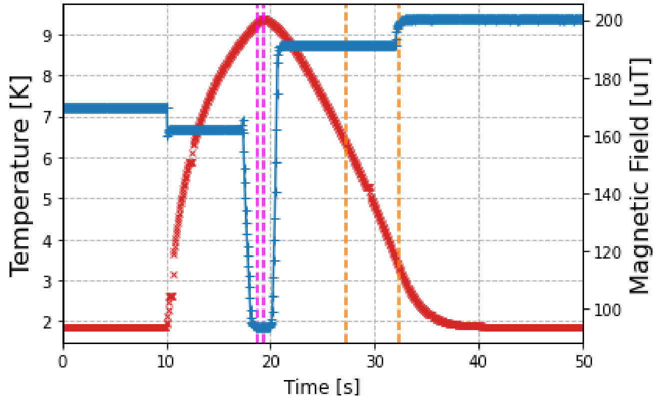
- Complementary measurement: VSM

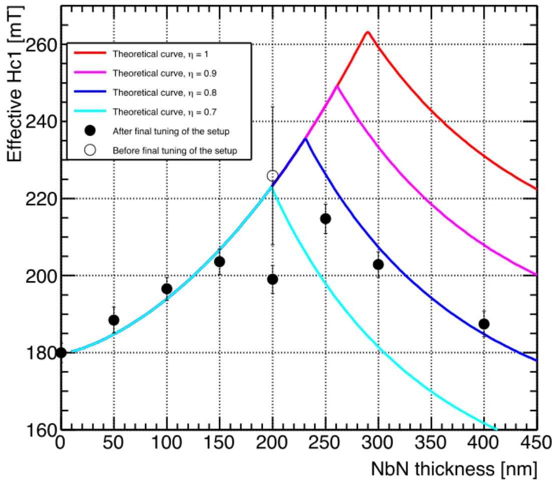
4 Supplementary Measurements

5 Outlook

Goal of my thesis

- Magnetic studies of SIS coatings
- Two main aspects:
  - Magnetic Flux Expulsion
    - Investigations on T_c
  - Determination of H_{C1}
    - Penetration depth λ_L
    - Film thickness d





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

Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

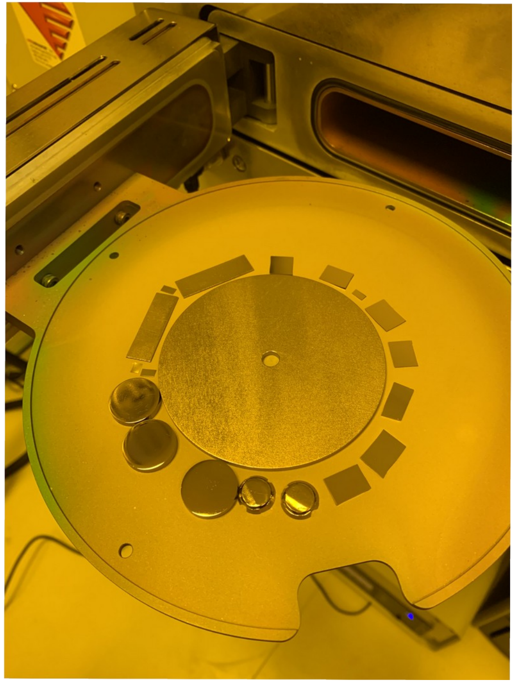
1

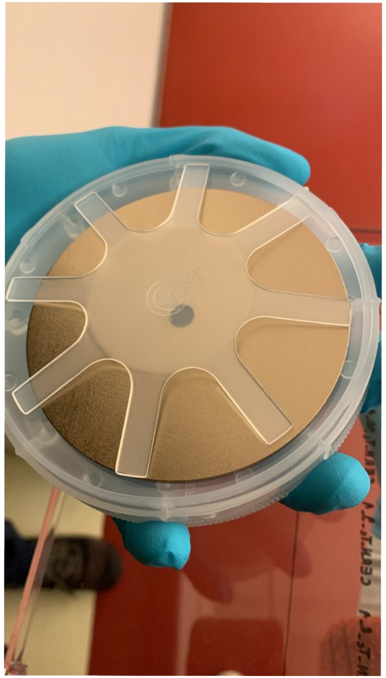
Status Update of PEALD System

Status of PEALD System

- Various problems
  - O-Ring broke, was replaced
  - Scratch at chamber door, valve opening/closing, plasma head…
- Started first coating campaign
  - So far all coatings and annealings went as expected
- Working on optimization of process parameter

Status of PEALD System





2

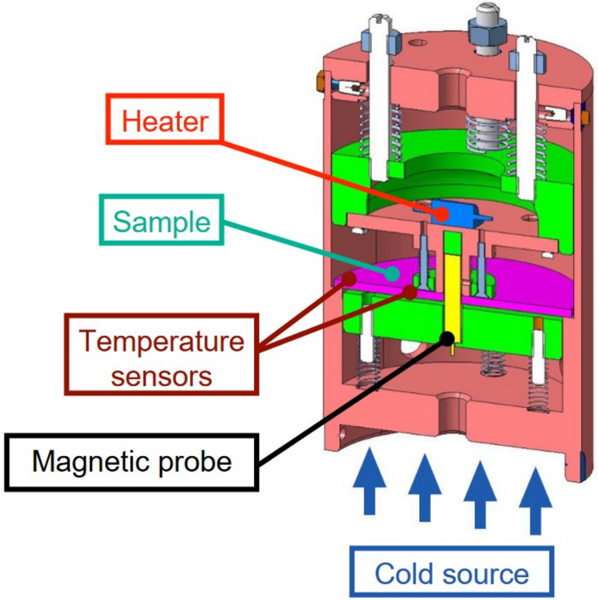
Flux Expulsion Measurements at CERN

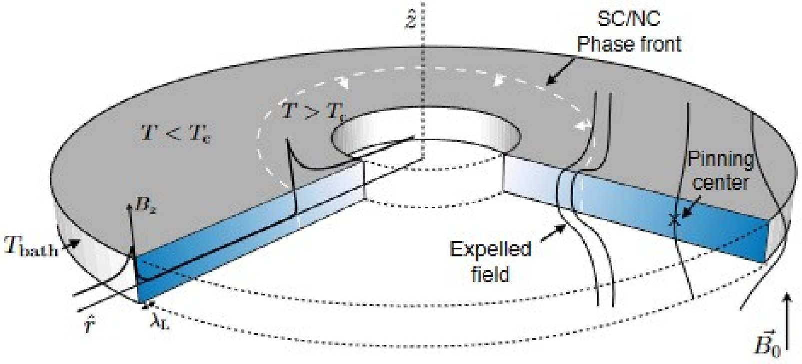
Flux Expulsion Measurements at CERN

- Discovery of **Flux Ratcheting** on Nb/AlN/NbTiN
- System modification of Flux Lens Experiment
  - Installation of new flux probe
  - Moved to new cryostat
- Further investigations on T_C

Flux Lens and disc-shaped samples

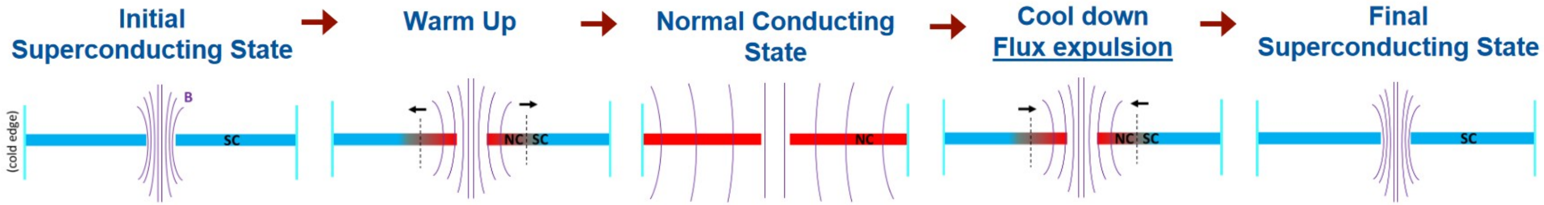
- Collimate expelled flux from superconducting sample at controlled cool-down conditions





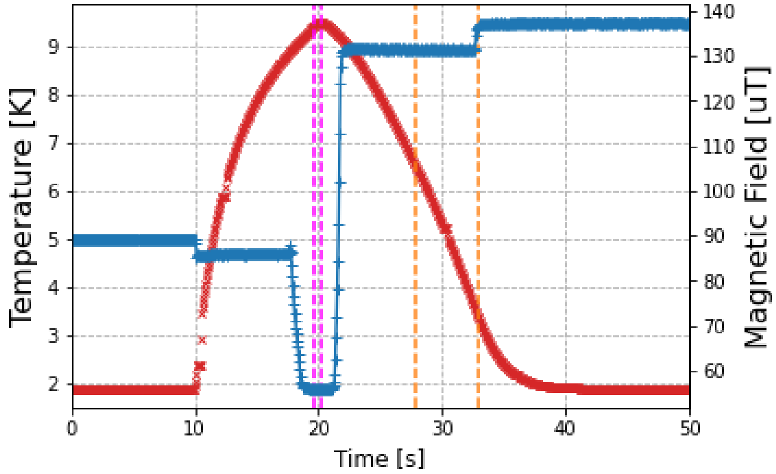
Bartolome et al. „Flux expulsion lens“ CERN SRF Workshop 2022

Flux Expulsion Measurements at CERN



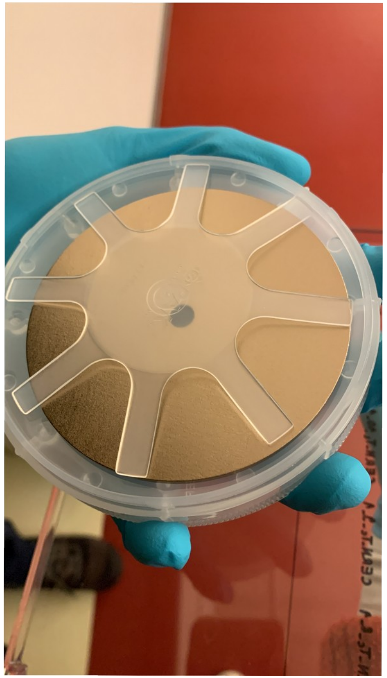
Bartolome et al. „Flux expulsion lens" CERN SRF Workshop 2022

Flux Expulsion of a ratcheting pulse:

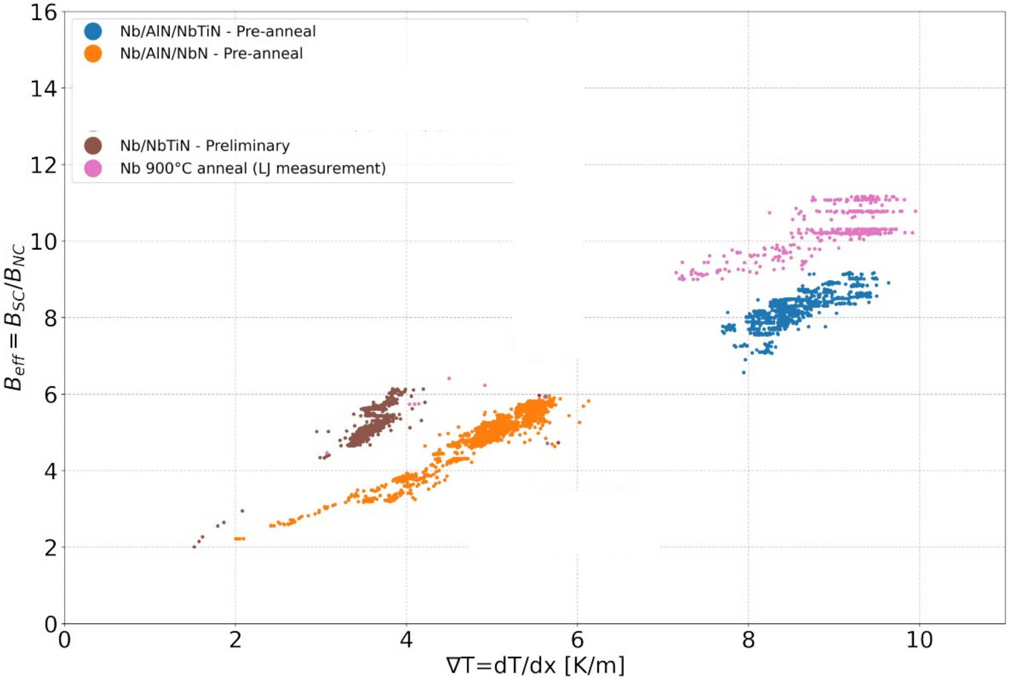


Sample Preparation

Live table - Update as samples change				
	Initial structure		Current placement	Comments
Samples	Structure	Anneal Temp		
DESY_0_1	Nb	800	DESY	Sacrificial sample - Becomes Nb/AlN
DESY_1_1	Nb	900	DESY	Becomes SISIS
DESY_2_2	Nb/NbTiN	800	CERN	Annealed
DESY_3_2	Nb/AlN/NbTiN	800	CERN	Annealed
DESY_4_2	Nb/AlN/NbN	800	CERN	Annealed
DESY_5_2	Nb/AlN/NbTiN	900	CERN	Annealed
DESY_6_2	Nb/NbTiN	900	CERN	Annealed
DESY_7_1	Nb	900	DESY	

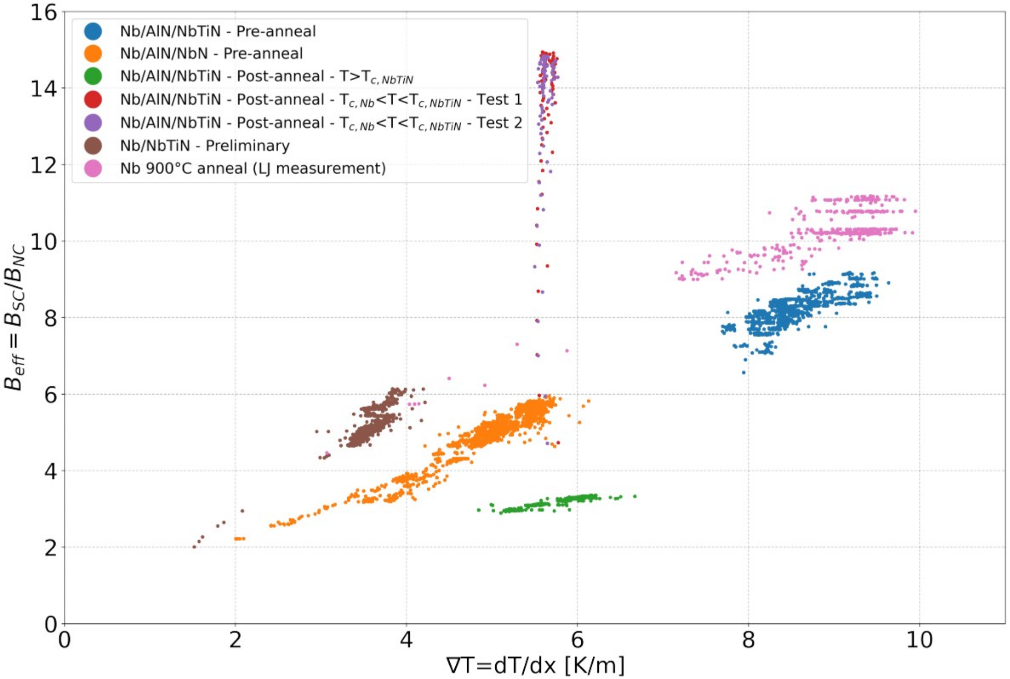


Flux Expulsion and Ratcheting



- **Pre-anneal**: Flux expulsion dominated by Nb substrate

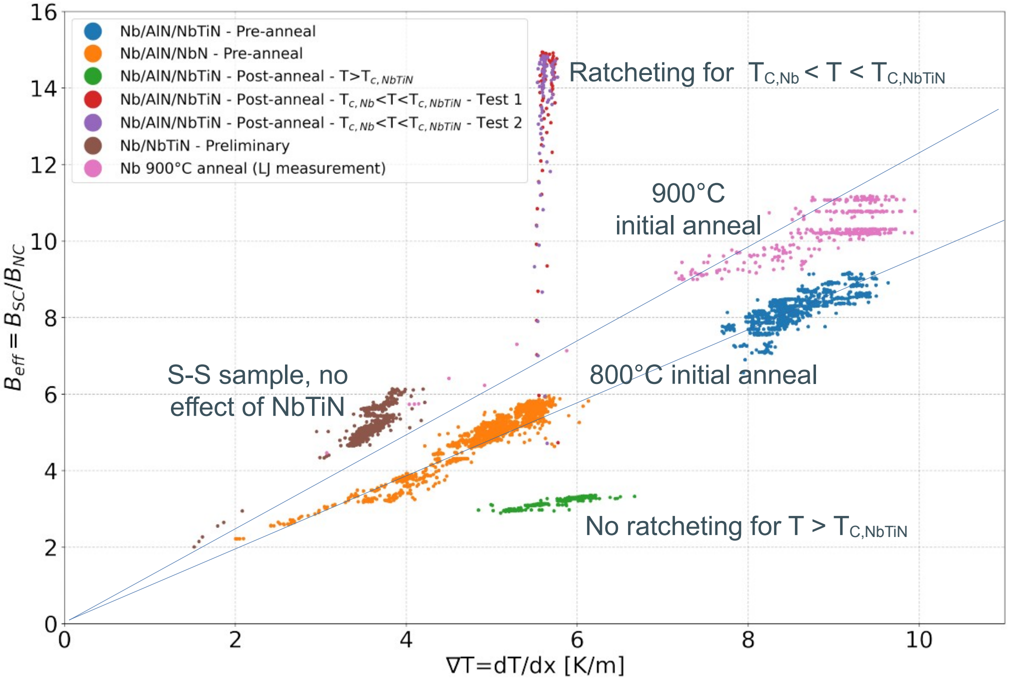
Flux Expulsion and Ratcheting



- **Pre-anneal**: Flux expulsion dominated by Nb substrate

Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

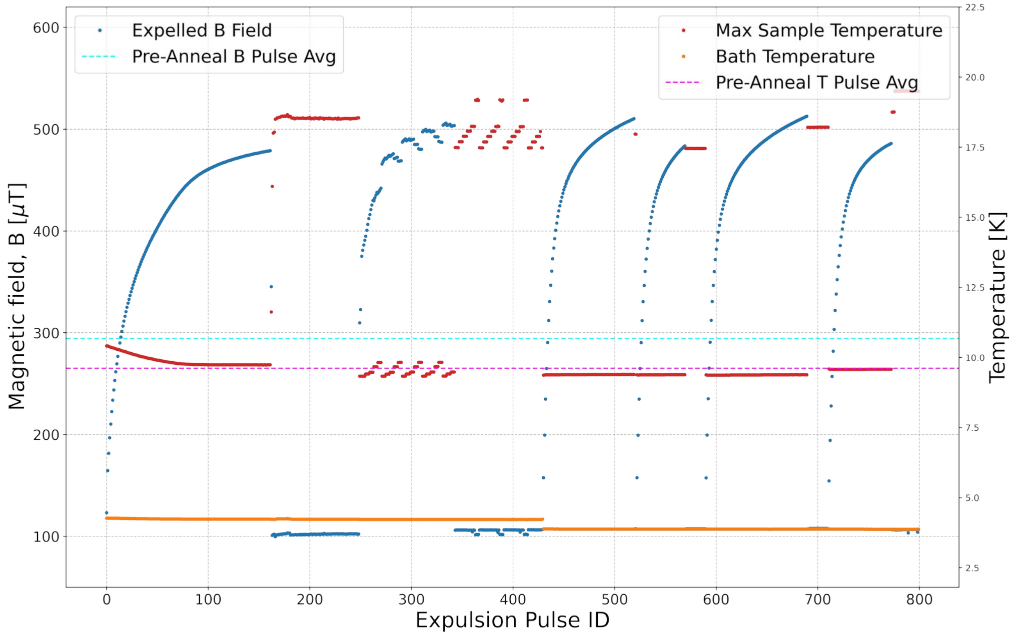
Flux Expulsion and Ratcheting



Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

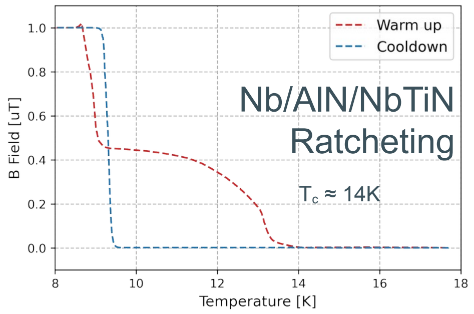
- Sample preparation with 900°C annealing has better expulsion than 800°C annealing
- $T_{C,NbTiN} \approx$ 14K (possibly underestimated)
- S-S sample shows no effect of NbTiN layer
- Nb/AlN/NbTiN looks worse for $T > T_{C,NbTiN}$ → More pinning sites!

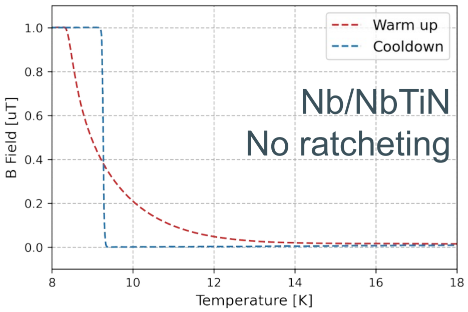
Flux Ratcheting for Nb/AlN/NbTiN



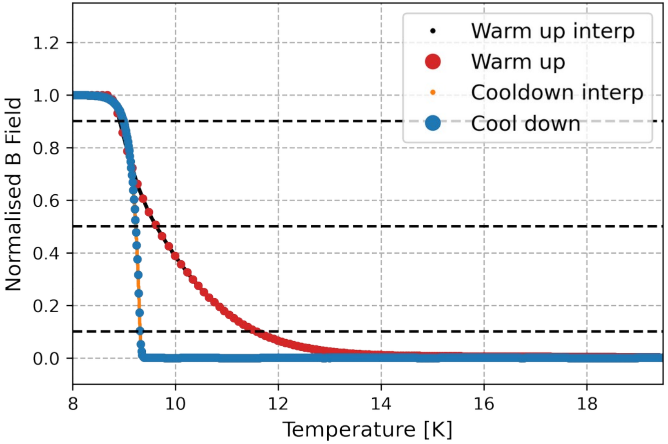
- SIS sample
- B_{SC} changes for variying T_{max}
- **RATCHTING IN REPRODUCIBLE**

No ratcheting for Nb/NbTiN and Nb/AlN/NbN - T_C check





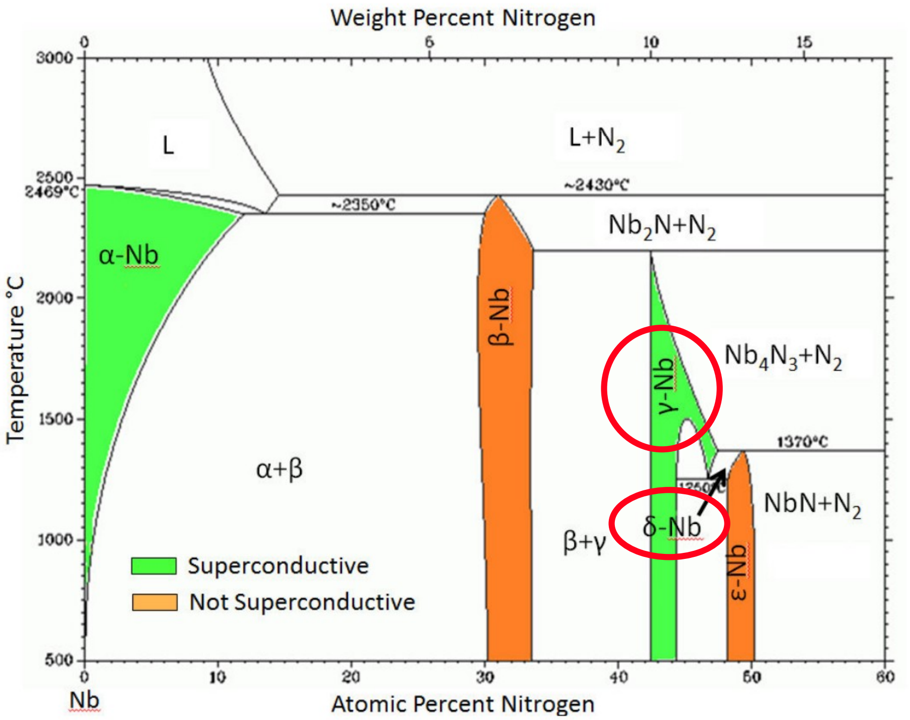
Nb/AlN/NbN →
<u>No ratcheting</u>



Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

- Ratcheting occurs for SIS with NbTiN, stepwise transition
- **But NOT** for S-S with NbTiN
  → Assumption: Oxygen uptake during annealing, $T_{C,NbTiN}$ surpressed
- **And NOT** for SIS with NbN
  → We don't achieve needed NbN phase during annealing

NbN phases



https://www.desy.de/f/students/2014/reports/hilbig_mario.pdf

Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

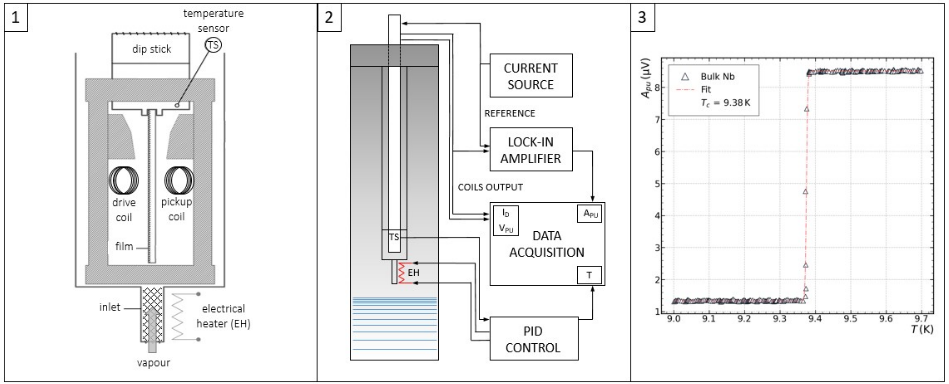
- Assumption: We form γ-phase with $8.9<T_C<11K$
- NbN δ-phase with T_C of 17.3K is relevant for SRF

  → Forms above 1250°C annealing, not possible for us

Soon: 1300°C annealing in Darmstadt.

T_C measurement at CERN

- Measurement of coated Nb samples
- Contactless inductive measurement



Fonnesu, D. et al. [doi:10.18429/JACoW-SRF2021-SUPFDV018] (2021)

	Initial structure		Current placement	Comments
Samples	Structure	Anneal Temp		
CERN_Tc_1_2	Nb/AlN/NbTiN	800	CERN	Annealed @900°C 1h
CERN_Tc_2_1	Nb/AlN/NbTiN	800	CERN	
CERN_Tc_3_2	Nb/NbTiN	800	CERN	Annealed @900°C 1h
CERN_Tc_4_1	Nb/NbTiN	800	CERN	
CERN_Tc_5_2	NbAlN/NbN	800	DESY	Annealed @900°C 1h
CERN_Tc_6_1	NbAlN/NbN	800	DESY	
CERN_Tc_7_1	Nb/NbN	800	DESY	Will be annealed
CERN_Tc_8_1	Nb/NbN	800	DESY	
CERN_Tc_9_1	Nb/NbN	800	DESY	1300°C anneal in Darmstadt
CERN_Tc_9_2	Nb	800	DESY	Becomes SISIS

T_C measurement at CHyN

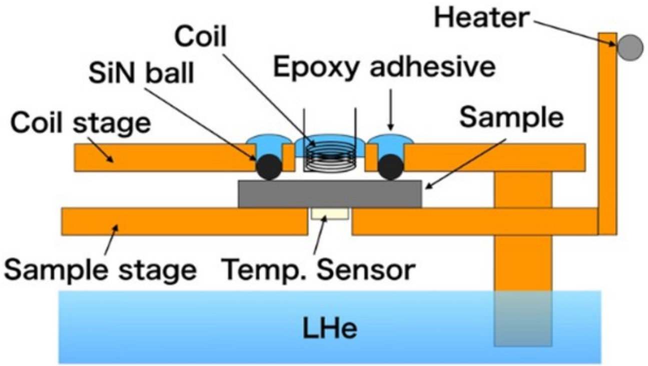
- Dynacool system
- Measurement of electrical properties of coated Si samples
- **First results of Nb/AlN/NbTiN**
    - Not annealed: 8.51K
    - Annealed: 15.95K

3

Determination of H_{c1} at KEK

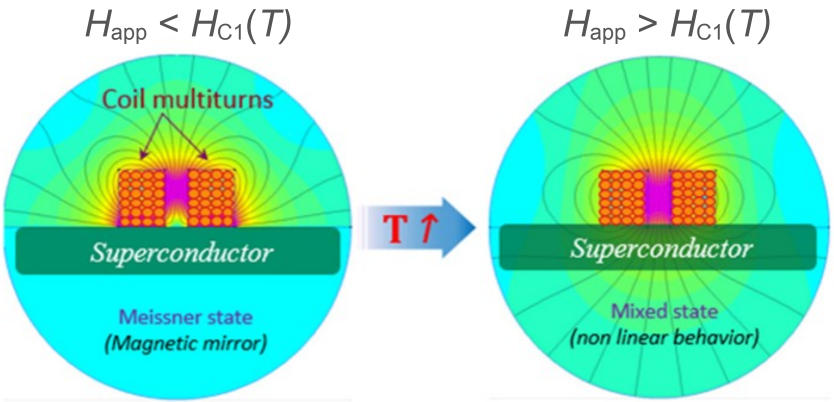
Determination of H_{c1} at KEK

Setup



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

Principle



Aburas, M. et al. [THPB038] (SRF2017)

Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

Coating Plan KEK

d_s / nm	NbTiN
25	Yes
50	Yes
60	Yes – 2x w & 1x w/o 120°C
75	Yes – 2x w & 1x w/o 120°C
100	Yes – 2x w & 1x w/o 120°C
125	Yes
S(IS)²	Yes - 2x
∑	14

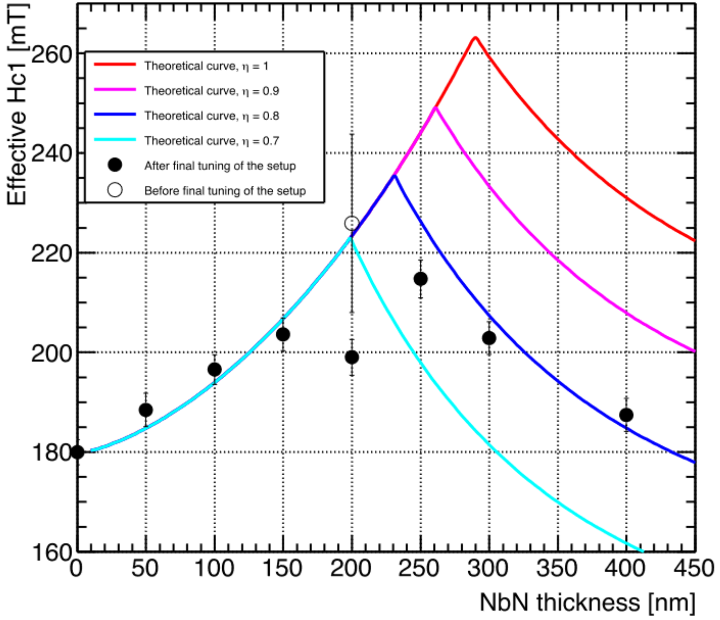
UHH Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

- One S-S sample coated with NbN

  → to be annealed in Darmstadt
- System modifications @KEK
- First 5 samples will be send to KEK within next weeks!

Determination of H_{c1}

- Input parameters
  - London penetration depth λ_L
  - Film Thickness d



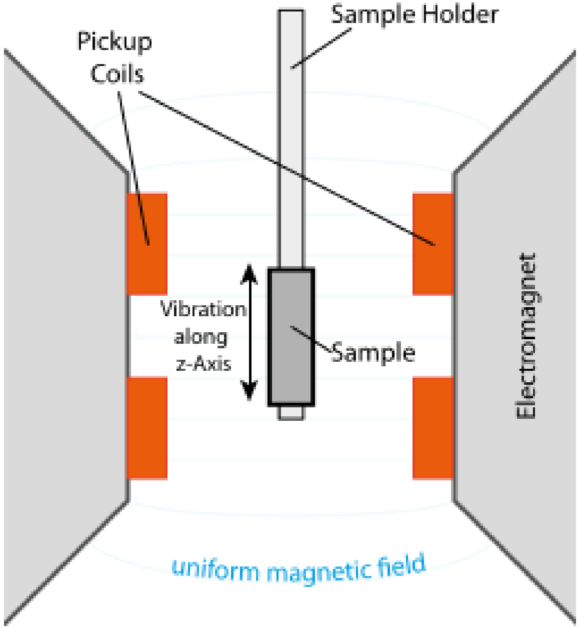
Ito, H. et al. [arXiv: 1907.03410] (2019)

Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

Complementary Measurements: VSM @ CHyN

- Vibrating-Sample magnetometry
- Measure magnetic moment m vs.
  - H to obtain H_{C1}
  - T to obtain T_C

  of zero-field-cooled samples
- Possible for both Nb and Si

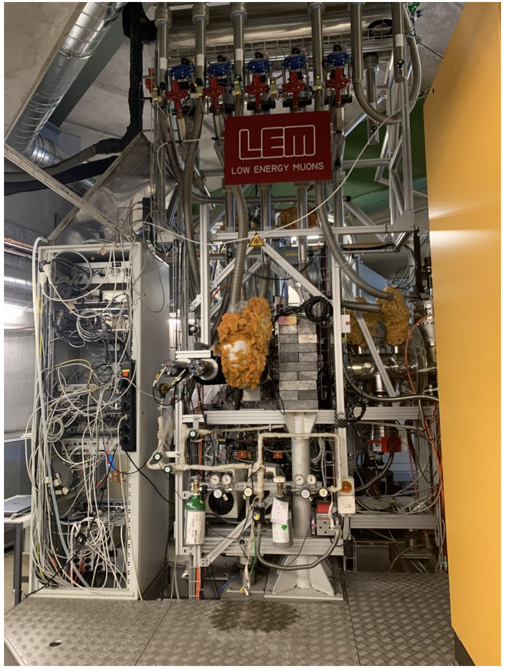


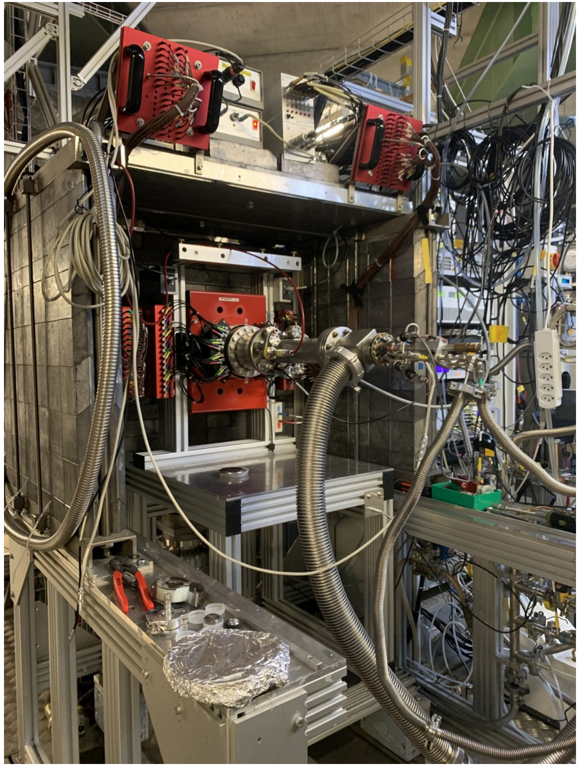
https://en.wikipedia.org/wiki/Vibrating-sample_magnetometer

4

Supplementary Measurements

LE-μSR Experiment at PSI



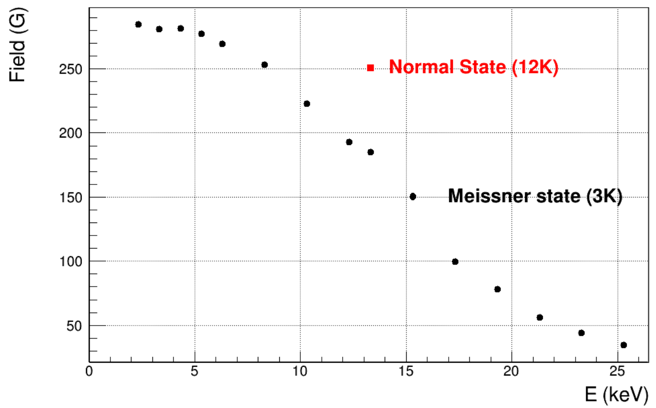


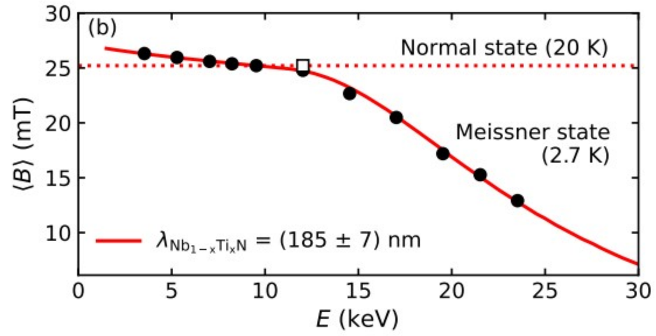
Determination of λ_L

- Depth-resolved measurement of Meissner-Screening profile
- Field screening considering counter-current-flow induced by substrate

$$B(z) = B_0 \times \begin{cases} 1, & z \leq 0, \\ D_{S-S}^{-1} \left[\cosh\left(\frac{d_s - z}{\lambda_s}\right) + \left(\frac{\lambda_{sub}}{\lambda_s}\right) \sinh\left(\frac{d_s - z}{\lambda_s}\right) \right], & 0 < z \leq d_s, \\ D_{S-S}^{-1} \left[\exp\left(-\frac{z - d_s}{\lambda_{sub}}\right) \right], & z > d_s, \end{cases}$$

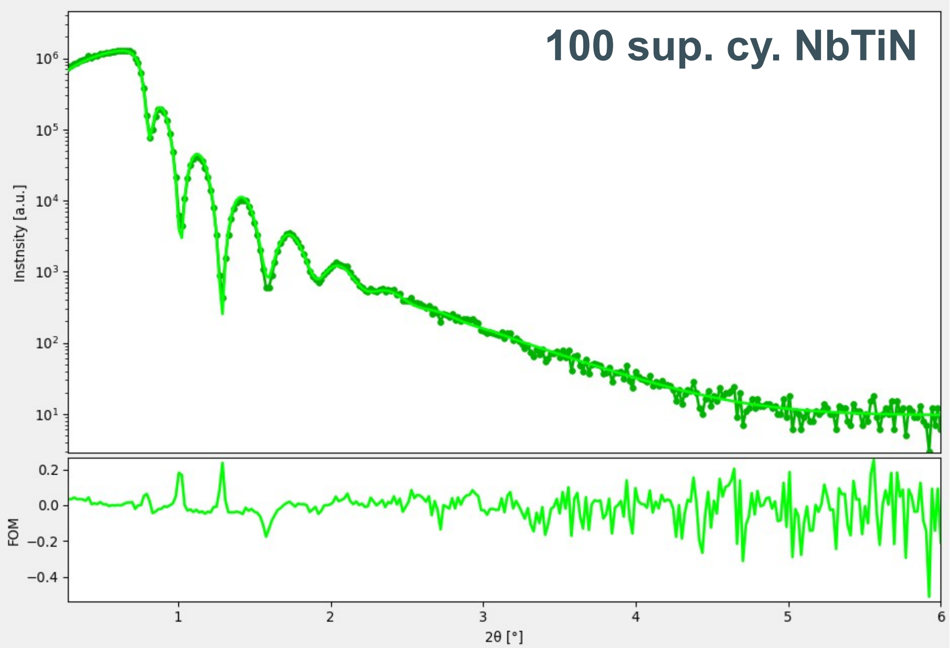
$$D_{S-S} = \cosh\left(\frac{d_s}{\lambda_s}\right) + \left(\frac{\lambda_{sub}}{\lambda_s}\right) \sinh\left(\frac{d_s}{\lambda_s}\right)$$





Asaduzzaman, Md et al. [arXiv:2304.09360v1] (2023)

XRR for NbTiN



- Film thickness d as input parameter for H_{C1} determination
  → determine growth per cycle with XRR
- Good fit for 100 supercycles of NbTiN (25nm thickness)
- Remeasurements for
  → Various # (super)cycles
  → NbTiN, TiN

Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

5

Outlook

The Coating Plan

COATINGS

TO DO:
Precursors?
EP Nb samples
Check T for AIN with Rakshith
Isabel & Rakshith discussion T of AIN coating

REMEMBER:
19 December Power Shut Down @ CHyN
Enough Si for XRR, SEY

OVERVIEW:
Campaign 1 => 8xKEK, 1x Flux Lens, 8x Tc, 8x Nb ref, and Si + 3 Nb sheets
Campaign 2 => 6xKEK, 1x Flux Lens, 5x Nb ref, and Si + Nb sheet

SPARE KEK SAMPLE -> Nr. 20
SPARE Tc SAMPLES -> CERN_Tc_9_1, CERN_Tc_10_1 (for Multilayer coating?)
SPARE FLUX LENS SAMPLES -> DESY_0_1 (Sacrificial sample - Becomes Nb/AIN), DESY_7_1

SHIPPING TO CERN:
CERN, Bat 252 1-026, 1211 GENEVA 23, Switzerland
SY-RF-SRF: Superconducting RF section
Alick Macpherson

NOTES:
AIN @ 100, 125°C is bad, @ 150, 200°C it's good but blisters
Nb precursor refill when more than 5000 cycles done (max. 6000)
Nanowires for Rak!!
15nm AIN 273 cycles
60nm NbTiN 243 super cycles (1 supercycle = 1*TiN + 3*NbN)
60nm NbN 1000 cycles
TBTDEN = Nb precursor
TDMAT = Ti precursor
TMA = Al precursor

CAMPAIGN 1

For?	thin film	layers	thickness (approx. nm)	AIN cyc	NbN cyc	TiN cyc	approx. duration (h)	days	which samples?	Priority Run Nb.	comments	COATING	ANNEALING
KEK, CERN	AIN (15nm) /NbTiN	SIS	60	273	729	243	100 (10 + 90) -> 83h	4	KEK_3, KEK_23, KEK_4 CERN_Tc_1_1, CERN_Tc_2_1 DESY_5_1 Si & Nb ref (260, 261) Nb sheet	1	KEK 2x w & 1x w/o 120°C High T for AIN (because of DESY_5_1)	06.11.23	10.11.23
CERN	NbTiN	SS	60	0	729	243	90 →72h	4	CERN_Tc_5_1, CERN_Tc_6_1 DESY_6_1 Si & Nb ref (262, 263) 2x Nb sheet	2		10.11.23	14.11.23
CERN	AIN (15nm) /**NbN**	SIS	60	273	1000	0	100 (10 + 90) → 88h	4	CERN_Tc_3_1, CERN_Tc_4_1 Si & Nb ref (264)	3	**Annealing in Darmstadt**	13.11.23	freitag
CERN	**NbN**	SS	60	0	1000	0	90	4	CERN_Tc_7_1, CERN_Tc_8_1 Si & Nb ref (265)	4	**Annealing in Darmstadt**	freitag	
KEK	AIN (15nm) /NbTiN	SIS	75	273	911,25	303,75	125 (10 + 115)	5	KEK_5, KEK_19 Si & Nb ref	5	KEK 1x w & 1x w/o 120°C		
KEK	AIN (15nm) /NbTiN	SIS	100	273	1215	405	160 (10 + 150)	6,5	KEK_7, KEK_18 Si & Nb ref	6	KEK 1x w & 1x w/o 120°C		
KEK	AIN (15nm) /NbTiN	SIS	50	273	607,5	202,5	100 (10 + 80)	4	KEK_2 Si & Nb ref	7			
SUM				1365	6191,75	1397,25		31,5					

CAMPAIGN 2

For?	thin film	layers	thickness (approx. nm)	AIN cyc	NbN cyc	TiN cyc	approx. duration (h)	days	which samples?	Priority	comments	COATING	ANNEALING
KEK	AIN (15nm) /NbTiN	SIS	75	273	911,25	303,75	125 (10 + 115)	5	KEK_6 Si & Nb ref	8	KEK 1x w 120°C		
KEK	AIN (15nm) /NbTiN	SIS	100	273	1215	405	160 (10 + 150)	6,5	KEK_8 Si & Nb ref	9	KEK 1x w 120°C		
KEK	AIN (15nm) /NbTiN	SIS	25	273	303,75	101,25	50 (10 + 40)	2	KEK_1 Si & Nb ref	10			
KEK, CERN	AIN (15nm) /NbTiN	S(IS)^2 125 total	125	273	1518,75	506,25	170 (10 + 75 + 10 + 75)	7	KEK_12, KEK_13 DESY_1_1 CERN_Tc_9_1, CERN_Tc_10_1 Si & Nb ref Nb sheet	11			
KEK	AIN (15nm) /NbTiN	SIS	125	273	1518,75	506,25	200 (10 + 190)	8	KEK_10 Si & Nb ref	12			
SUM				1365	5467,5	1822,5		28,5					

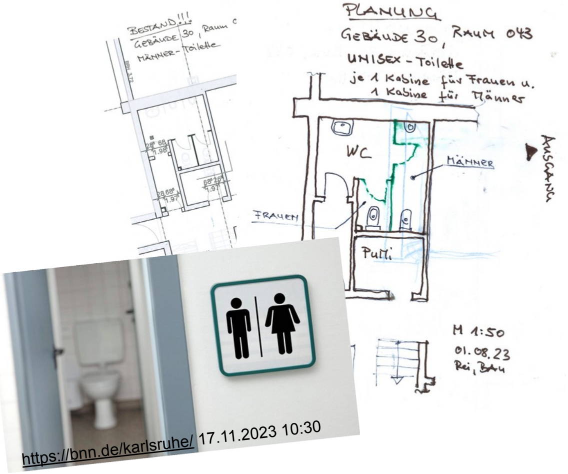
Outlook

Next weeks

- Send samples to Darmstadt, CERN
- Continue coatings for KEK until CHyN shutdown
- Send first batch of samples to KEK

Next months

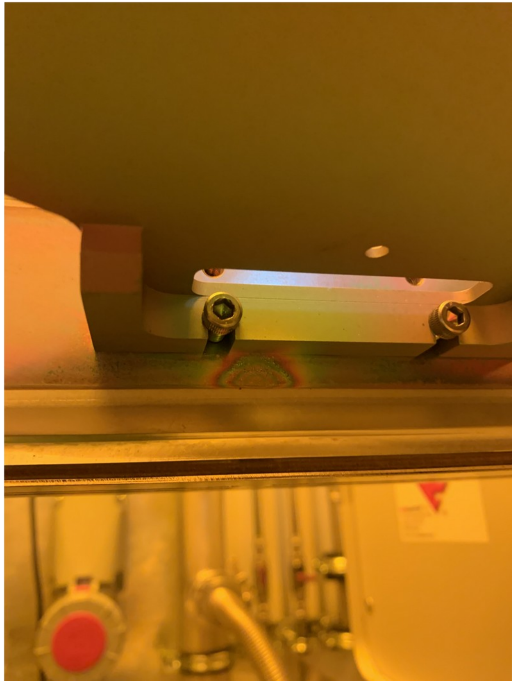
- Measurements at KEK
- Ongoing studies at CERN
- Work on data analyses to obtain H_{c1}



RECONSTRUCTION STARTS NEXT WEEK!!

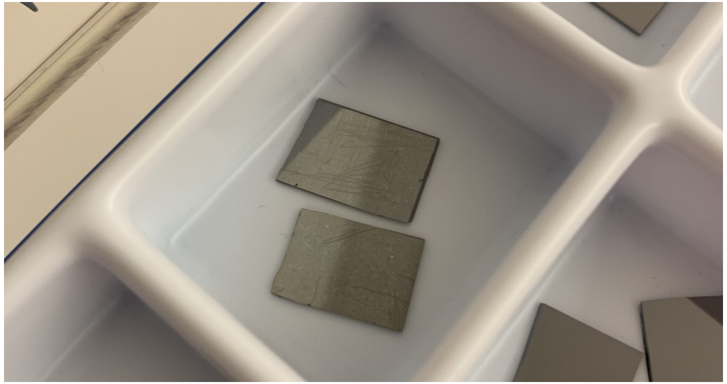
Backup

Status of PEALD System

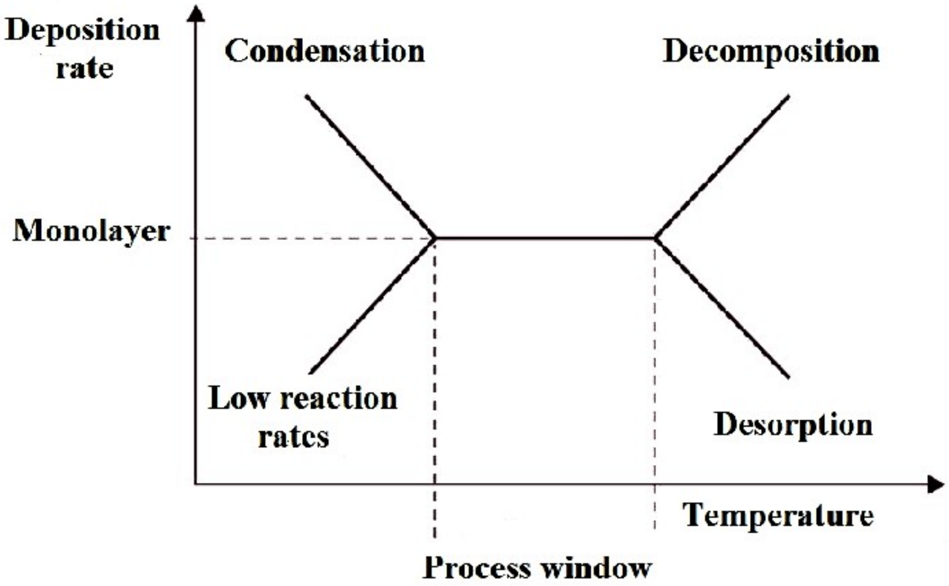


Process Parameter Studies (Rakshith)

- Low T (~100°C) → dusty, dirty surface
- High T (~200°C) → blisters

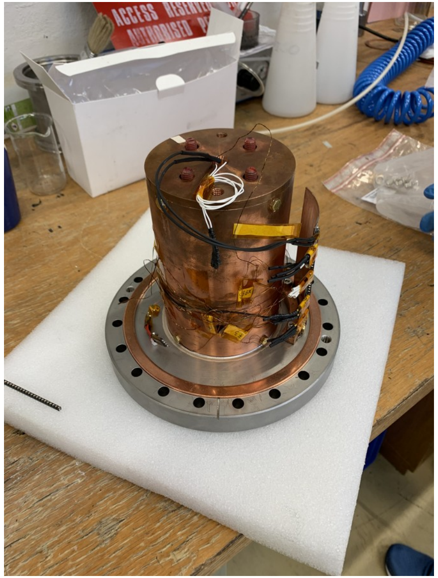


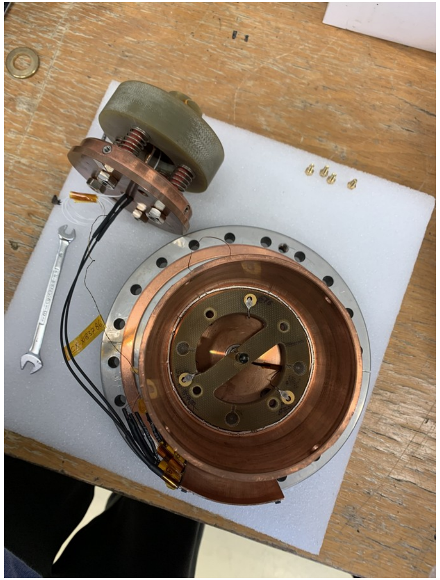
350 cycles AlN @ 105°C + 250°C 8h heating



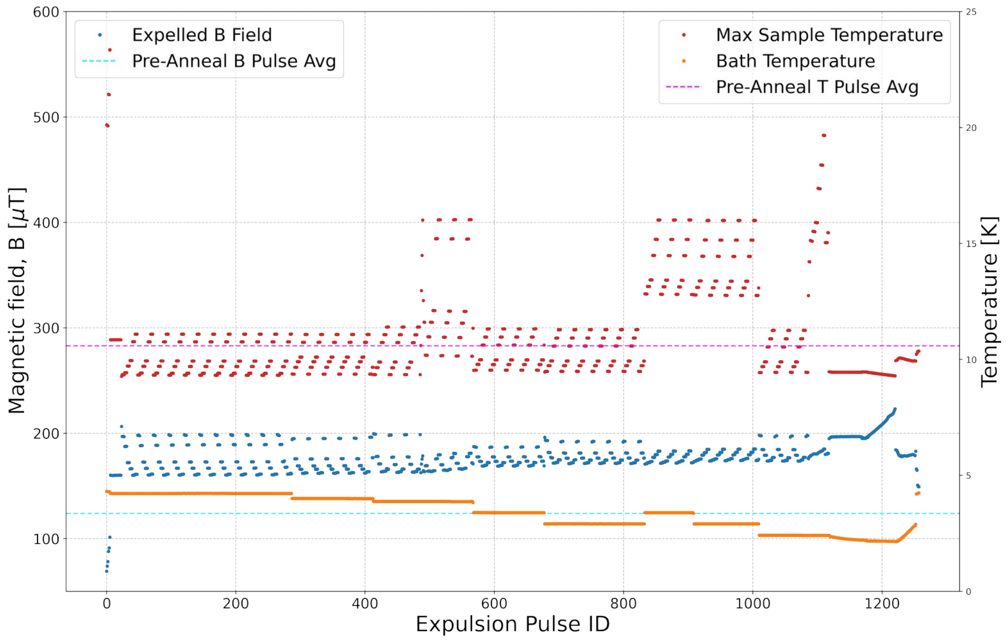
Dobrzanski, Leszek & Szindler, Marek & Szindler, Magdal (2015). Surface morphology and optical properties of Al2O3 thin films deposited by ALD method. 73. 18-24.

Installation of new Flux Probe



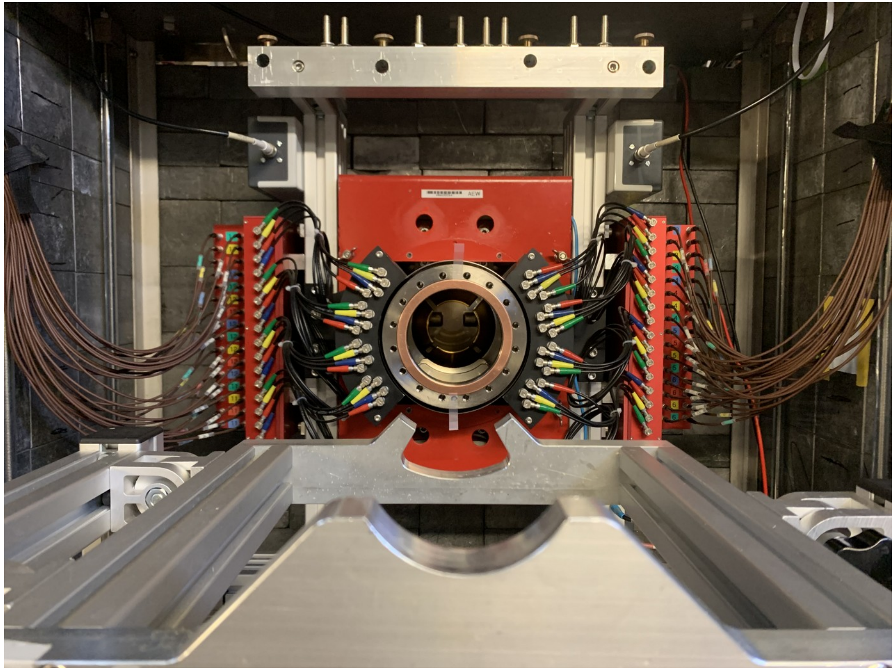


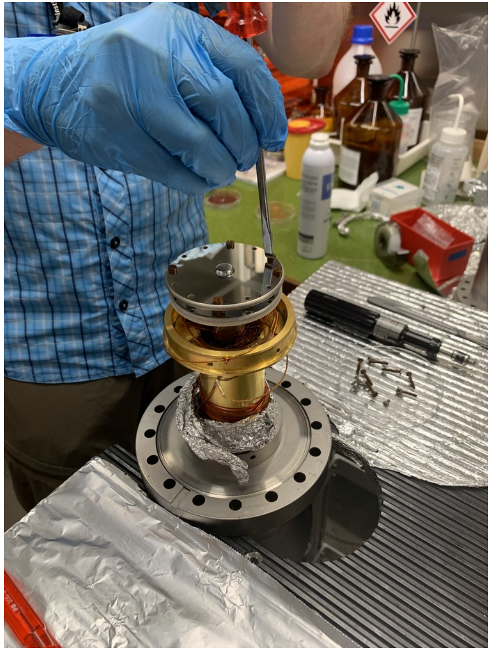
No ratcheting for Nb/NbTiN



- **S-S sample**
- Measured $T_{C,Nb} < T < T_{C,NbTiN}$
- No increase of T_C after 900°C anneal
- Similar behaviour as before annealing

LE-μSR Experiment at PSI



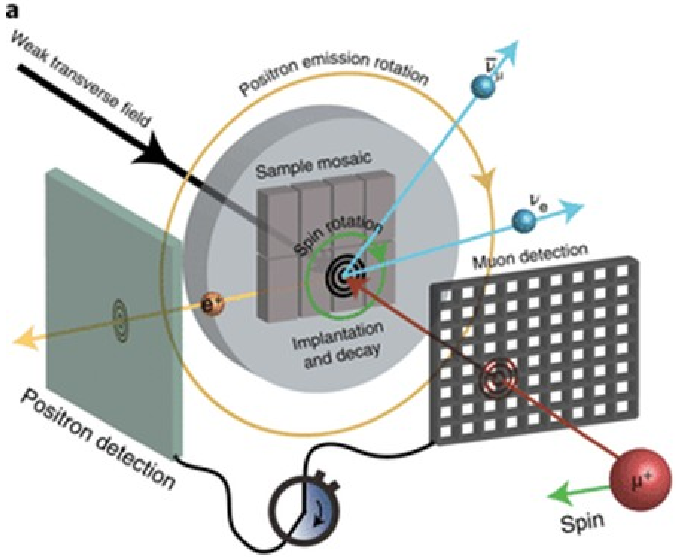


Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

Determination of London penetration depth at PSI

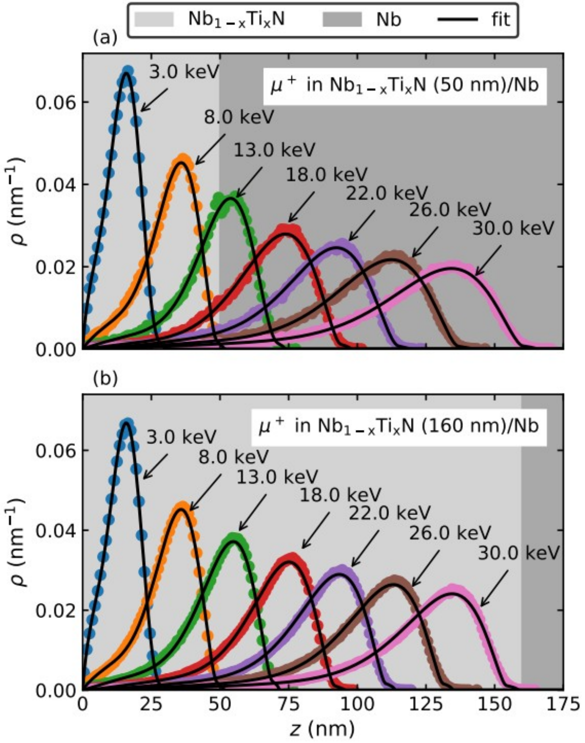
LE-μSR technique

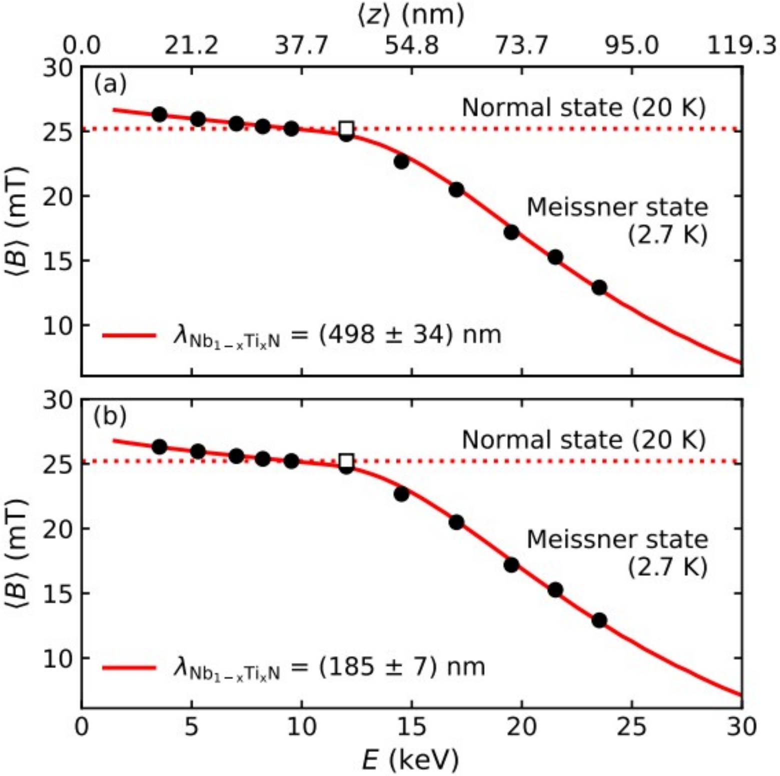
- Depth-resolved measurement of Meissner-Screening profile



Fowlie, J. et al. [DOI:10.1038/s41567-022-01684-y] (2022)

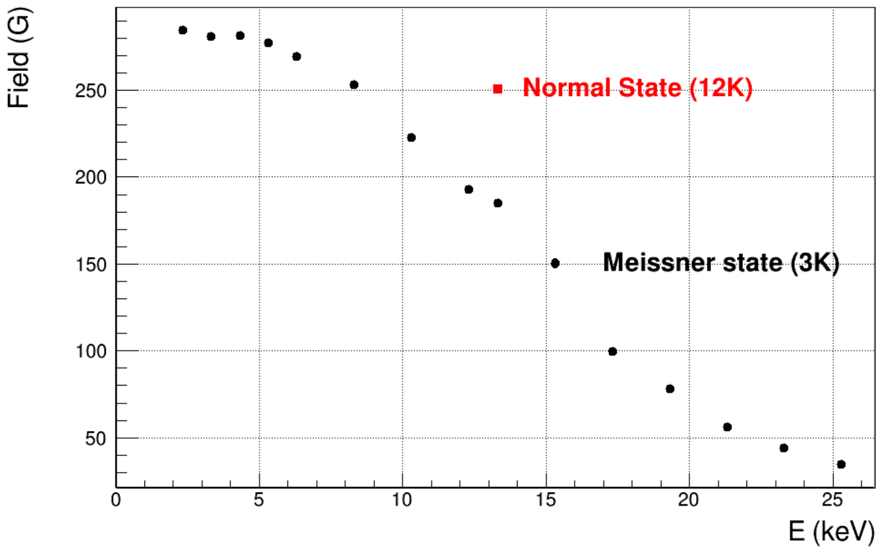
Determination of London penetration depth at PSI





Asaduzzaman, Md et al. [arXiv:2304.09360v1] (2023)

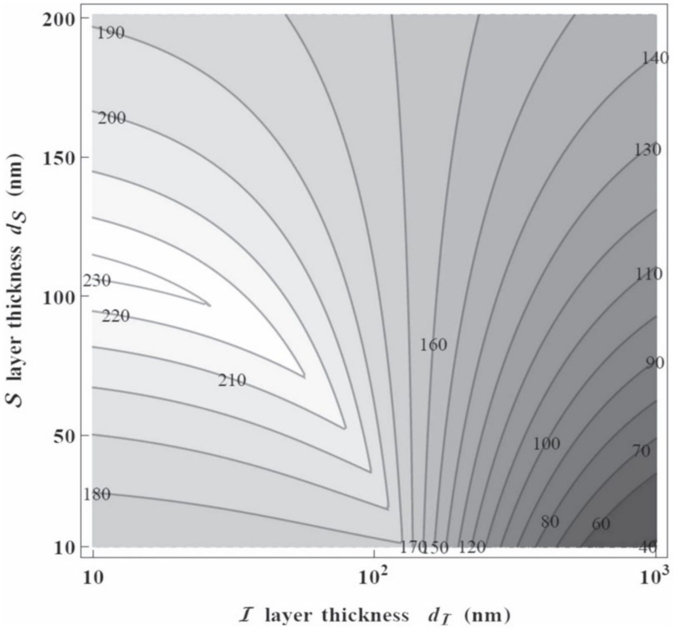
LE-μSR Measurement



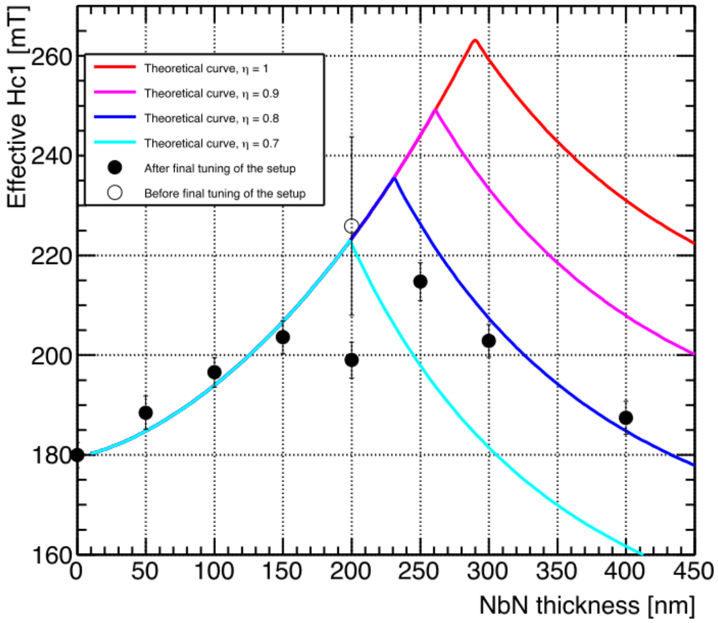
- Measured B field within sample as function of penetration depth
- $\lambda_{L,NbTiN}$ for analysis of H_{c1}
- 900°C annealed Nb/NbTiN sample (S-S sample)

→ Problem: Measurements at CERN show low Tc of Nb/NbTiN. **Affect on λ_L?**

Determination of H_{c1}



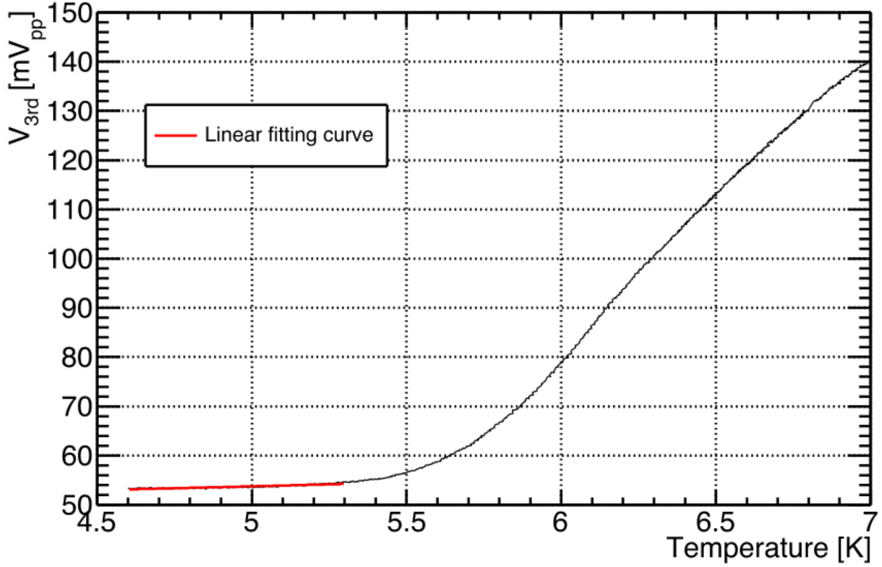
Kubo, T. *Supercond. Sci. Technol.* 30 023001 (2017)

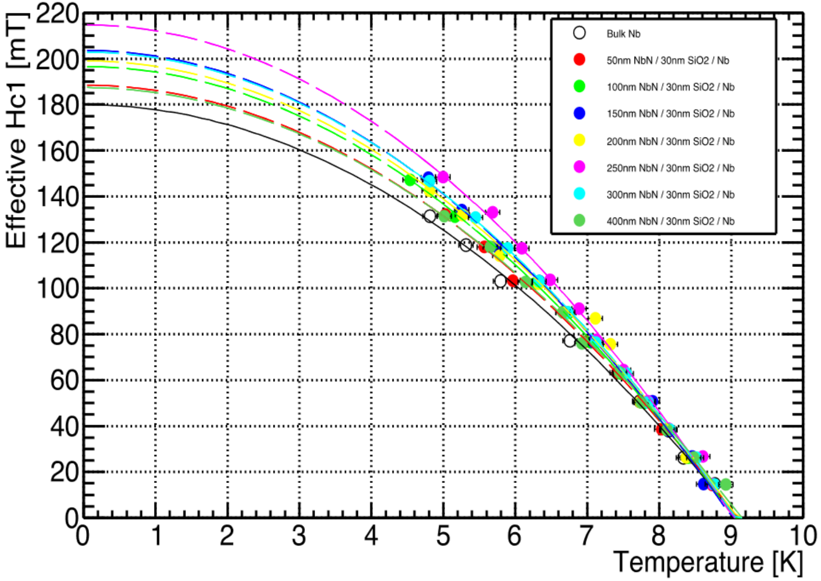


Ito, H. et al. [arXiv: 1907.03410] (2019)

Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

Determination of H_{c1}

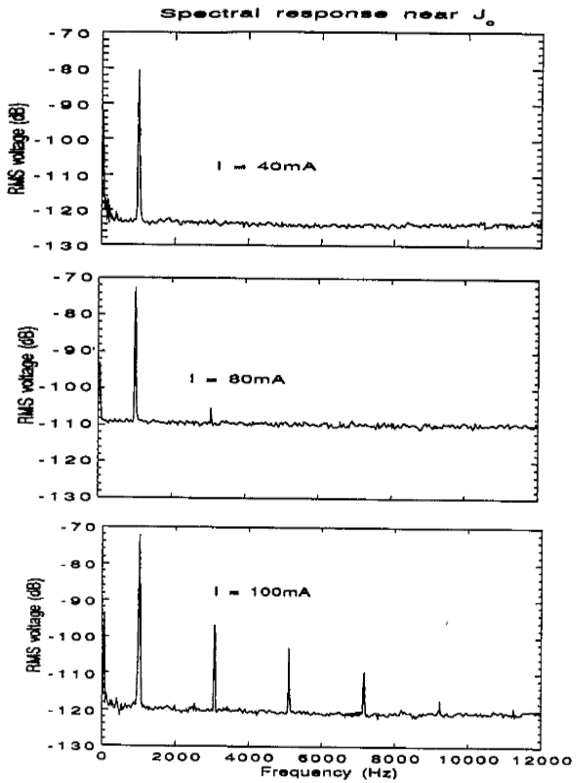




Ito, H. et al. [arXiv: 1907.03410] (2019)

Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

Determination of H_{c1}



Claassen, J.H. et al. [Rev Sci Instrum 62, 996–1004] (1991)

Universität Hamburg
DER FORSCHUNG | DER LEHRE | DER BILDUNG

Contact

Lea Preece
Master Student

Universität Hamburg
Institute for Experimental Physics
Luruper Chaussee 149
22761 Hamburg

Room: 045, 30

+49 40 8998-2394
lea.preece@desy.de