



SUPERCONDUCTING TECHNOLOGIES

FOR THE NEXT GENERATION
OF ACCELERATORS

WORKSHOP

Sergio Calatroni

History and Potential of Thin Film Technology
for SC Cavities

Outline

- Why thin films
- Nb sputtering on Cu cavities
 - Technology
 - Advantages and disadvantages
 - State of the art
- Future prospects
 - Energetic condensation
 - Multilayers
 - High(er) T_c

Nb bulk and Nb/Cu



or



?



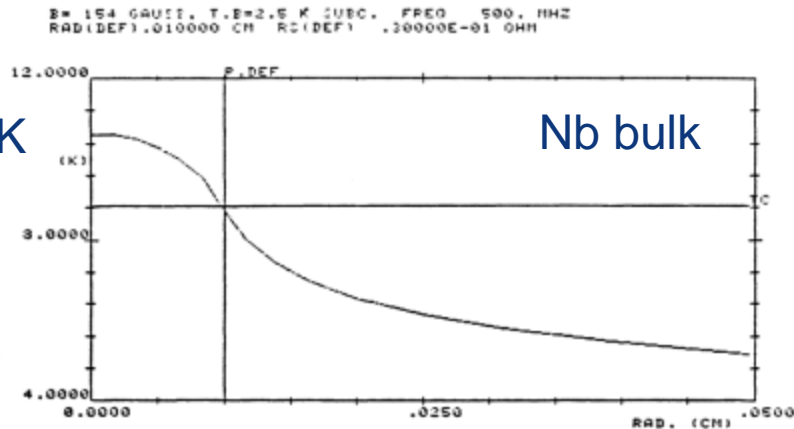
Basic cost advantages

Thin films on Cu OFE cavities

- Cu OFE sheets: 10 EUR/kg
- Nb RRR 300 sheets: 800 EUR/kg
- Simpler cryostats: helium tank can be made of stainless steel instead of titanium
- Conduction cooling is possible

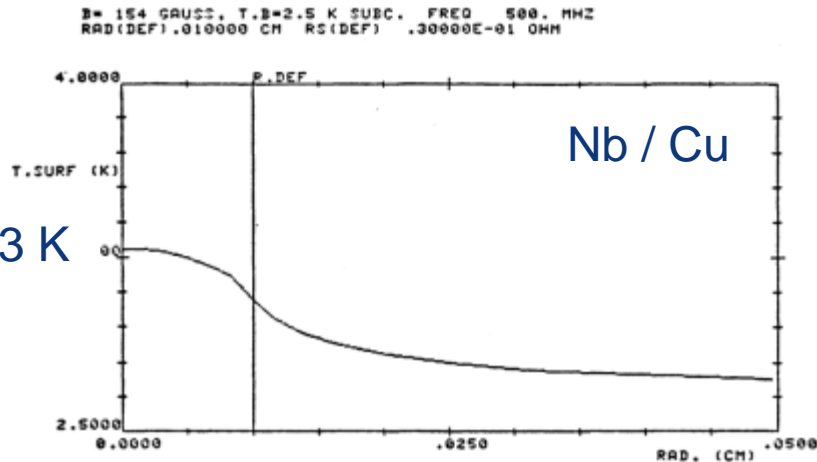
Basic historic motivation

Nb: 10.2 K



Temperature distribution calculation for a 100 micron radius steel defect embedded either in niobium or in copper

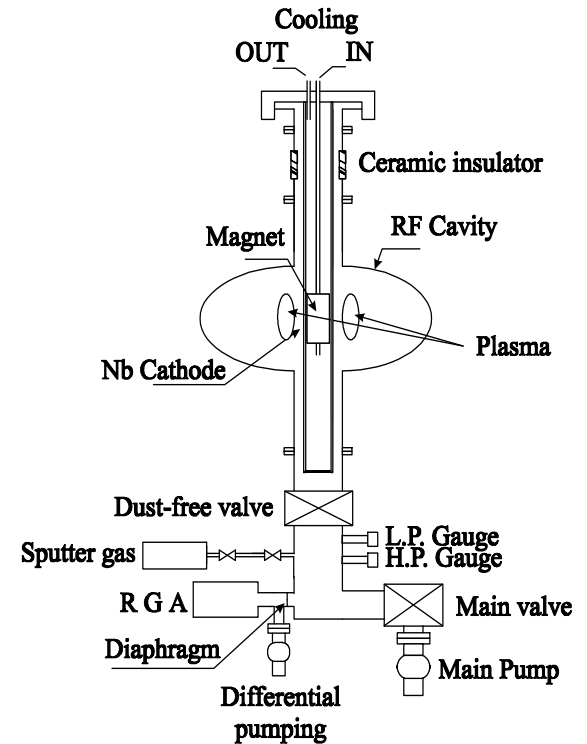
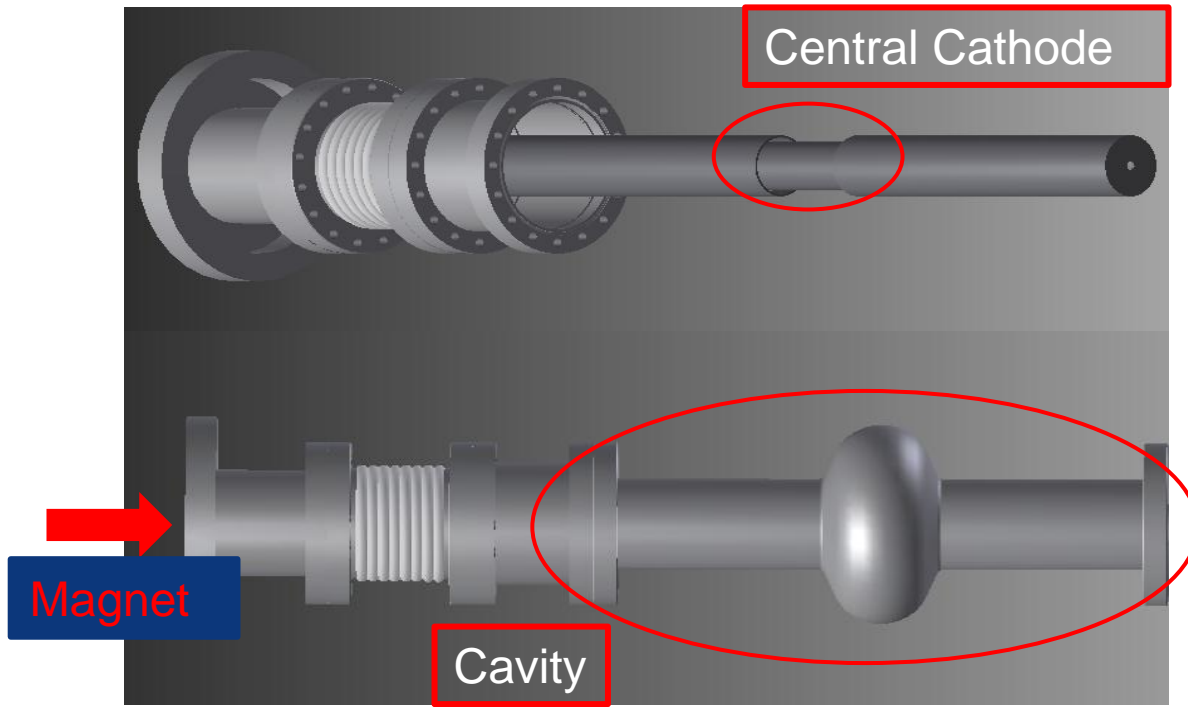
Nb/Cu: 3.3 K



Joachim Tuckmantel - Thermal effects in superconducting RF cavities: some new results from an improved program
CERN-EF-RF-84-6. - 1984.

Quenches cannot happen because of the stabilising effect of the copper with respect to thermo-magnetic breakdown

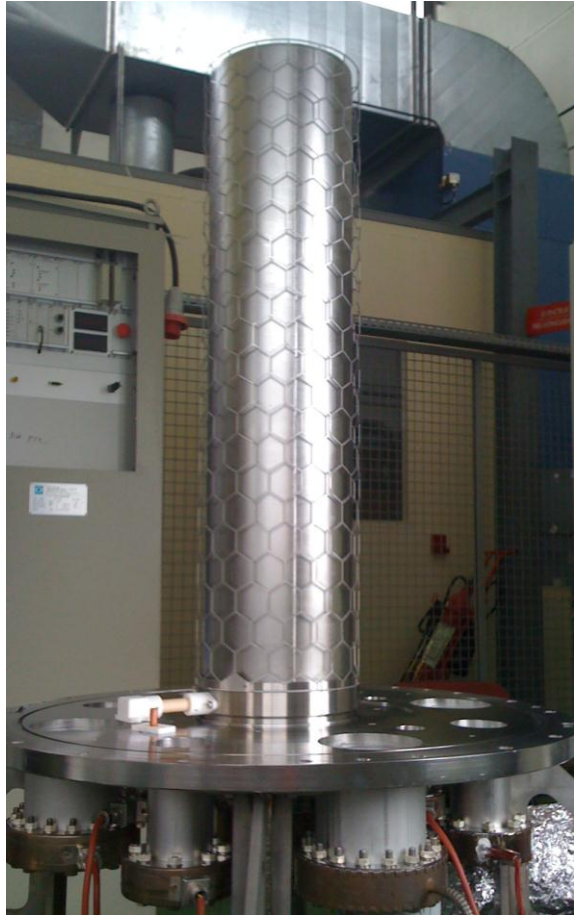
Niobium Magnetron Sputtering



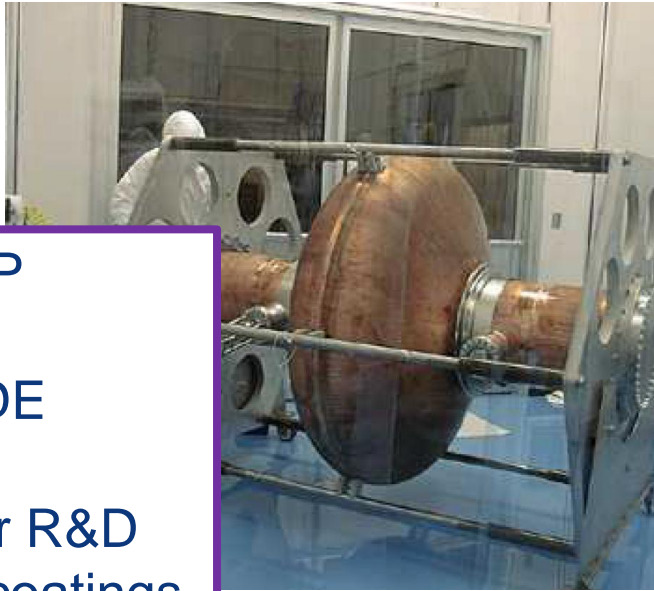
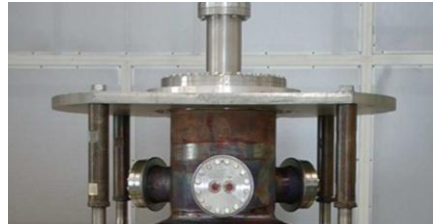
- Sputtering parameters (1.5 GHz):
 - Sputter gas pressure of 1.5×10^{-3} mbar (Ar or Kr)
 - Plasma current stabilized at 3A - DC
 - Sputter potential ~ -360 V
 - Coating temperature is 150 °C.

- “Standard films” characteristics:
 - RRR: 11.5 ± 0.1
 - Argon content: 435 ± 70 ppm
 - Grain size: 110 ± 20 nm
 - T_c : 9.51 ± 0.01 K
 - Strain: $\Delta a_{\perp} / a_{\perp} = 0.636 \pm 0.096$ %

Diode sputtering

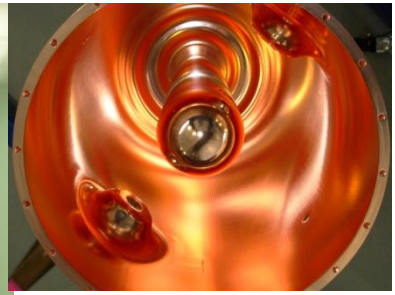
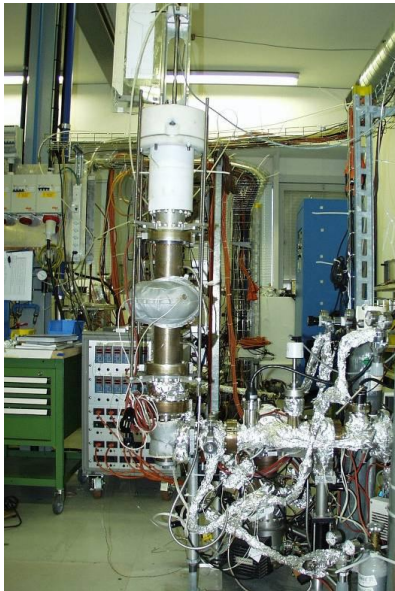


Coating of beta = 1 and lower beta cavities

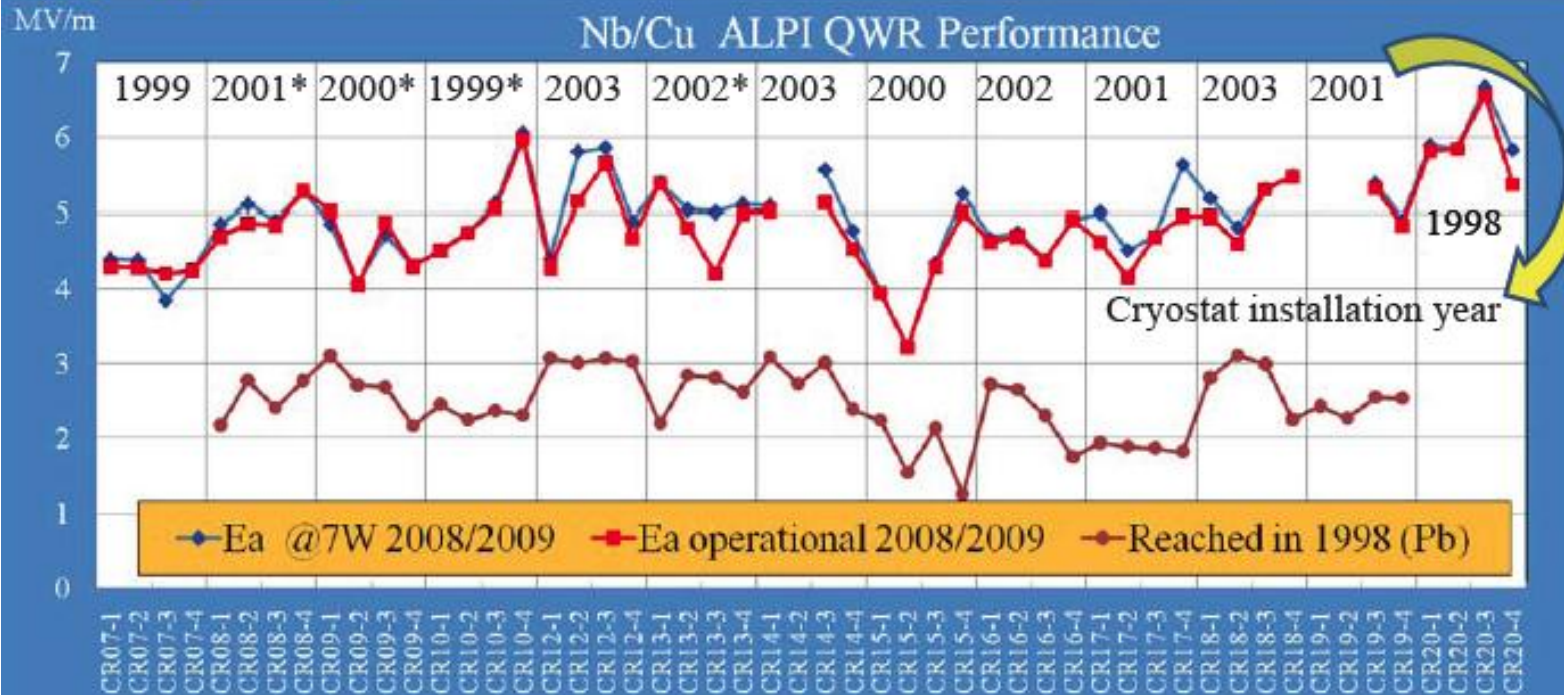


288 Nb/Cu cavities installed in LEP
16 Nb/Cu cavities installed in LHC
32 Nb/Cu projected for HIE-ISOLDE

About 300 coatings on 1.5 GHz for R&D
Low-beta studies, several 10's of coatings
(+ Soleil prototypes, 3HC, 200 MHz
Cornell...)



Nb/Cu ALPI QWR Performance



Nb/Cu Average Ea @7W: 4.83MV/m
 Nb/Cu Operational Ea : 4.70 MV/m
 Pb/Cu Average Ea: 2.48 MV/m

* Recent cryostat maintenance

56 cavities total

Effect of external magnetic field

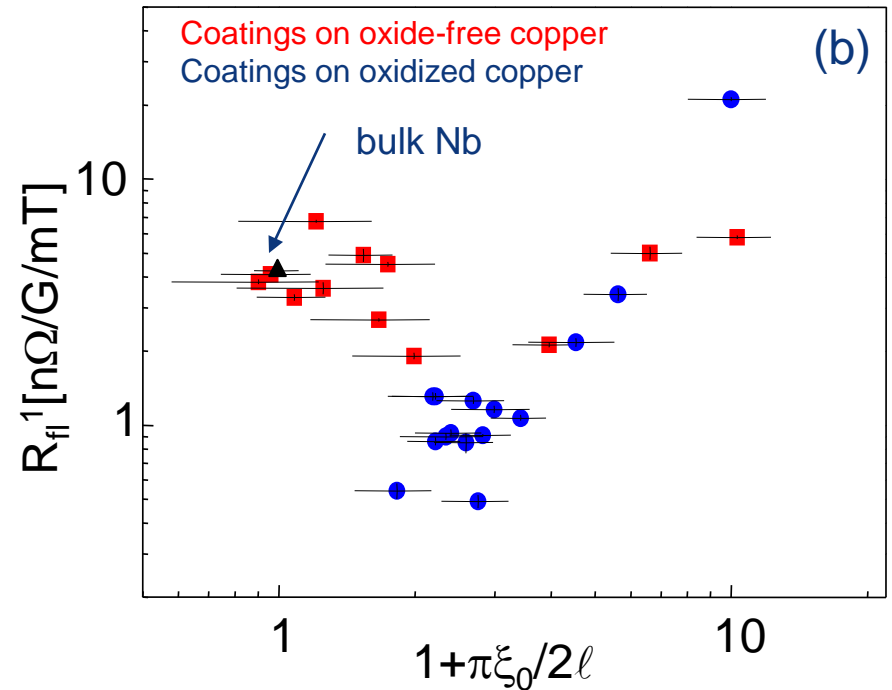
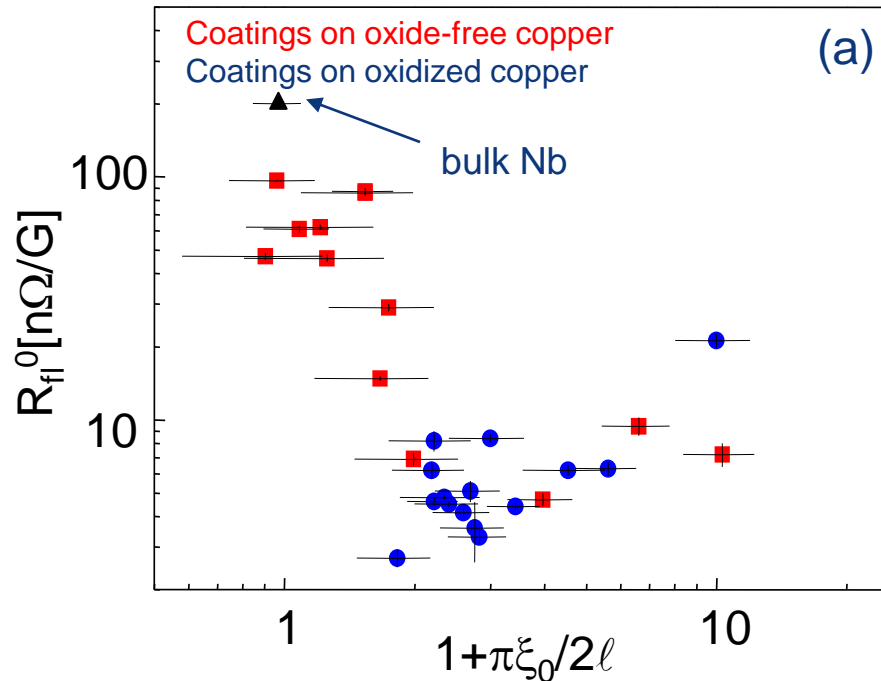
Losses due to trapped external magnetic field at 1.7 K are characterized as

$$R_{\text{fl}} = (R_{\text{fl}}^0 + R_{\text{fl}}^1 H_{\text{RF}}) H_{\text{ext}}$$

The minimum values are obtained using krypton as sputter gas:

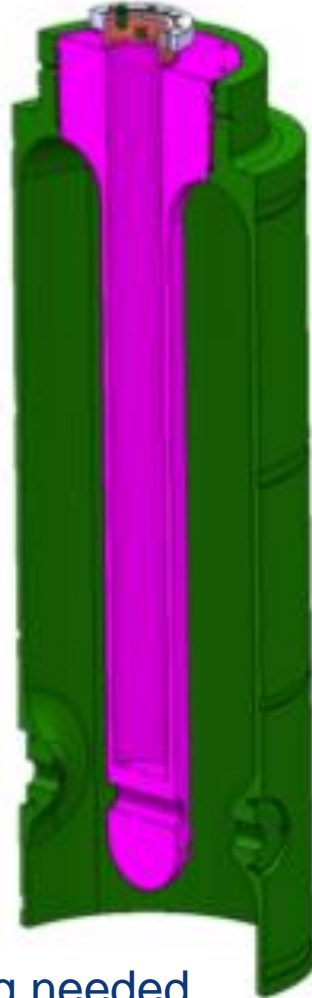
$$R_{\text{fl}}^0 = 3 \text{ n}\Omega/\text{G}$$

$$R_{\text{fl}}^1 = 0.4 \text{ n}\Omega/\text{G/mT}$$



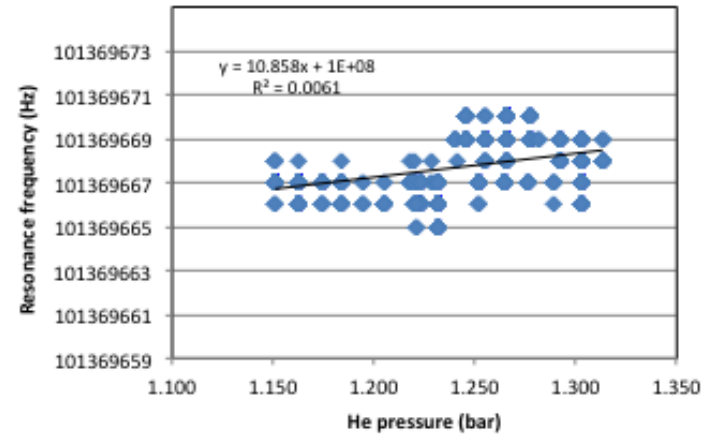
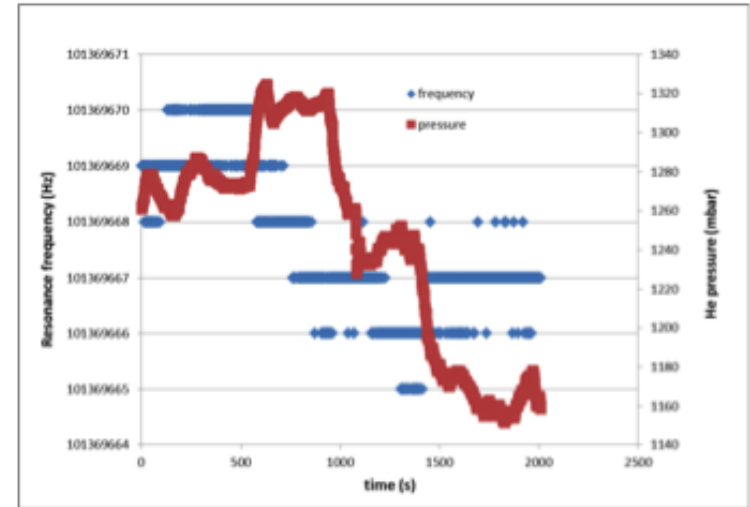
No need for magnetic shielding considerably simplifies cryostat design

Mechanical stability



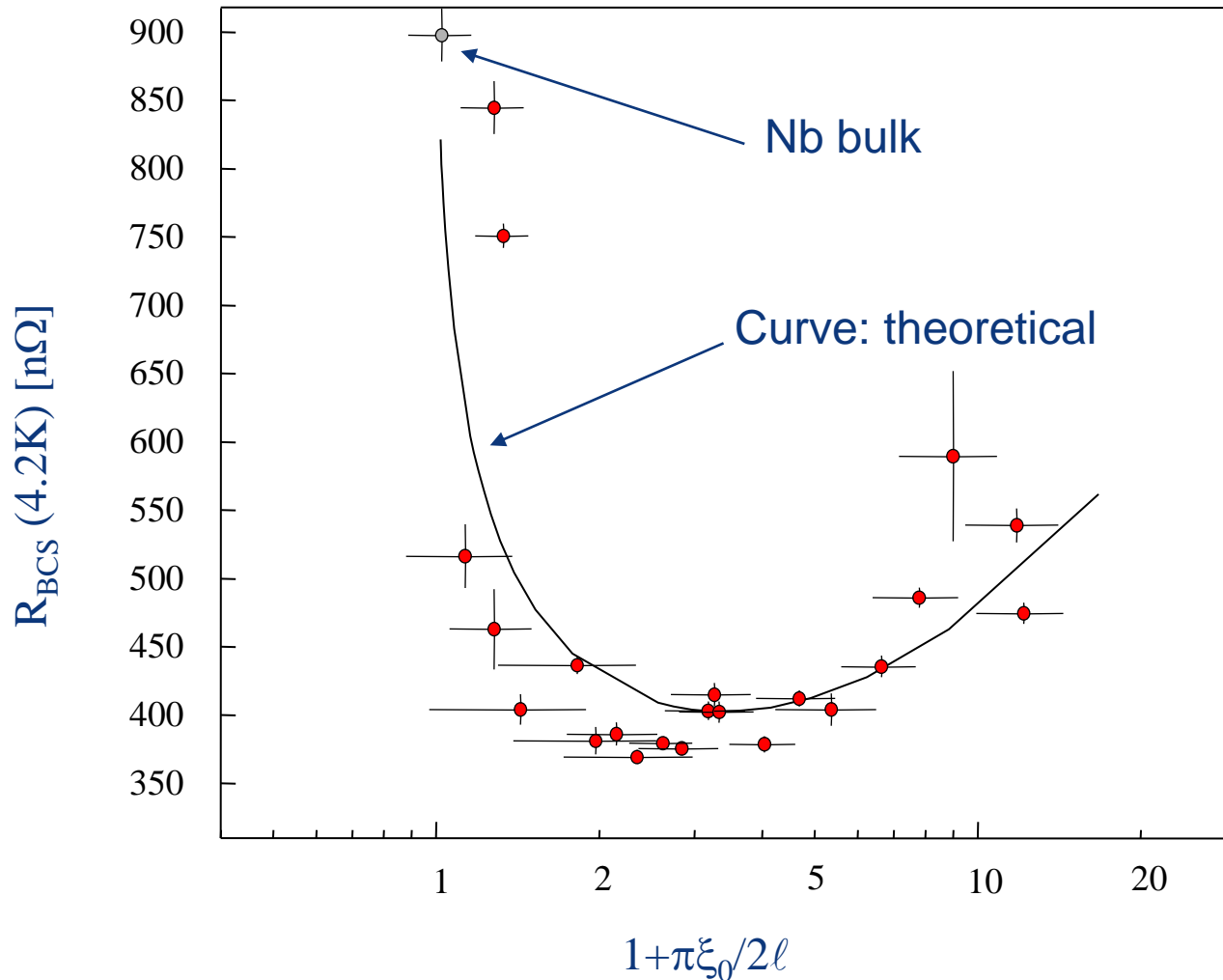
No dynamic tuning needed

QP1 (new design) ~ 0.01 Hz/mbar



Much easier operation compared to bulk:
lower microphonics, feedback systems, etc.

BCS resistance at zero RF field



R_{BCS} at 4.2 K
(1.5 GHz)

Nb bulk: ~900 nΩ

Nb films: ~400 nΩ

R_{BCS} at 1.7 K
(1.5 GHz)

Nb bulk: ~2.5 nΩ

Nb films: ~1.5 nΩ

Lowest R_{BCS} allows achieving highest Q values even at 1.7 K

LEP success rate

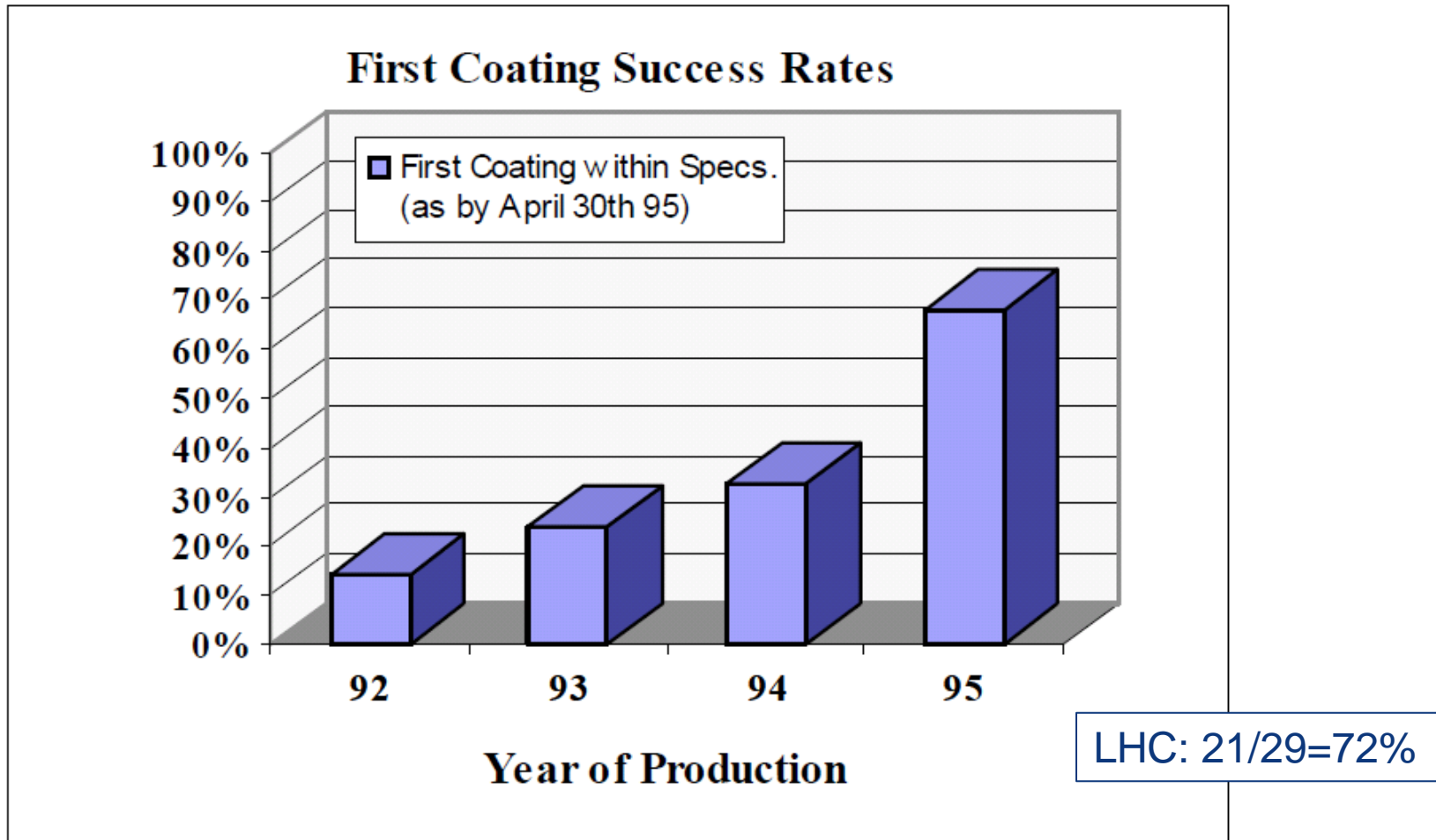


Fig.5: Total first coating success rates in industry

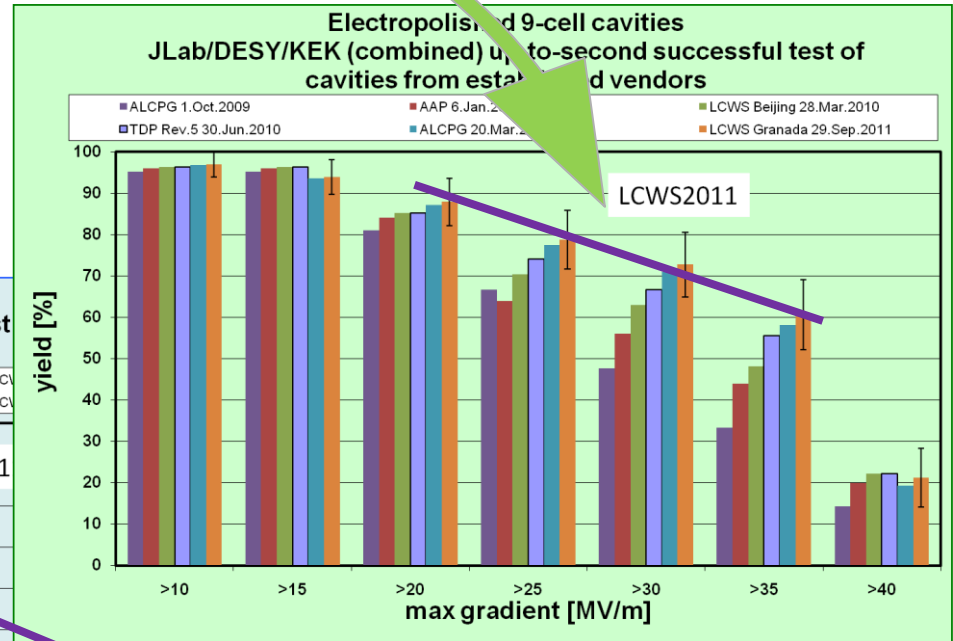
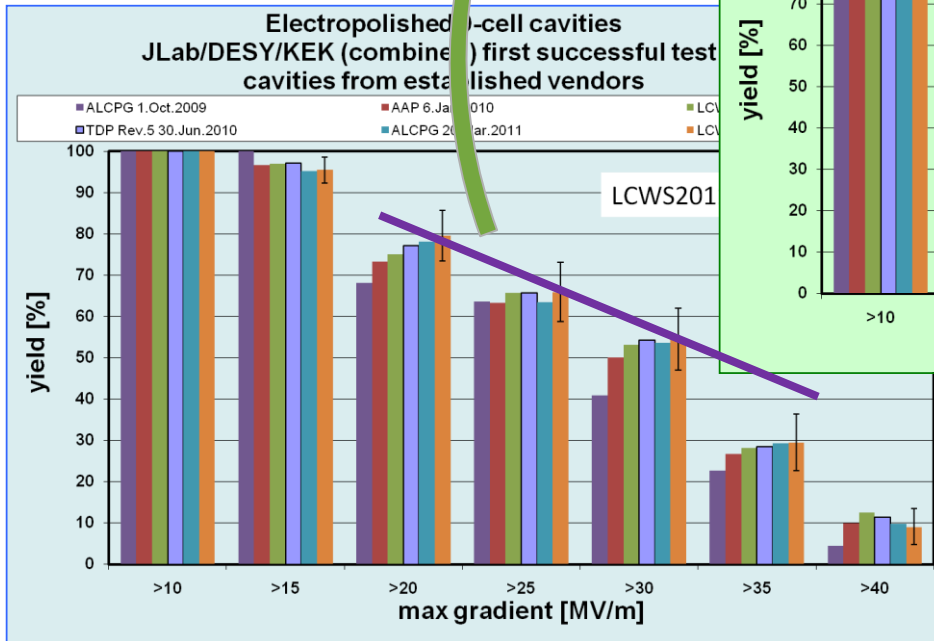
ILC success rate



Gradient Yield: Time Progression

with most recent update, Sept. 2011

1st pass

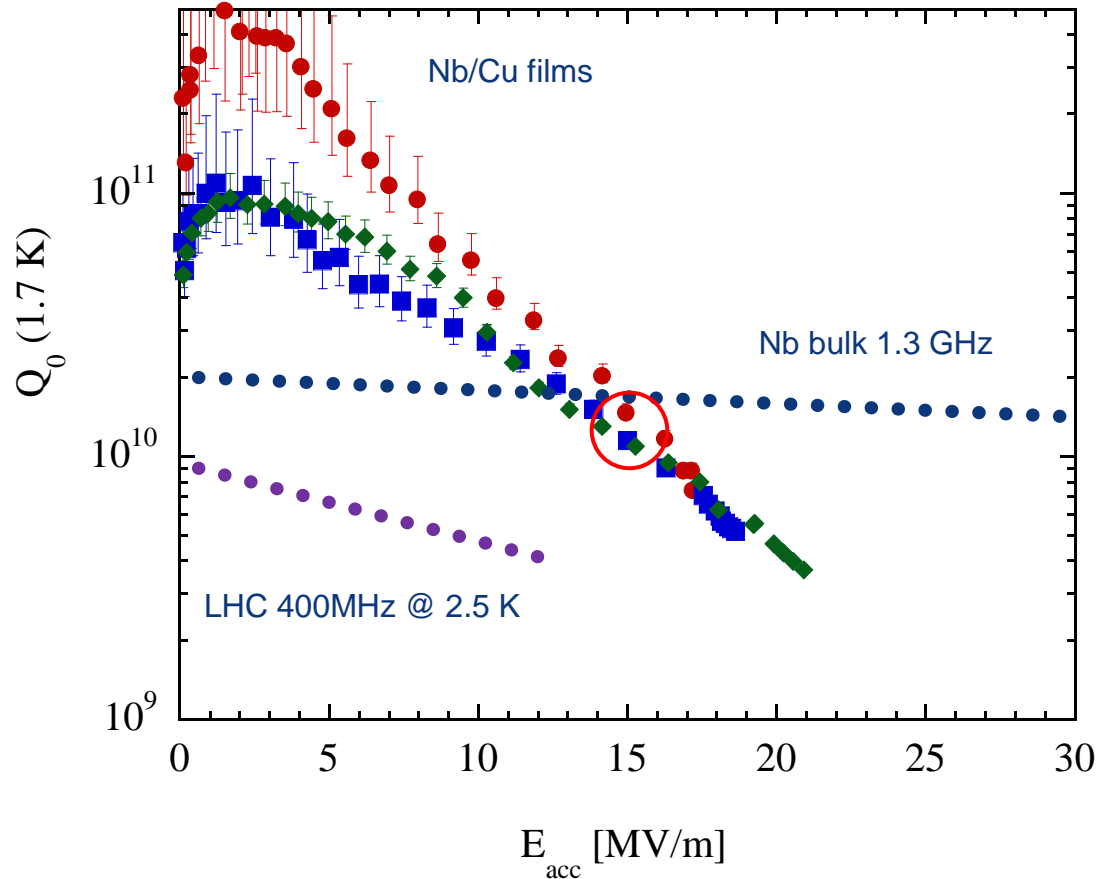


2nd pass



State of the art

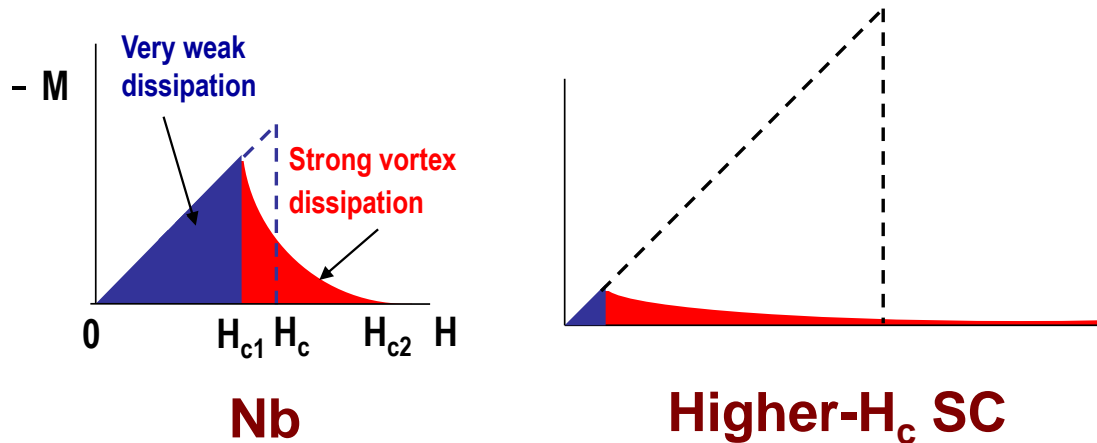
1.5 GHz Nb/Cu cavities, sputtered with Kr @ 1.7 K ($Q_0=295/R_s$)



$Q = 1 \times 10^{10}$ @ 15 MV/m is a value that would make film cavities a competitive option for new high energy proton accelerators

Limitation of H_{c1} : from A. Gurevich

Superconducting Materials



Very weak dissipation at $H < H_{c1}$ ($Q = 10^{10}-10^{11}$)
 Q drop due to vortex dissipation at $H > H_{c1}$

Nb has the highest lower critical field H_{c1}

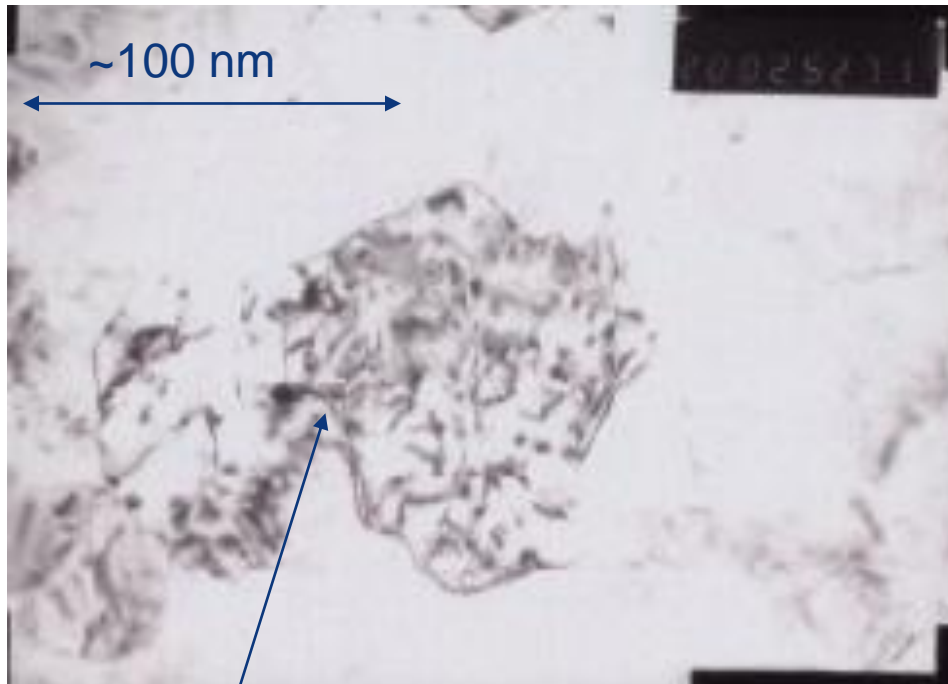
Material	T_c (K)	$H_c(0)$ [T]	$H_{c1}(0)$ [T]	$H_{c2}(0)$ [T]	$\lambda(0)$ [nm]
Pb	7.2	0.08	na	na	48
Nb	9.2	0.2	0.17	0.4	40
Nb ₃ Sn	18	0.54	0.05	30	85
NbN	16.2	0.23	0.02	15	200
MgB ₂	40	0.43	0.03	3.5	140
YBCO	93	1.4	0.01	100	150
Nb films	9.45	0.2	0.03÷0.05	0.8÷1.2	40

$$H_{c1} = \frac{\phi_0}{4\pi\lambda^2} \left(\ln \frac{\lambda}{\xi} + 0.5 \right)$$

Thermodynamic critical field H_c (surface barrier for vortices disappears)

$$H_c = \frac{\phi_0}{2\sqrt{2}\pi\lambda\xi}$$

TEM view – film defects



Crystallographic defects can be at the origin of reduced H_{c1} compared to bulk Nb

$$\frac{1}{\ell_{total}} = \frac{1}{\ell_{intragrain}} + \frac{1}{D}$$

RRR of films: $10 \div 30$

⇒

mfp of films $30 \div 100$ nm

Grain size of films > 100 nm

⇒

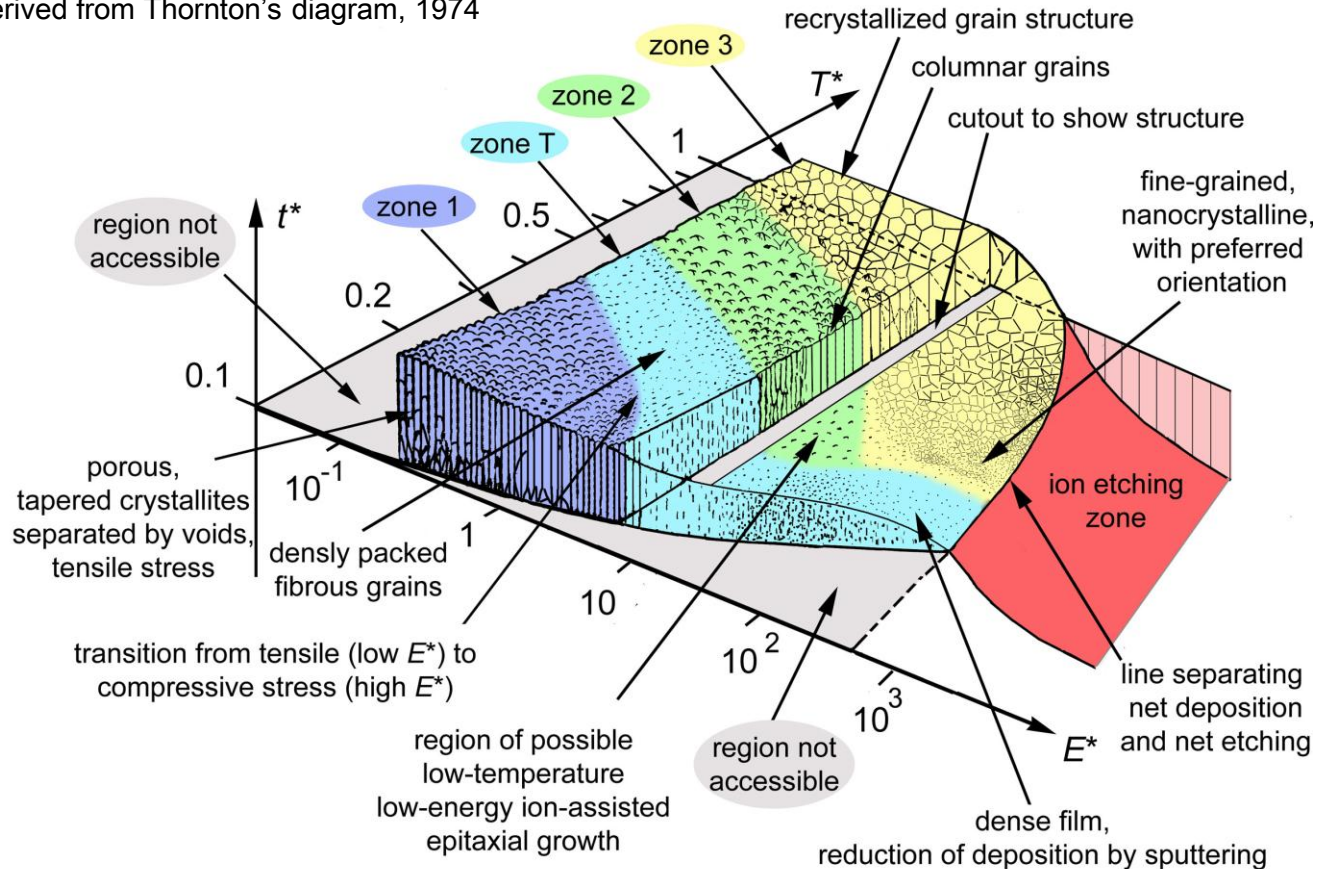
RRR limited by intragrain defects in most cases

The goal is to make films as bulk-like as possible in terms of microstructure structure. The grain size does not seem to be a major issue

Structure zone model from A. Anders

Generalized Structure Zone Diagram

derived from Thornton's diagram, 1974



© Andre Anders, 2010

A. Anders, Thin Solid Films **518**, 4087 (2010).

Energetic condensation

Additional energy provided by fast ionised particles arriving at a surface with energy $\gg eV$

⇒ Changes in the film growth process:

- ❑ Residual gases are desorbed from the substrate surface
- ❑ Chemical bonds are easily broken, thus affecting nucleation processes & film adhesion
- ❑ Enhanced mobility of surface atoms
- ❑ Arriving Nb ions penetrate 2-3 atomic layers
- ❑ Ions arrive with normal incidence

- ⇒ Changes in
- Morphology
 - Microstructure
 - Stress

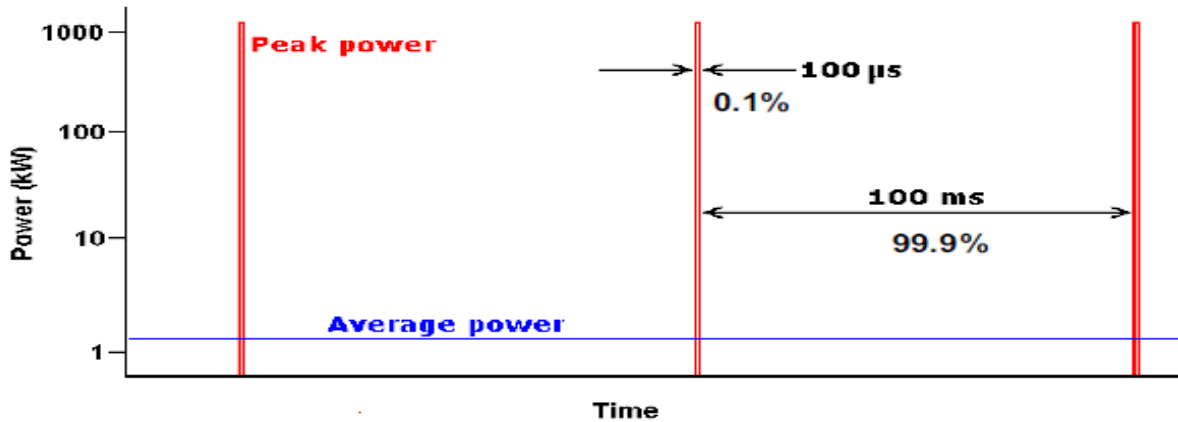
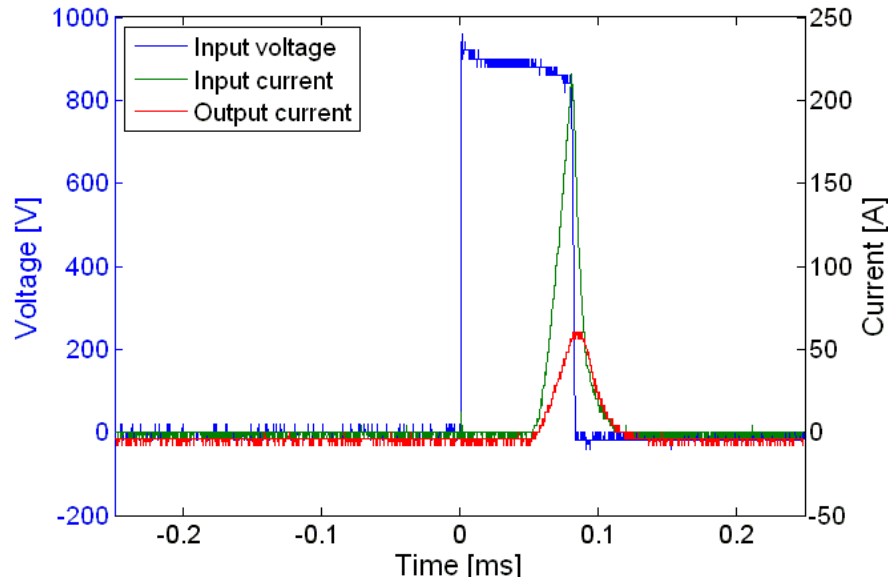
Energetic condensation around the world

	Deposition Methods	Deposition Rate (nm/s)	Ions Energy (eV)	Ion Flux (Nb ions/cm ² -s)	Flux Power (W/cm ²)
1	Magnetron Sputtering, CERN	1.67	<1 (therm.)	~3X10 ¹⁵ (neutrals)	2.4X10 ⁻³
2	HIPIMS Sputtering, CERN and LBNL	0.1	10-80 (bias)	~3X10 ¹⁴	2.4X10 ⁻³
3	UHV Cathodic Arc, INFN + IPJ	10 (no filtered) 2 (filtered)	10-100 (bias)	~1.8X10 ¹⁶ (no filtered)	2.9X10 ⁻¹ (DC Mode)
4	Cathodic Arc, AASC CED™	560 (instant rate, only last 1ms, duty cycle 0.05%)	100-170 (bias)	~1X10 ¹⁸ (inst. rate, last 1ms)	16 (inst. rate, last 1ms)
		0.28 (~1 monolayer/s) (avg. rate.)		~5X10 ¹⁴ (avg. rate)	8X10 ⁻³ (avg. rate)
5	ECR Nb Plasma, JLAB	2.25[new] 0.5	>63 64-280	~1X10 ¹⁵	~7 X10 ⁻² (at 100eV)



Elaborated from a table by C. Reece, JLAB

HIPIMS at CERN

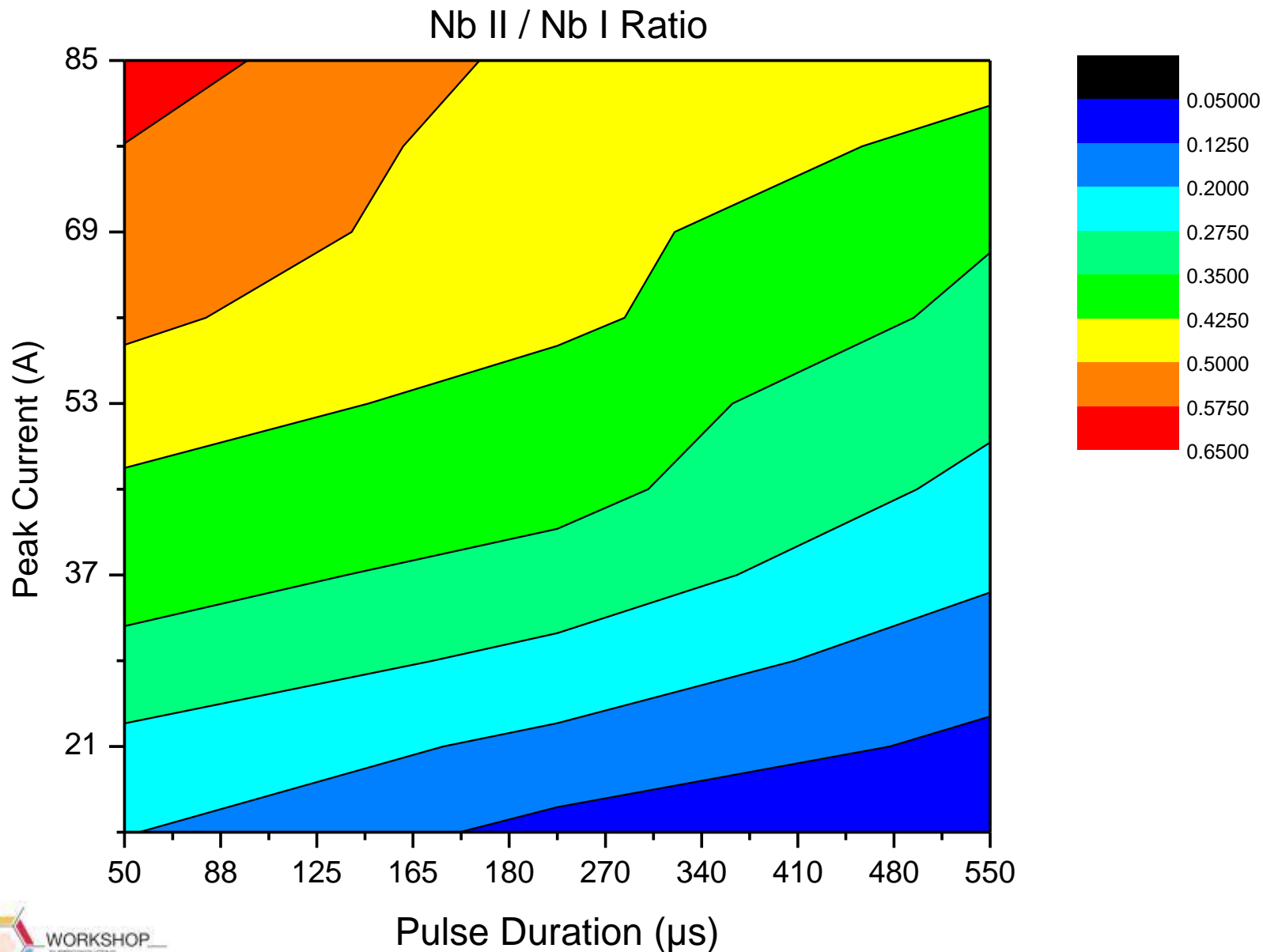


Example of a duty cycle.

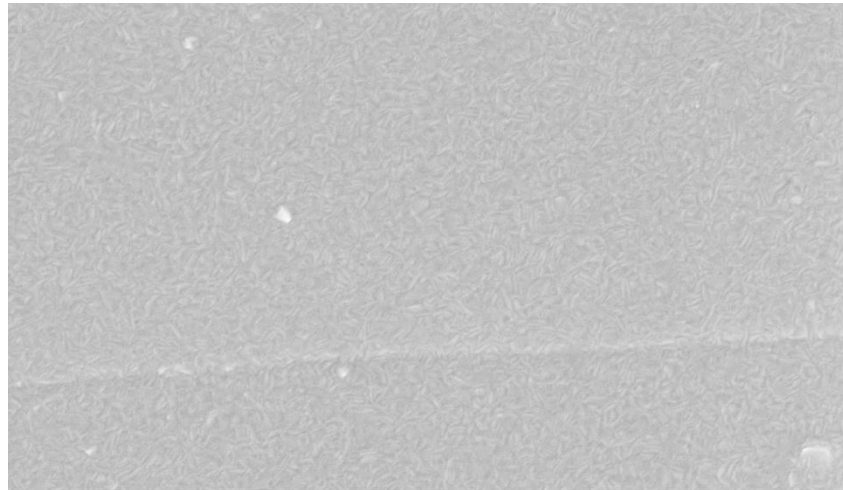


The amount of sputtered Nb in a pulse can be so large that its “pressure” is larger than the noble gas pressure. As a consequence also the Nb becomes ionized

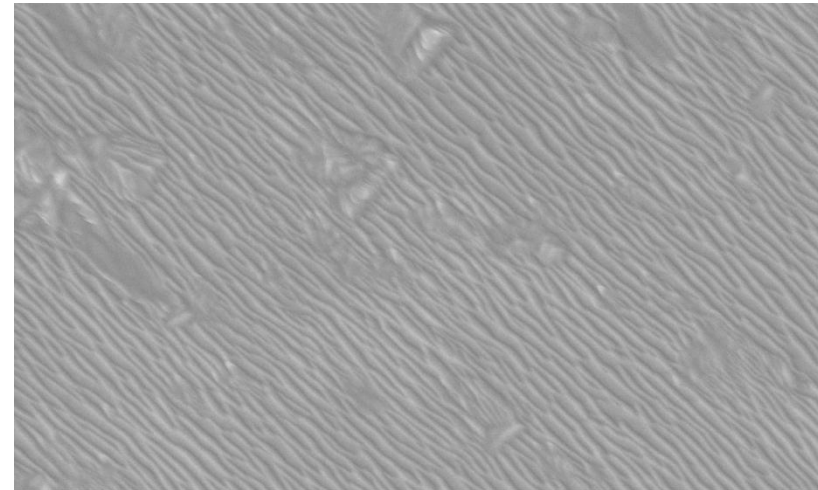
Ionisation ratio in HIPIMS



Comparison between DCMS and HiPIMS

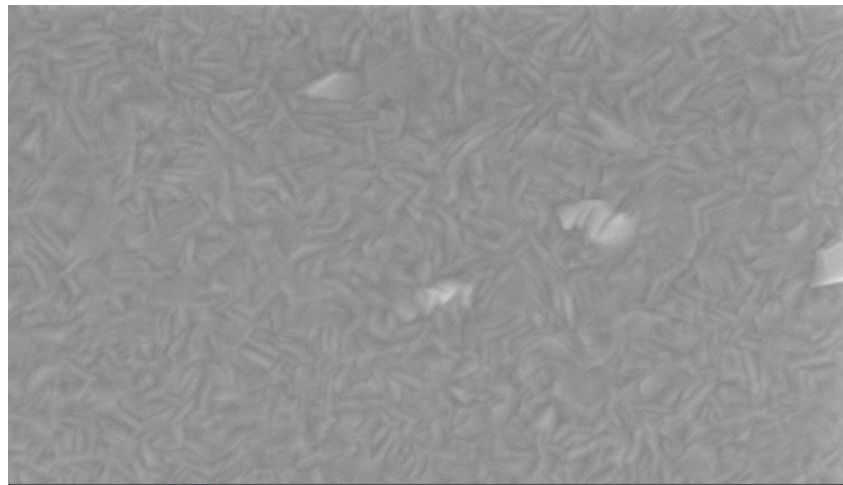


200 nm  EHT = 5.00 kV
WD = 0.9 mm Nb coating on Cu
DCMS Mag = 20.00 K X
Signal A = InLens Sample 1 Ignacio Aviles
Date :26 Apr 2012 

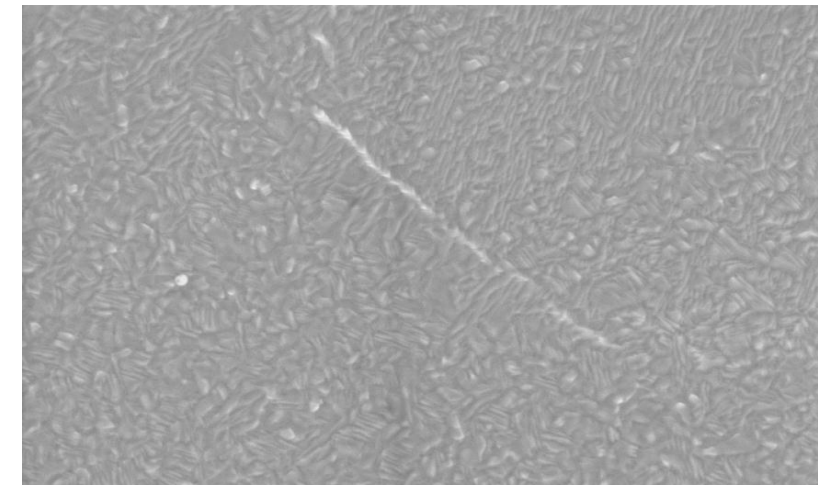


200 nm  EHT = 5.00 kV
WD = 0.7 mm Nb coating on Cu
HiPIMS Mag = 20.00 K X
Signal A = InLens Sample 2 Ignacio Aviles
Date :26 Apr 2012 

HiPIMS High Power (unbiased)



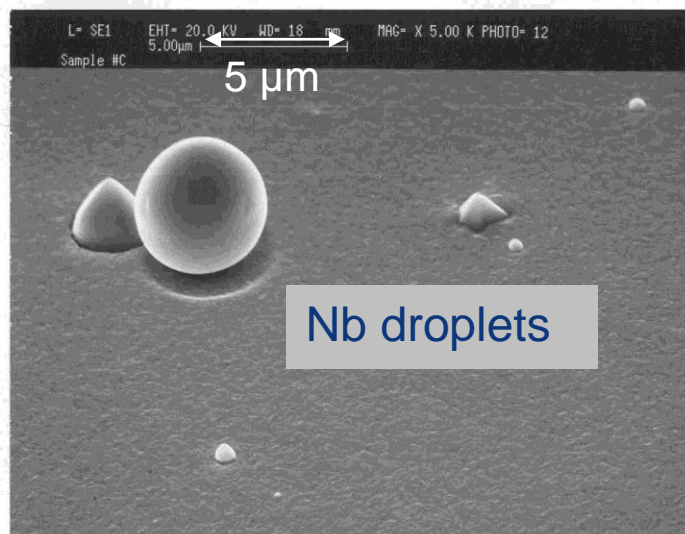
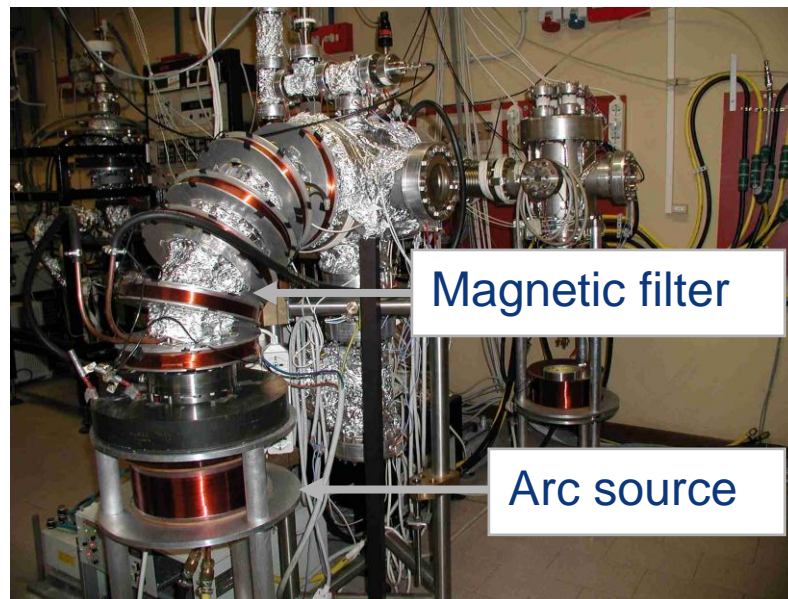
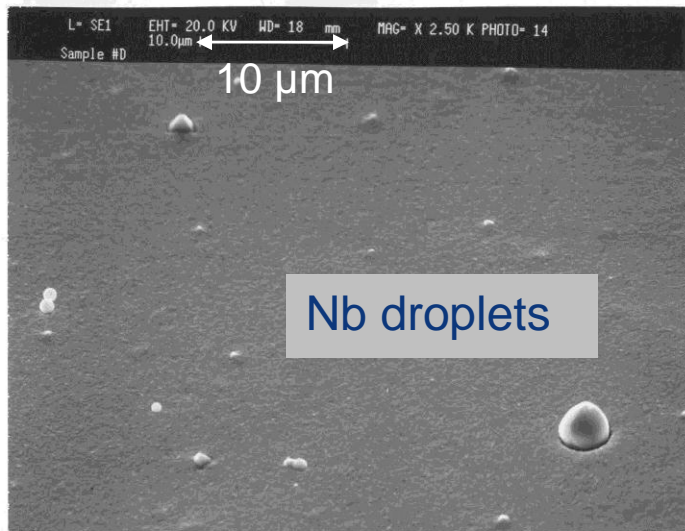
100 nm  EHT = 5.00 kV
WD = 0.9 mm Nb coating on Cu
DCMS Mag = 60.00 K X
Signal A = InLens Sample 1 Ignacio Aviles
Date :26 Apr 2012 



200 nm  EHT = 3.00 kV
WD = 1.2 mm HIE-ISOLDE
HiPIMS Nb coating on Cu Mag = 20.00 K X
Signal A = InLens Run 4 Holder 1 Ignacio Aviles
Date :23 Mar 2012 

HiPIMS Low Power (unbiased)

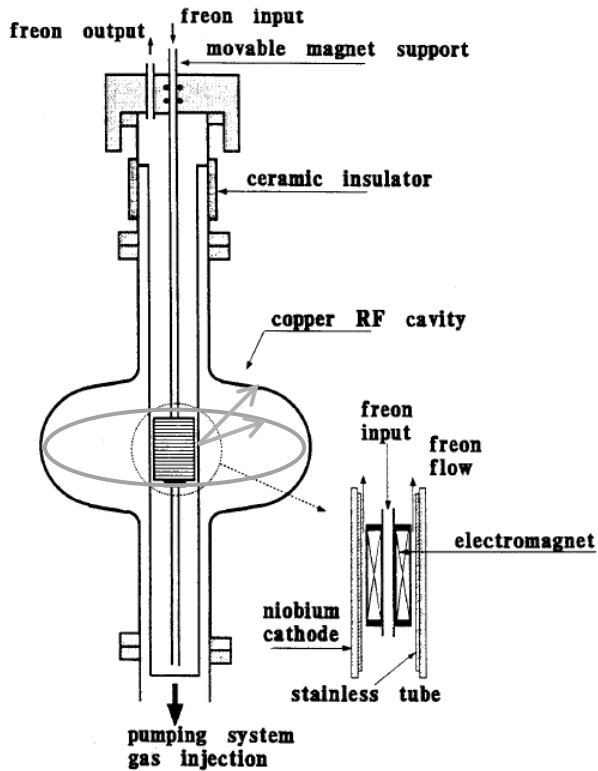
Plasma Arc INFN



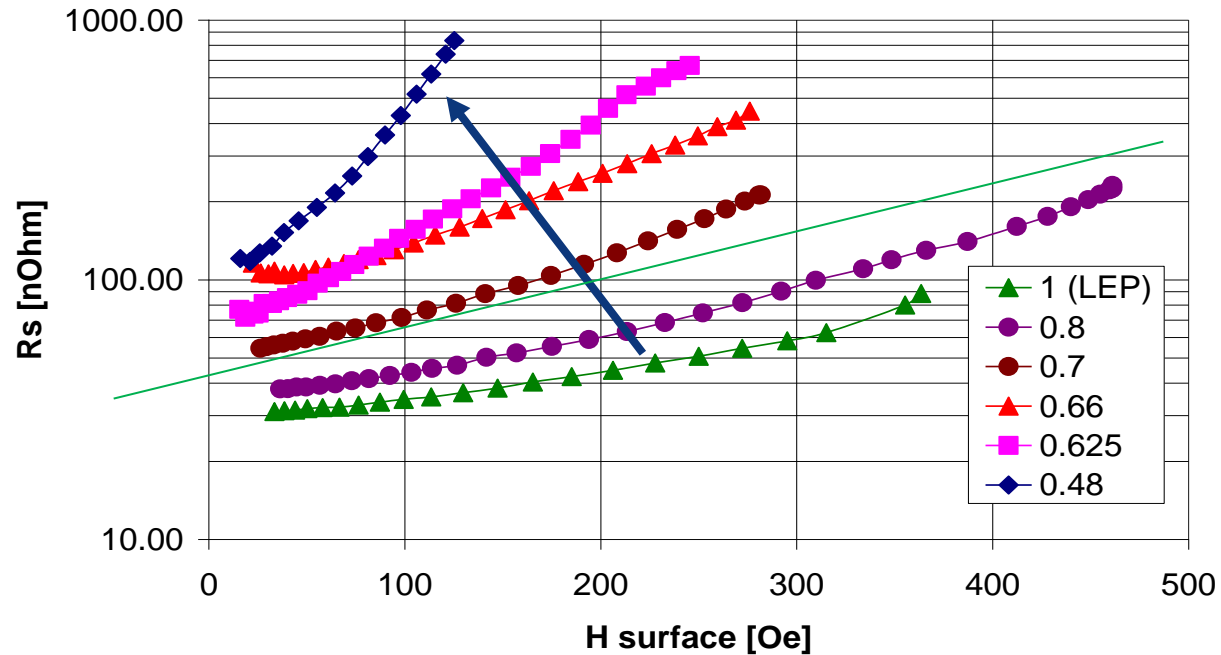
Linear arc with coaxial blind-shutter filter



Angle of incidence of the coating



Average angle of incidence increases

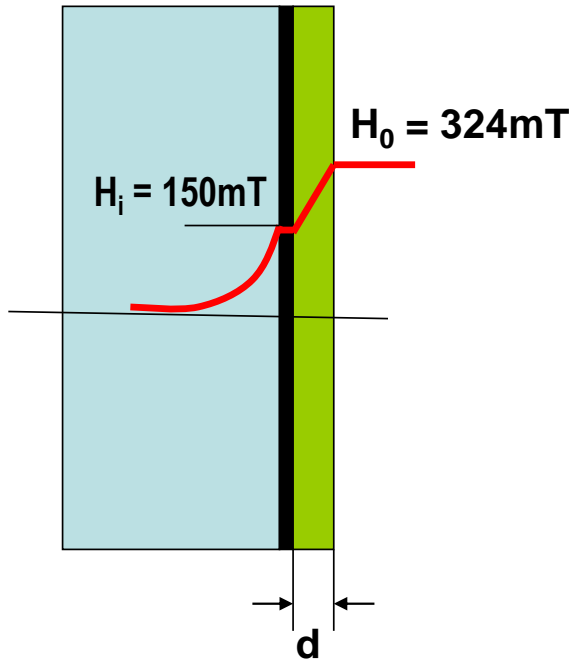


There is a "threshold" effect

Energetic condensation of ions with addition of a bias will likely help solving this problem

Multilayers. From A. Gurevich

A minimalistic solution



A Nb cavity coated by a single Nb_3Sn layer of thickness $d = 50\text{nm}$ and an insulator layer in between

If the Nb cavity can withstand $H_i = 150\text{mT}$, then the external field can be as high as

$$H_0 = H_i \exp(d / \lambda_0) = 150 \exp(50 / 65) = 323.7\text{mT}$$

Lower critical field for the Nb_3Sn layer with $d = 50\text{ nm}$ and $\xi = 3\text{nm}$: $H_{c1} = 1.4\text{T}$ is much higher than H_0

A single layer coating more than doubles the breakdown field with no vortex penetration, enabling $E_{\text{acc}} \sim 100\text{ MV/m}$

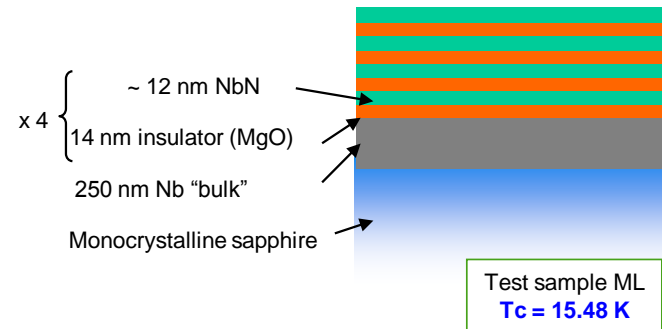
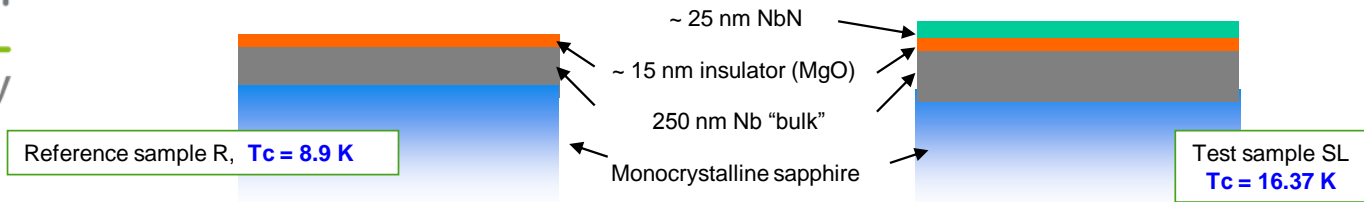
Multilayers by sputtering

First exp. results on high quality model samples

I r f u
cea
saclay

Choice of NbN:

- ML structure = close to Josephson junction preparation (SC/insulator compatibility)
- Use of asserted techniques for superconducting electronics circuits preparation:
 - Magnetron sputtering
 - Flat monocrystalline substrates

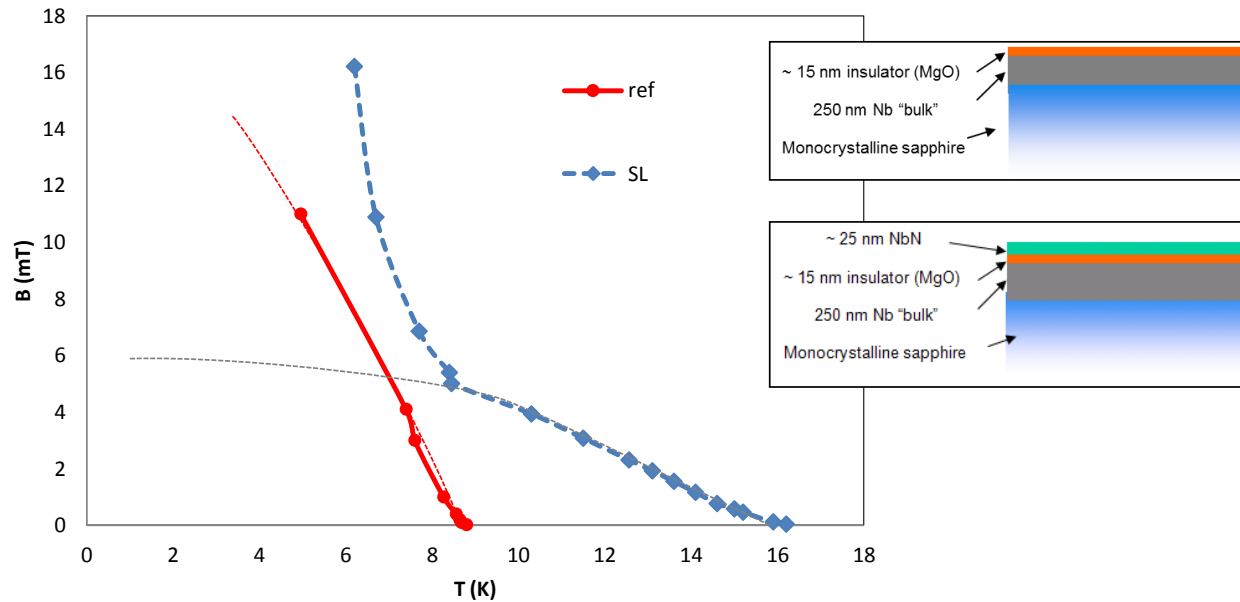


Collaboration with J.C. Villégier, CEA-Inac / Grenoble

NbN / MgO / Nb layers

Local magnetometry (3)

I r f u
cead
saclay

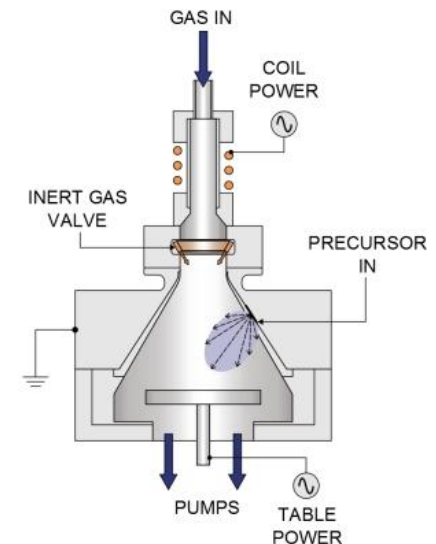
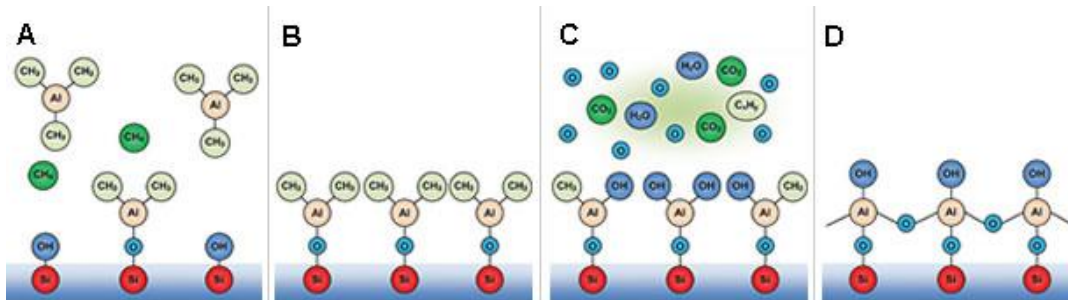


- **SL sample** : 250 nm Nb + 14 nm MgO + 25 nm NbN
- $8.90\text{K} < T_p < 16\text{K}$: behavior ~ NbN alone
- $T_p < 8.90\text{K}$, i.e. when Nb substrate is SC , $\Rightarrow B_{C1}^{SL} \gg B_{C1}^{Nb}$

A new idea: ALD

ALD (Atomic Layer Deposition): conformal coatings from gas precursors

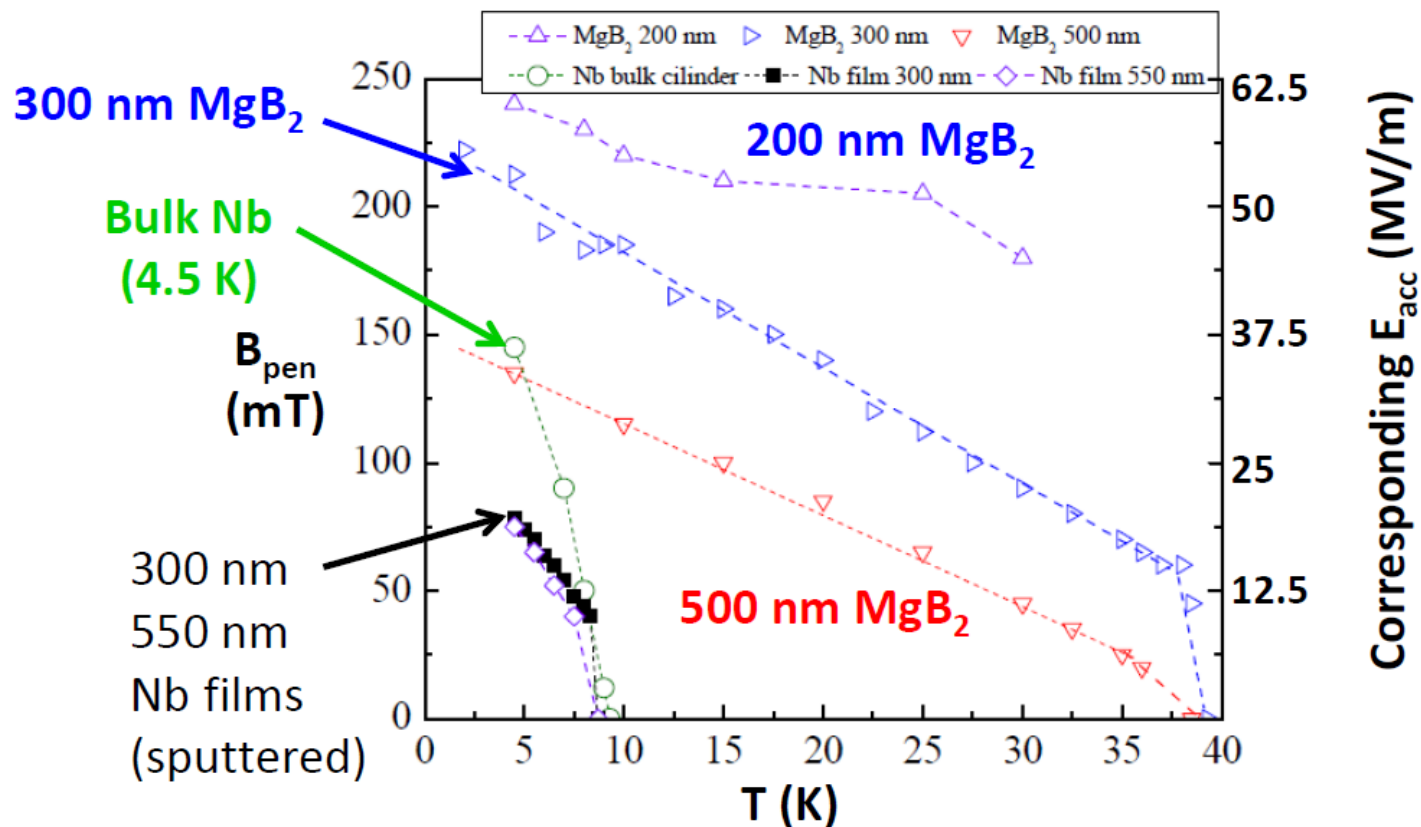
- Preliminary investigations (literature survey) done within EuCARD
- Testing going on at Argonne National Lab (USA)



Images: Oxford Instruments

MgB₂ multilayer

DC magnetization measurement results: MgB₂ thin films (<500 nm) prepared by STI show higher B_p than that of Nb



SRF2011, Chicago, IL, USA, 25-29 July 2011

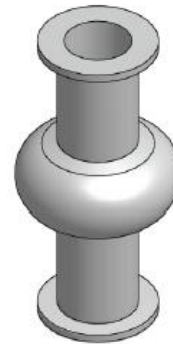
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MgB₂ thick films, preparing for cavity coating

MgB₂ work at Temple – 6 GHz Nb cavity coating system



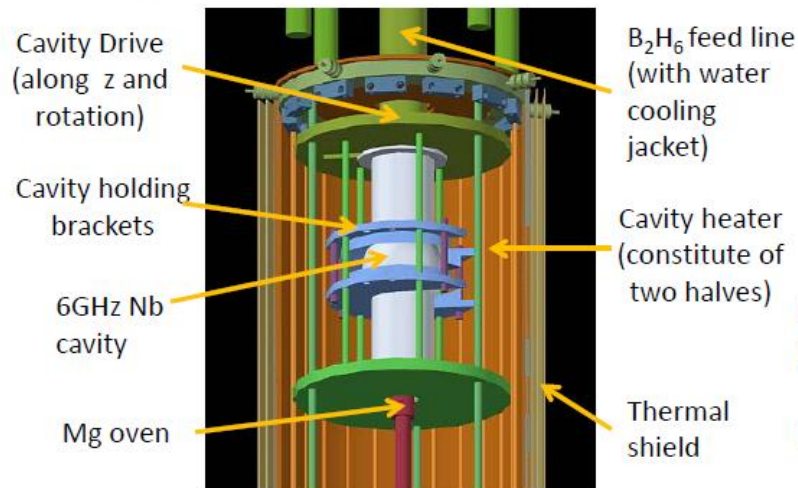
Integrated HPCVD system for cavity coating



6 GHz Nb cavity
(4" high, 1.5" dia.)



S.S. Dummy
(4" high, 1.5" dia.)



Cartoon of cavity coating system



Front view



under test



Top view

- ❑ System for in-situ coating a real 6GHz Nb cavity
- ❑ B₂H₆ and Mg feeding line fixed, the cavity will move vertically in z direction and rotate
- ❑ System is close to completion, heater testing finished, same-size stainless steel dummy will be first coated for uniformity test

Summary and outlook

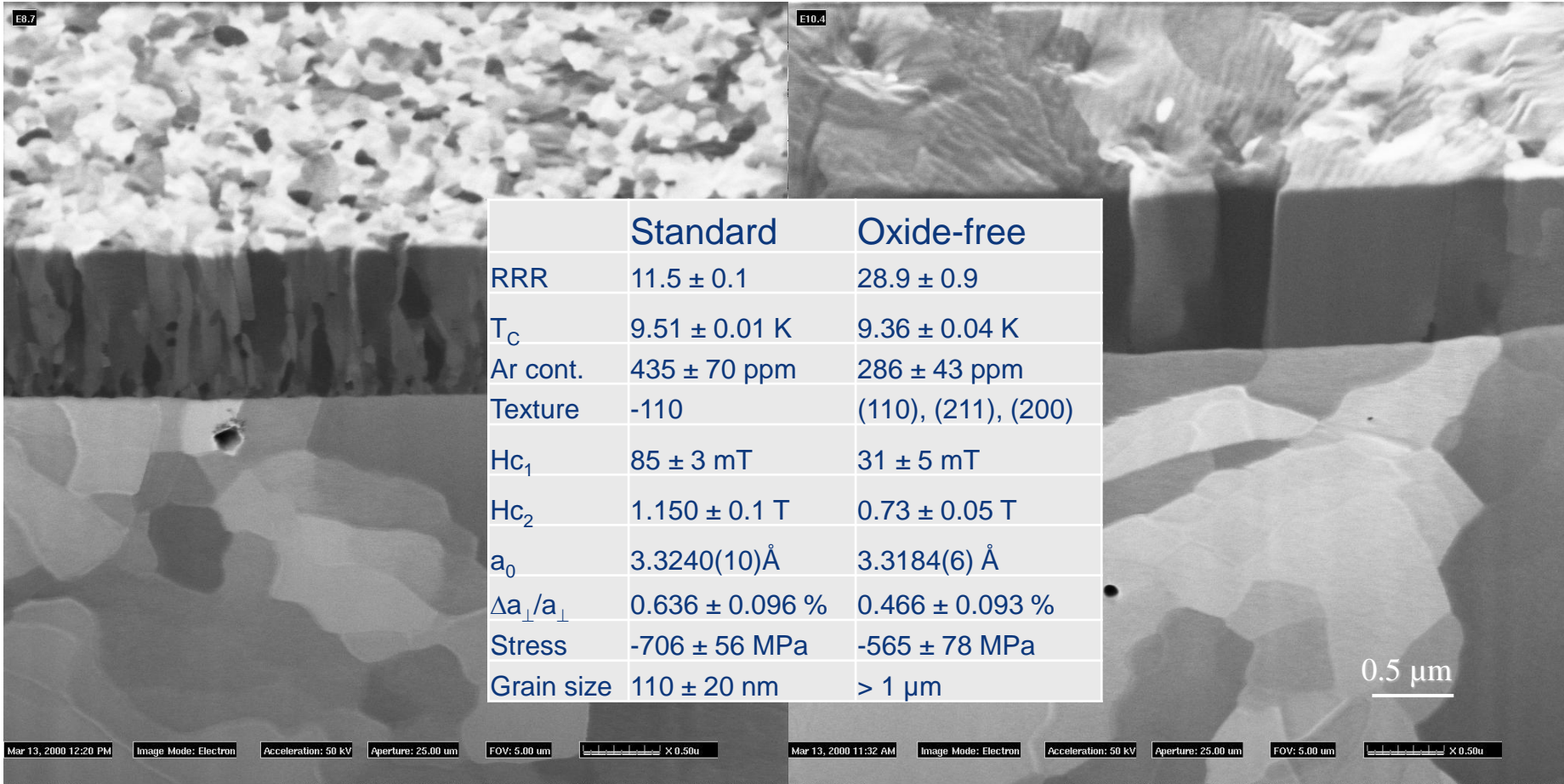
- Nb/Cu sputtered films investigated for > 25 years. State of the art results could be compatible with specs for high intensity proton accelerators.
- Nb/Cu cavities have clear advantages for operation stability. RIB facilities could benefit.
- Energetic deposition techniques have promise of improving over standard Nb/Cu -> will see soon
- Multilayers can theoretically improve even the performance of bulk Nb -> need R&D
- Higher Tc materials: potential for higher fields, probable limitation for high Q -> need R&D



Film analyses

Standard films

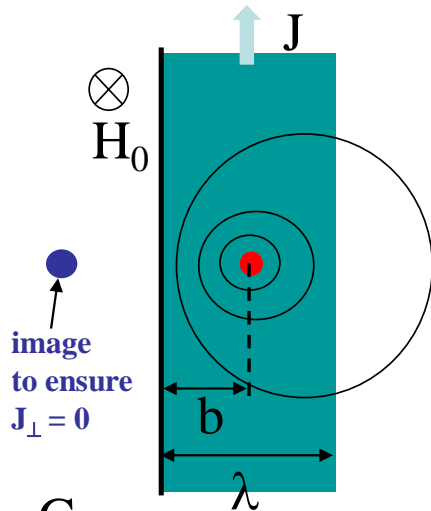
Oxide-free films



FIB cross sections courtesy: P. Jacob - EMPA

Multilayers. From A. Gurevich

Surface barrier: How do vortices get in a superconductor at $H > H_{c1}$?

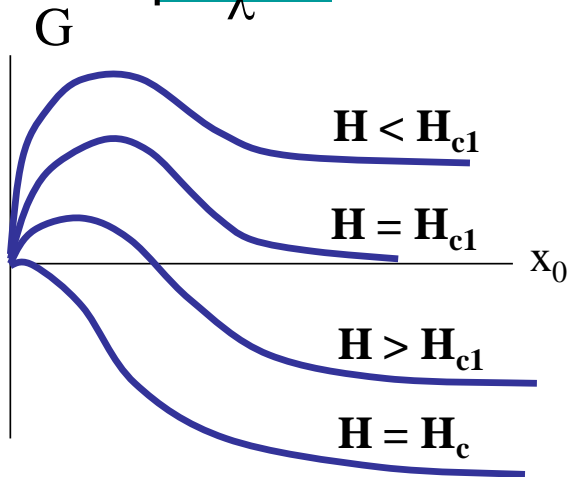


Two forces acting on the vortex at the surface:

- Meissner currents push the vortex in the bulk
- Attraction of the vortex to its antivortex image pushes the vortex outside

Thermodynamic potential $G(x_0)$ as a function of the position x_0 :

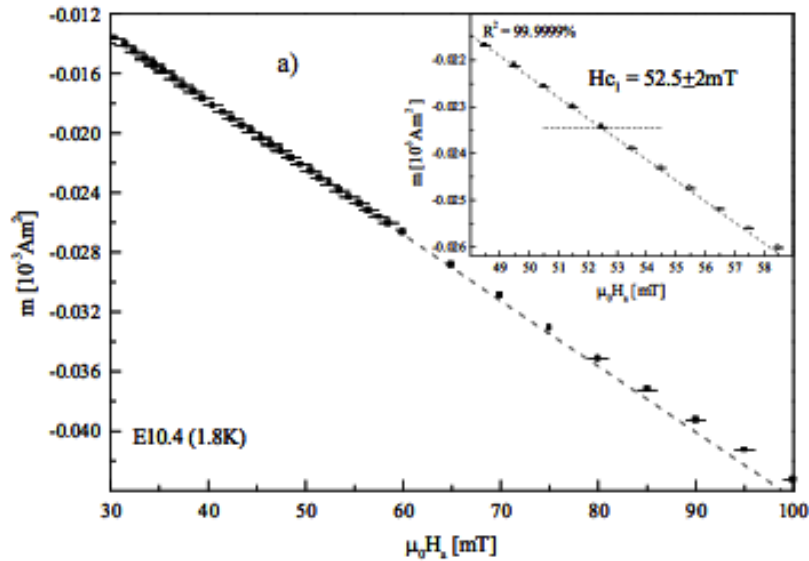
$$G(b) = \underbrace{\phi_0 H_0 e^{-x_0/\lambda}}_{\text{Meissner}} - \underbrace{0.5 H_v (2x_0)}_{\text{Image}} + H_{c1} - H_0$$



Vortices have to overcome the surface barrier even at $H > H_{c1}$ (Bean & Livingston, 1964)

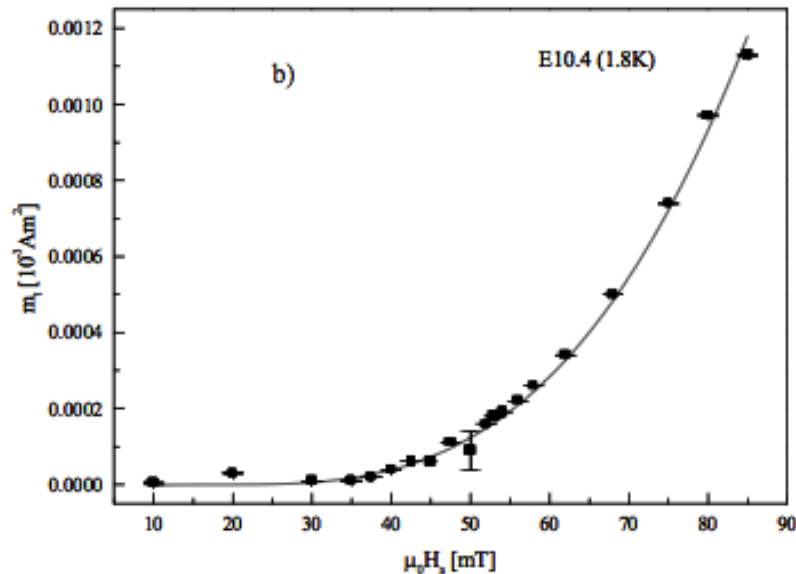
Surface barrier disappears only at the overheating field $H = H_c > H_{c1}$ at which the surface J becomes of the order of the depairing current density

Measurements of H_{c1} on Nb films



Magnetic moment with squid magnetometer filed parallel to the sample

$$H_{c1} = 52.5 \pm 2 \text{ mT}$$

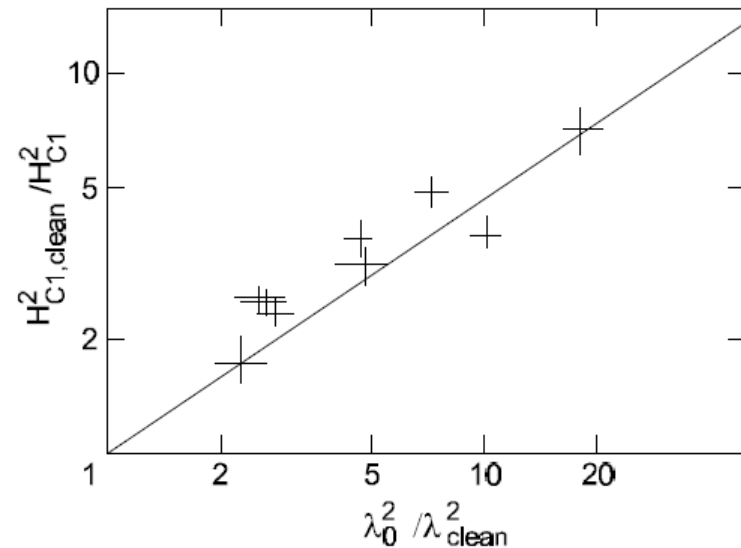
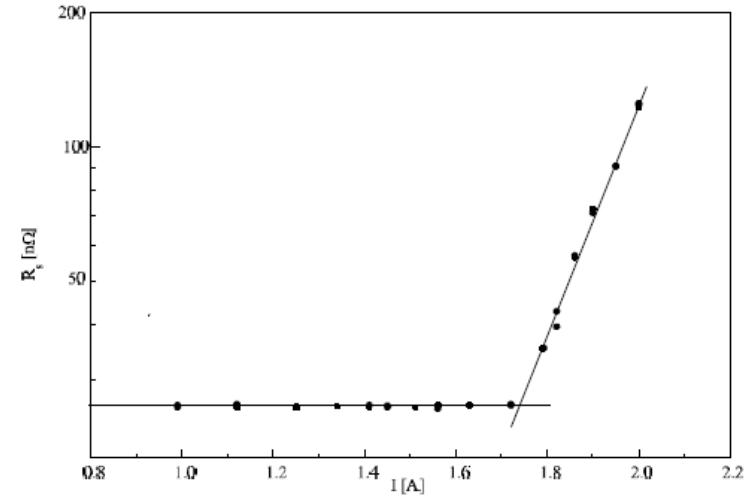
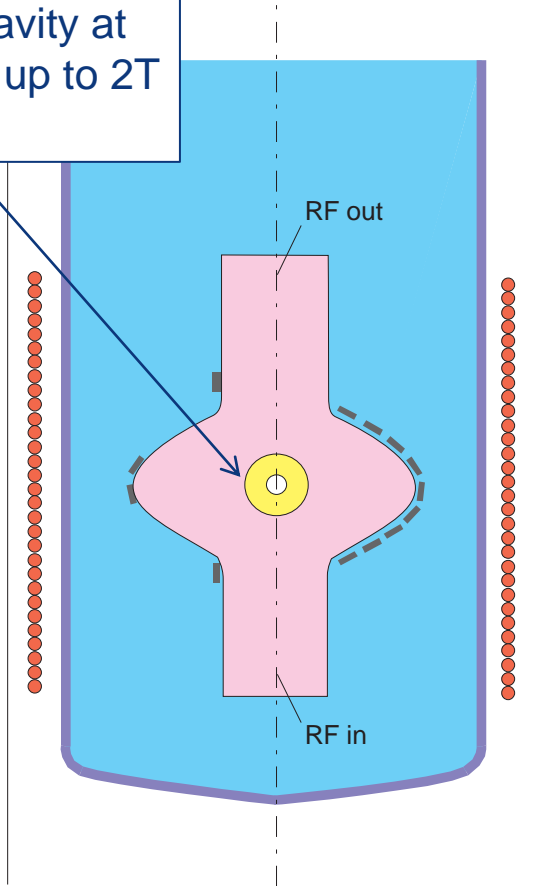


Same film, trapped magnetic moment with squid magnetometer (fit with V.V. Moshchalkov et al., Physica C 175 (1991) 407)

$$H_{c1} = 31 \pm 3 \text{ mT}$$

Measurement of H_{c1}

SC coil at the outer wall of the cavity at equator H_{ext} up to 2T over 3 cm²



$H_{penetration}$ films $\leq 0.6 H_{penetration}$ bulk

Local magnetometry (1)

I r f u



saclay

- 3rd harmonic measurement, coll. INFM Napoli

M. Aurino, et al., Journal of Applied Physics, 2005. 98: p. 123901.

Perpendicular field : field distribution can be determined analytically.

- If $r_{\text{sample}} > 4 r_{\text{coil}}$: Sample \equiv infinite plate approximation
- Applied field : perpendicular, induction (B) // surface (below B_{C1})

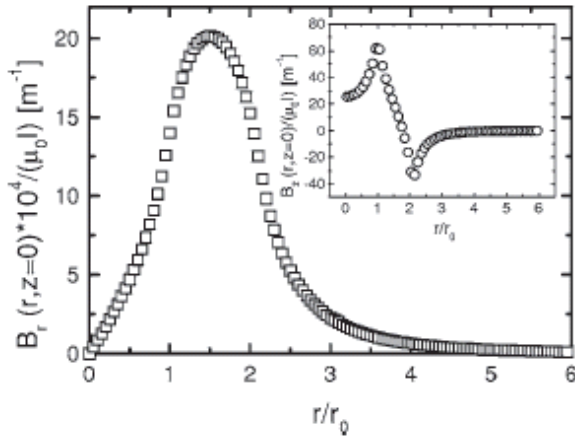
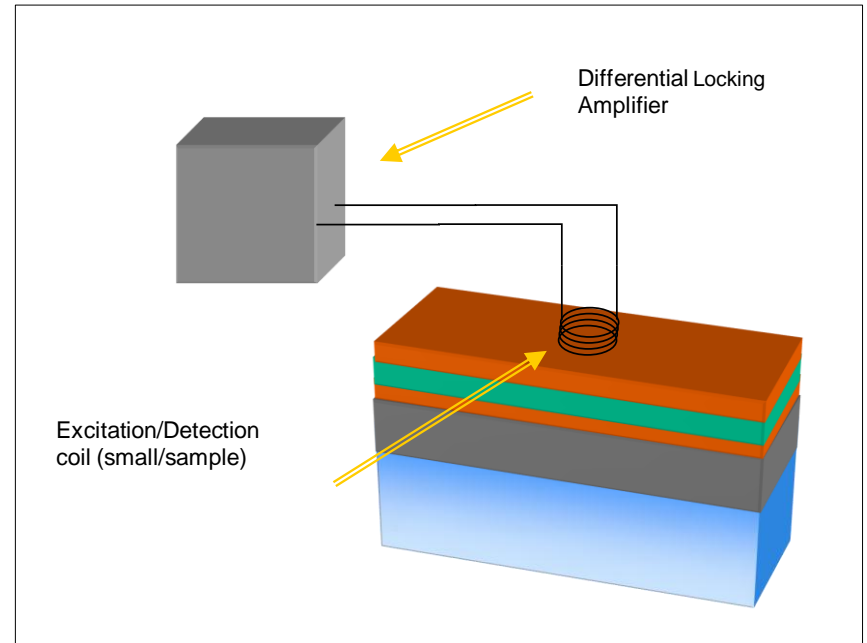
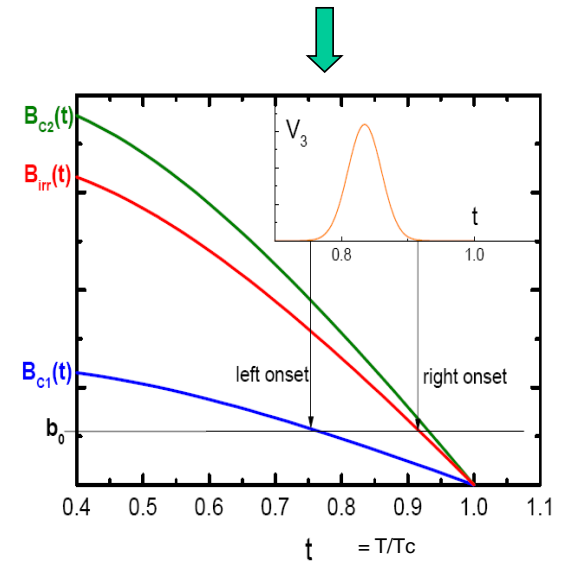
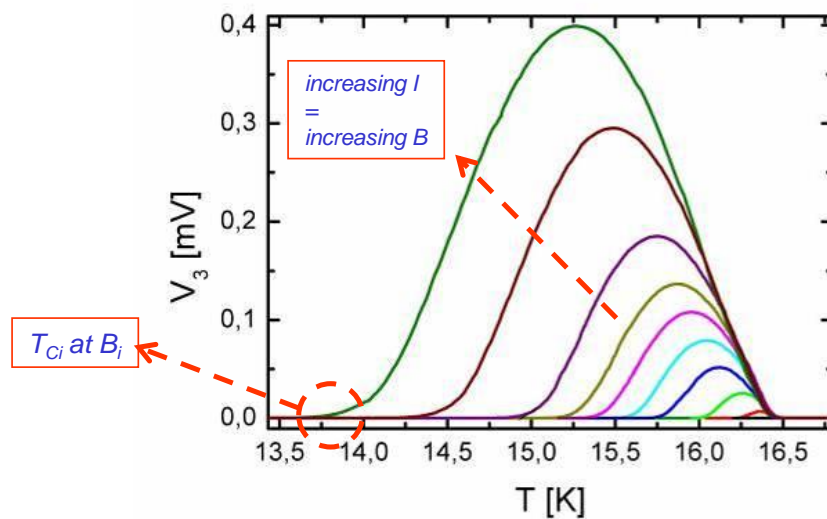


FIG. 4. Radial component of the total induction field (open squares) for a multiturn coil. In the inset the result for the normal component (open circles) is shown. Both components have been calculated at the sample surface as a function of r/r_0 .



Local magnetometry (2)

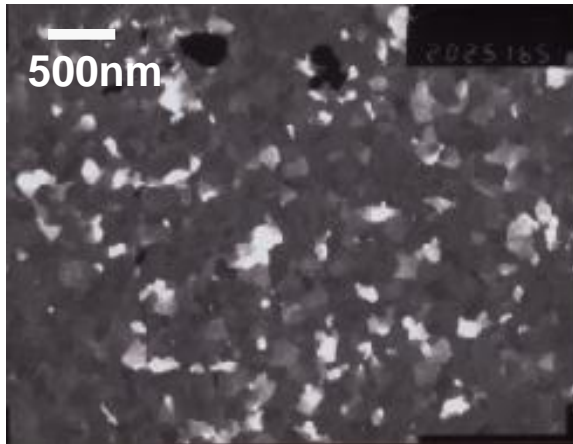
- 3rd harmonic measurement, coll. INFM Napoli
 - $b_0 \cos(\omega\tau)$ applied in the coil
 - temperature ramp
 - third harmonic signal appears @ T^{b_0} , when b_0 reaches $B_{C1}(T^{b_0})$
 - series of $b_0 \Rightarrow$ series of transition temperature $\Rightarrow B_{C1}(T)$



Sample SL : third harmonic signal for various b_0

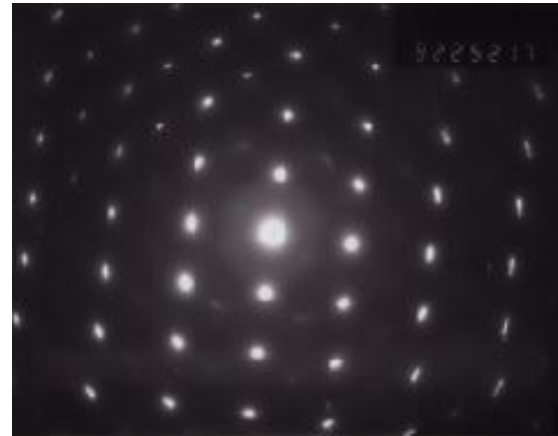
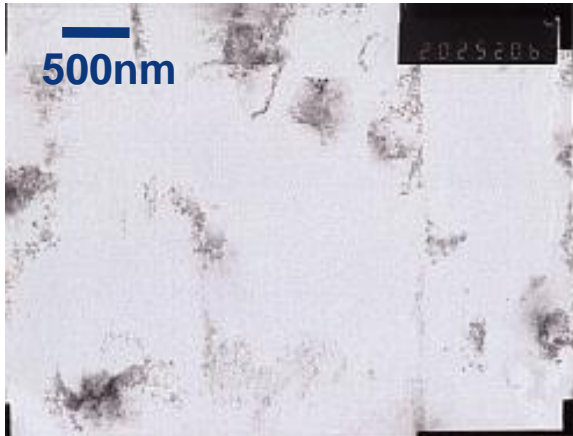
TEM micrographs in plan view

Standard



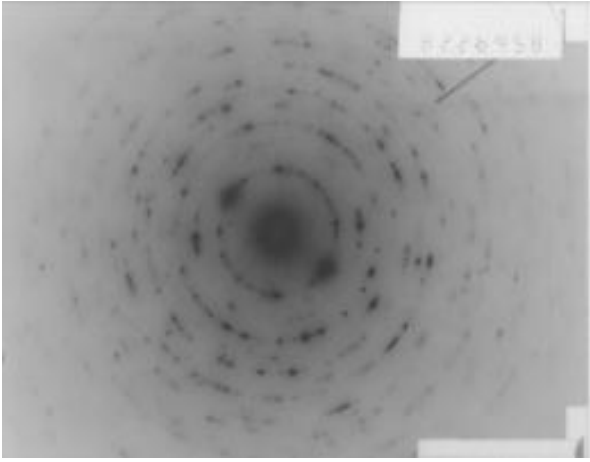
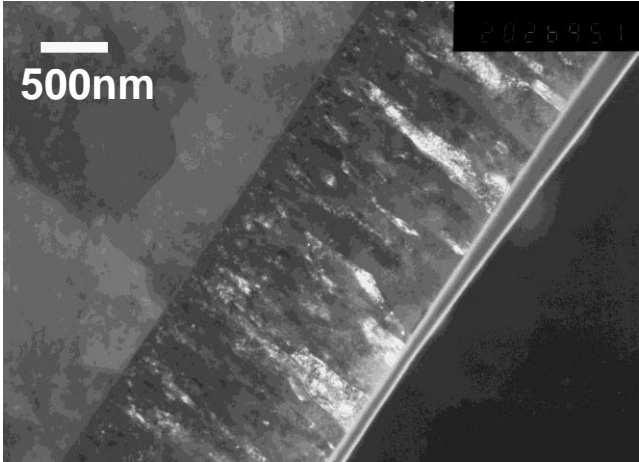
Grain size ~ 100 nm
Fibre texture
Diffraction pattern:
powder diagram

Oxide-free

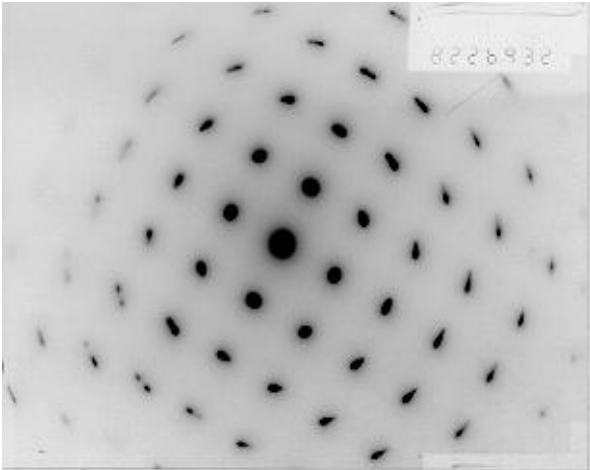
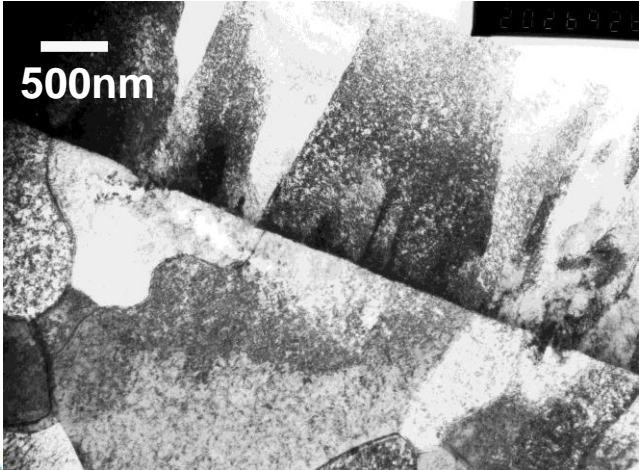


Grain size ~ 1-5 μ m
Heteroepitaxy
Diffraction pattern:
zone axis [110]

TEM micrographs in cross section



(110) fibre texture
⊥ substrate plane

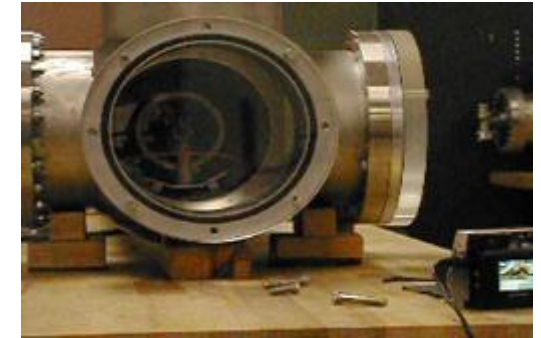
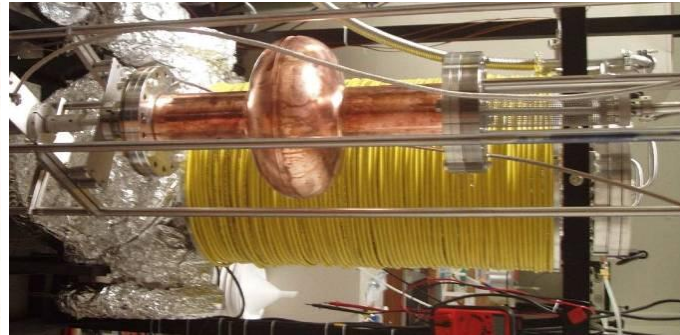


Heteroepitaxy
Nb (110) // Cu(010)
Nb (110) // Cu(111)
Nb (100) // Cu(110)

Standard

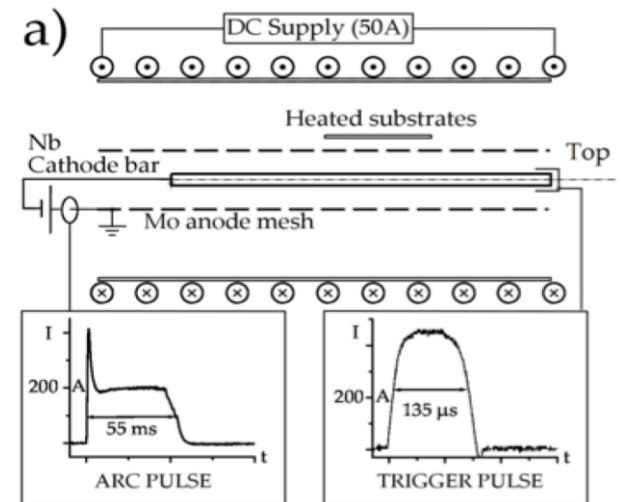
Oxide-free

Coaxial Energetic Deposition (CED)



Coaxial Energetic Deposition (CED™)

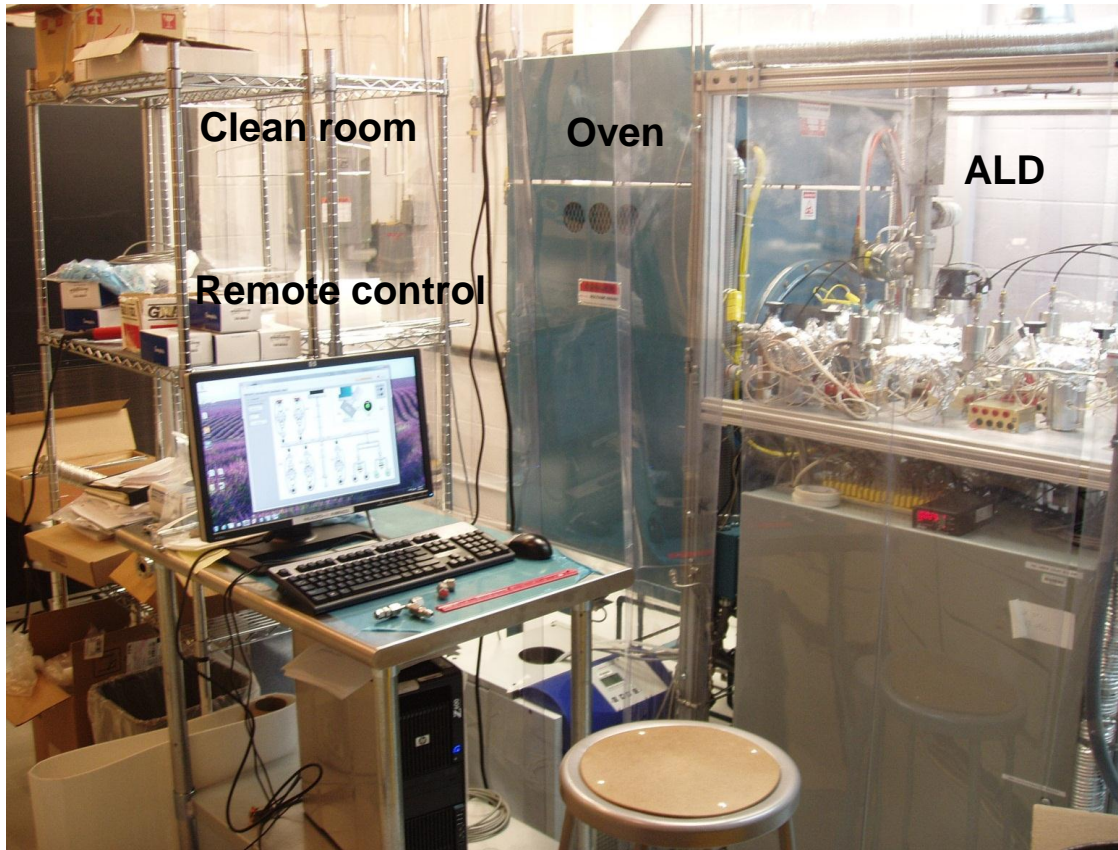
- CED coater uses “welding torch” technology
- Arc source is scalable to high throughputs for large scale cavity coatings
- Present version deposits ~1 monolayer/pulse ~1 ms



Atomic layer deposition – USA only



Atomic Layer Deposition Cavity System at ANL



- Clean room class 100
- UHV Oven: $650\text{C}-10^{-7}$ T
- Fully automatized ALD
- Long deposition overnight
- Growth of films:
12.5 K T_c (for now)
- Uniform on cavity scale

Thomas Proslie

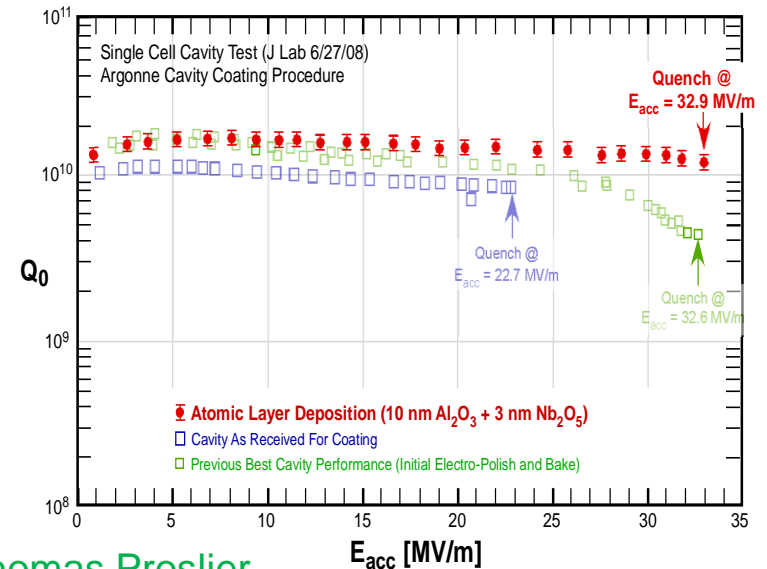
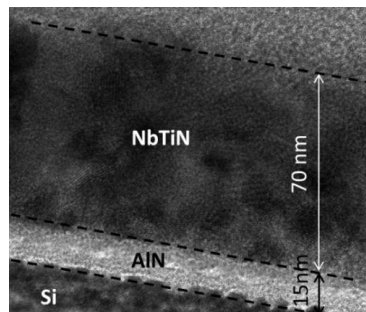
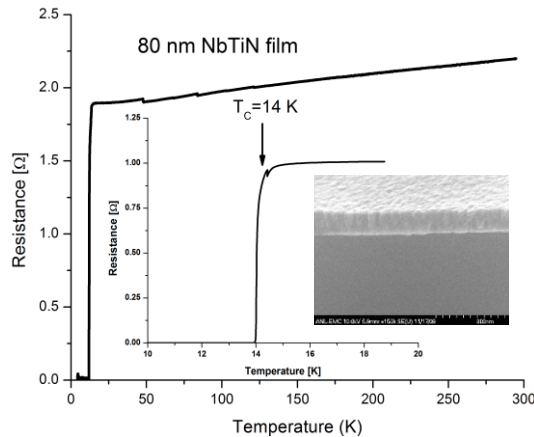
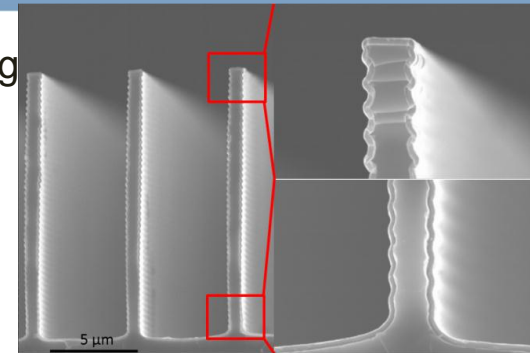


ALD



Atomic layer deposition of superconductors

- Self limiting surface reactions -> conformal coating
- Vapor phase precursors -> high aspect ratio



Thomas Proslier

E_{acc} [MV/m]

