Superconductive Materials

Part 13

Materials for SRF - Thin films

Outline

- Motivation for thin films in SRF cavities
- How to realize a thin film coating?
- State of the art in Nb thin films (accelerators using thin film technology)
- Characteristics of Nb thin films
- R&D on Nb thin films

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Why thin films for SRF?

1. Reduce material cost
2. Change the surface properties (bulk properties ≠ surface properties)
3. Use materials with poor mechanical properties (but excellent SRF properties)
4. Realize complex structures

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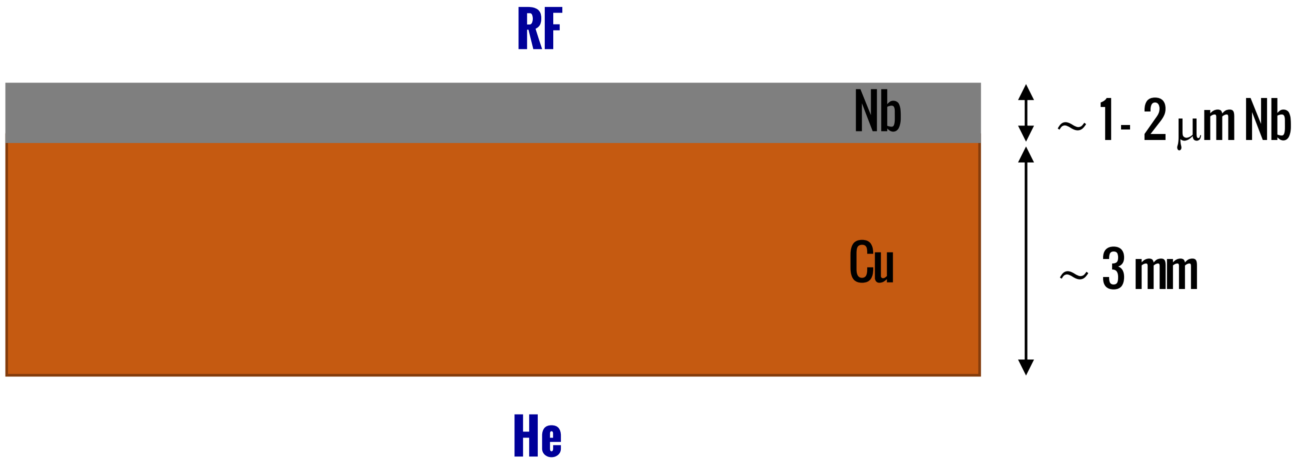
Reduce material cost

RF penetration in Nb is **limited by λ_L** (less than 100 nm)

Not necessary more than 1 micron of Nb at the surface



Cu is almost **100 times cheaper** than high pure Nb

It is possible to **increase the mechanical stability** of the cavities increasing wall thickness

Why thin films for SRF?

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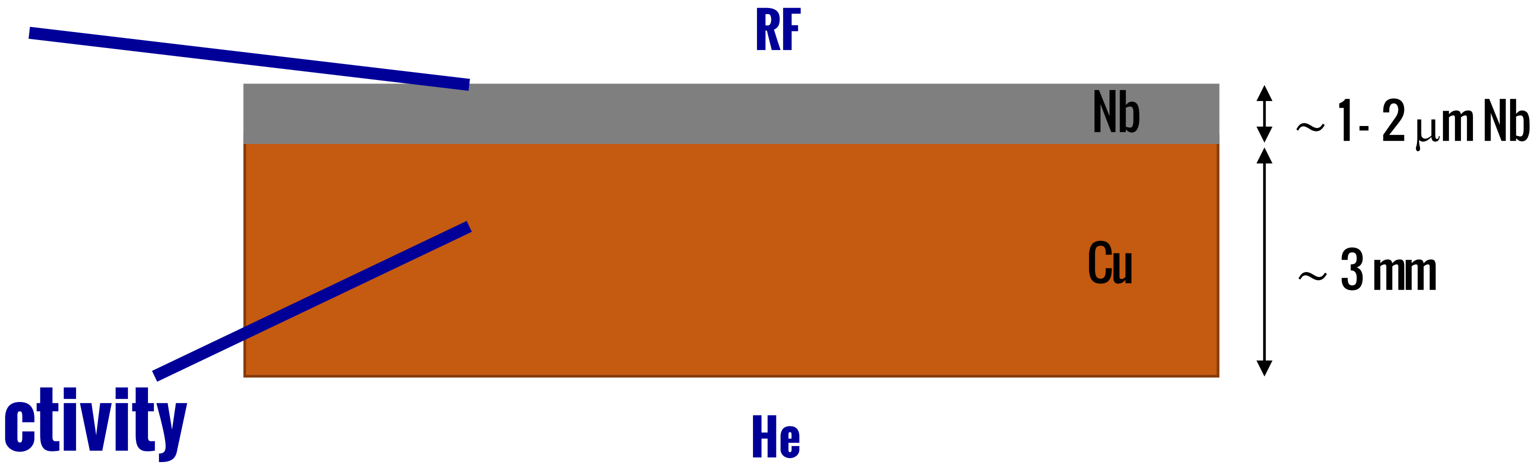
Motivation for thin films in SRF

Change the surface properties

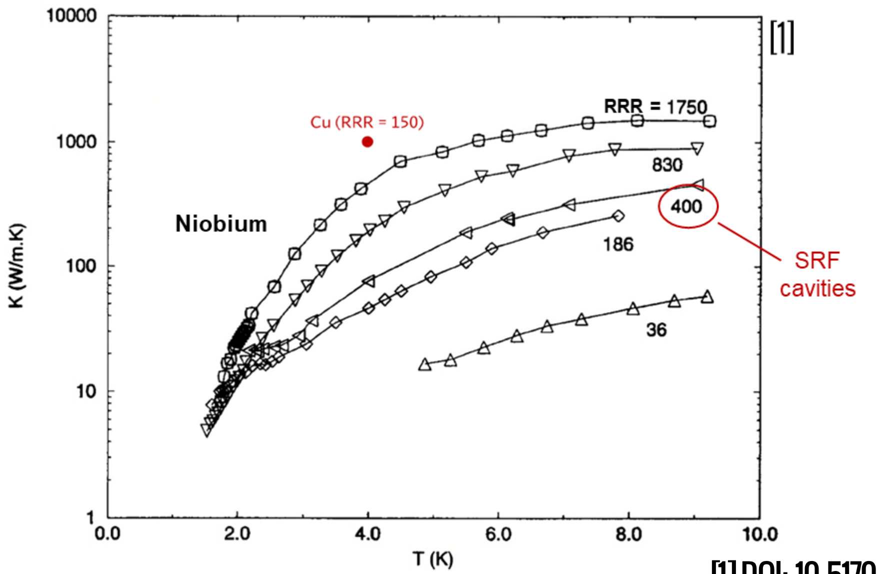


Surface: **Low Rs**

Bulk: **High thermal conductivity**

Cu presents high thermal conductivity

→ resistance to quench



[1]

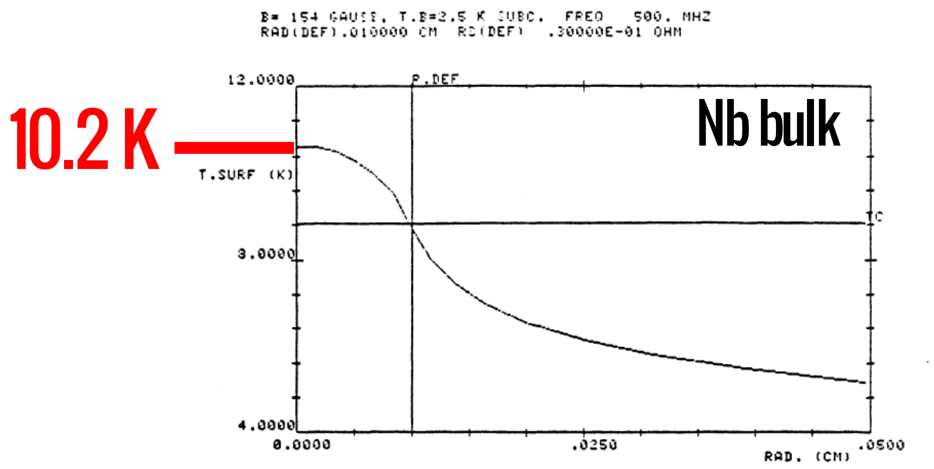
[1] DOI: 10.5170/CERN-1996-003.191

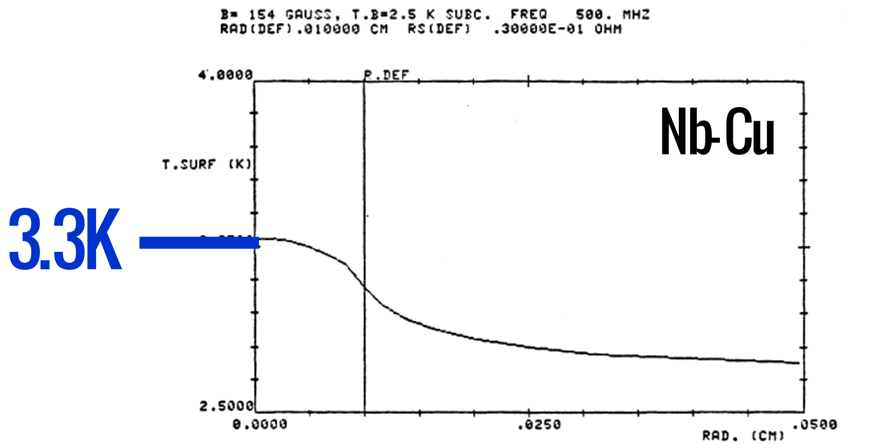
Temperature distribution in Nb-Cu

Temperature distribution simulation for an iron based defect imbedded in Nb or Cu

Copper prevent Quench

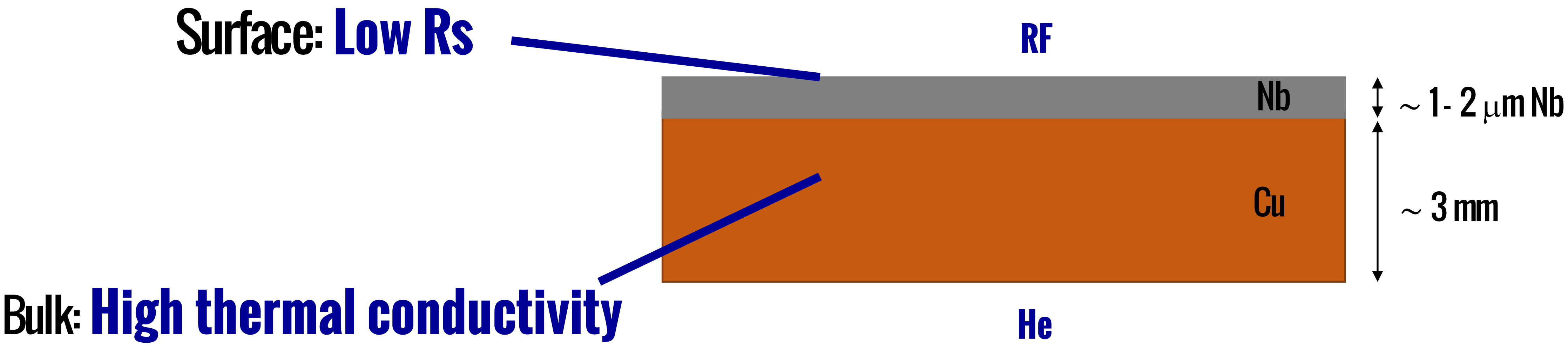
due to thermo-magnetic breakdown





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

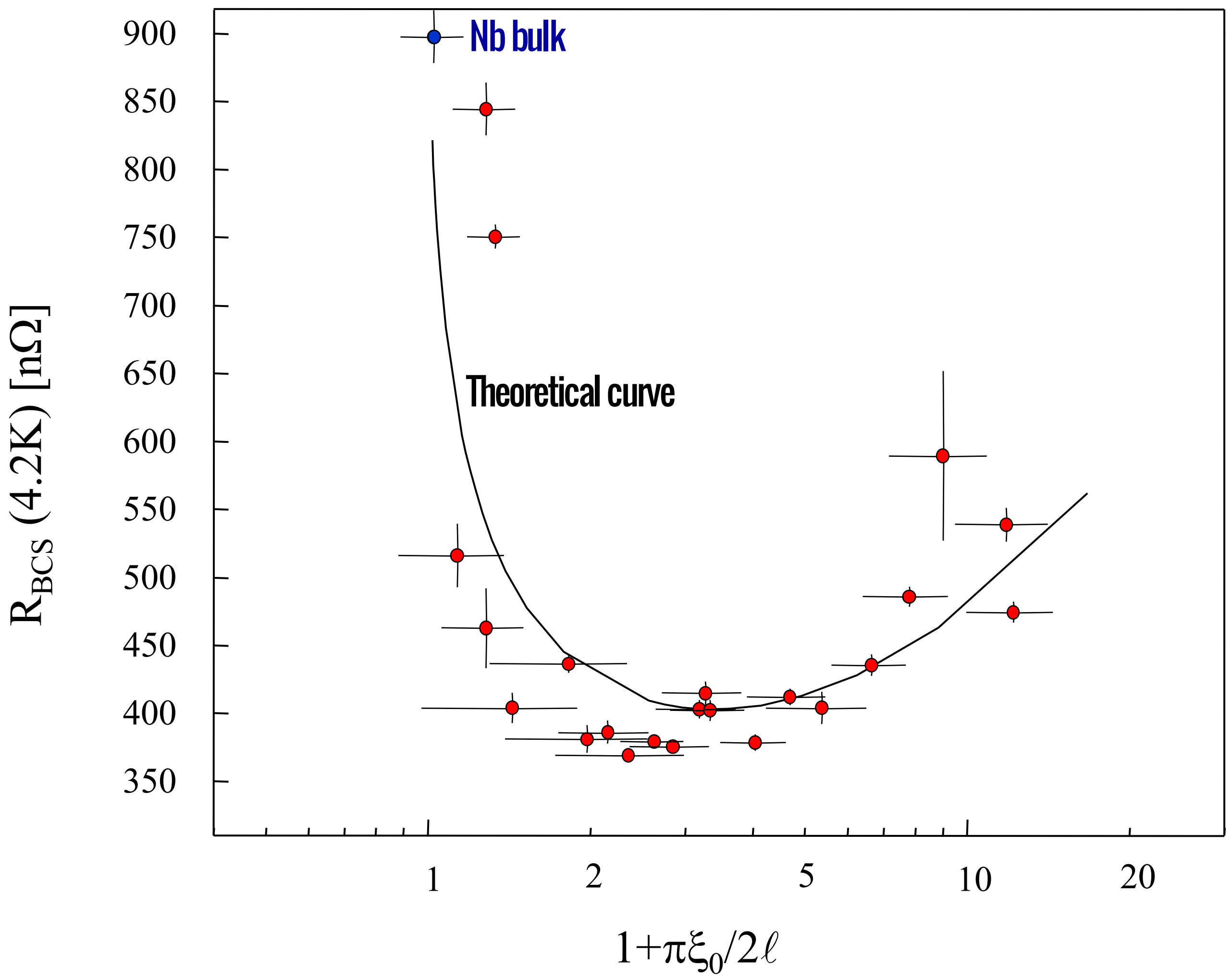
Change the surface properties



Cu presents high thermal conductivity → resistance to quench

Nb surface resistance could be modulated

BCS resistance depends on mean free path

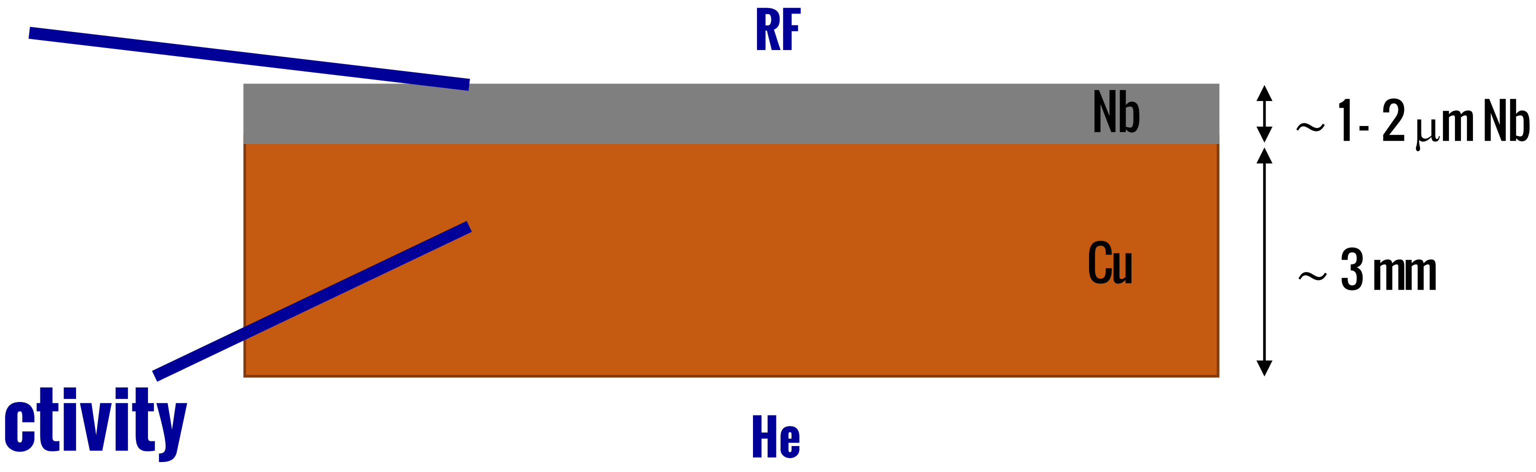


R_{BCS} @ 4.2 K
Nb bulk: ~900 nΩ
Nb films: ~400 nΩ

R_{BCS} @ 1.7 K
Nb bulk: ~2.5 nΩ
Nb films: ~1.5 nΩ

Benvenuti C et al 1999 Physica C 316 153

Change the surface properties



Surface: **Low Rs**

Bulk: **High thermal conductivity**

Cu presents high thermal conductivity → resistance to quench

Nb surface resistance could be modulated

Safer handling for the chemical surface treatments

Cu polishing VS Nb polishing

Nb Chemical Polishing

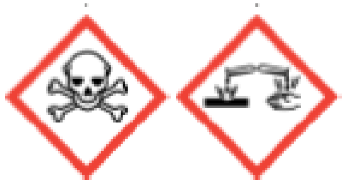
BCP composition (1:1:1 or 1:1:2)

- **HF - Hydrofluoric acid (49%)**
- HNO_3 - Nitric acid (70%)
- H_3PO_4 - Phosphoric acid (85%)

Nb Electrochemical Polishing

EP bath composition (1:9)

- **HF - Hydrofluoric acid (49%)**
- H_2SO_4 - Sulphoric acid (96%)



Cu Chemical Polishing

SUBU5 composition

- sulfamic acid (5g/l)
- hydrogen peroxide 32% (50ml/l)
- n-butanol 99% (50ml/l)
- ammonium citrate (1g/l)

Cu Electrochemical Polishing

EP bath composition (3:2)

- H_3PO_4 - Phosphoric acid (85%)
- N-buthanol (99%)

No HF for Cu polishing

No chemical post treatment on Nb film necessary

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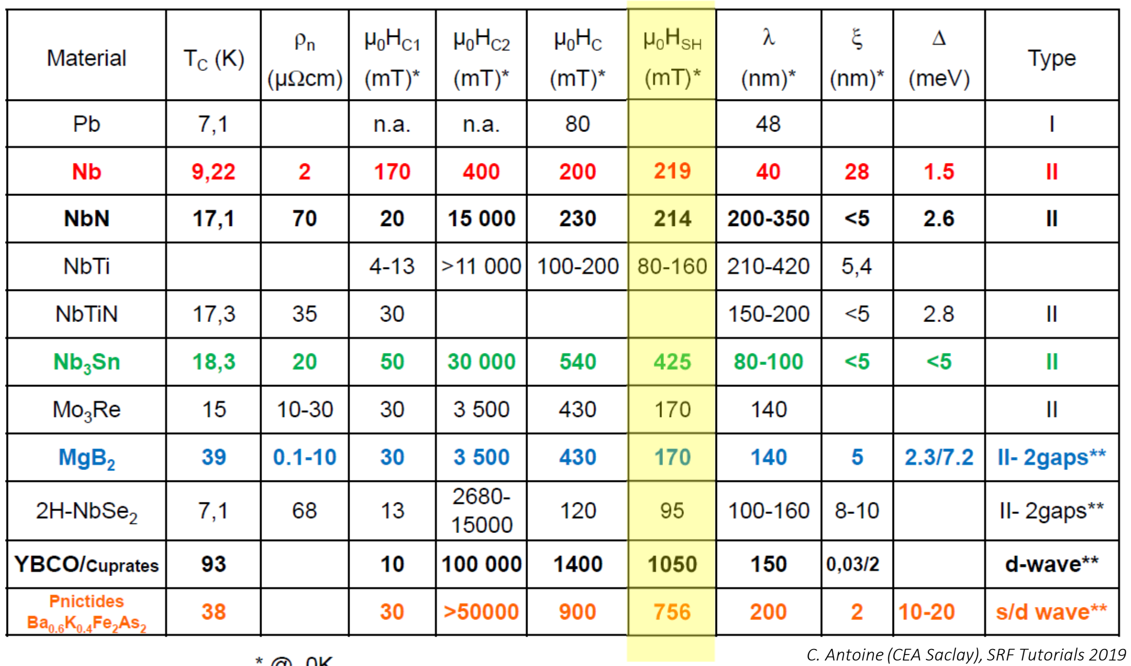
Use materials with poor mechanical properties (but excellent SRF properties)

A15 materials (Nb_3Sn, V_3Si, Nb_3Ge, etc.) present high T_c and High H_{c1} but are very brittle: can not be used as bulk materials for SRF cavities

(WAIT THE NEXT LECTURE)



Material	T_C (K)	ρ_n ($\mu\Omega$cm)	μ_0H_{C1} (mT)*	μ_0H_{C2} (mT)*	μ_0H_C (mT)*	μ_0H_{SH} (mT)*	λ (nm)*	ξ (nm)*	Δ (meV)	Type
Pb	7,1		n.a.	n.a.	80		48			I
Nb	**9,22**	**2**	**170**	**400**	**200**	**219**	**40**	**28**	**1.5**	**II**
NbN	**17,1**	**70**	**20**	**15 000**	**230**	**214**	**200-350**	**<5**	**2.6**	**II**
NbTi			4-13	>11 000	100-200	80-160	210-420	5,4		
NbTiN	17,3	35	30				150-200	<5	2.8	II
Nb_3Sn	**18,3**	**20**	**50**	**30 000**	**540**	**425**	**80-100**	**<5**	**<5**	**II**
Mo_3Re	15	10-30	30	3 500	430	170	140			II
MgB_2	**39**	**0.1-10**	**30**	**3 500**	**430**	**170**	**140**	**5**	**2.3/7.2**	**II- 2gaps****
2H-$NbSe_2$	7,1	68	13	2680-15000	120	95	100-160	8-10		II- 2gaps**
YBCO/cuprates	**93**		**10**	**100 000**	**1400**	**1050**	**150**	**0,03/2**		**d-wave****
Pnictides $Ba_{0.6}K_{0.4}Fe_2As_2$	**38**		**30**	**>50000**	**900**	**756**	**200**	**2**	**10-20**	**s/d wave****

* @ 0K
** 2D => orientation problems ?

C. Antoine (CEA Saclay), SRF Tutorials 2019

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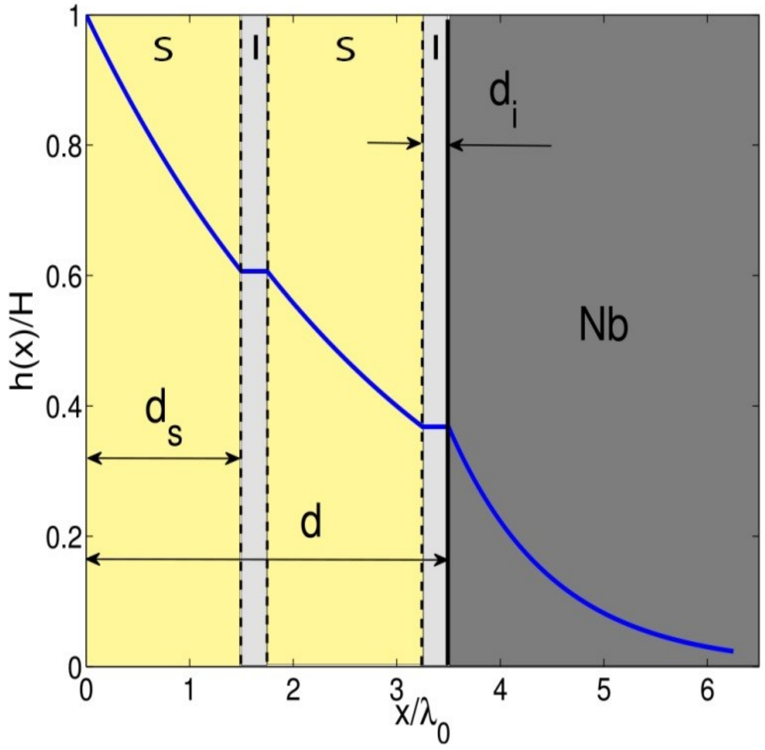
4. **Realize complex structures**

Realize complex structures

SIS Multilayer

(WAIT THE NEXT LECTURE)



Alex Gurevich, Appl. Phys. Lett. 88, 012511 (2006)

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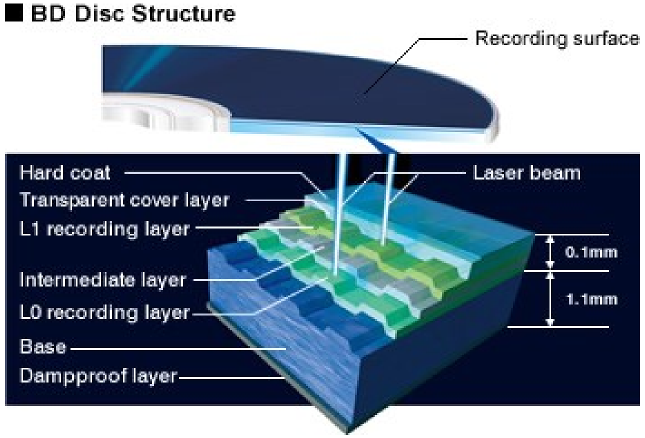
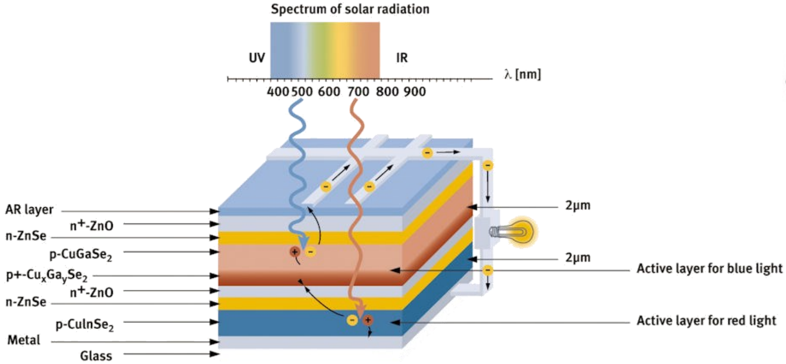
Thin film applications

Some of the most utilized applications of thin film deposition processes include:

- Single and multilayer films and coatings
- Nanolayered materials
- Optical films for transmission and reflection
- Decorative films
- Decorative and wear-resistant (decorative/functional) coatings
- Permeation barriers for moisture and gases
- Corrosion-resistant films
- Electrically insulating layers for microelectronics
- Coating of engine turbine blades
- Coating of high strength steels to avoid hydrogen embrittlement
- Diffusion barrier layers for semiconductor metallization
- Magnetic films for recording media
- Transparent electrical conductors and antistatic coatings
- Wear and erosion-resistant (hard) coatings (tool coatings)
- Dry film lubricants
- Composite and phase-dispersed films and coatings
- Nanocomposite materials
- Thin-walled freestanding structures and foils





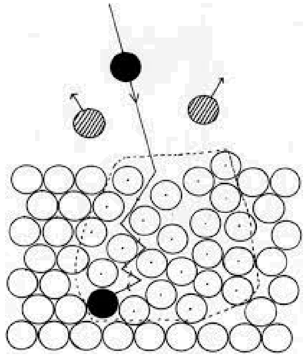
Thin film deposition techniques

Chemical Deposition

- **Plating/Electroplating**
- **Dip/Spin coating**
- **Chemical Vapour Deposition**
  - Thermal CVD
  - Plasma Enhanced CVD
  - Atomic Layer Deposition (ALD)

Phisical Deposition

- **Physical Vapour Deposition**
  - Evaporation
  - Laser Ablation
  - Plasma Spray
  - Sputtering
  - Cathodic Arc



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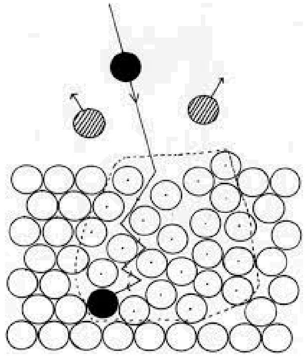


PVD deposition techniques

How to realize a thin film coating

Physical vapor deposition processes (often just called thin film processes) are atomistic deposition processes in which material is vaporized from a solid or liquid source in the form of atoms or molecules and transported in the form of a vapor through a vacuum or low pressure gaseous (or plasma) environment to the substrate, where it condenses

Donald M. Mattox,
Handbook of Physical Vapor Deposition PVD) Processing

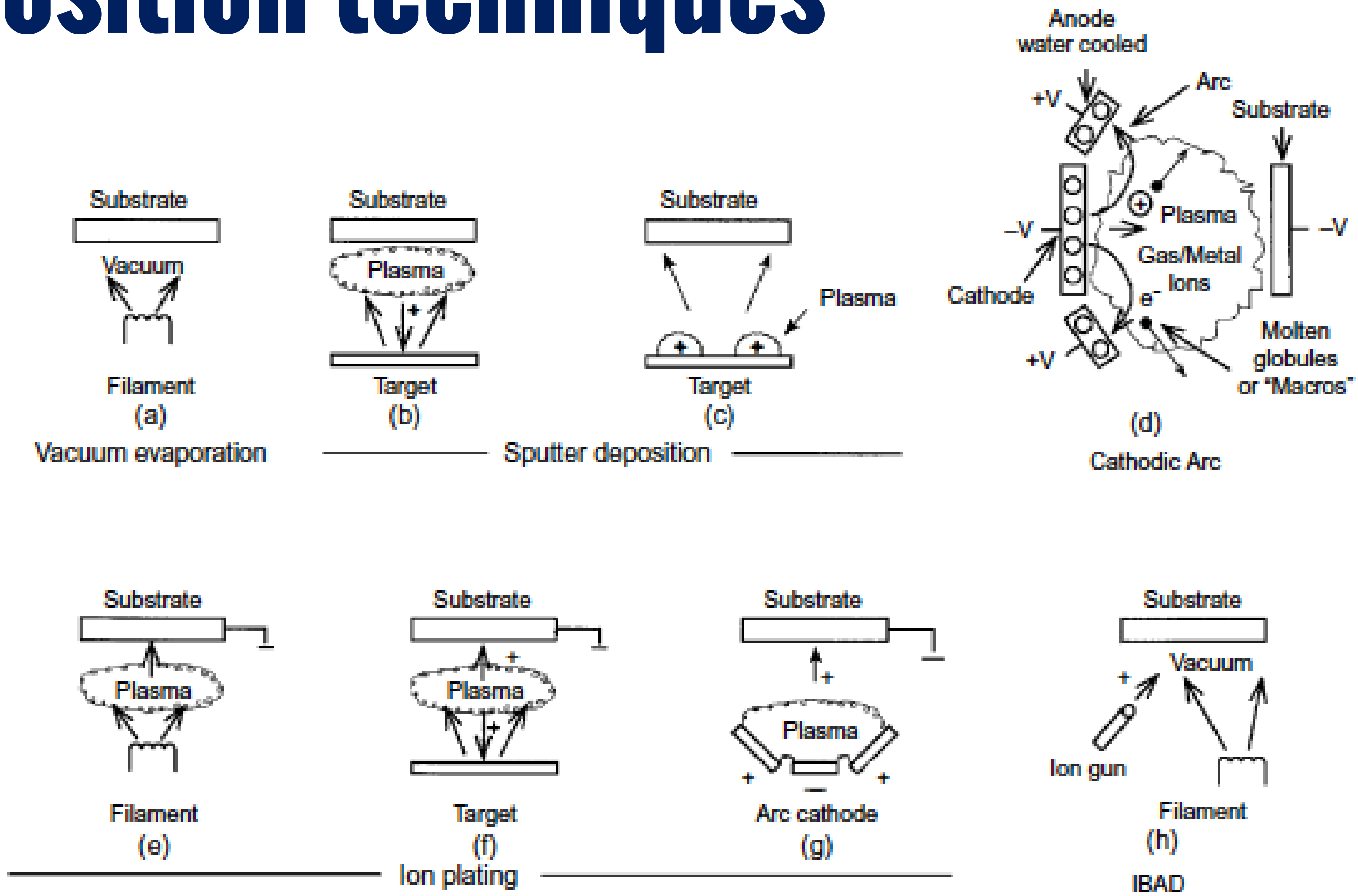
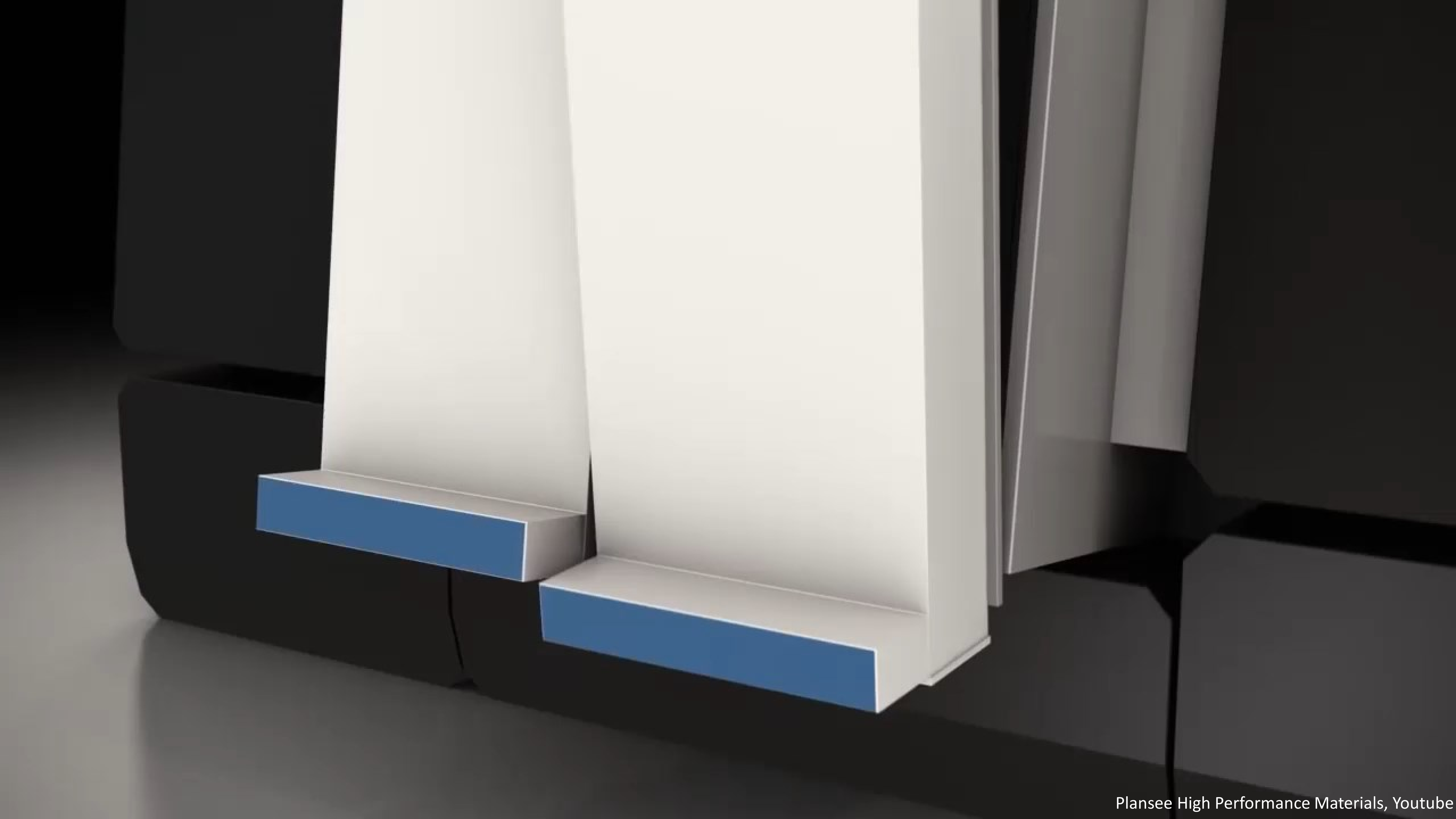


Figure 1.1: PVD Processing Techniques: (a) Vacuum Evaporation, (b) and (c) Sputter Deposition in a Plasma Environment, (d) Sputter Deposition in a Vacuum, (e) Ion Plating in a Plasma Environment with a Thermal Evaporation Source, (f) Ion Plating with a Sputtering Source, (g) Ion Plating with an Arc Vaporization Source, and (h) Ion Beam-Assisted Deposition (IBAD) with a Thermal Evaporation Source and Ion Bombardment from an Ion Gun



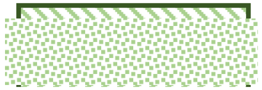
Plansee High Performance Materials, Youtube

Interface

The depositing film material may diffuse and react with the substrate to form a "interfacial region"

Nb-Cu case

Abrupt



Graded

Weak chemical reaction between atoms and substrate

Low deposition temperature

Surface contamination

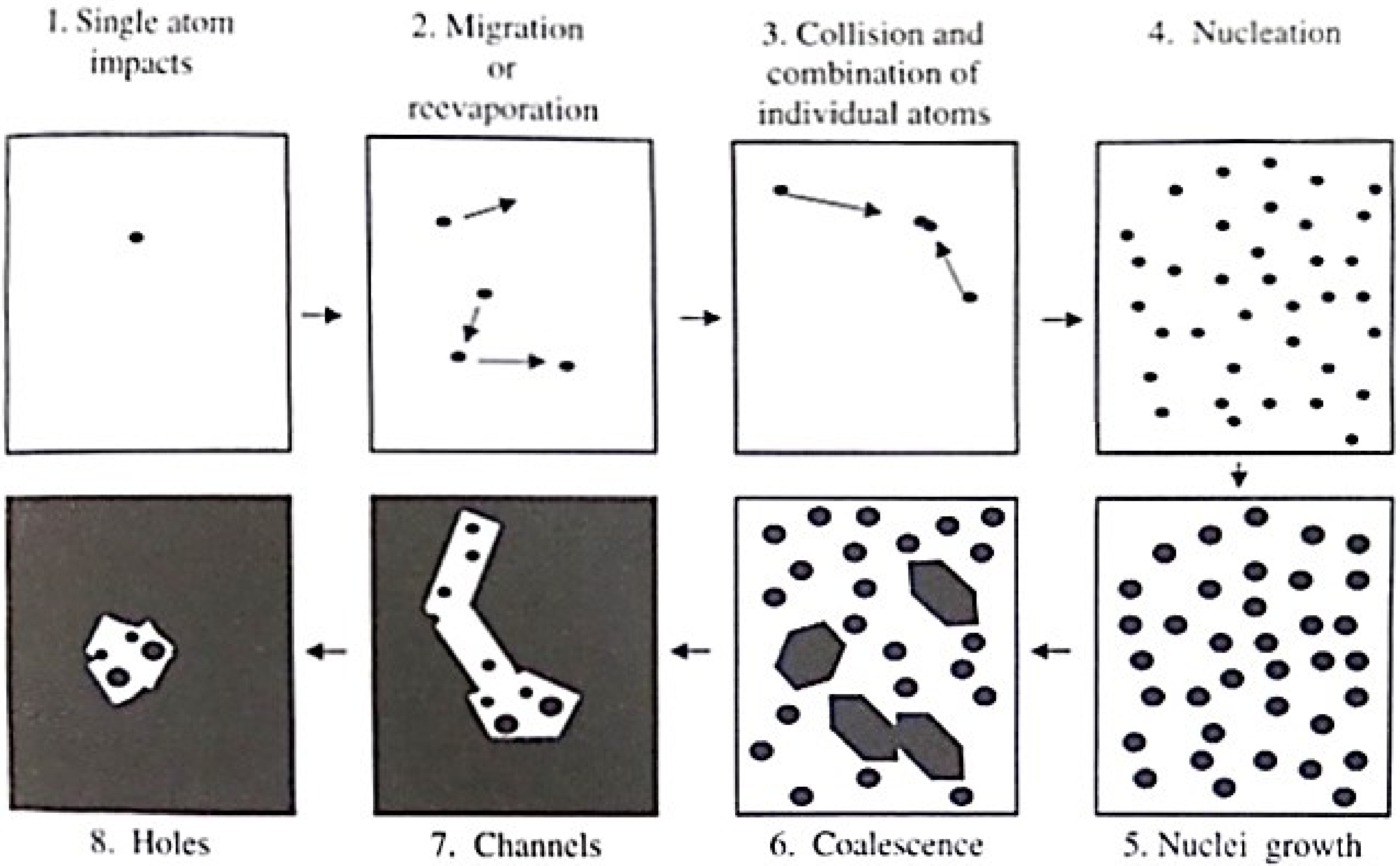
Low nucleation density

By diffusion (solubility, temperature, time, contaminations)

Chemical reaction (oxygen-active metals on oxide substrates)

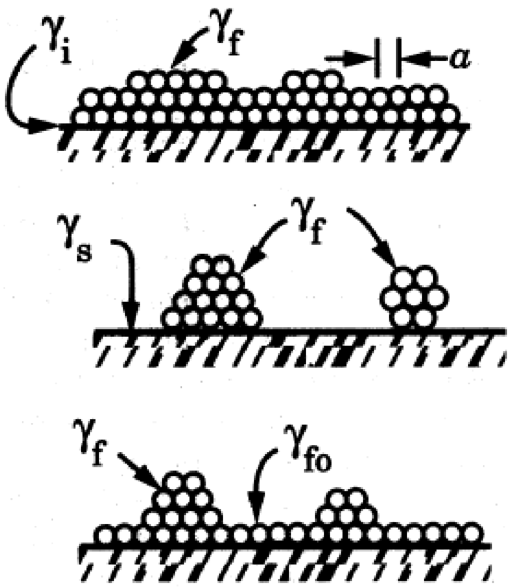
By co-deposition or implantation of energetic ions of the material

Nucleation stages



Phases of film growth: Nuclei growth

Nuclei grow by collecting adatoms which either impinge on the nuclei or migrate over the surface



binding energy atom-atom **<** binding energy atom-surface

Layer by layer growth *(Frank-van der Merwe)*

binding energy atom-atom **>** binding energy atom-surface

Island growth *(Volmer-Weber)*

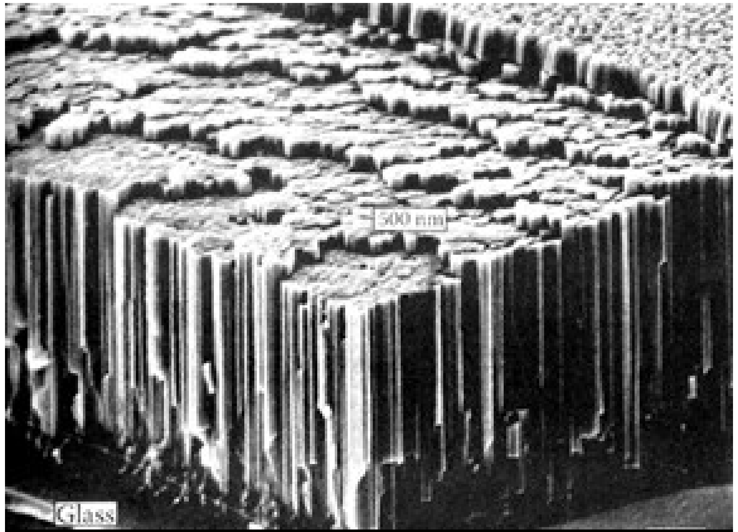
binding energy atom-atom **=** binding energy atom-surface

Layer by layer + island growth *(Stranski-Krastanov)*

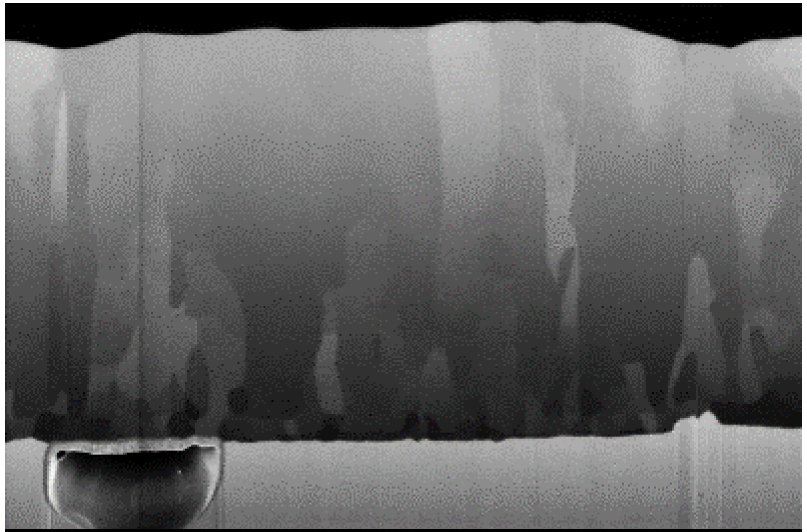
Phases of film growth: Film growth

Is the evolution of the nucleation, where arriving atoms are deposited on the previously deposited material

Usually exhibits a columnar morphology

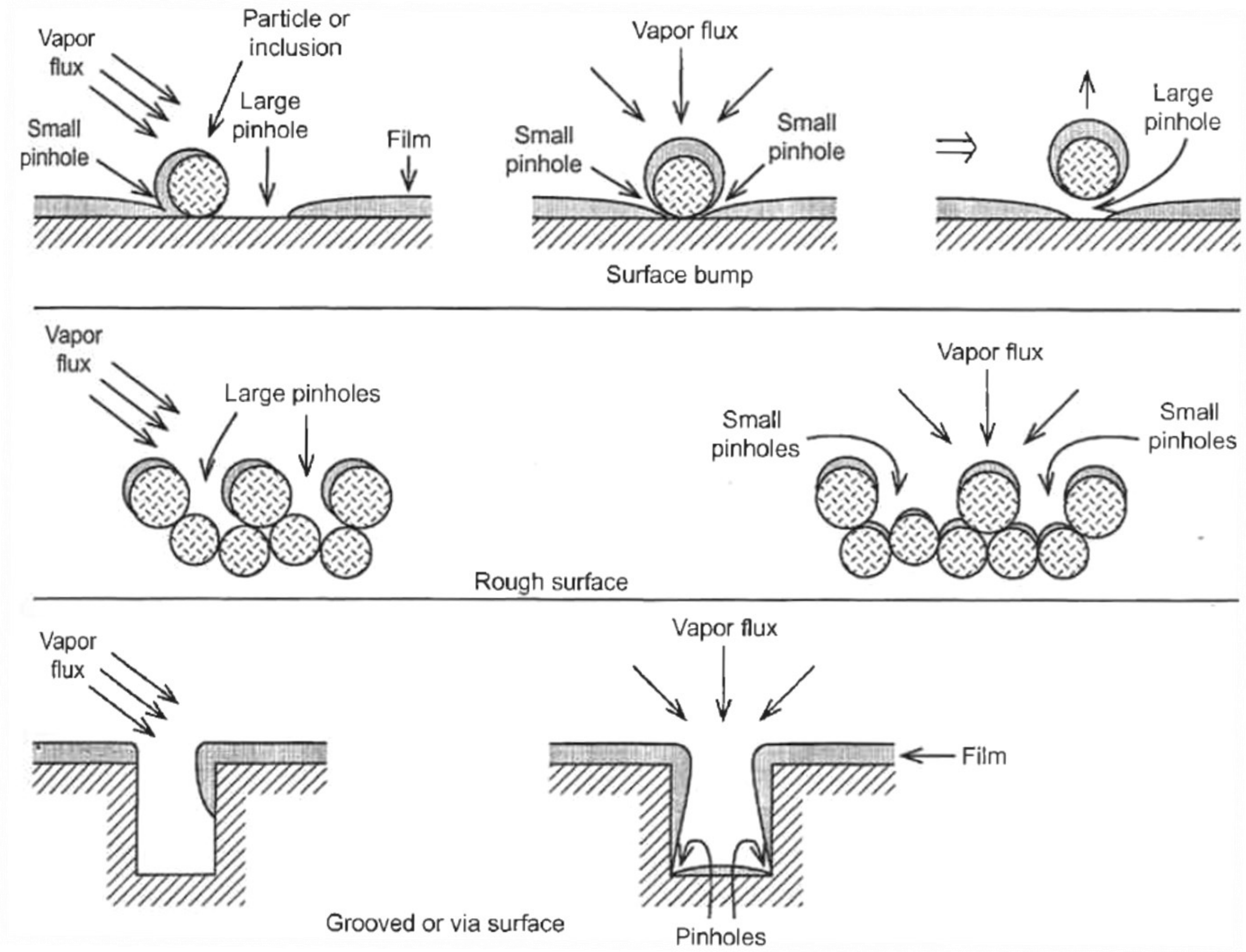


TiN on glass from Macleod's book

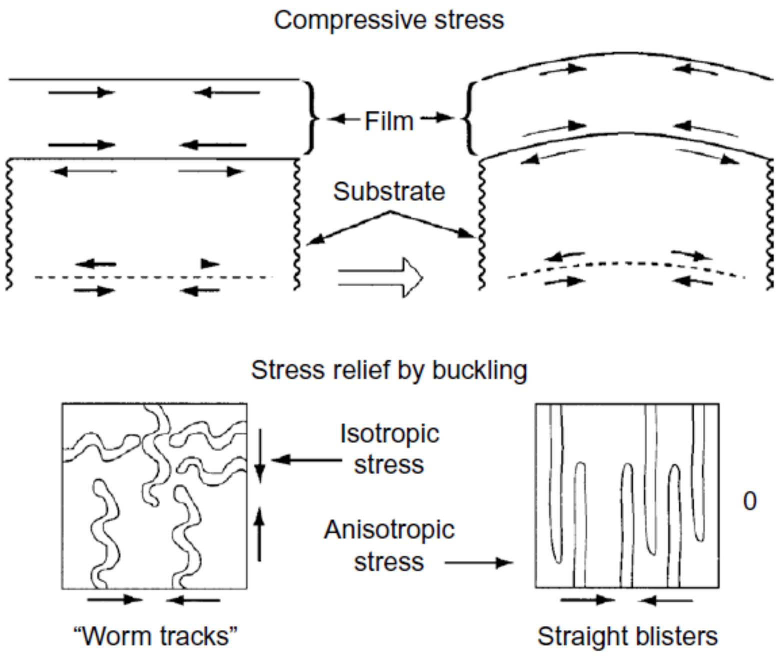


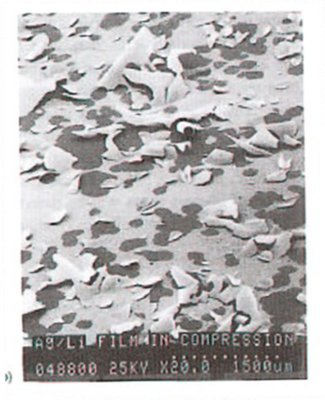
Nb on Cu (A. Sublet, CERN)

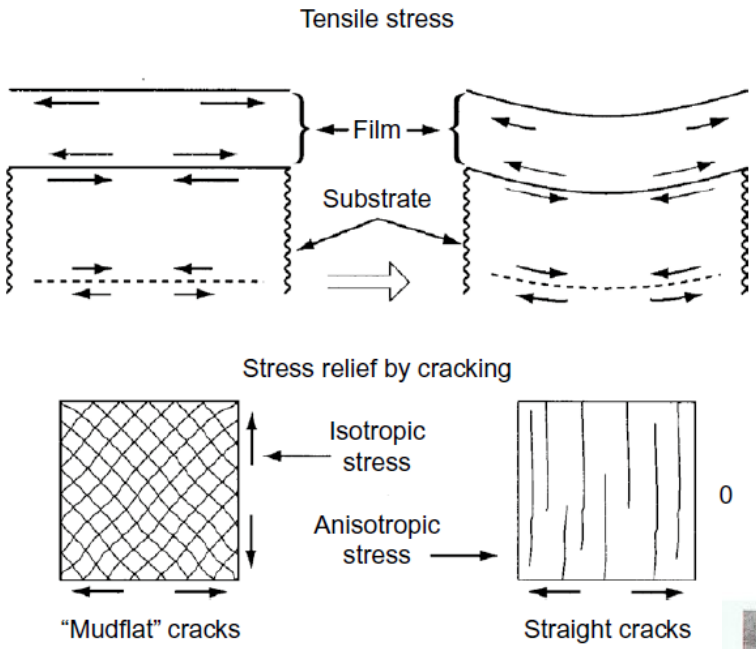
On growth and adhesion



Stress on thin films

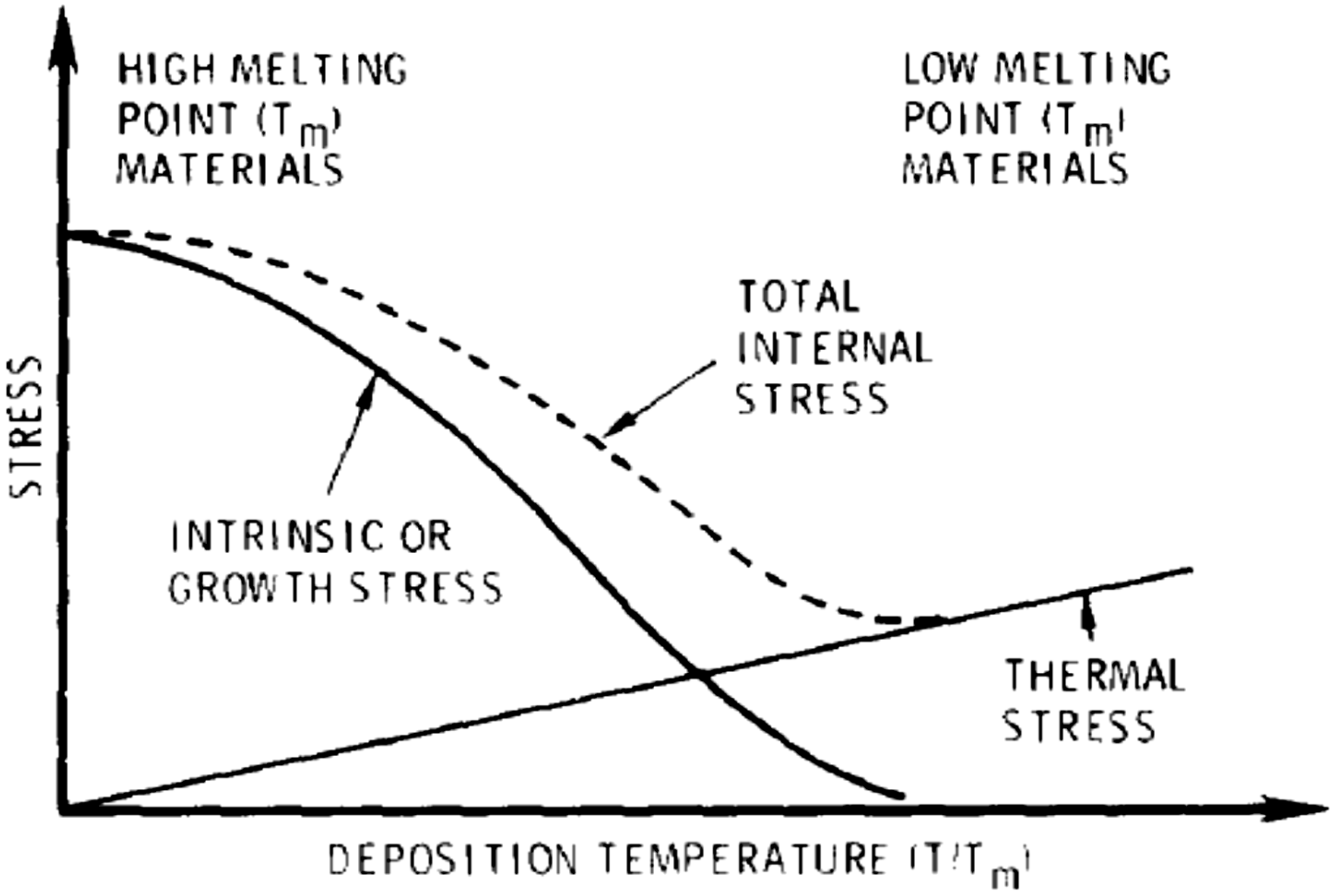






Donald M. Mattox, Handbook of Physical Vapor Deposition PVD) Processing

How to reduce film stress? Temperature effect



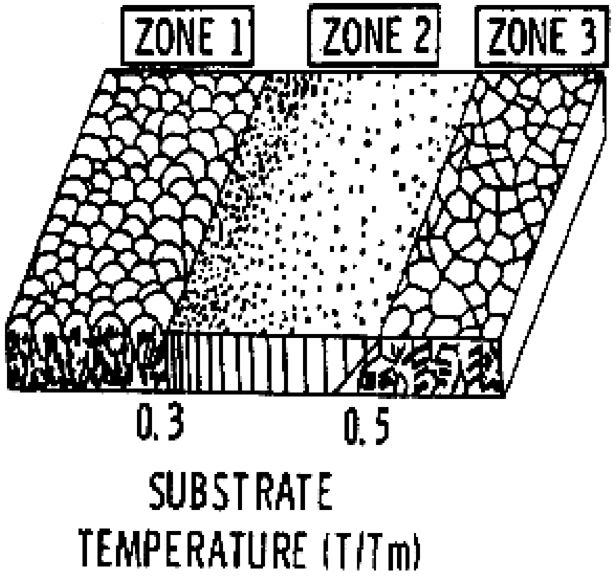
J. A. Thornton and D. W. Hoffman, “Stress-related effects in thin films,” *Thin Solid Films*, 1989

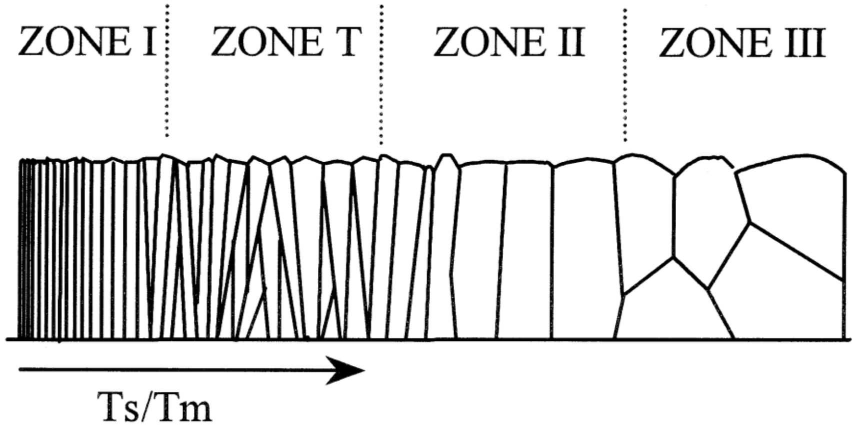
Temperature effect and Structure Zone Diagram (SZD)

- Based on the compilation of the experimental results, is a guideline for "predicting" the structure of deposited thin films

- 1^{st} proposed in 1969 by Movchan & Demchishin for films deposited by thermal evaporation.

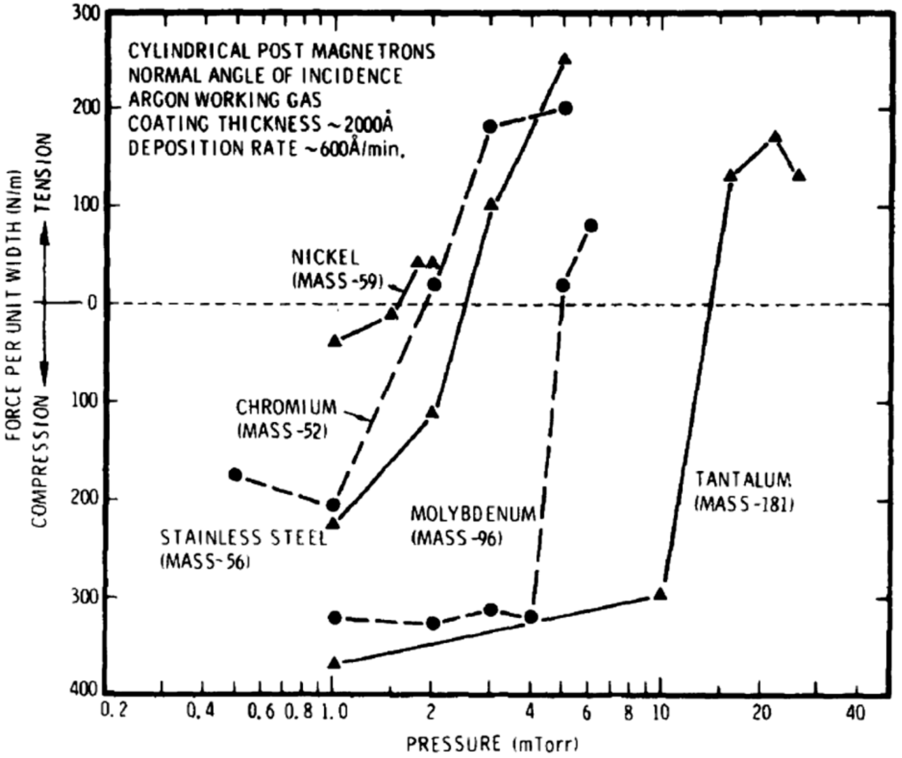
$$T_h = \frac{T}{T_m}$$ Homologous temperature





How to reduce film stress? Pressure effect

ZERO STRESS PRESSURE



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How to reduce film stress? Pressure effect

How to realize a thin film coating

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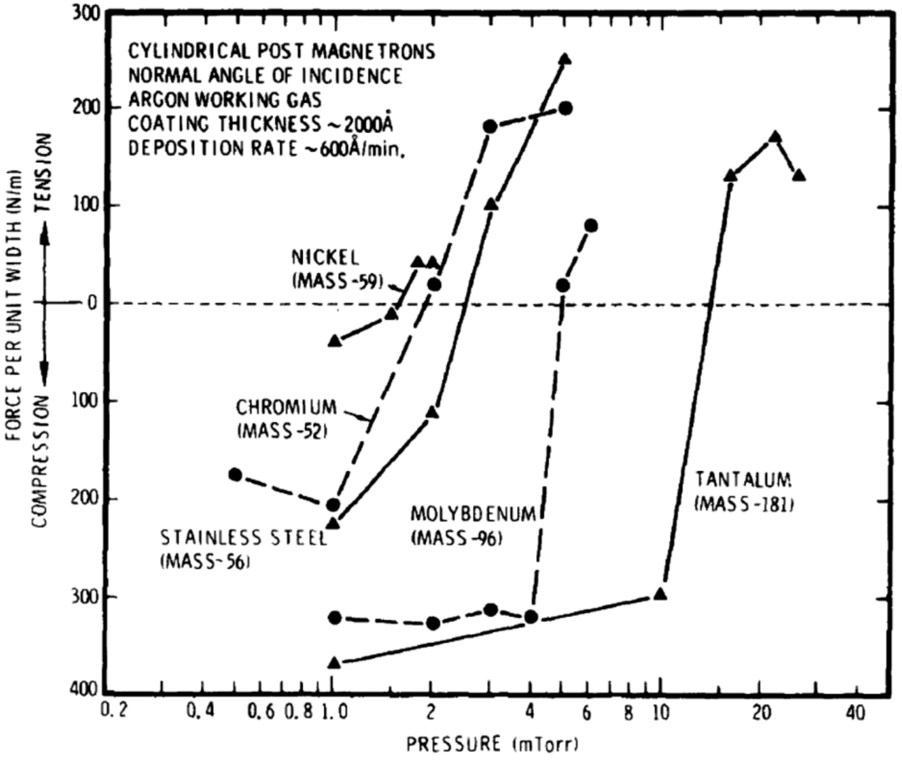

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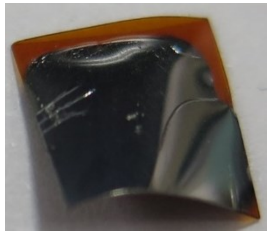
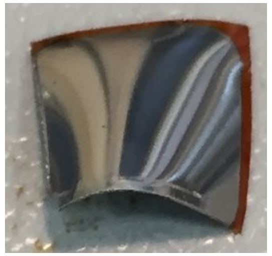
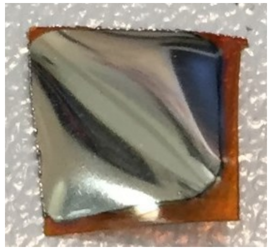


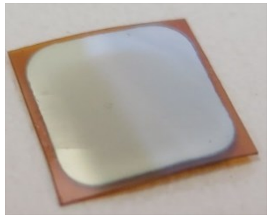
$7 \cdot 10^{-3}$ mbar



$9 \cdot 10^{-3}$ mbar



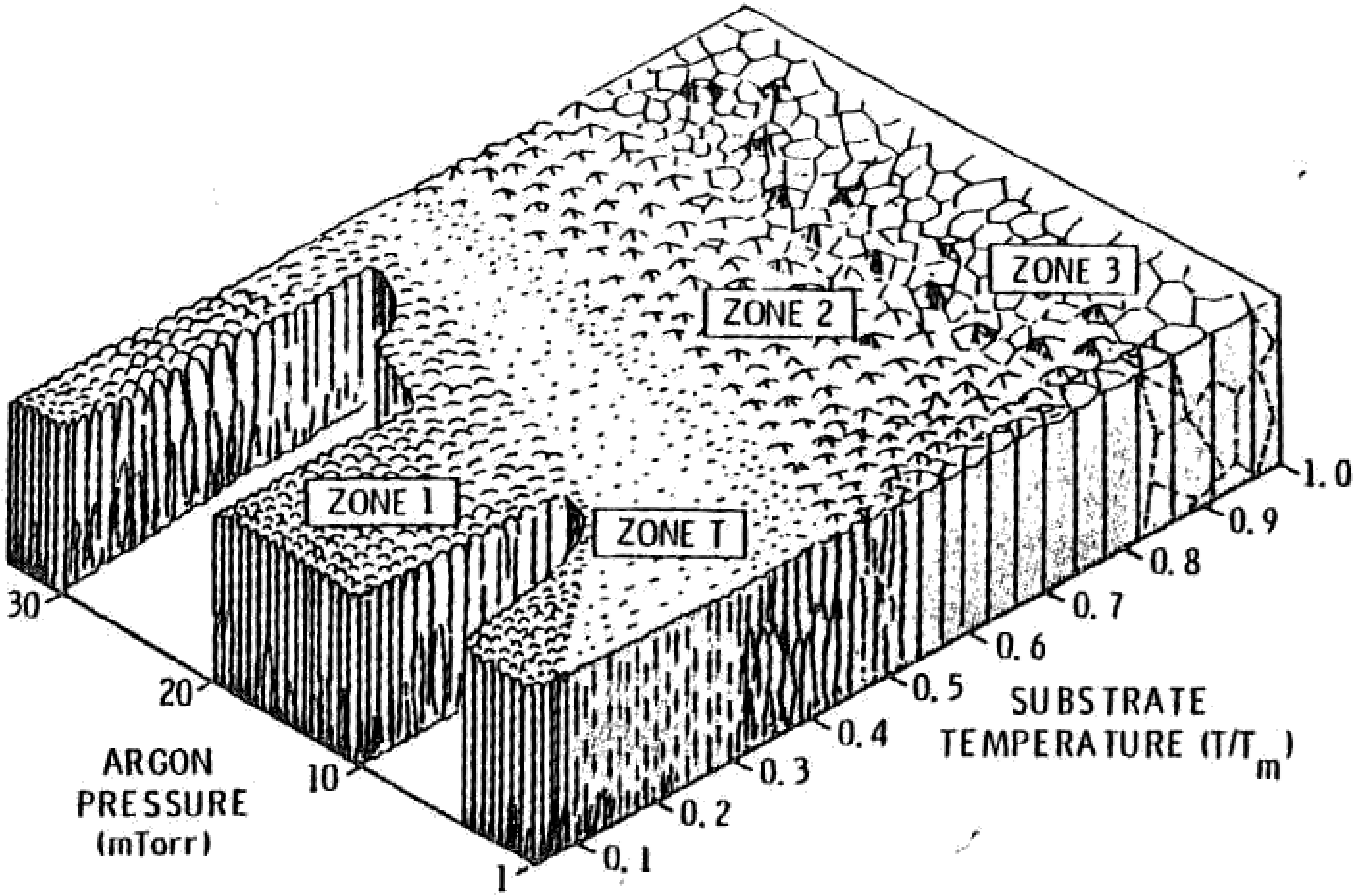
$2 \cdot 10^{-2}$ mbar



$5 \cdot 10^{-2}$ mbar

Thorton Structure Zone Diagram

- ZONE 1: characterized by a fine-grained structure of textured and fibrous grains, pointing in the direction of the arriving vapor flux. The morphology is caused by the low mobility of the adatoms that produce a continued nucleation of grain.
- ZONE T: a dense fibrous structure with a smooth, highly reflective surface. Diffusion is “remarkable” but grain boundary diffusion is strongly limited. Ionic bombardment of the growing film can move the morphology from zone 1 to zone T.
- ZONE 2: surface diffusion sets in, leading to uniform columnar grains.
- ZONE 3: dense films with large grains, drive by bulk diffusion and recrystallization.



J.A. Thornton and D.W. Hoffman, Thin Solid Films, vol. 171, no. 1, pp. 5–31, 1989

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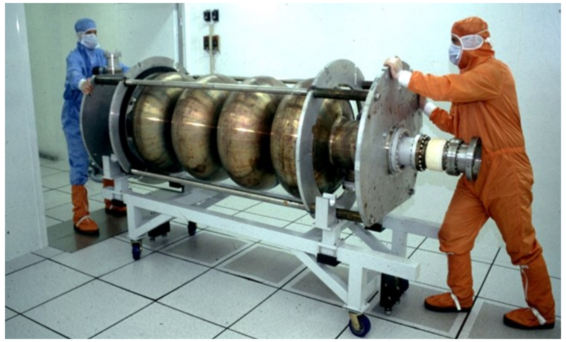
Nb film is a well known technology

- 288 Nb/Cu cavities installed in LEP @ CERN
- 56 Nb/Cu cavities installed in ALPI @ LNL INFN
- 16 Nb/Cu cavities installed in LHC @ CERN
- 20 Nb/Cu cavities installed in HIE-ISOLDE @ CERN

- R&D in many different labs: CERN, INFN, JLAB, STFC, Cornell, IMP, ...



90’s LEP2: 350MHz 4-cells

1998-2004 ALPI: 160 MHz QWR

2000’s LHC: 400MHz 1-cell

2010’s HIE-ISOLDE: 100MHz QWR

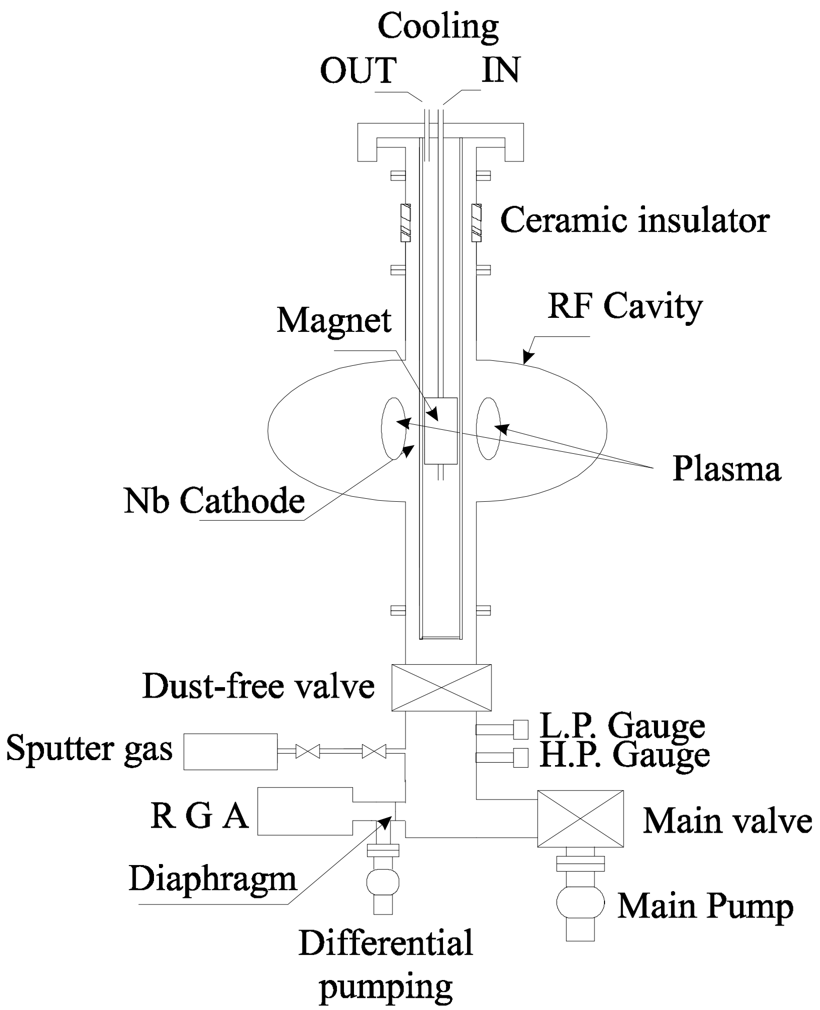
Elliptical cavities @CERN - LEP2, LHC and 1,5 GHz R&D

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**
- Thickness: 1.5 μm

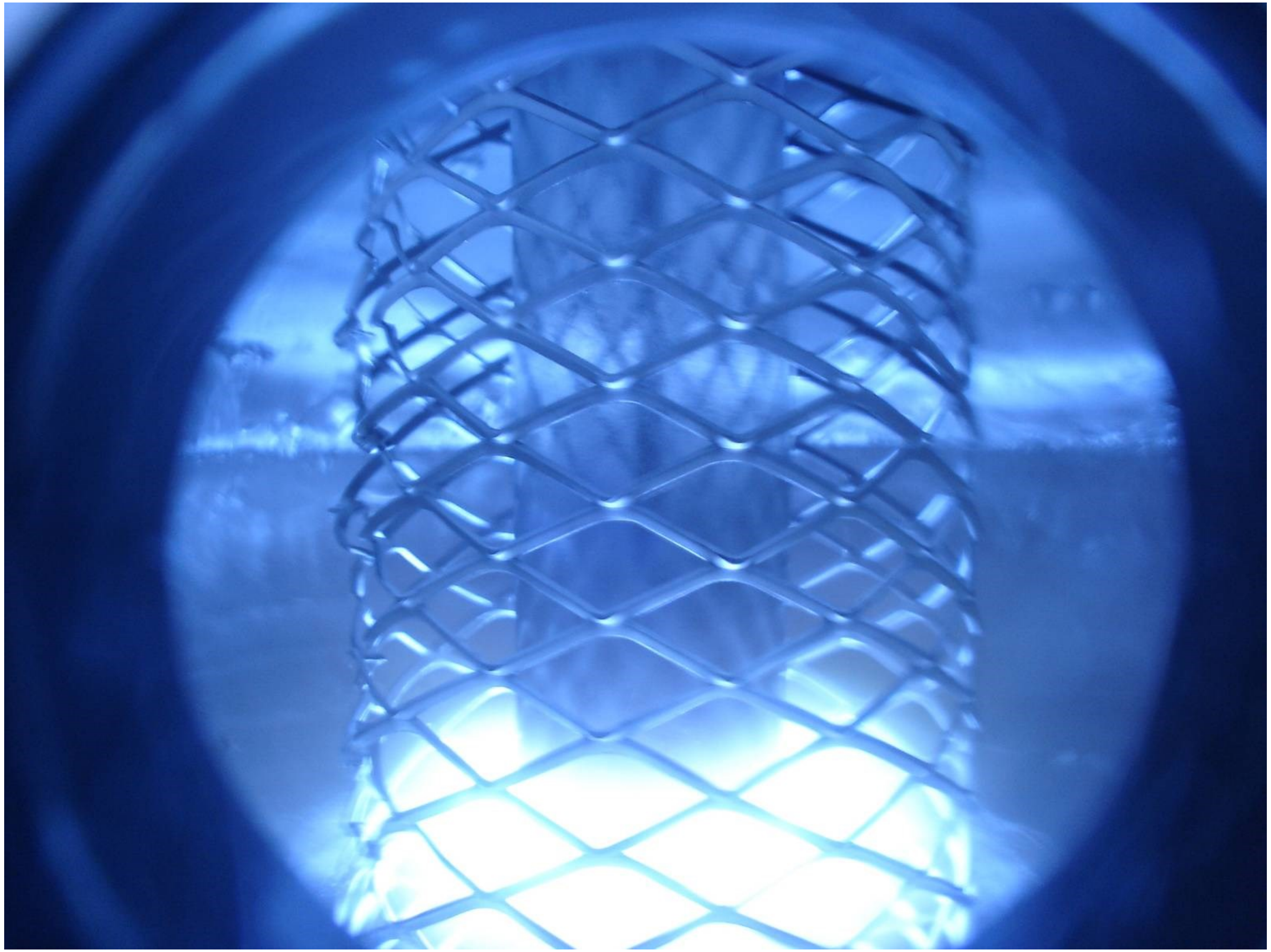
SPUTTERING PARAMETERS (1,5 GHz)

- RRR: 11.5 ± 0.1
- Argon content: 435 ± 70 ppm
- Grain size: 110 ± 20 nm
- **Tc: 9.51 ± 0.01 K**

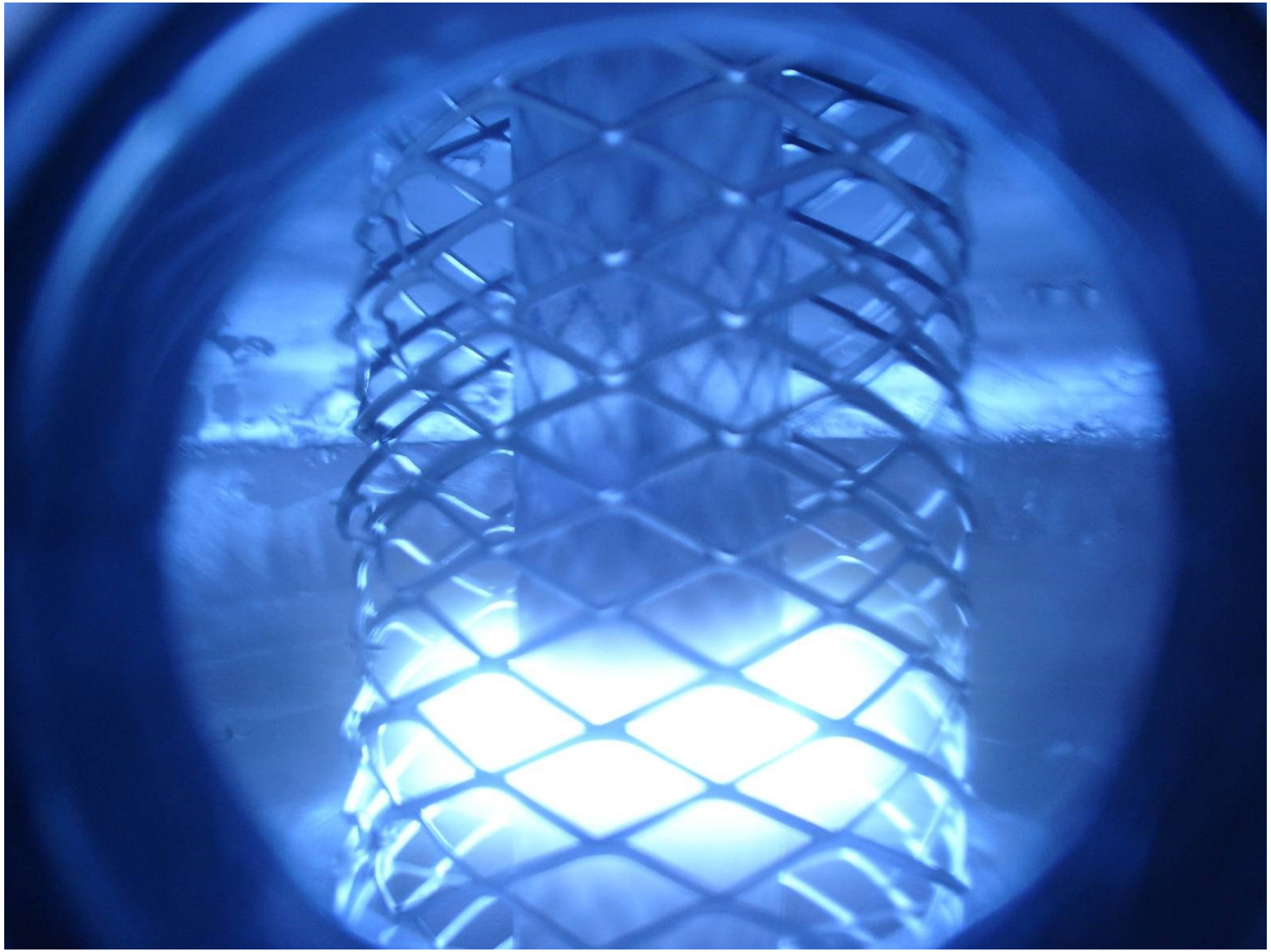


Courtesy of S. Calatroni (CERN)

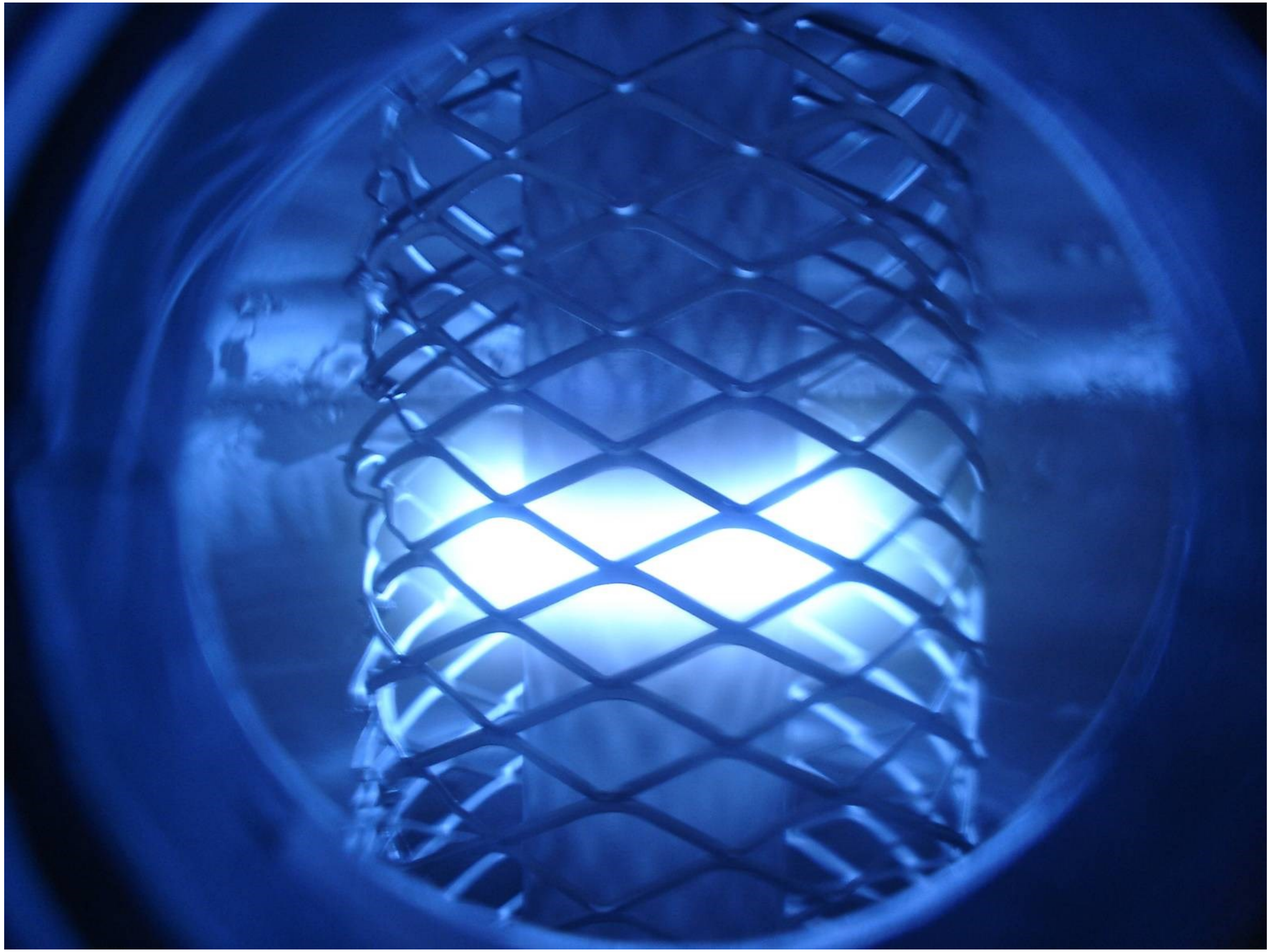
1.5 GHz Cavity Sputtering System @LNL



1.5 GHz Cavity Sputtering System @LNL



1.5 GHz Cavity Sputtering System @LNL



1.5 GHz Cavity Sputtering System @LNL

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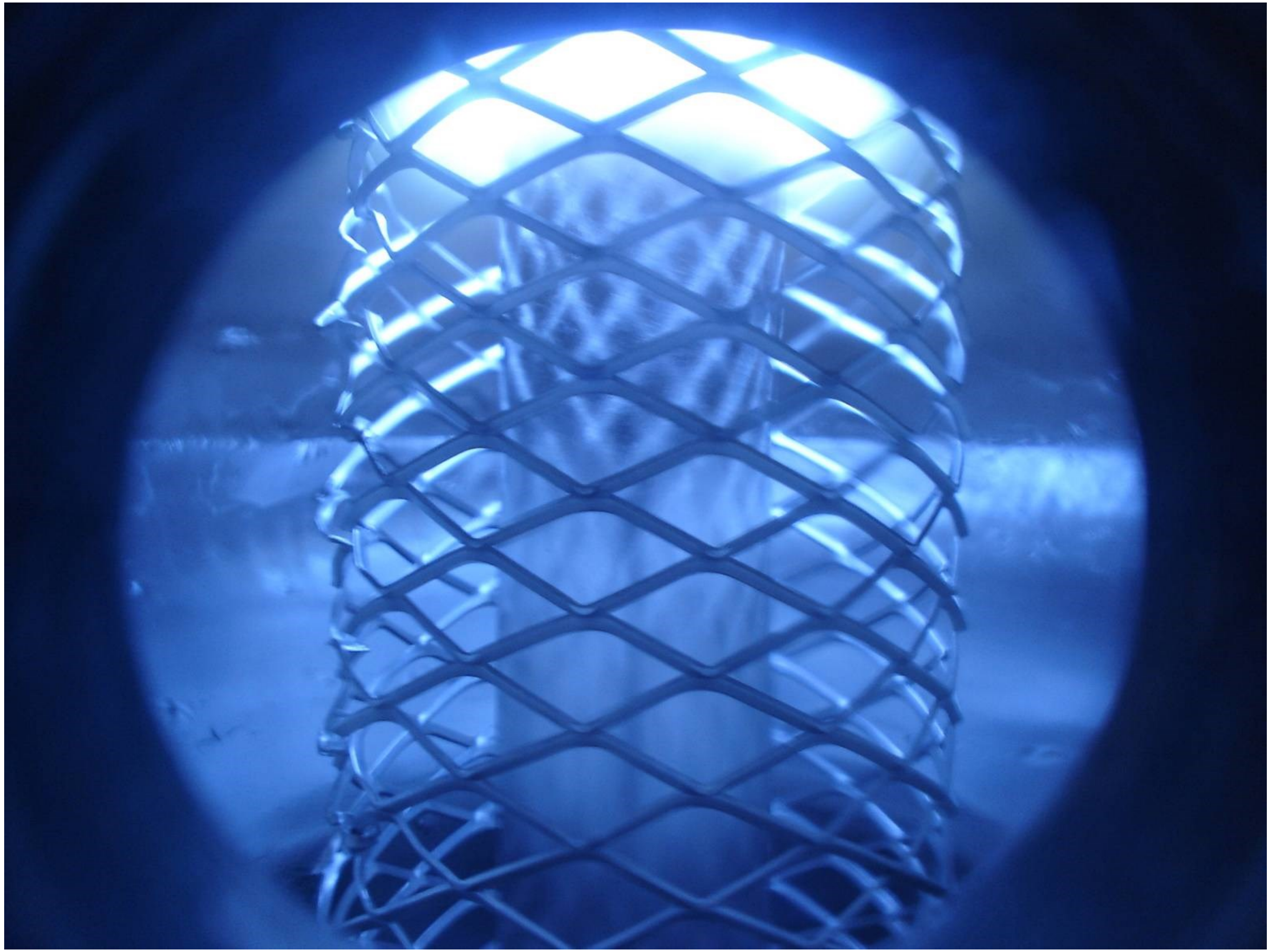
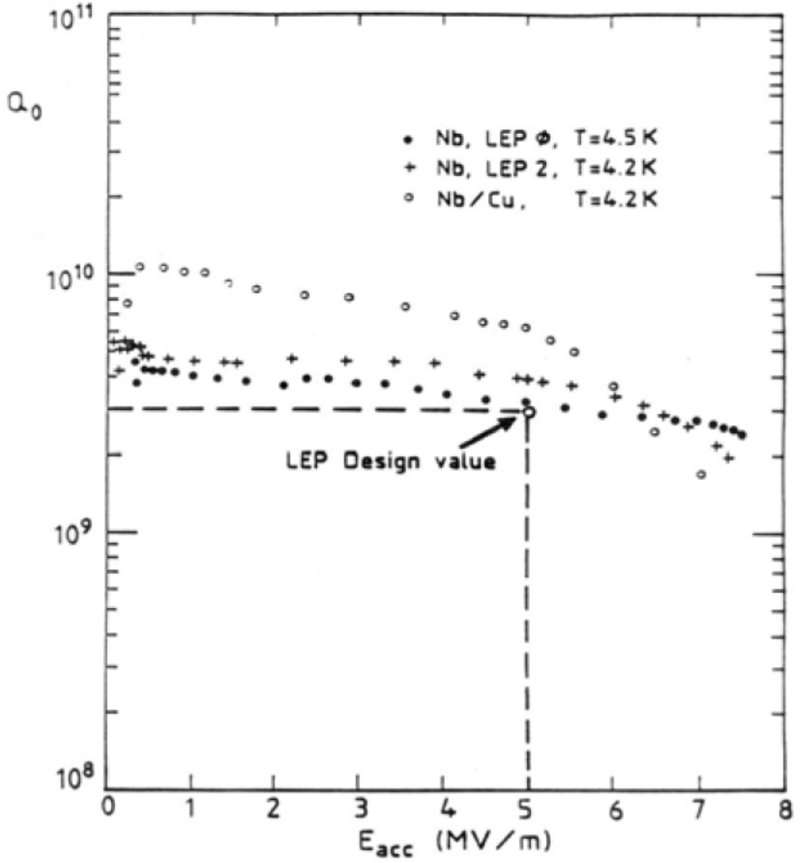


LEP2 Performances



G. Arnolds-Mayer et al. 1988 Proc. of the 3rd Workshop on RF Superconductivity

Eight pre-series 4-cell cavities for LEP were built at CERN, the remaining 264 were **made by three European industrial suppliers**

No thermal quench (contrary to bulk Nb)

Higher performances compare to bulk Nb

Nb bulk cavities performance in the eighties were limited by the poor Nb thermal conductivity (RRR of 40)

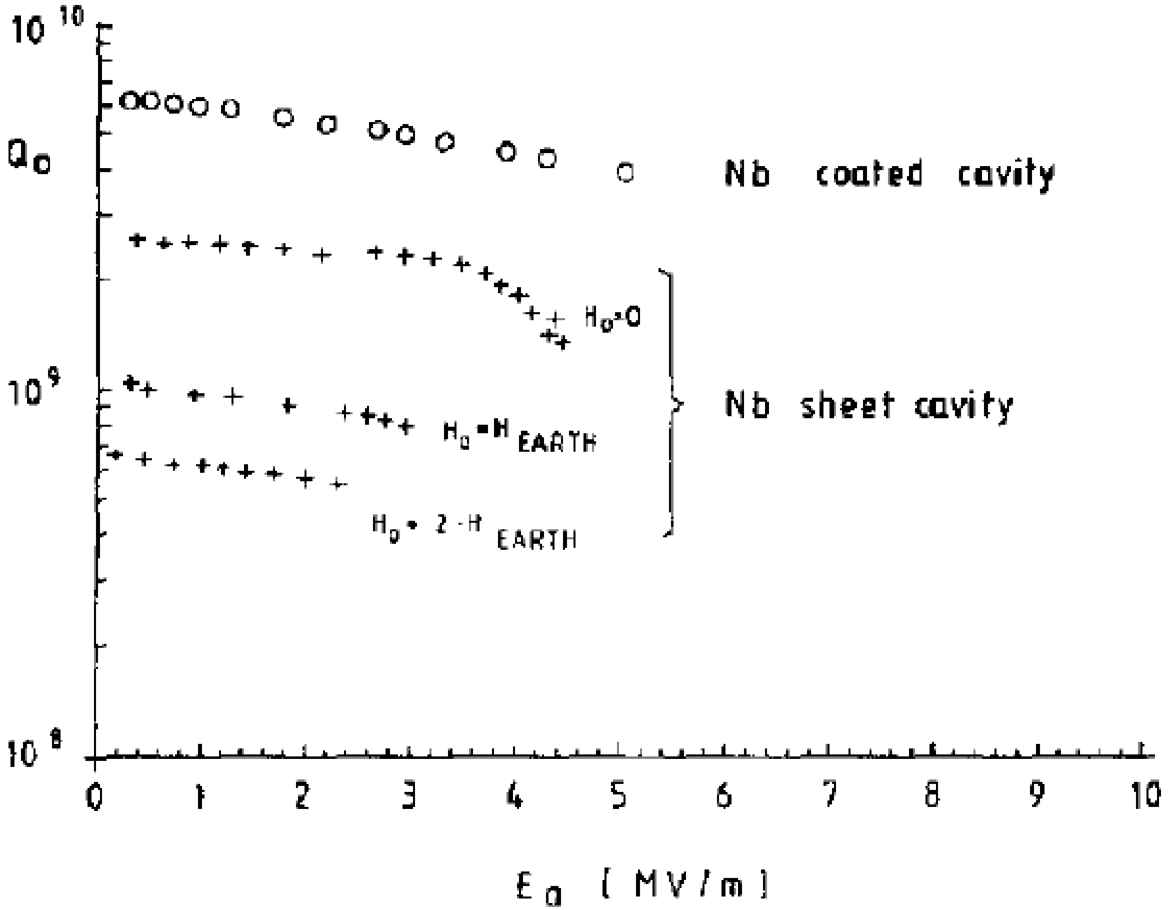
Unexpected advantage

Almost insensitive to the Earth's magnetic field

- Bulk Nb: 100 nΩ/Gauss
- Nb films: 1 nΩ/Gauss

cheaper cryostats

Not necessary complex magnetic shielding of the cavities



C. Benvenuti et al. I Physica B 197 (1994)

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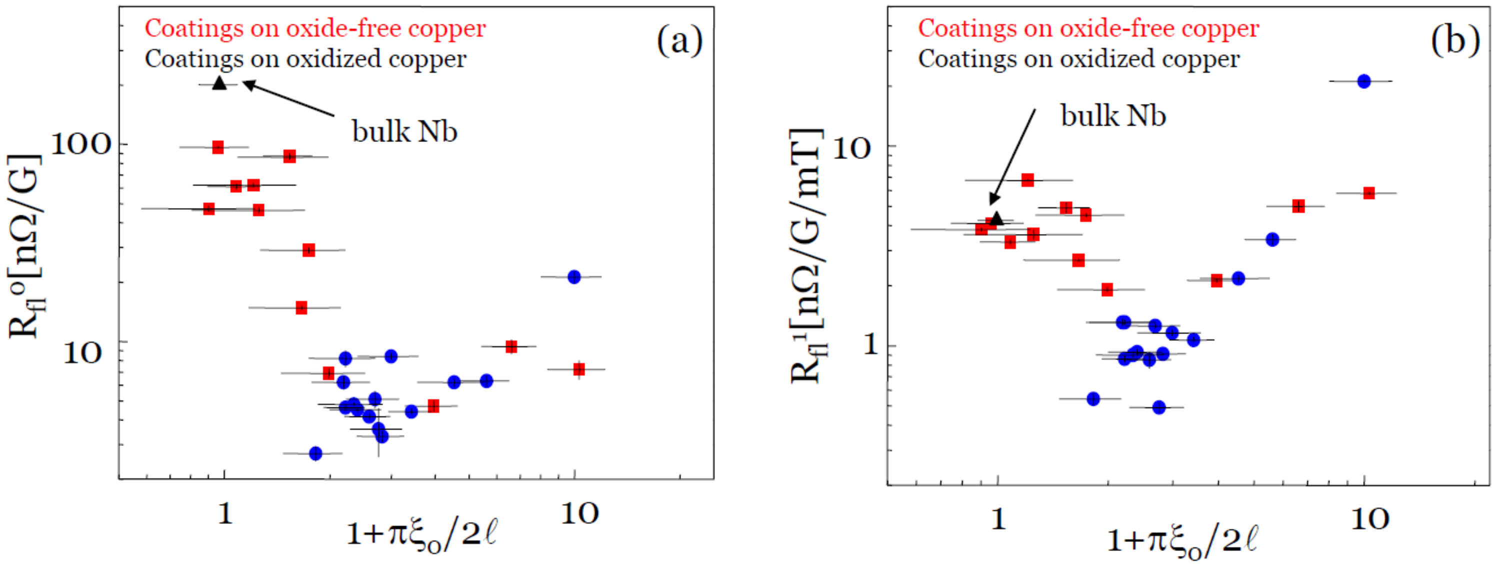
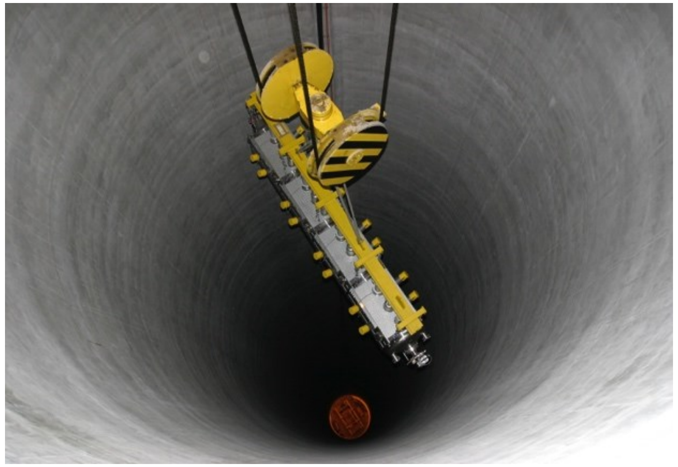


$$R_{fl} = (R_{fl}^0 + R_{fl}^1 H_{RF})\, H_{ext}$$

C. Benvenuti et al., Physica C 316 (1999) 153–188

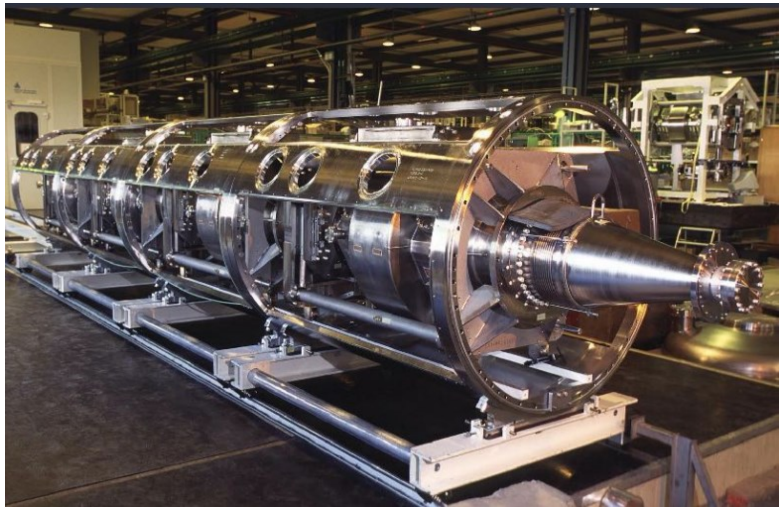
LHC cavities: from substrate fabrication to installation in the tunnel

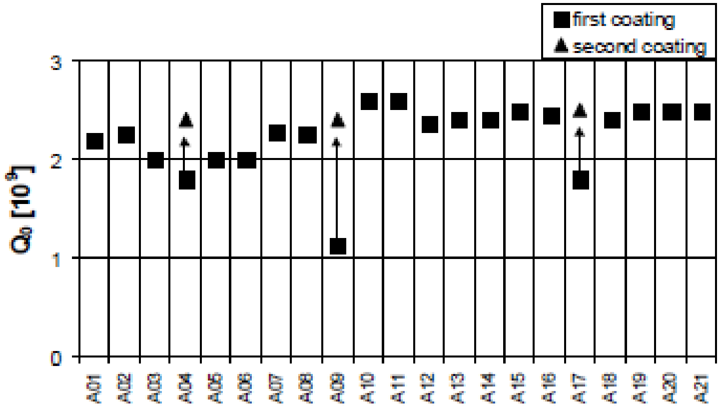


LHC Point 4, ~ 100 m deep shaft → LHC tunnel

Courtesy of A. Sublet (CERN)

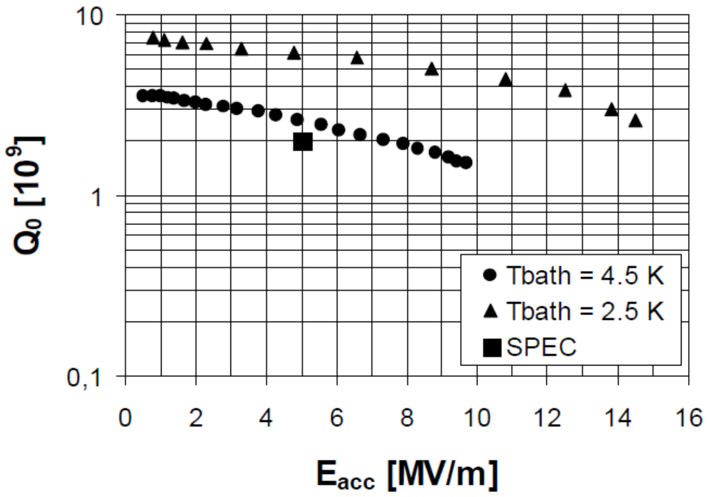
LHC

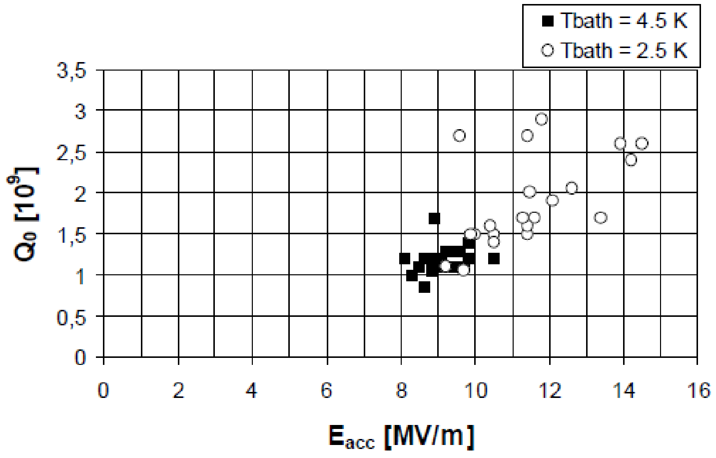




Distribution of cavity quality Q0 at a gradient of Eacc = 5 MV/m and a bath temperature of 4.5 K

21 cavities 400 MHz cavities produced at ACCEL

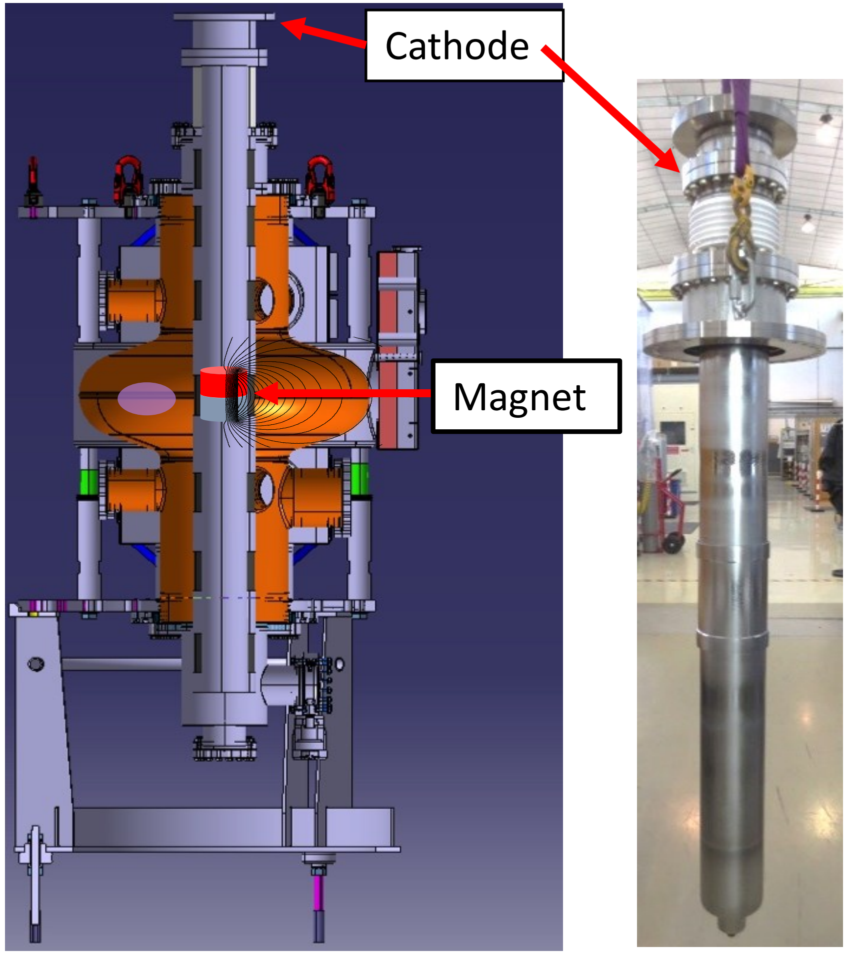




Highest gradients Eacc and quality factors Q0 at the highest gradients achieved at bath temperatures of 4.5 K and 2.5 K

S. Bauer *et al.*, "Production of Nb/Cu sputtered superconducting cavities for LHC," 1999

LHC cavities coating setup



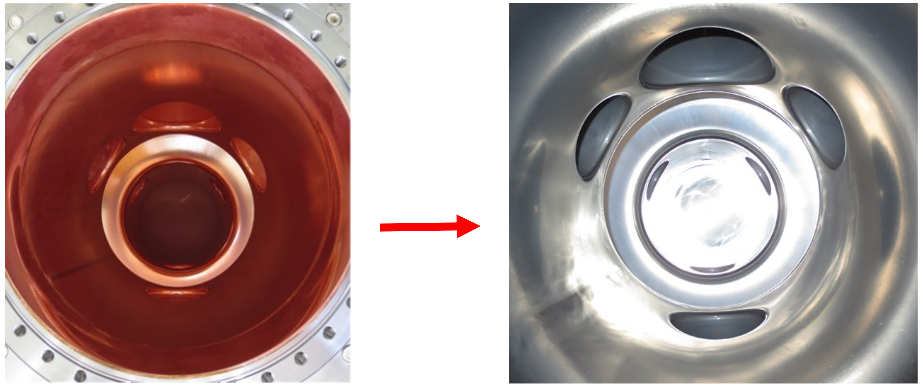
LHC cavity coating setup

- **Cavity as UHV chamber** (10^{-10} mbar base vacuum)
- Cavity = anode, grounded
- Nb cylindrical cathodes tubes
- **movable electromagnet inside**, liquid cooled

→ DC-magnetron sputtering, **6 kW, 1.10^{-3} mbar Kr**

→ Cavity bake-out (bake-out tent) to 180°C
→ Coating 7 steps for the 7 different electromagnet positions
→ Duration = 1h 20' at low temperature (150°C)
→ Nb layer thickness ~ 2 mm

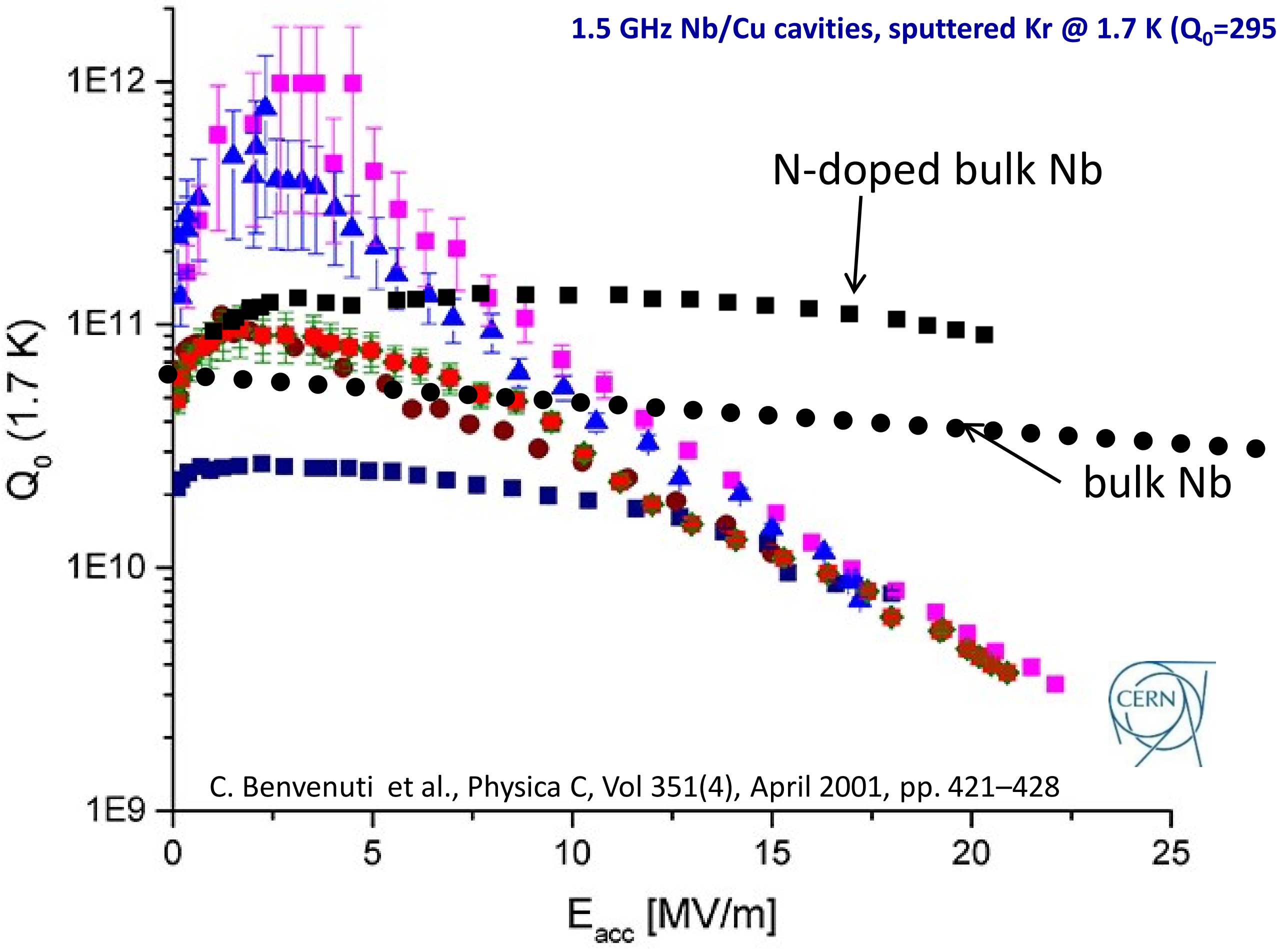
8 spare cavities produced



Before and after coating views from cavity main aperture

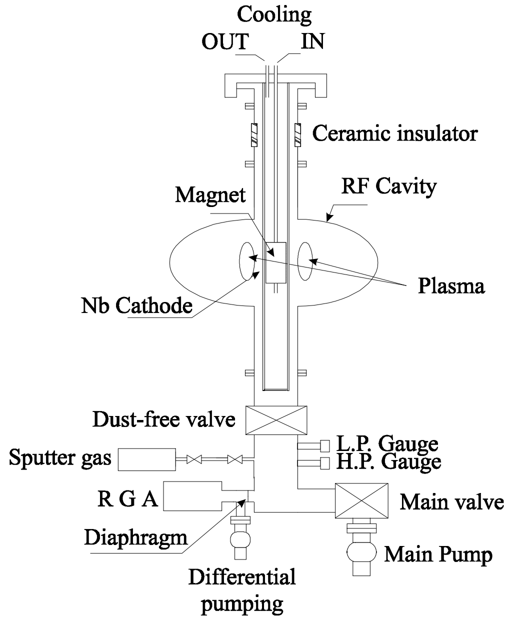
A. Sublet, Thinfilms Workshop 2018

R&D on 1,5 GHz - State of the art for Nb-Cu cavities



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

C. Benvenuti et al., Physica C, Vol 351(4), April 2001, pp. 421–428

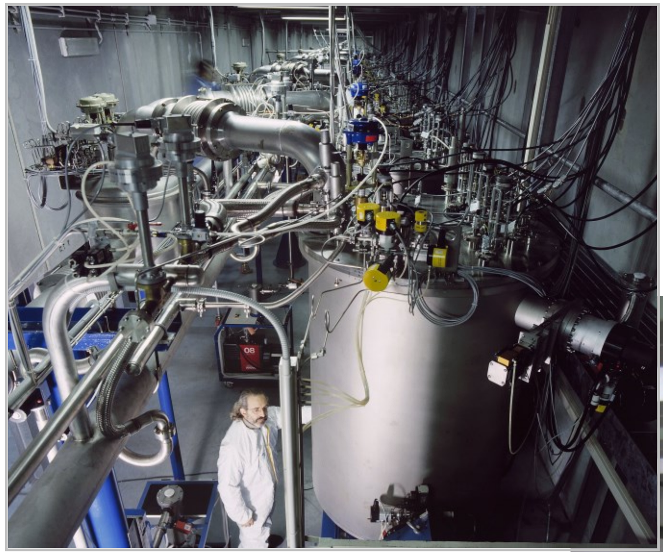


High Q at low field

Strong Q-slope still present

Increasing complexity:
Quarter Wave Resonator

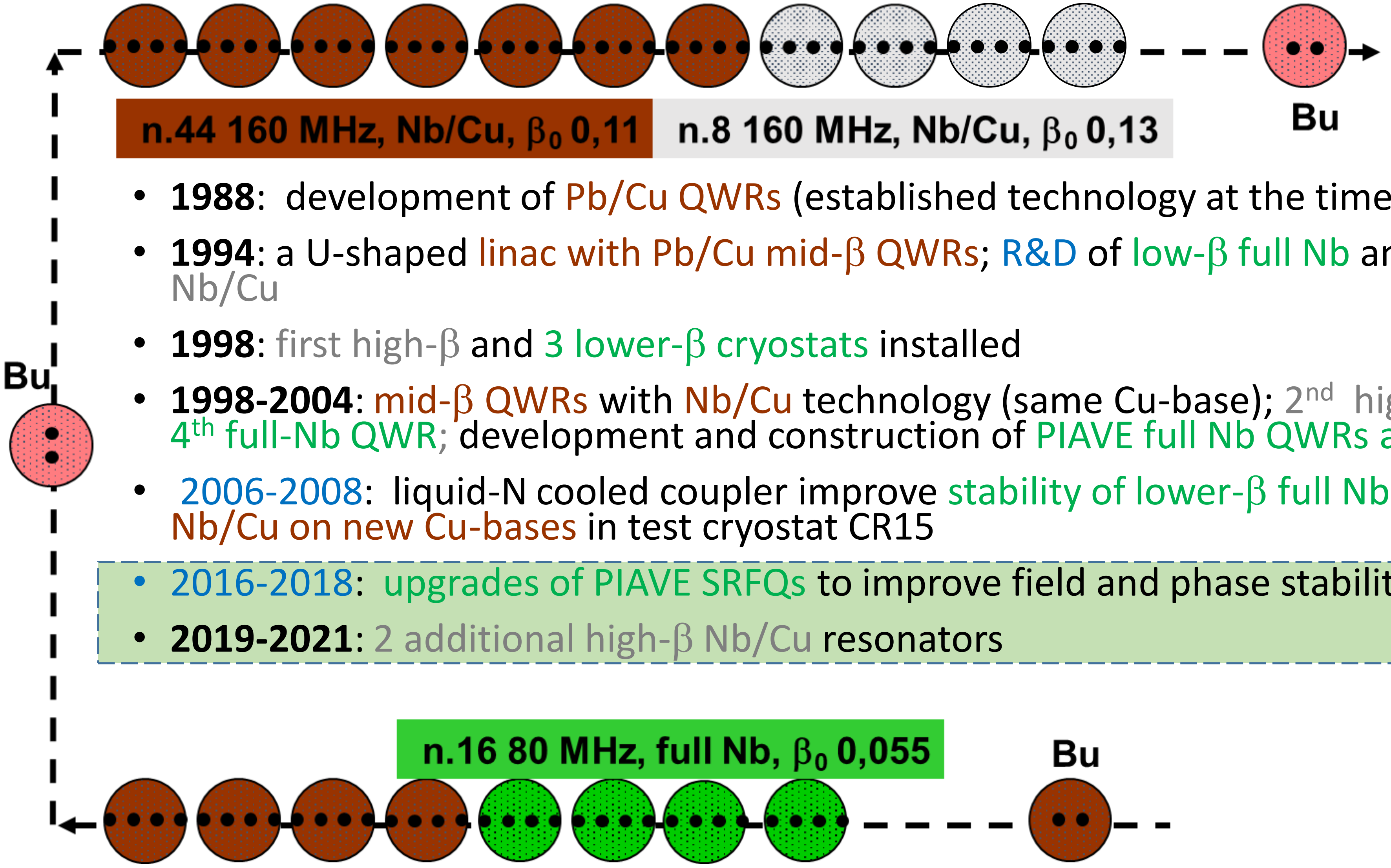
ALPI @ LNL - 160 MHz QWR



A more complex geometry than elliptical cavities

State of the art in Nb thin films

QWR in ALPI @ LNL INFN

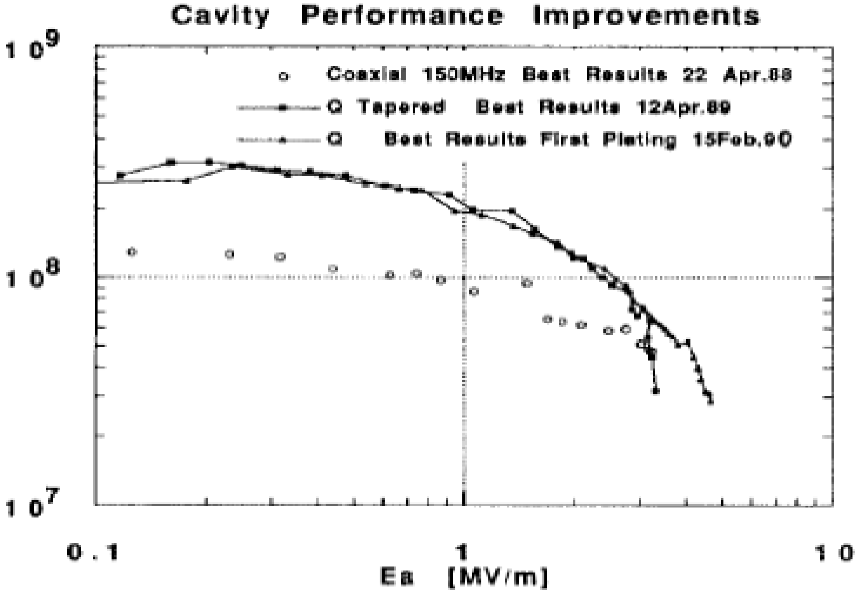


- **1988**: development of Pb/Cu QWRs (established technology at the time)
- **1994**: a U-shaped linac with Pb/Cu mid-β QWRs; R&D of low-β full Nb and high-β Nb/Cu
- **1998**: first high-β and 3 lower-β cryostats installed
- **1998-2004**: mid-β QWRs with Nb/Cu technology (same Cu-base); 2nd high-β QWR, 4th full-Nb QWR; development and construction of PIAVE full Nb QWRs and SRFQs;
- 2006-2008: liquid-N cooled coupler improve stability of lower-β full Nb QWRs; Nb/Cu on new Cu-bases in test cryostat CR15
- 2016-2018: upgrades of PIAVE SRFQs to improve field and phase stability
- **2019-2021**: 2 additional high-β Nb/Cu resonators

G. Bisoffi – Long-term SRF Experience at INFN-Legnaro, TTC Meeting – Vancouver 2019

The early ALPI with mid-β Pb/Cu QWRs


1992: ALPI during first assembly



E_a = 2.3÷2.7 MV/m

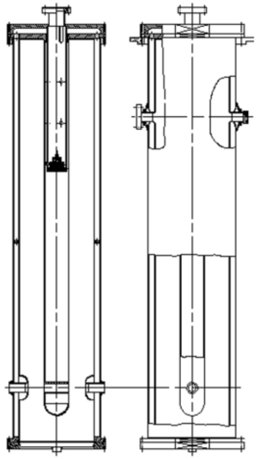
β_0=0,11

Reliable operation

Cheaper than full Nb, **mechanically stable, not** susceptible to **quench**, ideal for complicated geometries

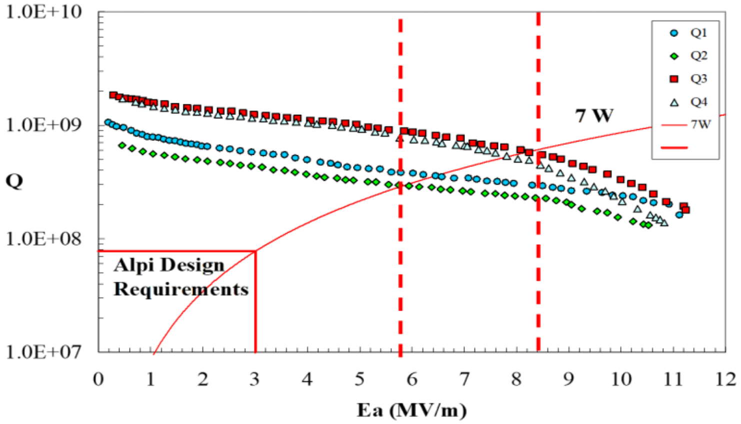
Limited performance, and some **degradation of E_a**

R&D on full-Nb and Nb/Cu QWRs

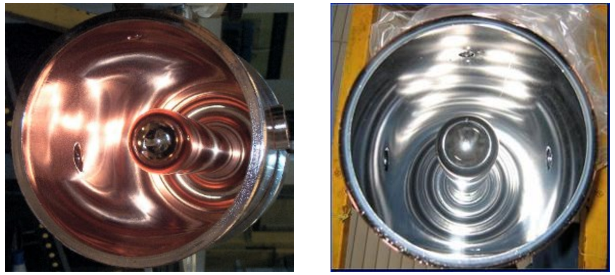


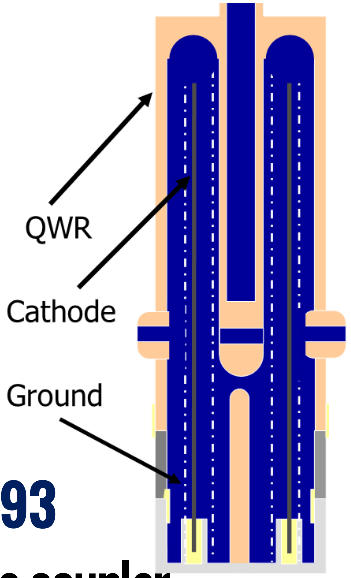
First 80 MHz low-β full Nb cavity in 1993

(double wall thin Nb outer conductor, equipped with original mechanical dampers)



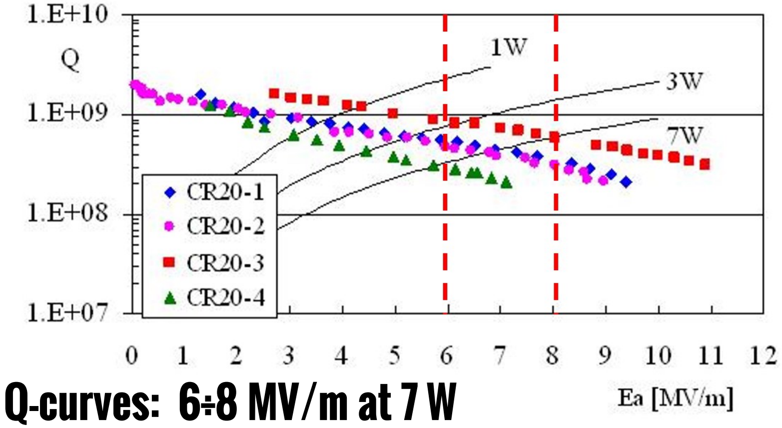
Off-line Q-curves: 6÷8 MV/m at 7 W





First 160 MHz high-b Nb/Cu cavity in 1993

(geometry optimized for sputtering, capacitive coupler far from the shorting plate)



Off-line Q-curves: 6÷8 MV/m at 7 W

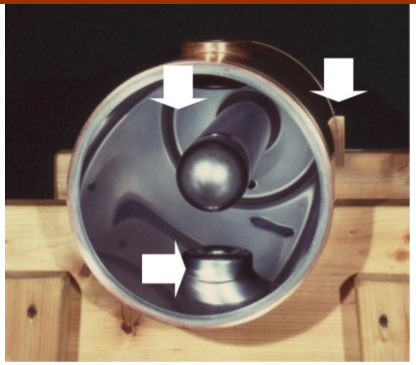
Higher mechanical stability
Less sensitive to microphonic effects

G. Bisoffi – Long-term SRF Experience at INFN-Legnaro, TTC Meeting – Vancouver 2019

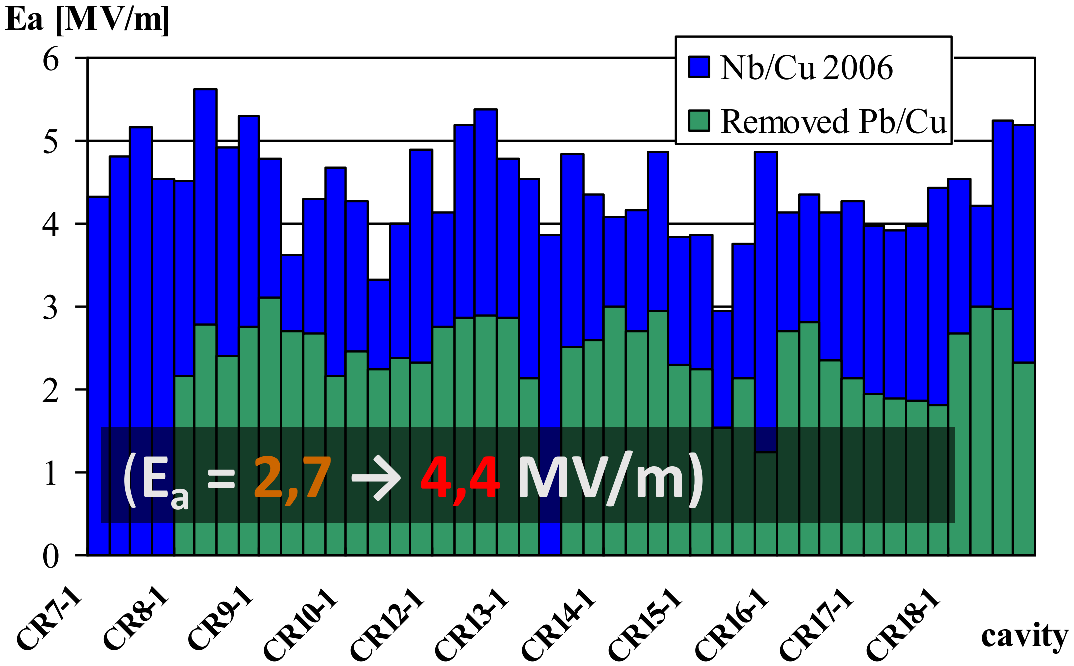
The next step: apply Nb/Cu to mid-β QWRs…

n.44 160 MHz, Nb/Cu, β_0 0,11 **n.8 160 MHz, Nb/Cu, β_0 0,13**

Bu



… on a non-Nb/Cu-optimized geometry and Cu-brazed joints (smaller radii on end pkate and beam-ports, beam coupler hole in high-H region)



(E_a = 2,7 → 4,4 MV/m)

Bul

n.16 80 MHz, full Nb, β_0 0,055

Bu

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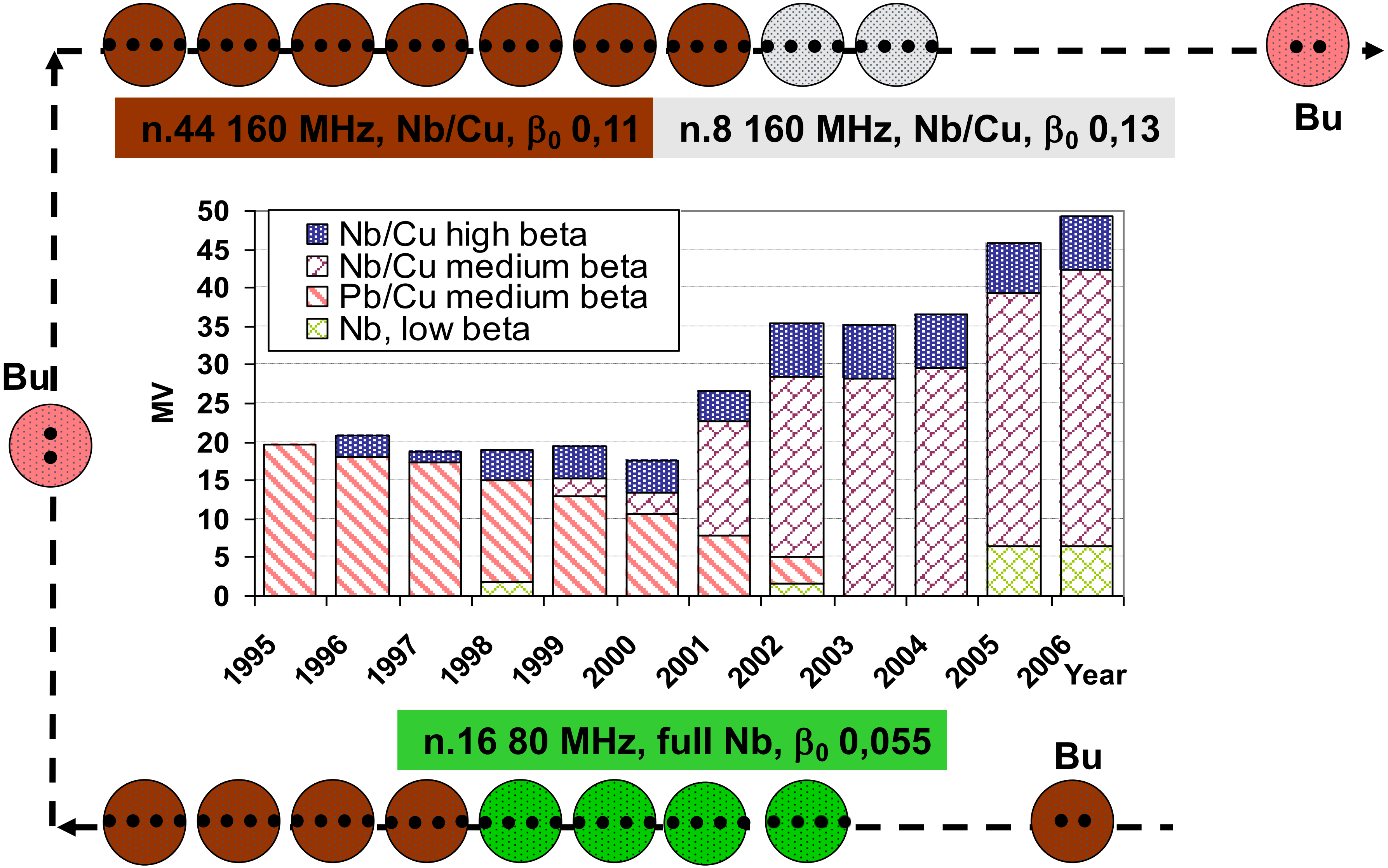
ALPI V_{eq} from 20 to 48 MV



n.44 160 MHz, Nb/Cu, β_0 0,11

n.8 160 MHz, Nb/Cu, β_0 0,13

n.16 80 MHz, full Nb, β_0 0,055

G. Bisoffi – Long-term SRF Experience at INFN-Legnaro, TTC Meeting – Vancouver 2019

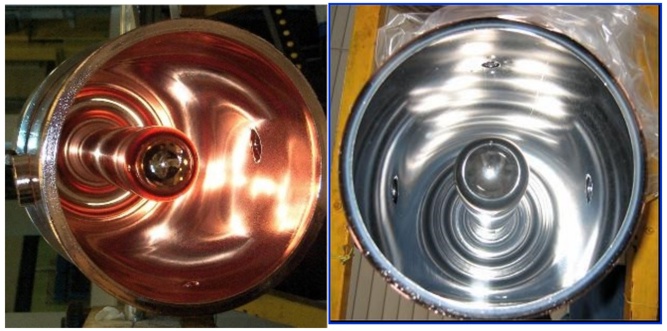
ALPI QWR - Effect of the substrate

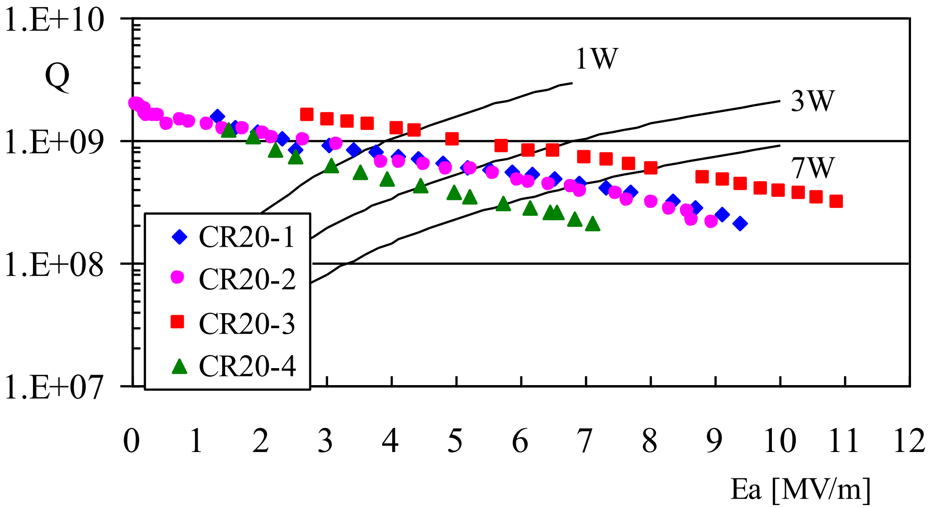
High β QWRs (β=0.13, 160 MHz)

Drilled by a billet of OFHC Cu, 99.95% grade

No brazed joints, beam ports jointed by indium gaskets

Rounded shorting plate



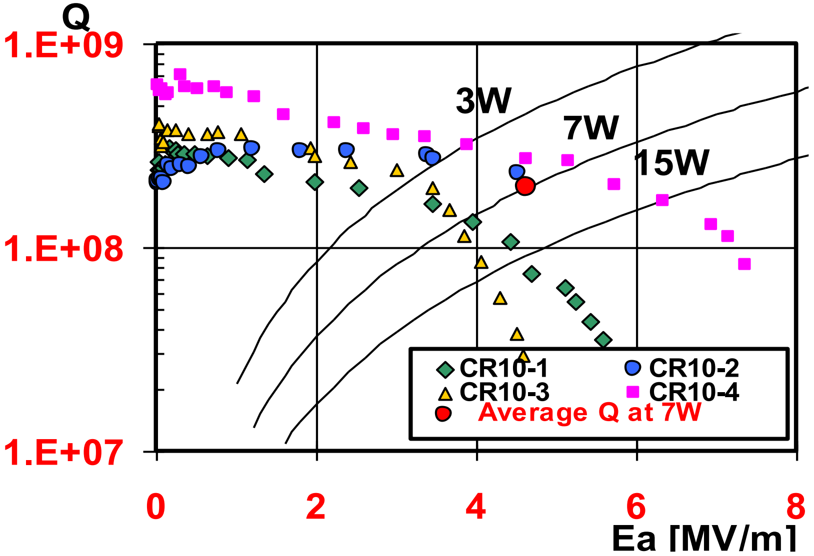


Medium β QWRs (β=0.13, 160 MHz)

Brazed joints (especially the ones in the outer resonator surface)

Flat shorting plate

Beam ports shape



A. Porcellato, Nb Sputtered Cu QWR, Thin Films Workshop LNL 2006

Nb/Cu sputtering advantages

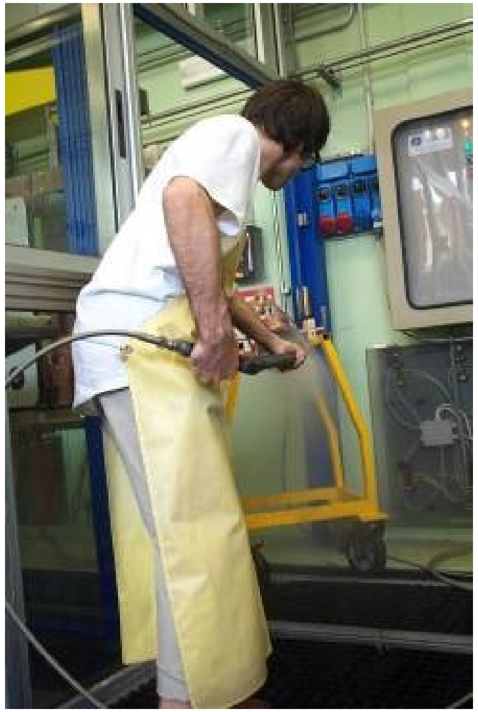
Mechanical stability (mechanical vibrations are not an issue)
Frequency not affected by changes He bath Dp (<0.01Hz/mbar)
Reduced over-coupling (smaller amplifier, coupler do not need cooling, rf lines have reduced size and limited rf dissipation)
High thermal stability (less prone to hot spots, conditioning easier)
Stiffness (in case of loss of isolation vacuum leak)
Absence of Q-disease (less demand on cryogenic system cooling velocity and reliability)
Insensitivity to small magnetic fields (no magnetic shielding)
High Q of the N.C. cavity (easier coupling in N.C state)
Absence of In vacuum joints (vacuum leaks less probable)
Price (both material and construction)

The lower performance of Nb/Cu cavities at high fields, due to the more pronounced Q-slope of Nb/Cu resonators, is not an issue in QWRs as it is in b>0.5 cavities, because beam dynamic constraints require to limit the accelerating gradient in the low b section of linacs to values well reachable by Nb sputtered resonators

A. Porcellato, Nb Sputtered Cu QWR, Thin Films Workshop LNL 2006

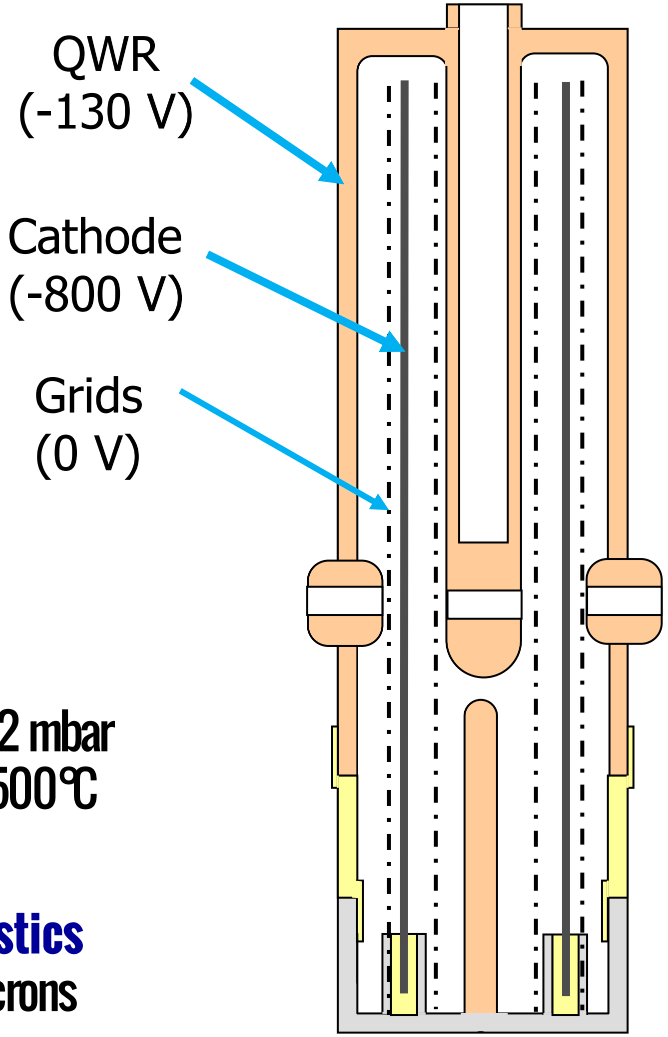
ALPI QWR - Substrate preparation

- Electropolishing (20μm, 2 hours)
- Rinsing (water, ultrasonic water, HPR)
- Chemical polishing (10μm, 4 min, SUBU5)
- Passivation (sulphamic acid)
- Rinsing (water, ultrasonic water, HPR)
- Drying (ethanol, nitrogen)



A. Porcellato, Nb Sputtered Cu QWR, Thin Films Workshop LNL 2006

ALPI QWR - Sputtering process



QWR
(-130 V)

Cathode
(-800 V)

Grids
(0 V)

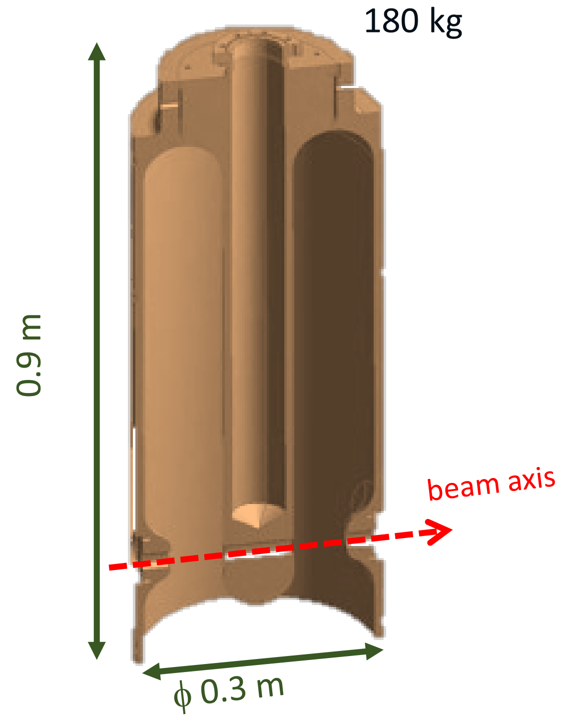
Parameters
Argon pressure: 0.2 mbar
Substrate T: 300-500°C

Film characteristics
Thickness: 1-2 microns
RRR: 9-20

A. Porcellato, Nb Sputtered Cu QWR, Thin Films Workshop LNL 2006

HIE-ISOLDE @ CERN

Superconducting linear accelerator for energy upgrade of ISOLDE radioactive ion beam facility



Cryomodule clean room assembly

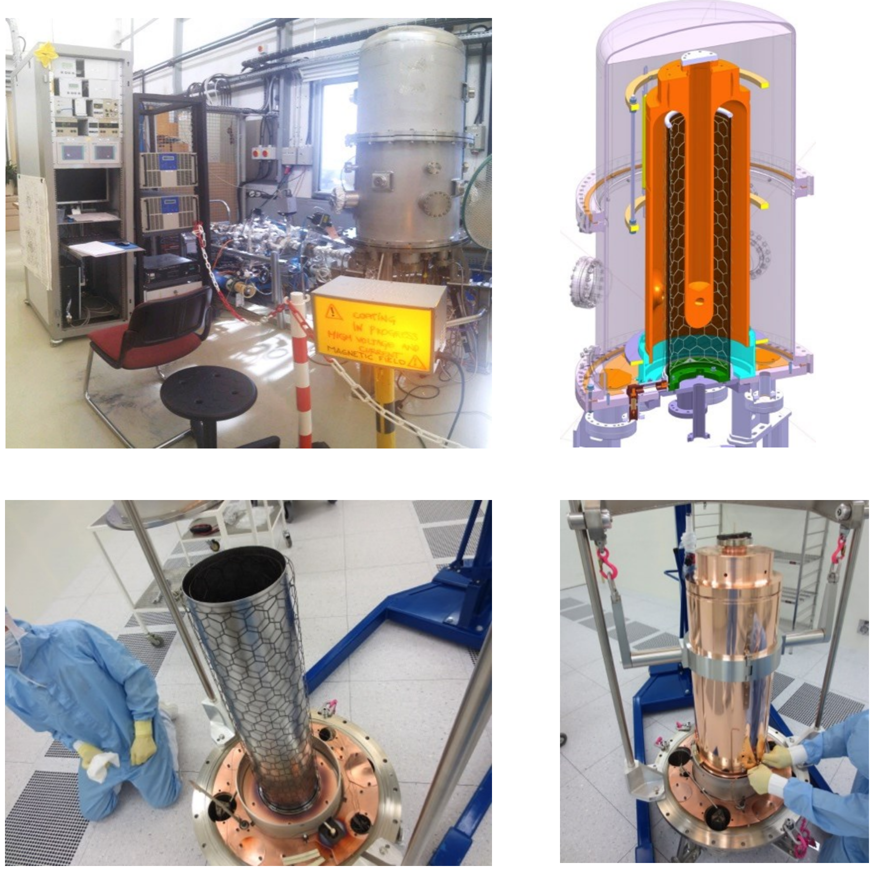
4 cryomodules in HIE-ISODLE Linac

Courtesy of A. Sublet (CERN)

HIE-ISOLDE @ CERN

Same sputtering **configuration** as in **ALPI**

Clean room assembly improvement



Cavity in UHV chamber (10^{-8} mbar base vacuum)

3D-forged Cu cavity substrate, biased at -80 V

Nb cylindrical cathode used on both sides, not cooled

DC-bias diode sputtering, 8 kW, Ar 0.2 mbar

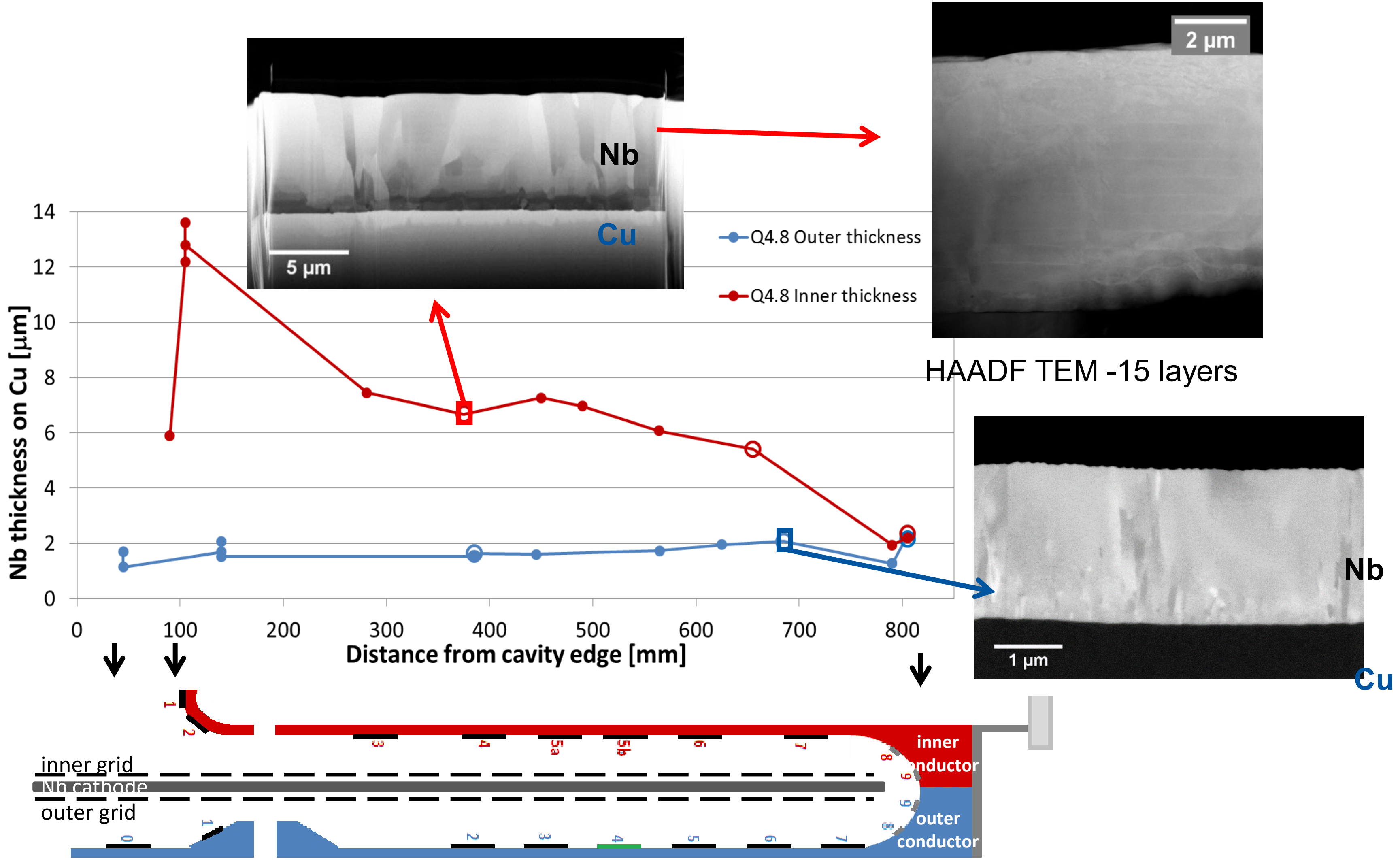
Coating at high temperature (300 → 620℃)

Done in 15 run/cool-down cycles (4 days)

Nb layer thickness ranging from 1.5 mm to 12 mm

Courtesy of A. Sublet (CERN)

HIE-ISOLDE film characteristics

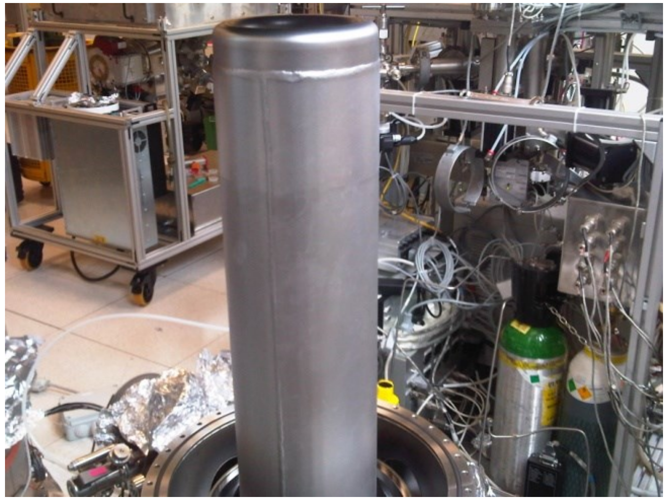


HAADF TEM -15 layers

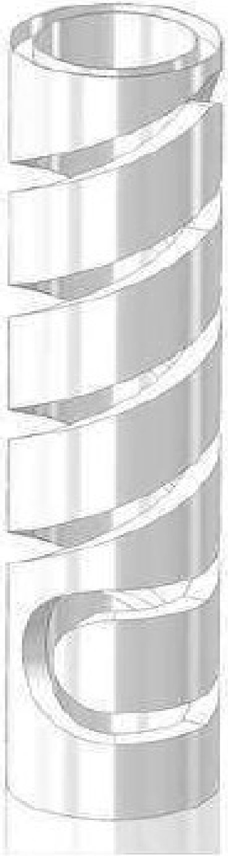
→ Influence of “multilayered” Nb film/dislocations/morphology on the RF performances?

A. Sublet (CERN), Thinfilms Workshop, LNL 2018

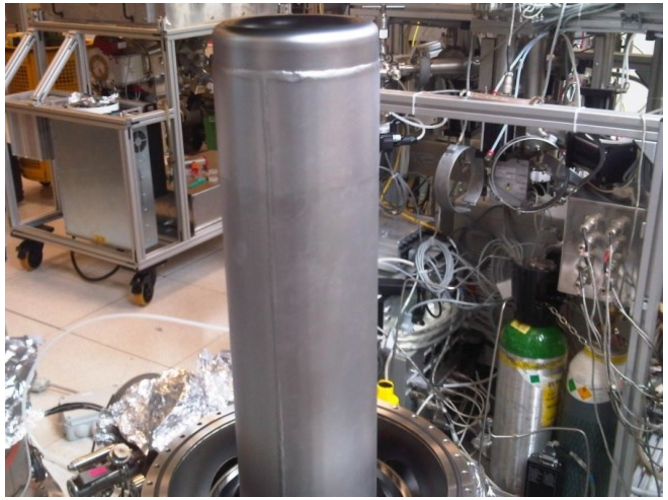
HIE ISOLDE R&D @LNL



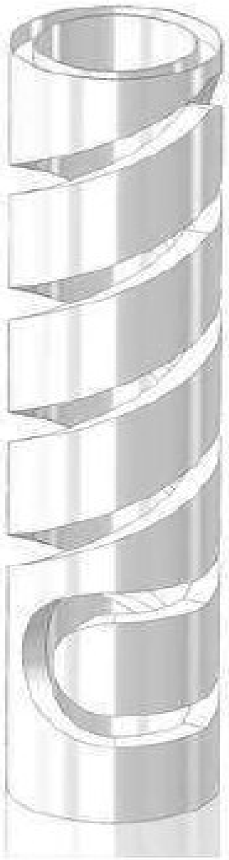
Helicoidal magnetic configuration

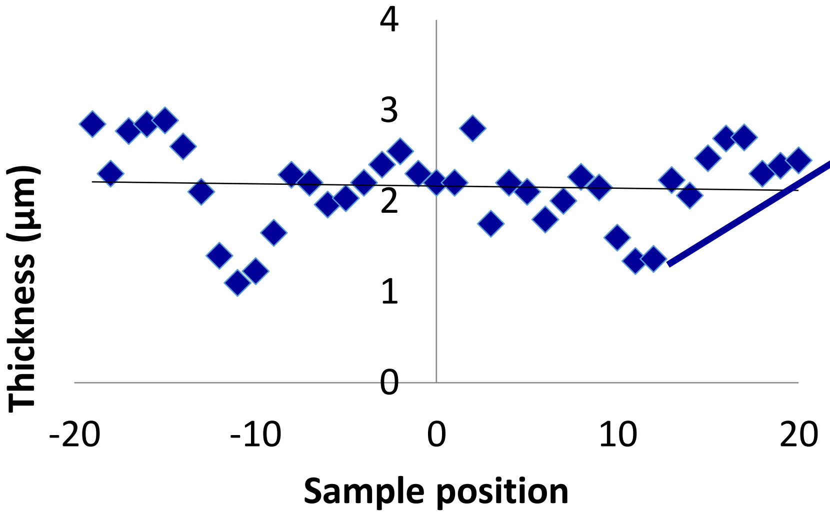


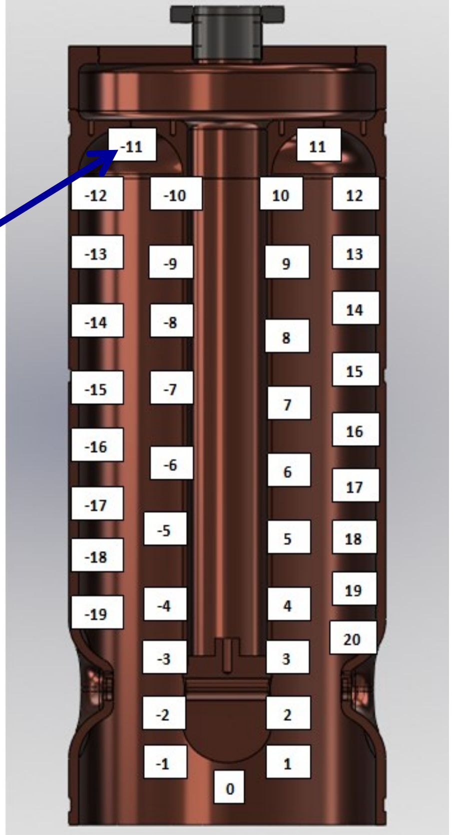
HIE ISOLDE R&D @LNL



Helicoidal magnetic configuration





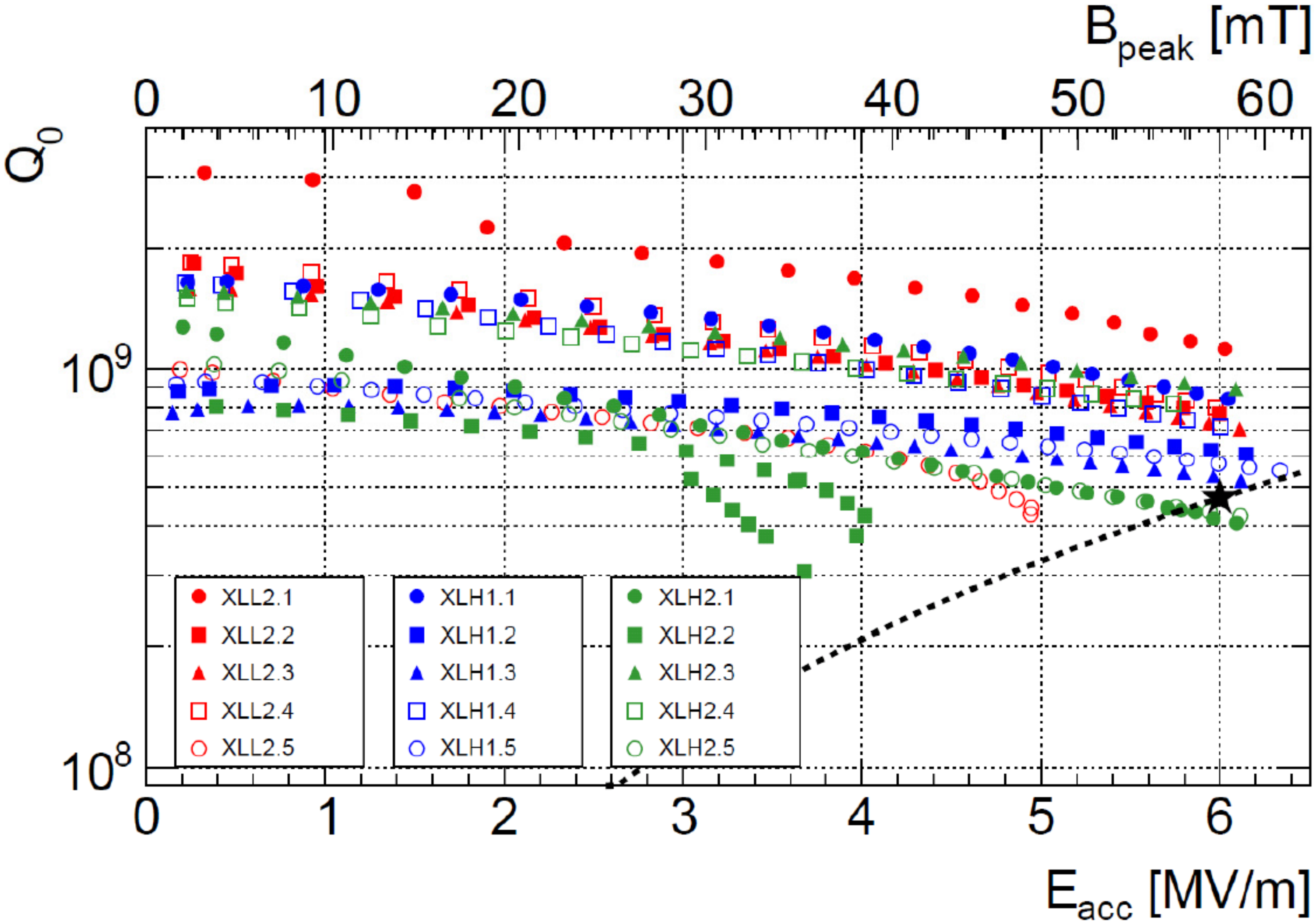


Good thickness uniformity

R&D stopped due to difficulties in the handling of the chemical polishing

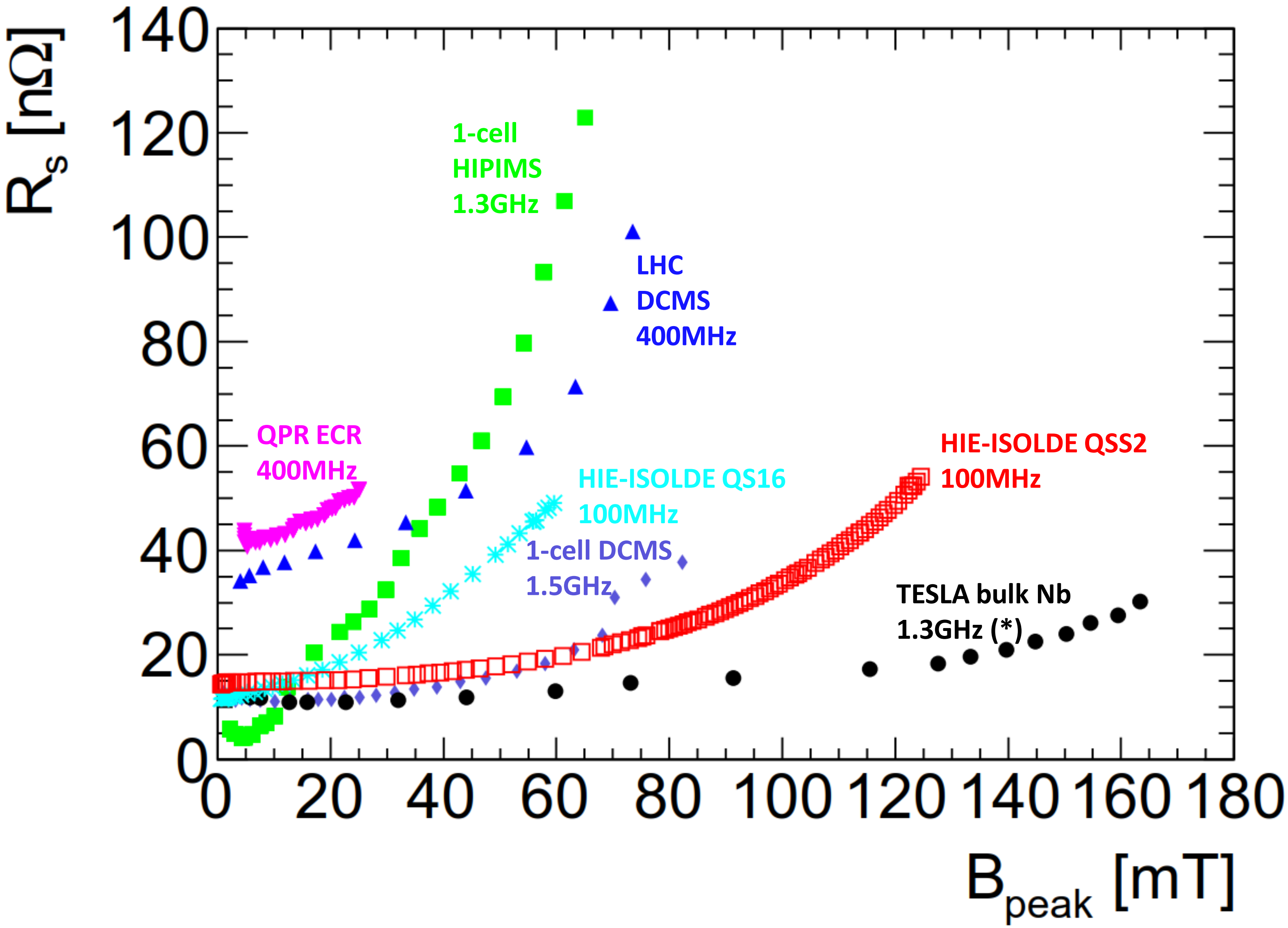
(HIE ISOLDE QWR larger than ALPI QWR)

HIE-ISOLDE QWR Performances



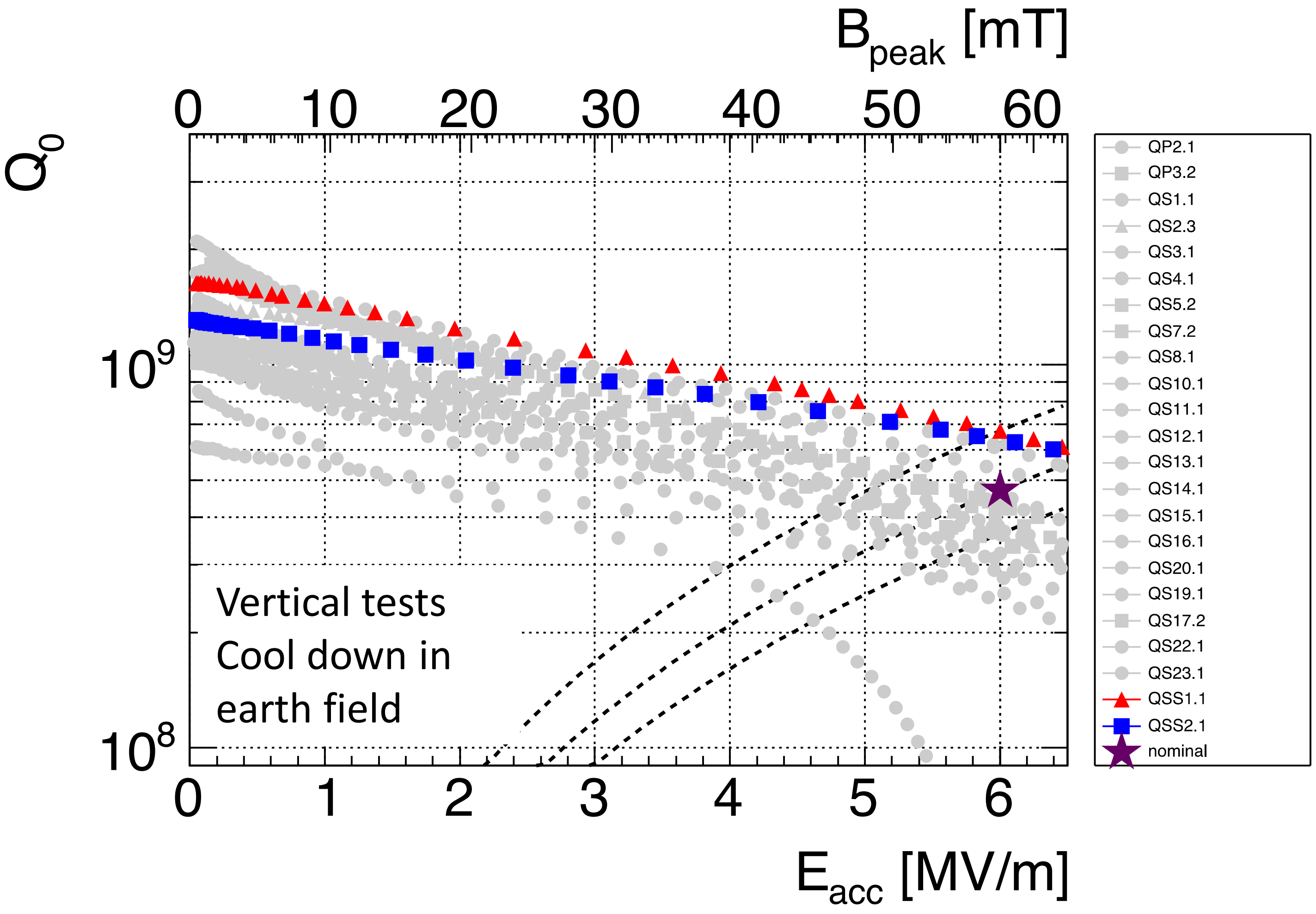
Courtesy of W. Venturini (CERN)

State of the art of Nb-Cu films around 2 K



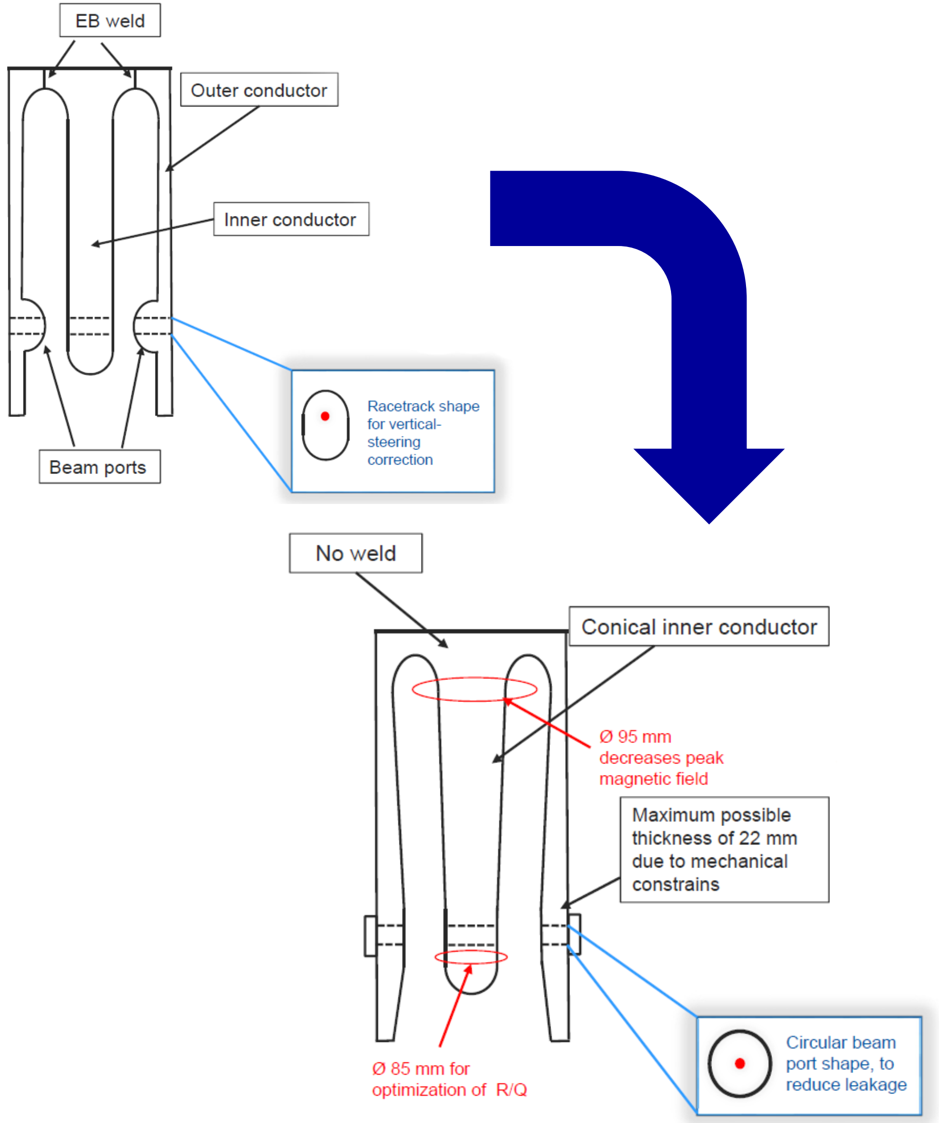
Courtesy of W. Venturini (CERN)

HIE-ISOLDE QWR Seamless Design

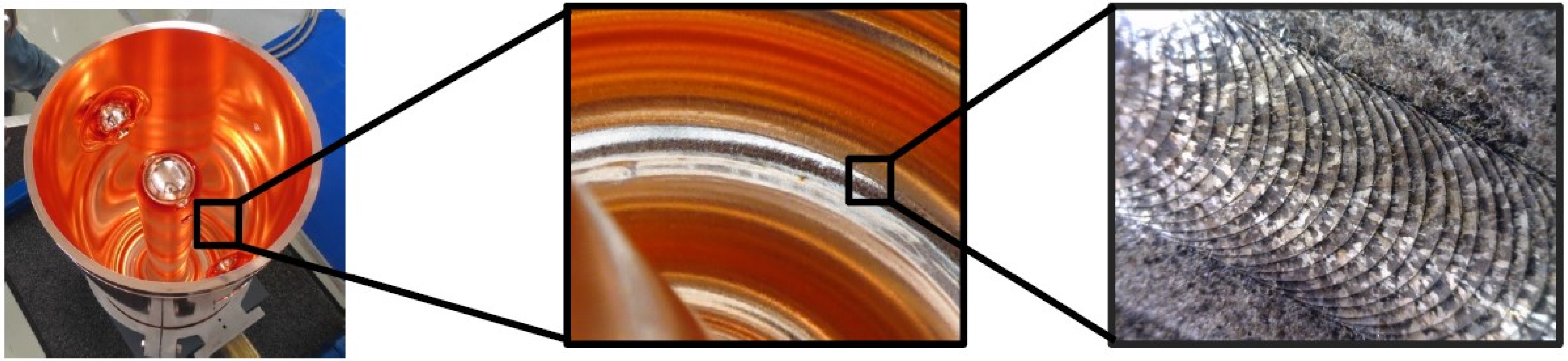


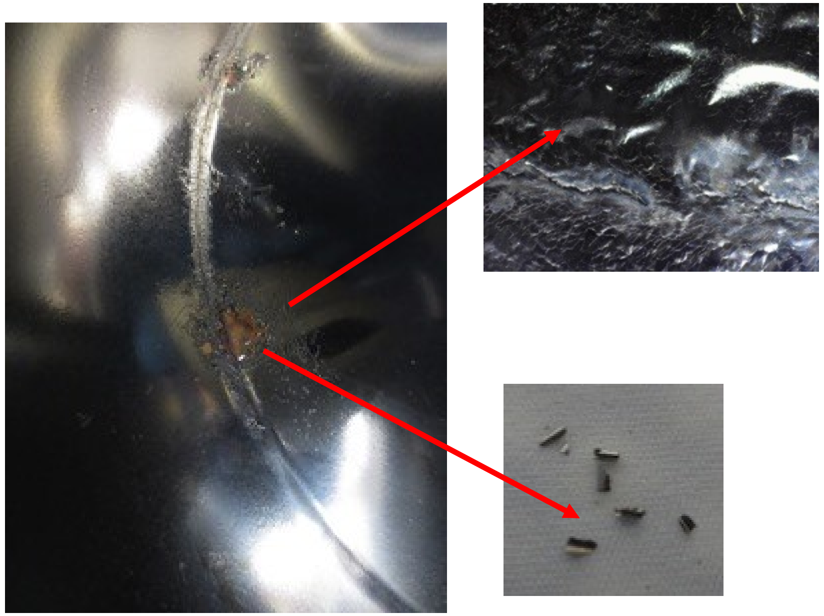
Vertical tests
Cool down in
earth field

Courtesy of W. Venturini (CERN)



Motivation through the seamless design





- → **longitudinal fracture** along the weld
- → Source of chemicals trap/release
- → oxidation/contamination → peel-off

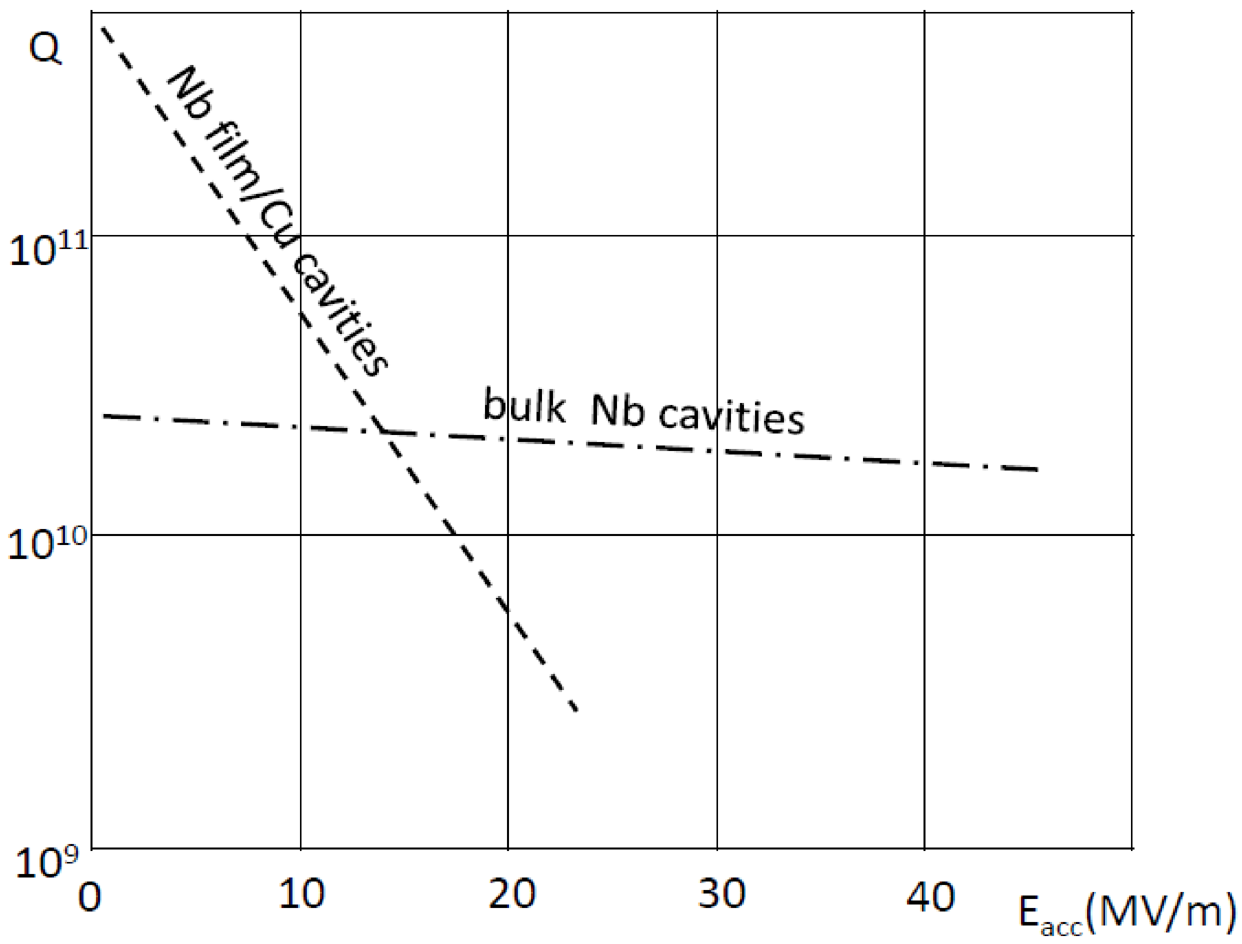
Courtesy of A. Sublet (CERN)

QWR Main Lesson learned: the substrate is important!

Outline

- Motivation for thin films in SRF cavities
- How to realize a thin film coating?
- State of the art in Nb thin films (accelerators using thin film technology)
- **Characteristics of Nb films**
- R&D on Nb films

Q-slope problem



Unsolved problem since 1990s

Several theory proposed

Depinning of trapped flux

Low HC1

Early vortex penetration due to roughness

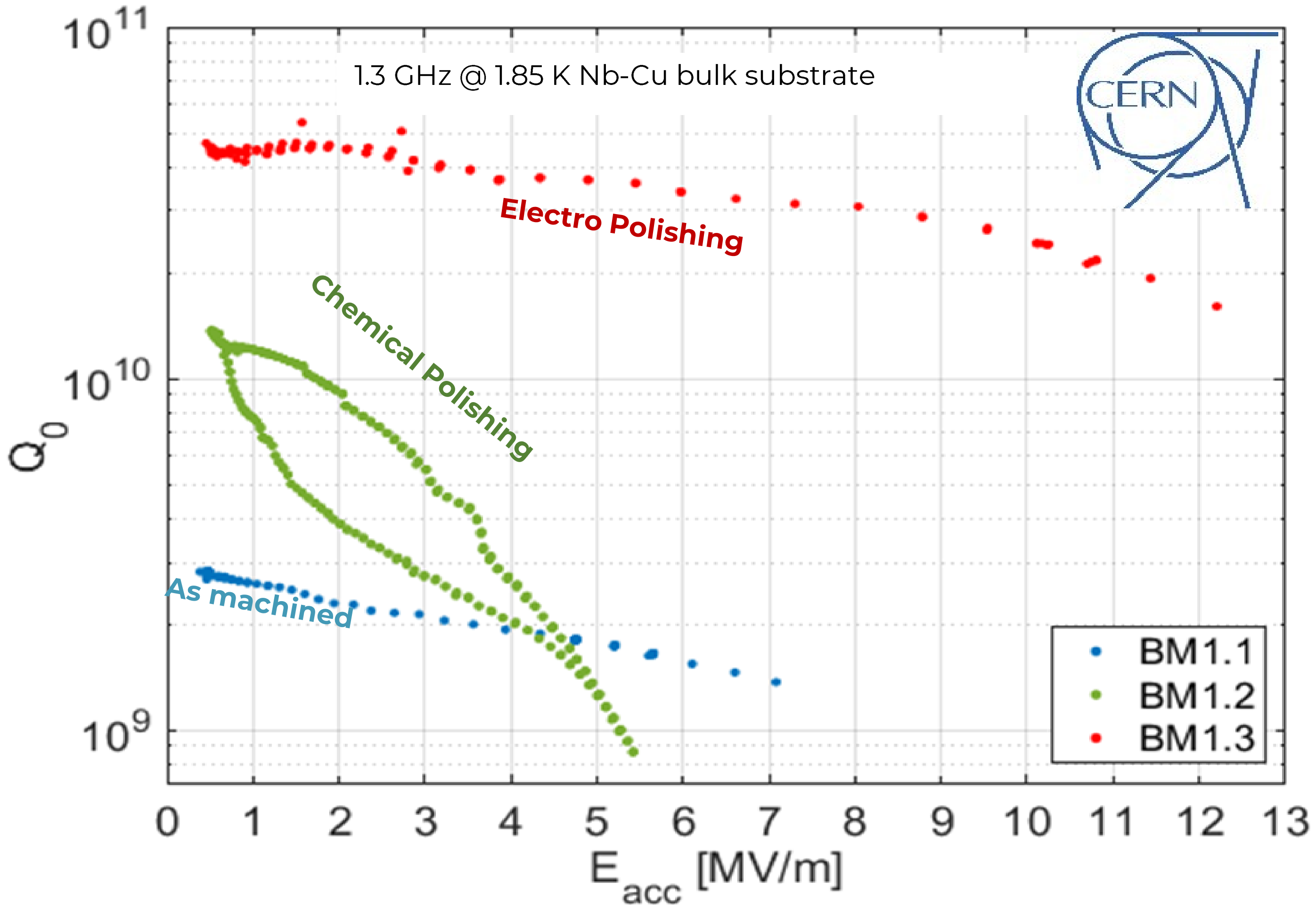
Grain boundaries

Bad thermal contact at the interface

Not intrinsic problem of the films

Substrate is important

Effect of Polishing

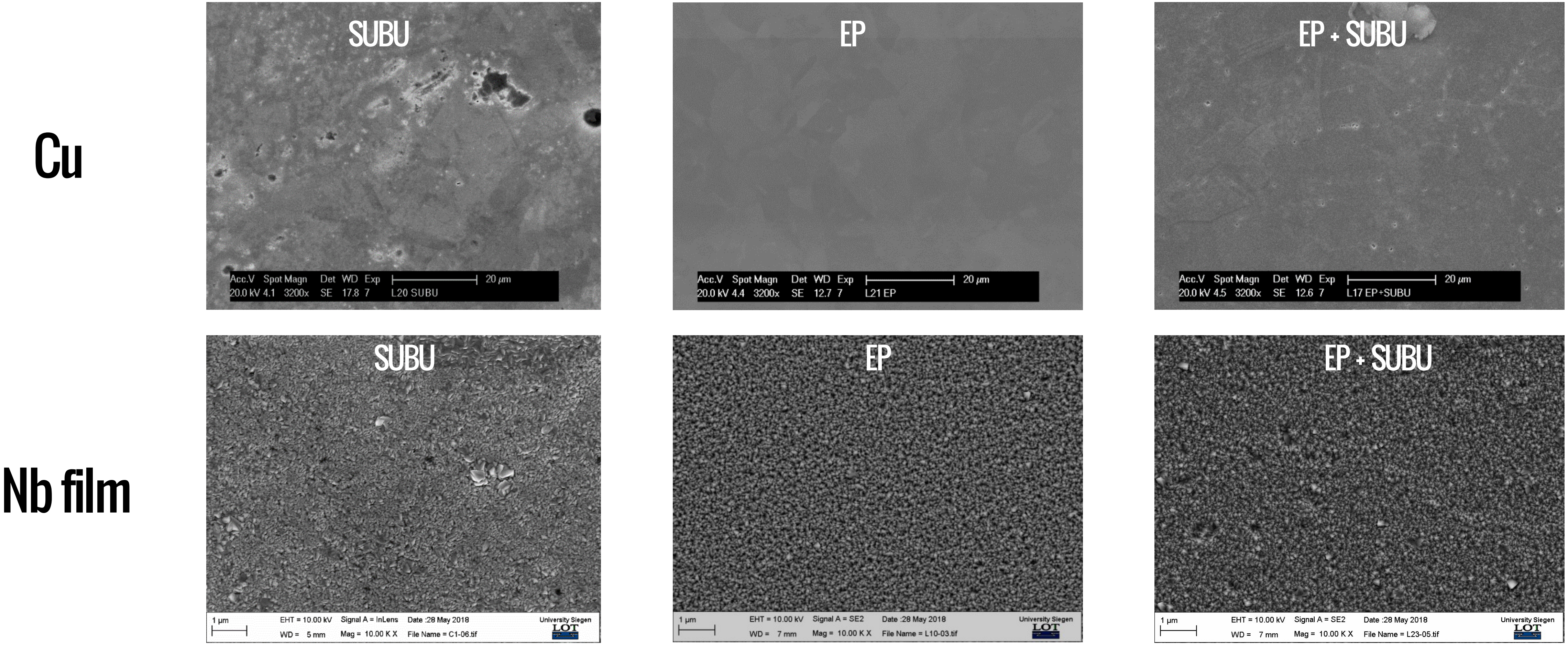


L. Vega Cid, TTC meeting 2022 (elaborated)

Cu substrate plays a fundamental role in SRF performances

Roughness and defects reduction by **surface treatments are mandatory** for a good and uniform SRF coating

Effect of Polishing



PVD film mimate the surface morphology

Pira et al., SRF 2018

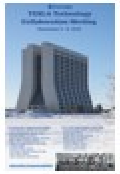
INFN
LNL

Cristian **Pira**
On behalf of SRF LNL Group

Plasma Electrolytic Polishing

TTC meeting 2023
Fermilab, 5 December 2023

TESLA TECHNOLOGY
T T C
COLLABORATION

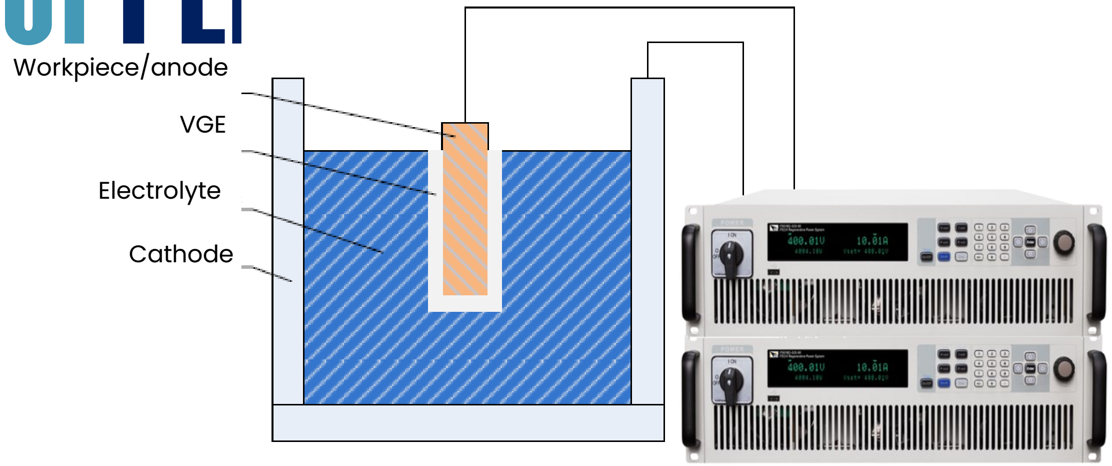


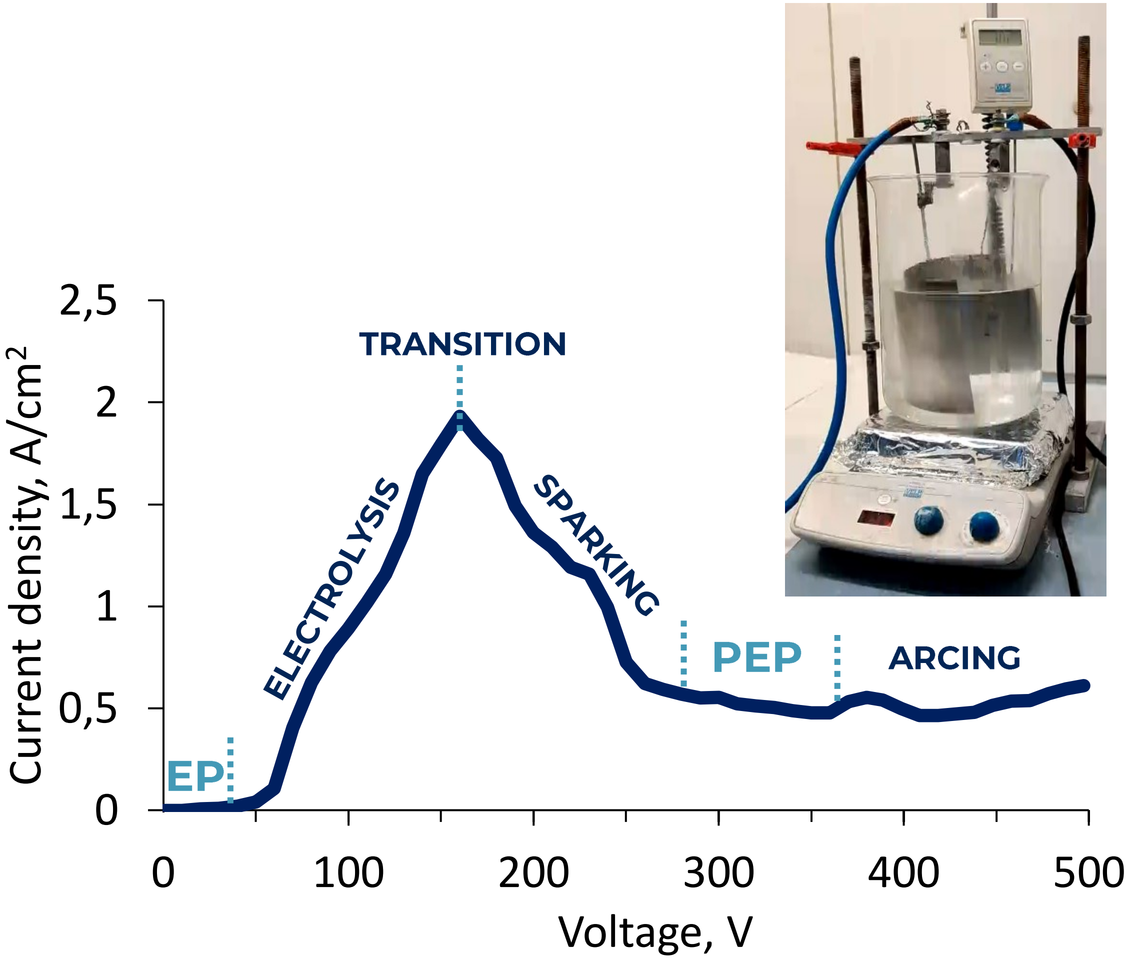
iFAST

SAMARA
INFN CSN5 EXPERIMENT

This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under GA No 101004730.

Basics of PEP

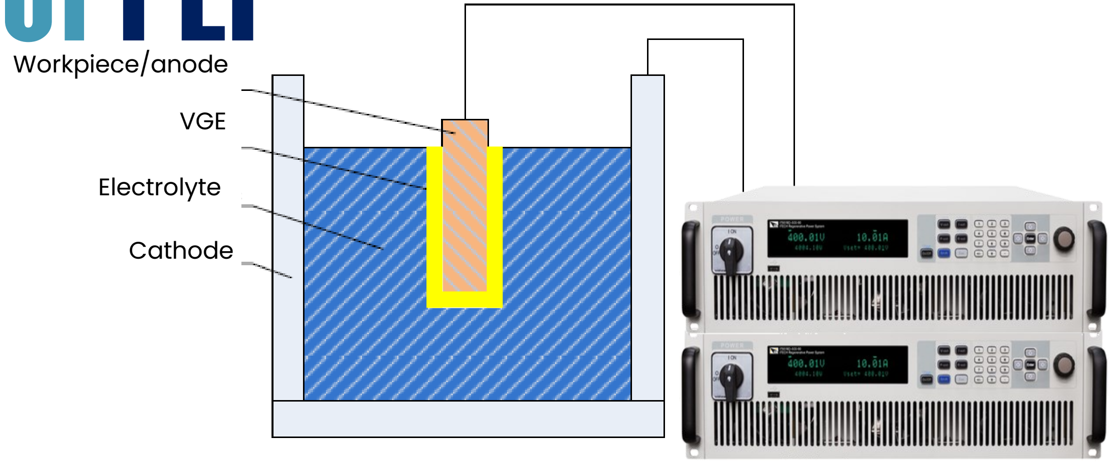


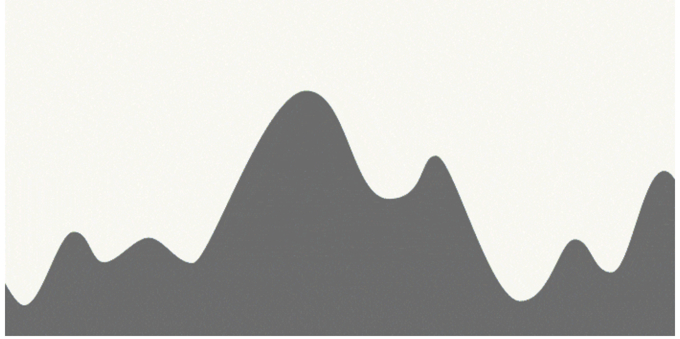


Pira C. et. Al, SRF Proceeding 2021

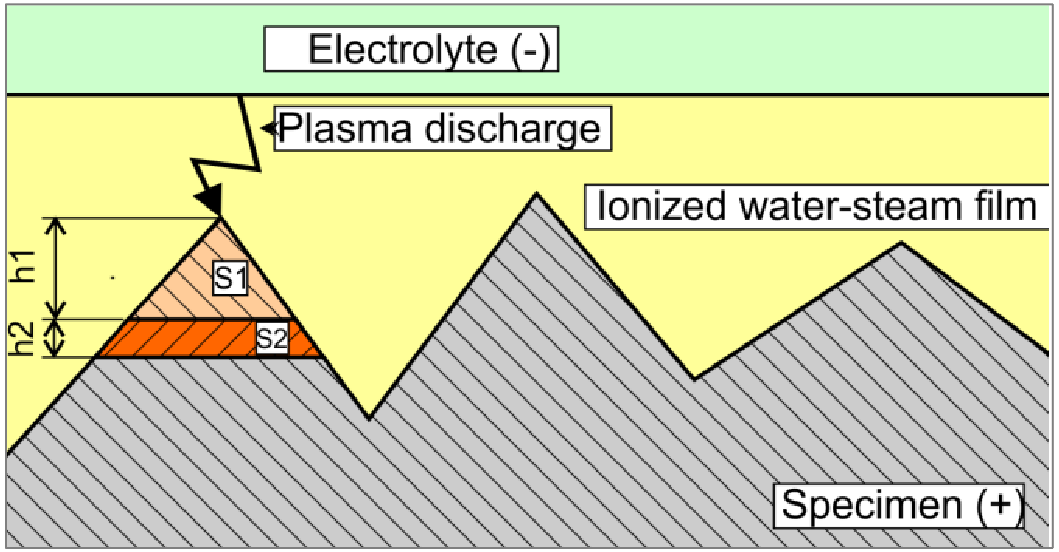
	EP	PEP
Bath	Concentrated acid solutions	Diluted water-salt solutions
Area **chatode: anode**	1:1	10:1
Working voltage	2-25 V	260 – 340 V
Current density	0,03 A/cm^2	0,2-0,8 A/cm^2
Temperature	4-60 C° (lower is better)	60-90 C°

Basics of PEP





Vapor Gas Envelope



Vana, D et. al, Int. J. Mod. Eng. Res. 2013

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PEP Advantages

Green

Diluted water solutions,
enviromentally friendly

Fast

The fastest
non-destructive polishing

Plasma
Electrolytic
Polishing

Equal thickness removal yield
lowest roughness among
competitors

Efficient

Less sensitive to the
cathode shape!
AM compatible

Versatile

PEP is Green

Plasma Electrolytic Polishing

- Lower chemicals cost

Roughly 5x cheaper per 1 L

- Easier storage
- Easier and cheaper wastes proceeding
- Less security risks

No Acids in the chemical bath!
No HF for Nb!

INFN PEP Patented Bath

Ammonium Fluoride NH_4F 2-6 %
Sodium Fluoride NaF 0,5 – 2 %

BCP 1:1:2	EP Nb 1:9	PEP Nb
Quantity of chemicals (w. %)		
79 %	93 %	~5%

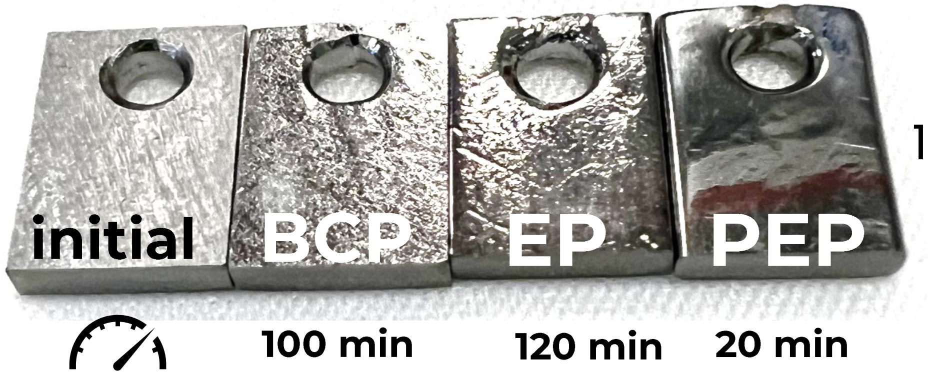
PEP is Faster

Plasma Electrolytic Polishing

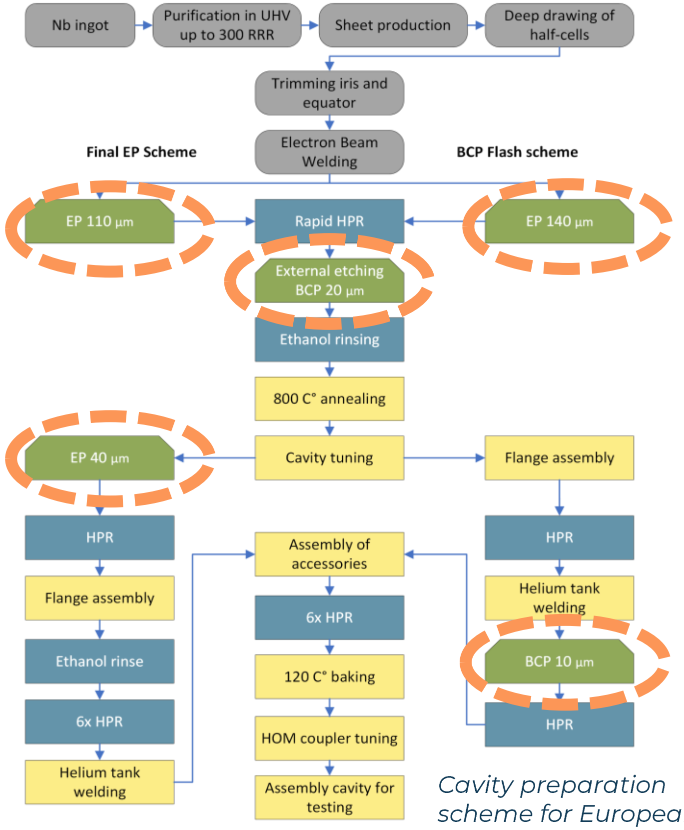
PEP is at least

6x times **faster** than EP!

In cavity mass production it would be huge advantage!



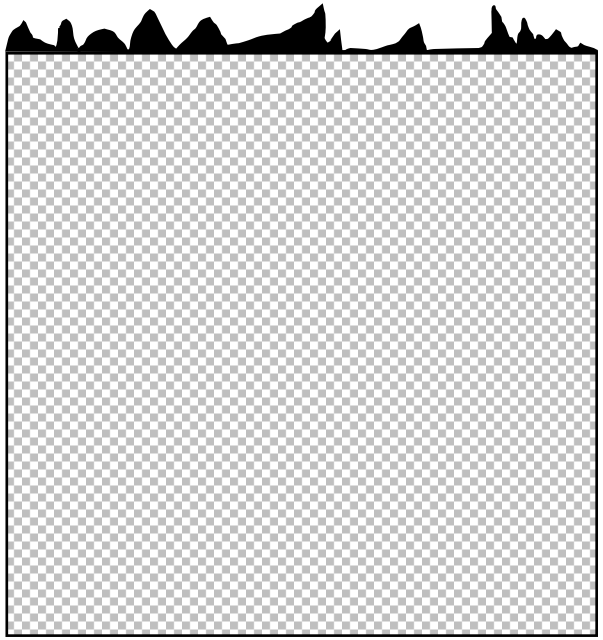
100 µm removed



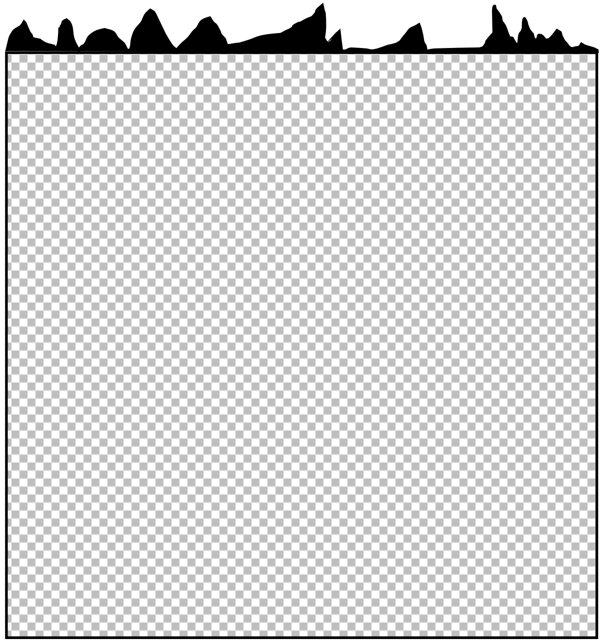
Cavity preparation scheme for European XFEL facility

PEP is Efficient

Plasma
Electrolytic
Polishing



EP

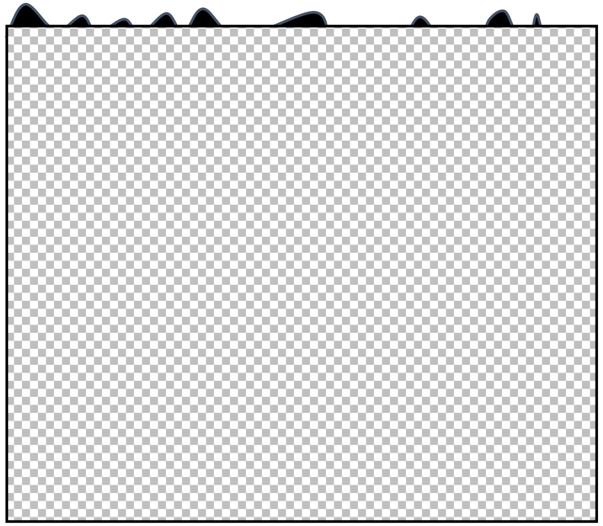


PEP

Removal of equal quantity of materials leads to lower roughness comparing to other treatments

PEP is Efficient

Plasma
Electrolytic
Polishing



EP

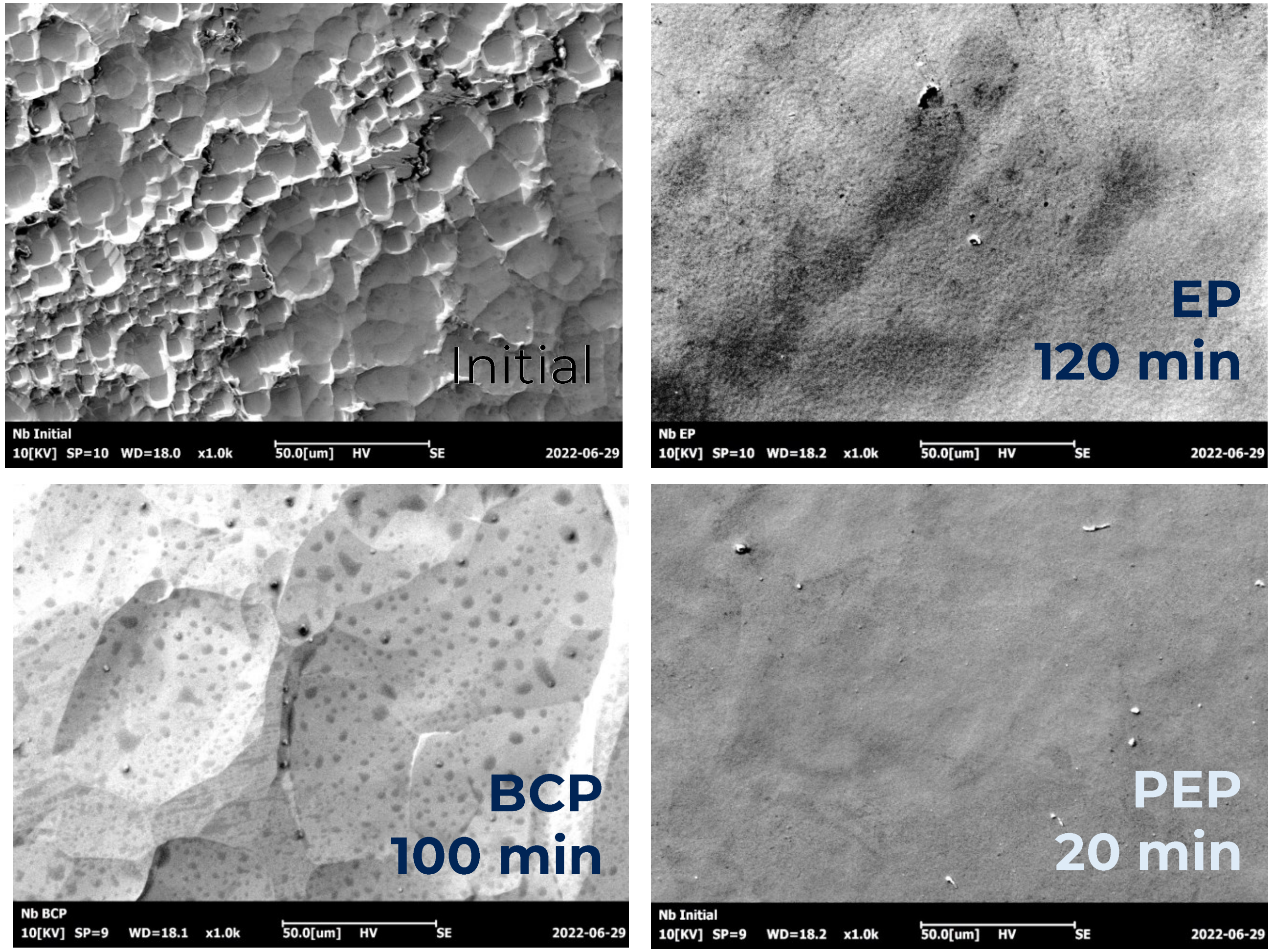
PEP

Removal of equal quantity of materials leads to lower roughness comparing to other treatments

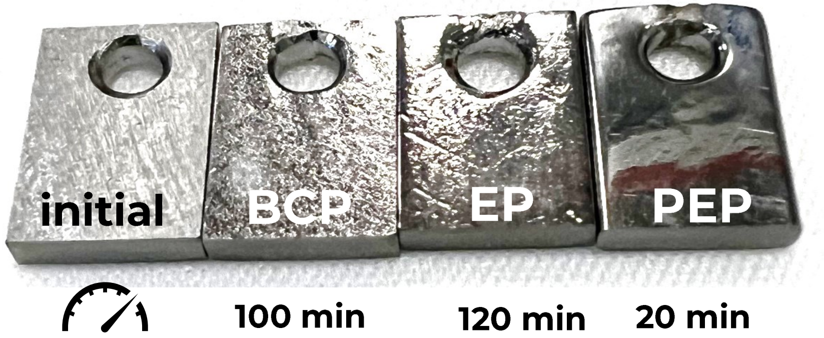
PEP is Efficient

Comparision with EP and BCP

Plasma
Electrolytic
Polishing



Nb, Magnification **1000x**;
100 µm Removal

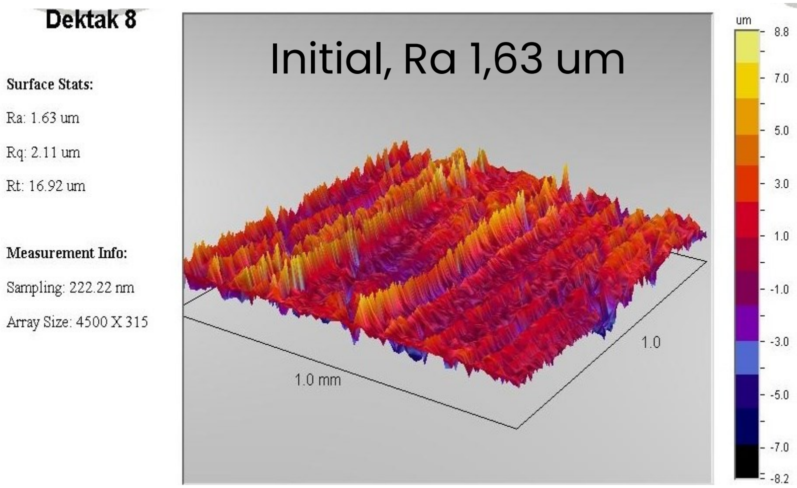


Both micro and macro **roughness is improved significantly**

PEP is Efficient

Comparision with EP and BCP

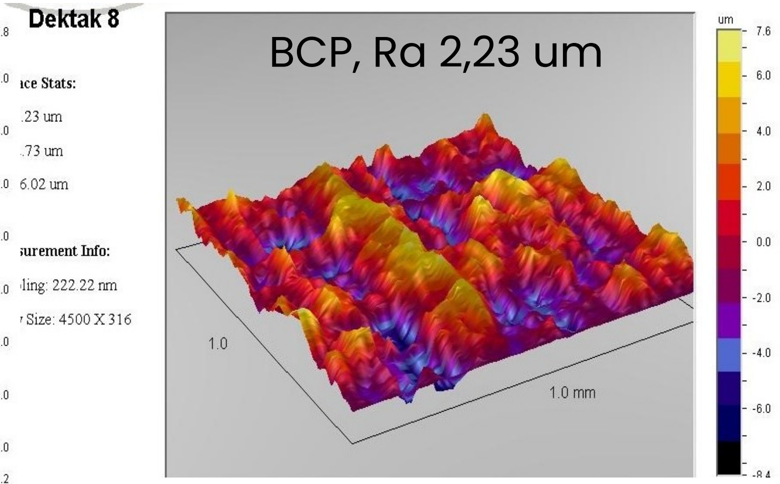
Plasma Electrolytic Polishing


Initial, Ra 1,63 um

Dektak 8

Surface Stats:
Ra: 1.63 um
Rq: 2.11 um
Rt: 16.92 um

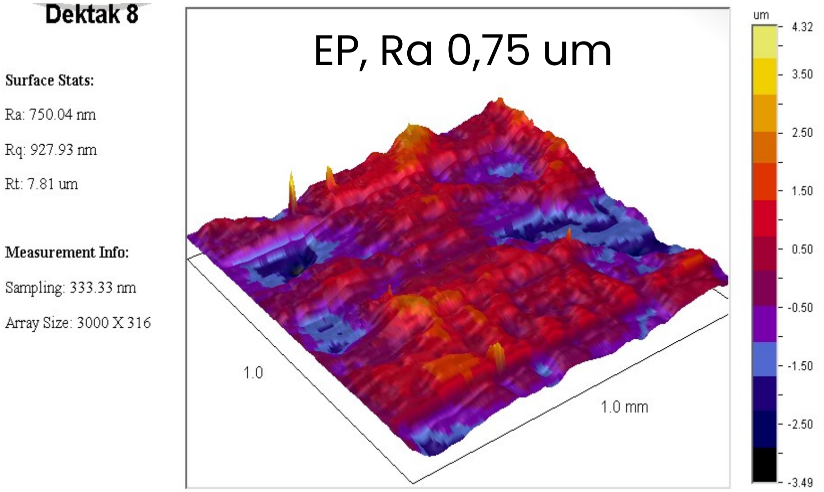
Measurement Info:
Sampling: 222.22 nm
Array Size: 4500 X 315


BCP, Ra 2,23 um

Dektak 8

ıce Stats:
.23 um
.73 um
6.02 um

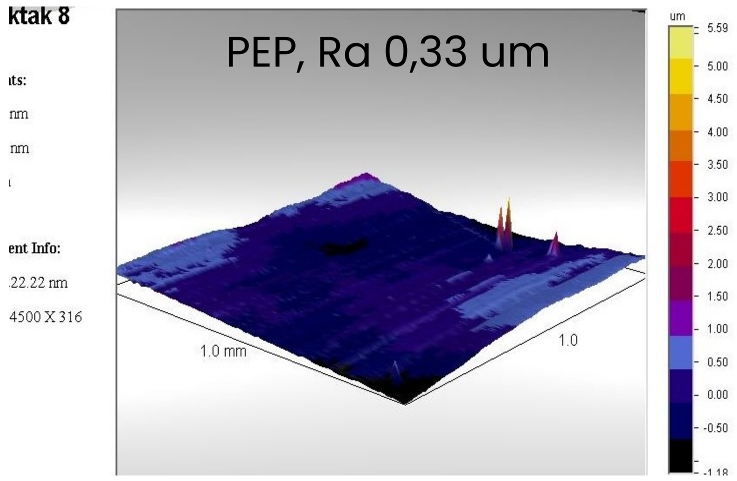
ısurement Info:
ıling: 222.22 nm
ı Size: 4500 X 316


EP, Ra 0,75 um

Dektak 8

Surface Stats:
Ra: 750.04 nm
Rq: 927.93 nm
Rt: 7.81 um

Measurement Info:
Sampling: 333.33 nm
Array Size: 3000 X 316

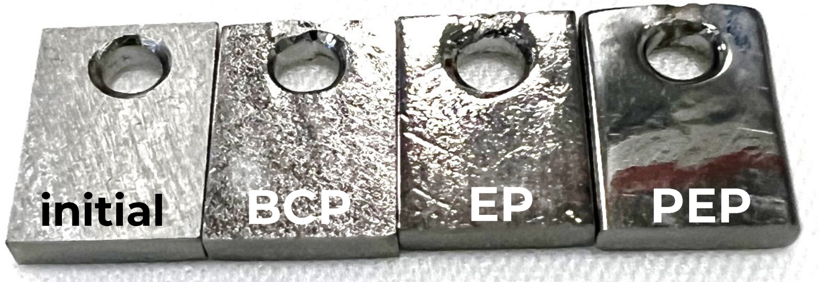

PEP, Ra 0,33 um

ktak 8

ıts:
nm
nm

ent Info:
22.22 nm
4500 X 316

Nb, Magnification **1000x**;
100 μm Removal



100 min 120 min 20 min

Both micro and macro
roughness is improved significantly

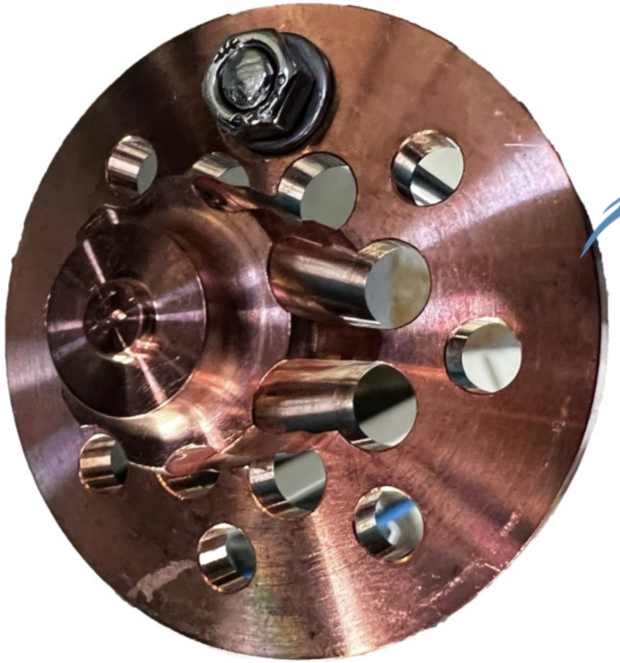
PEP is Efficient

Real Example: Photocathode

In collaboration with DESY.

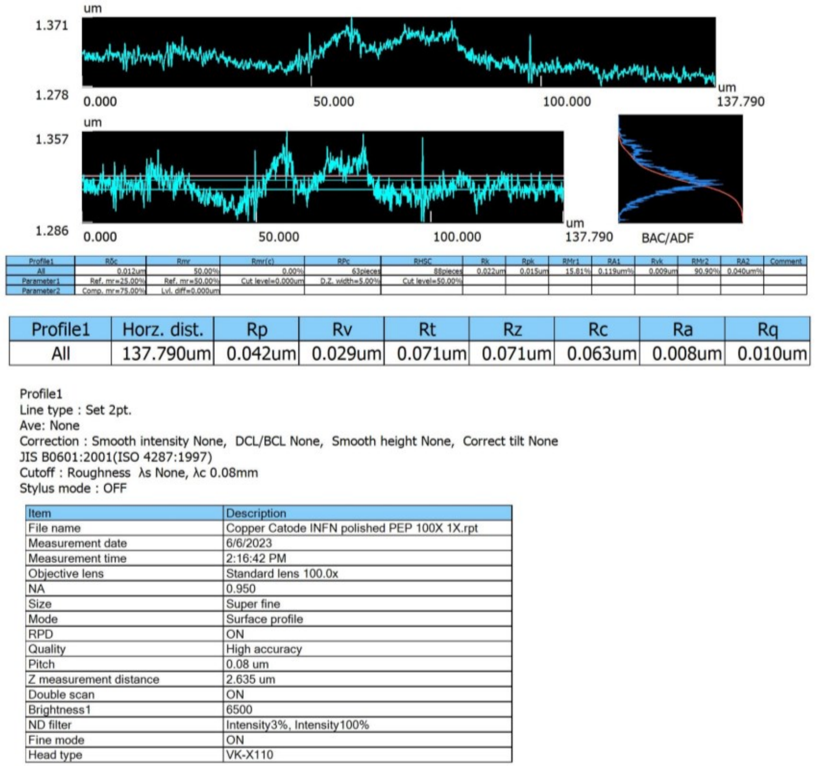
Plasma
Electrolytic
Polishing

Initial



After 4 min PEP

Ra ~ 8 nm!!!



Profile1	Rδc	Rmr	Rmr(c)	RPc	RHSC	Rk	Rpk	RMr1	RA1	Rvk	RMr2	RA2	Comment
All	0.012um	50.00%	0.00%	63pieces	88pieces	0.022um	0.015um	15.81%	0.119um%	0.009um	90.90%	0.040um%	
Parameter1	Ref. mr=25.00%	Ref. mr=50.00%	Cut level=0.000um	D.Z. width=5.00%	Cut level=50.00%								
Parameter2	Comp. mr=75.00%	Lvl. diff=0.000um											

Profile1	Horz. dist.	Rp	Rv	Rt	Rz	Rc	Ra	Rq
All	137.790um	0.042um	0.029um	0.071um	0.071um	0.063um	0.008um	0.010um

Profile1
Line type : Set 2pt.
Ave: None
Correction : Smooth intensity None, DCL/BCL None, Smooth height None, Correct tilt None
JIS B0601:2001(ISO 4287:1997)
Cutoff : Roughness λs None, λc 0.08mm
Stylus mode : OFF

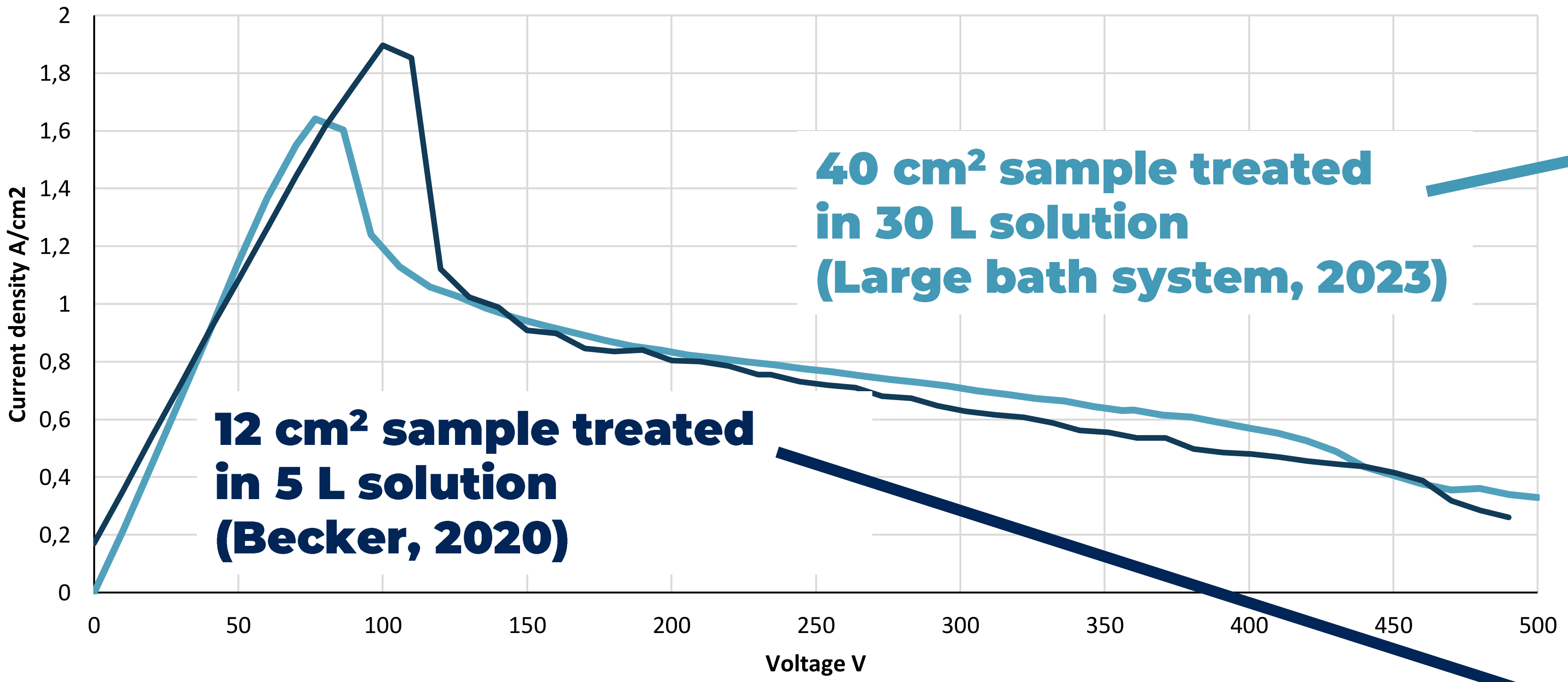
Item	Description
File name	Copper Catode INFN polished PEP 100X 1X.rpt
Measurement date	6/6/2023
Measurement time	2:16:42 PM
Objective lens	Standard lens 100.0x
NA	0.950
Size	Super fine
Mode	Surface profile
RPD	ON
Quality	High accuracy
Pitch	0.08 um
Z measurement distance	2.635 um
Double scan	ON
Brightness1	6500
ND filter	Intensity3%, Intensity100%
Fine mode	ON
Head type	VK-X110

PEP is Versatile

Scaling to large area

Plasma
Electrolytic
Polishing

Current/Voltage curve Cu;



40 cm² sample treated in 30 L solution (Large bath system, 2023)

12 cm² sample treated in 5 L solution (Becker, 2020)

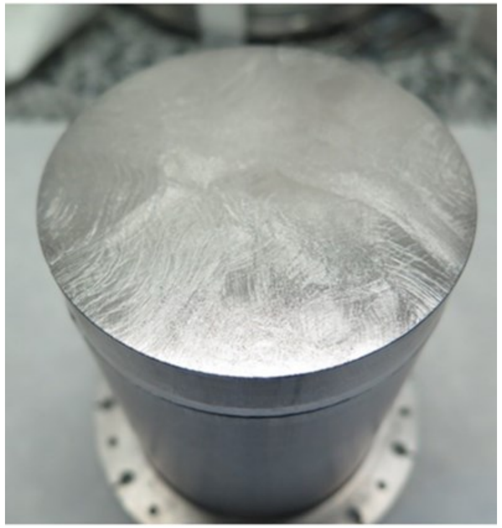
PEP is Versatile

Scaling Nb is a challenge

Plasma
Electrolytic
Polishing

In collaboration with

HZB Helmholtz Zentrum Berlin



Initial
(Bad BCP)

300 V
30 min

First run

300 V
30 min

Second run

PEP is Versatile

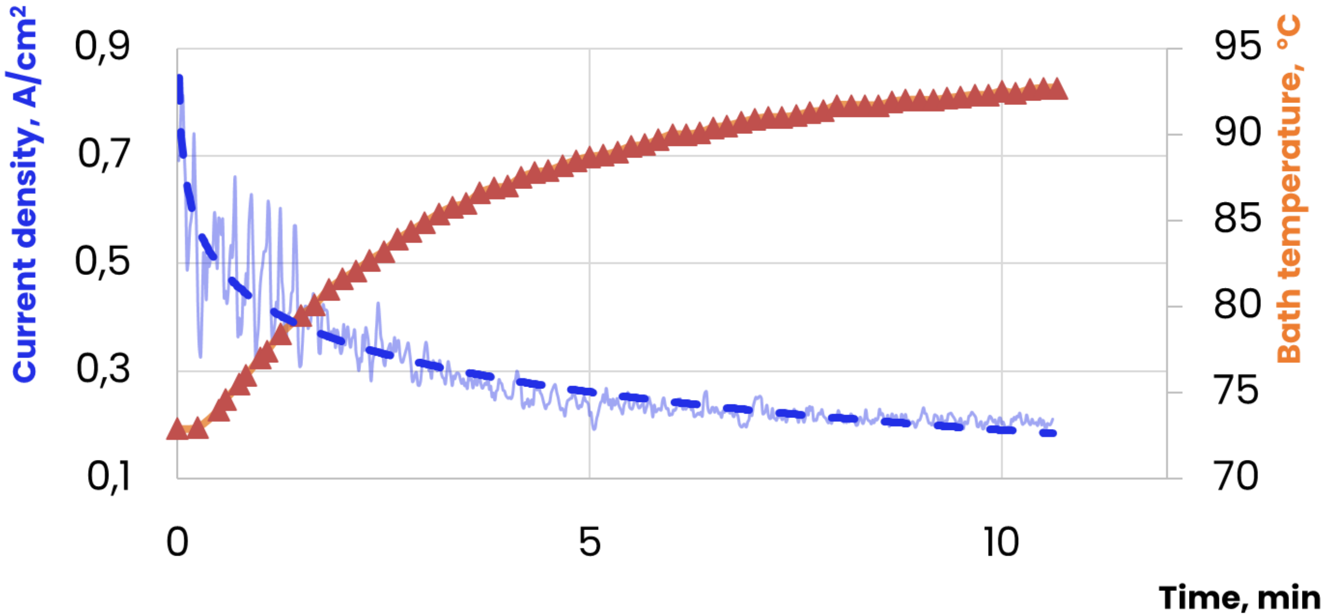
Scaling Nb is a challenge

Plasma
Electrolytic
Polishing

Initial	70 °C	75 °C	85 °C
Average	80 °C	85 °C	88 °C

TFSRF'22 Chyhyrynets et.al.

Current density is inversally proportional to Temperature



Temperature Gradients must be avoided

PEP is Versatile

Cu has no scaling problem

Plasma
Electrolytic
Polishing

The solution used for Cu PEP is **SUBU5**

Double effect: PEP+Chemical Polishing

In collaboration with
HZB Helmholtz Zentrum Berlin

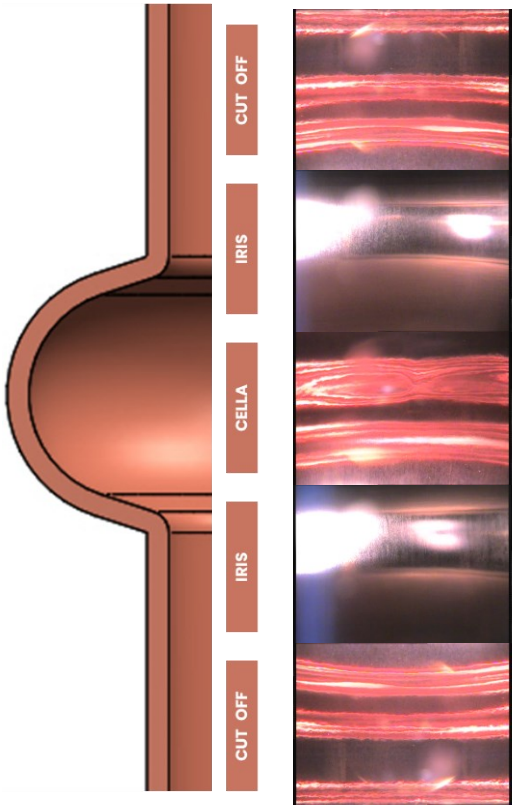
93

PEP is Versatile

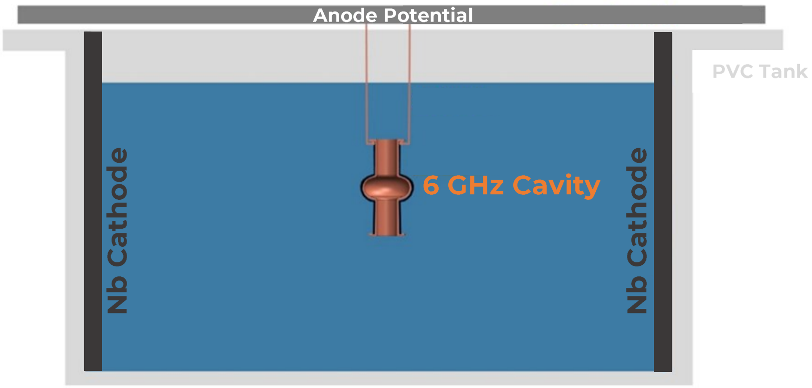
Plasma
Electrolytic
Polishing

Cu 6 GHz cavity succesfully polished

70 µm removed in 10 minutes
30 A (100 cm^2 → 1.3 GHz ~ 300 A)



No internal cathode,
Only external cathode!



Conclusions

- **PEP** is a promising **alternative** polishing technique **for SRF**
- **Greener, Faster, More Efficient** and versatile than EP and BCP
- **Scaling Nb** PEP to large area **is challenging** (Temperature gradients must be avoided)
- **PEP on elliptical cavity** geometry **proved** on Cu

Effect of the Cu substrate forming process

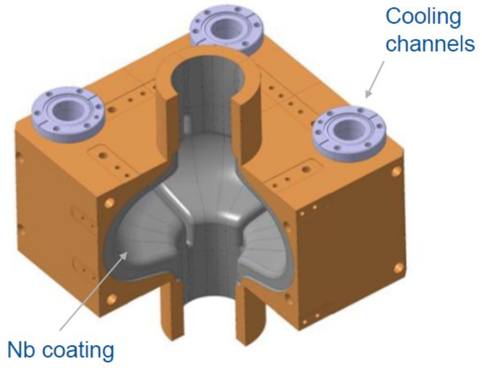
Cu substrate plays a fundamental role in SRF performances

Cavity fabrication

Different possibilities:

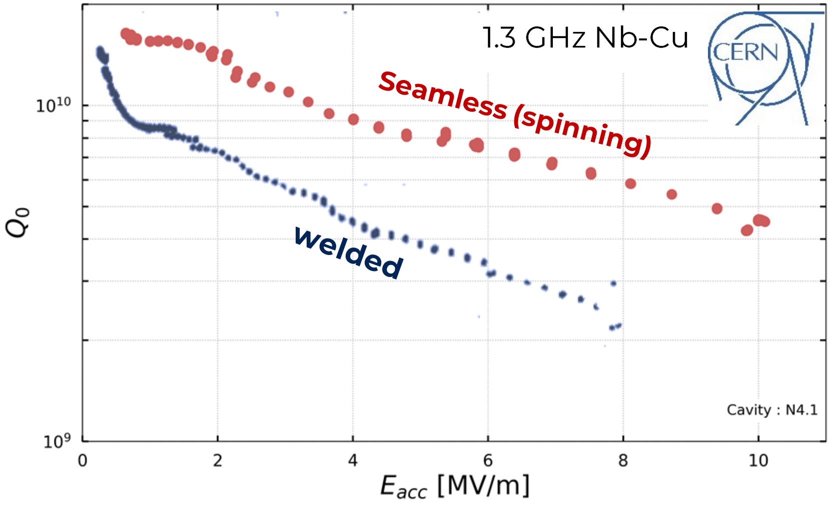
Welding/seamless

Spinning, hydroforming, electroforming…



SWEEL cavity

Simpler coating procedure

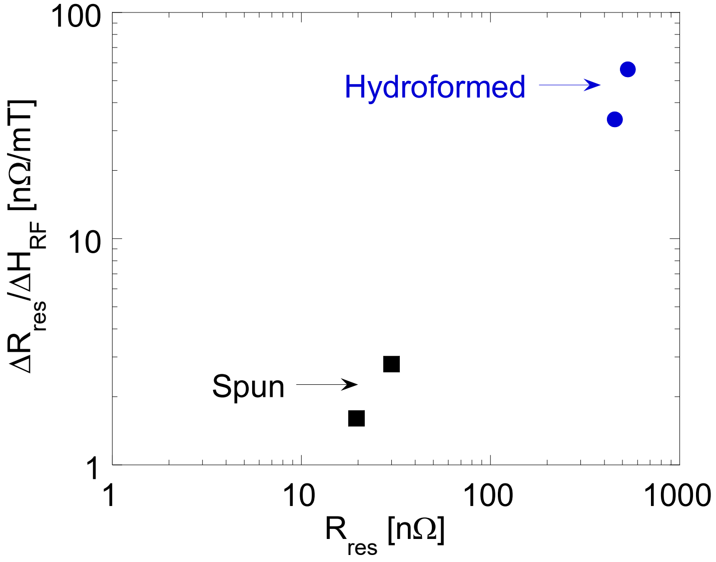


L. Vega Cid, TTC meeting 2022 (elaborated)

Different proofs of **seamless** RF performances **superiority**

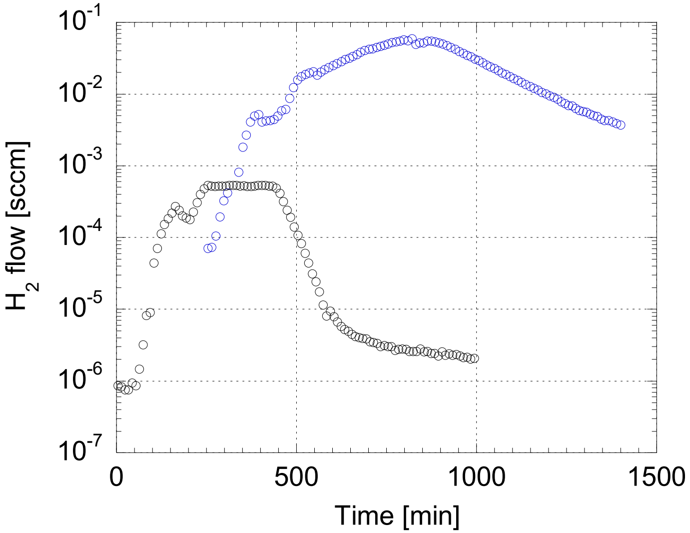
(Hie-ISOLDE, ALPI-INFN, CERN studies, …)

Effect of the Cu substrate forming process



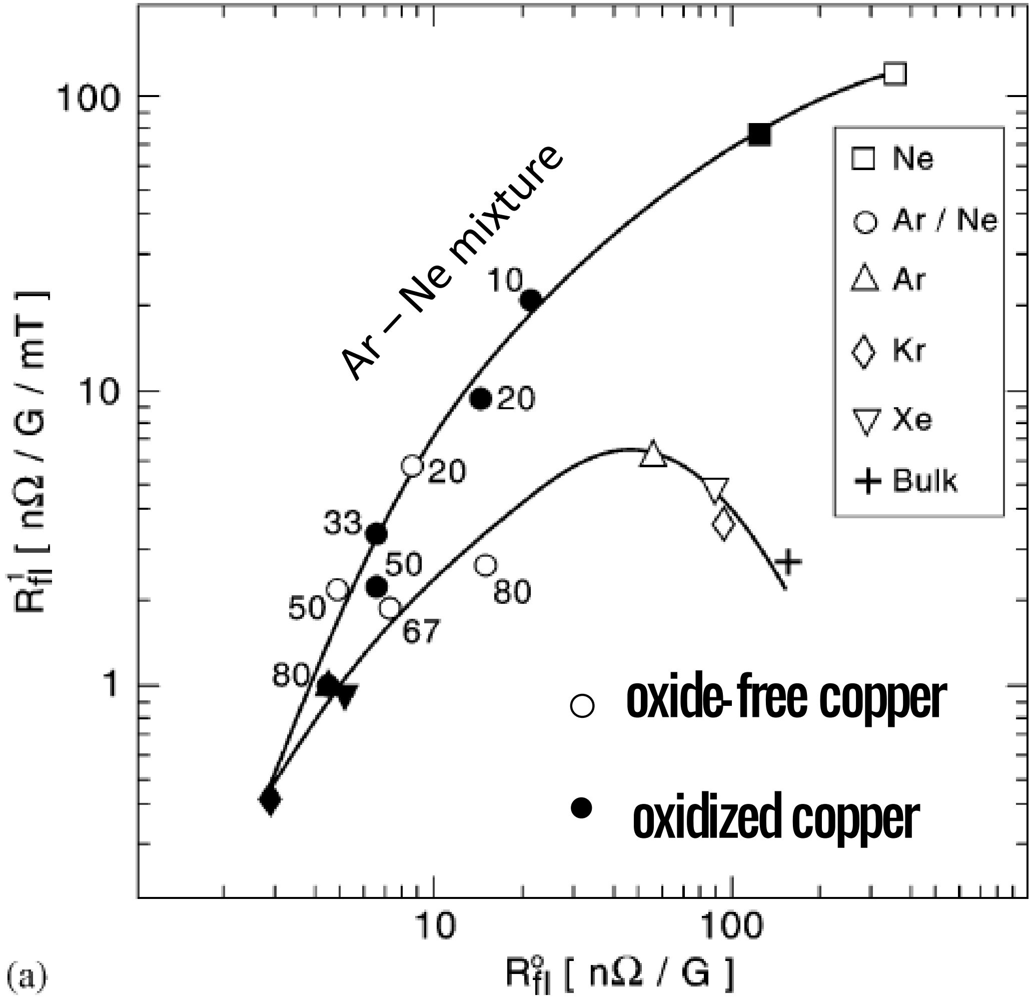
Coatings on oxide-free **hydroformed** cavities consistently **worse than** for **spun cavities**? Why?

Possible answer: a larger quantity of **hydrogen** was migrating into the film from the hydroformed cavity



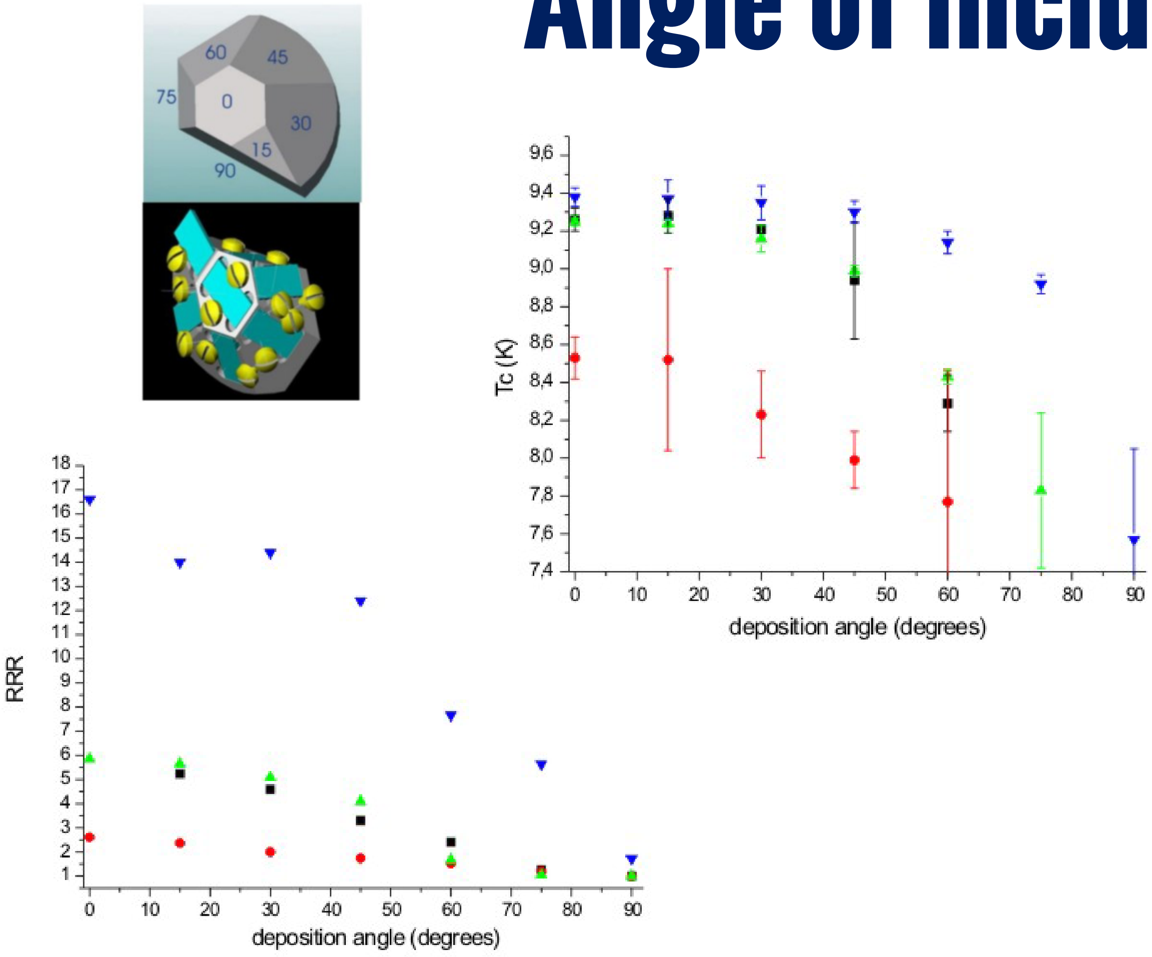
S. Calatroni (CERN), SRF 2001

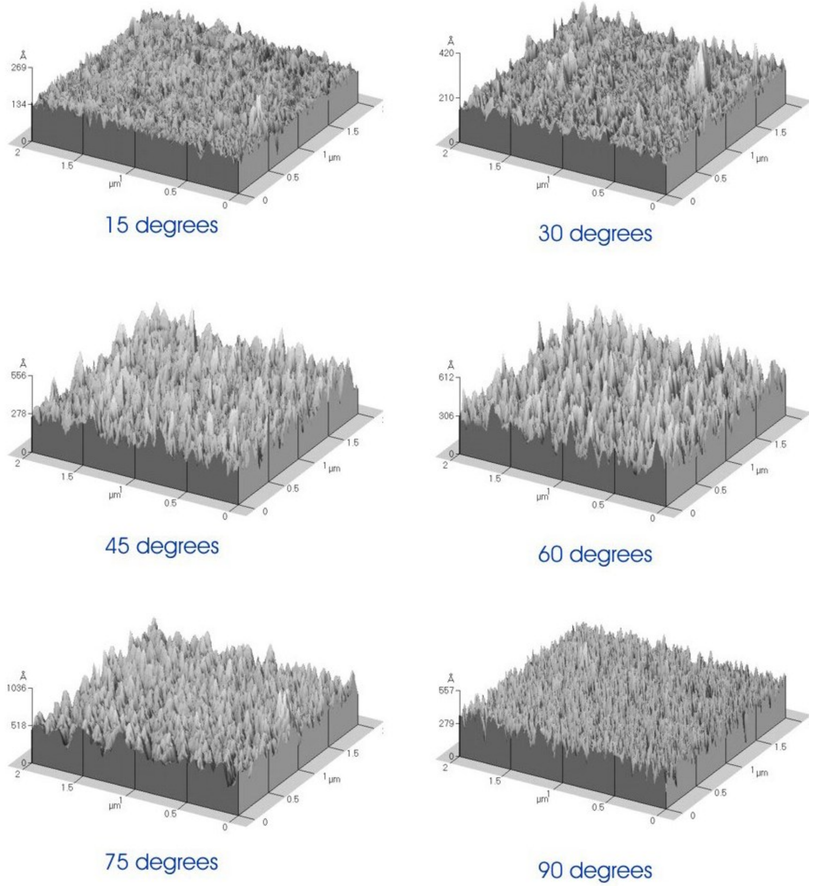
Effect of the gas



S. Calatroni (CERN), SRF 2001

Angle of incidence of coating





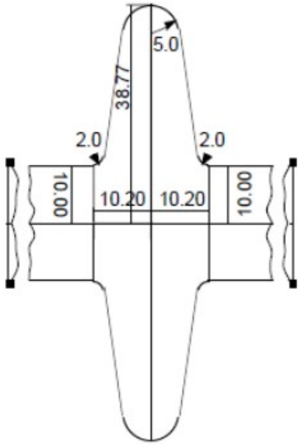
Superconducting properties of niobium films depends on deposition angle between target and substrate

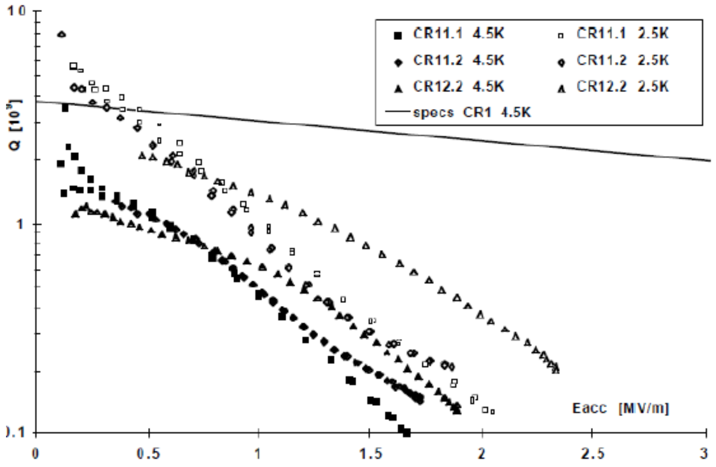
The effect is related to change in the coating morphology

D. Tonini et al, Morphology of niobium films sputtered at different target-substrate angle, SRF99, THP11

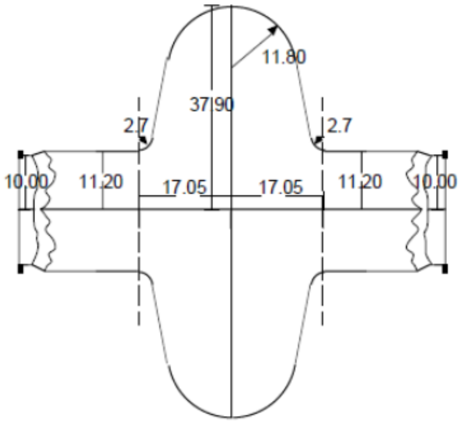
Angle of incidence of coating

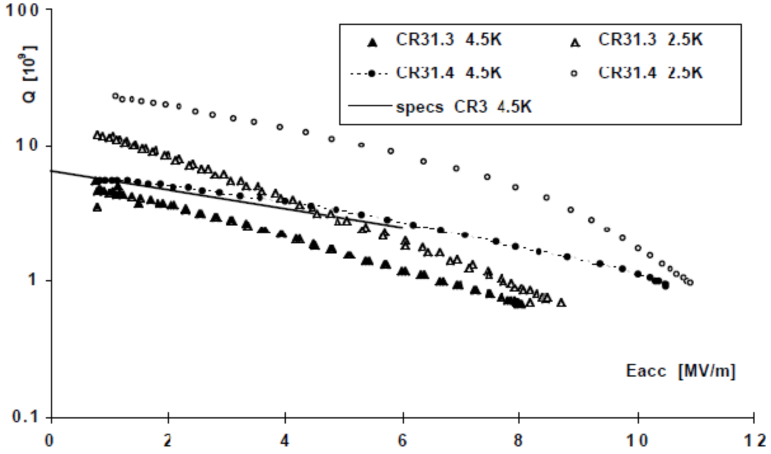
β = 0.48





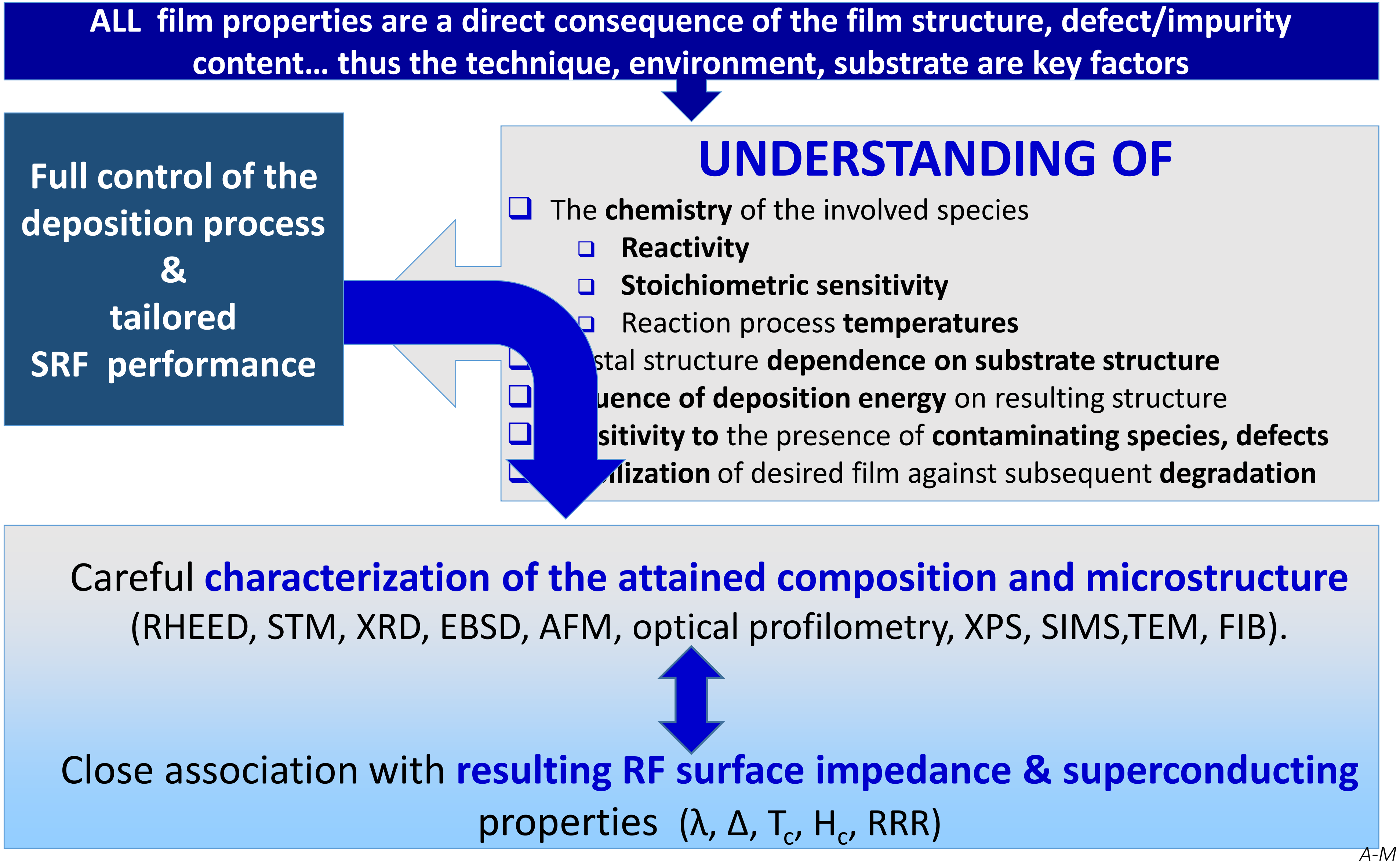
β = 0.8





C. Benvenuti et al, Production and test of 352 MHz Niobium Sputtered Reduced Beta cavities, 1997, SRF97D25

Next generation Nb films



ALL film properties are a direct consequence of the film structure, defect/impurity content… thus the technique, environment, substrate are key factors

Full control of the deposition process
&
tailored
SRF performance

UNDERSTANDING OF

- ❑ The **chemistry** of the involved species
  - ❑ **Reactivity**
  - ❑ **Stoichiometric sensitivity**
  - ❑ Reaction process **temperatures**
- ❑ stal structure **dependence on substrate structure**
- ❑ **uence of deposition energy** on resulting structure
- ❑ **sitivity to** the presence of **contaminating species, defects**
- ❑ **ilization** of desired film against subsequent **degradation**

Careful **characterization of the attained composition and microstructure** (RHEED, STM, XRD, EBSD, AFM, optical profilometry, XPS, SIMS,TEM, FIB).

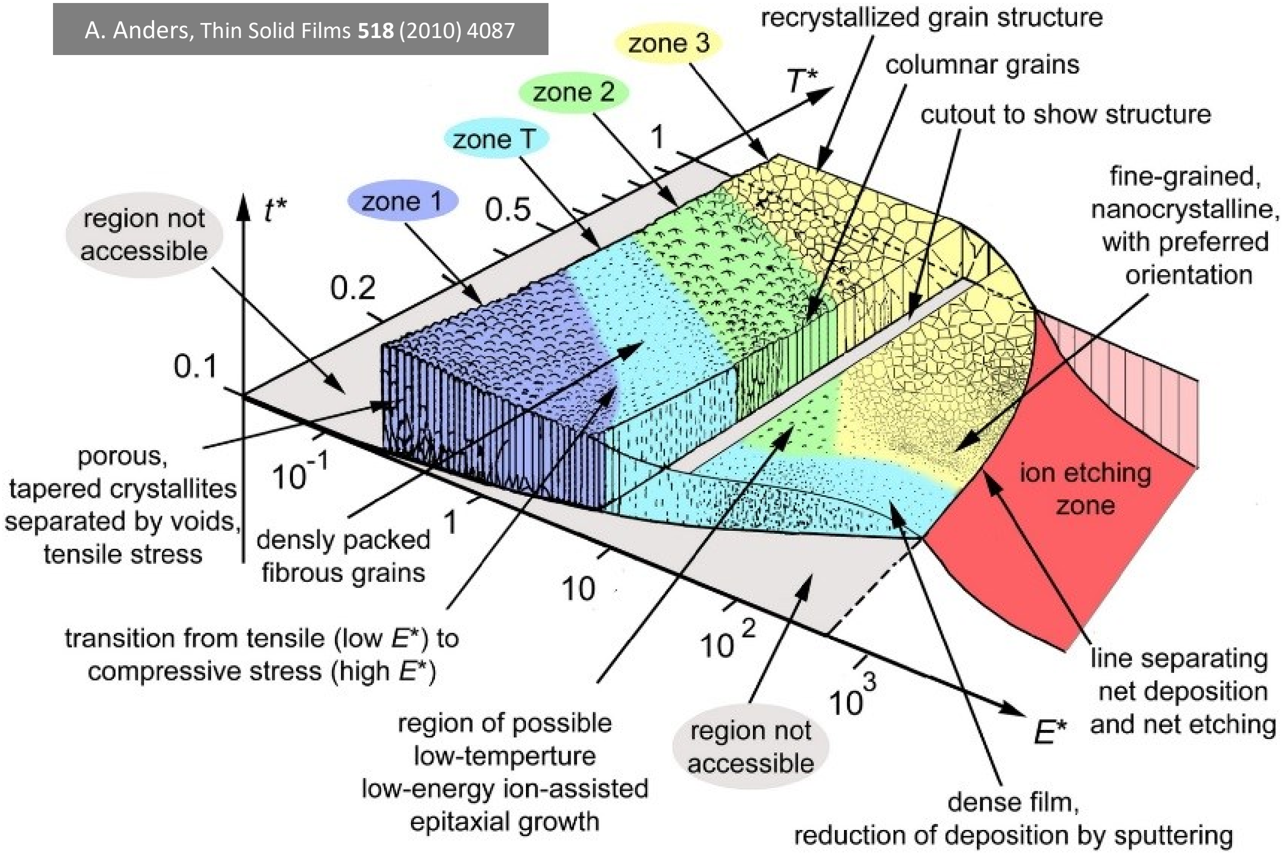
Close association with **resulting RF surface impedance & superconducting** properties (λ, Δ, T_c, H_c, RRR)

A-M Valente, SRF2017 Tutorials

Energetic Condensation

Generalized Structure Zone Diagram

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



Additional energy provided by fast particles arriving at a surface:

- residual gases desorbed from the substrate surface
- chemical bonds may be broken and defects created thus affecting nucleation processes & film adhesion
- enhanced mobility of surface atoms

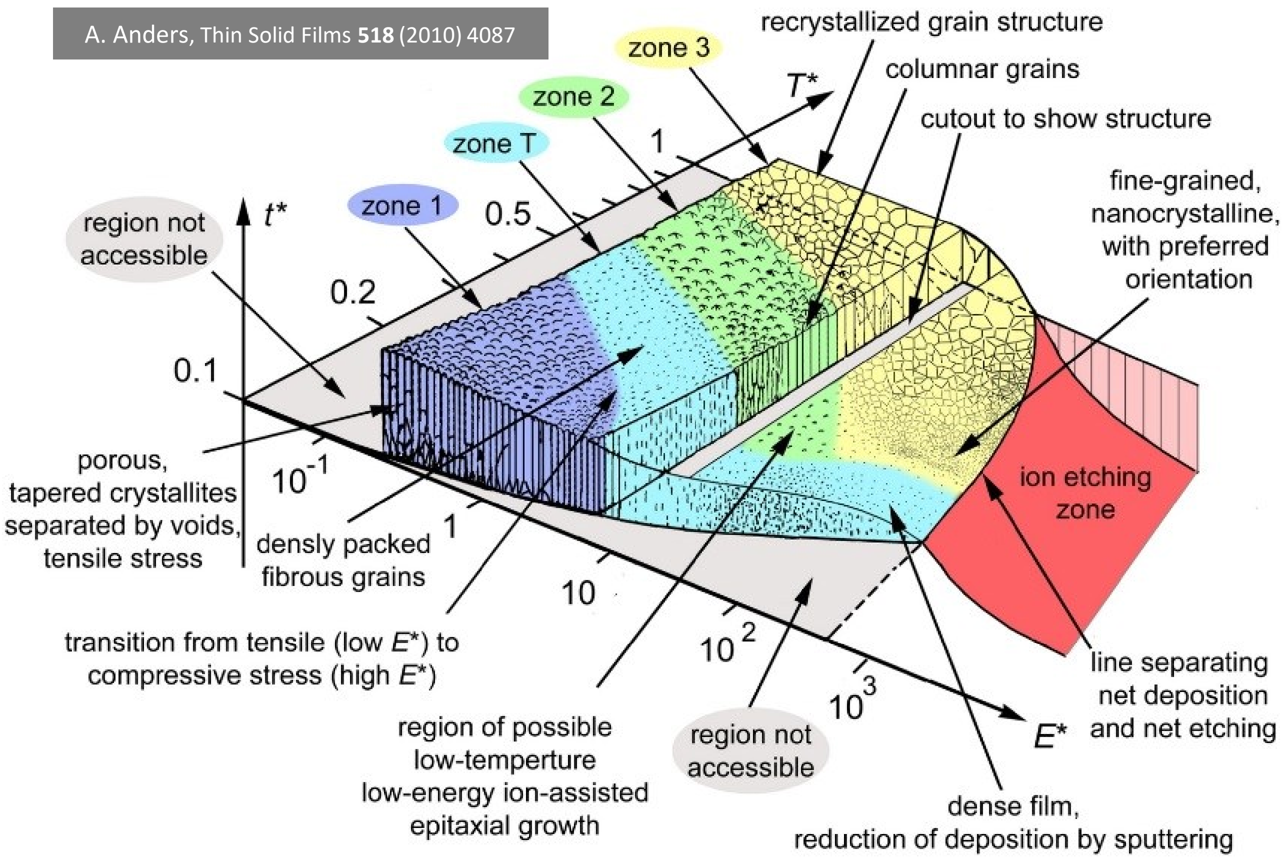
Changes & control in:

- Film density
- morphology
- microstructure
- Stress
- low-temperature epitaxy

A-M Valente, SRF2017 Tutorials

Energetic Condensation

Generalized Structure Zone Diagram


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

A variety of techniques with distinct technologies

- Vacuum Arc Plasma & Coaxial Energetic Deposition (CED)
- Electron cyclotron Resonance (ECR)
- High Impulse Power Magnetron sputtering (HiPIMS)

A-M Valente, SRF2017 Tutorials

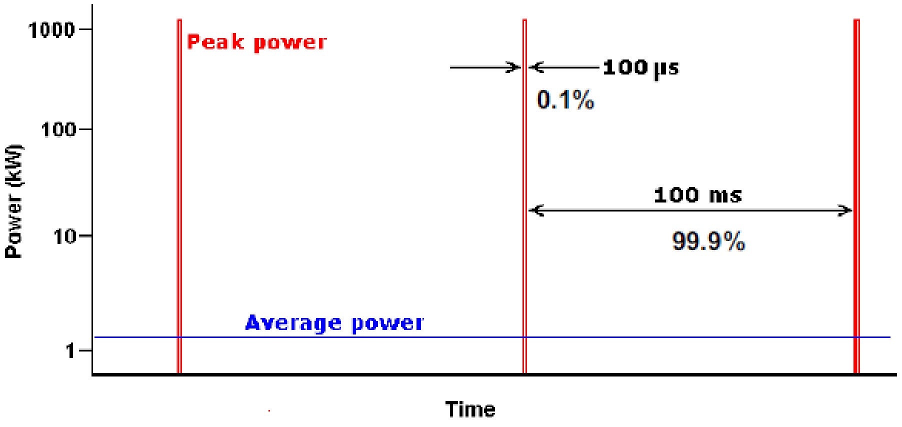
HiPIMS

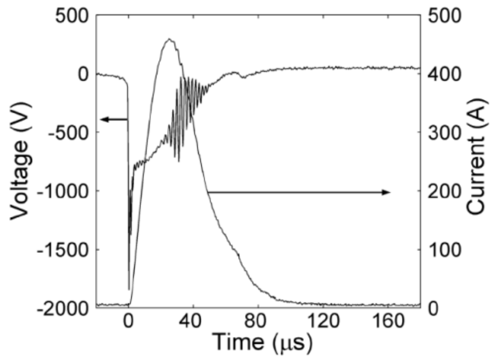
CERN (G. Rosaz et al.)
Jefferson Lab (A-.M. Valente et al.)
STFC ASTeC (R. Valizadeh et al.)
Siegen University (M. Vogel et al.)

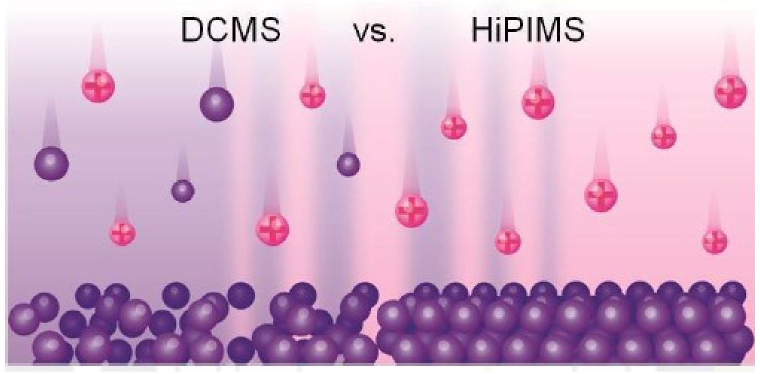
Lawrence Berkeley National Laboratories (A. Anders et al.)

HiPIMS

- **Pulsed sputtering** where the peak power exceeds the average power by typically two orders of magnitude
- The target material is **partially ionized**
- Large concentration of ions producing **high-quality homogeneous films**
- Possibility to self sustain discharge







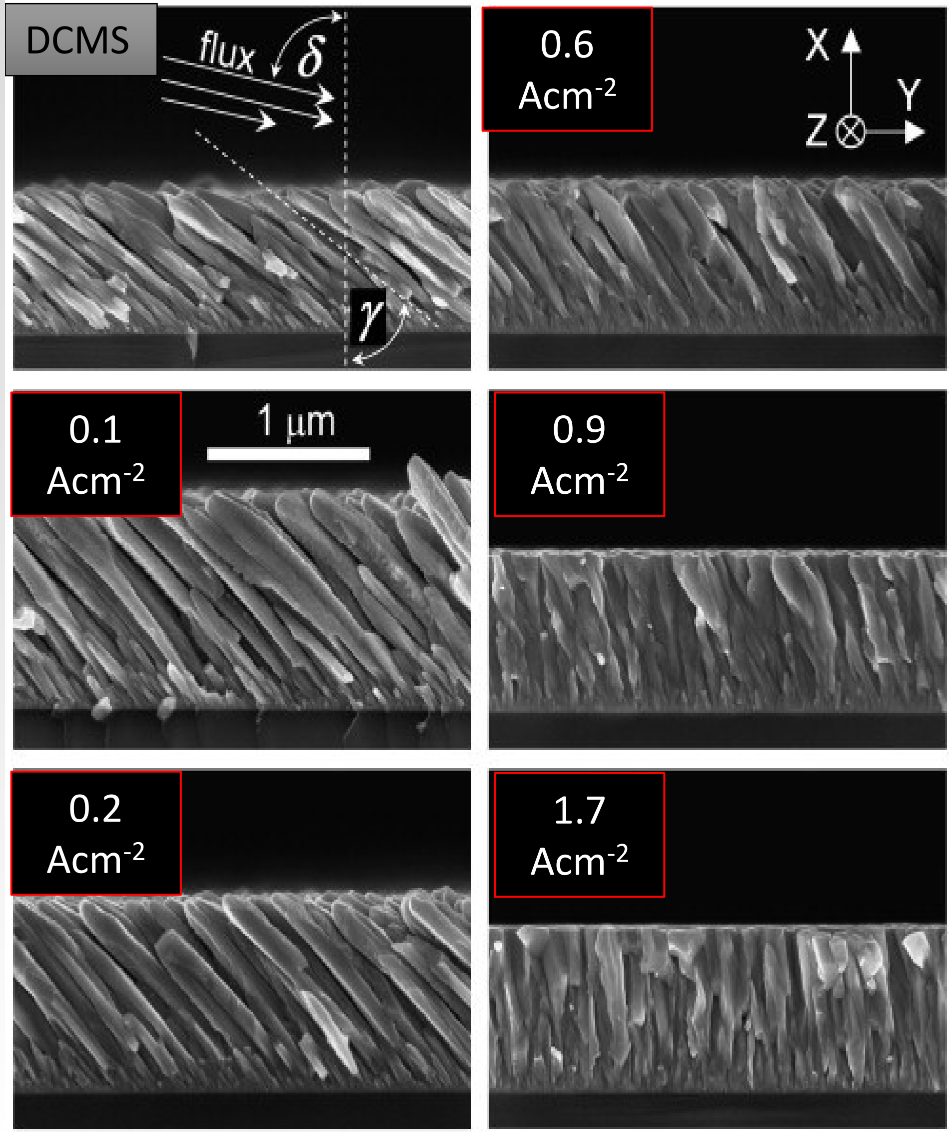
Very high purity
Excellent adhesion
better (normal) conductivity,
Large crystal grains, low defect density
Suppression of fiber structure
Superior density
Decreased roughness
Homogeneous coating even on complex-shaped surfaces
Phase composition tailoring
Interface engineering

Lower coating rate :
ions captured at the cathode

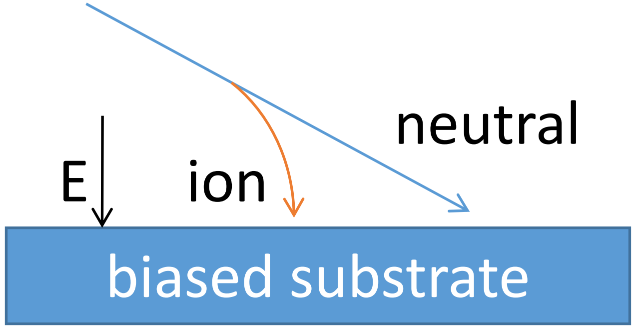
Very sensitive to cathode surface state (roughness), induced arcing

J. Böhlmark et al., J. Vac. Sci. Technol. A 23 (2005) 18

Conformal Coating



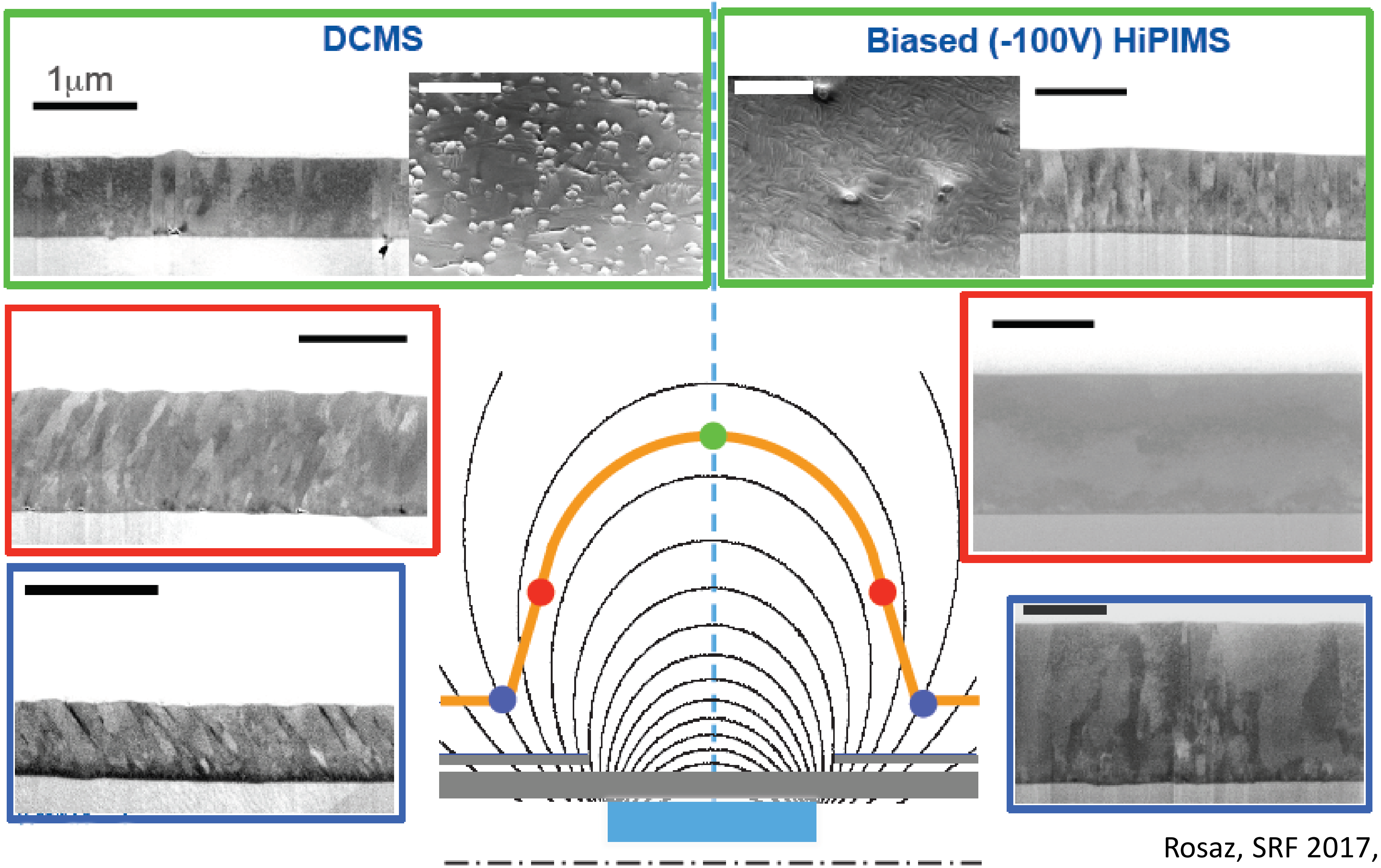
Inclination of columns is reduced at high target current densities due to high ion-to-neutral ratio



CrN Glancing Angle Deposition

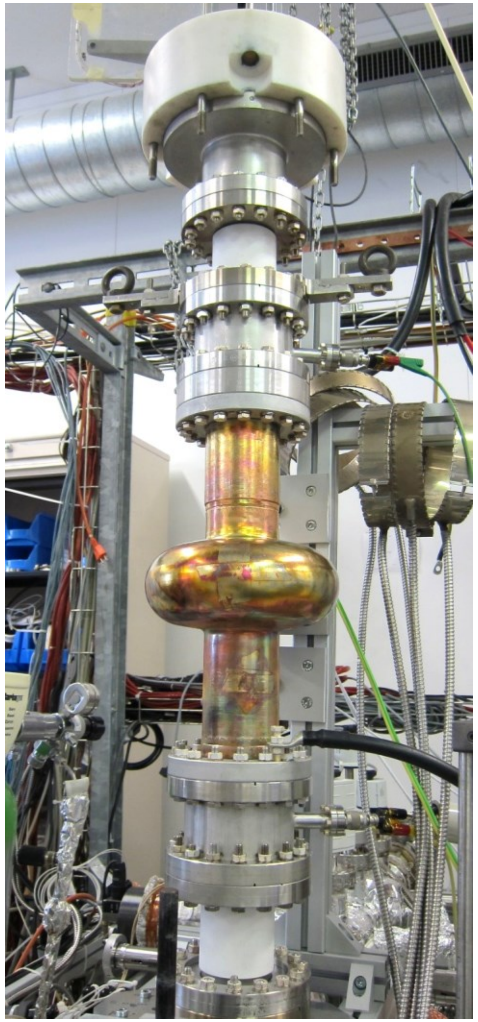
G. Greczynski, *et al.*, Thin Solid Films 519 (2011) 6354.

Conformal HiPIMS @CERN

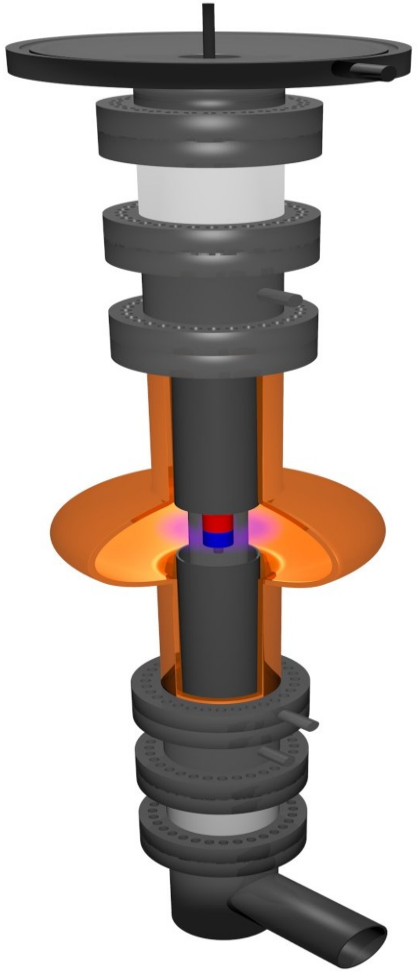


Rosaz, SRF 2017, Lanzhou (China)

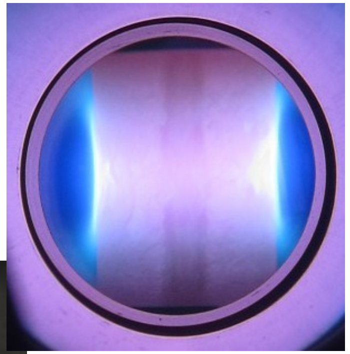
CERN HiPIMS Setup



1.3 GHz cavity coating setup



Nb cathode with permanent magnets inside and Nb anodes



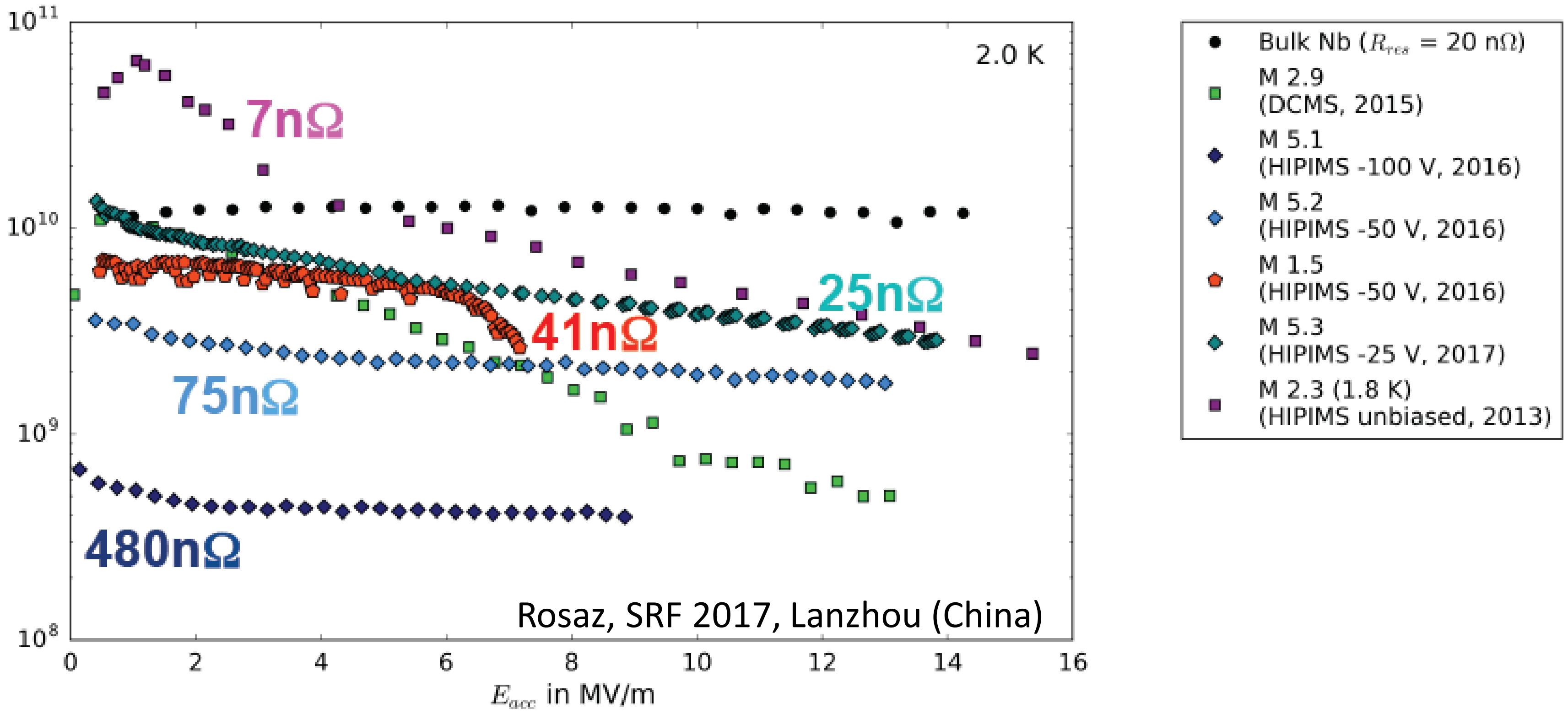
HiPIMS discharge

- **Same hardware as for DCMS**
- Pulsed Power supply
  - 1% duty cycle
  - Short pulses: 200 μs
  - High peak current (200 A vs 3 A for DCMS)
  - High peak power (80 kW peak for 1kW avg)
- Ionization of sputtered species
- Lower coating rate than DCMS

Courtesy of G. Rosaz (CERN)

R&D on Nb films

HiPIMS Results @ CERN

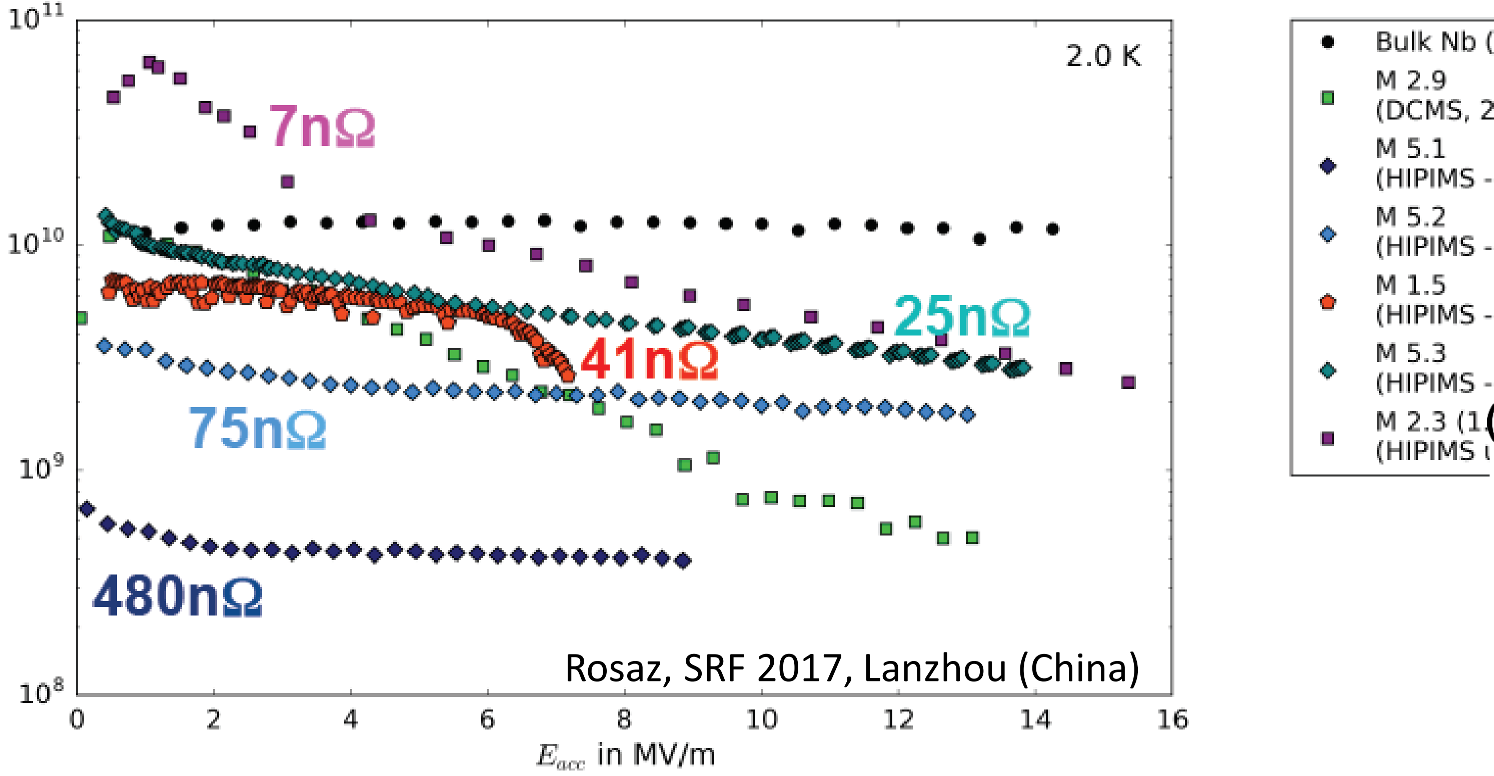


Rosaz, SRF 2017, Lanzhou (China)

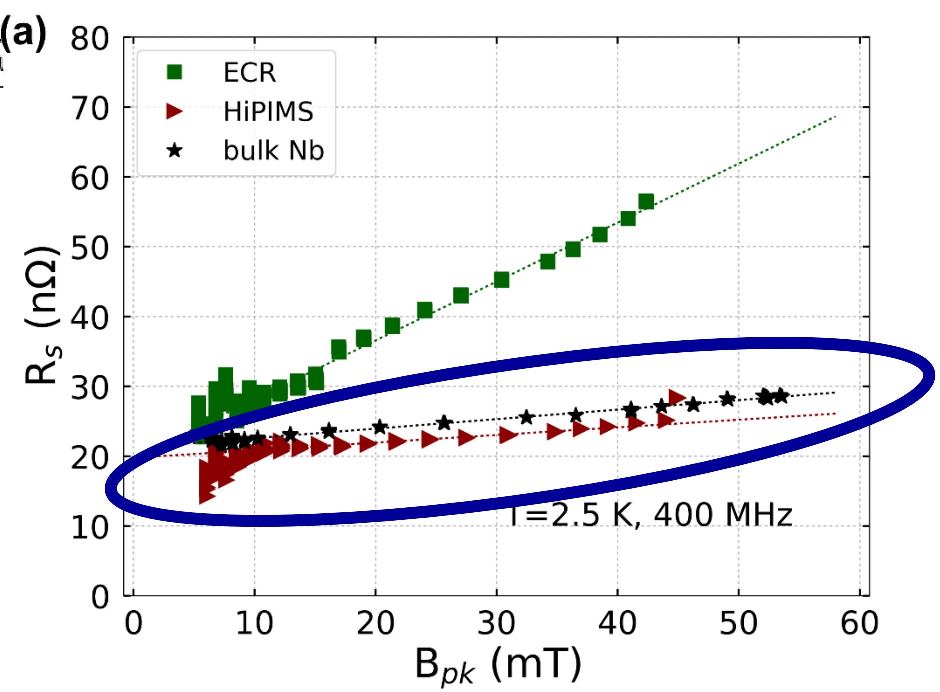
- High Bias does not give good results (gas implantation , stress)
- Lower pressure tends to better performances (contamination, stress)
- **Q-slope looks mitigated vs DCMS coating**

Courtesy of G. Rosaz (CERN)

HiPIMS Results @ CERN

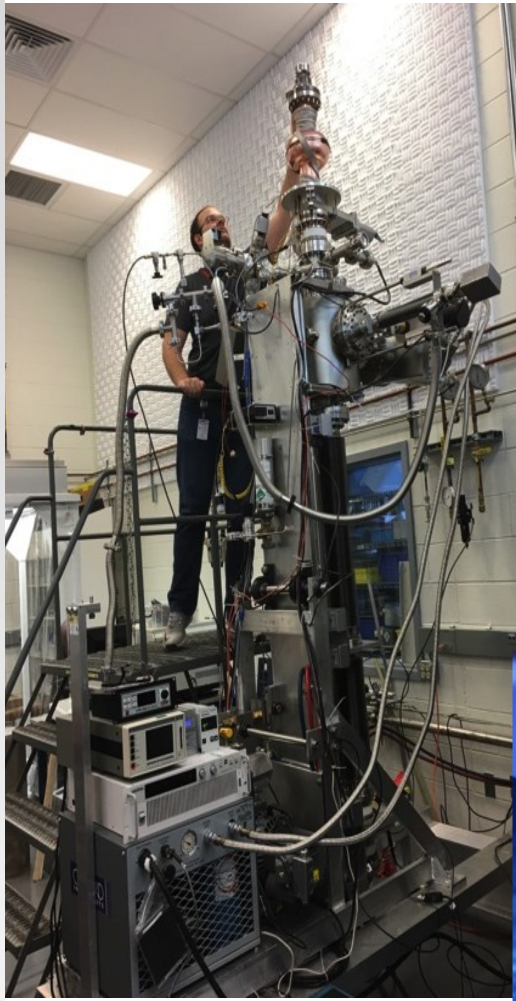


Rosaz, SRF 2017, Lanzhou (China)

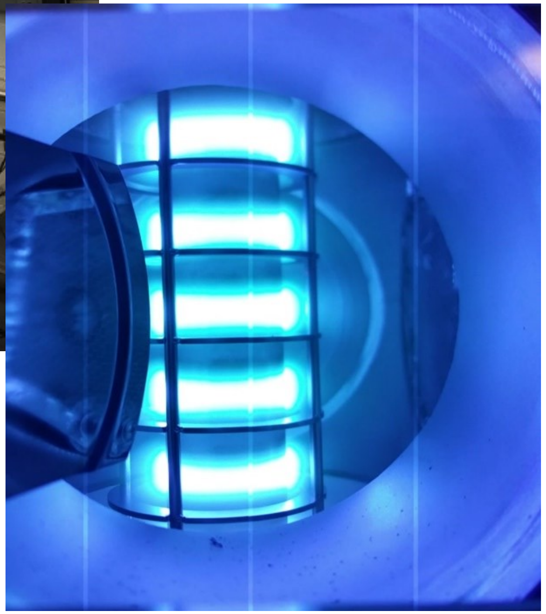


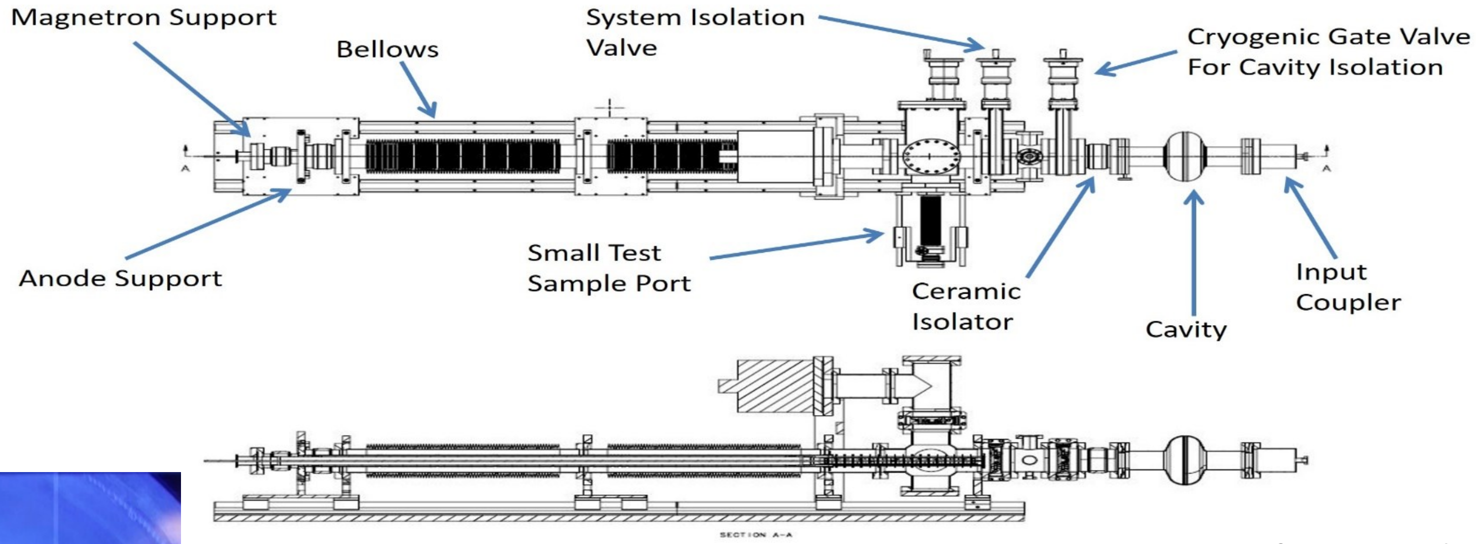
- High Bias does not give good results (gas implantation , stress)
- Lower pressure tends to better performances (contamination, stress)
- **Q-slope looks mitigated vs DCMS coating**

HiPIMS @ JLAB



System re-commissioned
June 2018



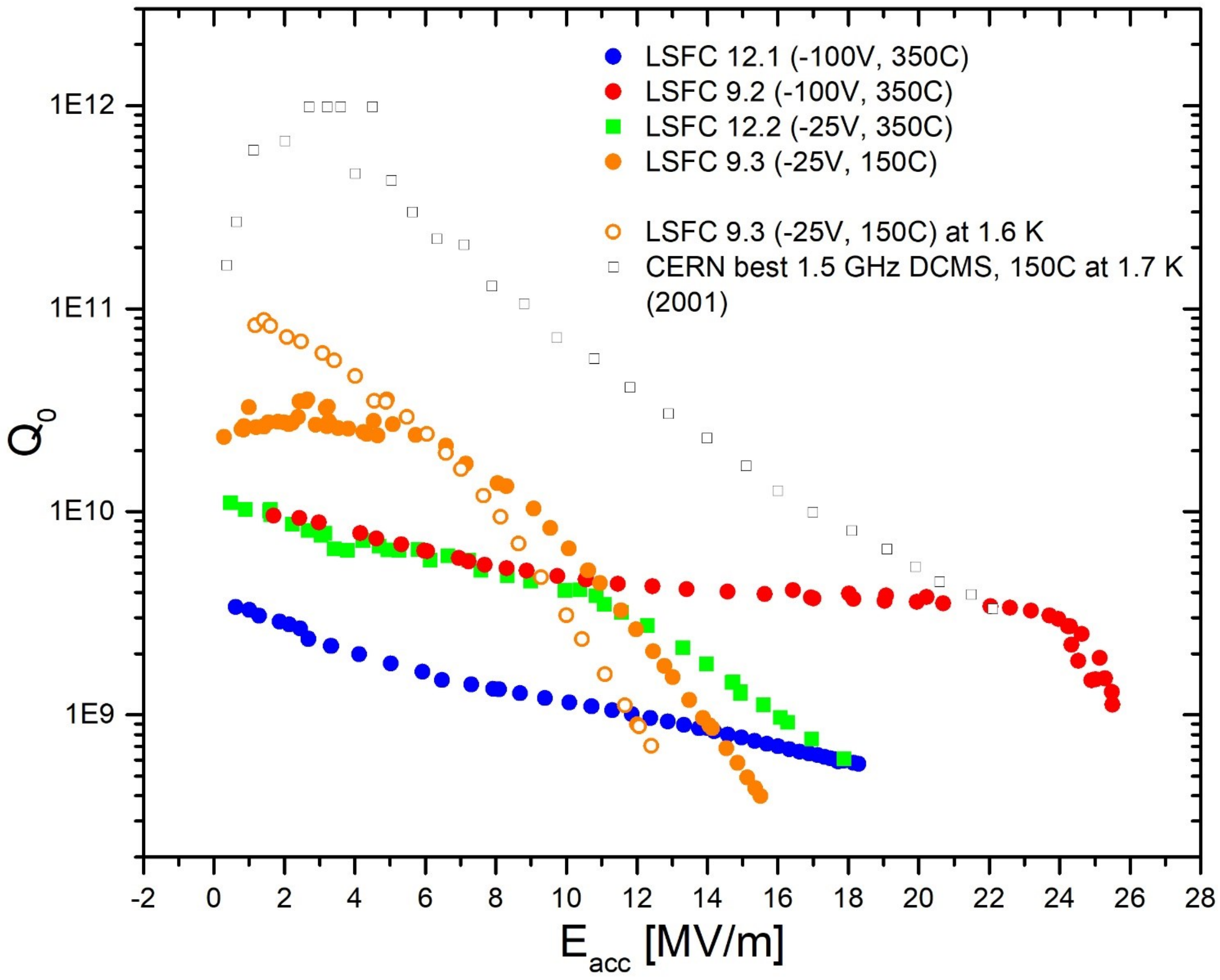


Courtesy of A.-M. Valente

- **Movable** cylindrical Nb **cathode**
- Background pressure in 10^{-9}-10^{-10} Torr
- Coating **temperatures up top 400 °C** under external nitrogen flow
- Kr atmosphere

Courtesy of A-M. Valente (JLAB)

HiPIMS Results @JLAB

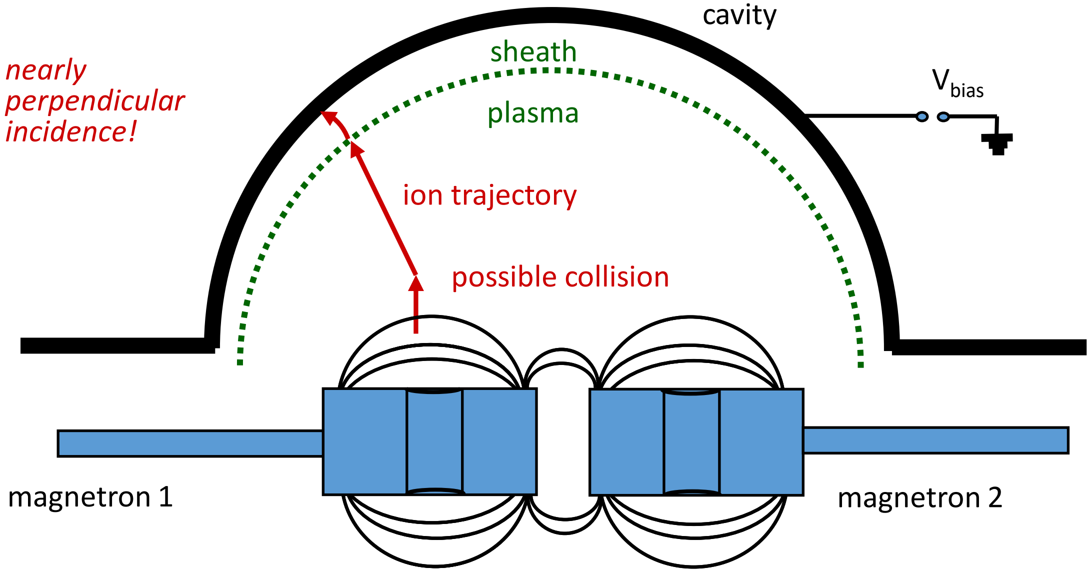


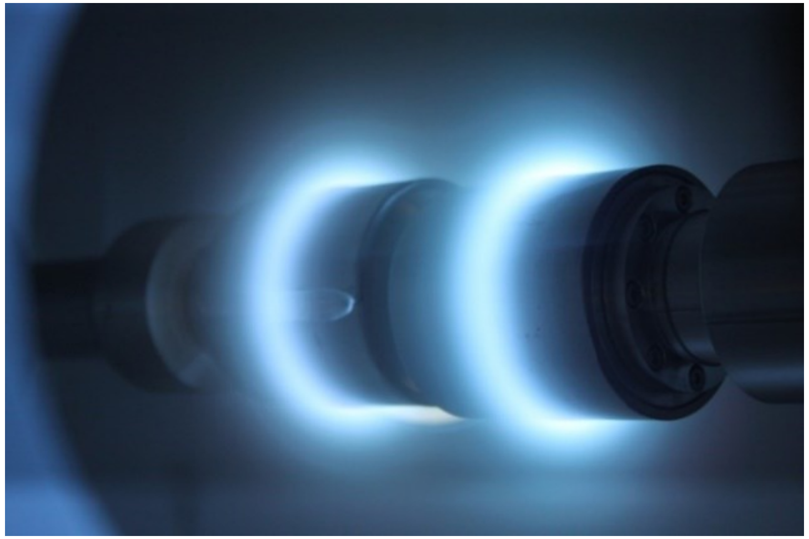
Some HiPIMS Nb/Cu cavities show **mitigation** of the characteristic **Q-slope**

Substrates are a possible **cause** of performance **limitation**

Courtesy of A-M. Valente (JLAB)

HiPIMS configuration @ LBNL





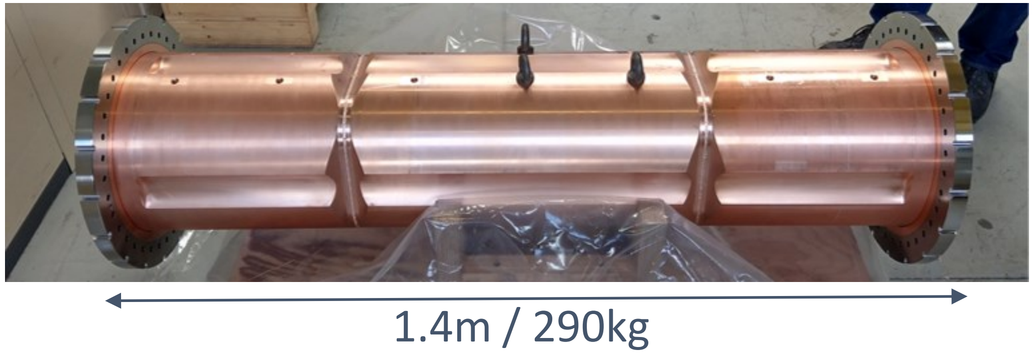
- HiPIMS **Dual Magnetron Configuration**
- Most effective for Biasing & influencing Ion Energies & Trajectories
- High power mode (above runaway threshold)
- Dominated by Nb emission
- No cavity RF tested

A. Anders, Thinfilms Workshop 2016, JLab

R&D on Nb films

The WOW cavity coating challenge

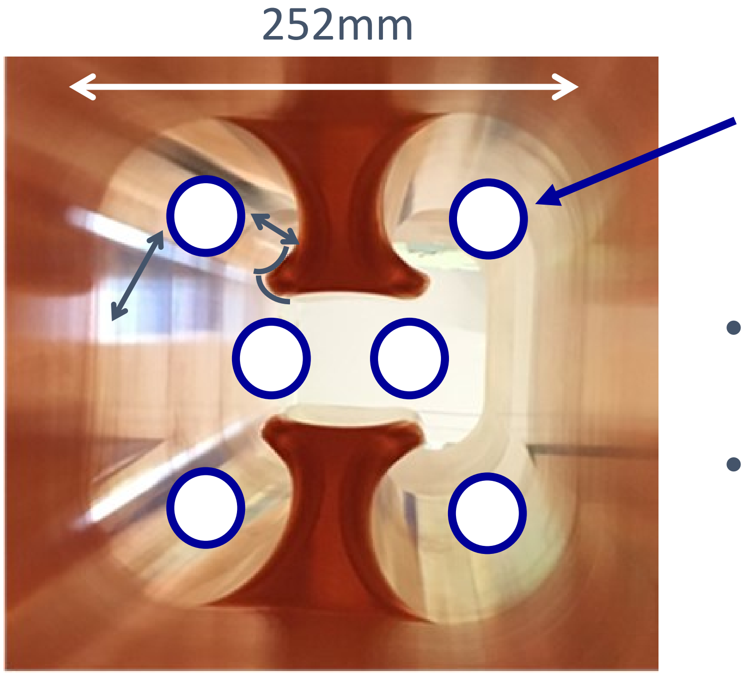
Wide-Open Waveguide (WOW) crab cavity (Nb/Cu), 1st prototype completed in 2018



HiPIMS +
- **negatively biased substrate**
- **positive pulse**

HiPIMS ↓ More metal ions

positive pulse ↓ High energy metal ions to densify the film



x6 Cylindrical magnetrons

- Distances (20 - 80 mm)
- Angles of incidence (0 - 90°)

F. Avino (CERN), TTC Meeting, CERN 2020

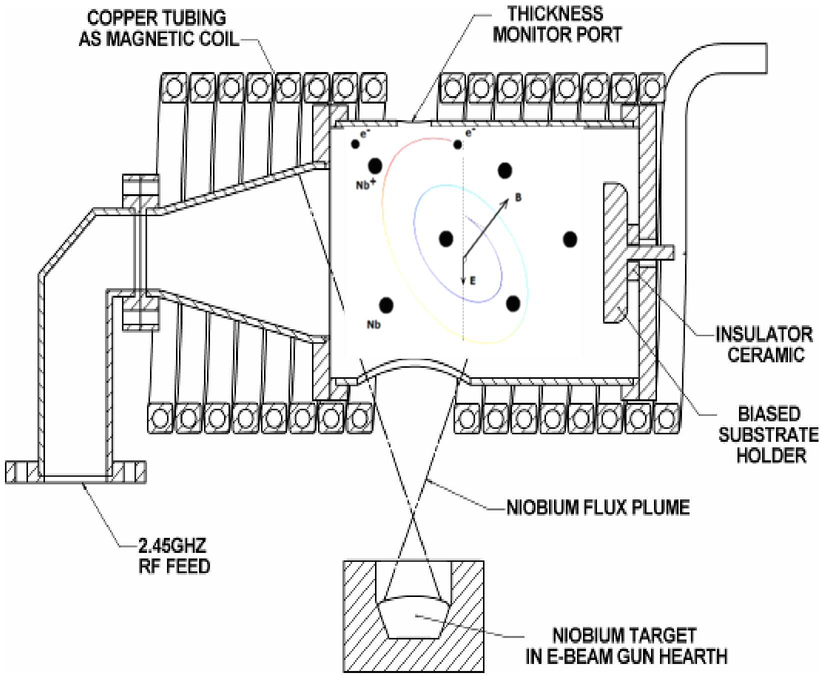
ECR

Jefferson Lab (A-.M. Valente et al.)

Energetic Condensation with ECR @JLAB

ECR DEPOSITION PROCESS

1. Nb is evaporated by e-beam in a separate vacuum chamber
2. Nb vapours are ionized by an ECR process
   - RF power (@2.45GHz)
   - Static B ⊥ E_{RF} with ECR condition
3. Nb ion are accelerate to the substrate (cavity) by a bias voltage



No working gas

Singly charged ions (64eV) produced in vacuum

Controllable deposition energy with Bias voltage

Excellent bonding , **No macro particles**

Good conformality

Scalability?

ECR film properties

	Substrate	RRR max
Insulating	a-Al_2O_3	591
	r-Al_2O_3	725
	c-Al_2O_3	247
	MgO (100)	188
	MgO (110)	424
	MgO (111)	270
	Al_2O_3 ceramic	135
	AlN ceramic	110
	Fused Silica	84
Metallic	**Cu (100)**	181
	Cu (110)	275
	Cu (111)	245
	Cu fine grains	**193**
	Cu large grains	**305**

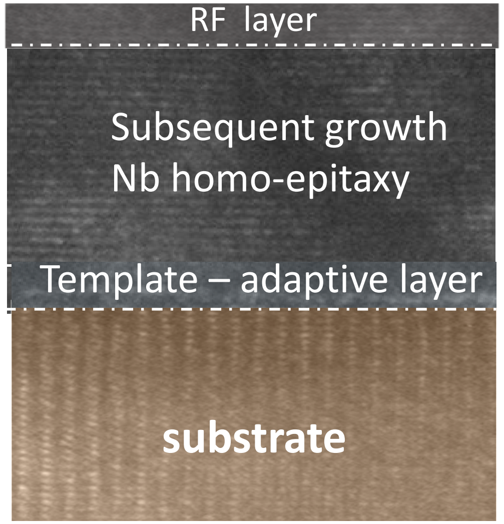
Courtesy of A-M. Valente (JLAB)

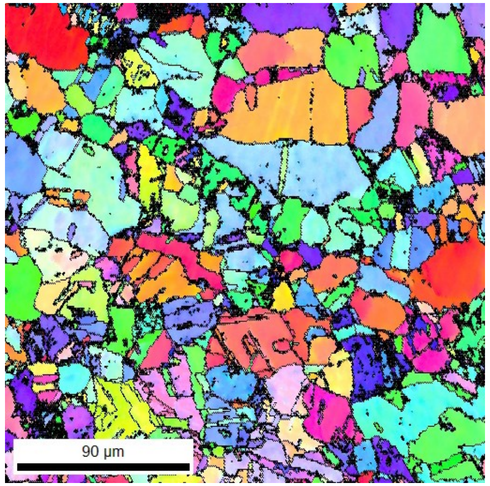
SEQUENTIAL PHASE FOR FILM GROWTH

- Interface
- Film nucleation (184 eV)
- Growth of appropriate template for subsequent deposition (64 eV)
- Deposition of final surface optimized for minimum defect density

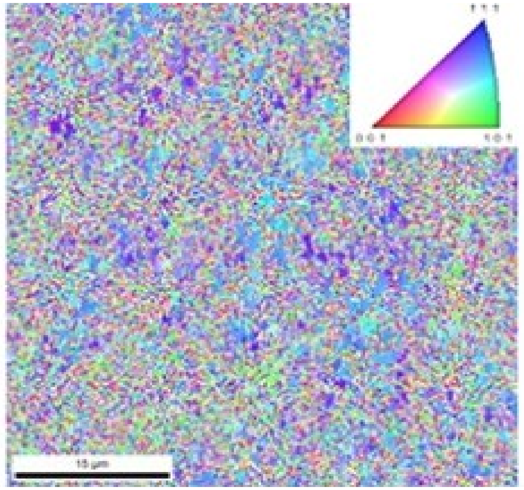
Bulk like properties

Opportunity for film engineering

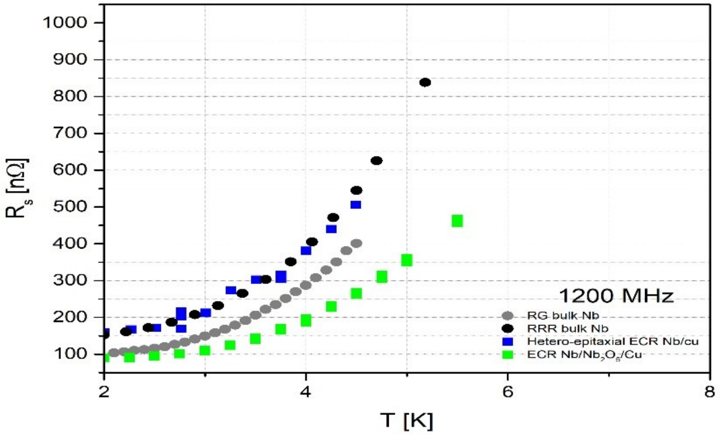




Hetero-epitaxial growth



Growth on amorphous interface

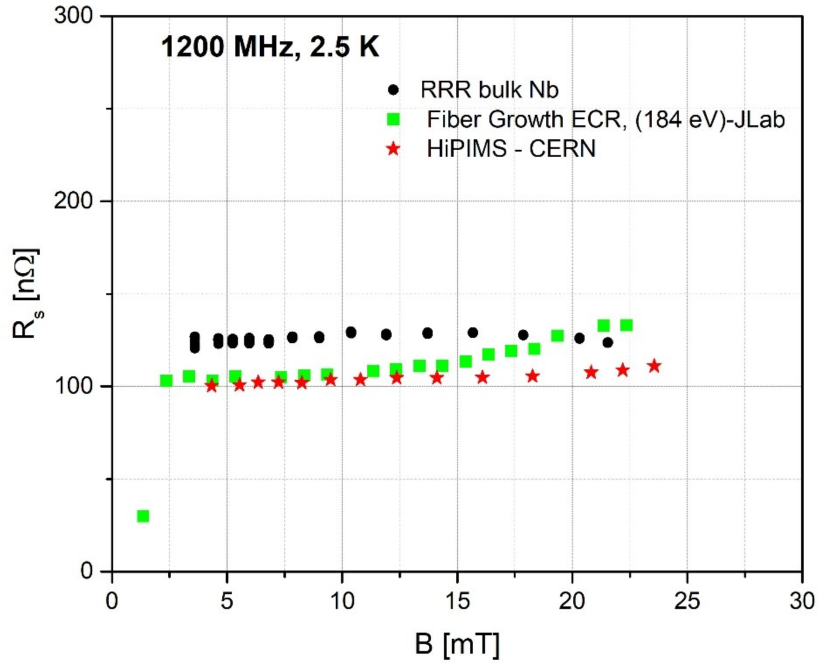


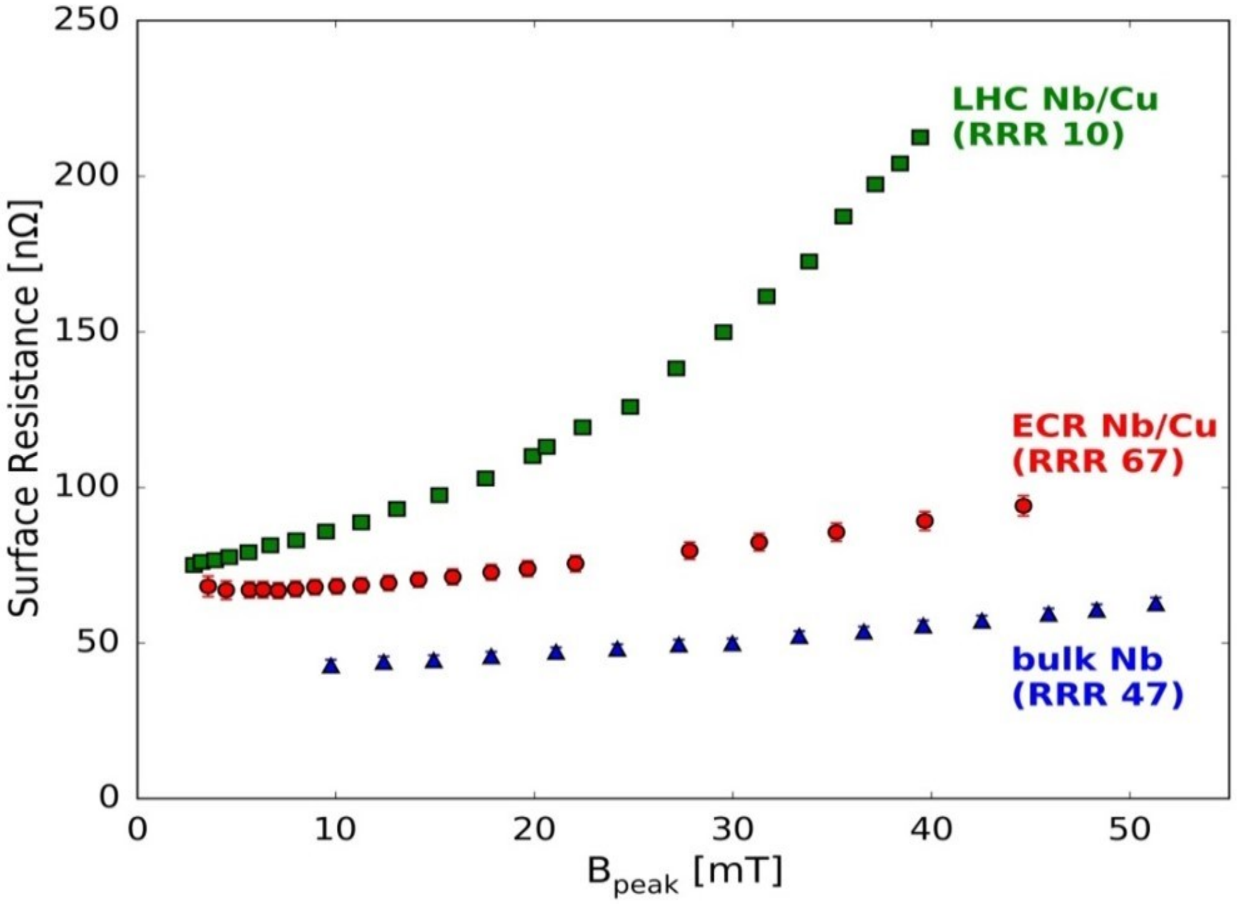
Fiber growth ECR Nb/Cu films perform better than hetero-epitaxial ones

ECR Results

Mitigation of Rs slope possible

Energetic Condensation Nb/Cu films show **similar RF behavior compare to bulk Nb** in QPR measurements





Courtesy of A-M. Valente (JLAB)

Chemical Vapour Deposition

Fundamental sequential steps in every CVD process

1. Convective and diffusive transport of reactants from the gas inlets to the reaction zone
2. Chemical reactions in the gas phase to produce new reactive species and by-products
3. Transport of the initial reactants and their products to the substrate surface
4. Adsorption (chemical and physical) and diffusion of these species on the substrate surface
5. Heterogeneous reactions catalyzed by the surface leading to film formation
6. Desorption of the volatile by-products of surface reactions
7. Convective and diffusive transport of the reaction by-products away from the reaction zone

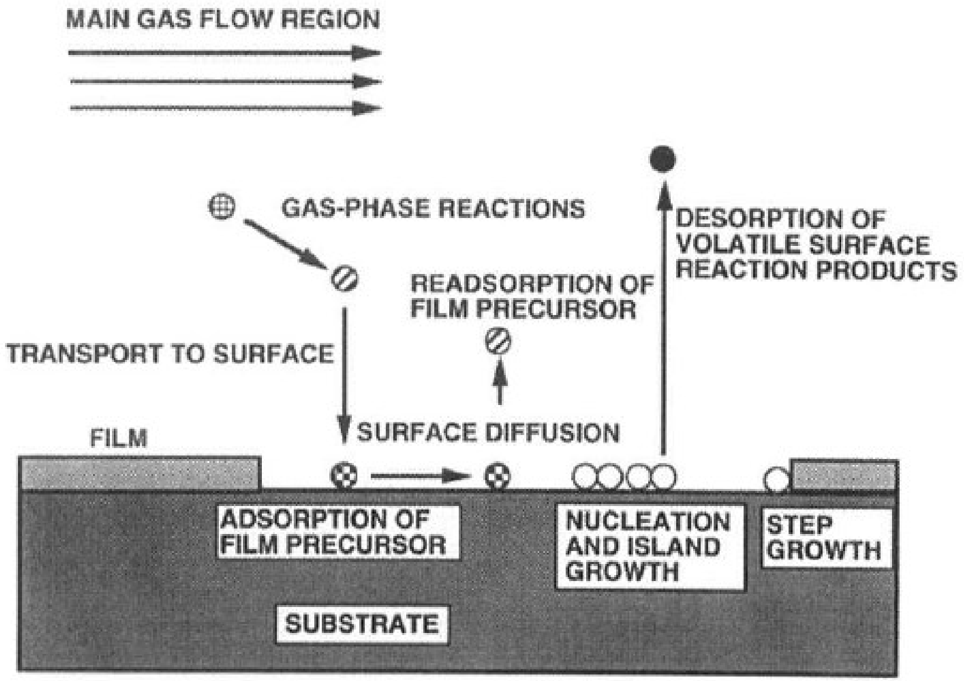


Figure 6-2 Sequence of gas transport and reaction processes contributing to CVD film growth. (From *Chemical Vapor Deposition*, edited by M. L. Hitchman and K. F. Jensen. Reprinted with the permission of Academic Press, Ltd., and Professor K. F. Jensen, MIT.)

CVD @ Cornell University and Ultramet

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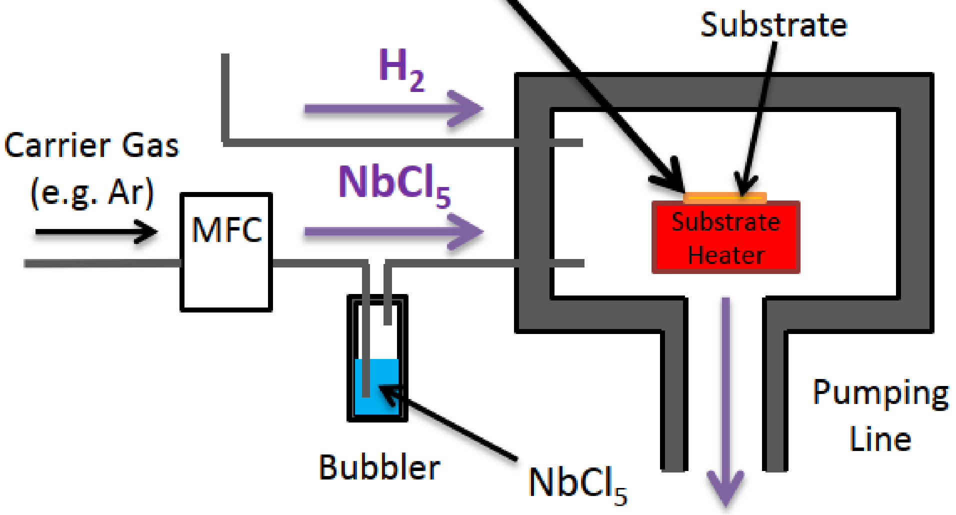
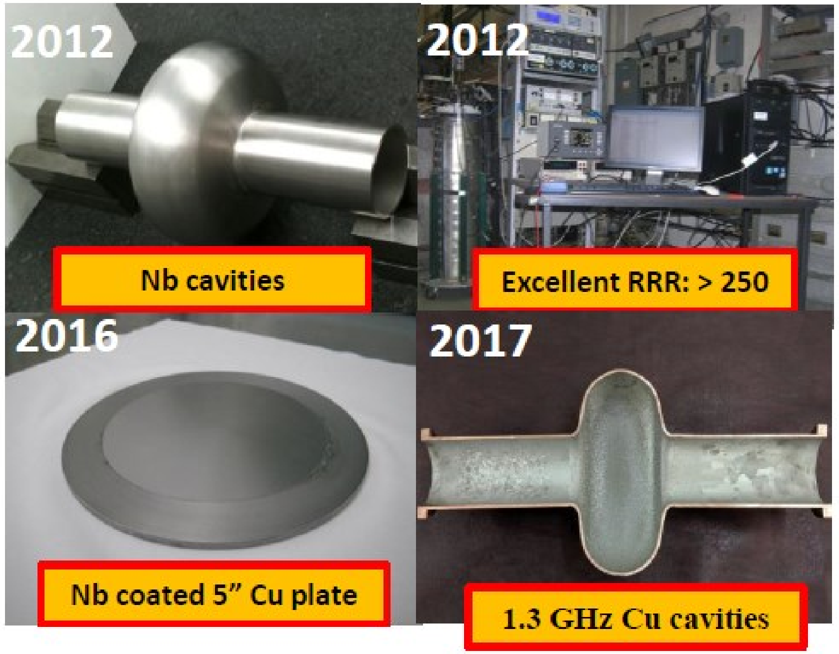
$$2\ NbCl5 + 5\ H2 \rightarrow 2\ Nb + 10\ HCl$$



Reactor diagram showing use of $NbCl_5$ to produce CVD niobium

P. Pizzolet al., (STFC) IPAC (2016)

CVD @ Cornell University and Ultramet



2012

Nb cavities

2012

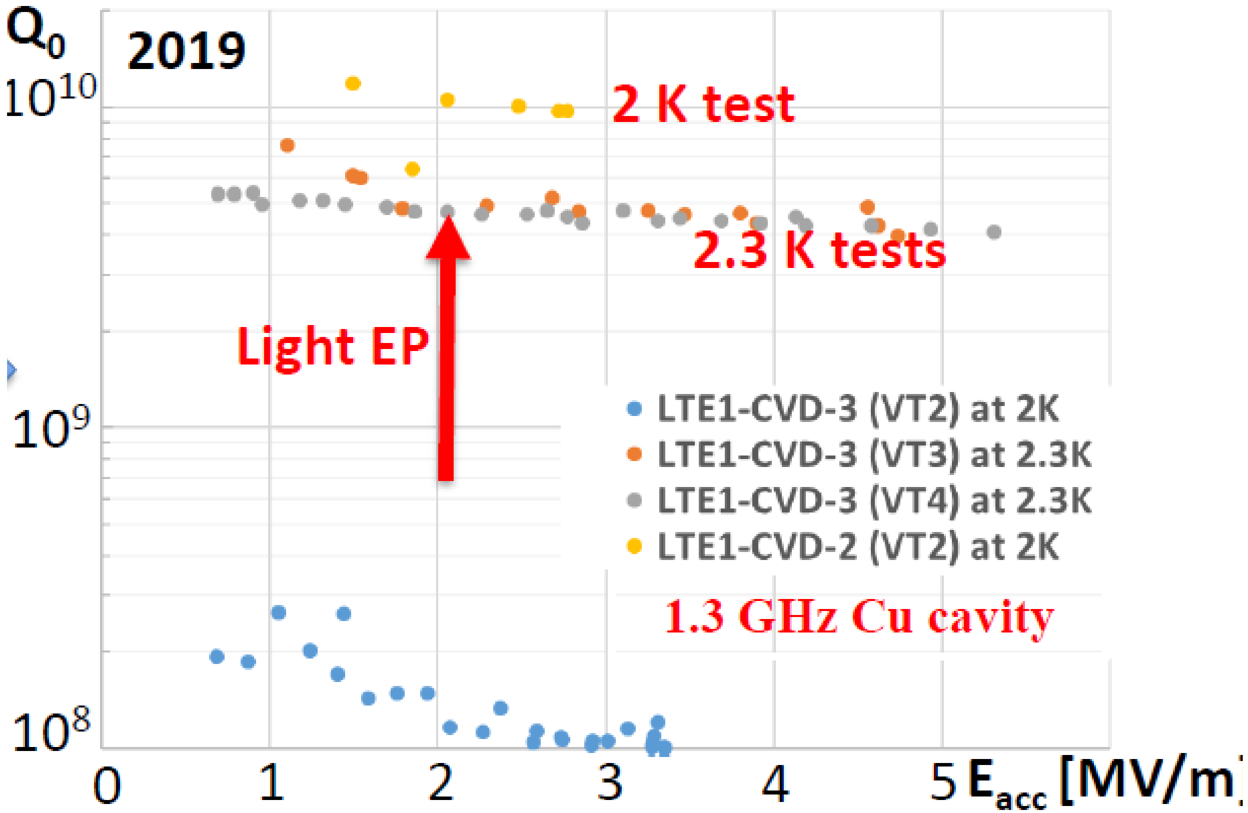
Excellent RRR: > 250

2016

Nb coated 5” Cu plate

2017

1.3 GHz Cu cavities



Film optimization & process scale-up

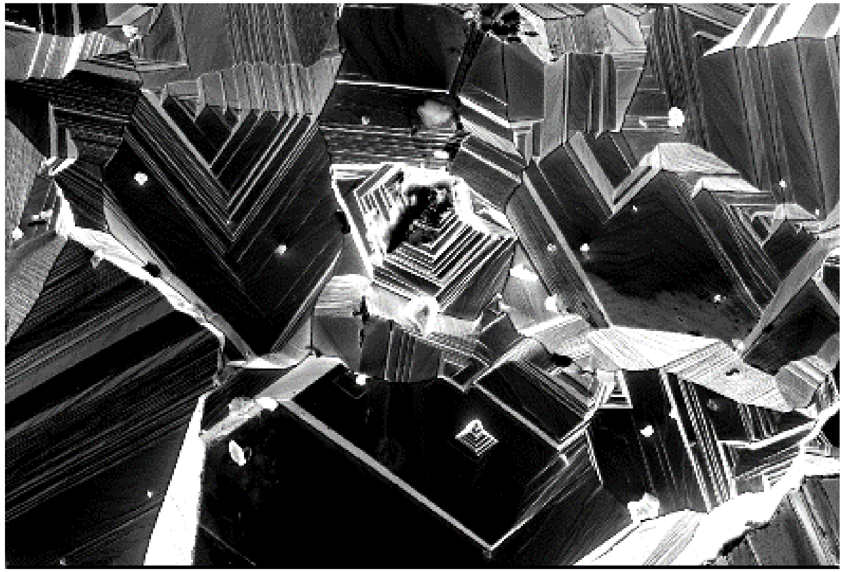
High purity (high RRR)

Excellent adhesion

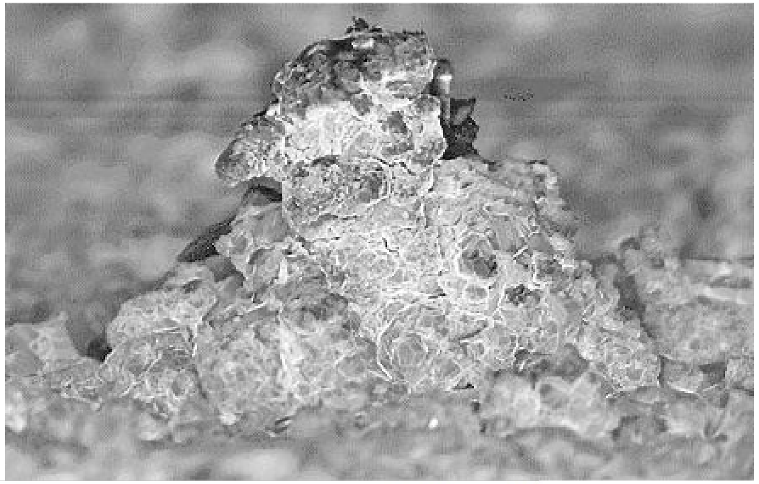
Full size cavity

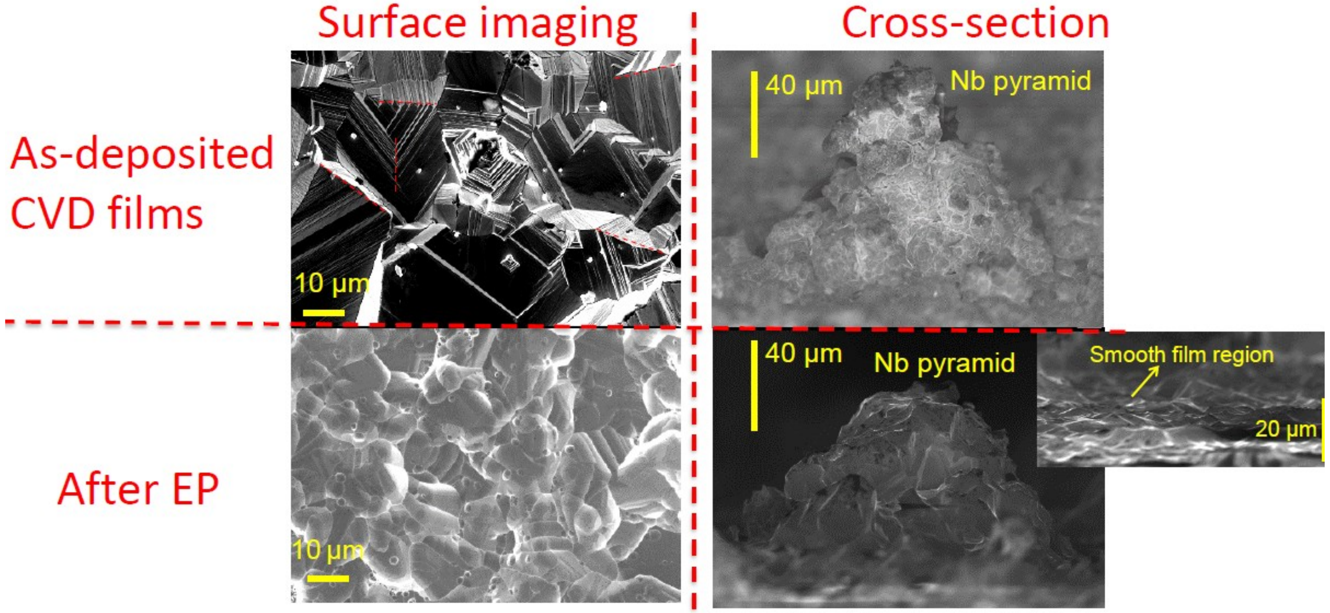
Zeming Sun Mingqi (Cornell), TTC Meeting, CERN 2020

CVD @ Cornell University and Ultramet



EP





Very Rough Surface

EP smooth pyramids

Zeming Sun Mingqi (Cornell), TTC Meeting, CERN 2020

Conclusions

Nb thin films are the optimum choice for low gradient/4.2 K applications

- Cost reduction
- $R_{BCS\ film} < R_{BCS\ bulk}$ → $Q_{0\ film} > Q_{0\ bulk}$
- Thermal stability
- Mechanical stability
- Less sensitivity to magnetic field trapping

Mitigation of Q-slope for high gradient applications seems possible

We need to understand the reason of the Q-slope

- Establish adequate process controls
- Mandatory have better substrates and chemical processes
- Need more RF measurements statistics

Other materials for SRF?

Superconductors for SRF?

Material	T_C (K)	ρ_n (μΩcm)	μ_0H_{C1} (mT)*	μ_0H_{C2} (mT)*	μ_0H_C (mT)*	μ_0H_{SH} (mT)*	λ (nm)*	ξ (nm)*	Δ (meV)	Type
Pb	7,1		n.a.	n.a.	80		48			I
Nb	**9,22**	**2**	**170**	**400**	**200**	**219**	**40**	**28**	**1.5**	**II**
NbN	**17,1**	**70**	**20**	**15 000**	**230**	**214**	**200-350**	**<5**	**2.6**	**II**
NbTi			4-13	>11 000	100-200	80-160	210-420	5,4		
NbTiN	17,3	35	30				150-200	<5	2.8	II
Nb_3Sn	**18,3**	**20**	**50**	**30 000**	**540**	**425**	**80-100**	**<5**	**<5**	**II**
Mo_3Re	15	10-30	30	3 500	430	170	140			II
MgB_2	**39**	**0.1-10**	**30**	**3 500**	**430**	**170**	**140**	**5**	**2.3/7.2**	**II- 2gaps****
2H-$NbSe_2$	7,1	68	13	2680-15000	120	95	100-160	8-10		II- 2gaps**
YBCO/cuprates	**93**		**10**	**100 000**	**1400**	**1050**	**150**	**0,03/2**		**d-wave****
Pnictides $Ba_{0.6}K_{0.4}Fe_2As_2$	**38**		**30**	**>50000**	**900**	**756**	**200**	**2**	**10-20**	**s/d wave****

* @ 0K
** 2D => orientation problems ?

C. Antoine, CEA Saclay

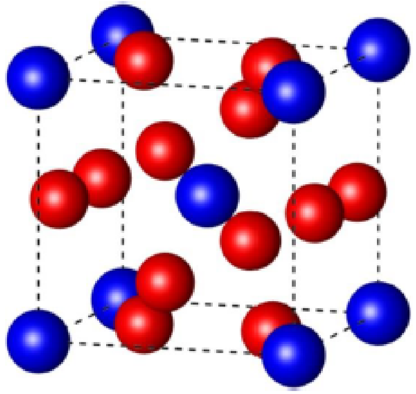
Nb_3Sn - in principle a great choice

Higher critical temperature

→ Operation at 4.2 K

Higher superheating field

→ Double the limit of niobium



Blue: tin

Red: niobium

Parameter	Niobium	Nb_3Sn
Transition temperature	9.2 K	18 K
Superheating field	219 mT	425 mT
Energy gap Δ/k_bT_c	1.8	2.2
λ at T = 0 K	50 nm	111 nm
ξ at T = 0 K	22 nm	4.2 nm
GL parameter κ	2.3	26

← Lower losses

← Higher gradients

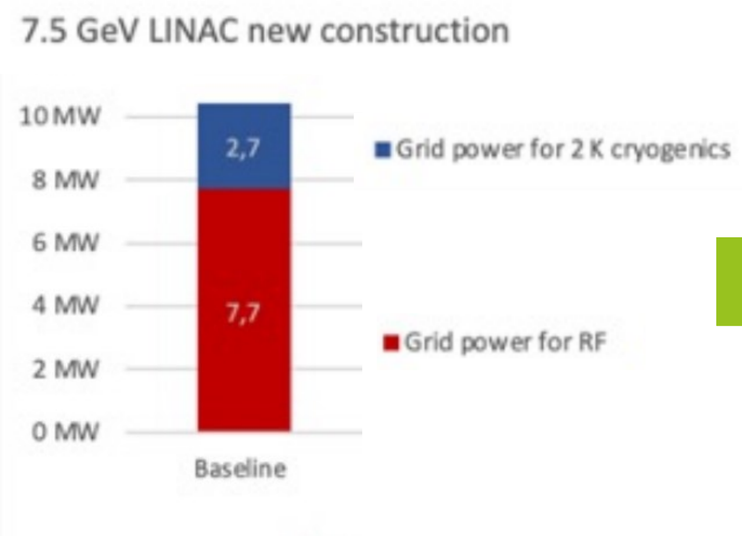
Nb_3Sn motivation

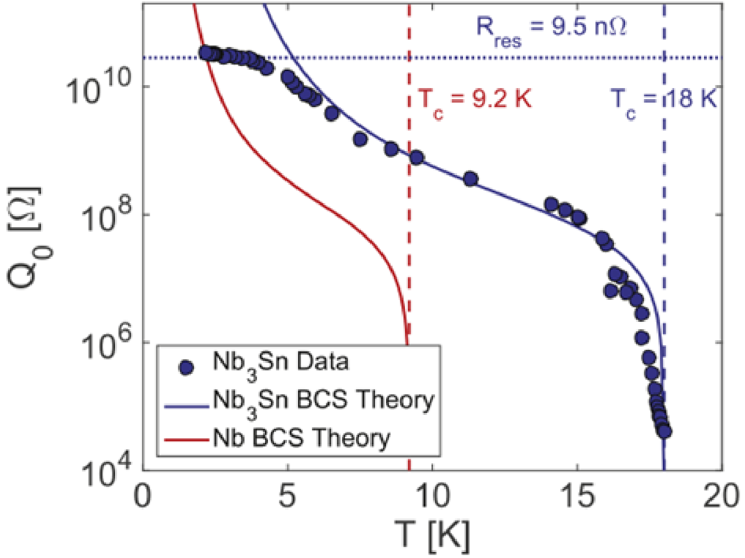
***Energy saving** is mandatory for **FCC-ee** and the **next generation accelerators**…*

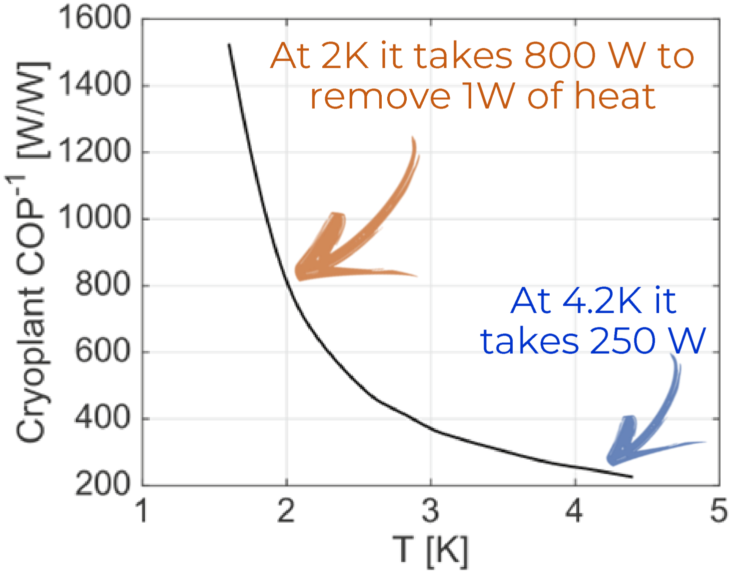
*… **cryogenics** is one of the **larger energy cost** in modern SRF accelerators*

Move from bulk Nb @2K to Nb_3Sn @4.5 K

reduces cryogenic power by a factor of 3







At 2K it takes 800 W to remove 1W of heat

At 4.2K it takes 250 W

Supercond. Sci. Technol. 30 (2017) 033004

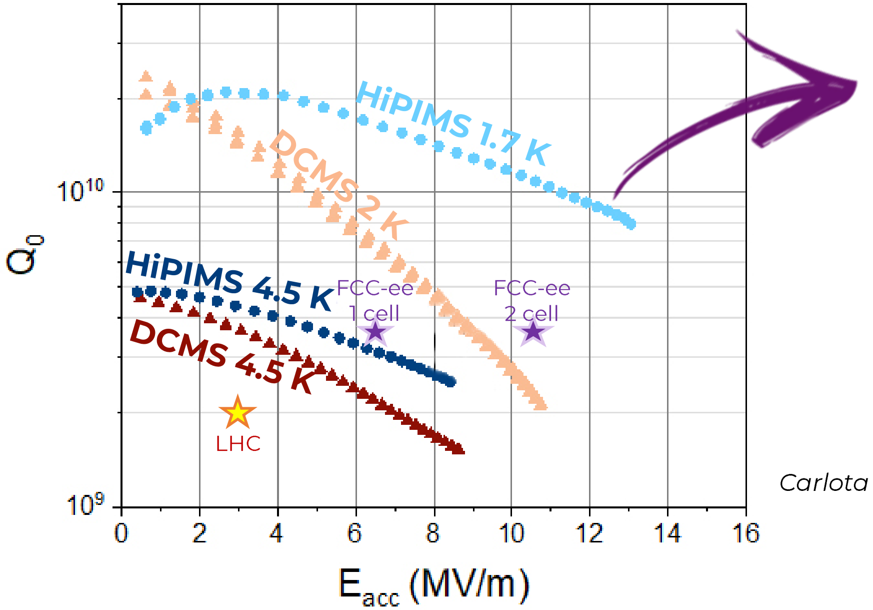
Nb_3Sn motivation

***Energy saving** is mandatory for **FCC-ee** and the **next generation accelerators**…*

*… **cryogenics** is one of the **larger energy cost** in modern SRF accelerators*

Move from **thin film Nb @4.5 K** to **Nb_3Sn @4.5 K**

Reduce T_{op}/T_c → Suppress R_{BCS} → Increase Q



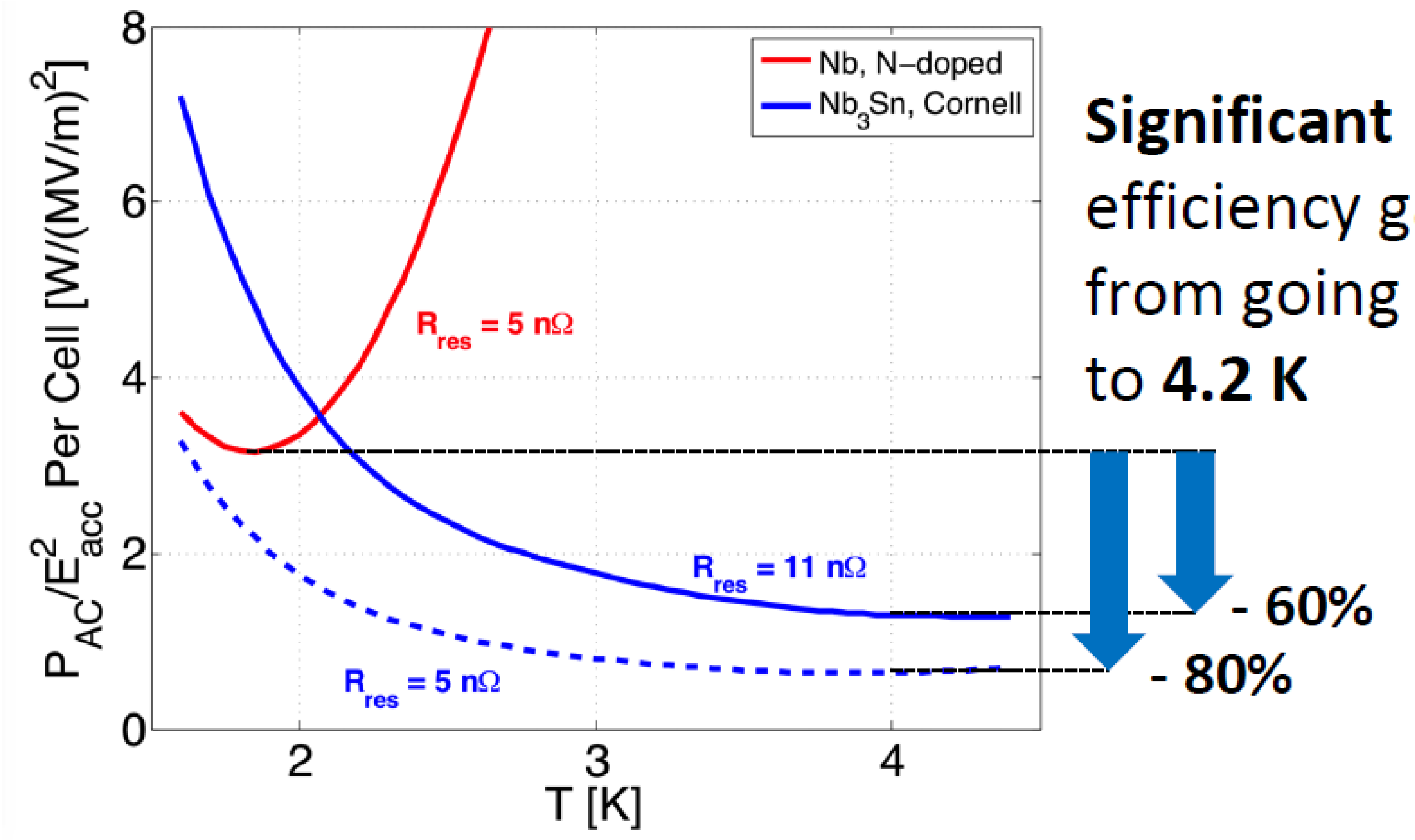
Expected Performances for Nb_3Sn

Carlota Pereira Carlos, FCC week 2023 (elaborated)

Effect of High Tc

Niobium → 45 MV/m

Nb_3Sn → 90 MV/m



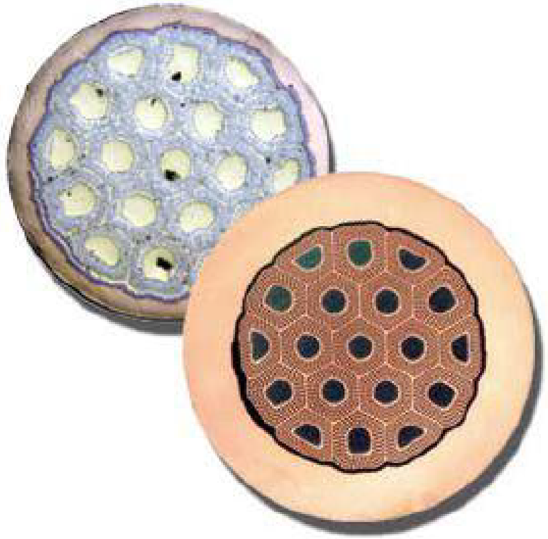
Significant efficiency gain from going to **4.2 K**

Nb_3Sn on Cu: Multiple challenges

- A15 are Brittle materials
- Complicated Phase Diagram
- Low melting point substrate
- Interface diffusion
- Coating Parameters
- Substrate preparation
- Target Production/Magnetron Design
- Trapped Flux
- Tuning

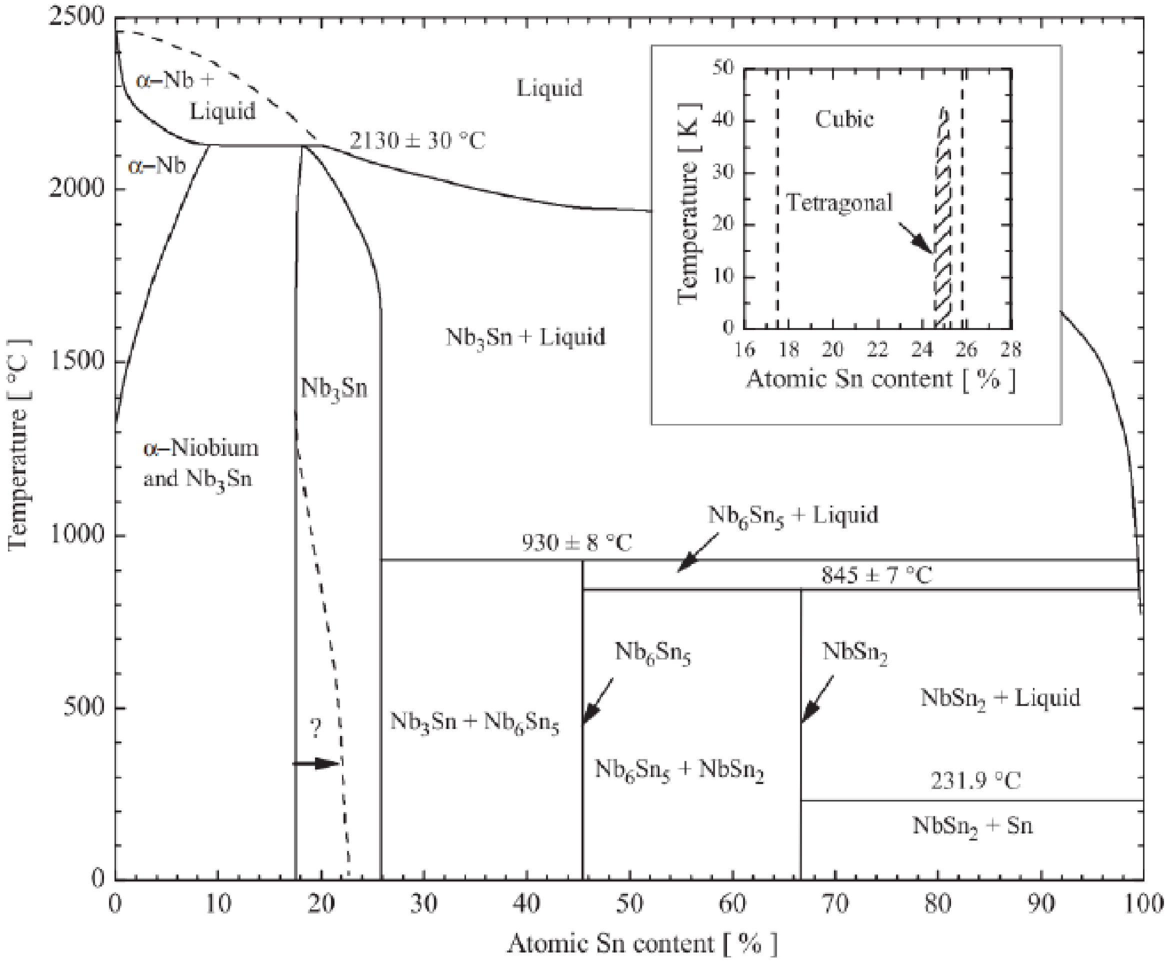
Nb_3Sn in magnets

Nb_3Sn is well known in the magnet community, where it has been used to generate fields above 10 Tesla



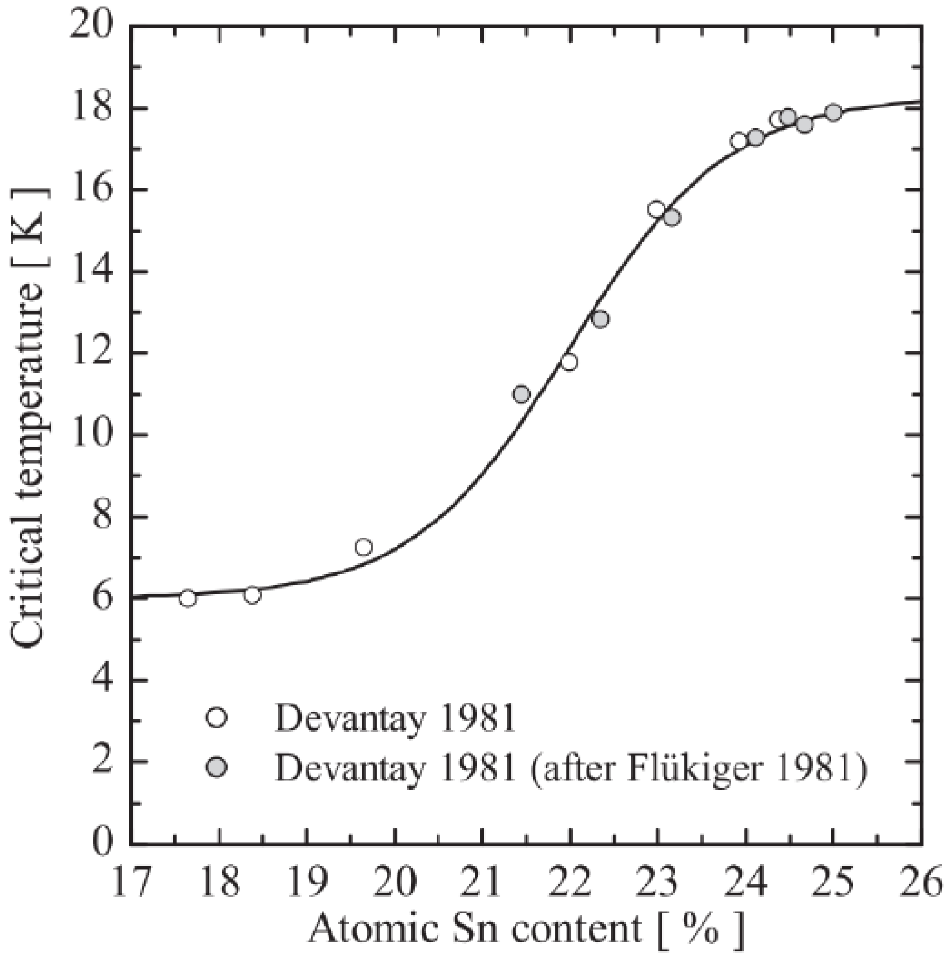
However, this material is designed to operate in the **mixed state**, and uses **copper** in the fabrication

Nb_3Sn phase diagram



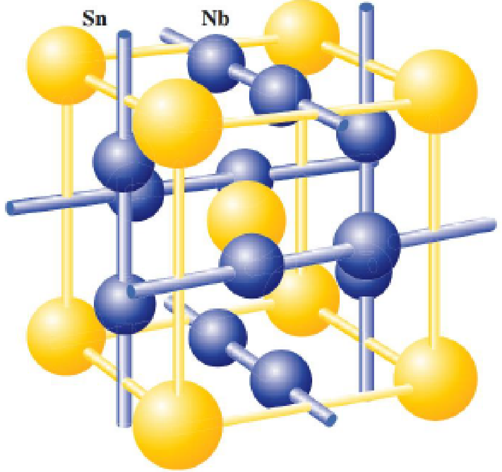
Above 950°C Nb_3Sn is the only binary phase

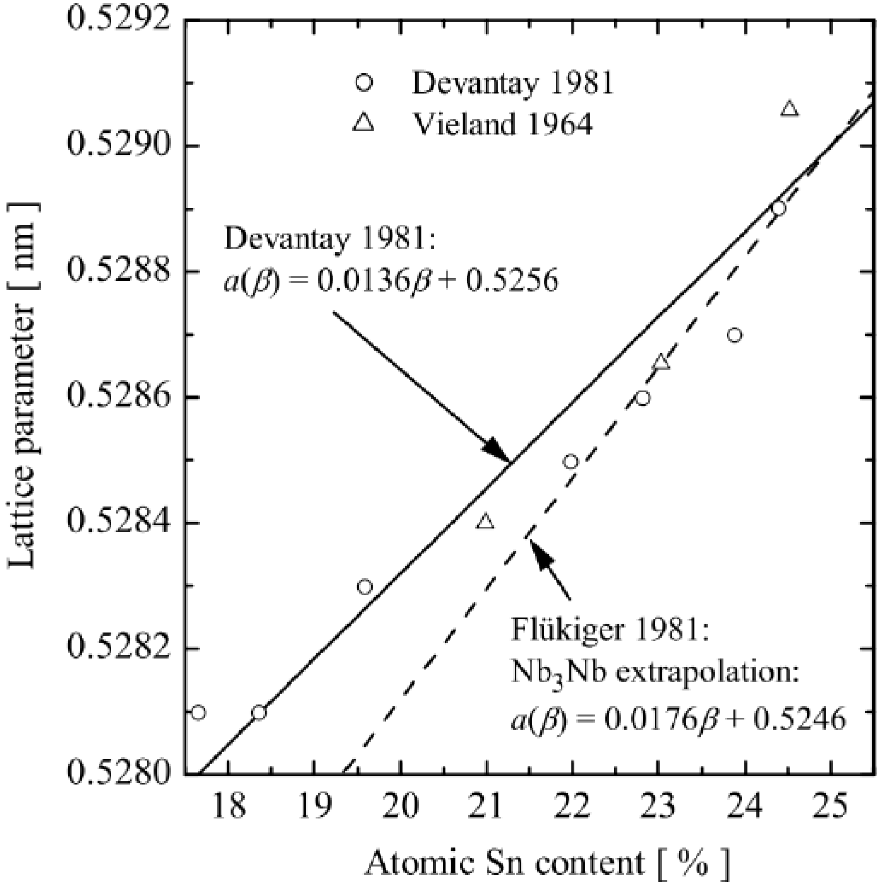
Tin-depleted Nb_3Sn



Tin-depletion of Nb_3Sn lowers T_c!

Changing lattice parameter

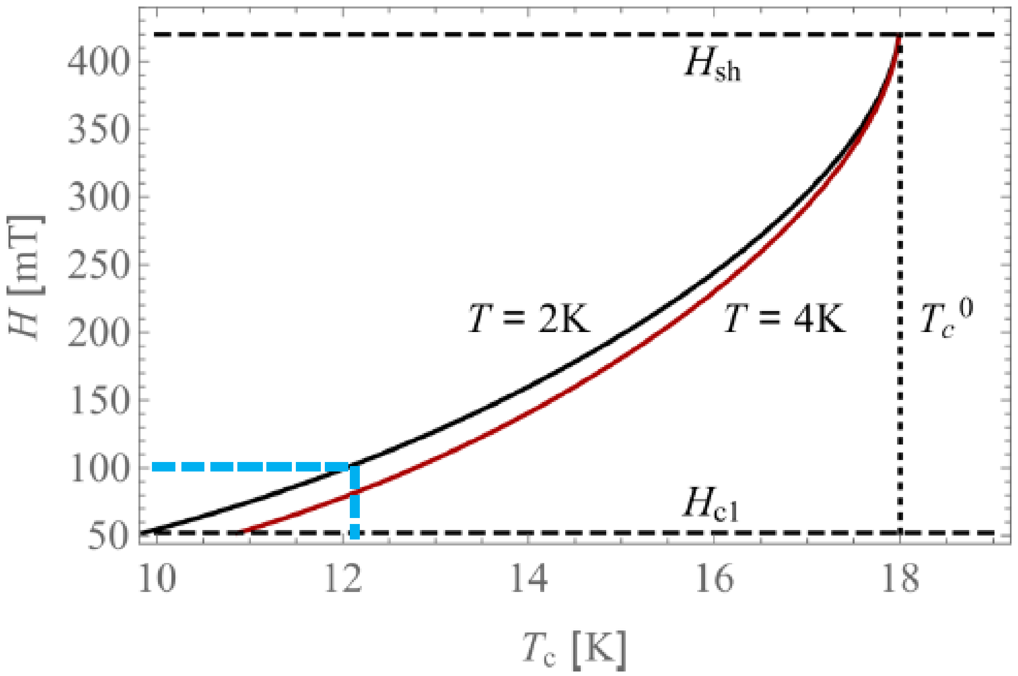


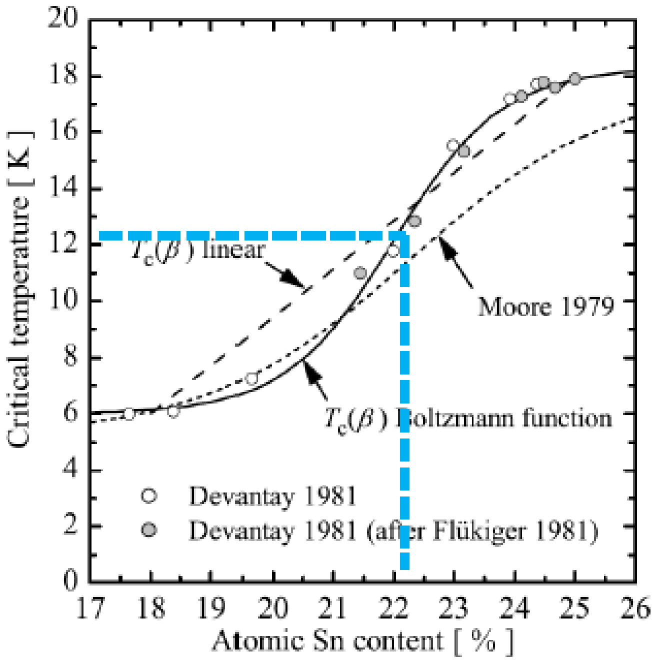


Tin depletion changes the lattice parameter

T_c suppression

A tin depletion of only **3%** reduces H_{sh} by **75%**





Flux entry could occur at tin-depleted surface defects

Moral of the story

Make stoichiometric Nb_3Sn!

25 atomic-% Sn

No exceptions!

Making Nb_3Sn a challenge!

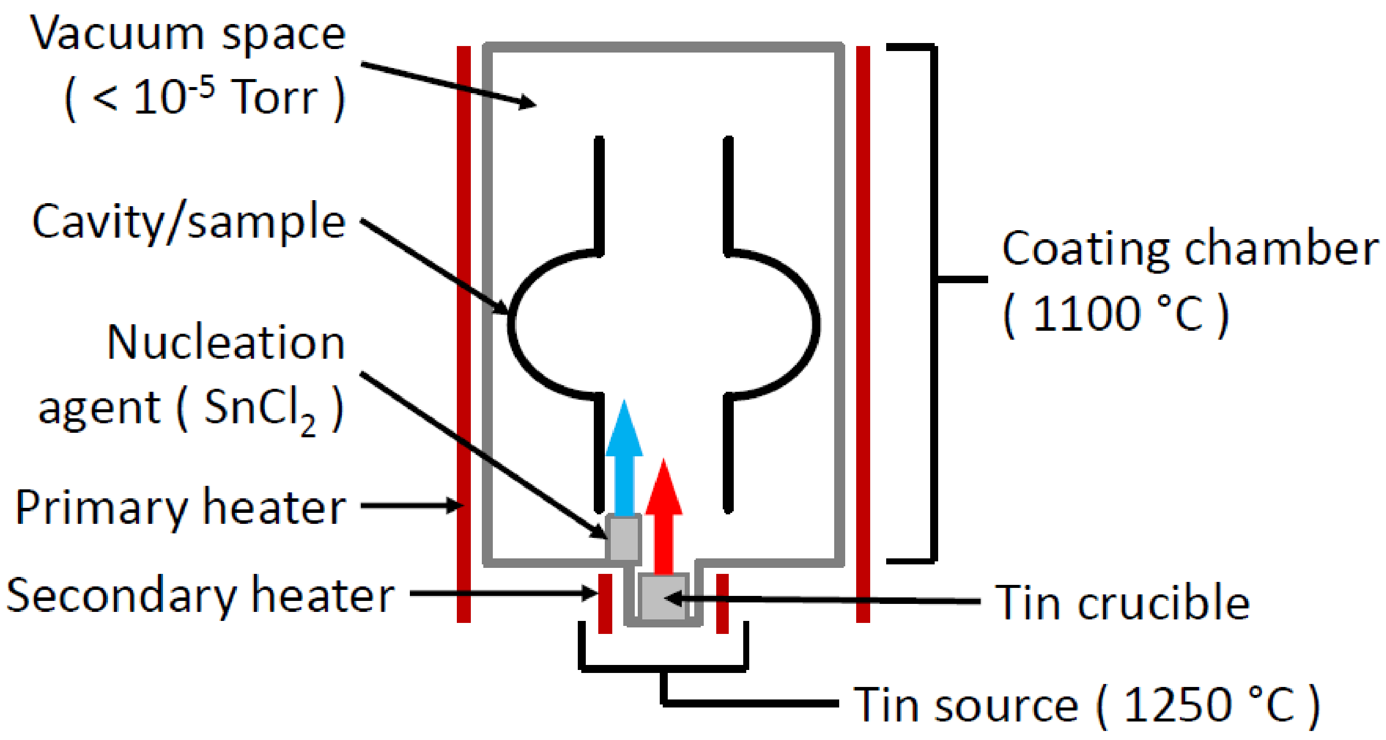
Nb_3Sn presents two major challenges:

- It is **brittle**
  - We cannot form it
- It has **low thermal conductivity**
  - Too thick, and we get thermal feedback

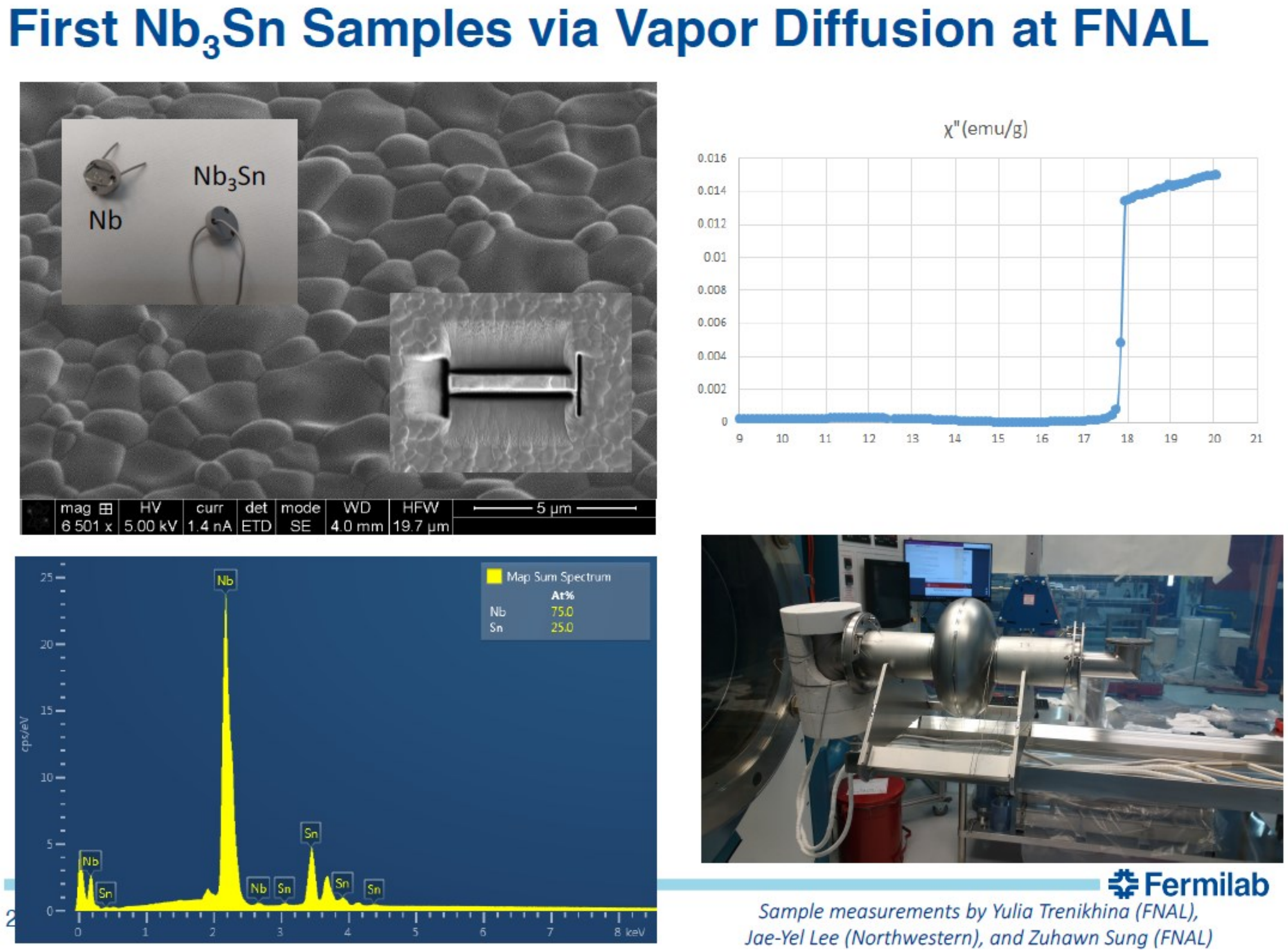
The answer: grow a **thin film** on a niobium substrate using the **vapour diffusion technique**

The vapour diffusion process

Coating furnace with separate **source hot-zone**



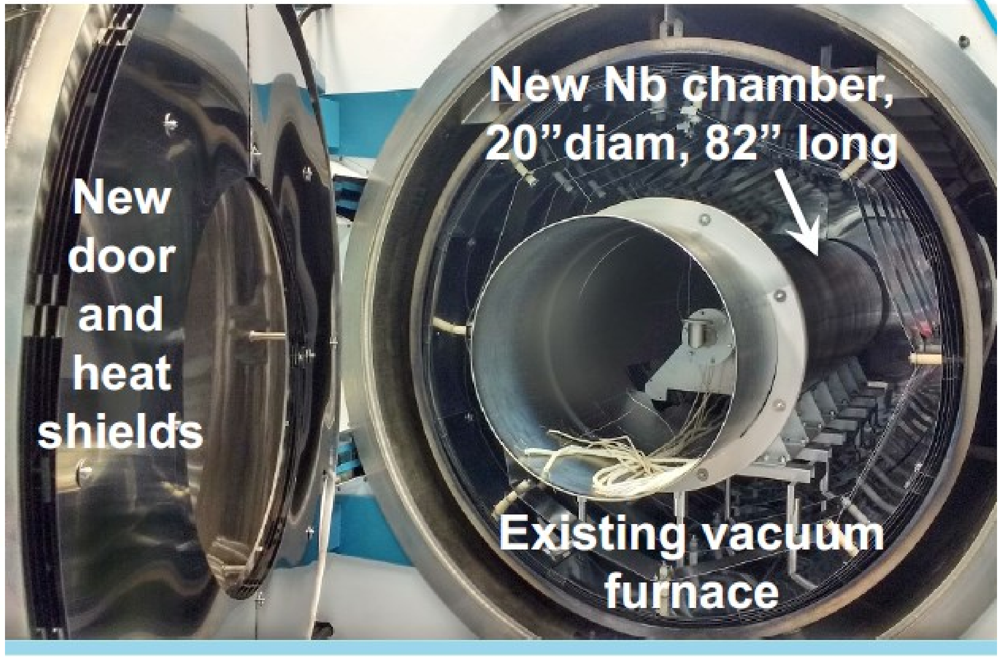
The vapour diffusion process



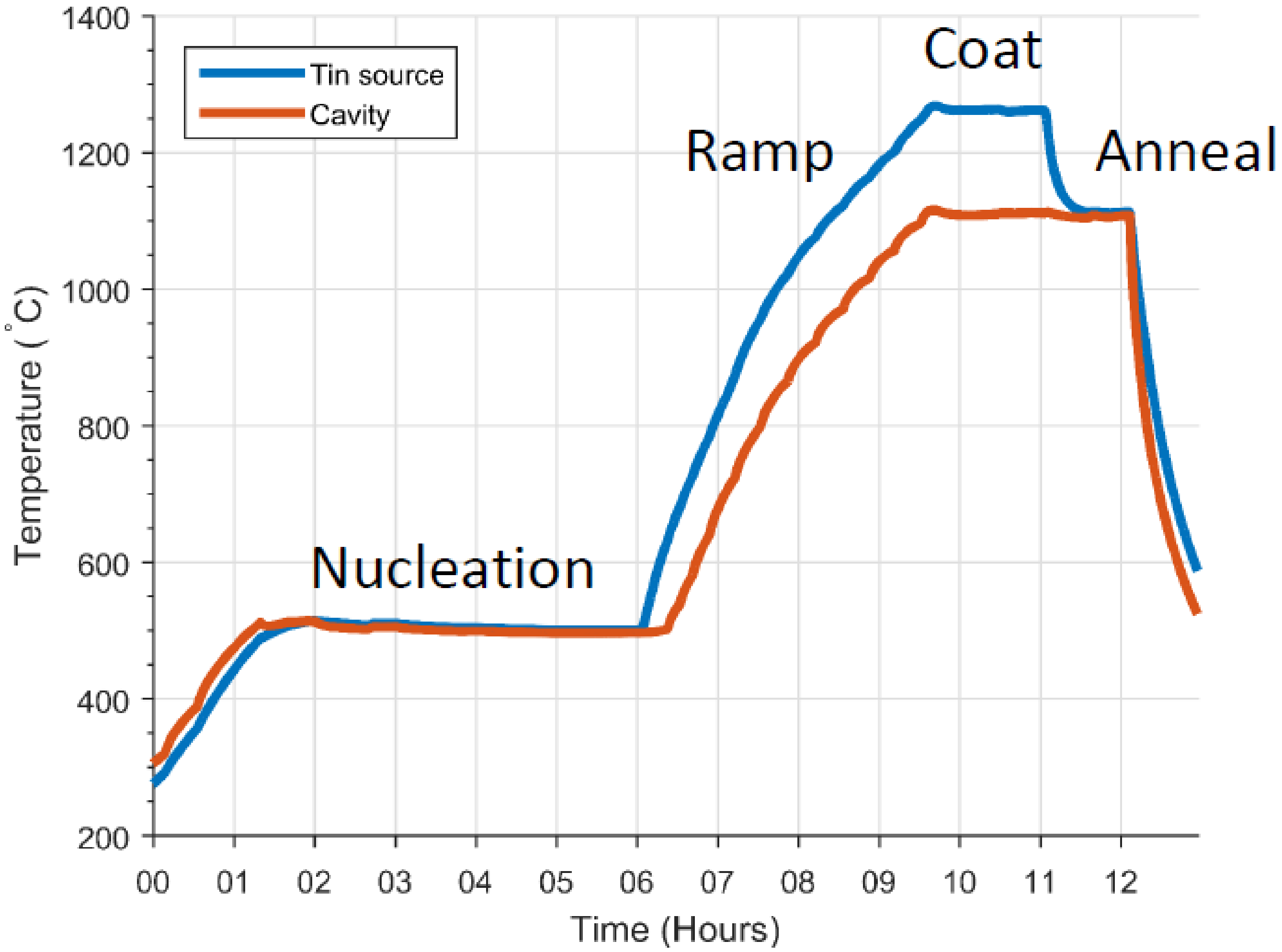
First Nb_3Sn Samples via Vapor Diffusion at FNAL

Fermilab

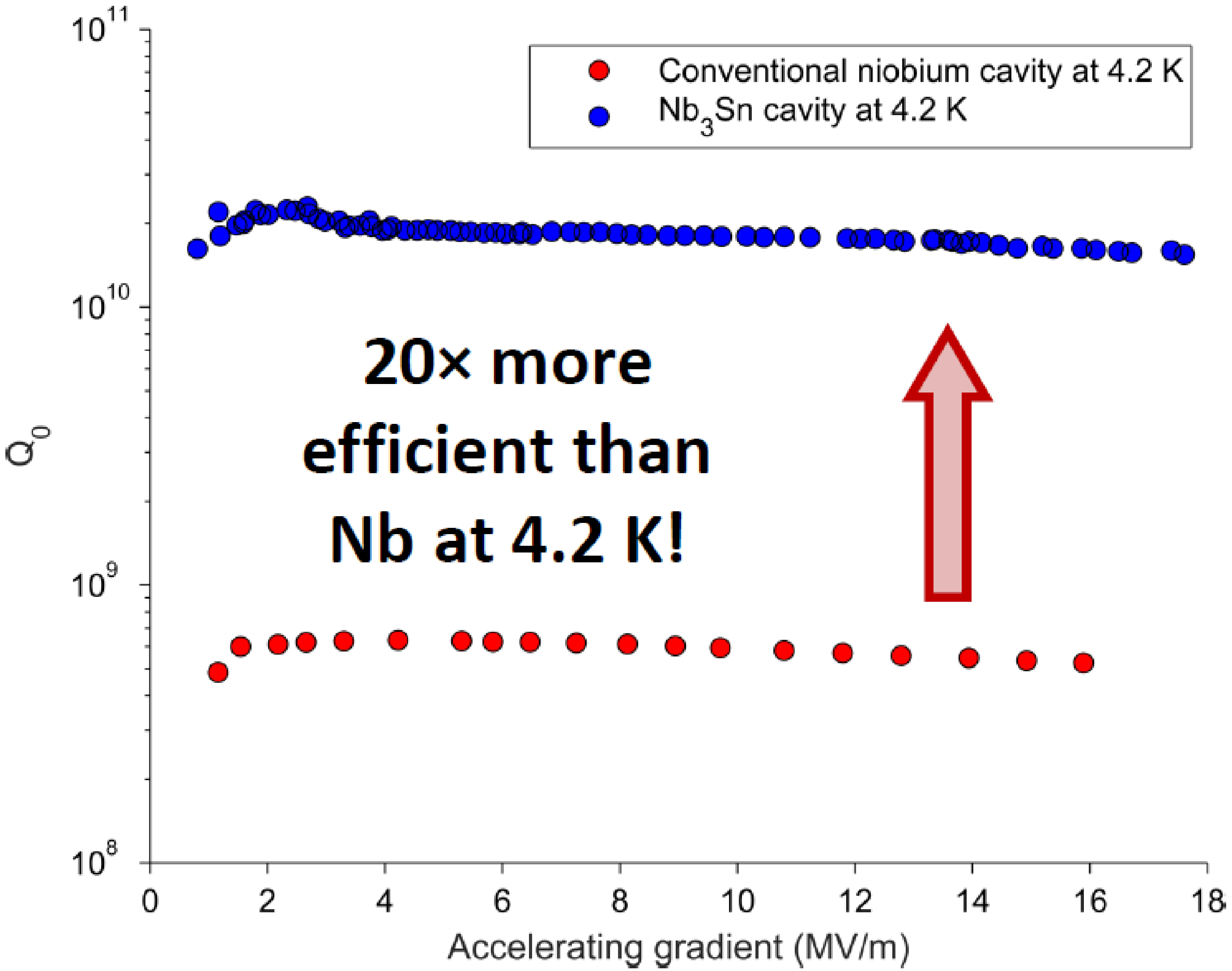
Sample measurements by Yulia Trenikhina (FNAL), Jae-Yel Lee (Northwestern), and Zuhawn Sung (FNAL)



Cornell coating profile



Comparision to Nb



20× more efficient than Nb at 4.2 K!

Nb_3Sn State of the art

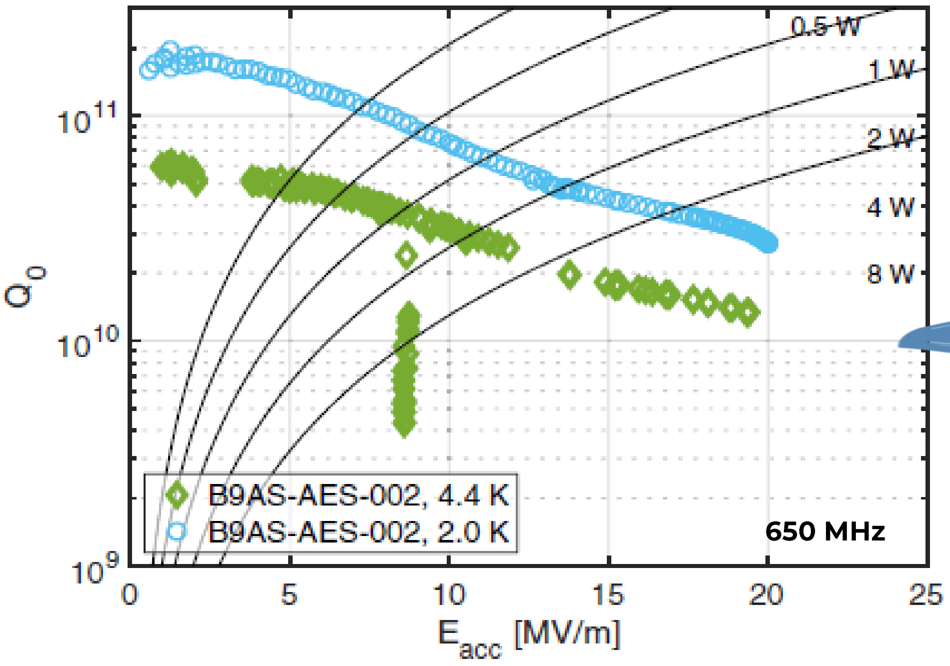
Main limit

Needed Nb bulk cavities

Not possible on **Cu**

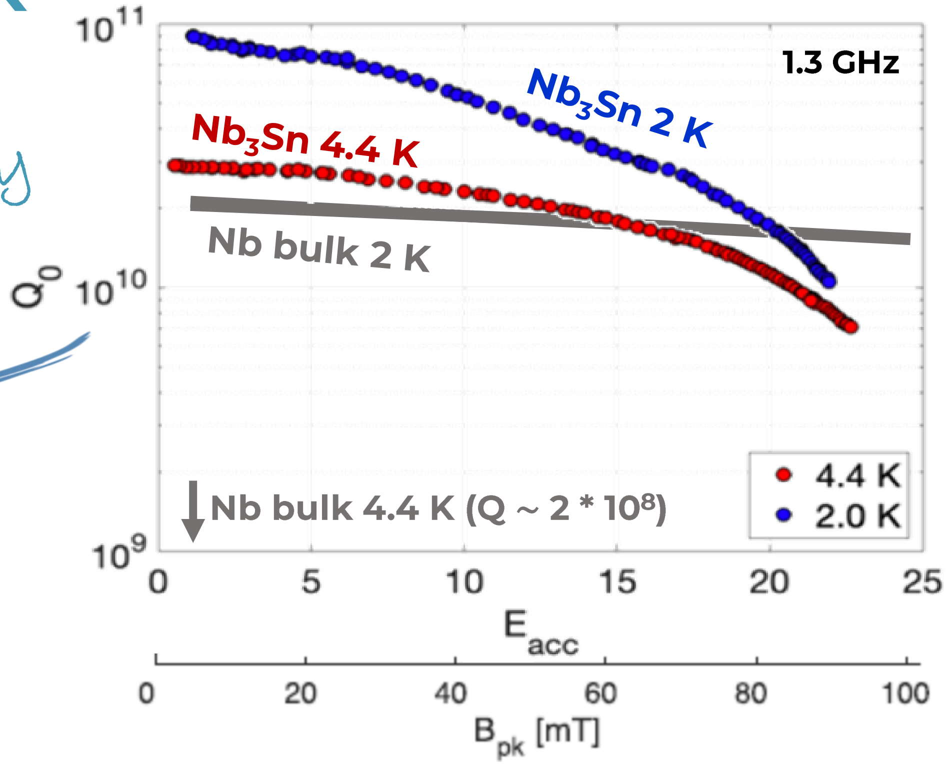
Vapour Tin Diffusion

Q_0 @ 4.4 K comparable to Nb bulk @ 2K



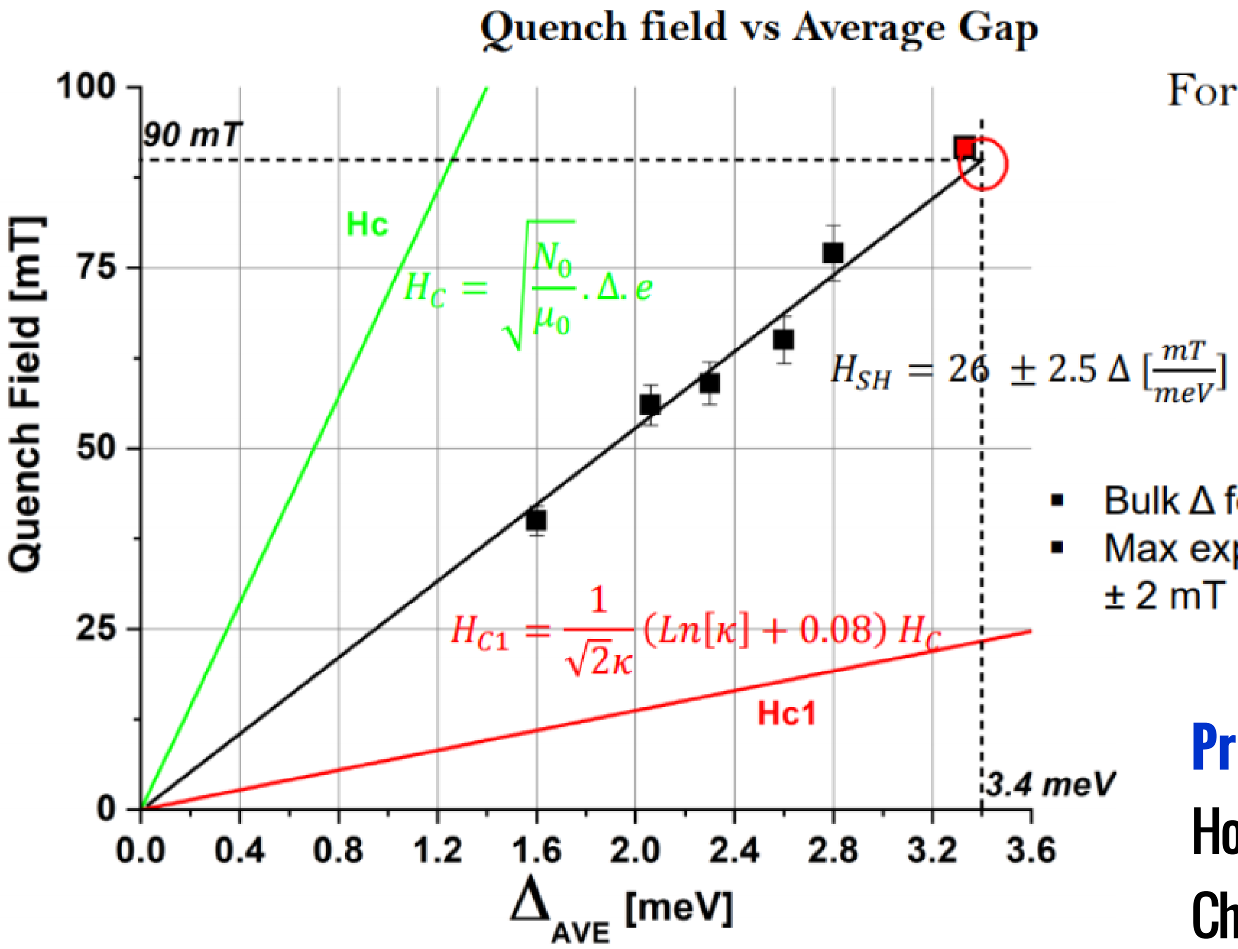
S. Posen, SRF 2019 proceedings

Low frequency
650 MHz



S. Posen, SRF 2019 proceedings (elaborated)

How far can Nb_3Sn go?



For large κ ($\lambda/\xi = 24$)

- Bulk Δ for Nb3Sn is 3.4 to 3.5 meV
- Max expected Quench field is ~ 91 ± 2 mT = 22 MV/m

Present results suggest we already reached the limit...

How is it possible change the H_{sh} slope?

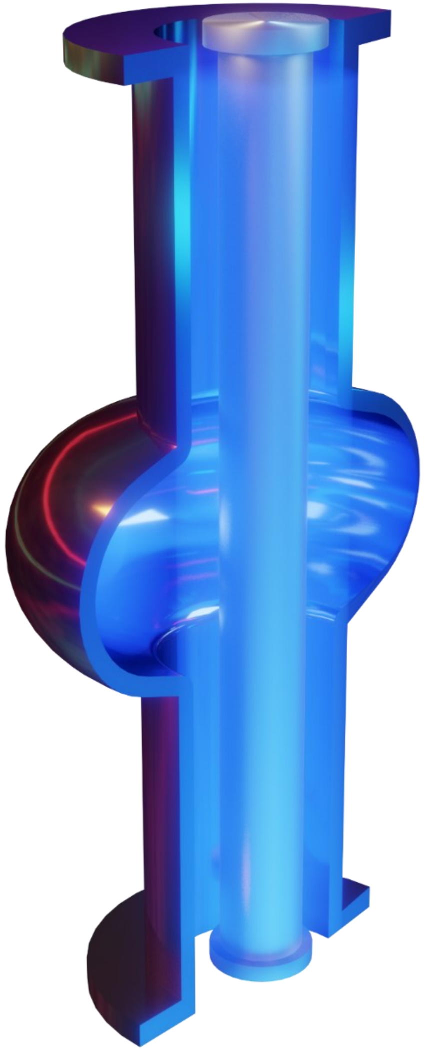
Changing the synthesis method?

T. Proslier, Correlations between Tunneling Spectroscopy and SRF cavity performances, TTC2020
https://indico.cern.ch/event/817780/contributions/3715517/attachments/1982513/3302032/TTC-Proslier.pdf

Different coating techniques for Nb_3Sn

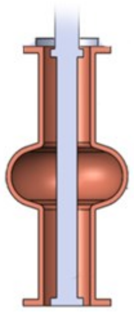
Diffusion	• Technique proved successful for magnet conductor application • Simple equipment compared to sputtering and CVD
Sputtering	• To sputter from a single target of correct stoichiometry (prepared by powder sintering) • Stoichiometry, Substrate Temperature, Deposition Rate, Deposition Thickness Can be varied independently
Co-sputtering	• Successful in synthesizing difficult materials like Nb_3Ge (highest Tc~23k),r V_3Si • Constituents are sputtered simultaneously onto a temperature controlled substrate • Stoichiometry dependent on relative positions of target & substrate (manipulated to get perfect stoichiometry) • Stoichiometry control difficult over large areas and if narrow stoichiometry range for A-15 phase
CVD	• MOCVD (*Metal Organic Chemical Vapour Deposition)*= CVD with metallorganic compound precursor • Precursor(s) in vapor phase chemically react on an heated substrate to grow a solid film • Deposition rate & structure of the film depend upon temperature & reagent concentration • Uniformity of temperature and flow of gaseous may be difficult with complex geometry

A-M Valente, SRF2017 Tutorials



Nb_3Sn on Cu Coatings @LNL

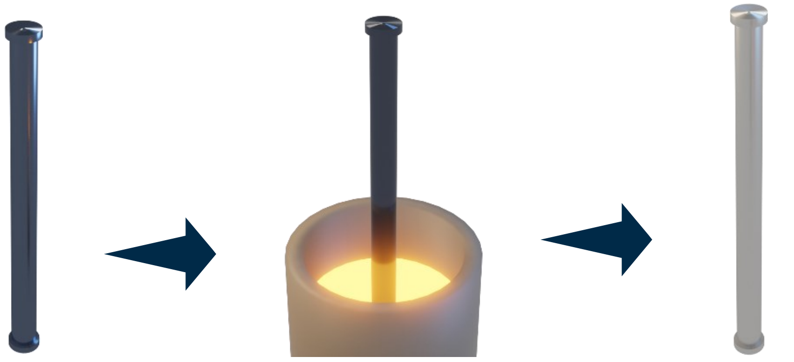
Nb_3Sn coatings: target production



Single target configuration easiest to scale onto elliptical geometry

Nb_3Sn cylindrical targets are not commercially available

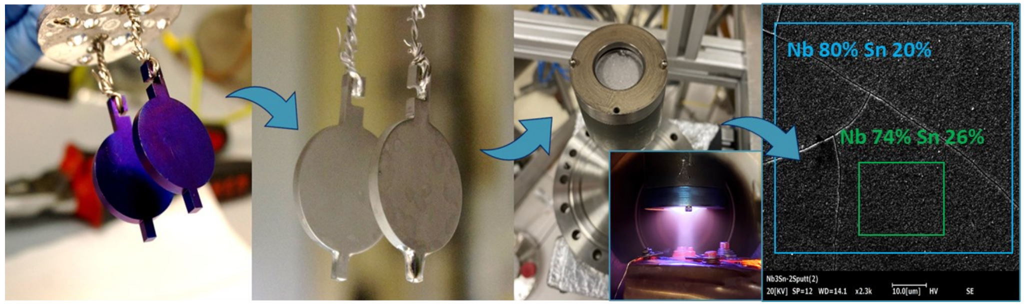
LNL Strategy for Nb_3Sn cylindrical targets production for 6 GHz cavities



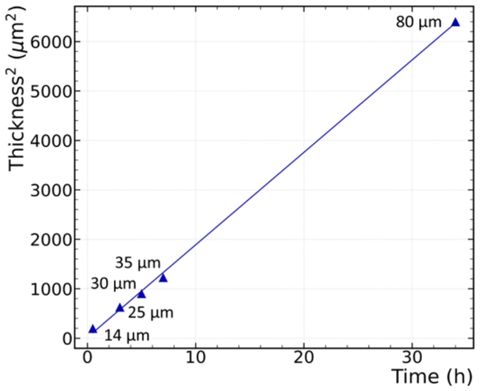
Raw Nb target

Dipping in liquid Sn

Nb_3Sn cylindrical target



Proof of concept

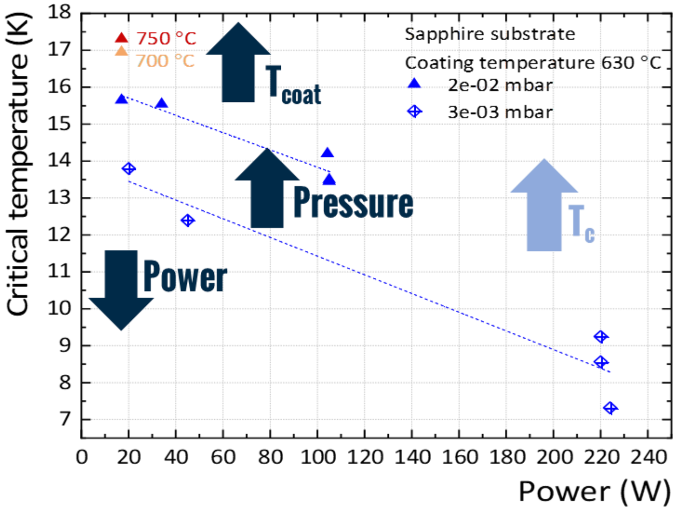


Nb_3Sn **thickness** related to **dipping time**

Possible **tin content modulation**

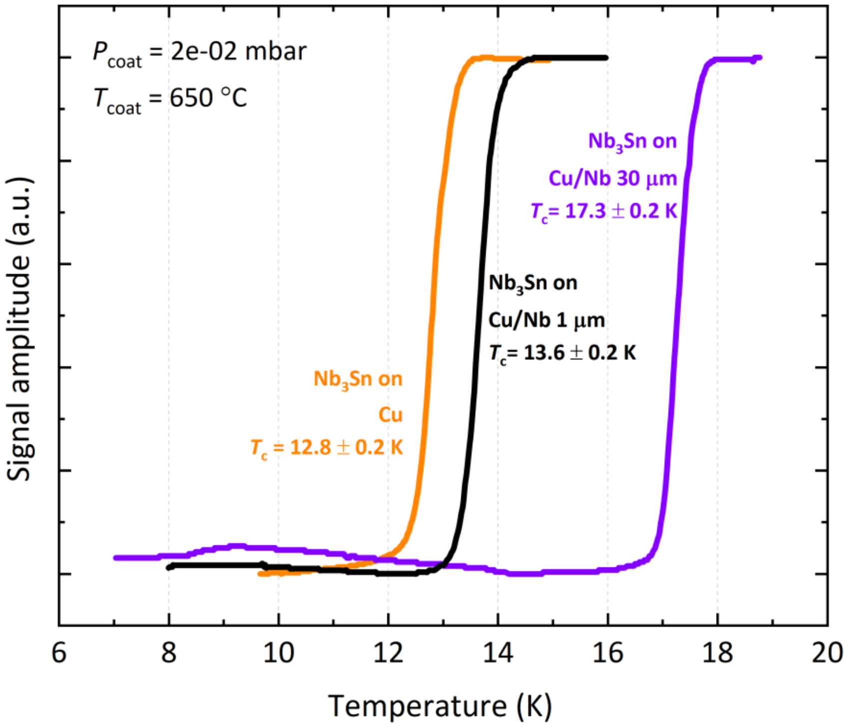
Nb_3Sn Coatings

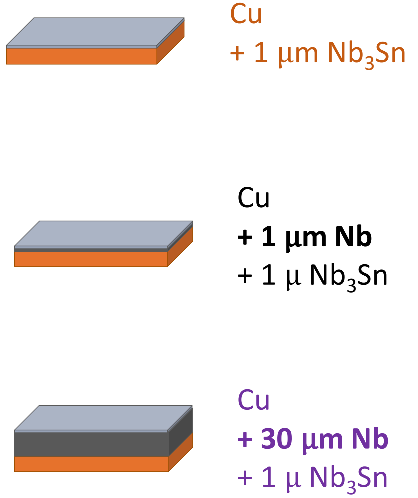
Long R&D phase on PVD Parameter Optimization



Optimized Coating Recipe

- Coating Parameters:
  - Pressure = $2*10^{-2}$ mbar
  - Power = 16 W
  - T substrate ≥ 600 C
- **Nb Thick Barrier Layer > 30 um**





Cu
+ 1 μm Nb_3Sn

Cu
+ 1 μm Nb
+ 1 μ Nb_3Sn

Cu
+ 30 μm Nb
+ 1 μ Nb_3Sn

A thick Nb buffer layer accommodates the Nb_3Sn coating

Nb substrate can be used to validate Nb_3Sn Coating Performances

First Nb_3Sn RF Results
(on a small Nb planar resonator)

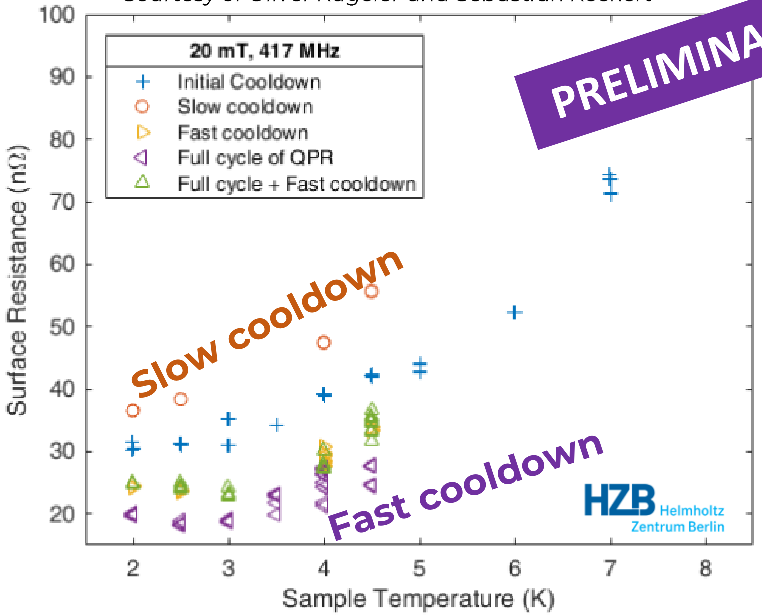
Rs of 23 nΩ
@ 4.5 K, 20 mT

Quench >70 mT
@ 4.5 K

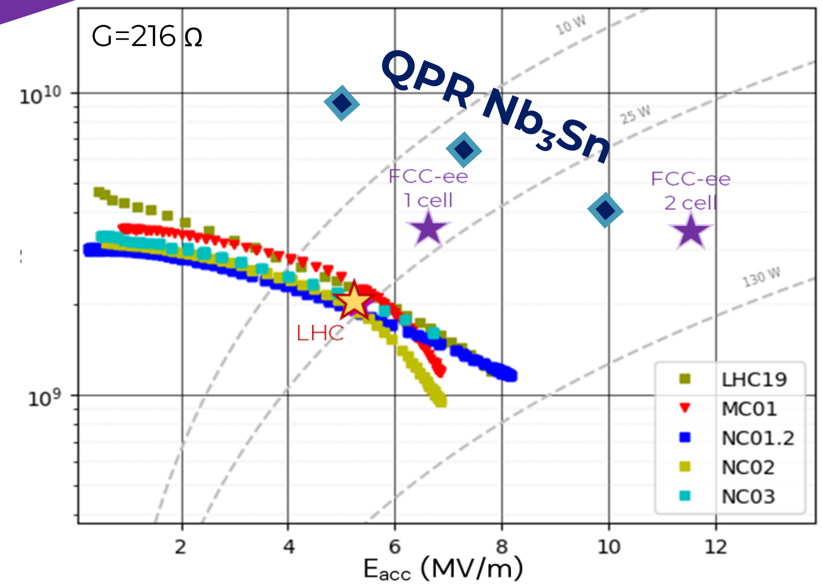
- **Nb_3Sn coating suffer flux trapping**
- **Cooldown procedure influence Rs**

Courtesy of Oliver Kugeler and Sebastian Keckert

PRELIMINARY RESULTS



LHC cavities Q vs E_{acc} @4.5 K



Equivalent to a Q of $9{\cdot}10^9$ @5 MV/m @4.5 K

Almost 1 order of magnitude better than LHC!!!

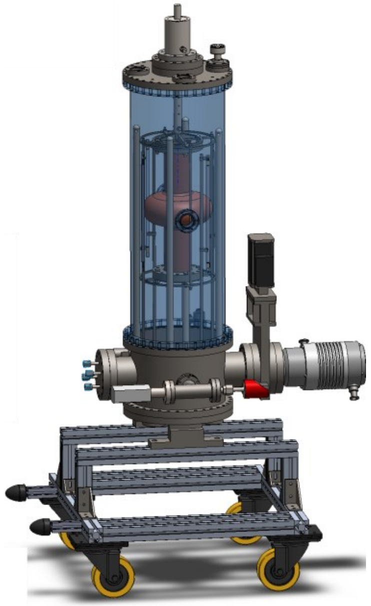
Room for improvement

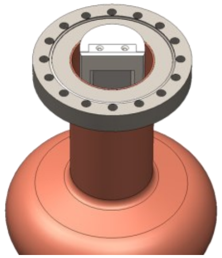
Nb_3Sn Path to Final Prototype

Nb_3Sn on bulk Nb to validate coating performances (2025)
on 1.3 GHz Elliptical Cavities (2025)

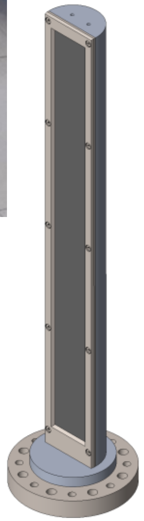
Develop Nb thick barrier/accommodation layer
on 1.3 GHz Elliptical Cavities (2025)
(proof of concept on 6 GHz cavities already done)

Nb_3Sn on Cu with thick Nb coating
on 1.3 GHz Elliptical Cavities (2026-2028)





- **1.3 GHz Vacuum system ready**
- **Magnetron source commissioned**



In parallel:

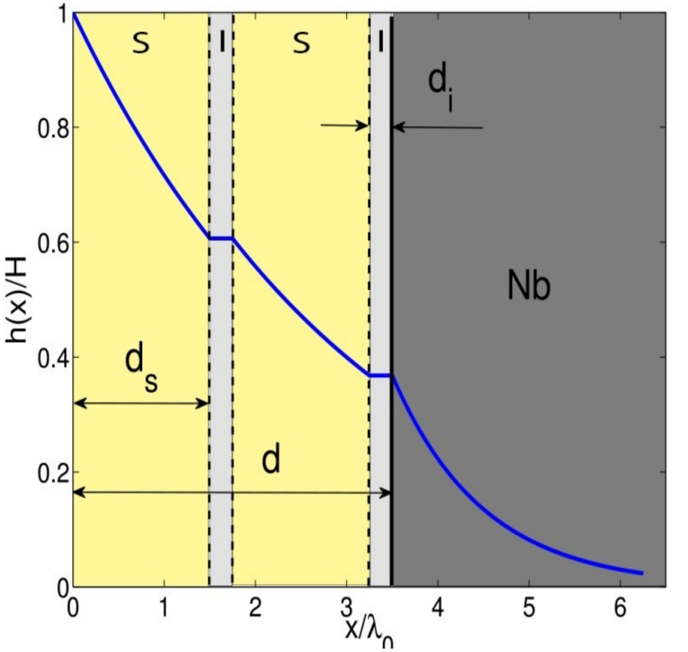
- *Study on alternative buffer layer* cea
- *Study on flux trapping* UKRI Science and Technology Facilities Council HZB Helmholtz Zentrum Berlin

SIS Multilayer

SIS Multilayer

Taking advantage of the high – Tc superconductors with much higher H_c without being penalized by their lower Hc1…

Alex Gurevich, Appl. Phys. Lett. 88, 012511 (2006)



$$B_{c1b} = \frac{\phi_0}{4\pi\lambda^2}\left(\ln\frac{\lambda}{\xi} + 0.497\right), \qquad d \gg \lambda,$$

$$B_{c1} = \frac{2\phi_0}{\pi d^2}\left(\ln\frac{d}{\xi} - 0.07\right), \qquad d \ll \lambda.$$

Alex Gurevich, Appl. Phys. Lett. 88, 012511 (2006)

Multilayer coating of SC cavities:
alternating SC and insulating layers with $d < \lambda$

Higher T_c thin layers provide magnetic screening of the Nb SC cavity (bulk or thick film) without vortex penetration

- Strong increase of H_{c1} in films allows using RF fields > H_c of Nb, but lower than those at which flux penetration in grain boundaries may become a problem=> no transition, no vortex in the layer
- high H_{C1} applied field is damped by each layer
- insulating layer prevents Josephson coupling between layers
- applied field, i.e. accelerating field can be increased without high field dissipation
- Strong reduction of BCS resistance (ie high Q_0) because of using SC layers with higher T_c, Δ (Nb_3Sn, NbN, etc)

Possibility to move operation from 2K to 4.2K

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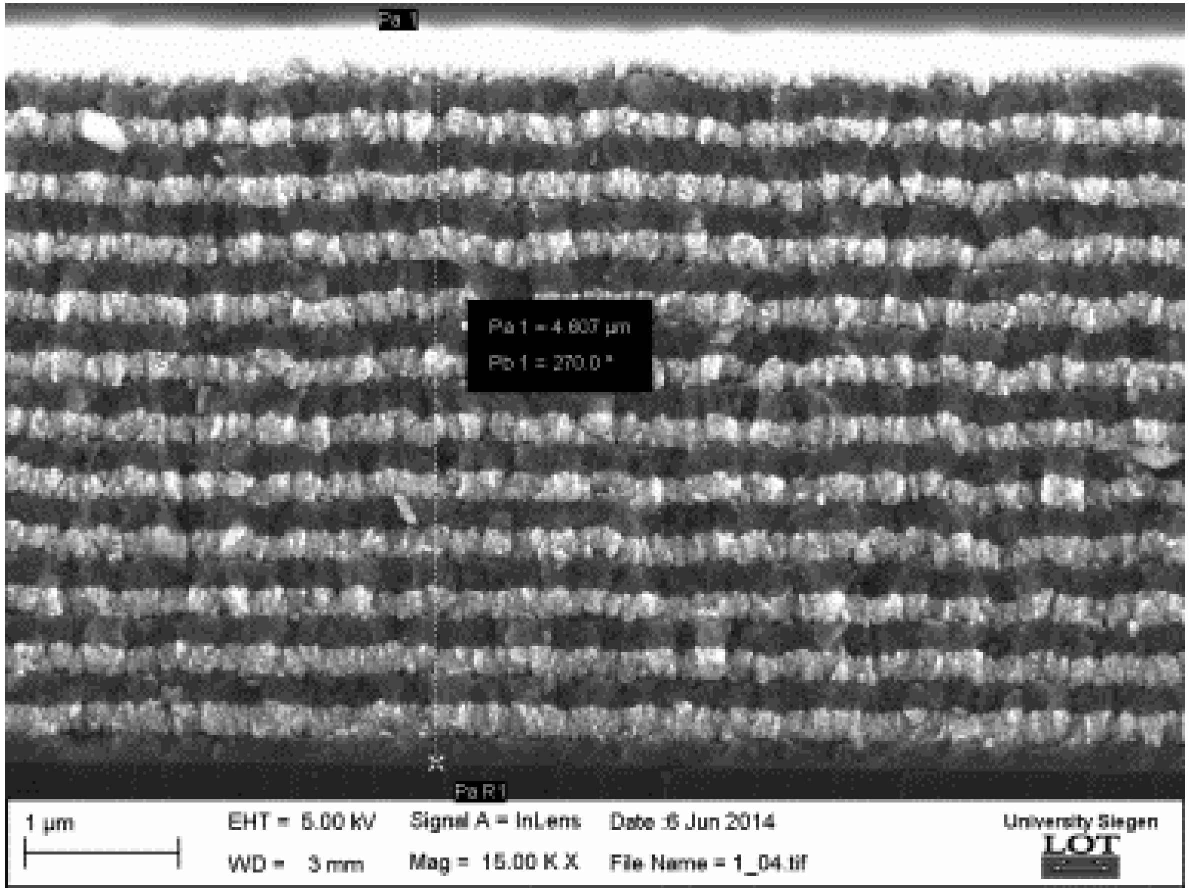
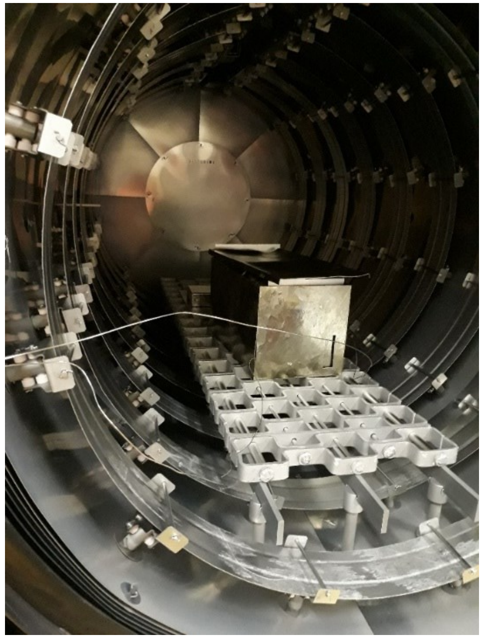
SIS First Attemps

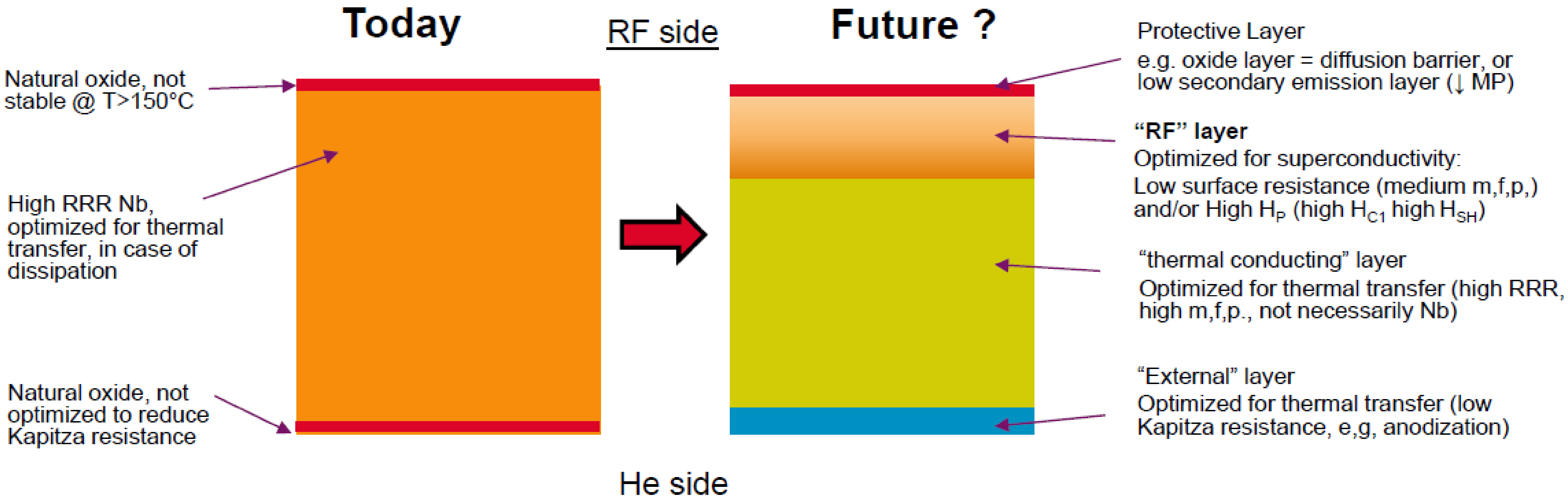


NbN - Nb multilayer @ Siegen University



Single Cell 1.3 GHz in a titanium box after ALD deposition of Al2O3 @ CEA

In the future?



Today

RF side

Future ?

Natural oxide, not stable @ T>150°C

High RRR Nb, optimized for thermal transfer, in case of dissipation

Natural oxide, not optimized to reduce Kapitza resistance

Protective Layer
e.g. oxide layer = diffusion barrier, or low secondary emission layer (↓ MP)

“RF” layer
Optimized for superconductivity:
Low surface resistance (medium m,f,p,) and/or High H_P (high H_{C1} high H_{SH})

“thermal conducting” layer
Optimized for thermal transfer (high RRR, high m,f,p., not necessarily Nb)

“External” layer
Optimized for thermal transfer (low Kapitza resistance, e,g, anodization)

He side

C. Antoine, CEA Saclay

Concluding remarks

- Niobium is getting close to its ultimate limits
- Superconducting cavities are dominated by their surface quality (Niobium AND other SC !)
- H_{SH} difficult to reach in real "accelerating cavities" (low T, large scale cavity fabrication, surface defects,…)
- Many long-standing problems of condensed matter physics and non-equilibrium superconductivity will have to be addressed to understand nonlinear surface resistance under strong rf fields
- Renewed activity on bulk-like Nb films (cost issues) and high H_{SH} SC e.g. Nb_3Sn or NbN (higher performances)
- ML structures seem to be a promising way to go beyond Nb for accelerator cavities Possibility to move from 2K to 4.2K: huge cost saving on refrigeration
- Multi-parameter materials optimization is required to revel the full SRF performance potential
- Look for higher Q_0, not only accelerating gradients

The interest & efforts for new materials research for SRF cavities application has been re-lighted and is gaining traction. Still a lot of work ahead!

Technological Revolution(s) In Perspective For SRF Cavities …

A-M Valente, SRF2017 Tutorials

Recommanded Literature

- R. Padamsee, J. Knobloch and T. Hays – « RF Superconductivity for Accelerators », Wiley-VCH, 2008
- J. P. Turneaure, J. Halbritter, and H. A. Schwettman. « The surface impedance of superconductors and normal conductors: The Mattis-Bardeen theory. » Journal of Superconductivity 4.5 (1991): 341-355
- A. Gurevich « Theory of RF superconductivity for resonant cavities. » Superconductor Science and Technology, 30(3), 034004 (2017).
- SRF Tutorials (https://jacow.org/Main/Proceedings?sel=SRF **and websites of the SRF conferences)**