20 Years of Experience with the Nb/Cu Technology for Superconducting Cavities and Perspectives for Future Developments

S. Calatroni for the
CERN SRF community

With many thanks to all
the SRF-films community

Introduction

- Why films (Nb films are the main subject of the talk)
- State of the art: high-field and low-beta applications
- Topics that need further R&D in order to be understood:
  - Effect of roughness
  - Effect of film structure
  - Effect of hydrogen
  - Effect of surface oxidation
- Conclusions and perspectives

Why films

- Advantages (primary objectives)
  - Thermal stability
  - Cost
  - Innovative materials (see MoP12, TuA13)
- Advantages (learned from experience)
  - Optimisation of R_{BCS} at 4.2 K (sputtered niobium films)
  - Reduced sensitivity to earth magnetic field
- Disadvantages (known from the beginning)
  - Fabrication and surface preparation (at least) as difficult as for bulk
- Disadvantages (learned from experience)
  - Steep R_{res} increase with RF field (sputtered niobium films)
  - Deposition of innovative materials is very difficult

State-of-the-art 20 years ago

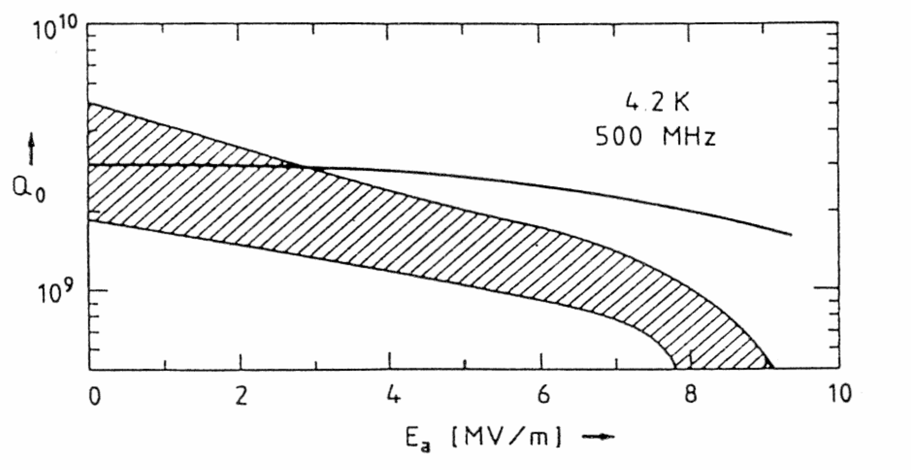


Figure 1. Typical $Q(E_a)$ curves of sheet metal (line) and sputter coated cavities (hatched).

LEP 272 Nb/Cu cavities 352 MHz (ACCEL, Ansaldo, CERCA)

LHC 16 Nb/Cu cavities 400 MHz Industrially made by ACCEL, who are also developing 500 MHz Nb/Cu cavities

See also MoA05, TuP30

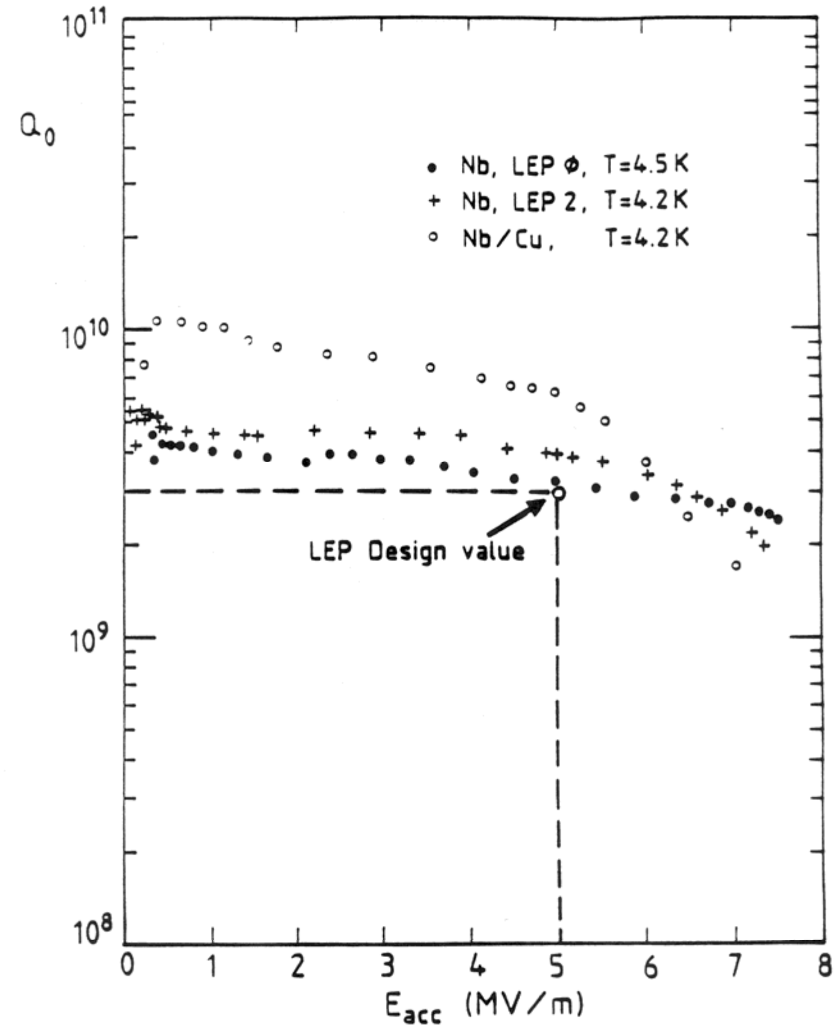
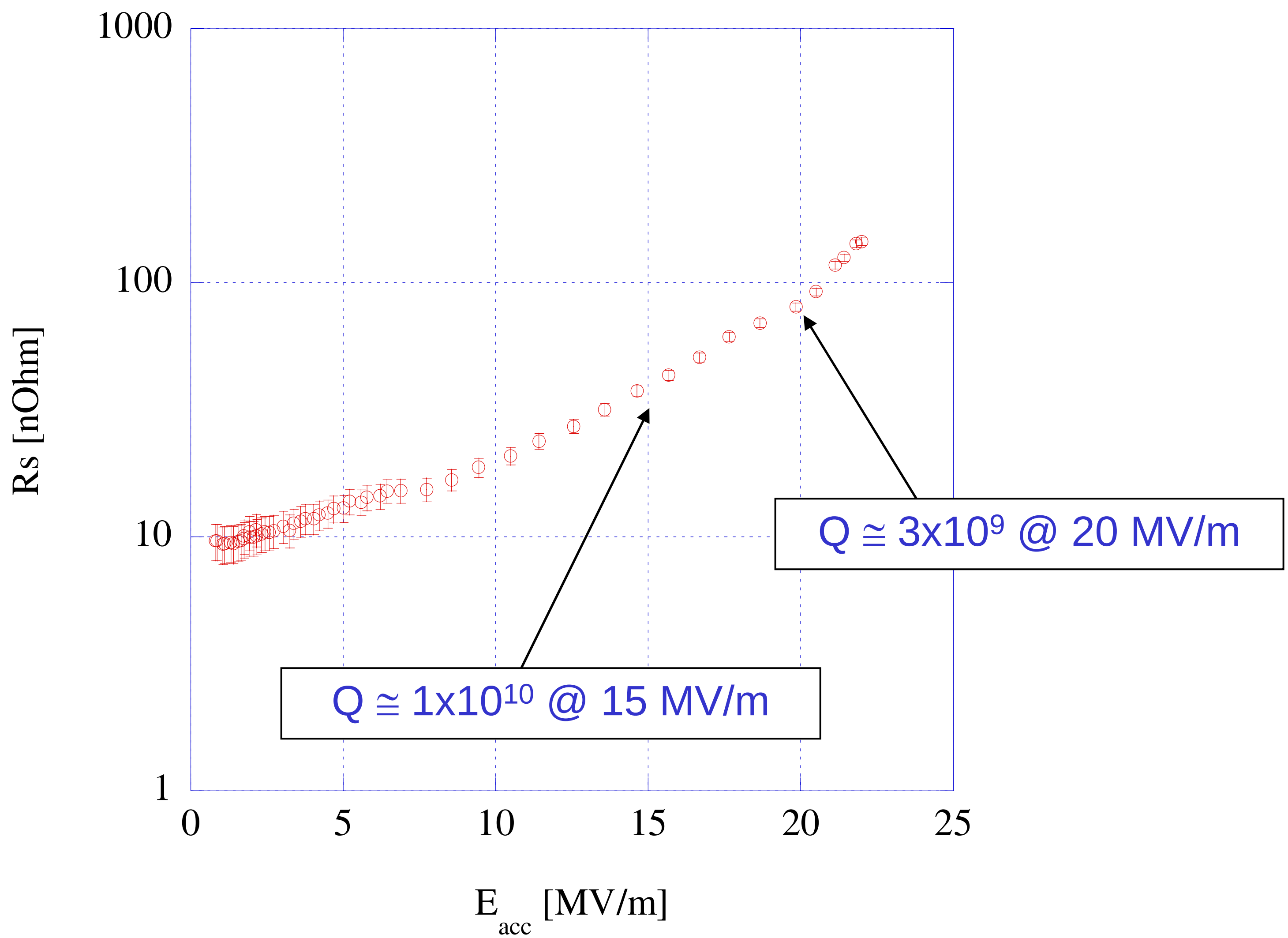


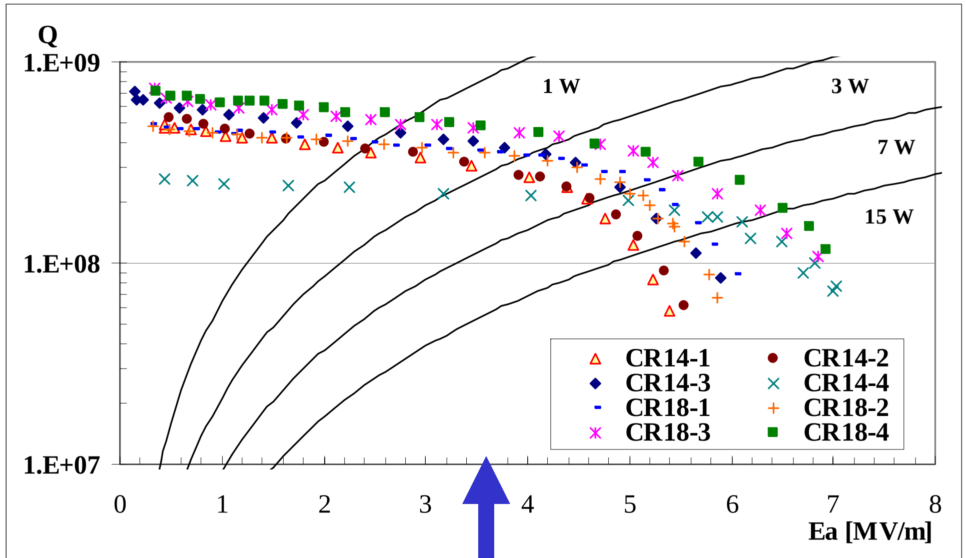
Fig. 2 Dependence of Q values on E_{acc}. Final values for 4-cell cavities LEP 0 and LEP 2 with $H_{ext} \leq 160$ mG and for a 350 MHz 4-cell Cu-cavity with a magnetron sputtered Nb-layer.

State-of-the-art at 1500 MHz – 1.7 K – single cell



28 MV/m reached in large cryostat

State of the art – QWR Nb-sputtered resonators for ALPI



Q-curves of QWRs installed in 2003

- Upgrading of ALPI medium β section, by replacement of electroplated Pb with a sputtered Nb film, completed in 2003; 46, β= 0.11 and 8, β= 0.13, 160 MHz Nb/Cu QWRs routinely used for beam acceleration. Average operational Ea > 4.4 MV/m @ 7W, very reliable and easy to put into operation, no deterioration with time. Some of them are reliably locked up to 6.5-7.3 MV/m without necessity of fast or "soft" tuners and/or strong overcoupling. Frequency not affected by changes in the He bath pressure (Δf <0.01 Hz/mbar!) .

- Nb Sputtering technology used at LNL also for producing end plates of both full Nb QWRs and SRFQs. See also MoP04.

Nb/Cu films – Philosophy

- There are two categories of films
  - Films which are intrinsically films
    - Thin, small grains, microstrained, under stress
    - Problems: defects & microstructure, (impurities), surface state
    - Examples: magnetron sputtered films on oxidised copper
  - The general trend is to move towards films which are bulk-like
    - Thick, large grains
    - Problems: hydrogen, surface quality
    - Examples: high-energy deposition techniques, annealed films, (Nb Cu-clad)

Of course a film from one family may as well present all the problems typical of the other family…

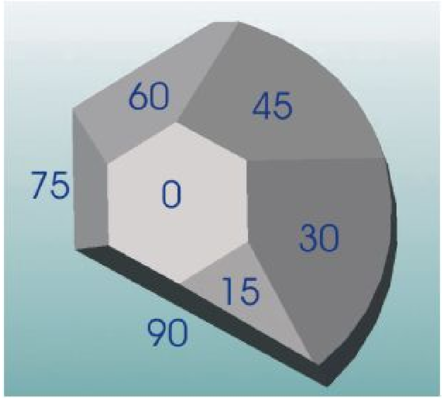
Research lines around the world

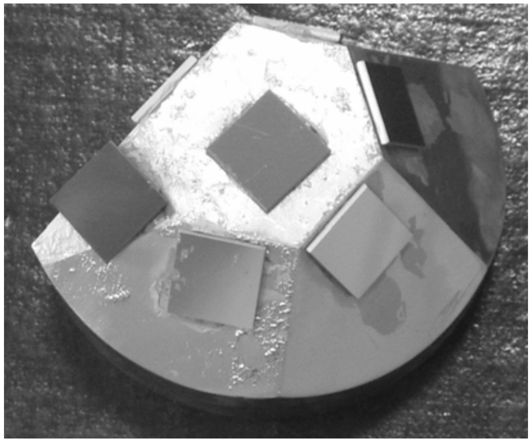
- Effect of substrate roughness
  - Non uniform coating, H enhancement (demagnetization), increased granularity
  - ➢ Optimisation of substrate preparation (electropolishing), study of angle-of-incidence effects, conformal coatings.
- Film structure – defects
  - H_{c1} reduction, hysteretic losses
  - ➢ Towards a bulk-like film: bias sputter deposition, high-energy deposition techniques, high-temperature annealing of films
- Effect of hydrogen
  - Hydrides formation
  - ➢ Measurements of H_2 contents, outgassings
- Oxidation
  - Localized states, corrosion of grain boundaries
  - ➢ Al_2O_3 cap layers

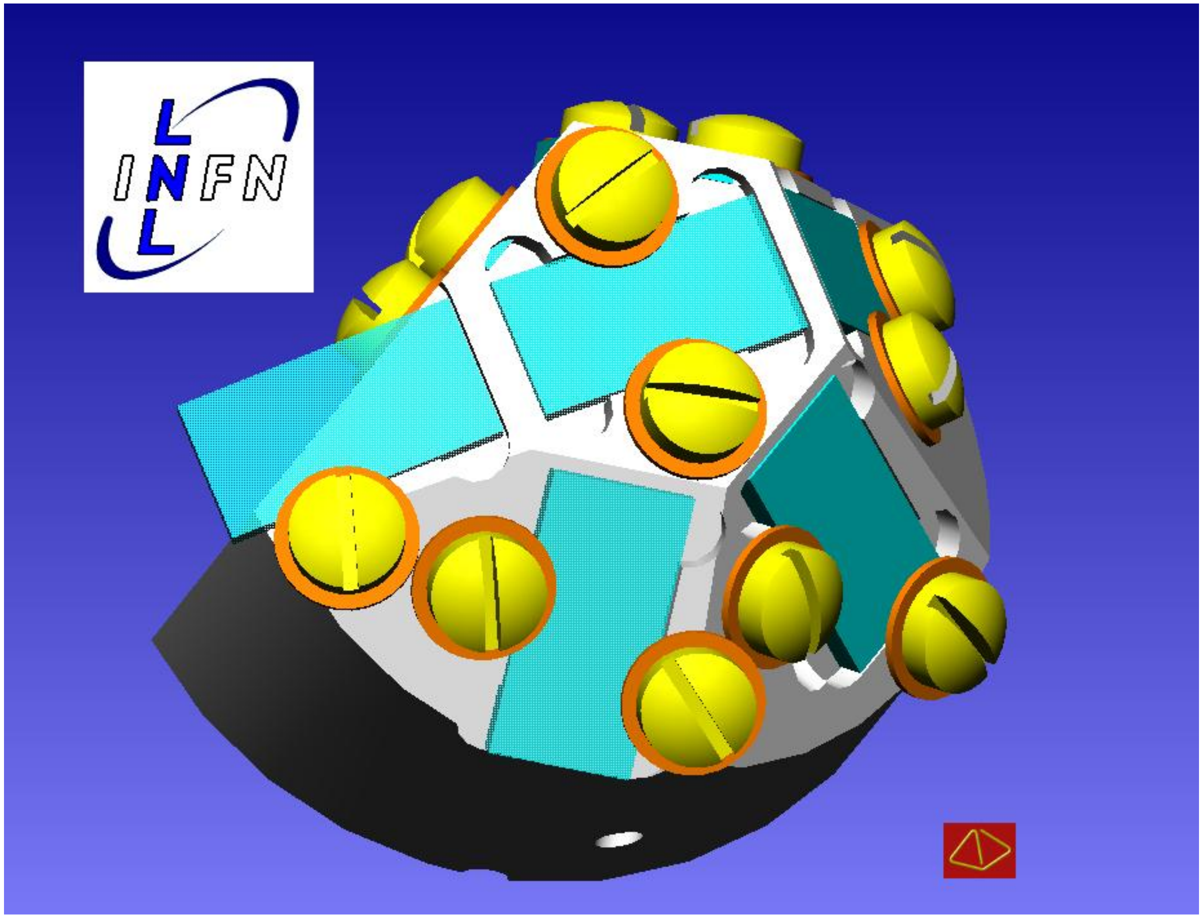
Research lines around the world

- Effect of substrate roughness
  - Non uniform coating, H enhancement (demagnetization), increased granularity
  - ➢ Optimisation of substrate preparation (electropolishing), study of angle-of-incidence effects, conformal coatings.
- Film structure – defects
  - H_{c1} reduction, hysteretic losses
  - ➢ Towards a bulk-like film: bias sputter deposition, high-energy deposition techniques, high-temperature annealing of films
- Effect of hydrogen
  - Hydrides formation
  - ➢ Measurements of H_2 contents, outgassings
- Oxidation
  - Localized states, corrosion of grain boundaries
  - ➢ Al_2O_3 cap layers

Nb coatings at various incidence angles (INFN –LNL)



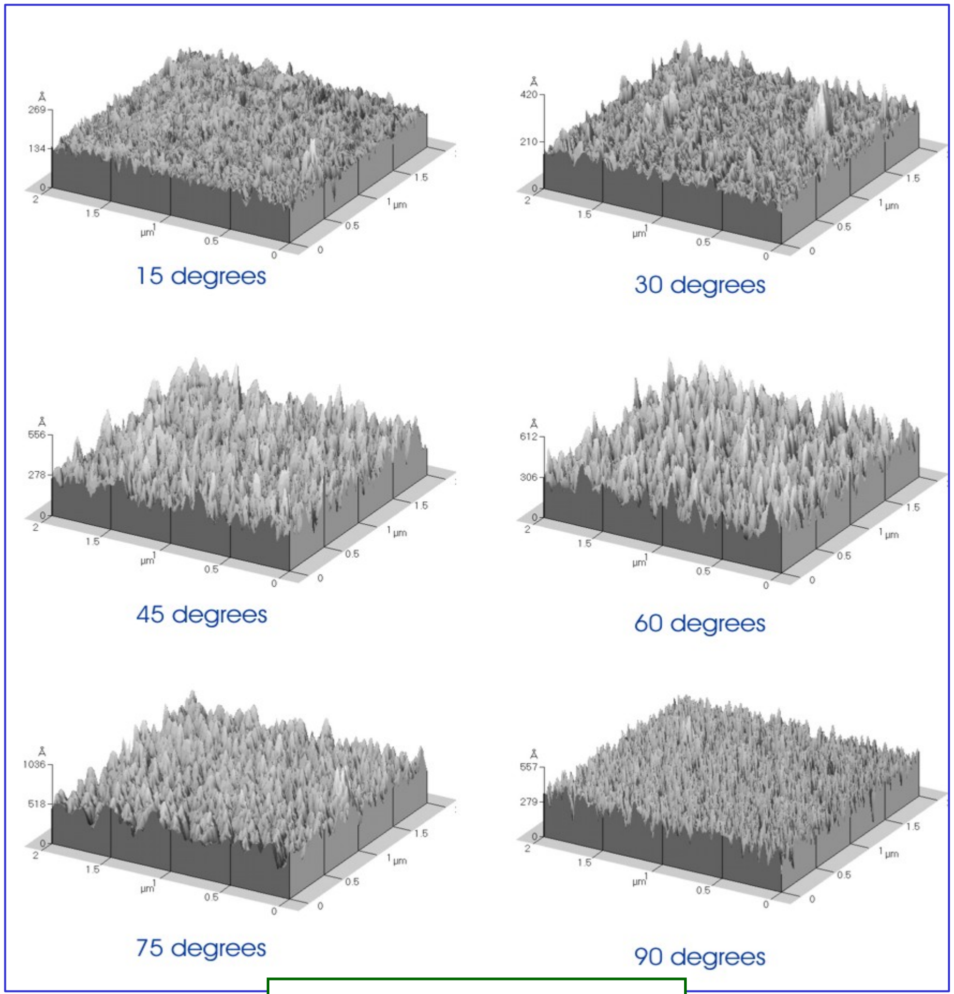




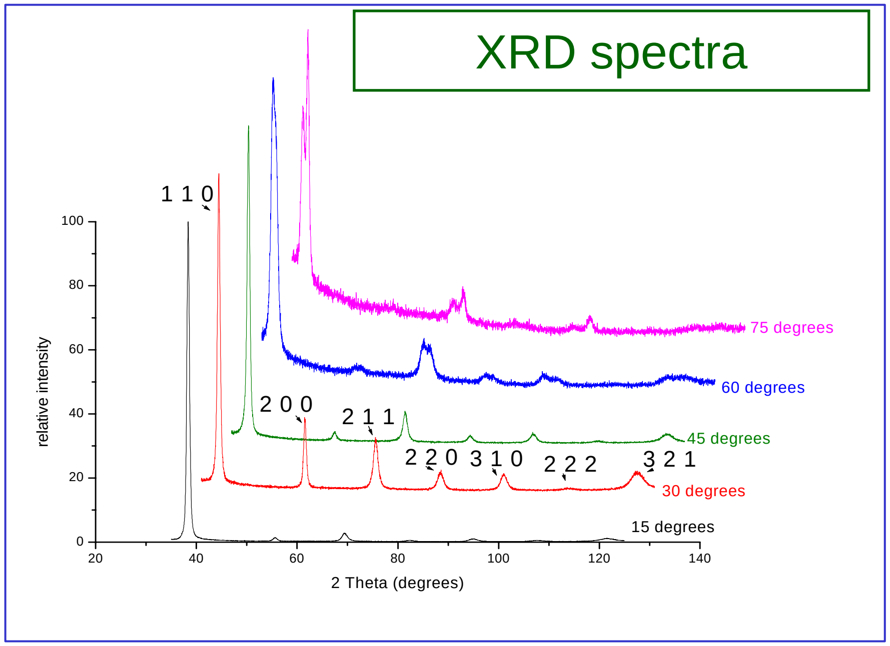
From: V. Palmieri, D. Tonini – INFN-LNL

See also TuA12

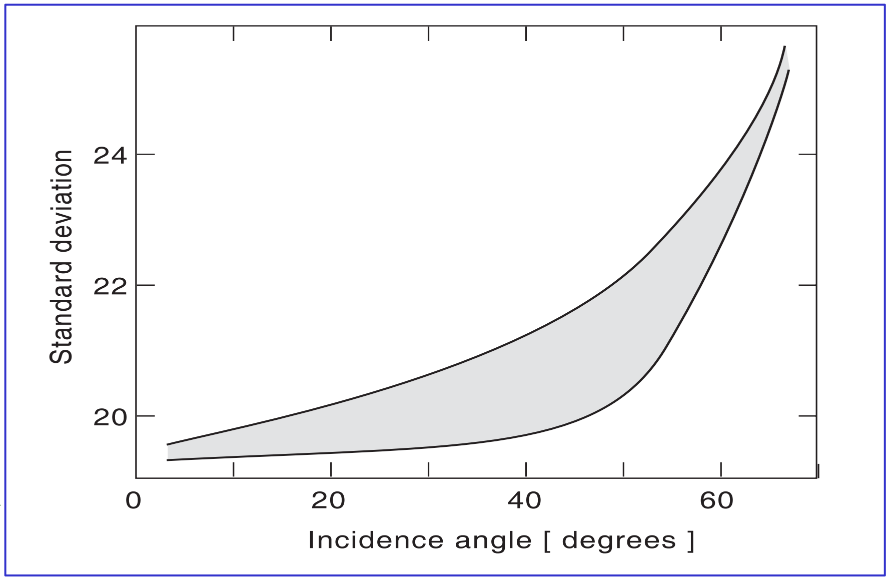
Variation of properties with incidence angle



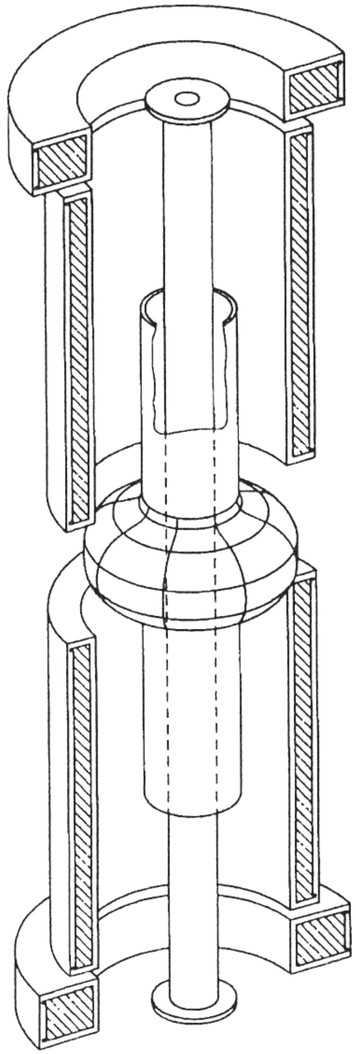
AFM roughness



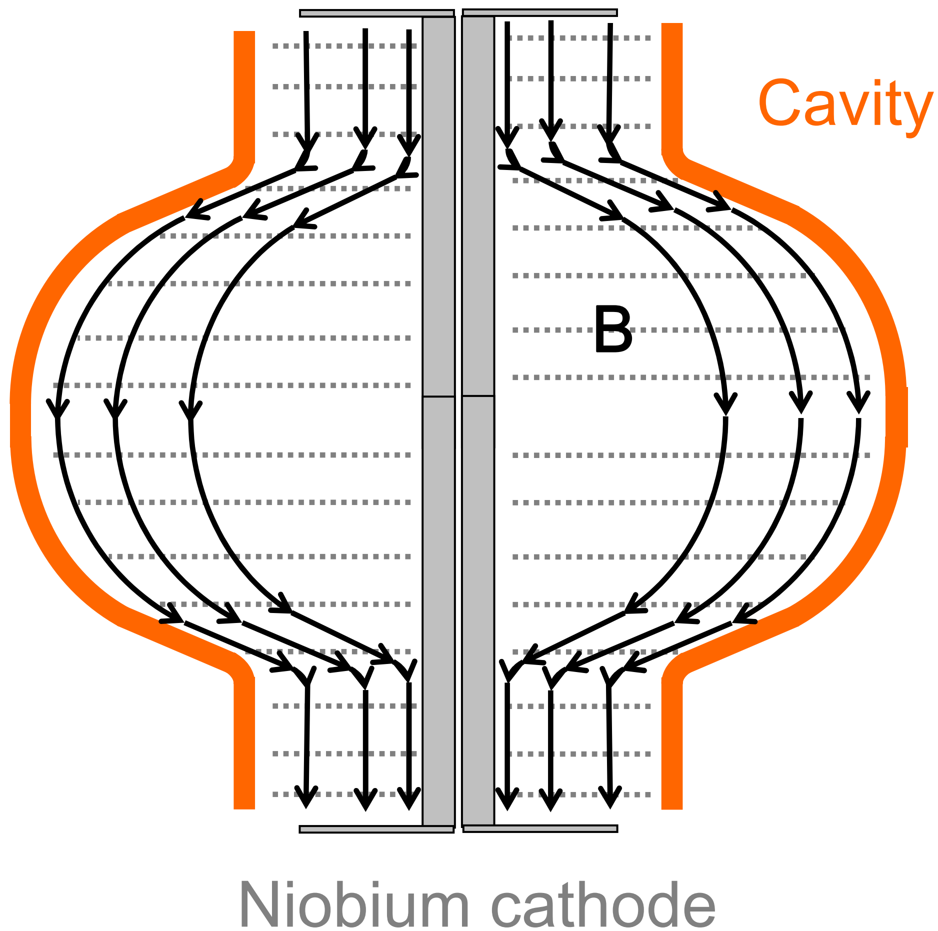
Std.Dev. of grey levels of SEM images
(CERN 1999)



Angle of incidence – post magnetron

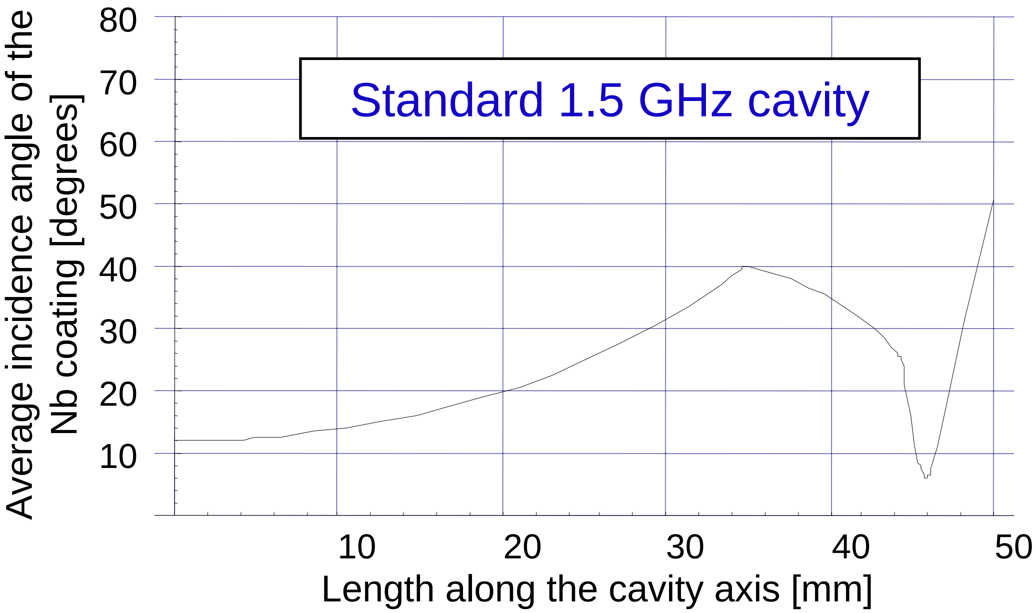


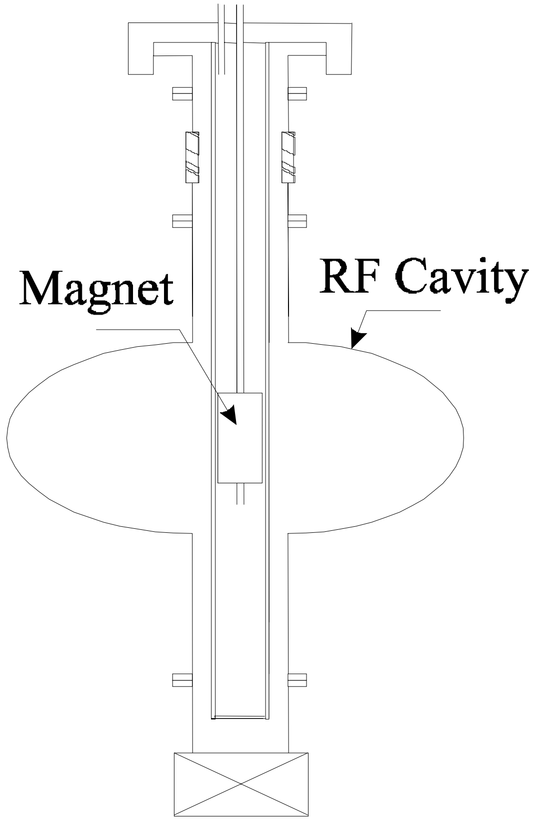
Magnetic field lines follow the cavity shape

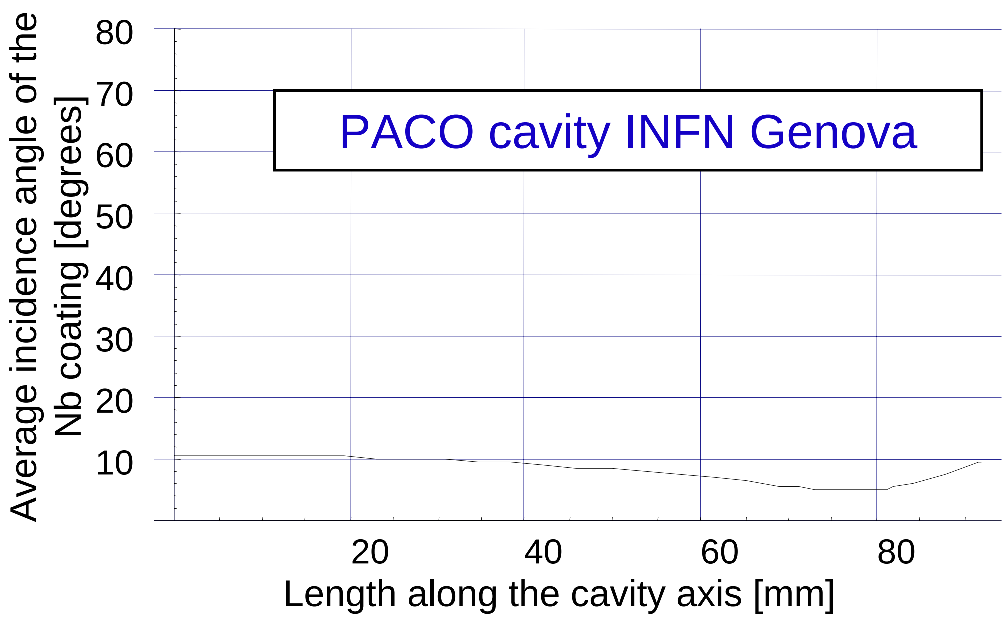


V. Palmieri, R. Preciso, V.L. Ruzinov, S. Yu. Stark, “A DC Post-magnetron configuration for niobium sputtering into 1.5 GHz Copper monocells”, Presented at the 7th Workshop on RF Superconductivity

Angle of incidence in spherical cavity





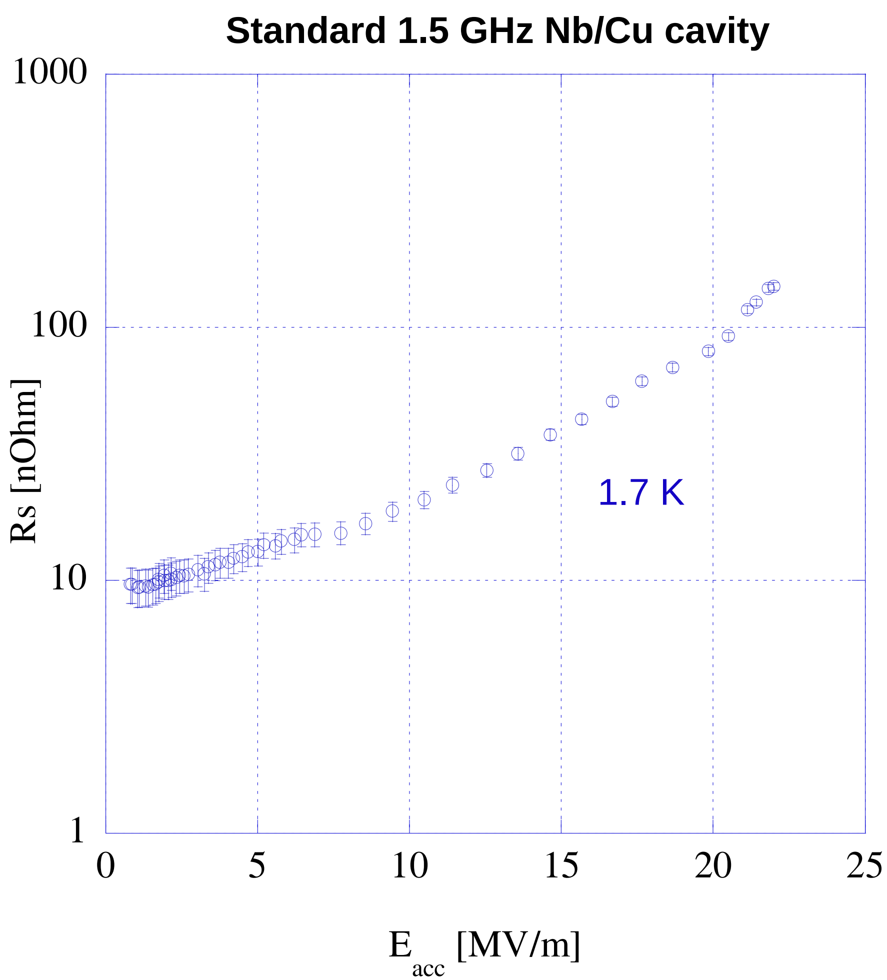


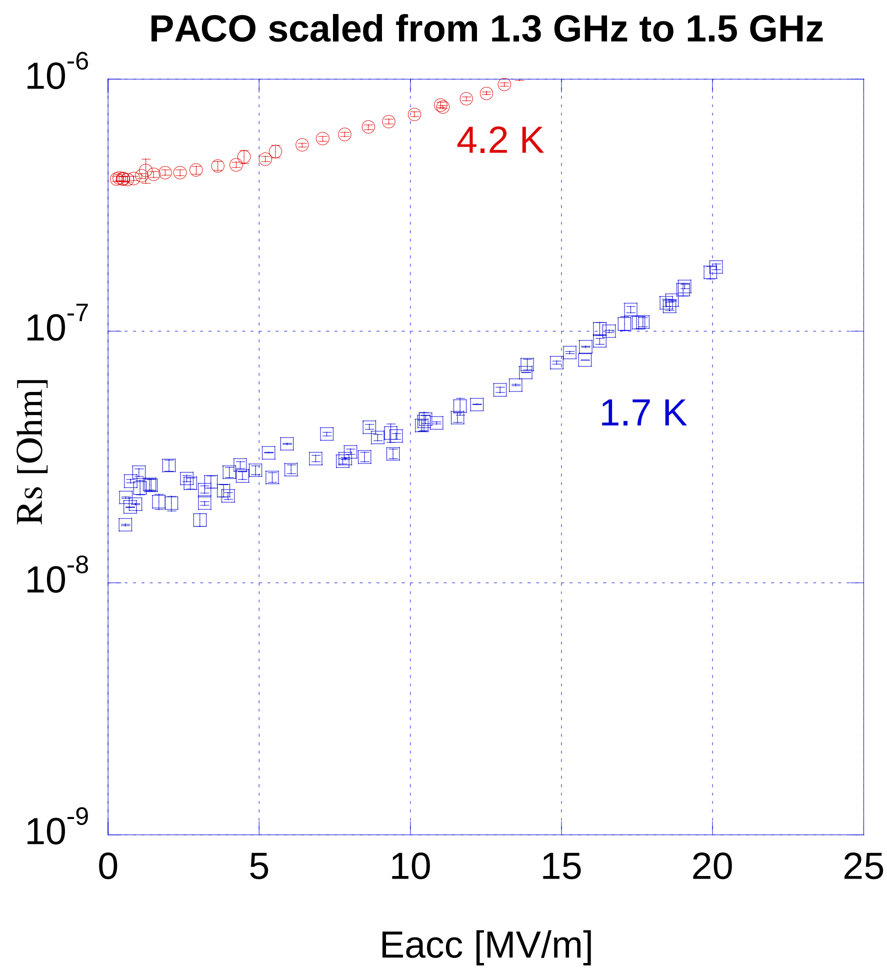
The cathode is not point-like

$\Rightarrow$

The incidence angle is always >0

Surface resistance of spherical cavity



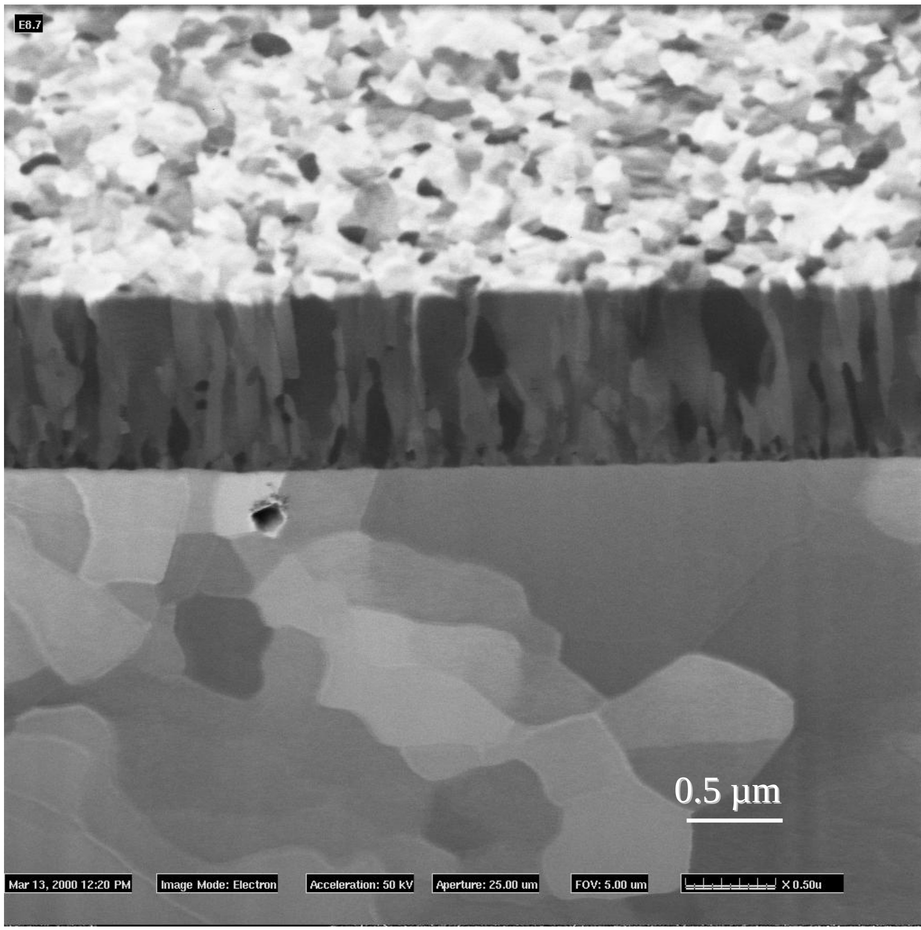


Reducing the angle of incidence does not change $R_s(E)$. However, the angle is always greater than zero, and whether this is creating any effect is only matter of speculation – for the time being

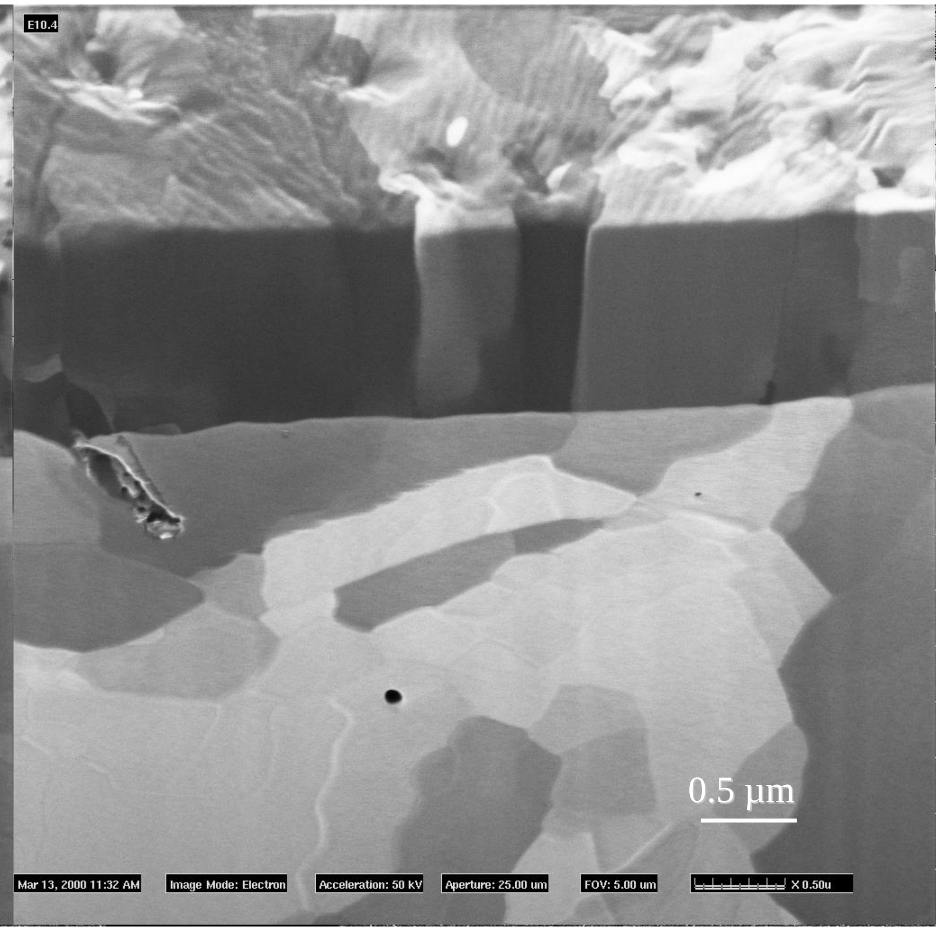
Film structure – FIB cross sections

Grain size with Focussed Ion Beam micrographs

Standard films



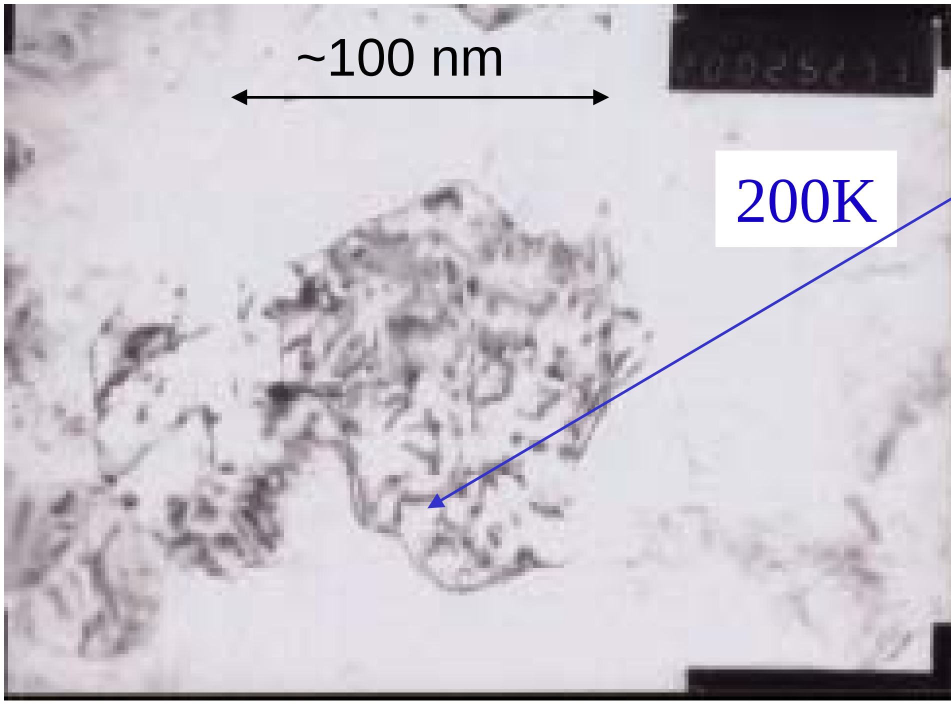
Oxide-free films

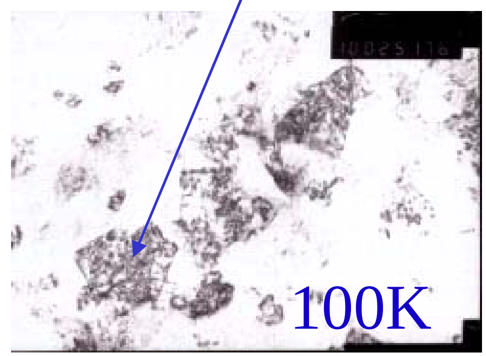


Courtesy: P. Jacob - EMPA

Defects – TEM views

Crystallographic defects

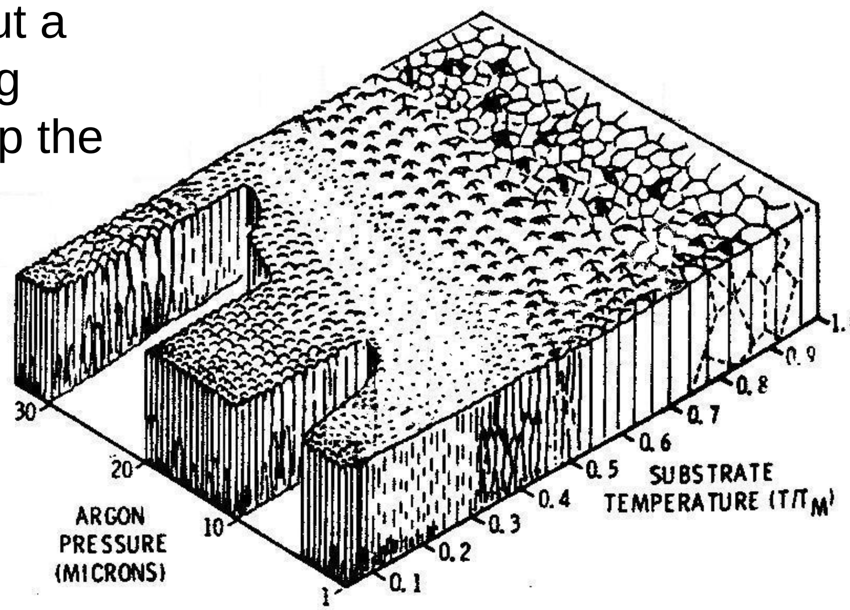




High-energy deposition techniques

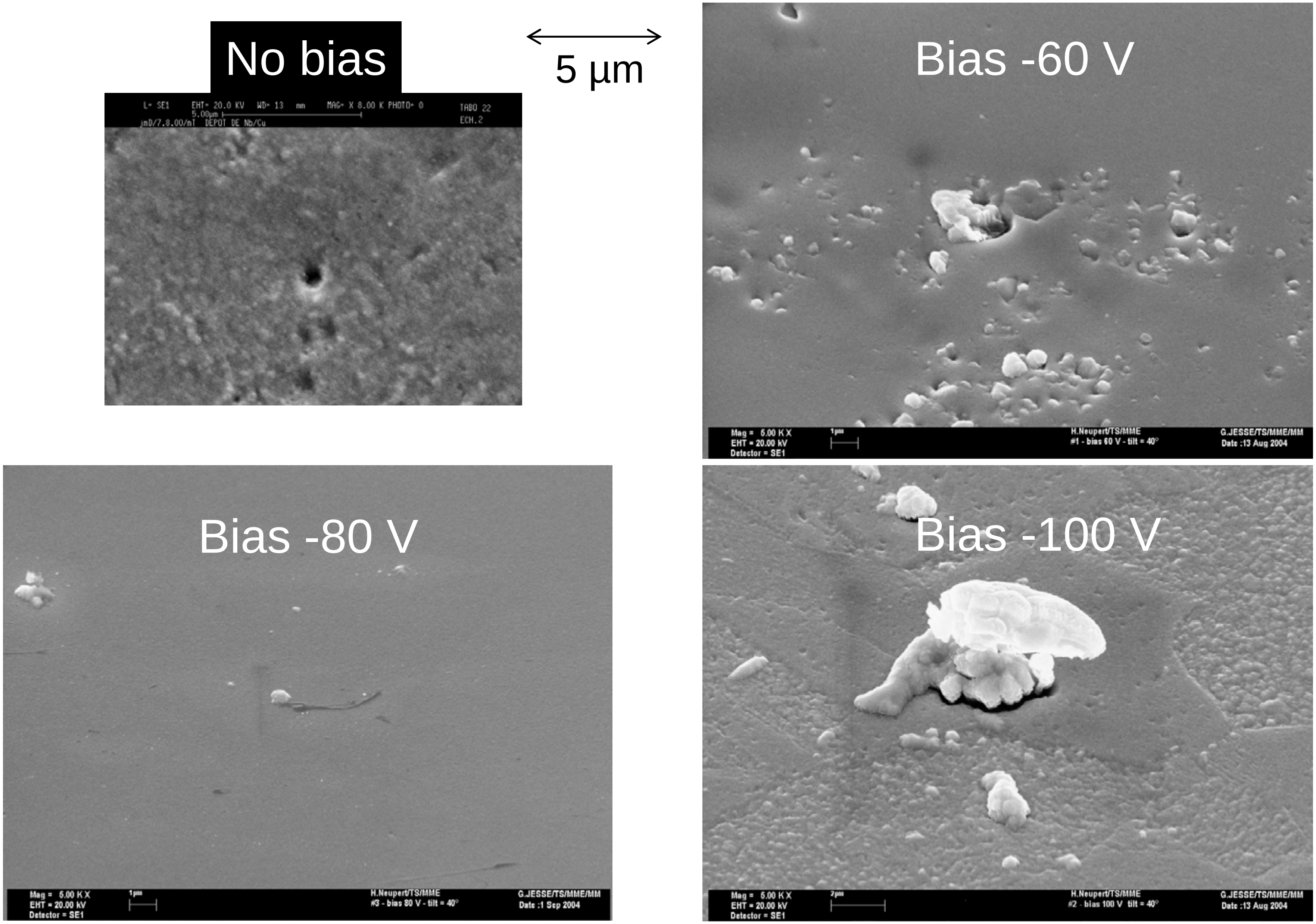
- Crystalline defects, grains connectivity and grain size may be improved with an higher substrate temperature which provides higher surface mobility (important parameter is $T_{substrate}/T_{melting\_of\_film}$)
- However the Cu substrate does not allow heating

➢ The missing energy may be supplied by ion bombardment
  - In bias sputter deposition a third electron accelerates the noble gas ions, removing the most loosely bound atoms from the coating, while providing additional energy for higher surface mobility
  - Other techniques allow working without a noble gas, by ionising and accelerating directly the Nb that is going to make up the coating
  - These techniques allow also to obtain "conformal" coatings that follow the surface profile better filling voids.

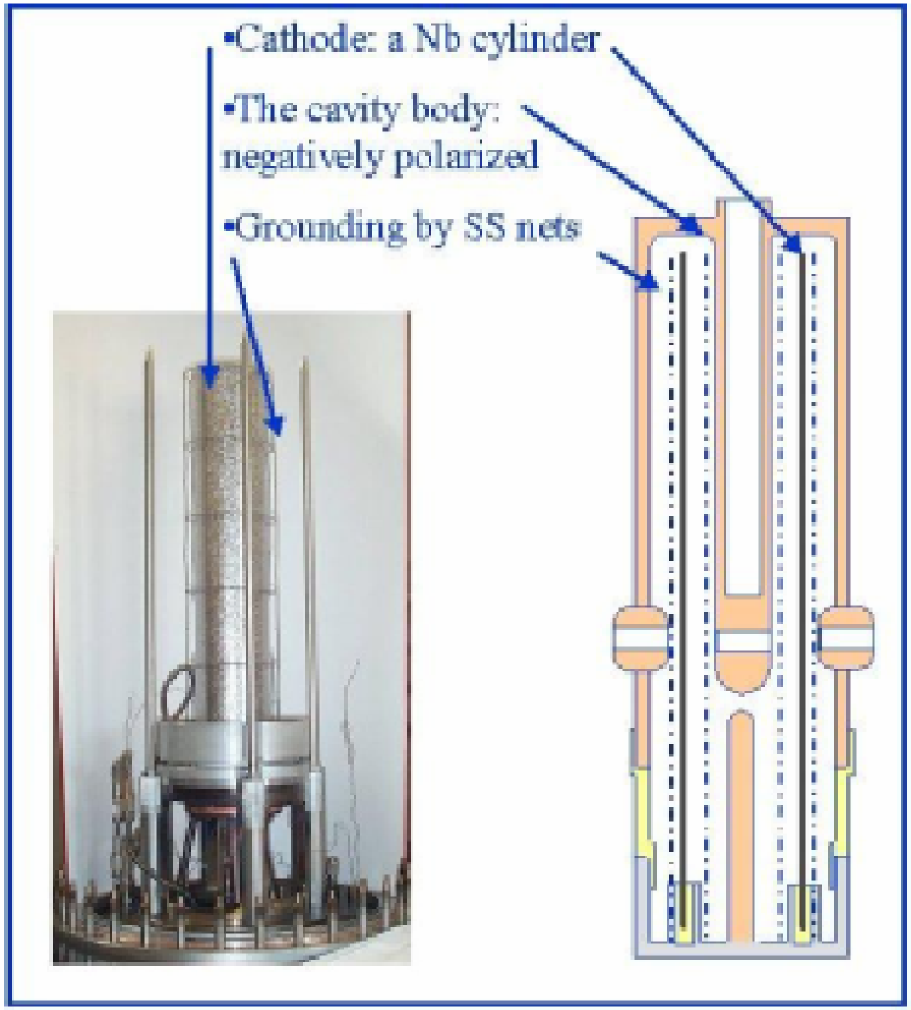


"Structure Zone Model"

Nb/Cu bias deposition – First SEM images at CERN

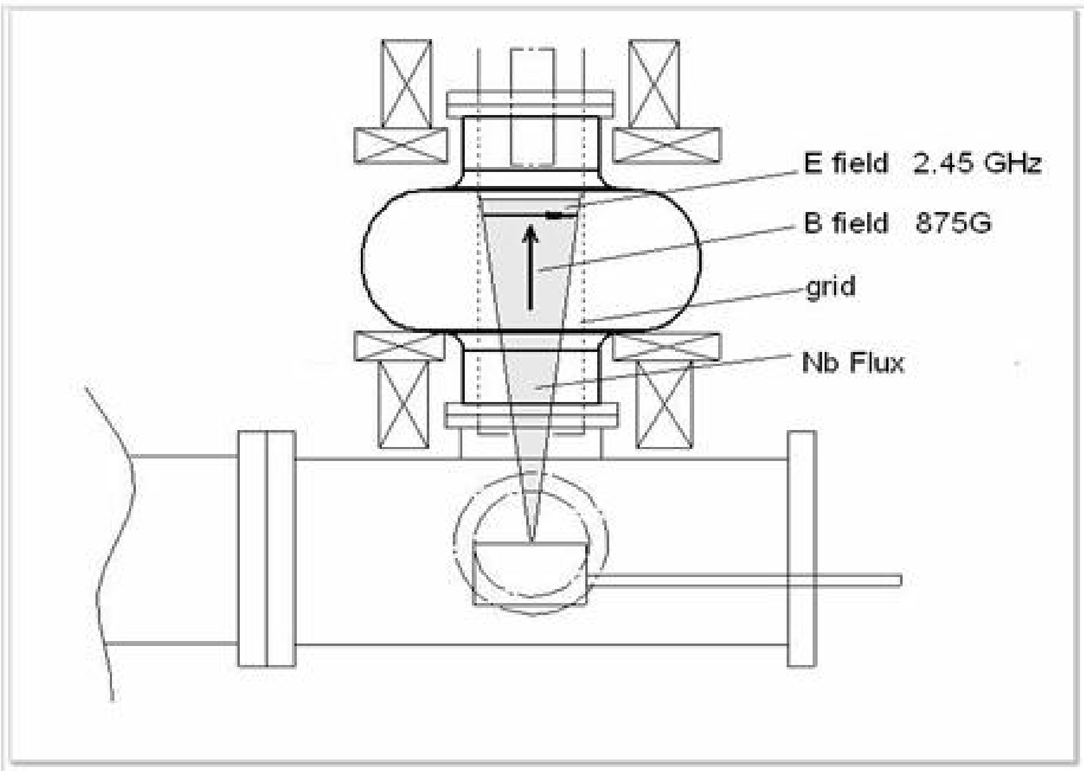


Biased sputtering at LNL



Evaporation + ECR (JLAB)

- Niobium is evaporated by e-beam, then the Nb vapours are ionized by an ECR process. The Nb ions can be accelerated to the substrate by an appropriate bias. Energies in excess of 100 eV can be obtained.



Generation of plasma inside the cavity
3 essential components:
Neutral Nb vapor
RF power (@ 2.45GHz)
Static B ⊥ ERF with ECR condition

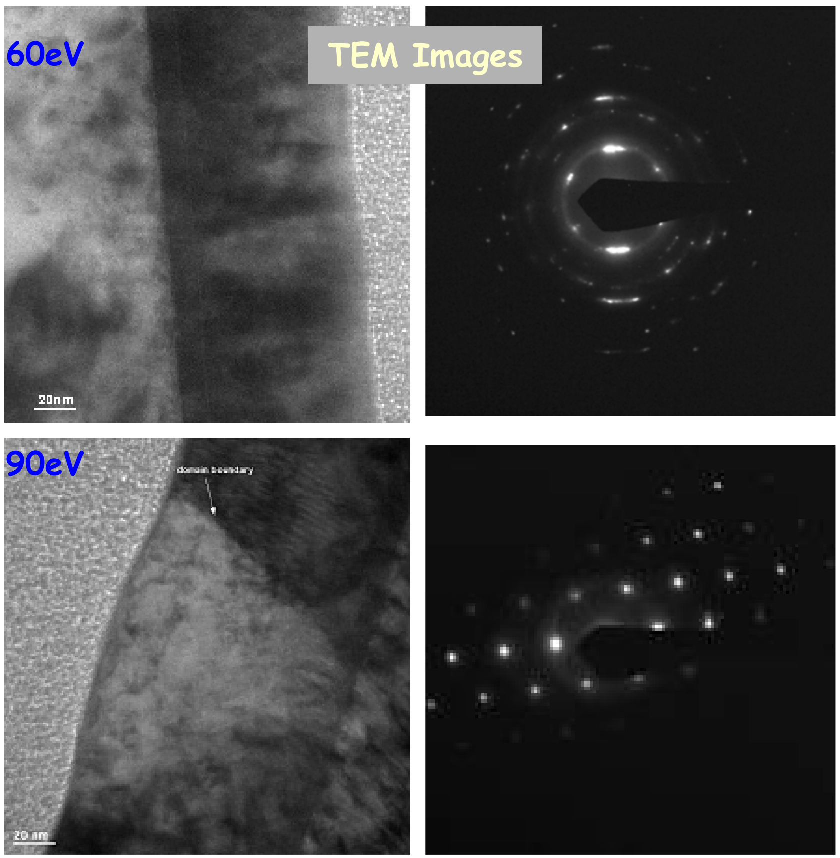
Why ECR?
No working gas
High vacuum means reduced impurities
Controllable deposition energy,
90-degree deposition flux
(Possible to help control the crystal structure)
Excellent bonding
No macro particles
Faster rate (Conditional)

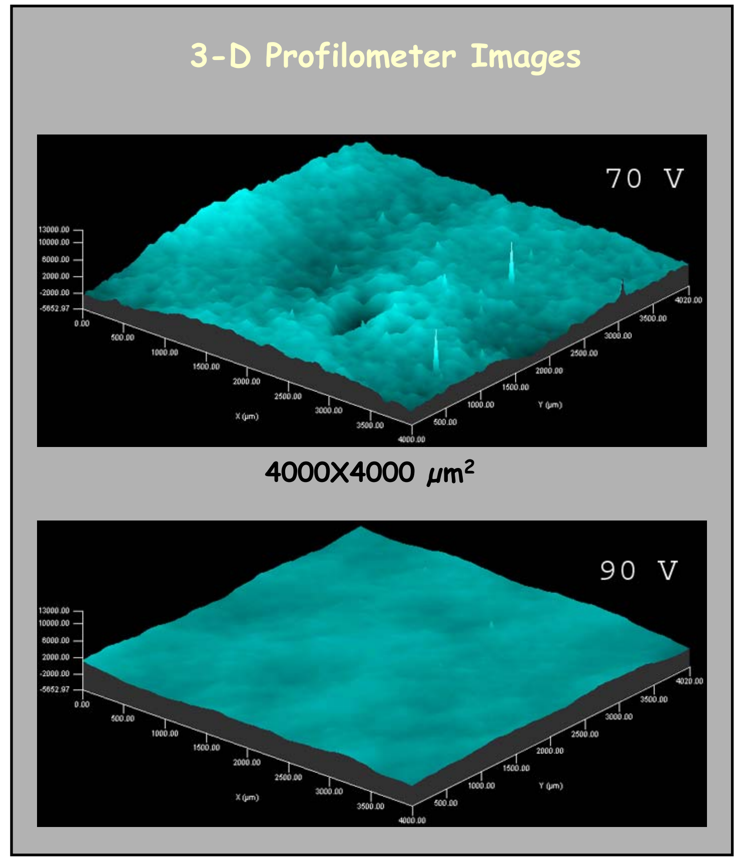
See also TuP61, TuP64

From: A.-M. Valente, G. Wu

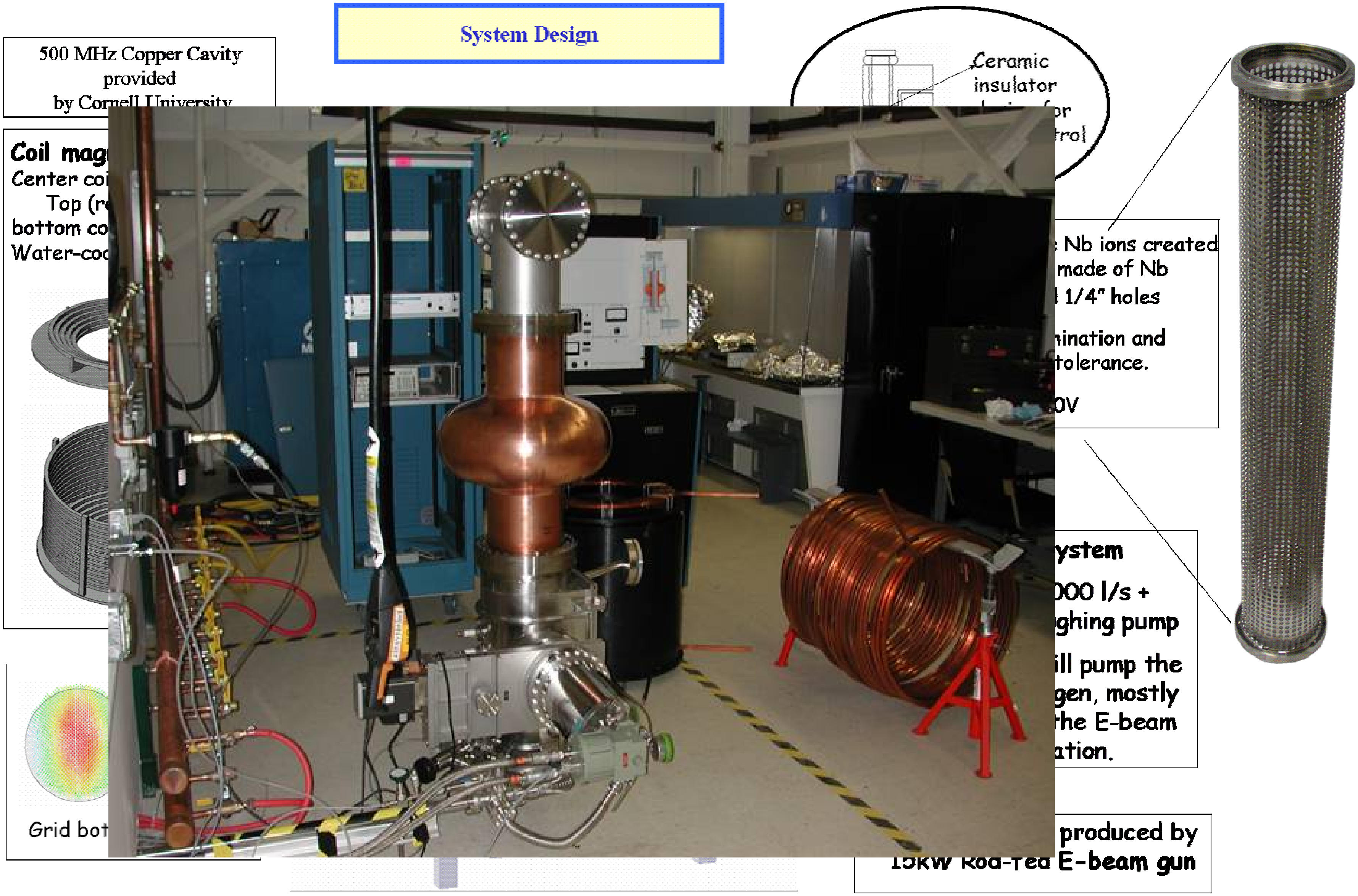
Evaporation + ECR: results on samples

- Obvious advantage: no noble gas for plasma creation
- Sample tests: good RRR and Tc, 100-nm grain size, lower defect density and smooth surfaces



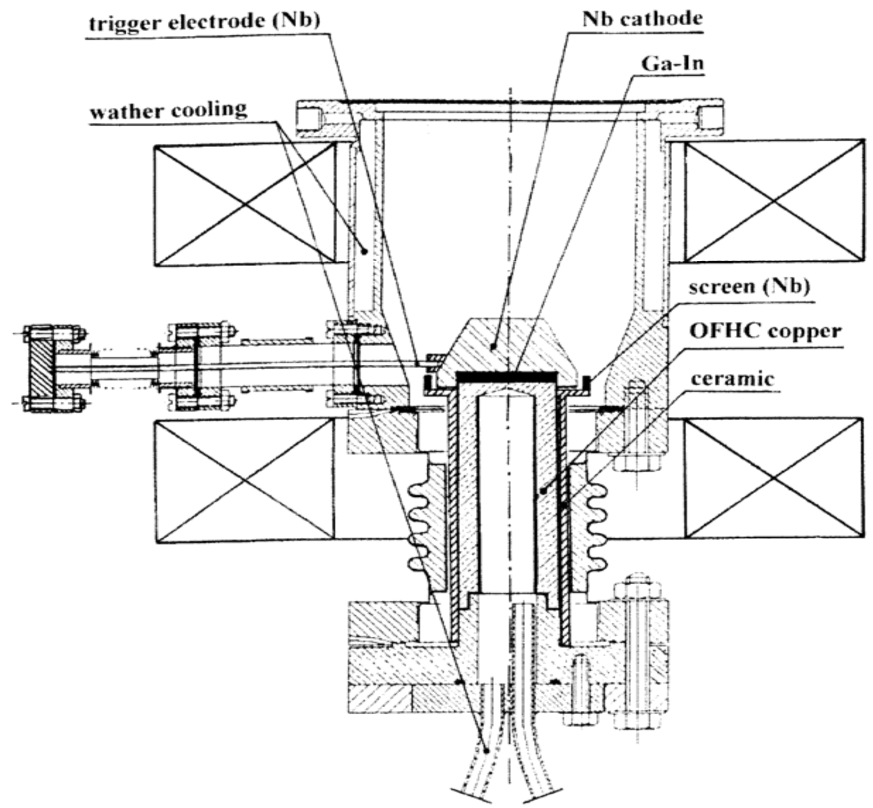


Application to cavities (JLAB)



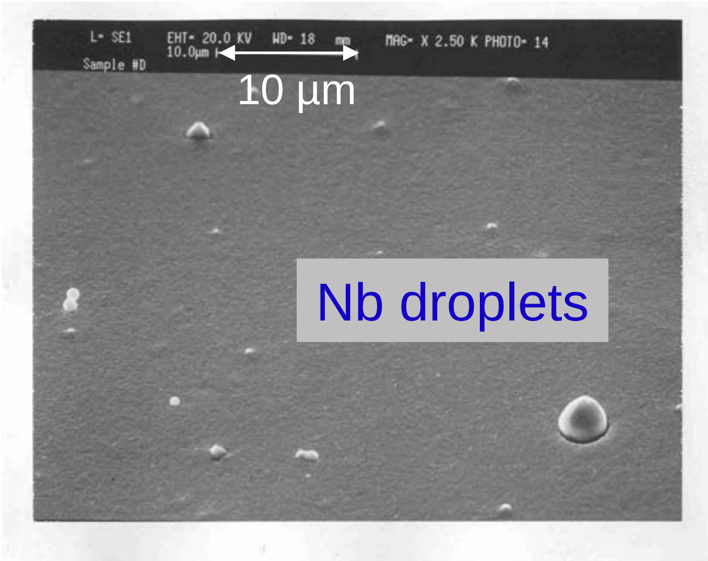
Plasma Arc (INFN)

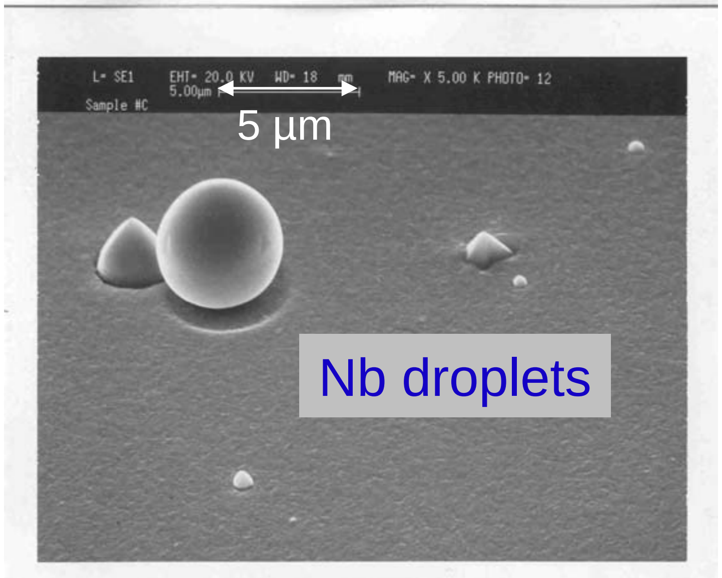
- In the plasma arc an electric discharge is established directly onto the Nb target, producing a plasma plume from which ions are extracted and guided onto the substrate by a bias and/or magnetic guidance
- Magnetic filtering (and/or arc pulsing) is also necessary to remove droplets
- A trigger for the arc is necessary: either a third electrode, or a laser
- Arc spot moves on the Nb cathode at about 10 m/s
- Arc current is 100-200 A
- Cathode voltage is ~ 35 V
- Ion current is 100-500 mA on the sample-holder (2-10 mA/cm^2)
- Base vacuum ~ 10^{-10} mbar
- Main gas during discharge is Hydrogen (~ 10^{-7} mbar)
- Voltage bias on samples 20-100 V

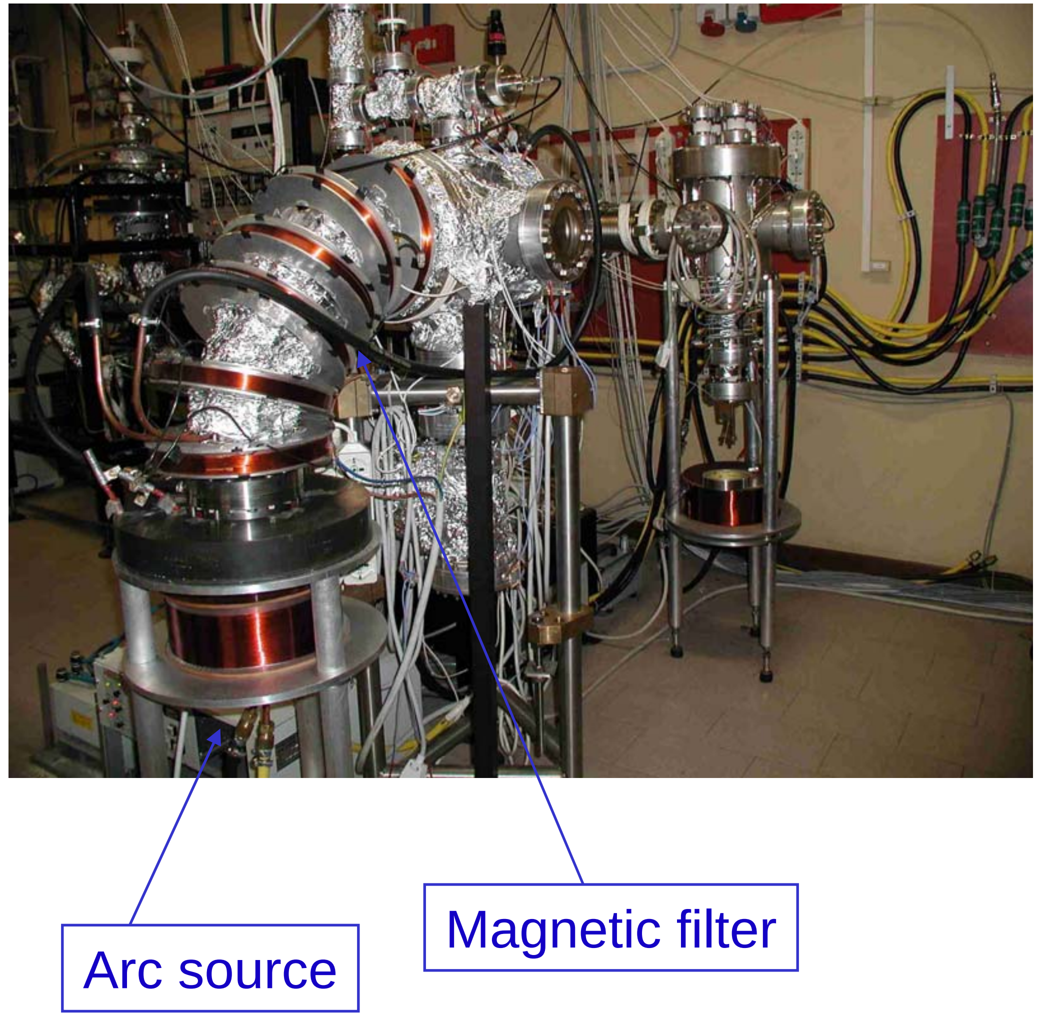


From: R. Russo, A. Cianchi, S. Tazzari

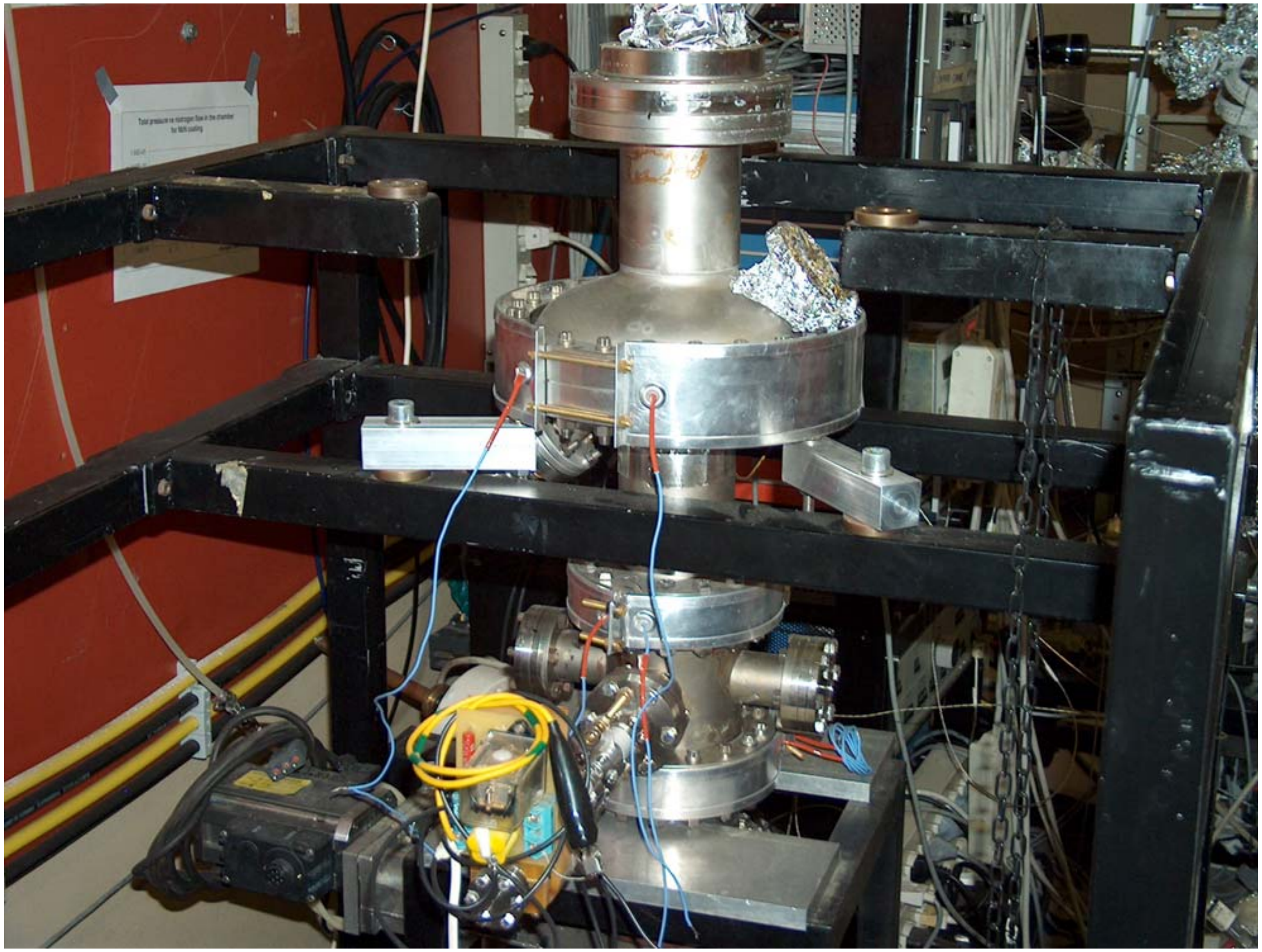
Plasma Arc – Need for filtering







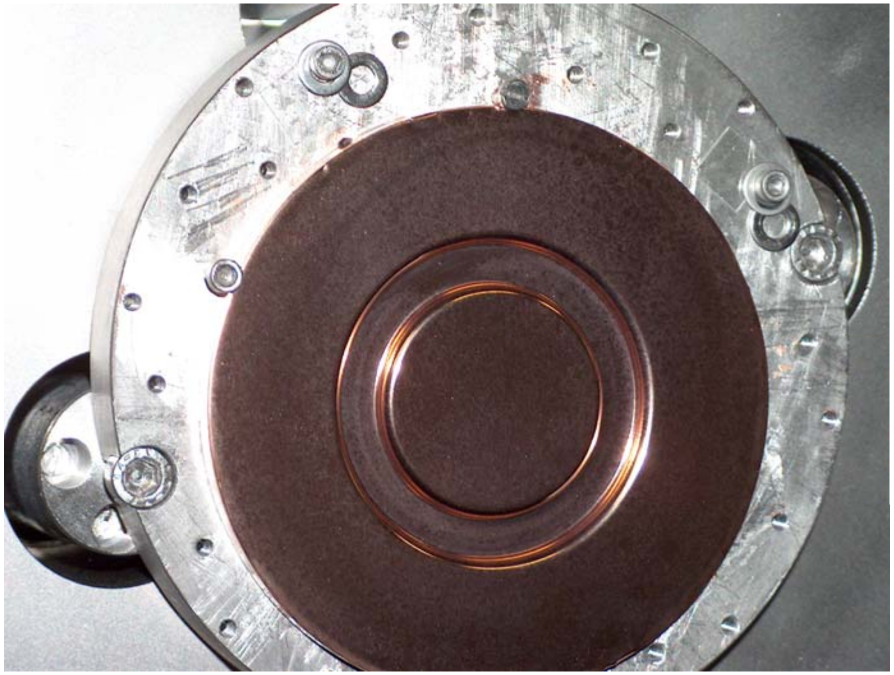
Planar arc – cavity deposition set-up



New ideas are put forward for using a planar arc for cavity coating

See also TuP62

Planar arc – RF measurements on samples!



Cu samples with Nb ARC-coating. Used as a baseplate of 6 GHz cavity operating in the TE011 mode. At low field, the surface resistance is in the range 3÷6 µOhm, as compared to the BCS Rs of 0.22 µOhm at 2.2 K and small mean free path. The Q remained constant up to a field of 300 Oe.

A baseline of 2.2 µOhm is measured with this cavity with a solid Nb plate

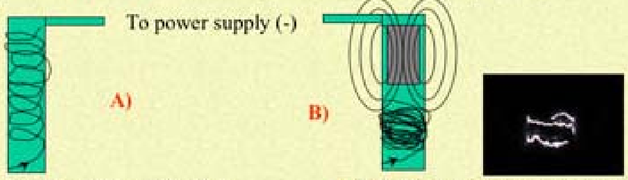
Liner arc for cavity deposition (Soltan Institute)

Cylindrical Arc Systems

Working principle

In a "cylindrical" arc the arc current flowing along the cathode generates a magnetic field that interacts with the arc plasma. This interaction constrains the arc spot to spiral around the cylindrical cathode in the direction of the current flow.

Modes of operation **A** and **B** schematically shown below are both possible:



A) : The arc is started at the positive side of the cathode and stopped on a floating potential electrode mounted at the opposite side of the cathode itself. Alternatively the current flow can be inverted as the spot reaches the opposite cathode end.

B) : A strong permanent magnet "reflects" the arc spot, confining its movement to the region below the magnet. Progress along the cathode is obtained by moving the magnet.

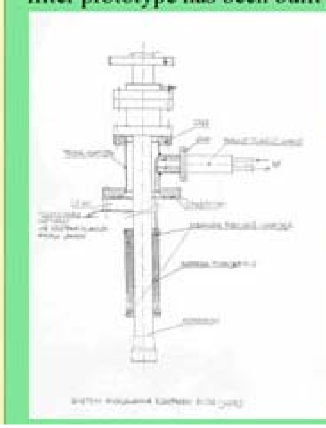
Laboratory in Rome: Cylindrical arc system and planar filtered arc

We have chosen solution B that allows controlling the arc movement like in the magnetron sputtering case because it makes coating of multicell cavities and control of the film thickness along the structure easier

The laboratory setup in Swierk in which the UHV system equipped with a cylindrical arc source shown in the photograph has been recently put into operation.

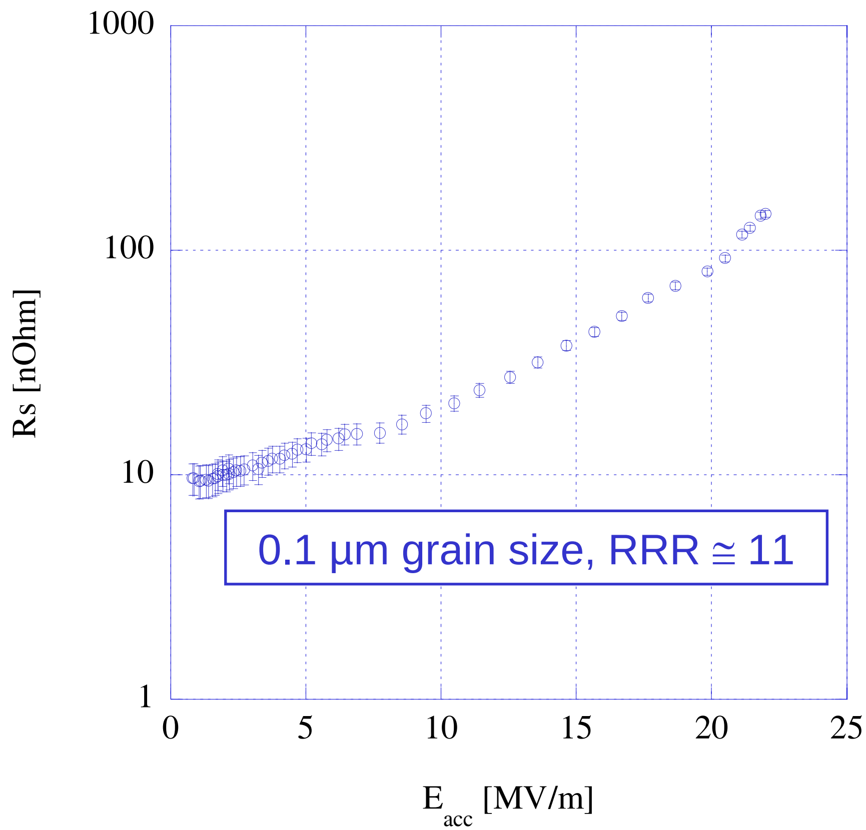
Filtering a cylindrical arc

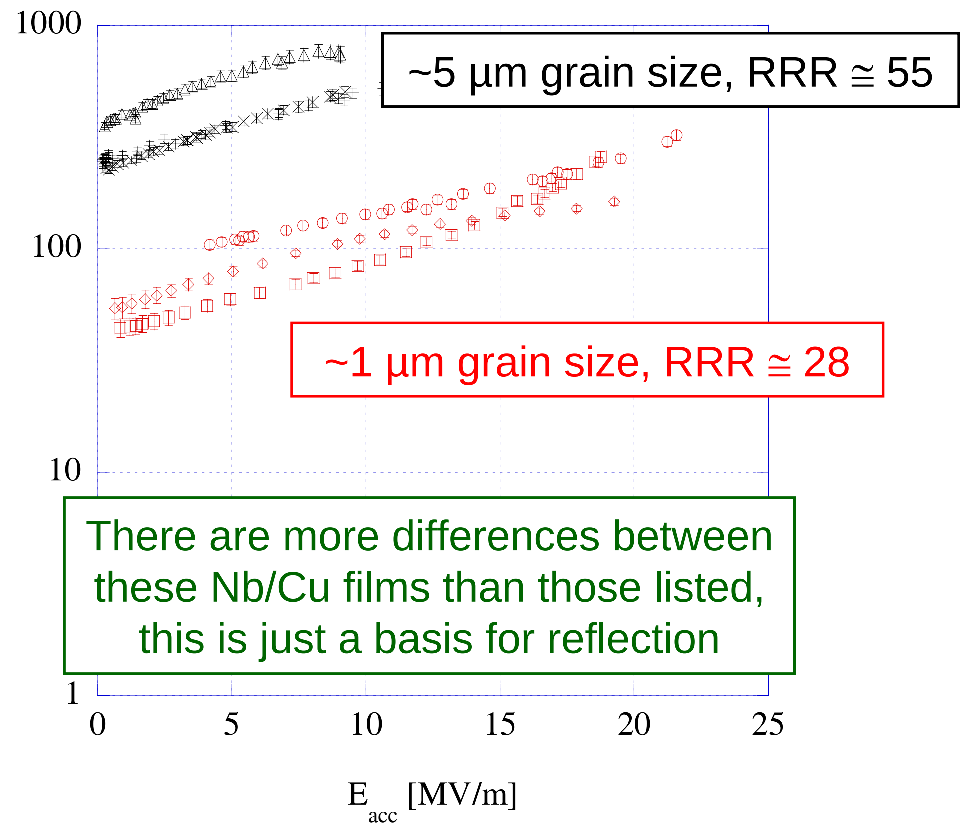
Macrodroplets, potential sources of field emission may represent a problem for the RF cavity performance. Experiments to verify this and to try and remove the droplets by HPWR or other methods are in progress. In addition, we have studied the possibility to filter such microdroplets in a cylindrical arc geometry. A first filter prototype has been built but not yet tested.



The filter, shown below, works as follows: a current driven through the copper tubing water cooled structure generating a magnetic field that guides plasma electrons (and ions) through the small gaps between pipes. There being no direct line of sight from cathode to anode, heavy particles can instead not get through.

Films as a bulk – Hydrogen becomes a problem!

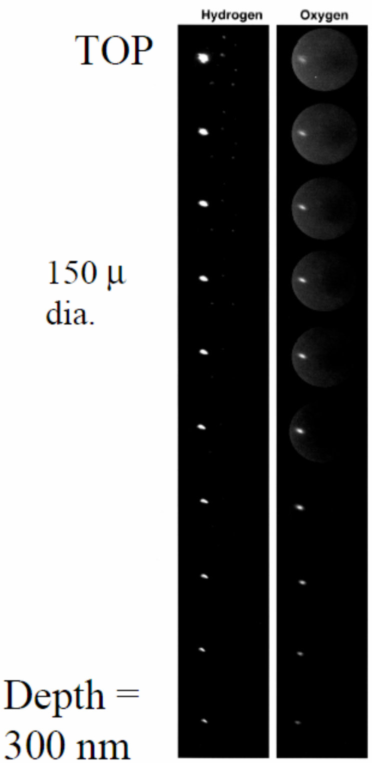




H_2 content is ~ 0.1 at. % for sputtered Nb/Cu films (in niobium bulk it is 0.02 at.%) and it is picked up from vacuum system during deposition.

A possible solution: high-temperature annealing, but it does not work with copper cavities. Proposal (L. Hand, W. Frisken): molybdenum cavities.

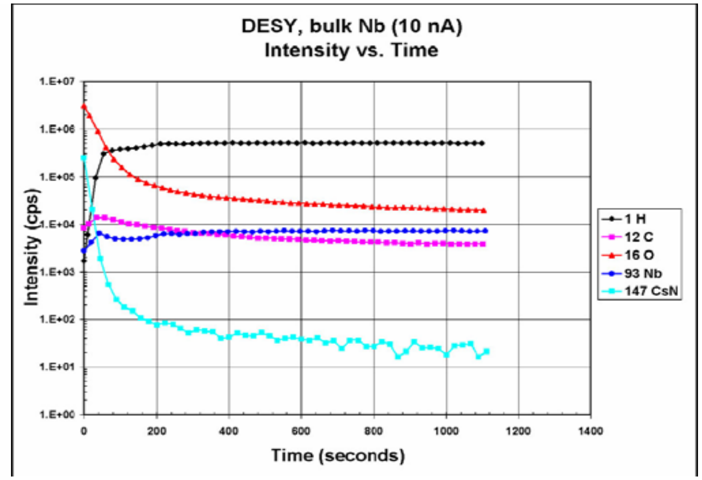
Film as a bulk – H_2 measurements

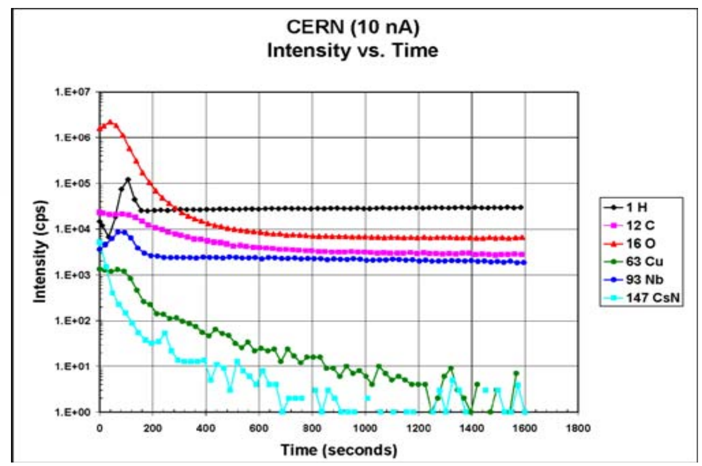


Depth profile in 30 nm steps, proceeding from top of film towards bottom.

Notice the conjunction of H+O in the brightest "river". <u>H visible here cannot be α phase.</u>

Oxygen is a known hydrogen "trap" in Nb.
The sensitivity for looking in grain boundaries is much less here than for EELS, but an image is produced by SIMS.



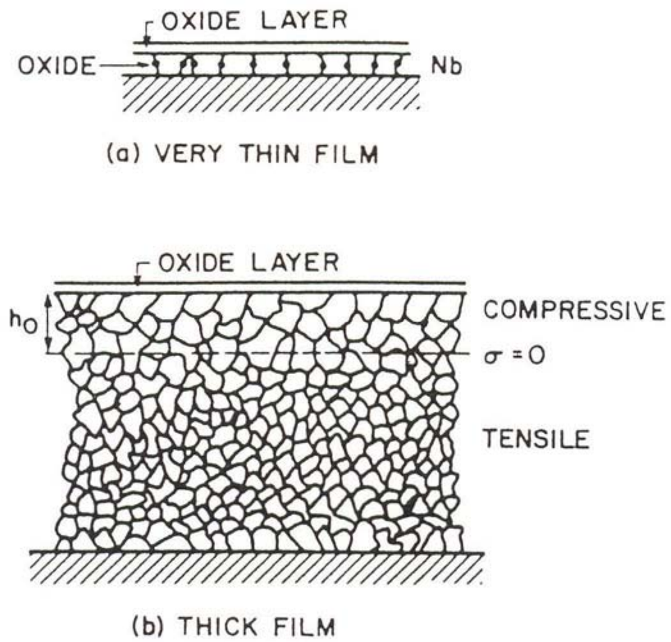


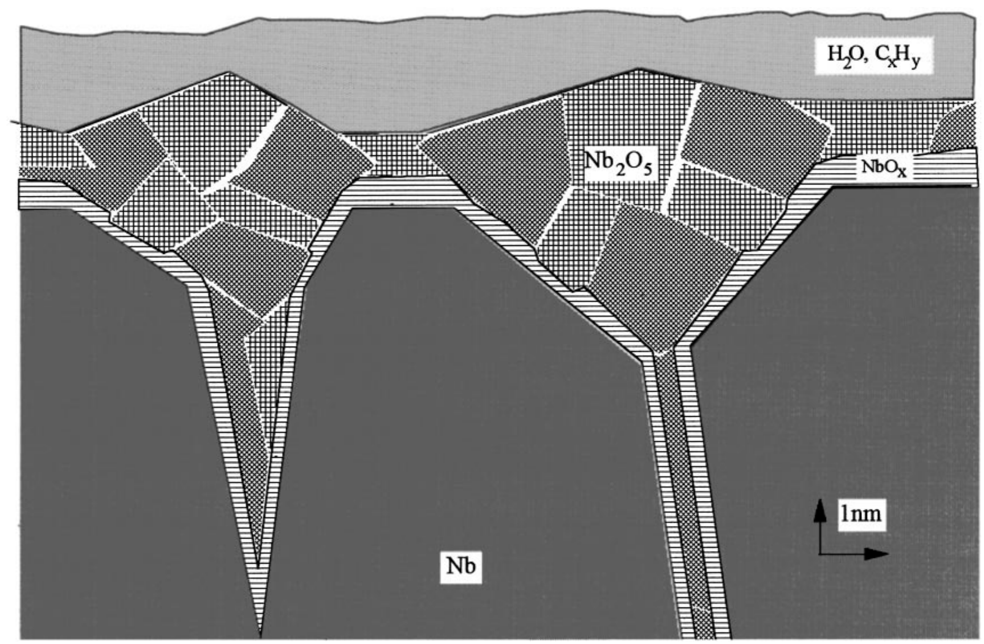
There are new results on measurements of H_2 content by measuring the lattice parameter and the total impurity content

See TuP16

From: L. Hand, Cornell U. – W. Frisken, York U.

Grain boundaries and surface oxidation



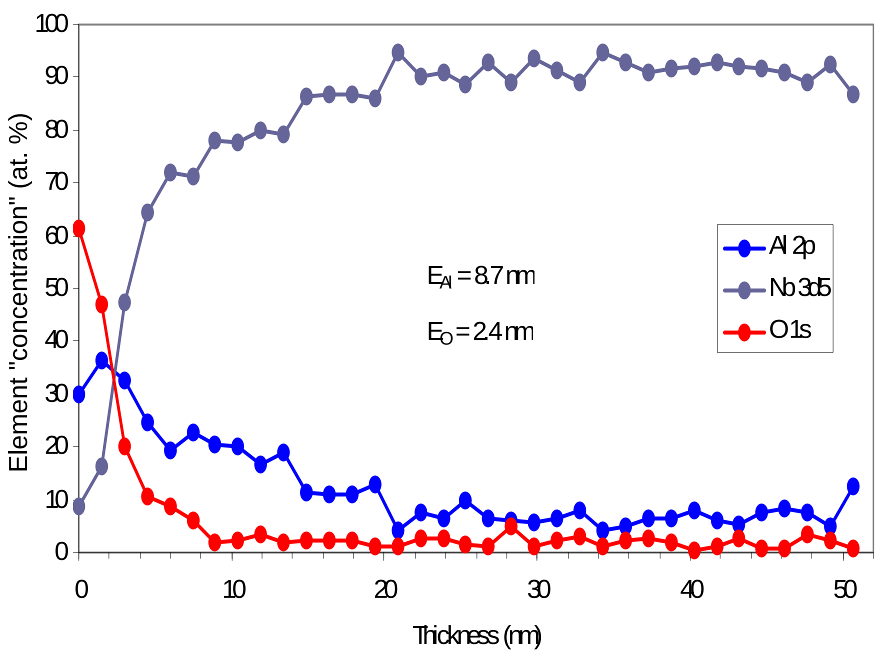


Famous drawings by Halbritter. Several effects might take place: ITE, flux penetration, H_{c1} depression, lower Tc, etc.

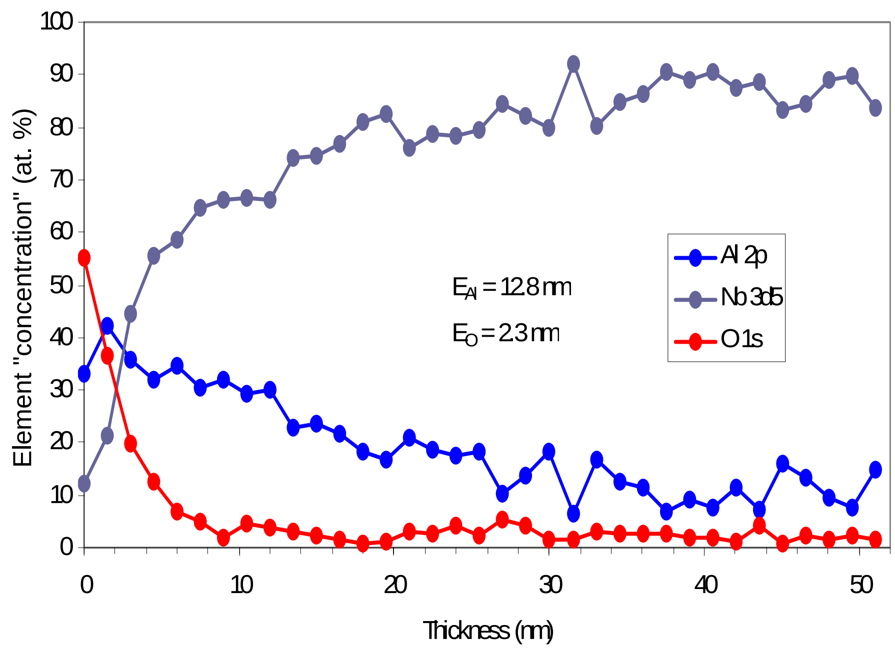
Possible solution – Al_2O_3 cap layers

- Technique routinely used for S-N-I-S Josephson junctions: a 5-nm thick Al layer is deposited onto the Nb base electrode, and let oxidize in air. Most of it is transformed to Al_2O_3 but some remains metallic.
- It is important to prevent any surface contamination of Nb prior to Al coating, to reduce the coalescence of the Al atoms.
- Other possible solution: NbN overlayer (J. Halbritter)

XPS depth profile



5 nm Al (nominal)



10 nm Al (nominal)

Conclusion

- Niobium films are still an option for particle accelerators at any beta, except for reaching the highest fields because of the increase of R_{res}
- The technique of choice is at present sputter deposition: a prerequisite for it is substrate design and its preparation

- R&D on ion deposition techniques to obtain bulk-like films is the new trend. An old enemy will then come into play: hydrogen
- Mastering of the oxide layer (through an Al_2O_3 cap?) should also become a central point of studies