Nb/Cu Technology – An Overview Of The Present Status

S. Calatroni

Introduction

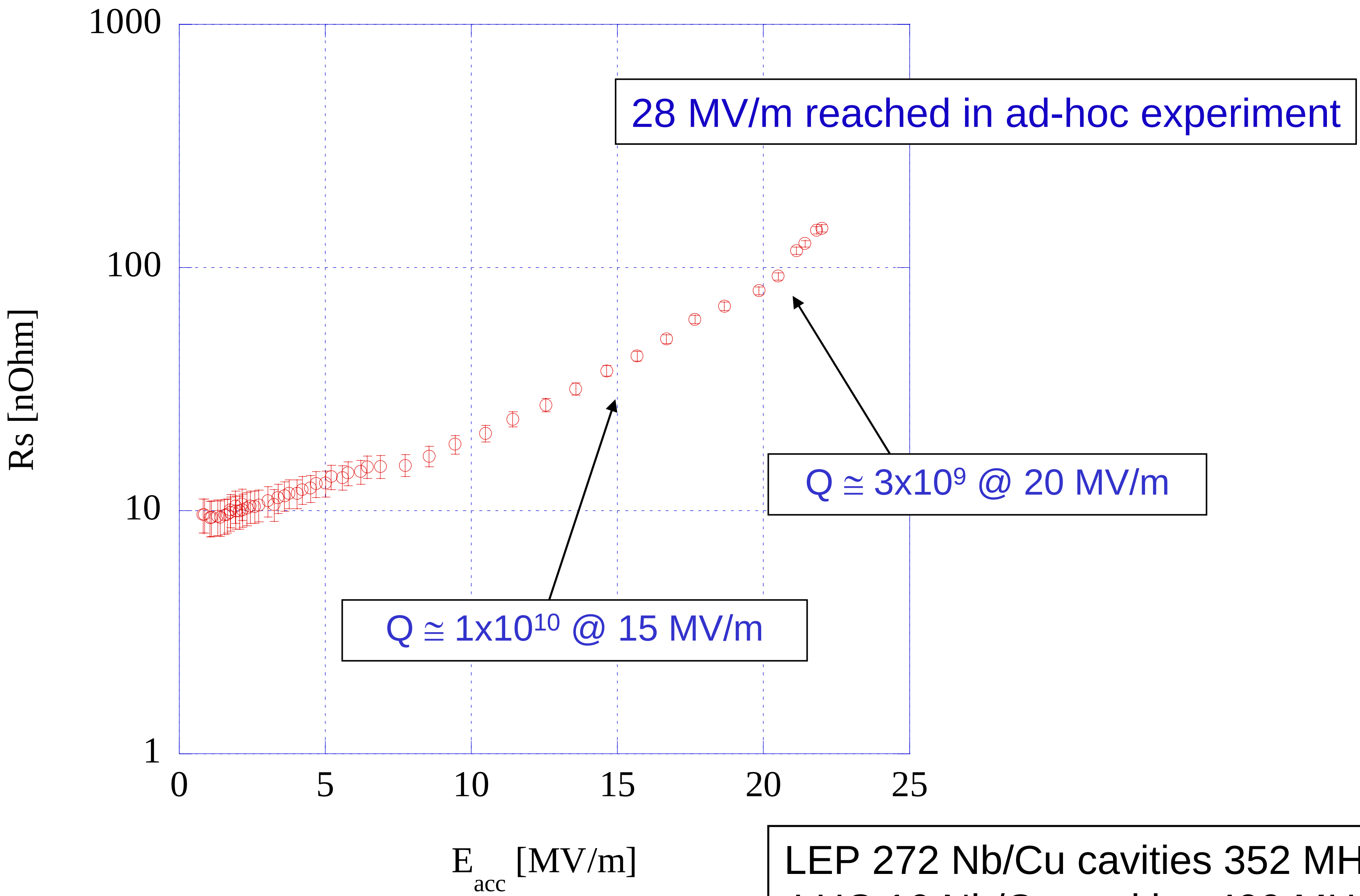
- Why films
- State of the art: high-field and low-beta applications
- Nb film R&D activities:
  - Roughness
  - Structure
  - Oxidation
- Conclusions

Why films

- Advantage (primary objectives)
  - Thermal stability
  - Innovative materials
  - Cost
- Advantage (learned from experience)
  - Optimisation of R_{BCS} at 4.2 K (sputtered niobium films)
  - Insensitive to earth magnetic field

- Against (understood from the beginning)
  - Fabrication and surface preparation (at least) as difficult as for bulk
- Against (learned from experience)
  - Steep R_{res} increase with RF field for Nb films
  - Deposition of innovative materials is very difficult

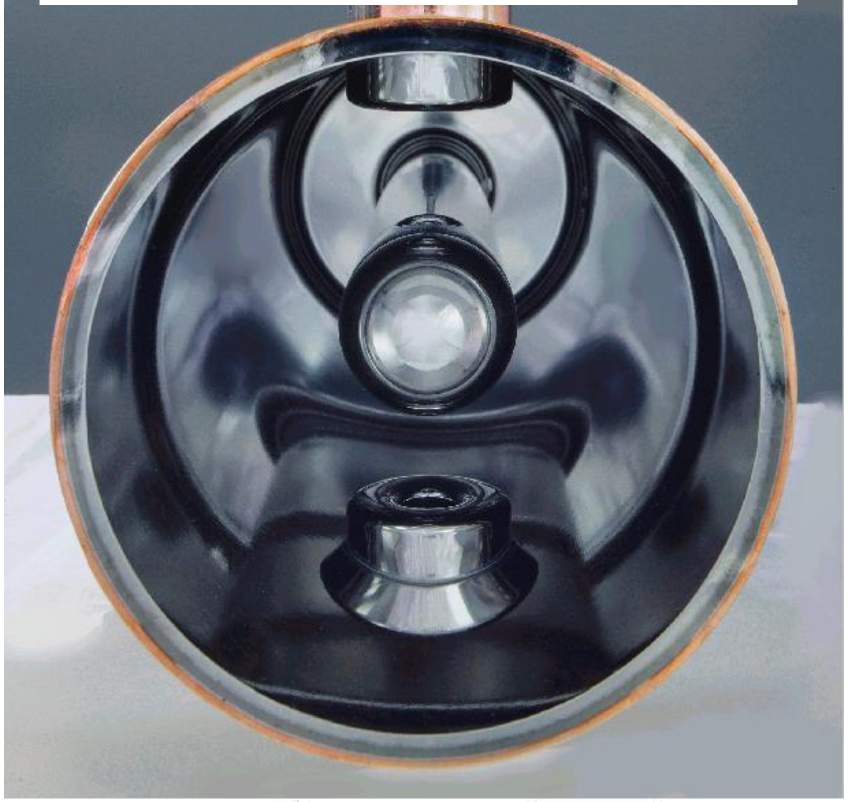
State-of-the-art at 1500 MHz – 1.7 K – single cell

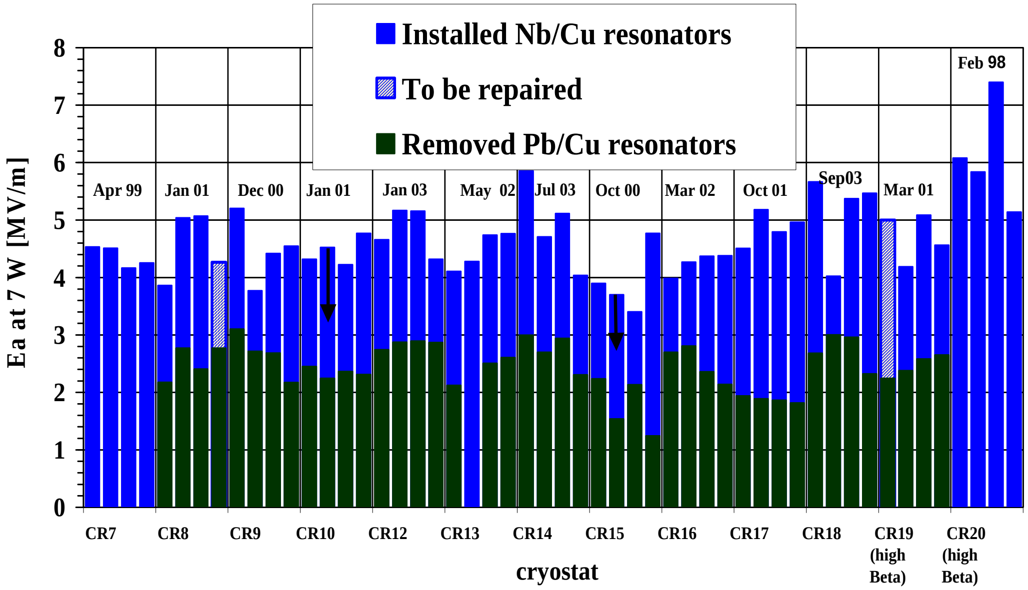


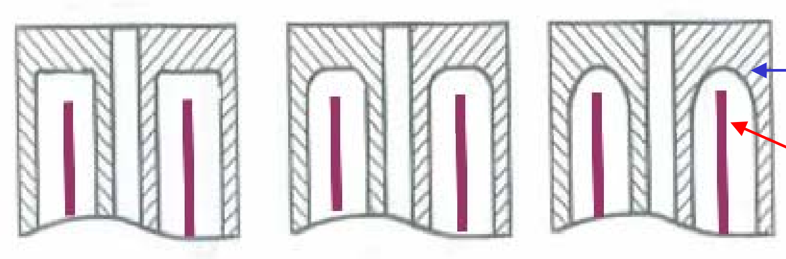
LEP 272 Nb/Cu cavities 352 MHz
LHC 16 Nb/Cu cavities 400 MHz

State of the art – QWR Nb-sputtered vs. Pb-plated

Inside view of ALPI QWR







The different angle shaping results in different deposition angles

Sputtering cathode

From: V. Palmieri, AM. Porcellato, S. Stark

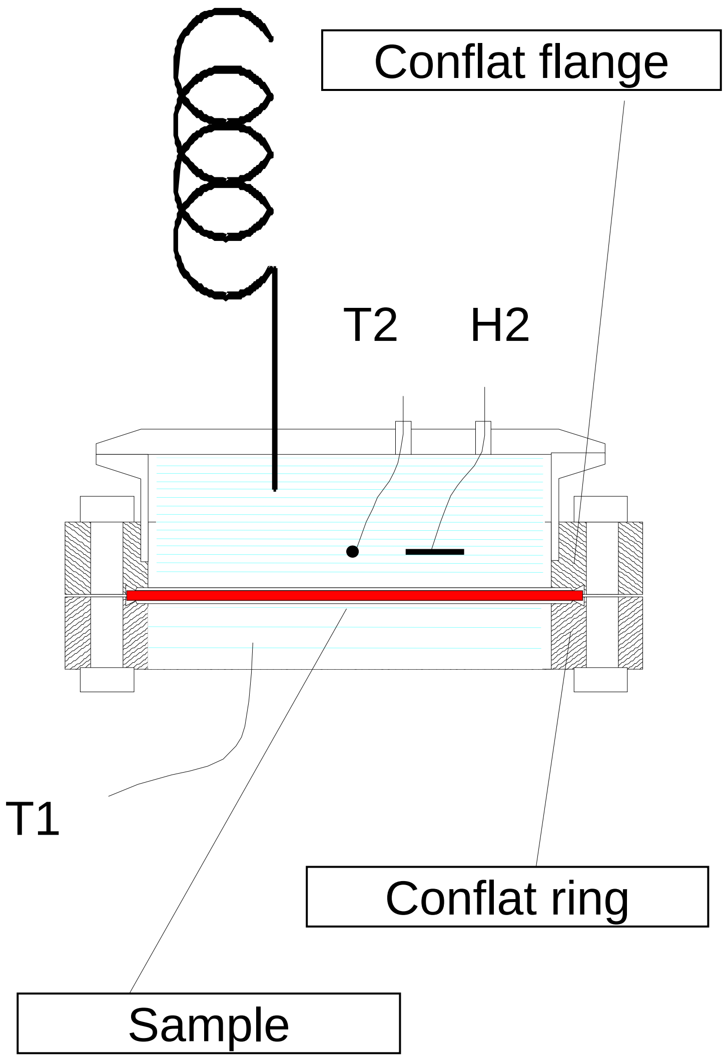
Nb/Cu films – Philosophy

- There are two categories of films

  - Films which are intrinsically films
    - Thin, microcrystalline, microstrained, under stress
    - Examples: CERN sputtered films on oxidised copper
    - Problems: defects, impurities, surface state

  - Films which try to mimic bulk
    - Thick, macrocrystalline
    - Examples: CERN sputtered films on oxide-free copper, high-energy deposition techniques, annealed films, (Nb Cu-clad)
    - Problems: hydrogen, surface quality

Thermal impedance film-substrate



The overall thermal impedance has been measured for pure Nb and Cu, and for Nb/Cu and Nb/Nb films, on 2-mm thick disks.

Cu (RRR=100) EP	4300 ± 200 $Wm^{-2}K^{-1}$
Cu EP + 1.5 µm Nb	4100 ± 200 $Wm^{-2}K^{-1}$
Nb (RRR=180) EP	1200 ± 200 $Wm^{-2}K^{-1}$
Nb EP + 1.5 µm Nb	1000 ± 200 $Wm^{-2}K^{-1}$

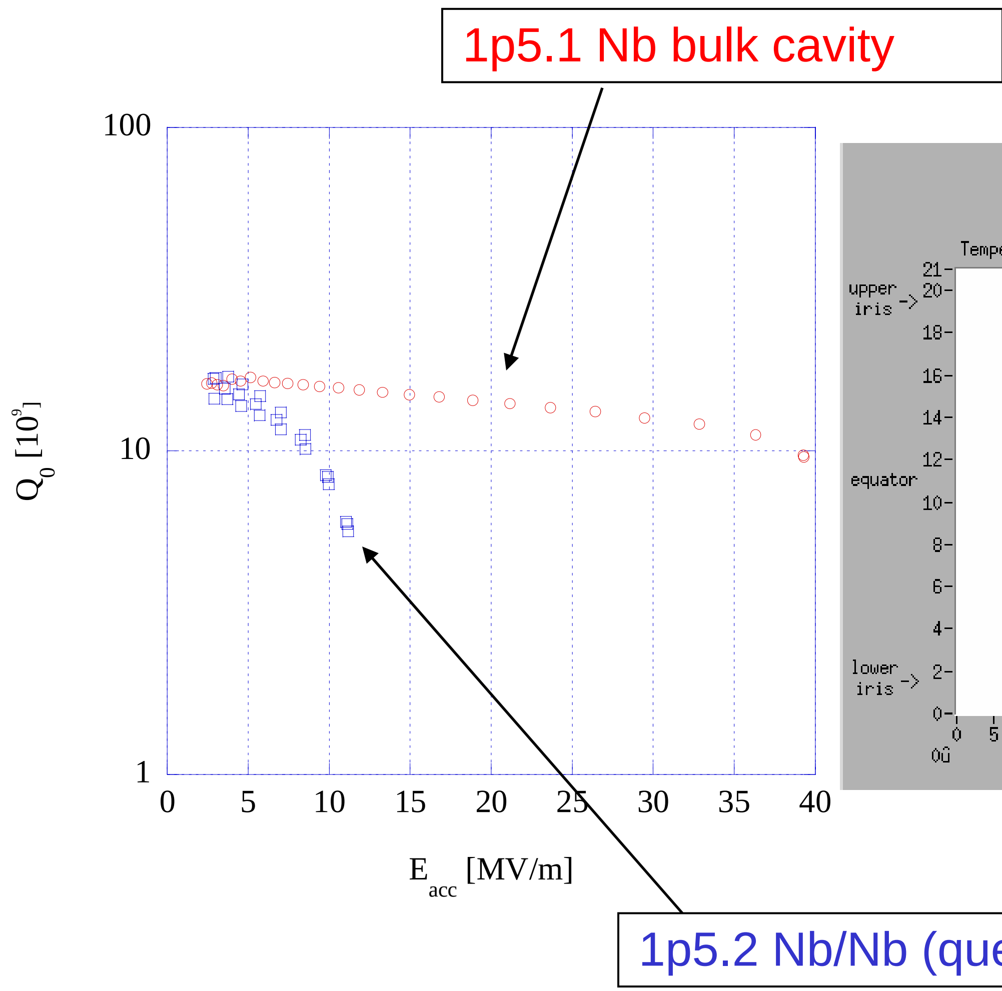
Nb (RRR=670)(extrap.) 2500 ± 200 $Wm^{-2}K^{-1}$

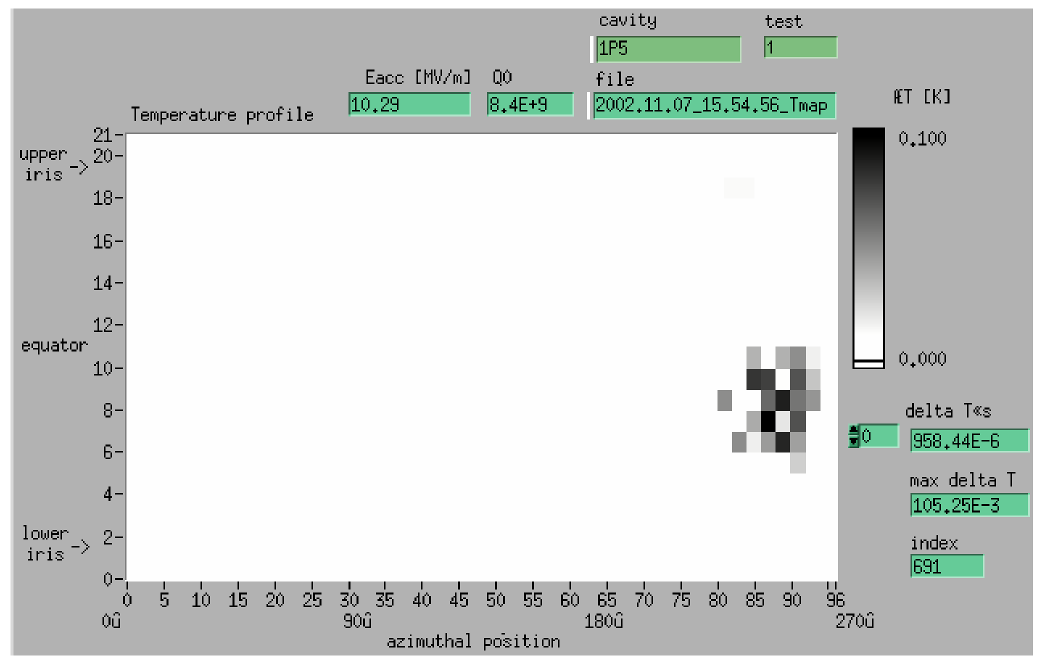
(Still lower than Nb/Cu, but Nb cavities performs better at high field !!)

The thermal impedance of the film (if existing) has no effect on R_{res} at high RF field

Thanks to: G. Vandoni, J-M Rieubland, L. Dufay

Intrinsic higher dissipation in films ?



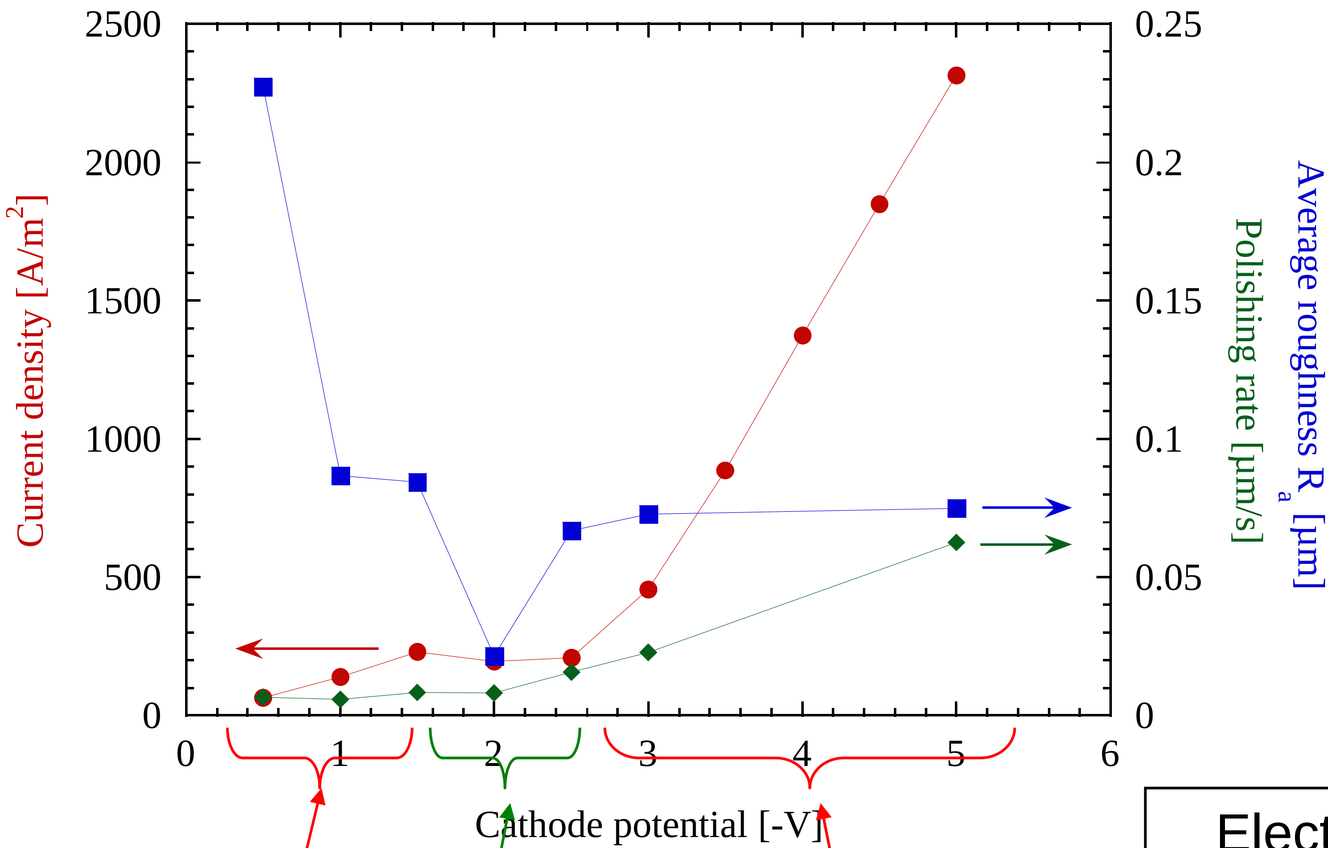


Thanks to: V. Palmieri, D. Reschke, R. Losito

Research lines around the world

- (Hydrogen)
- Effect of roughness
  - Optimisation of substrate preparation: electropolishing
  - Study of angle-of-incidence effects
- Film structure – defects
  - Bias sputter deposition (relevant also for roughness)
  - Towards a bulk-like film: high-energy deposition techniques
  - Towards a bulk-like film: high-temperature annealing of films
- Oxidation state
  - (Grain boundaries)
  - Surface effects: Al_2O_3 cap layers

Electropolishing – Polarization curve



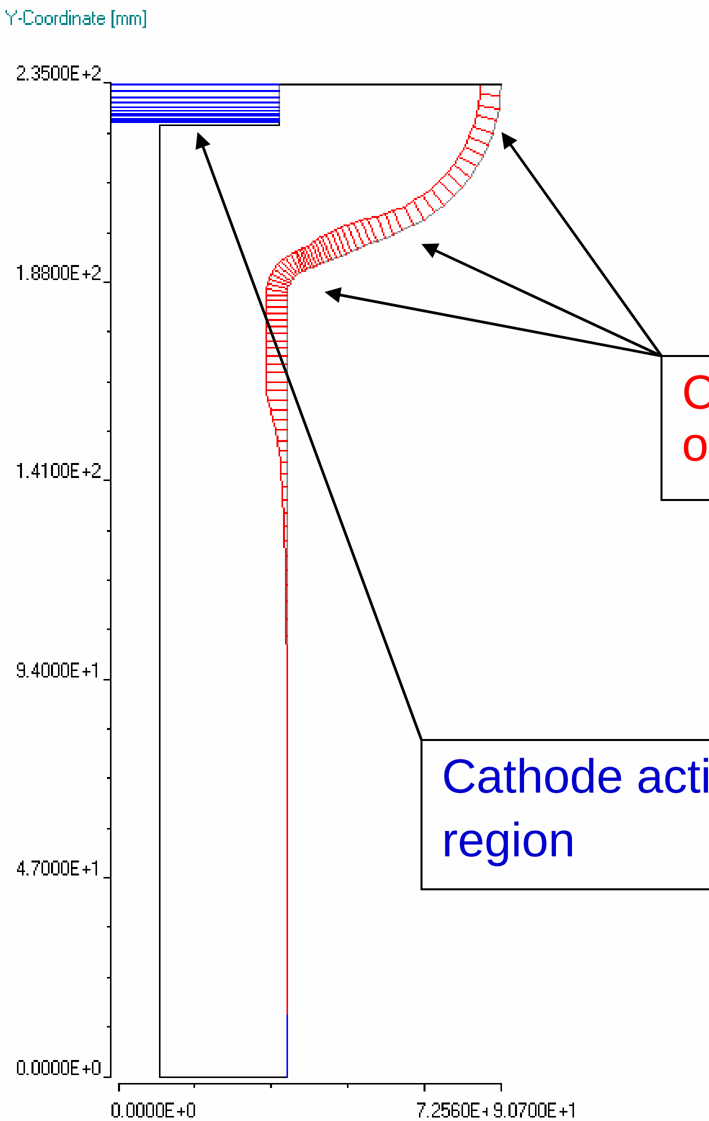
Production of $Cu(OH)_2$ on the surface!

Polishing

Production of O_2 bubbles!

Electrical resistance seen by the polishing current:
Diffusion layer ~ 0.1 Ω
Bath volume ~ 0.1 Ω
(Nb EP bath: 10 times less)

Electropolishing – Cathode design

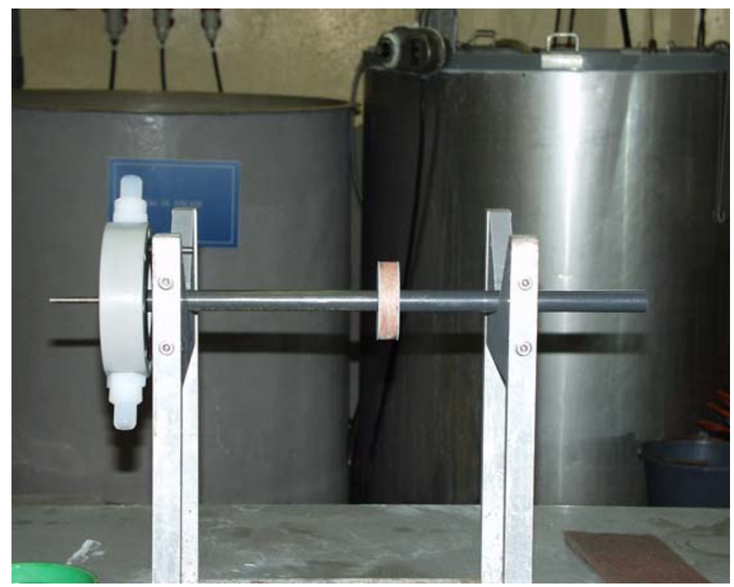


Numerical modelling of the cathode by simulation of the entire EP process with the Elsy 2D/3D computer code

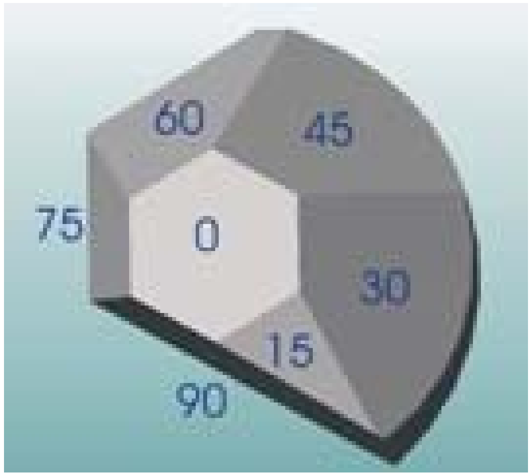
(www.elsyca.be)

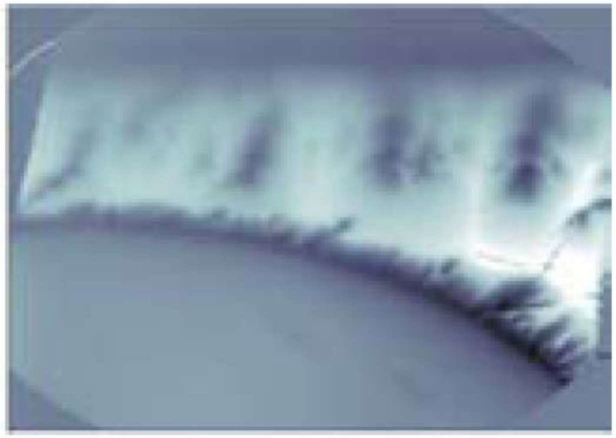
Current density is uniform over all the cell surface

Cathode active region



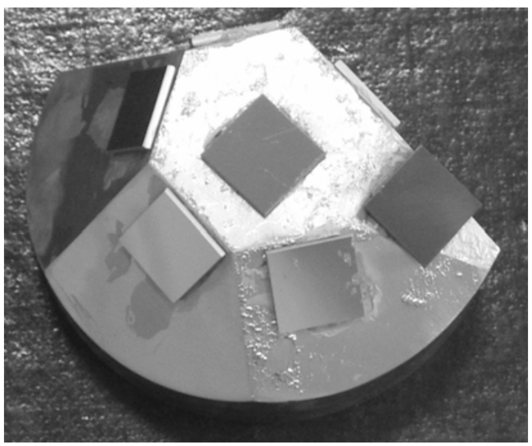
Nb coatings at various incidence angles





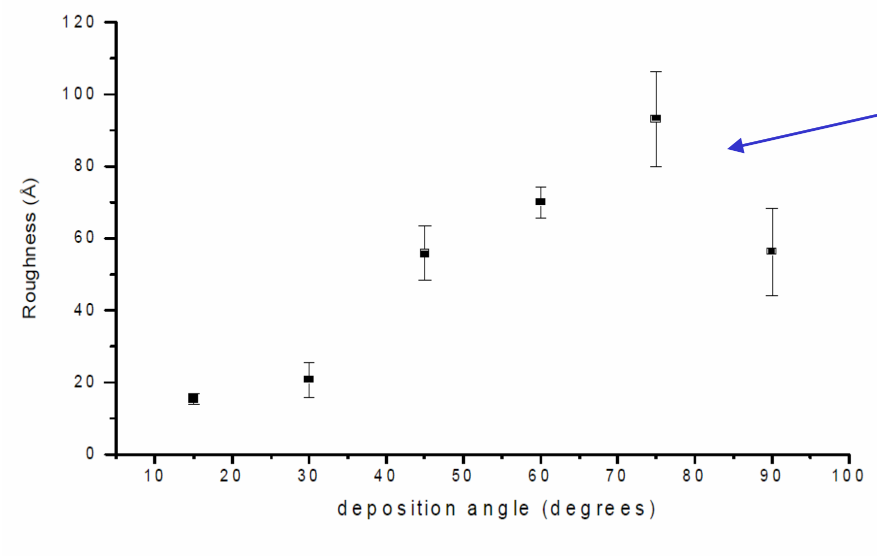
Sample at 0° angle. Bright areas represent trapped vortices in pinning sites, after an external field of 176 mT at T = 5 K. The image displays good connectivity in the central part of the film.

Sample at 45° angle, after an external field of 176 mT at T = 5 K. Dark lines are macroscopic defects and the sample stray field is closed inside them . The defects along perpendicular lines are visible, depending on the substrate morphology. Poor connectivity in the whole film.



From: V. Palmieri

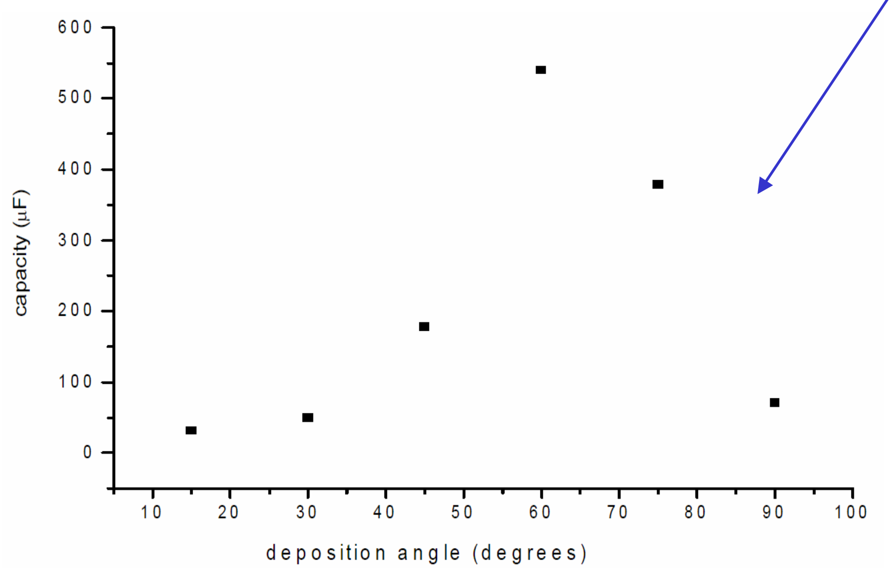
Variation of properties with incidence angle

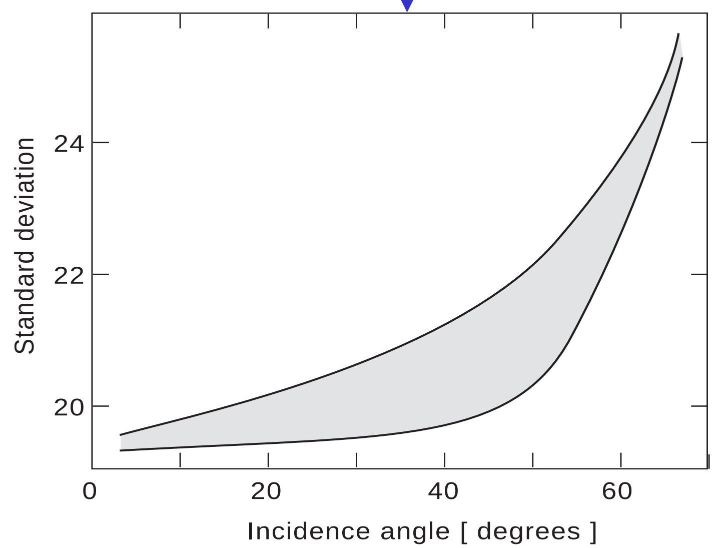


AFM roughness

Electrochemical Impedance Spectroscopy

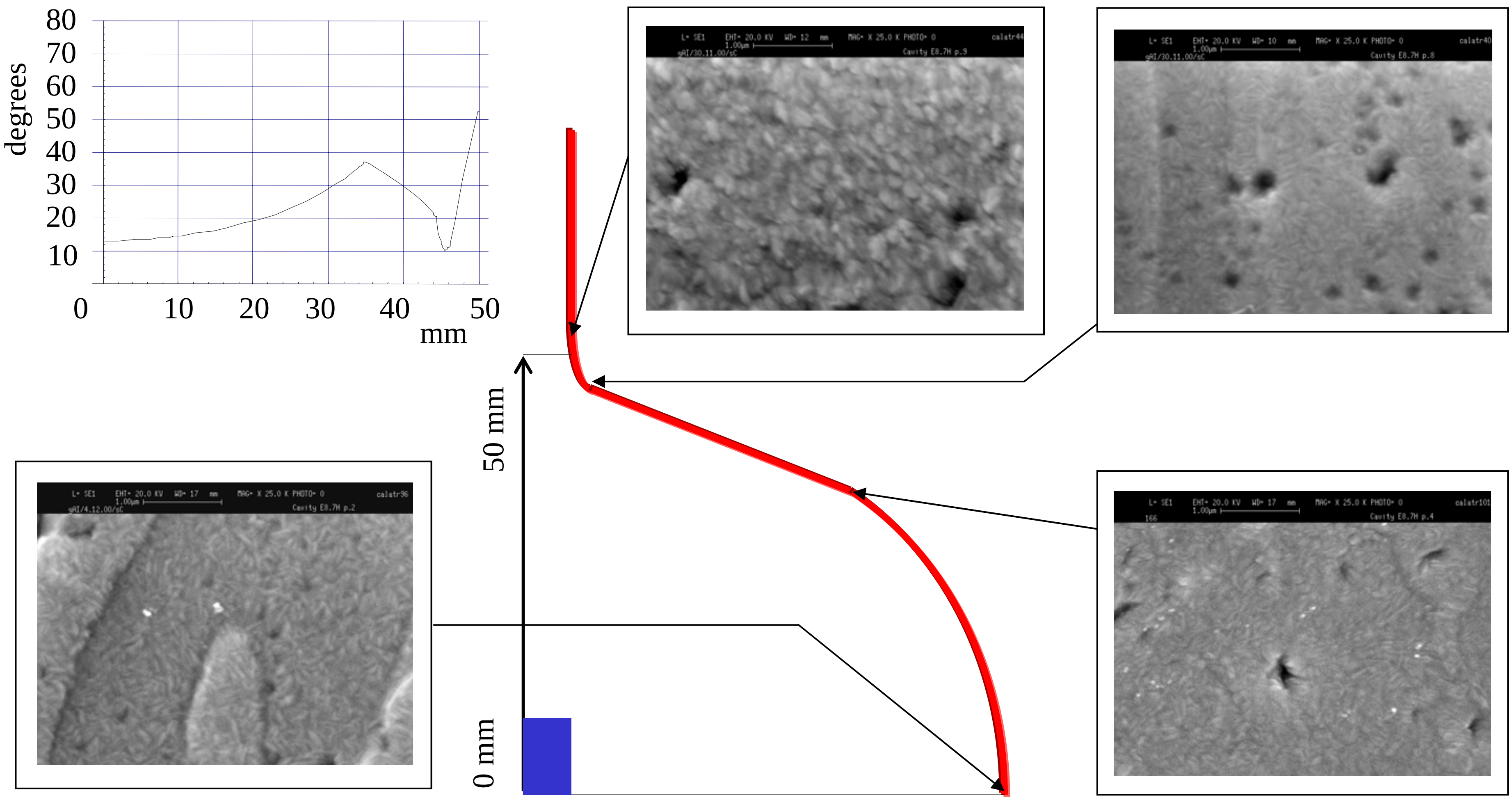
Std.Dev. of grey levels of SEM images (CERN 1999)





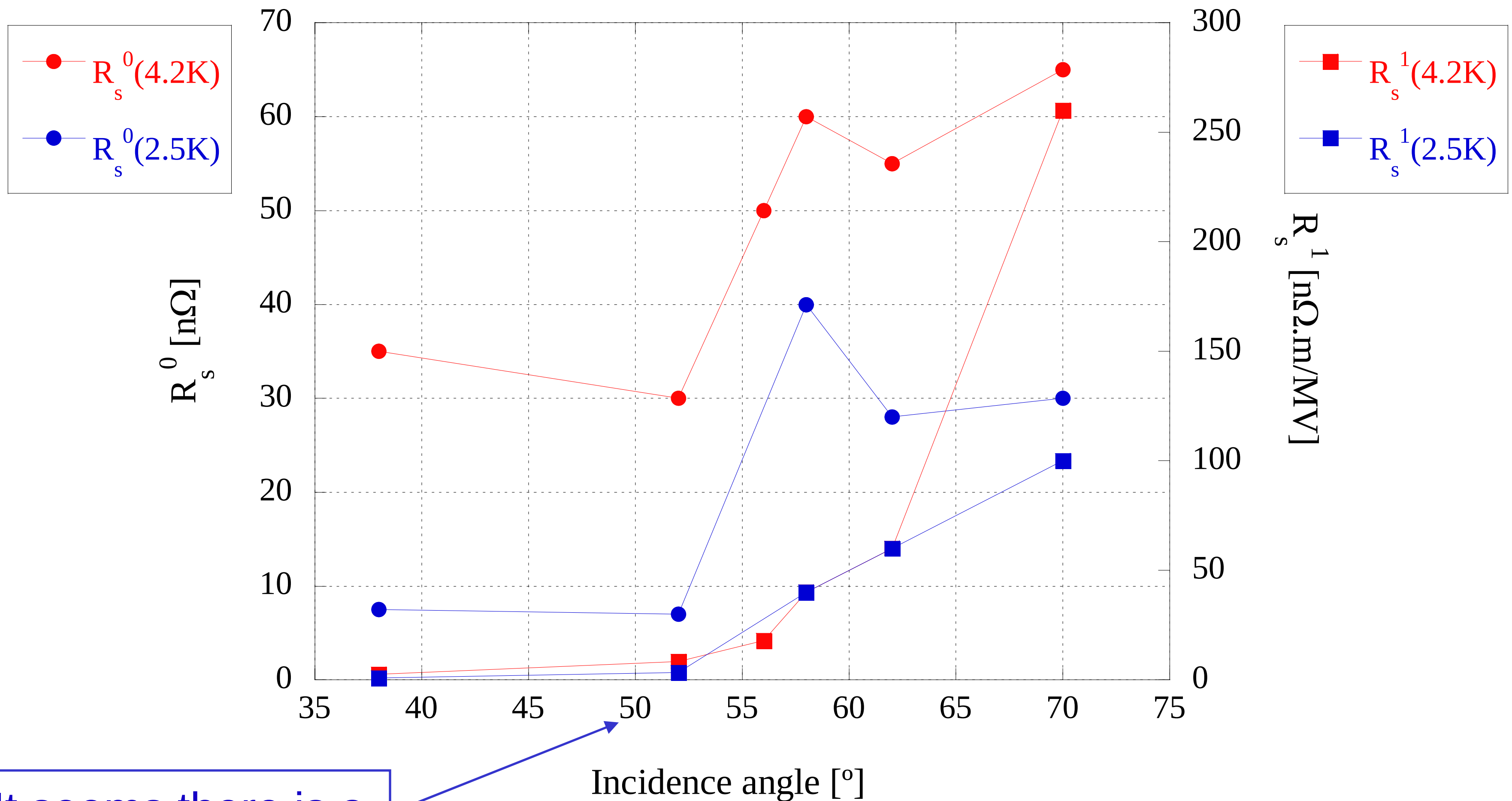
Roughness & incidence angle of Nb in 1.5 GHz cavities

SEM views of Nb film cut from 1.5 GHz cavity



Incidence angle and residual resistance in low-β cavities

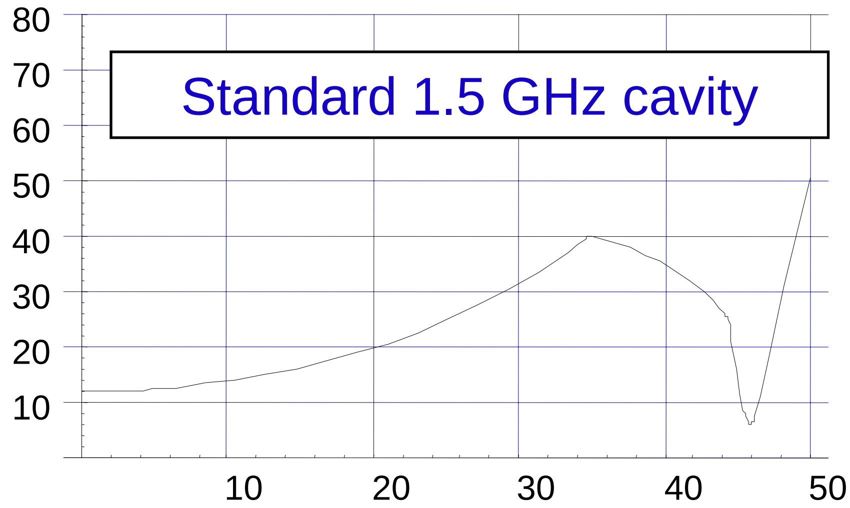
Correlation between the incidence angle of the film and the residual resistance, measured on 352 MHz Nb/Cu cavities

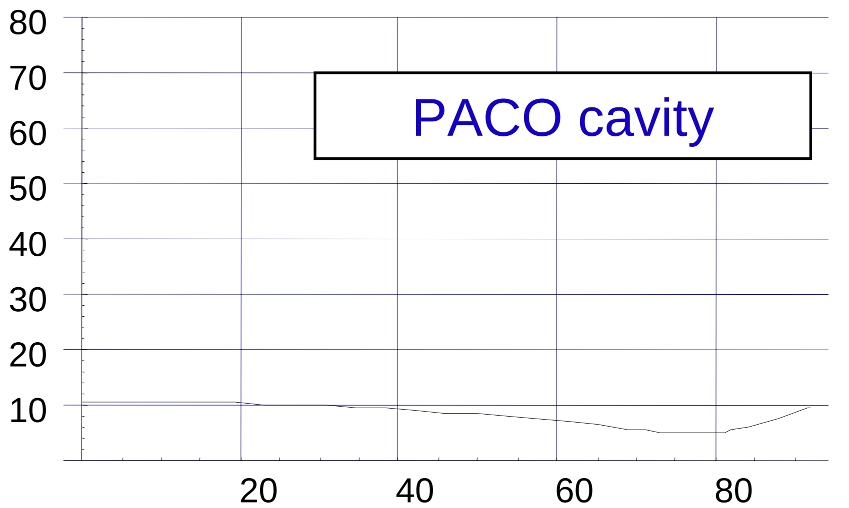


It seems there is a "threshold" effect

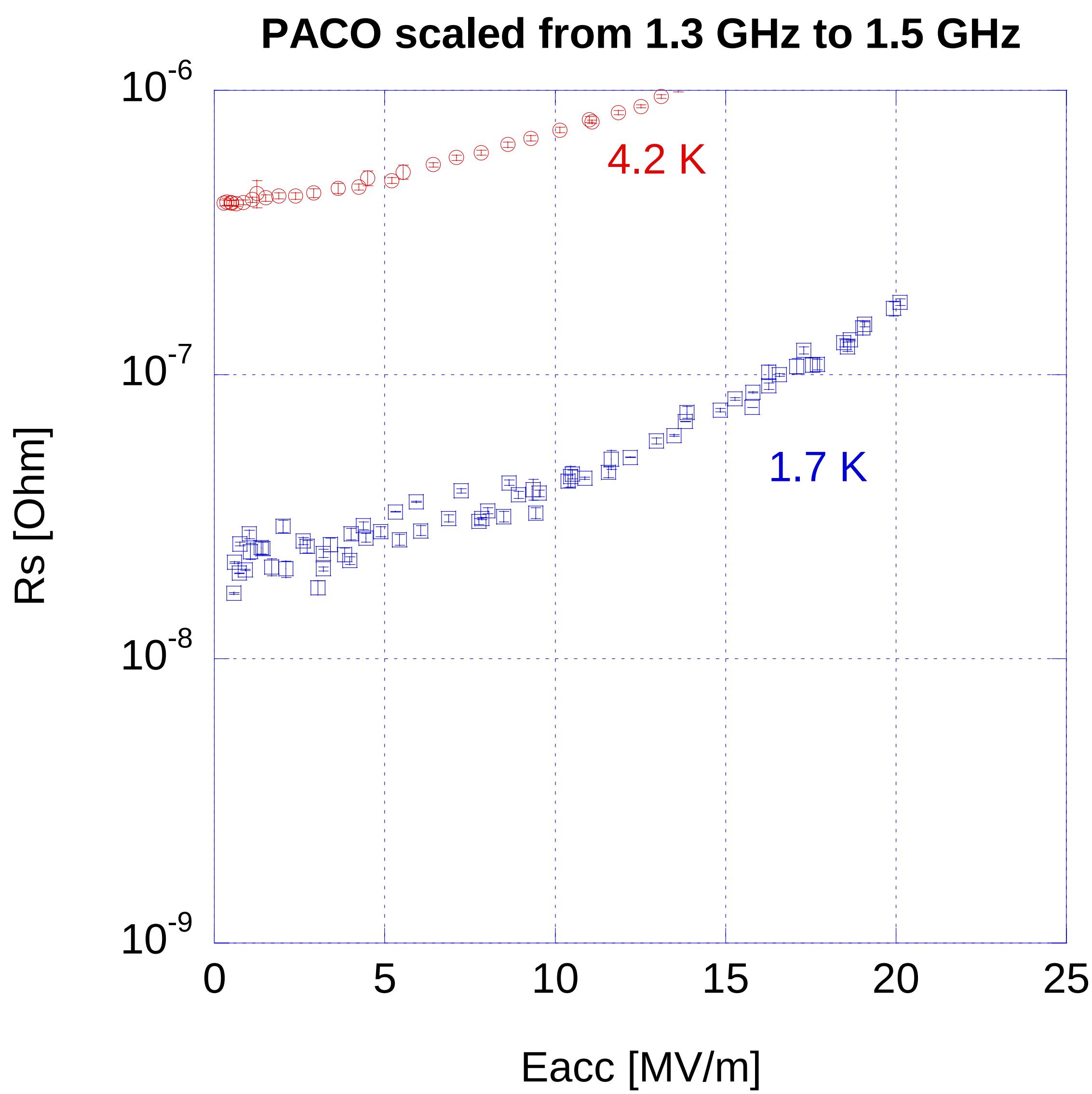
Angle of incidence in spherical cavity

Average incidence angle of the Nb coating. The abscissa is the length in mm along the cavity axis.





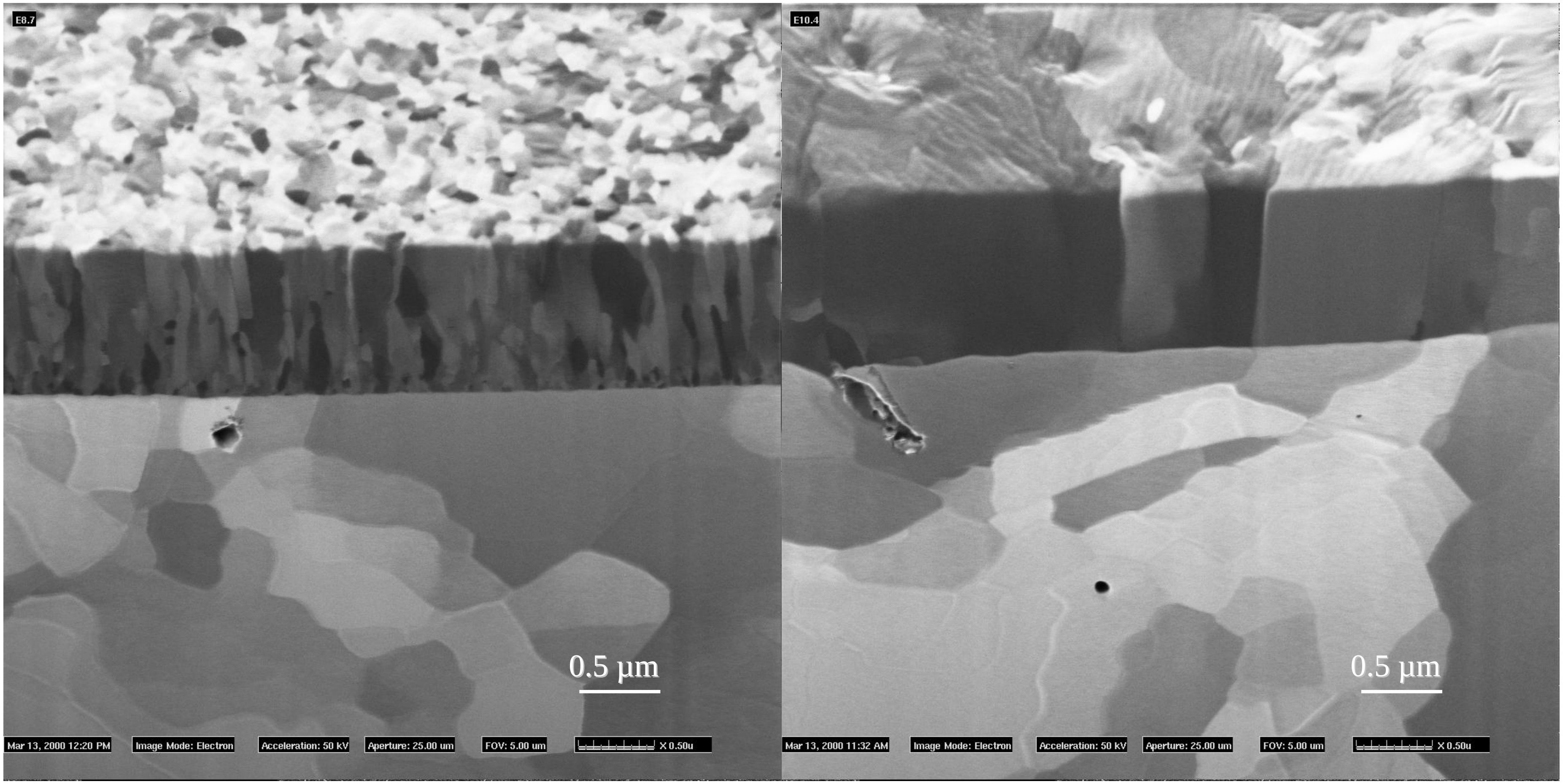
Surface resistance of spherical cavity



Film Structure – FIB cross sections

Grain size with Focussed Ion Beam micrographs

Standard films | Oxide-free films

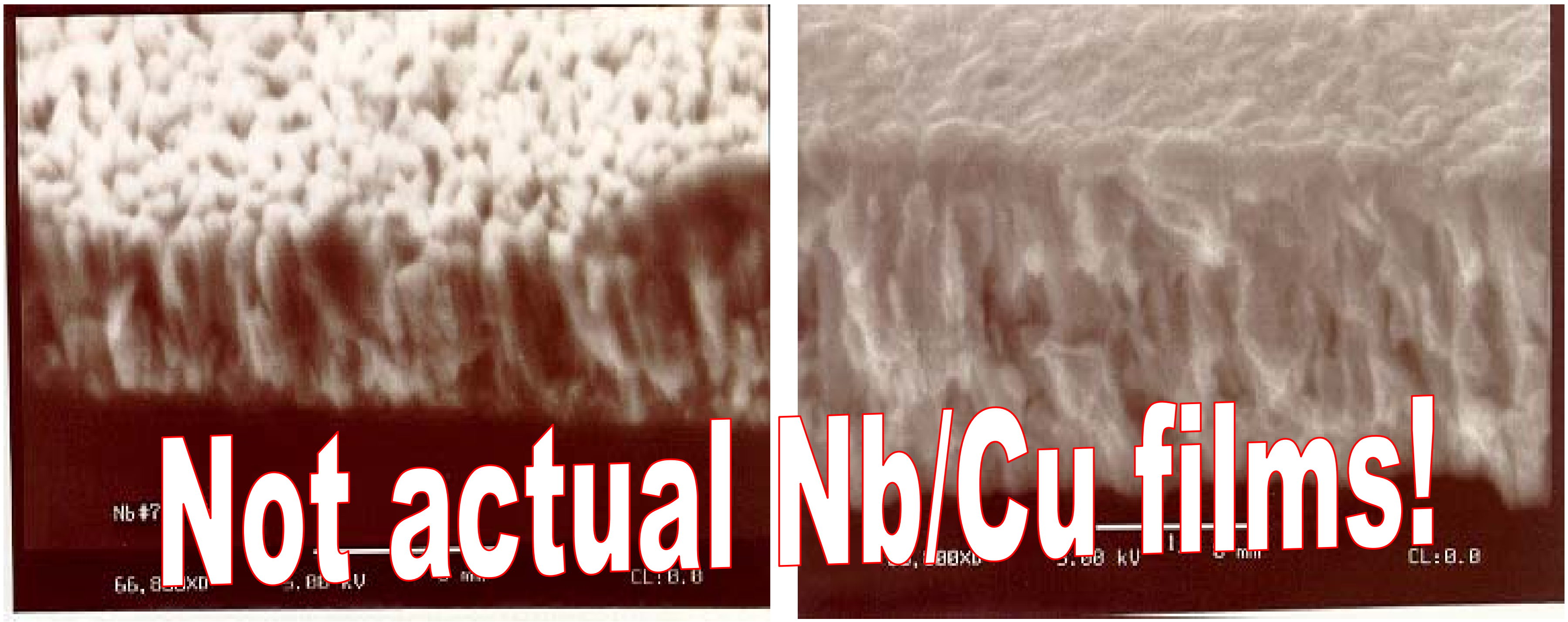


Courtesy: P. Jacob - EMPA

X

Some Nb films...

From talk of Rong-Li Geng at SRF 2003



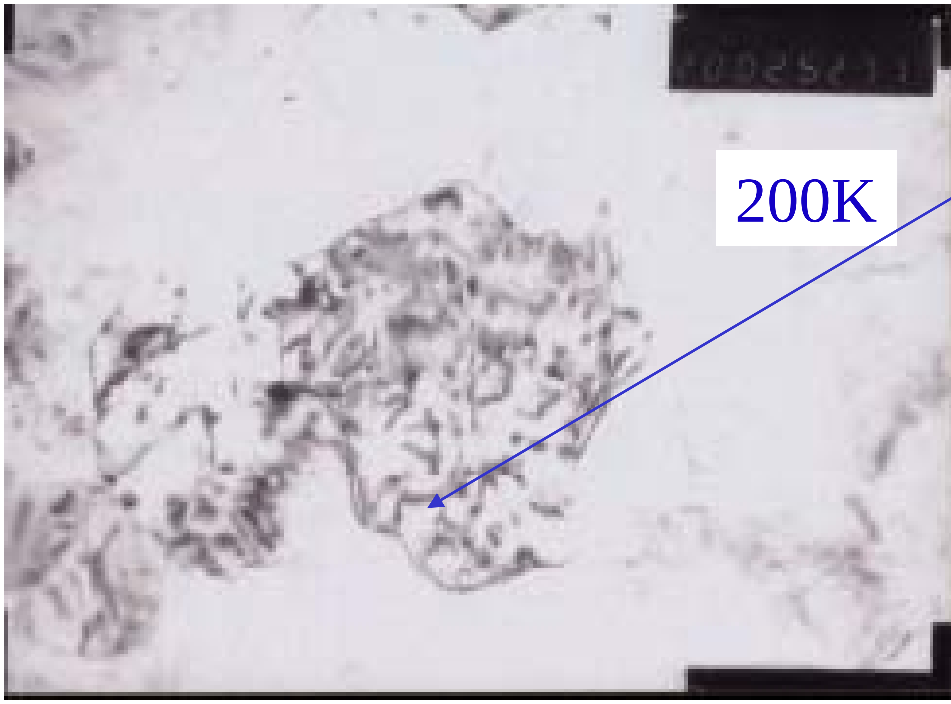
Without bias

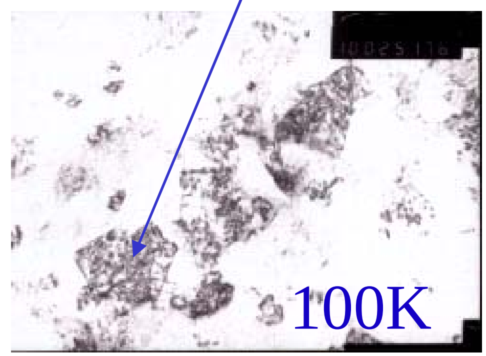
With bias voltage

Photo credit: K. Zhao et al., PKU

Defects – TEM views

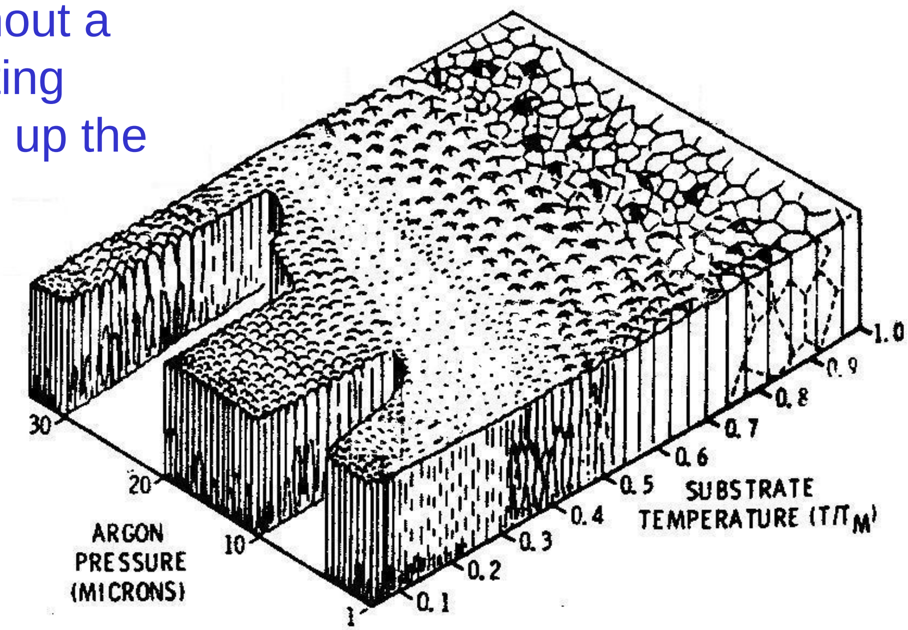
Crystallographic defects





High-energy deposition techniques

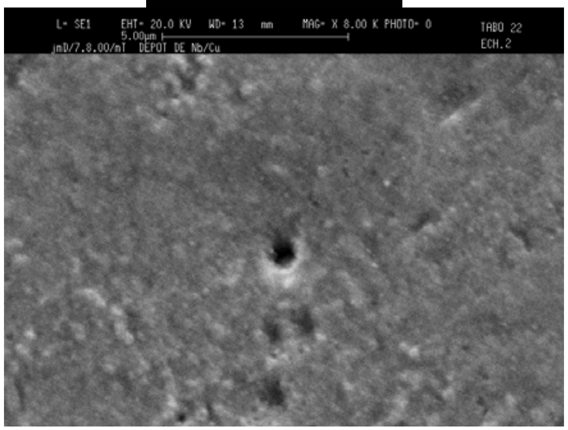
- Crystalline defects, grains connectivity and grain size may be improved with an higher substrate temperature which provides higher surface mobility (important parameter is $T_{substrate}/T_{melting\_of\_film}$)
- However the Cu substrate does not allow much freedom
- The missing energy may be supplied directly by ion bombardment
  - In bias sputter deposition a third electron accelerates the noble gas ions, removing the most loosely bounded atoms from the coating, while providing additional energy for better 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



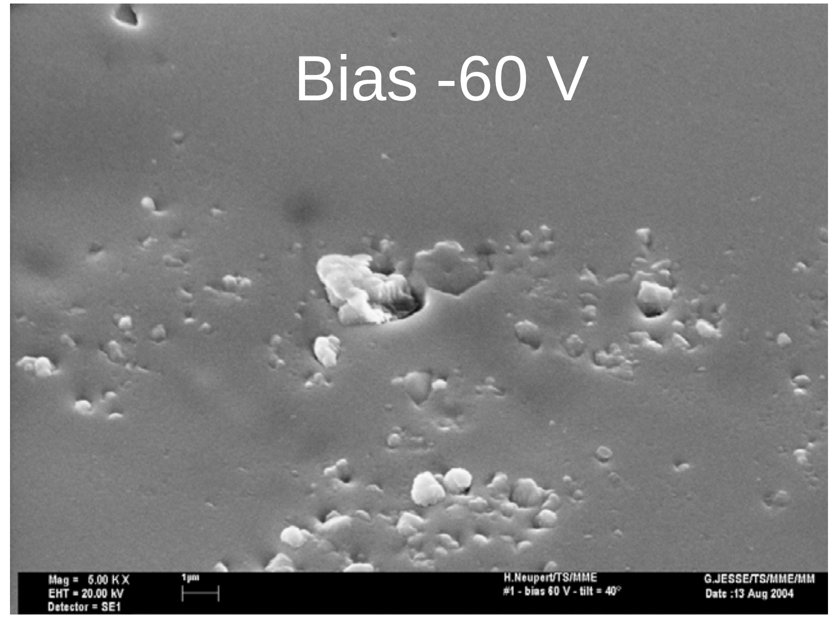
"Structure Zone Model"

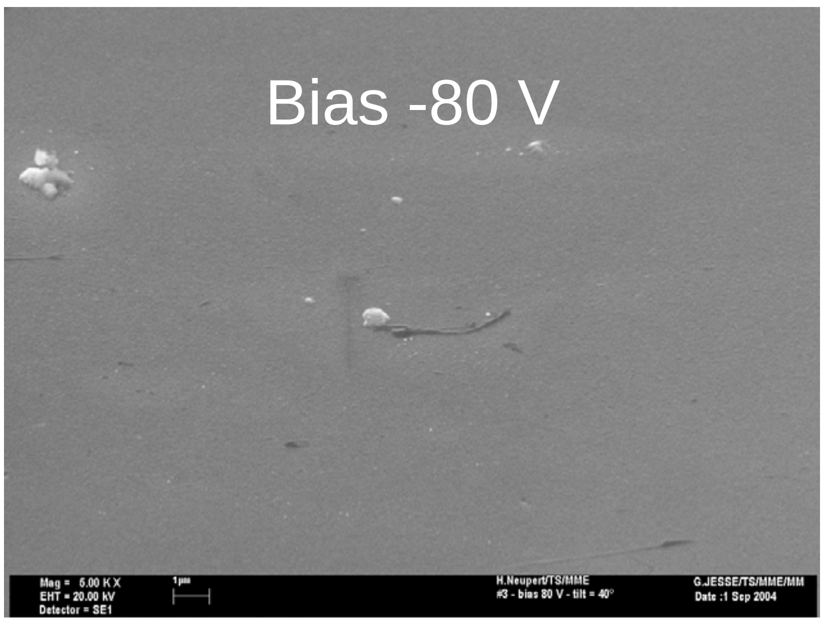
Nb/Cu bias deposition – First SEM images at CERN

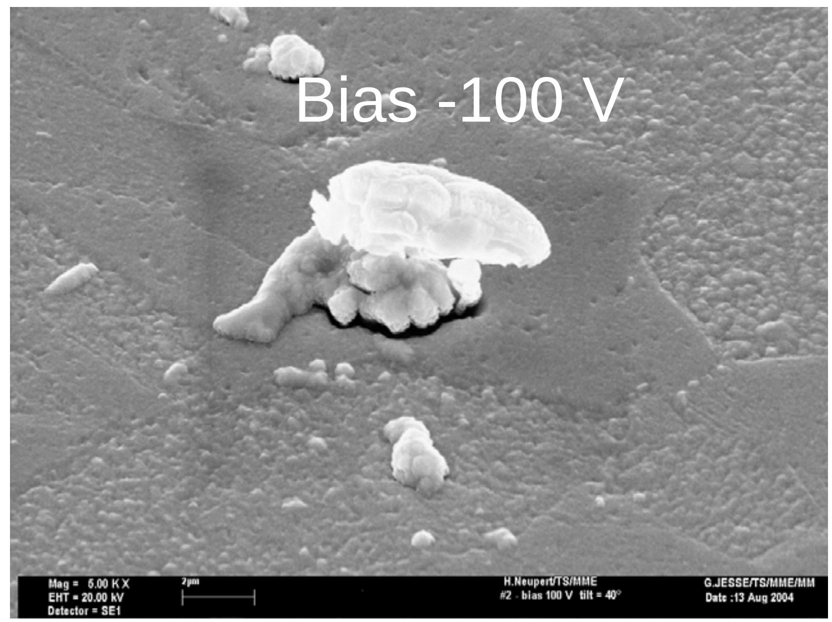
No bias



5 µm

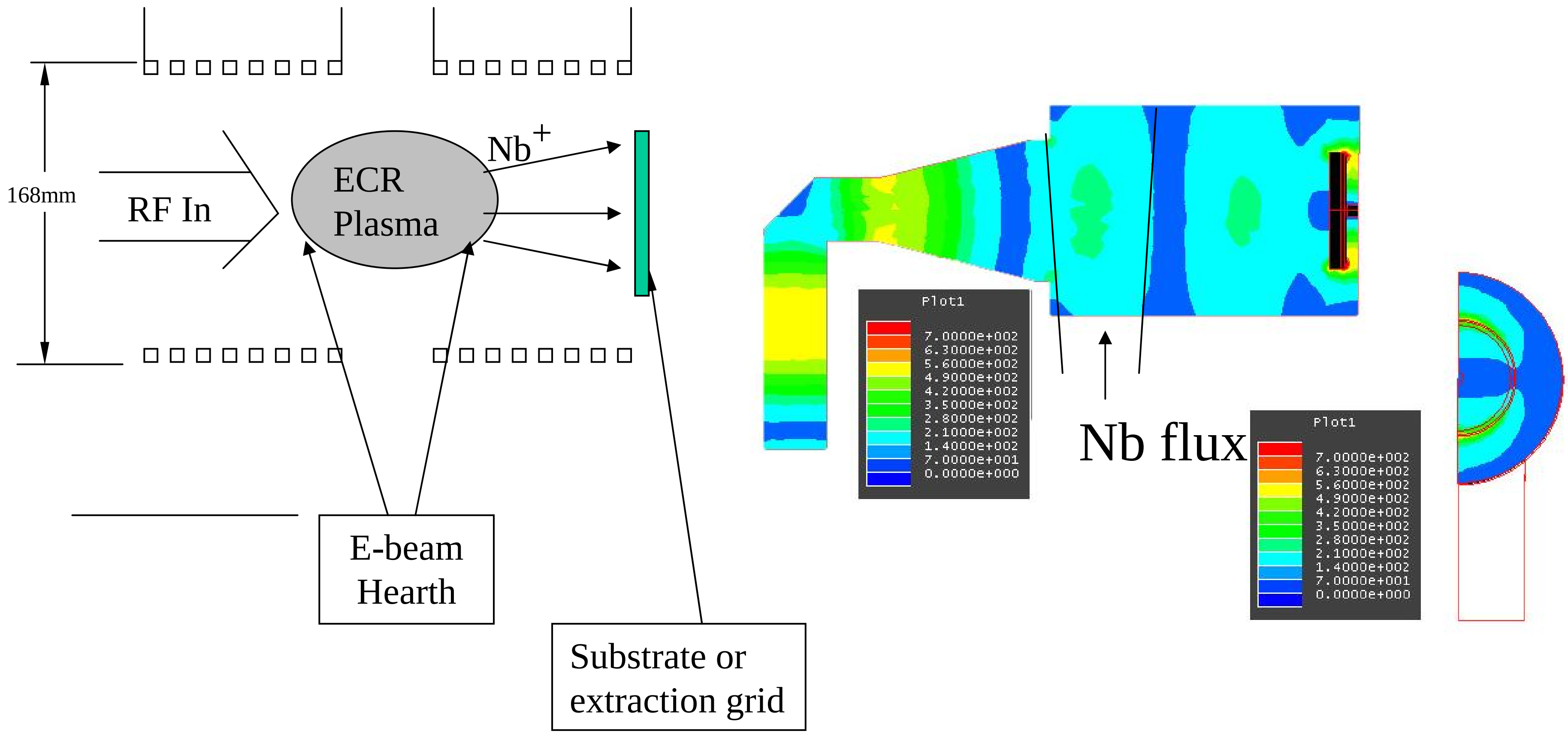






Evaporation + ECR (JLAB)

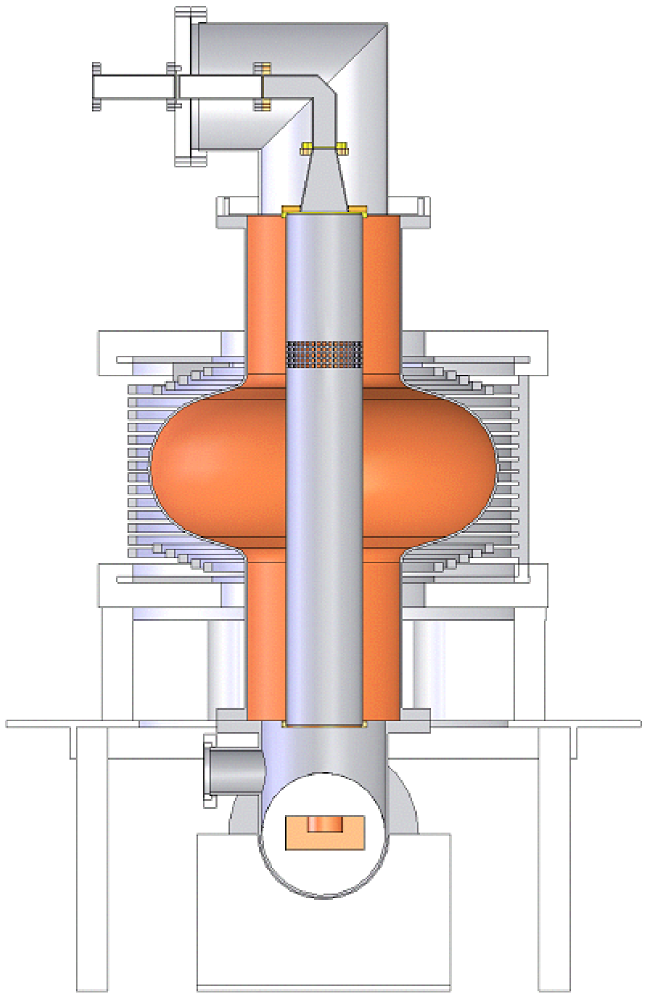
- Niobium is evaporated by e-beam, then the Nb vapours are ionized by an ECR process. The Nb ions can be accelerated to the substrate by an appropriate bias. Energies in excess of 100 eV can be obtained.



From: G. Wu

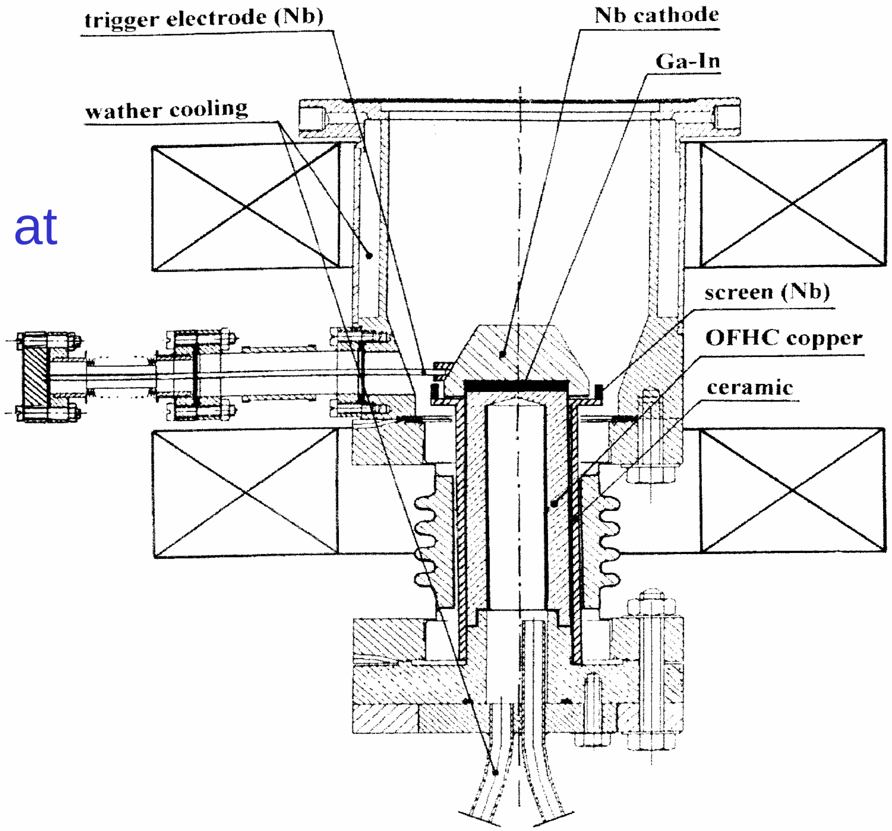
Application to cavities (JLAB)

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



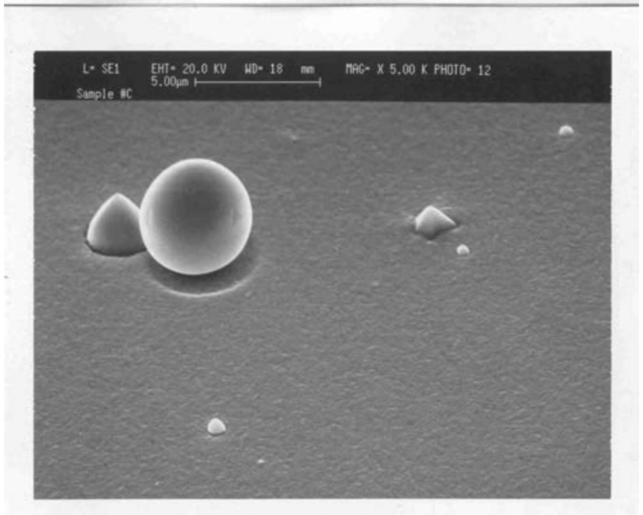
Plasma Arc (INFN)

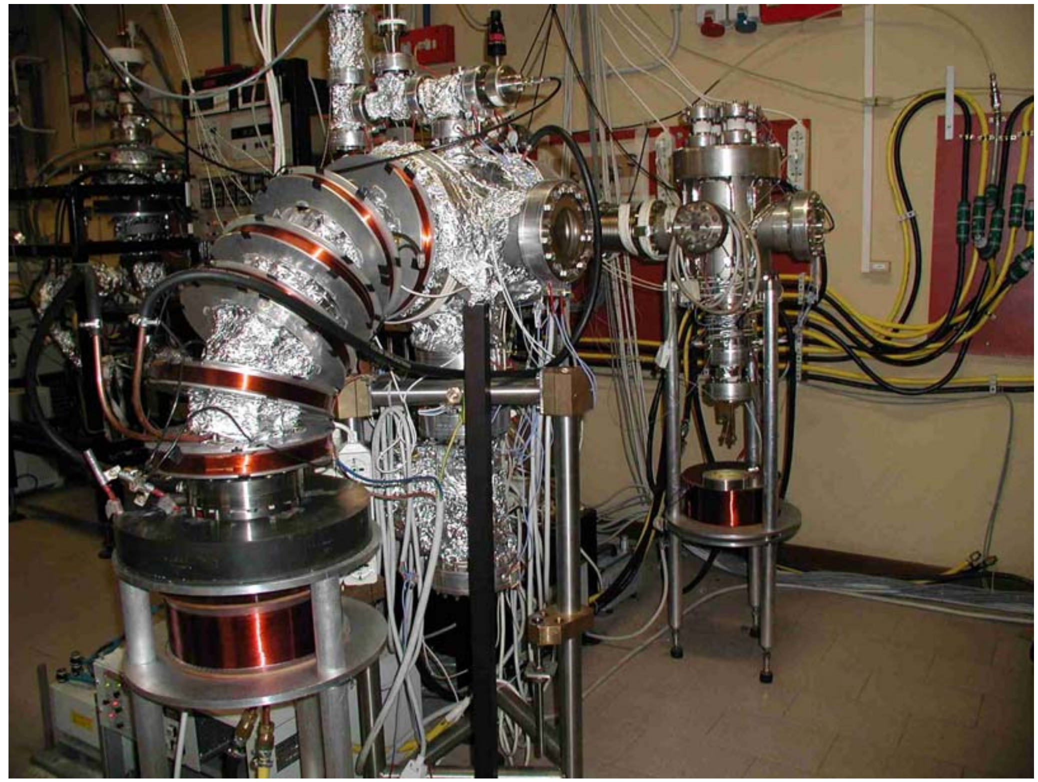
- In the plasma arc an electric discharge is established directly onto the Nb target, producing a plasma plume from which ions are extracted and guided onto the substrate by a bias and/or magnetic guidance
- Magnetic filtering (and/or arc pulsing) is also necessary to remove droplets
- A trigger for 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

Plasma Arc – Need for filtering (INFN)





Magnetic filtering

Nb droplets

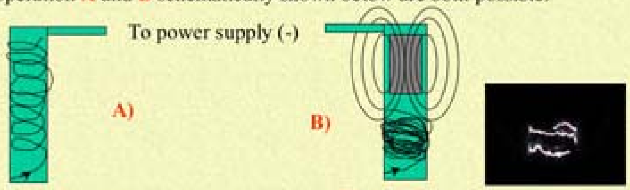
Liner arc for cavity deposition (INFN)

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.

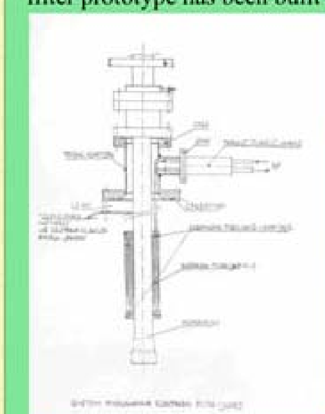
Laboratory in Rome: Cylindrical arc system and planar filtered arc

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

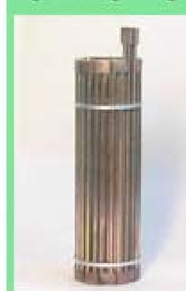
The laboratory setup in Swierk in which the UHV system equipped with a cylindrical arc source shown in the photograph has been recently put into operation.

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.

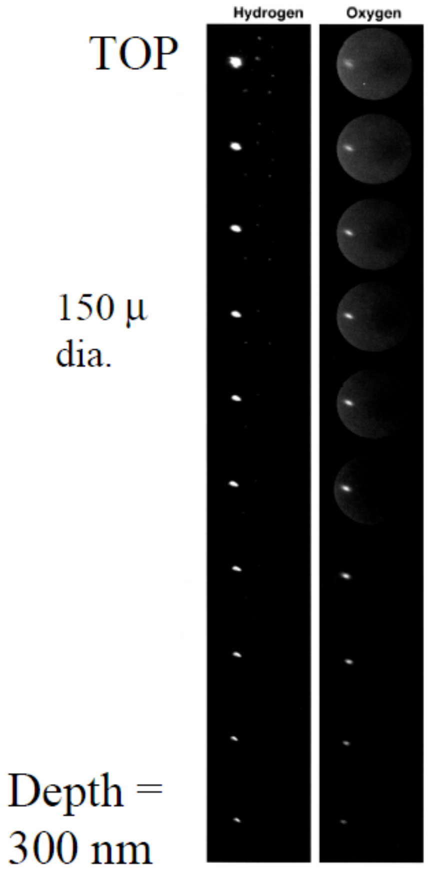


The filter, shown below, works as follows: a current driven through the copper tubing water cooled structure generating a magnetic field that guides plasma electrons (and ions) through the small gaps between pipes. There being no direct line of sight from cathode to anode, heavy particles can instead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



Film as a bulk – High temperature annealing

Annealing is a good option to increase the grain size and remove defects (CERN Proc. SRF1997). Innovative solution: Nb/Mo cavities + annealing at 800 ºC. This would be effective also in removing hydrogen, which has been found in large amounts in films (Saclay + CERN + Cornell)



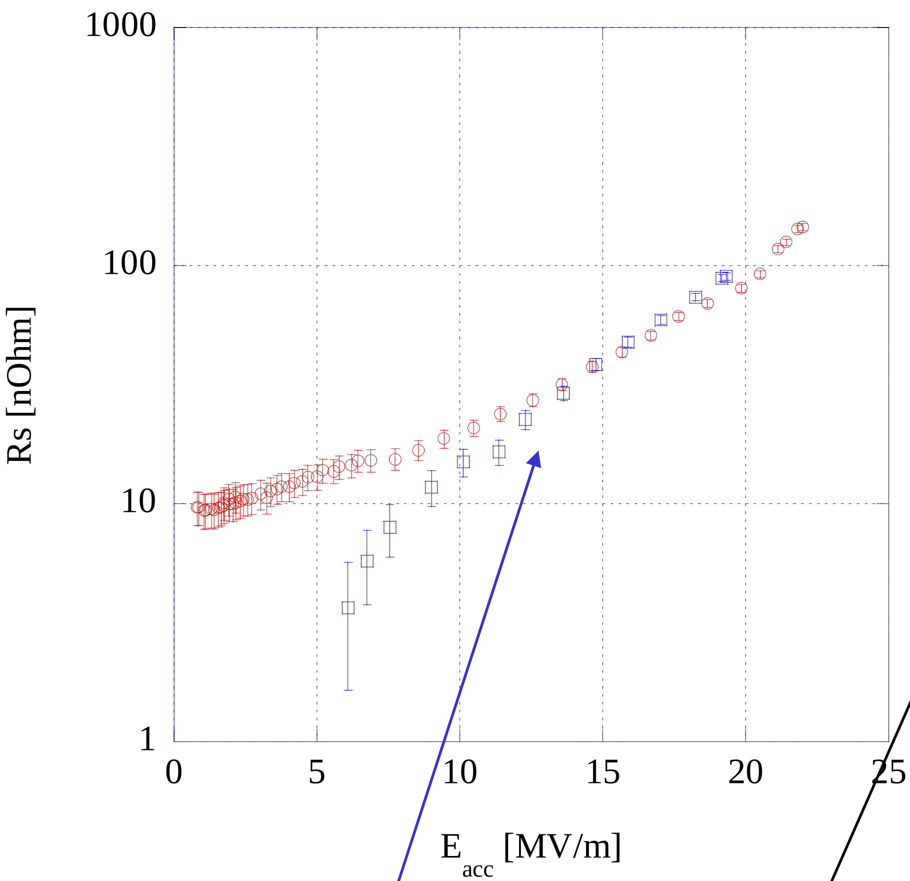
Depth profile in 30 nm steps, proceeding from top of film towards bottom.

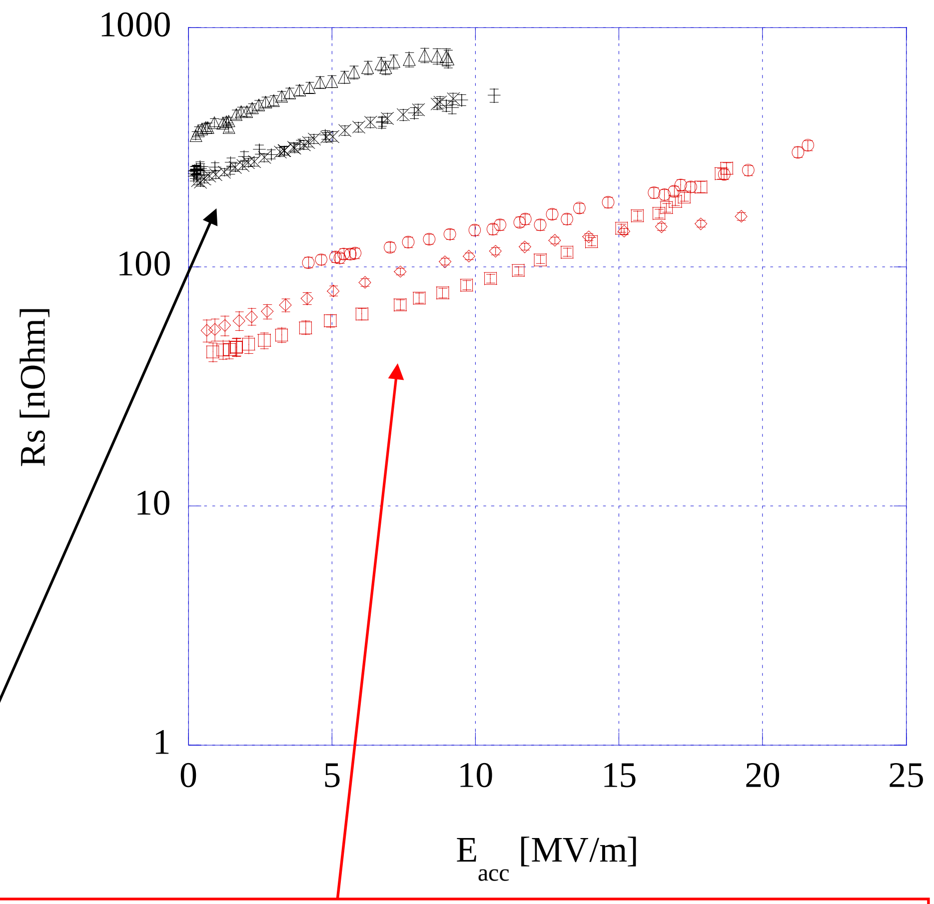
Notice the conjunction of H+O in the brightest "river". H visible here cannot be α phase.

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.

From: L. Hand

Films as a bulk – Effect of hydrogen





Coatings on oxidized copper substrate

Coatings on oxide-free copper (epitaxial growth, large grains, high RRR)

Coatings on oxide-free copper substrate annealed at 350 ºC (larger grains, higher RRR)

X

Annealed Cu grain size

50 µm ↕

Meridian cut

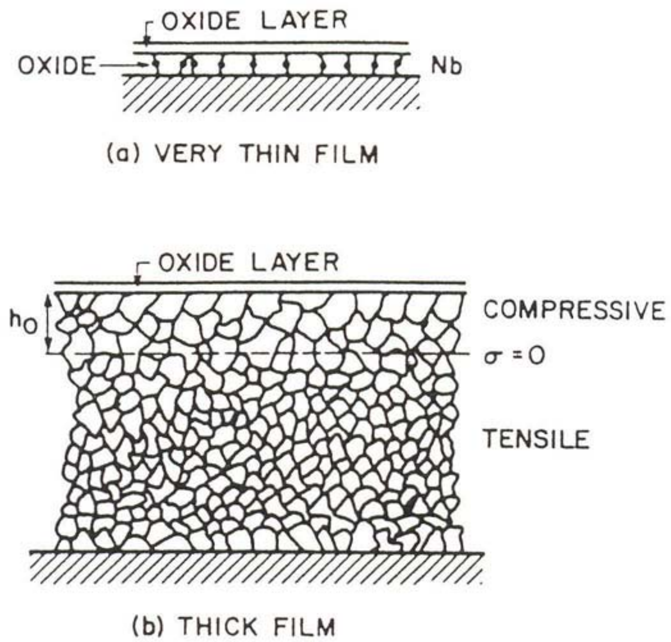
Annealed at 350 ºC

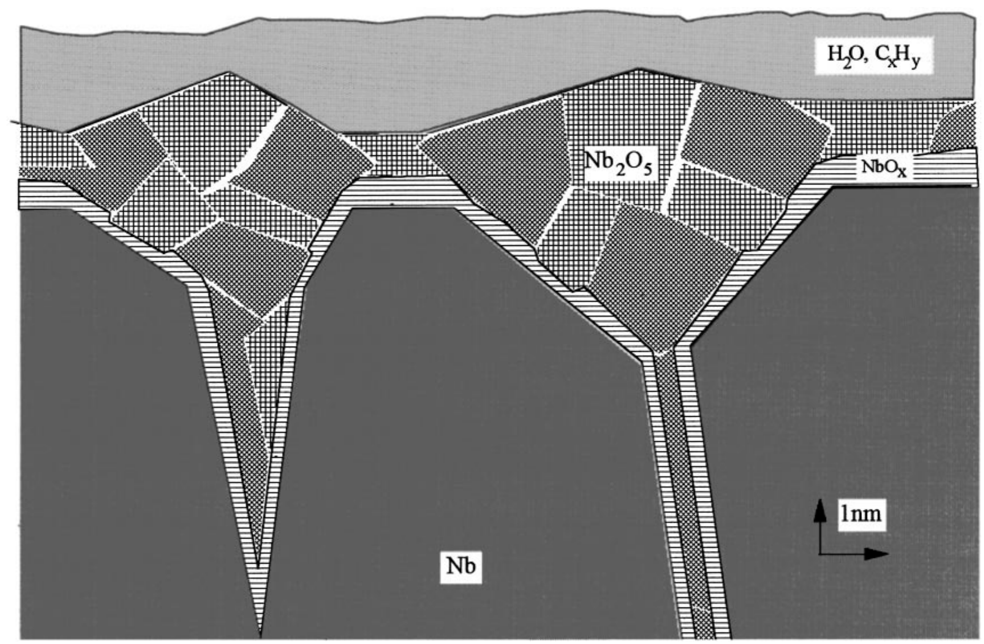
Standard Cu

50 µm ↕

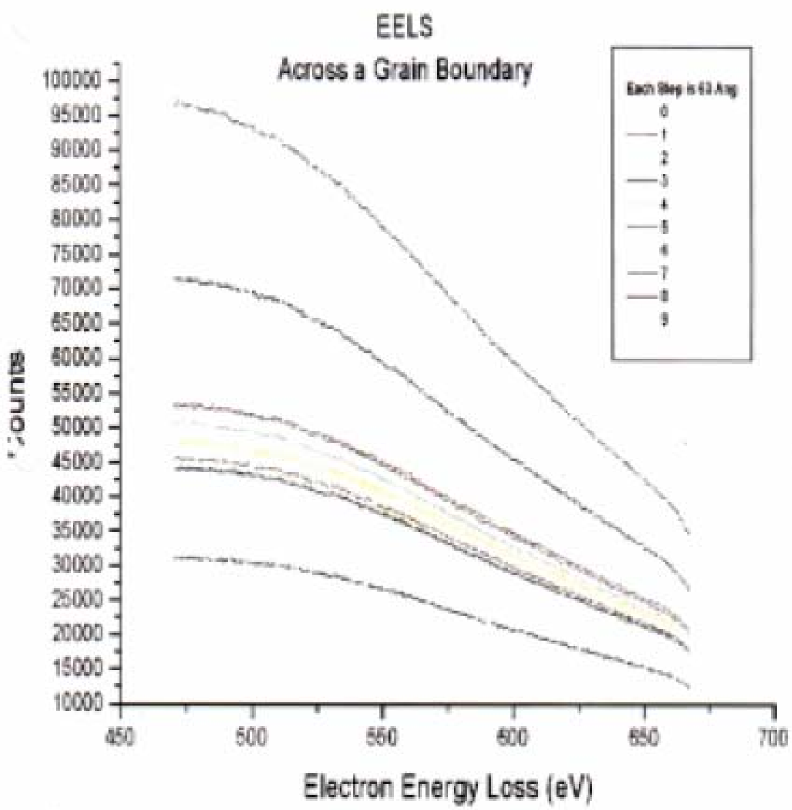
Equator cut

Grain boundaries and surface oxidation

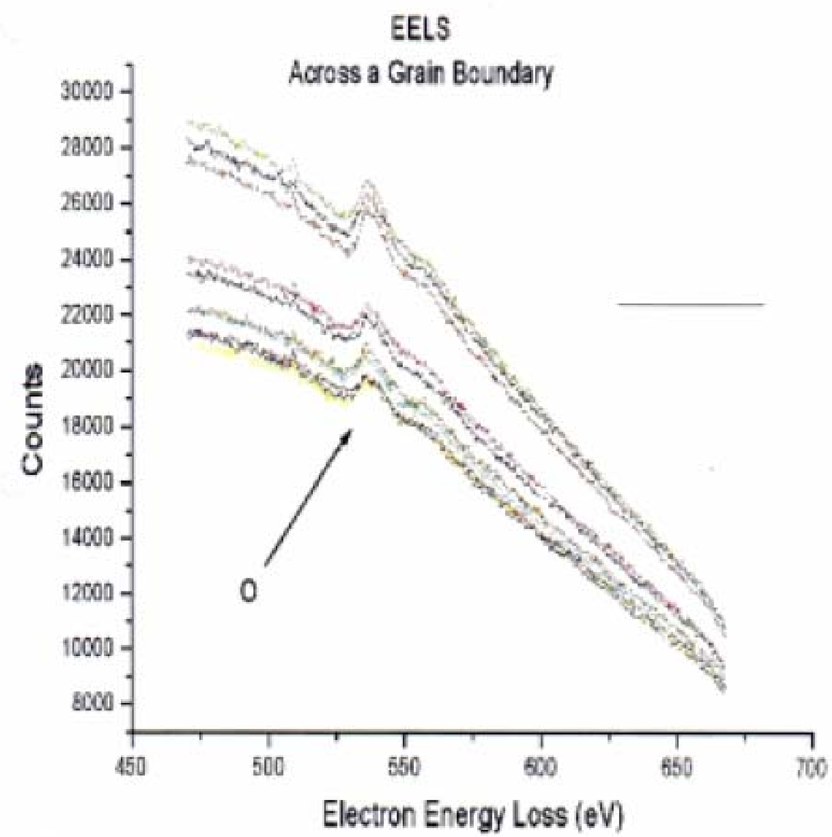




Famous drawings by Halbritter. Several effects might take place: ITE, flux penetration, H_{c1} depression, lower Tc, etc.



EELS scan showing no oxygen across a grain boundary



EELS scan showing oxygen across a grain boundary

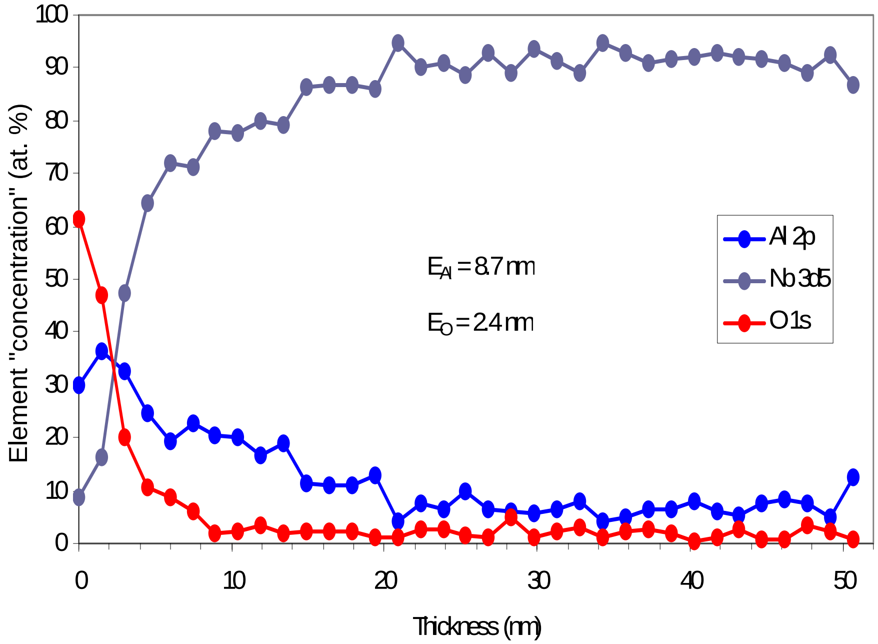
Right-hand graph shows presence of oxygen. Ultimate resolution of EELS is about 1 nm.

From: L. Hand

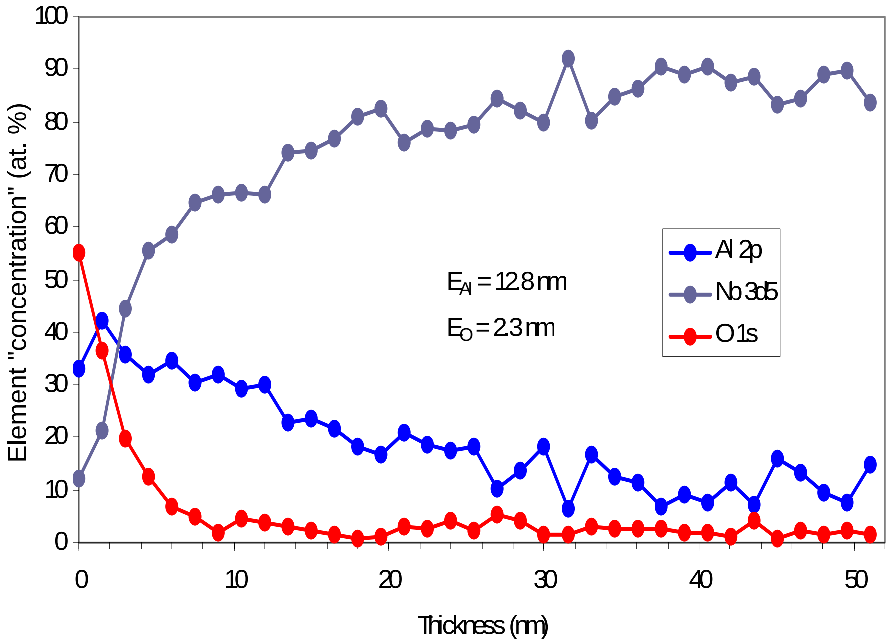
Possible solution – Al_2O_3 cap layers

- 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.

XPS depth profile



5 nm Al (nominal)



10 nm Al (nominal)

Conclusion

- Niobium films are a real option for accelerators at any beta
- The technique of choice is at present sputter deposition: a prerequisite for it is the understanding and the optimisation of substrate design and preparation

- High-field R_{res} increase of Nb/Cu films: several theories & ideas, no proven ones.
- Research into more bulk-like films is the general trend. Of course hydrogen will then be the main enemy.