Recent development in deposition of niobium films by UHVCA in SRF cavity

WP4.2

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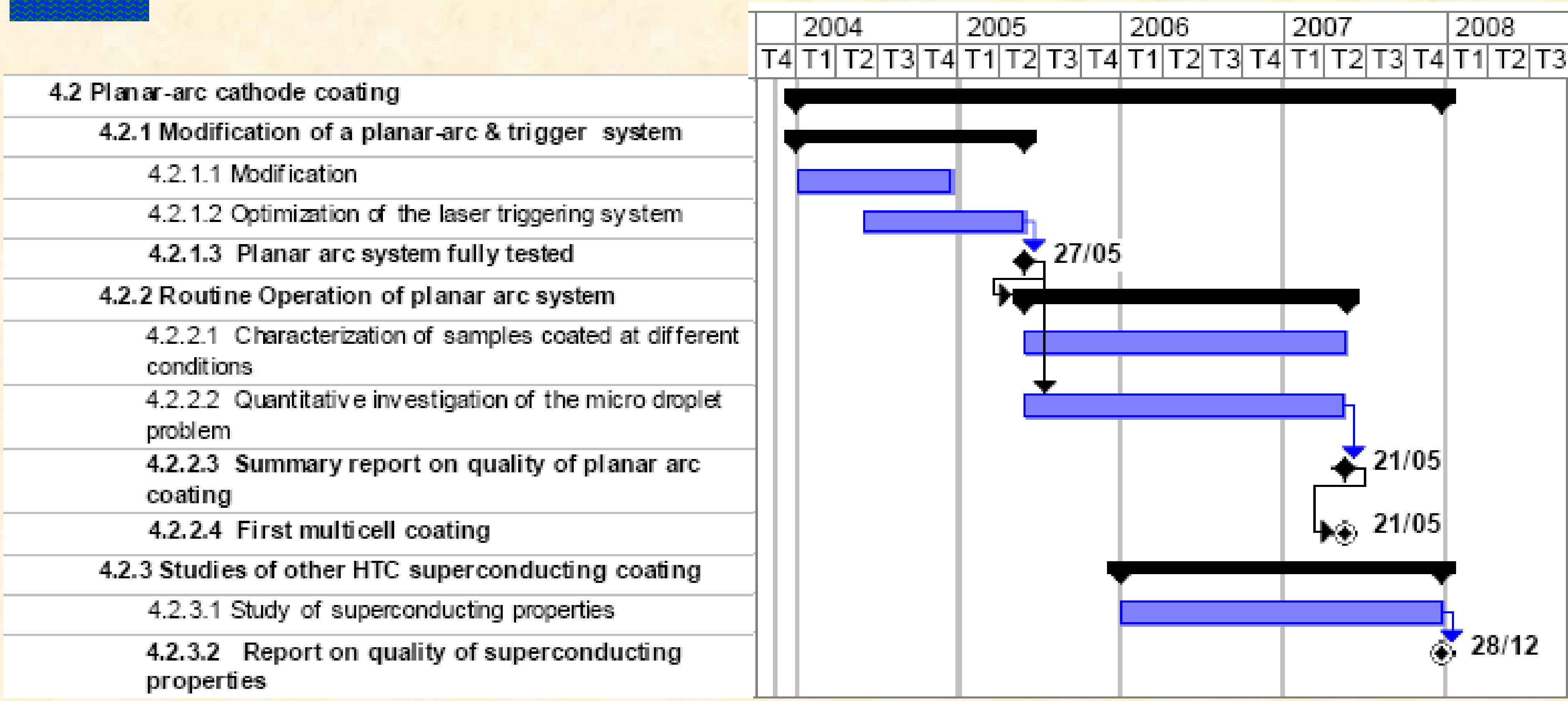
And also in collaboration with Saclay, DESY, Jlab, CERN, INFM...

Outline

- UHV arc set-up
- Results using the UHV arc sources
- The plasma transport
- Cavity deposition
- Problems and possible solutions

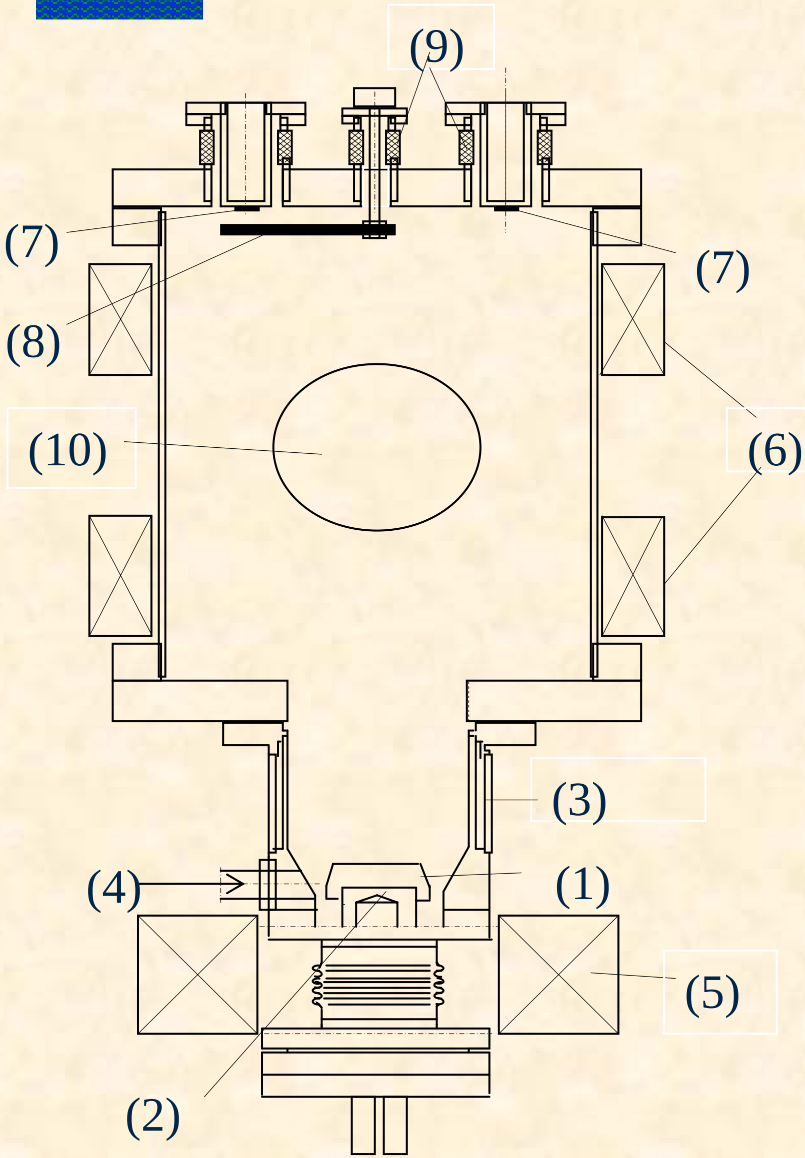


4.2 Planar-arc cathode coating

- 4.2.1 Modification of a planar-arc & trigger system
  - 4.2.1.1 Modification
  - 4.2.1.2 Optimization of the laser triggering system
  - **4.2.1.3 Planar arc system fully tested**
- 4.2.2 Routine Operation of planar arc system
  - 4.2.2.1 Characterization of samples coated at different conditions
  - 4.2.2.2 Quantitative investigation of the micro droplet problem
  - **4.2.2.3 Summary report on quality of planar arc coating**
  - **4.2.2.4 First multicell coating**
- 4.2.3 Studies of other HTC superconducting coating
  - 4.2.3.1 Study of superconducting properties
  - **4.2.3.2 Report on quality of superconducting properties**

No.	Task	Begin. of task	End of task	Status sept. 2007
4.2	Planar-arc cathode coating	01-01-04	30-11-07	**95%**
4.2.1	Modification of a planar-arc & trigger system	01-01-04	03-09-04	**100%**
4.2.1.1	Modification	01-01-04	16-04-04	**100%**
4.2.1.2	Optimization of the laser triggering system	19-04-04	03-09-04	**100%**
4.2.1.3	Planar arc system fully tested	03-09-04	27-05-05	**100%**
4.2.2	Routine operation of planar arc system	06-09-04	27-05-05	**100%**
4.2.2.1	Characterization of samples coated at different conditions	06-09-04	30-06-07	**95%**
4.2.2.2	Characterization of Nb-coated sapphire samples	06-12-04	08-02-05	**100%**
4.2.2.3	Characterization of Nb-coated copper samples	09-02-05	30-06-07	**95%**
4.2.2.4	Summary report on quality of planar arc coating	27-05-05	27-05-05	**100%**
4.2.3	Studies of other HTC superconducting coating	30-05-05	30-12-07	**50%**
4.2.3.1	Study of Nb film superconducting properties	30-05-05	30-06-07	**60%**
4.2.3.2	Report on quality of superconducting properties	30-06-07	30-11-07	**50%**

Planar arc configuration

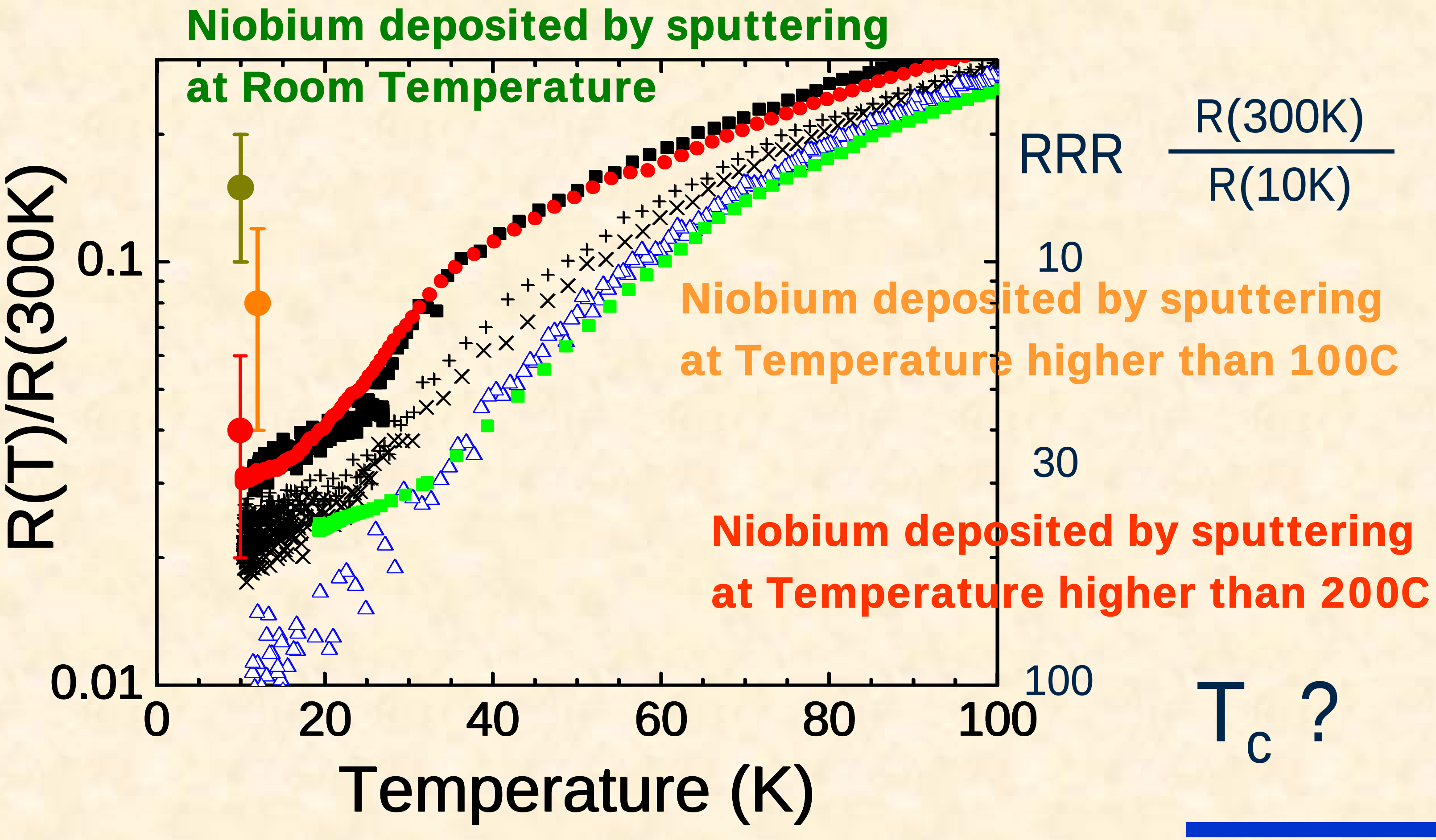


(1) The cathode
(2) Water cooled copper support
(3) Water cooled stainless steel anode
(4) Port for the arc ignition
(5) Magnetic coil
(6) Additional magnetic Helmholtz coil
(7) Sample holder
(8) Rotating shutter
(9) Electrically insulation
(10) CF100 pumping port

Cathode-substrates distance 50cm

R(T) Measurement

Nb film deposited by UHVCA on sapphire and copper at **room temperature**

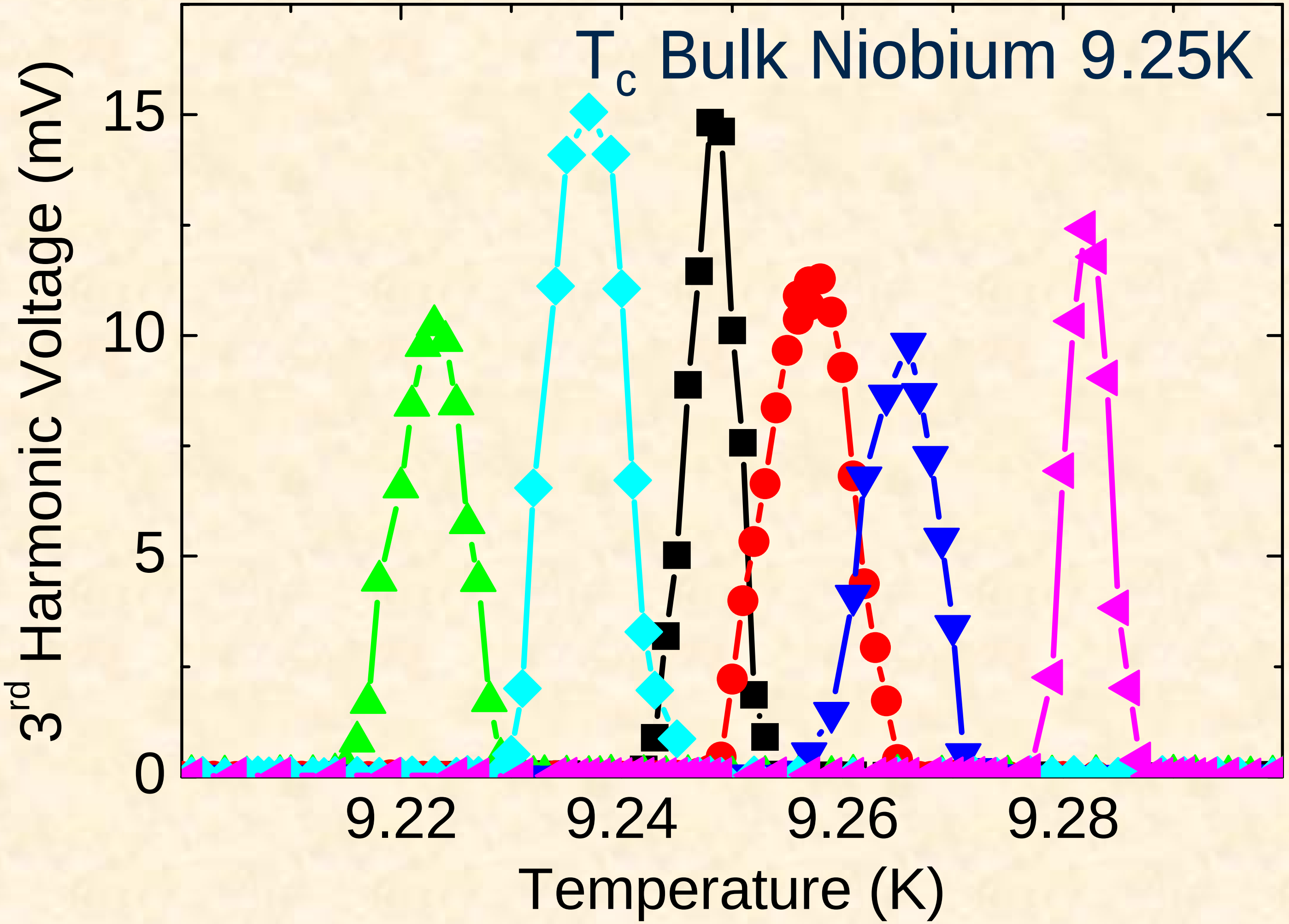


Niobium deposited by sputtering at Room Temperature

Niobium deposited by sputtering at Temperature higher than 100C

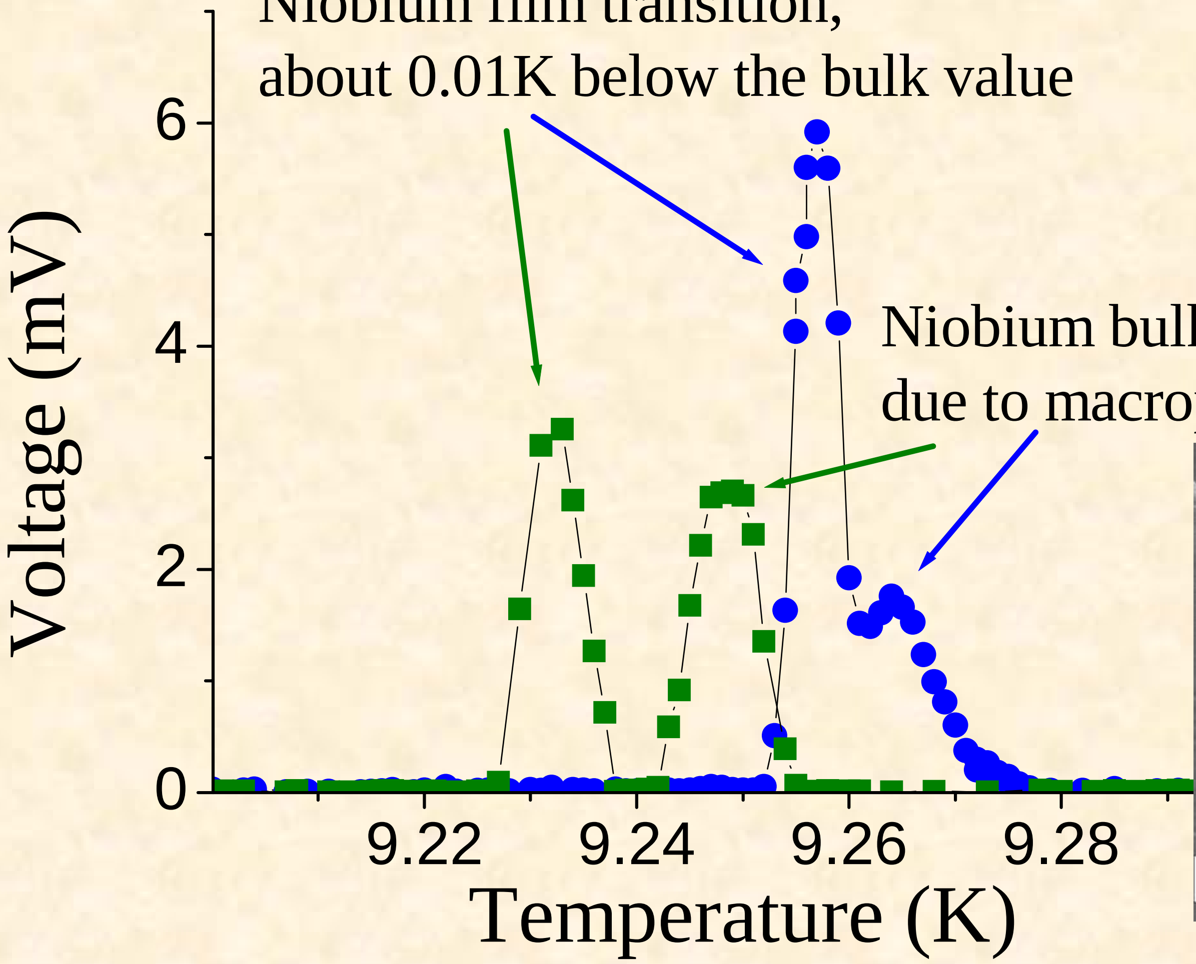
Niobium deposited by sputtering at Temperature higher than 200C

Inductive Tc measurements



R.Russo et al. Supercond. Sci. Technol., 18 (2005) L41-L44

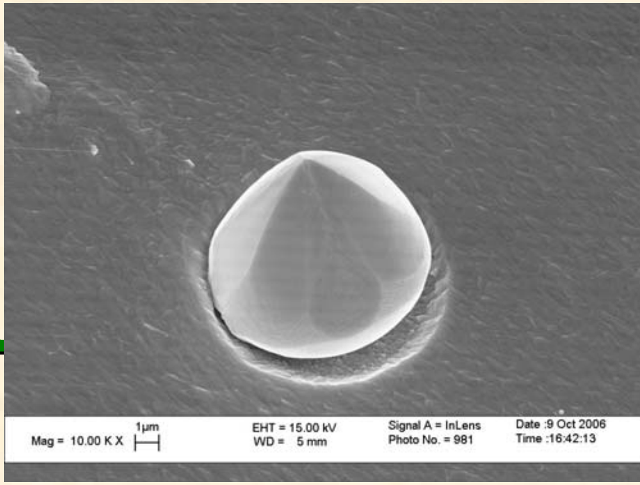
Effect of Macrodroplets on T_c



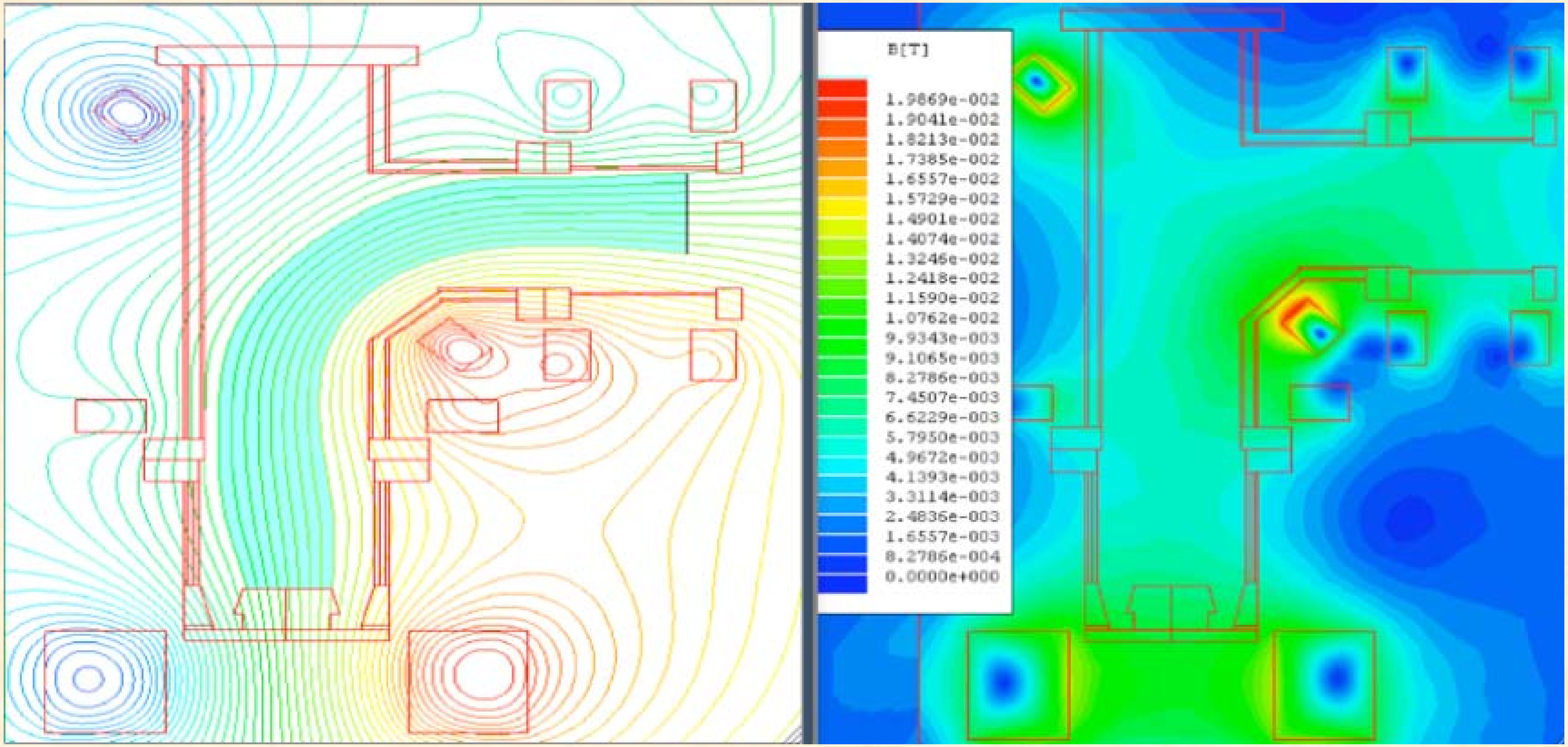
Niobium film transition,
about 0.01K below the bulk value

Niobium bulk transition
due to macroparticles

Nb Bulk has
T_c = 9.26K

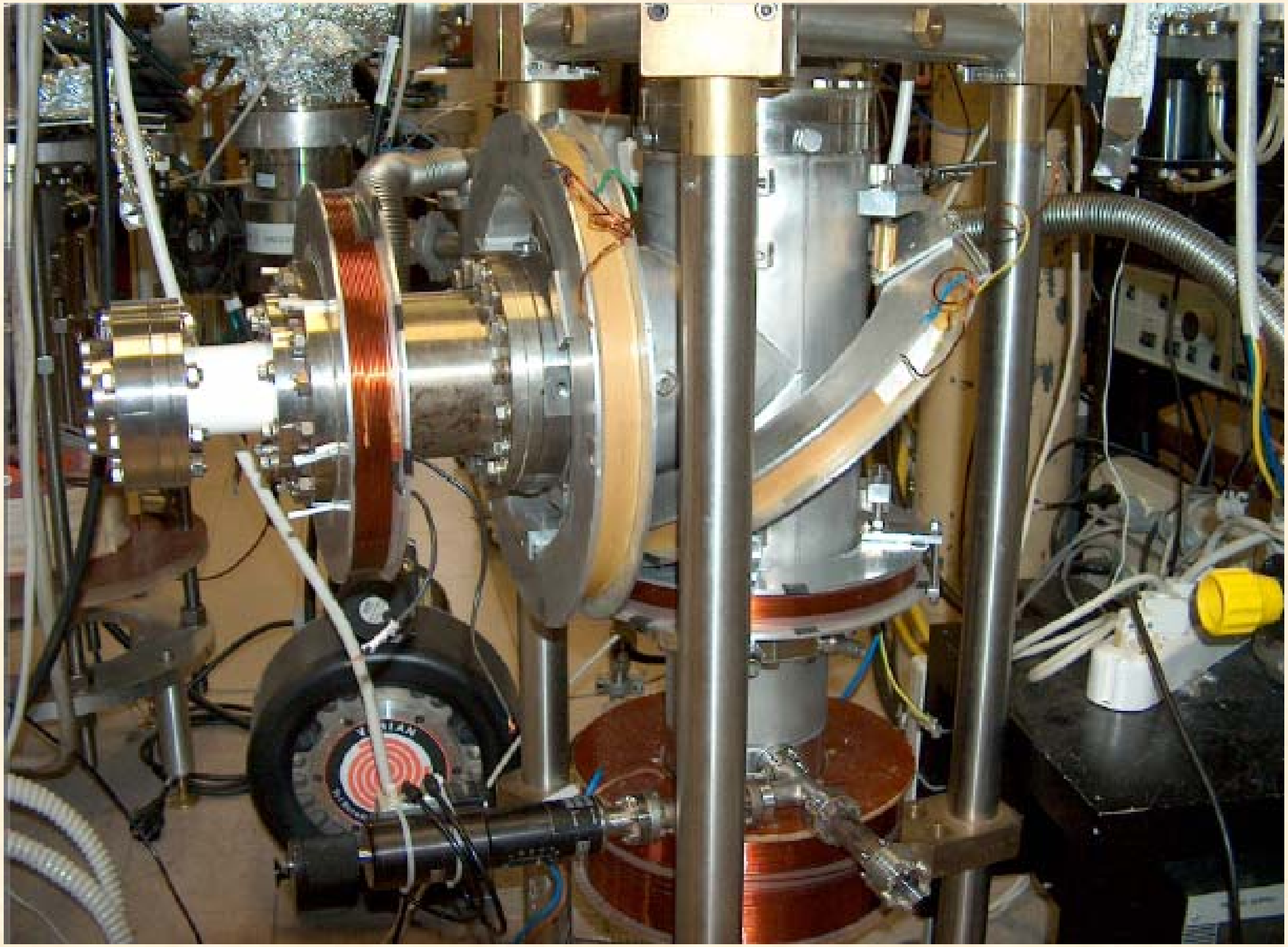


New UHV magnetic filter design

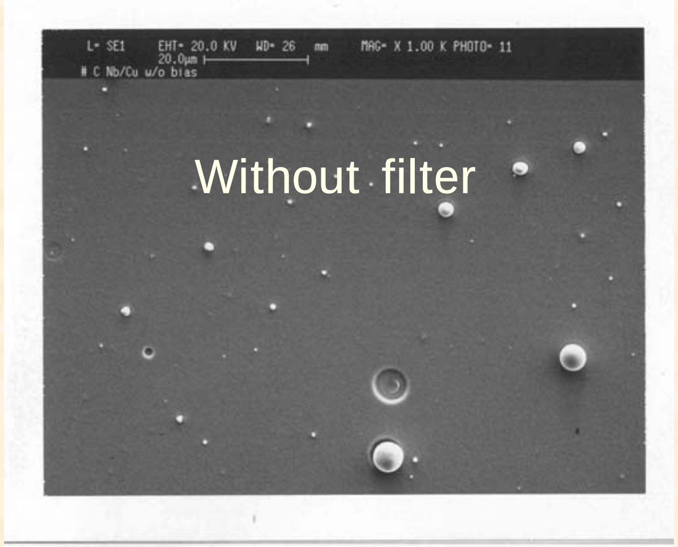


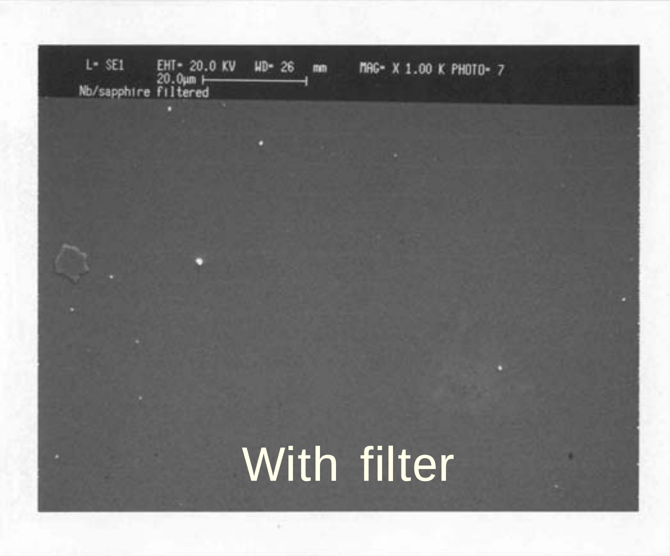
Courtesy of Soltan Institute for Nuclear Study, Swierck

New filter system in operation



Samples deposited using Filter

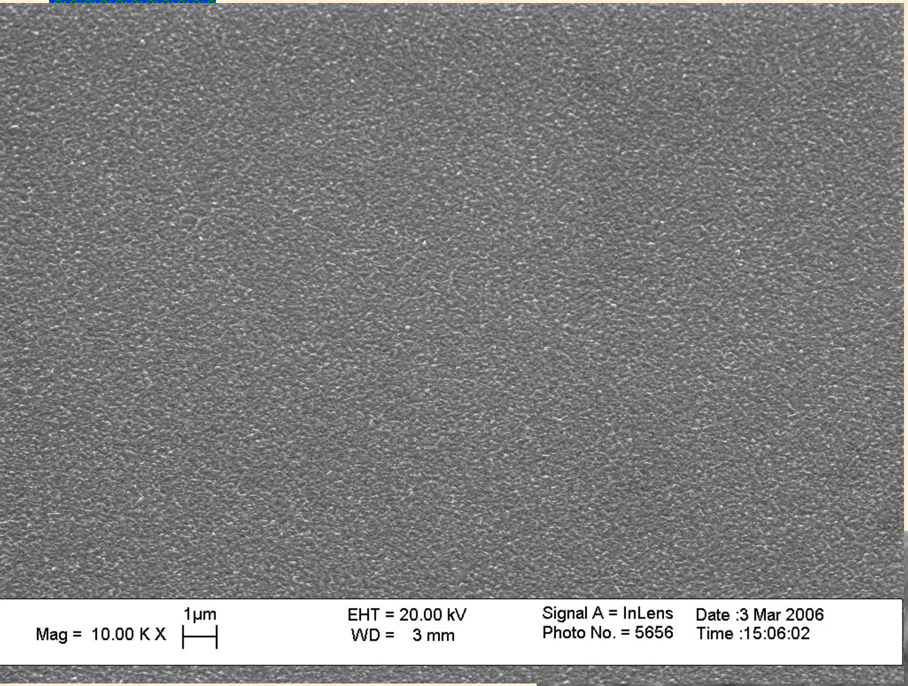


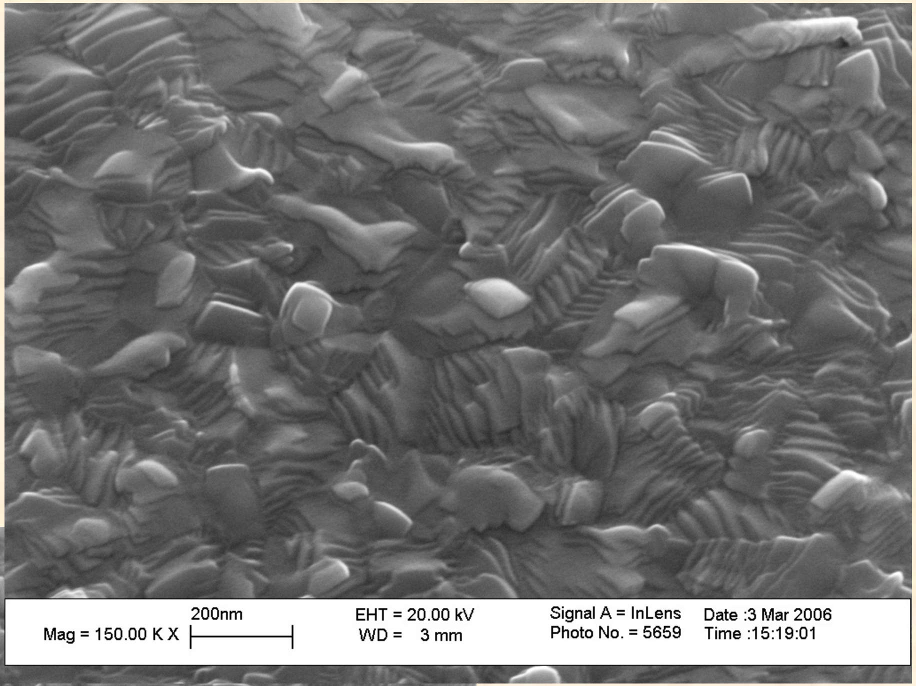


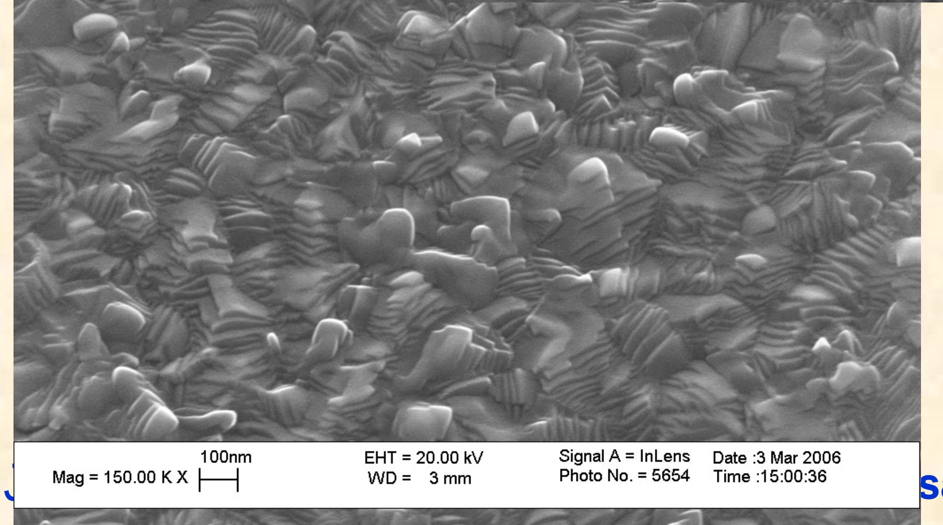
Number and dimensions of microdroplets are drastically reduced using filter.

The number and dimension of macrodroplets are difficoult to estimate because most defects on the surface are not microdroplets but simple small dust particles.

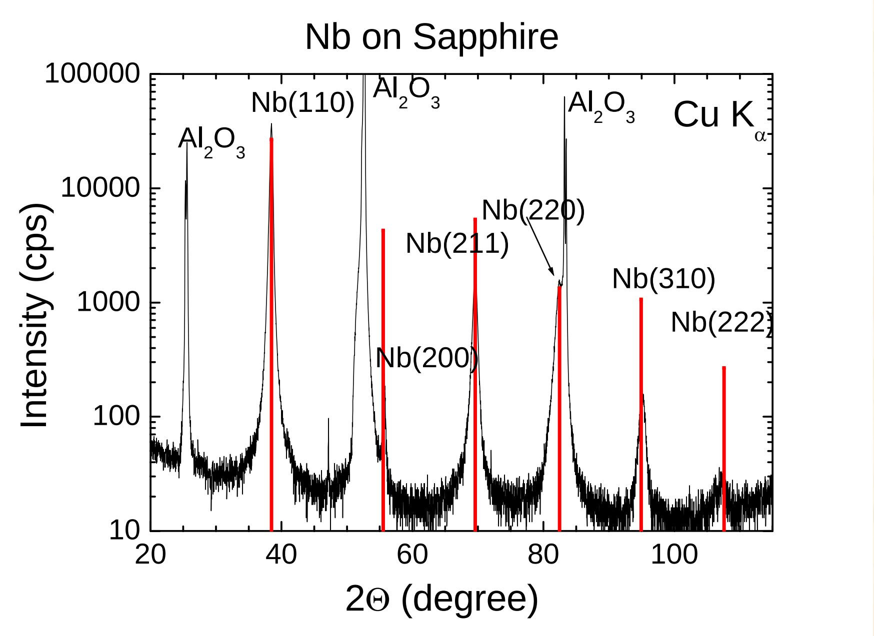
Nb film Morphology as seen by FEG-SEM

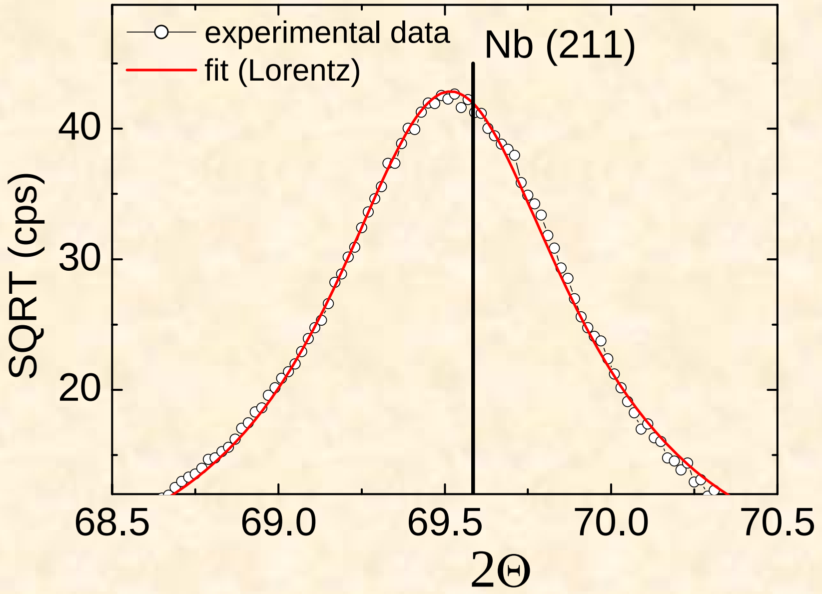






X-Ray diffraction pattern in Θ/2Θ





The best fit using all peaks gives a lattice parameter between 0.3299nm and 0.3313nm.

These values are similar to the bulk 0.3306nm and in agreement with T_c measurement.

The niobium films produced by arc deposition are less stressed than in the sputtering case (UHVCA $\Delta a_\perp/a_\perp < \pm 0.2\%$ Sputtering $\Delta a_\perp/a_\perp = 0.636 \pm 0.096\ \%$)

Lattice parameter Nb/Cu

$\theta/2\theta$

samples	V_{bias}	a_{Cu} (Å)	a_{Nb} (Å)
Pl06_08_Cu2	-23	3.610	**3.301**
Pl06_05_Cu2	-40	3.608	**3.301**
Pl06_04_Cu2	-60	3.610	**3.301**
Pl06_07_Cu2	-80	3.610	**3.302**

$\theta = 5^o$

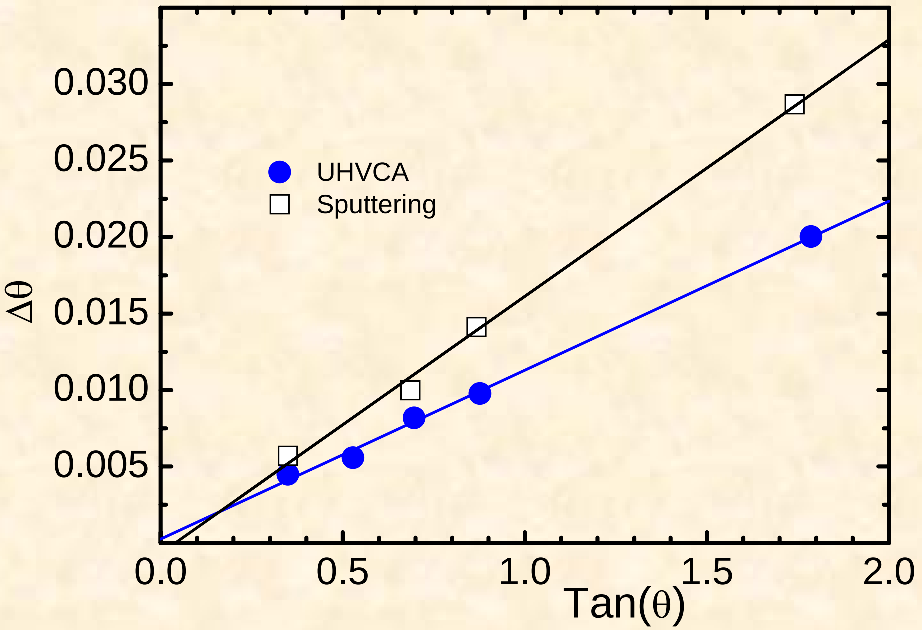
samples	**bias**	a_{Cu} (Å)	a_{Nb}(Å)
Pl06_08_Cu2	-23	3.610	**3.301**
Pl06_05_Cu2	-40	3.613	**3.299**
Pl06_04_Cu2	-60	3.611	**3.300**
Pl06_07_Cu2	-80	3.609	**3.301**

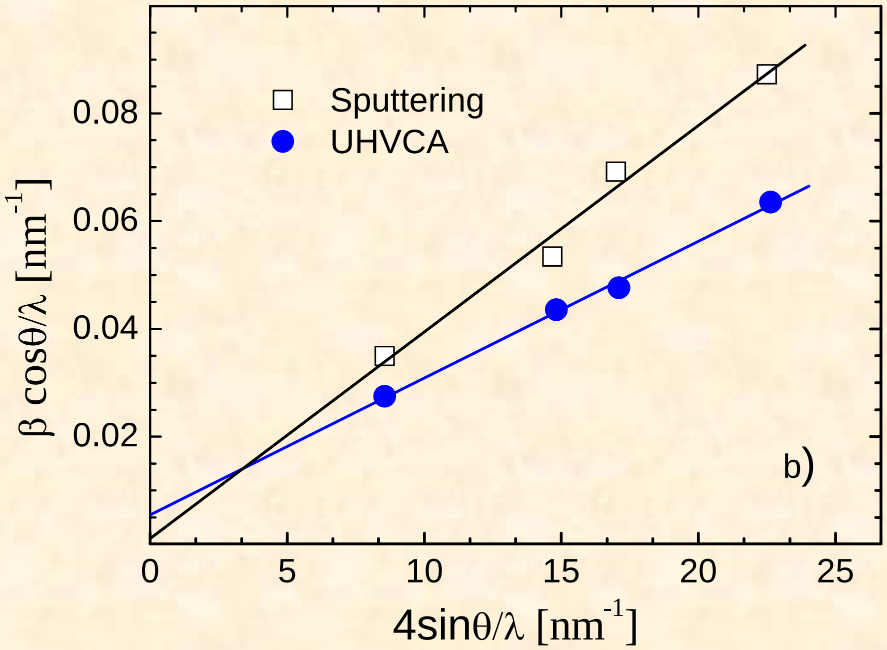
Nb Bulk 3.303 or 3.306 Å

$2\sigma = 0.003$ Å

Our Niobium film on copper substrate present the Nb bulk lattice parameter for all invistigated bias voltage (-20V -80V) and RRR values from 26 to 80.

Sputtering vs Arc: Strain and grain size



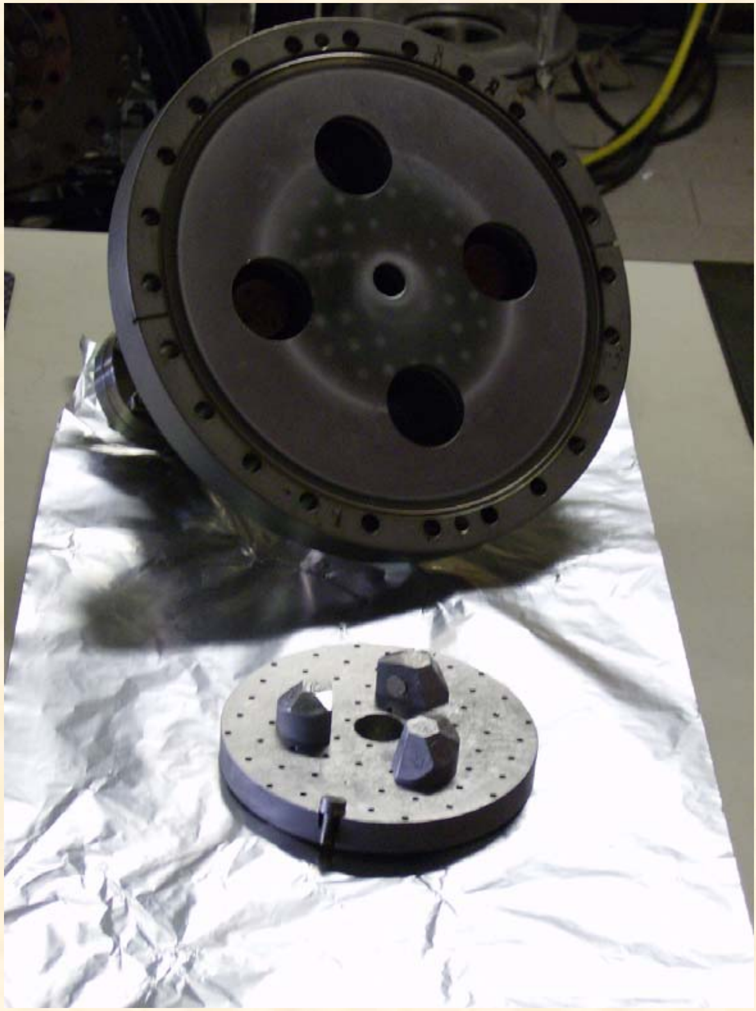


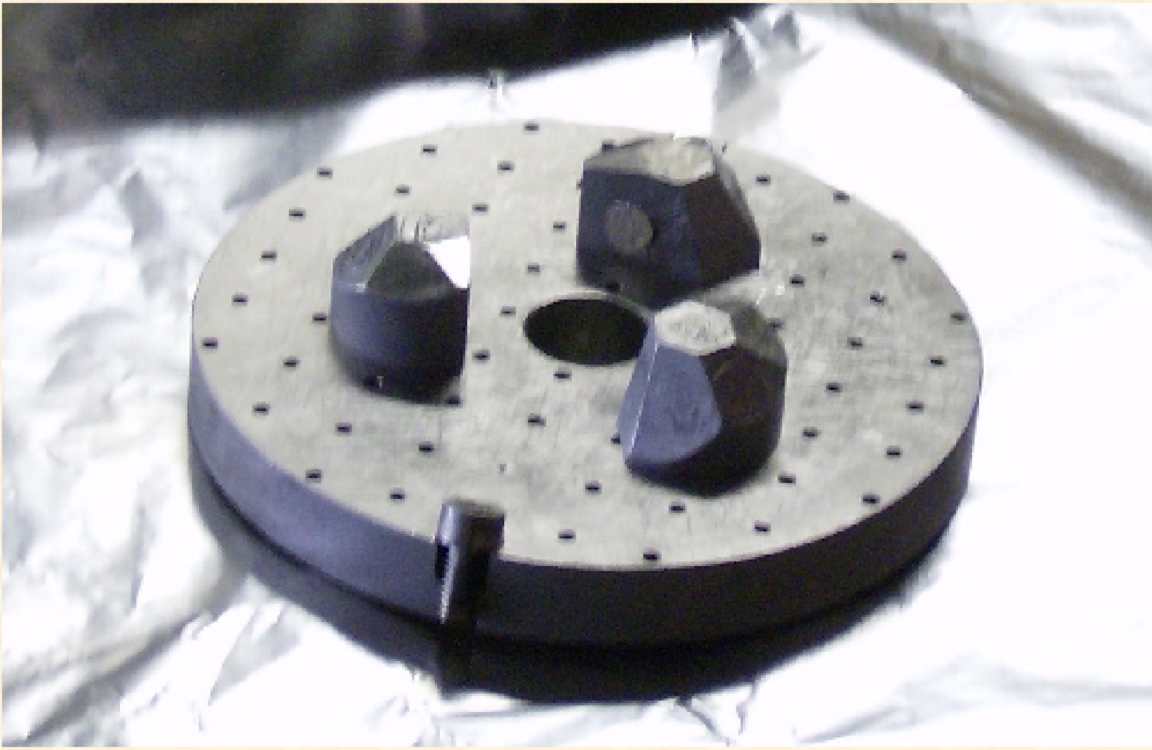
Nb Films deposited by UHV arc show lower strain and larger grain size (about 200nm with DC bias)

The multiangle sample holder

2 copper and 1 sapphire series of substrates for each depostion run.

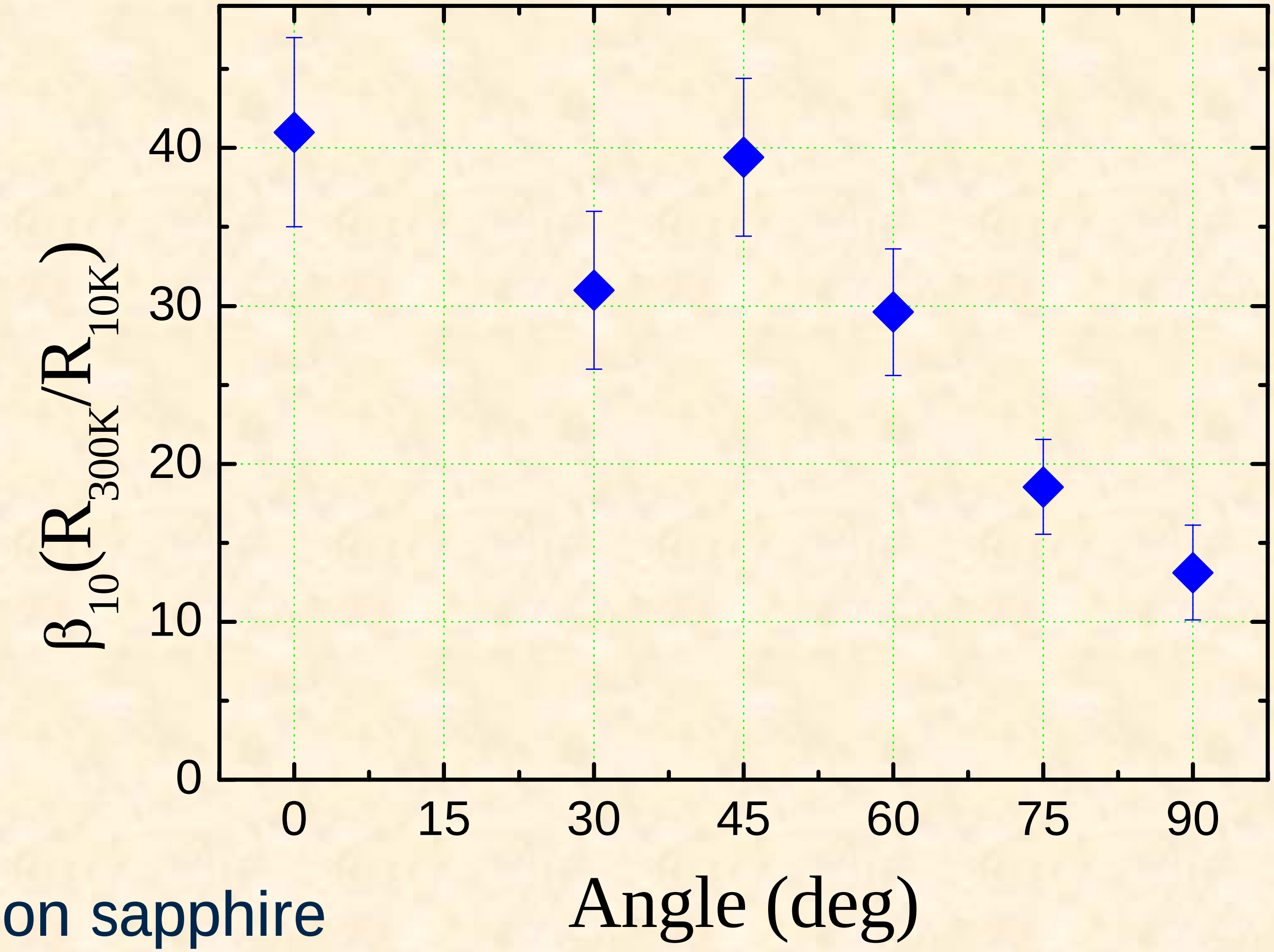
Deposition angles 0, 30, 45, 60, 75, 90





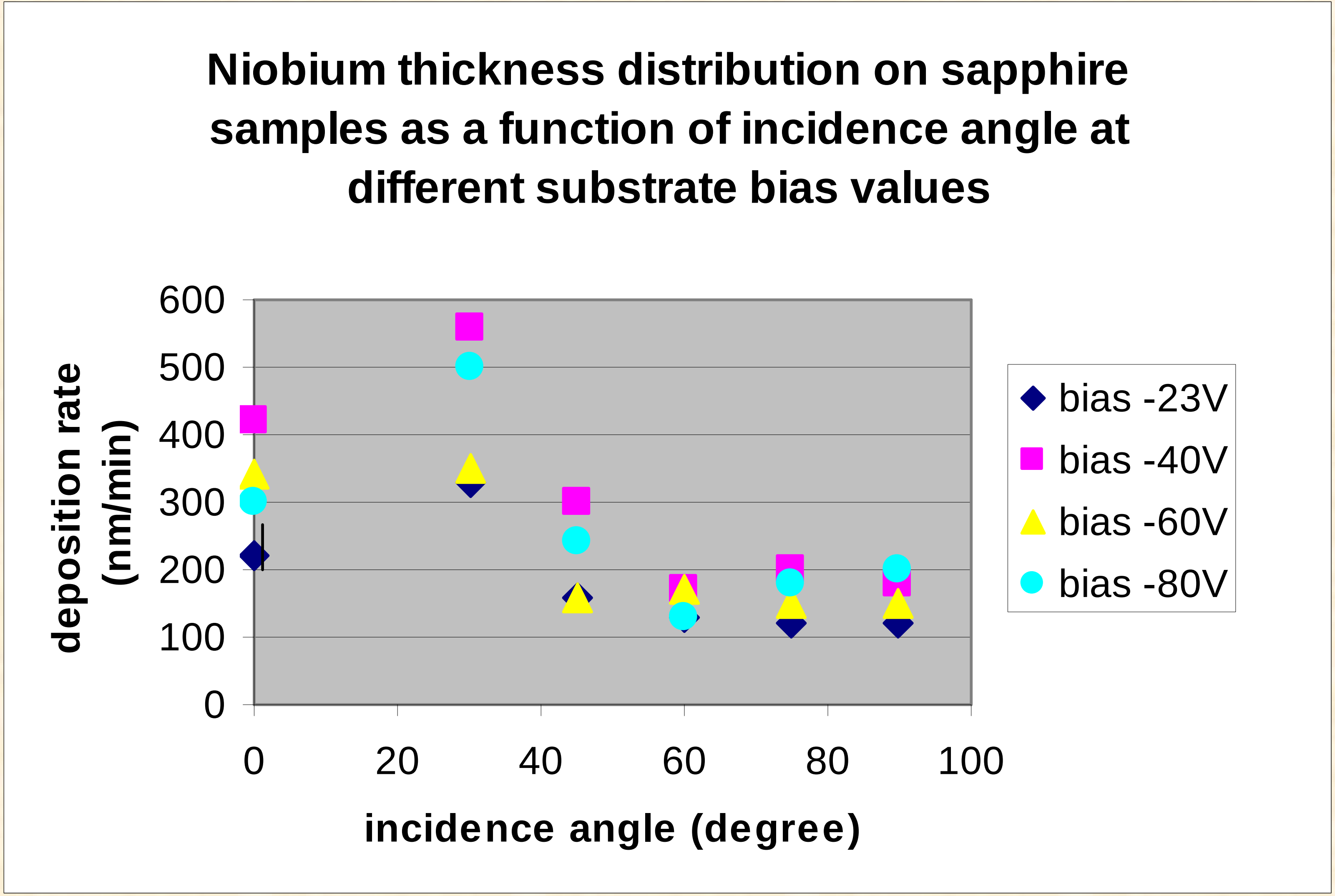
β vs Angle at low temperature (<100C)

Arc Current = 120A, Vbias = -40V

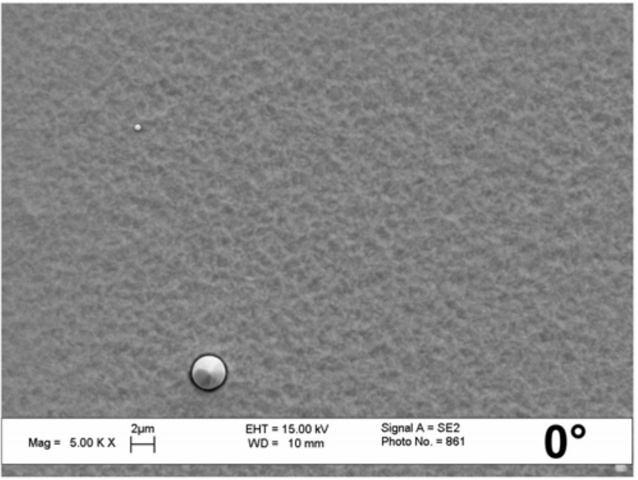


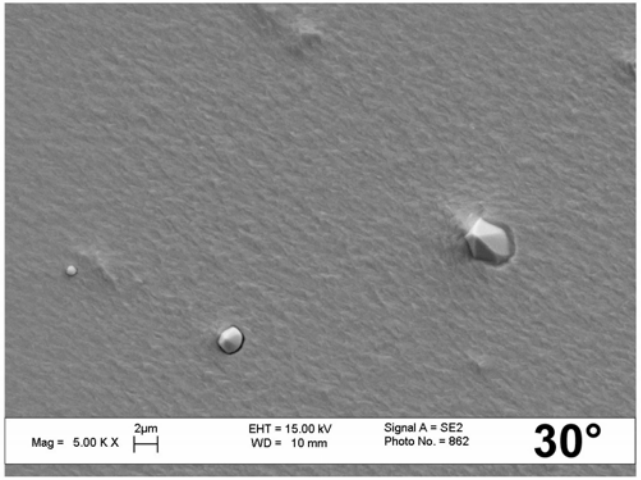
Nb on sapphire

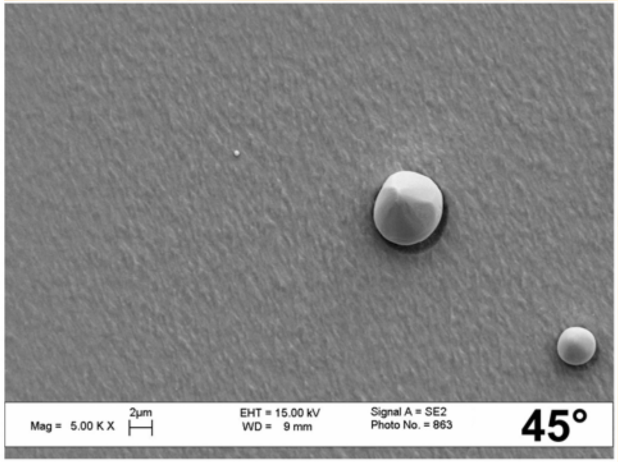
Deposition rate vs Angle

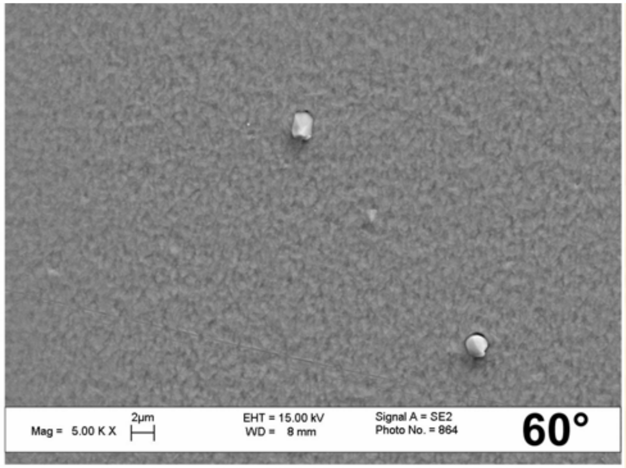


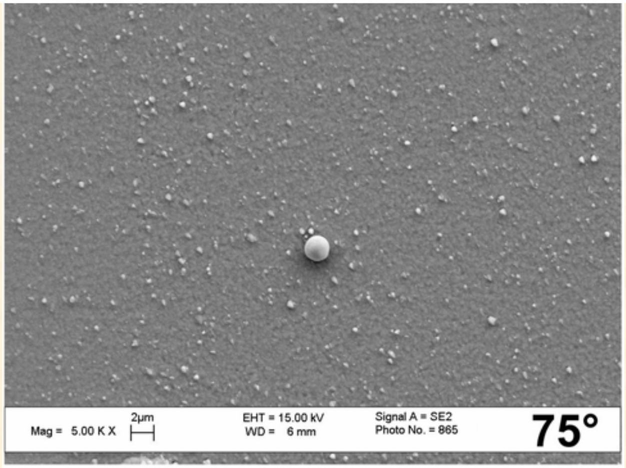
Morphology SEM (plan view)

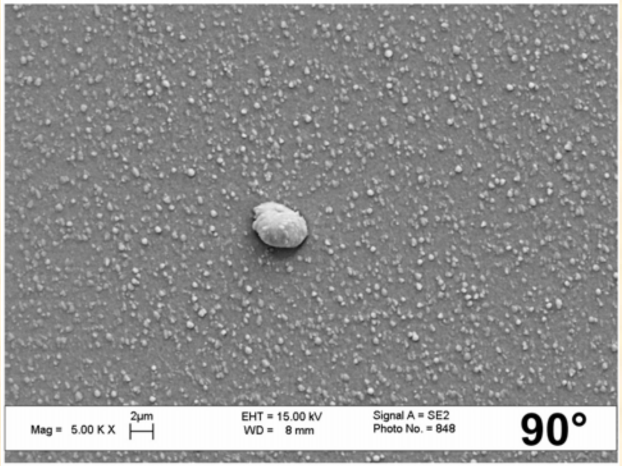




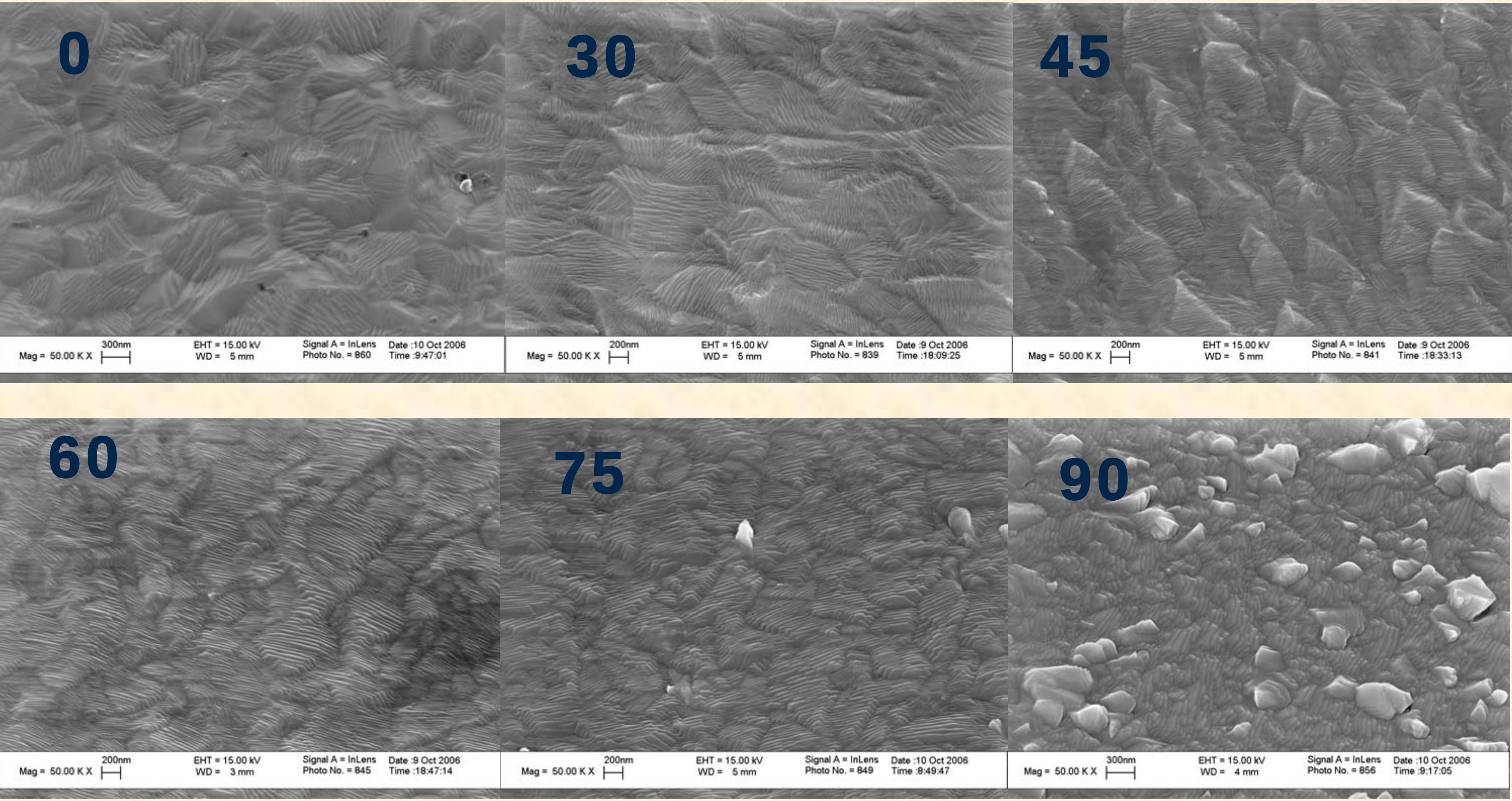




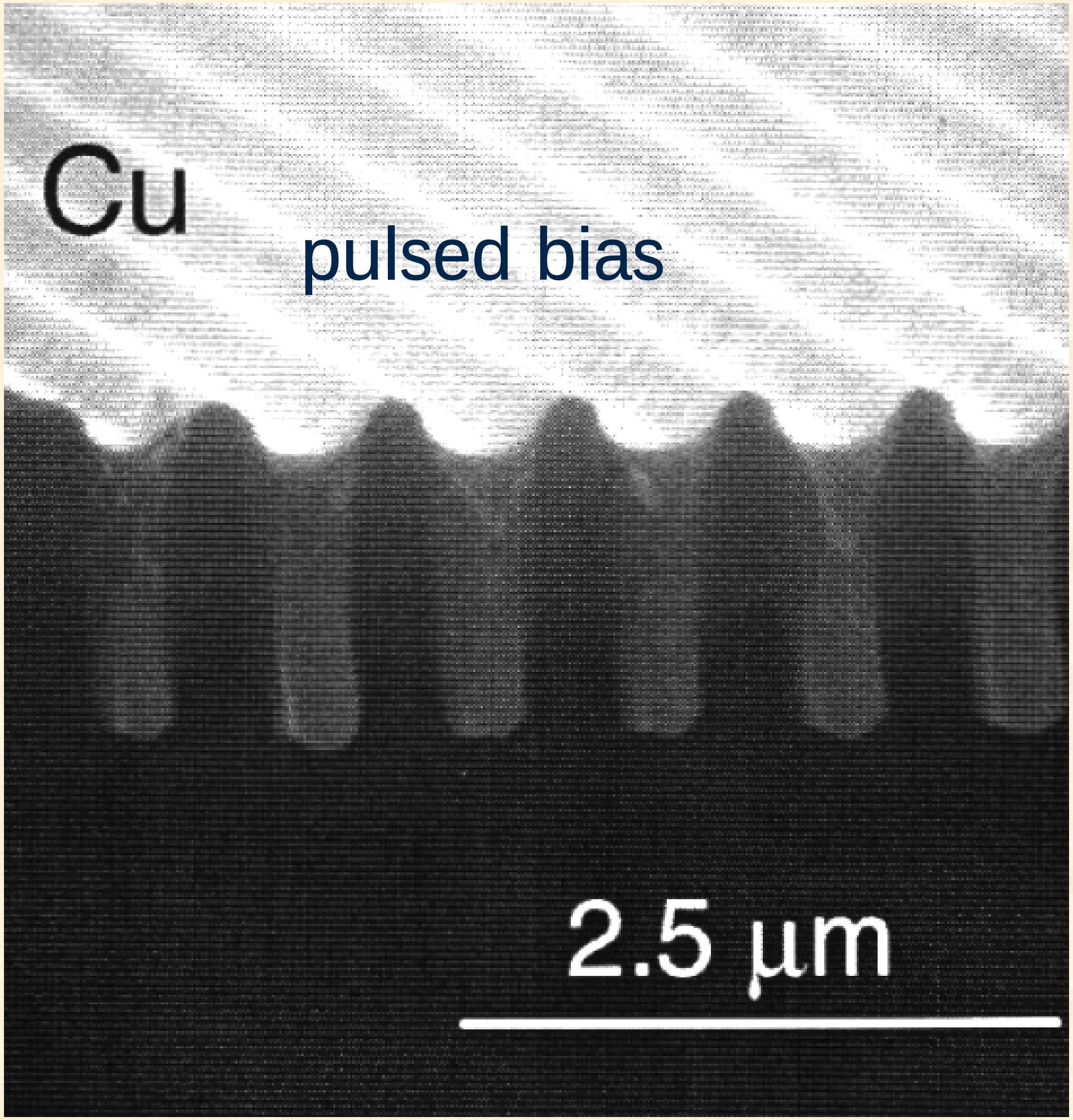




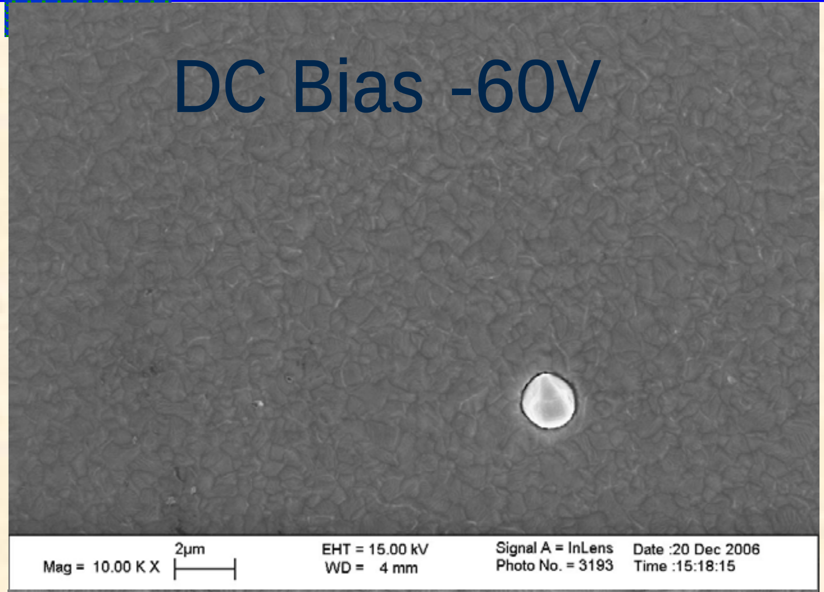
Morphology SEM (tilted view 35°)

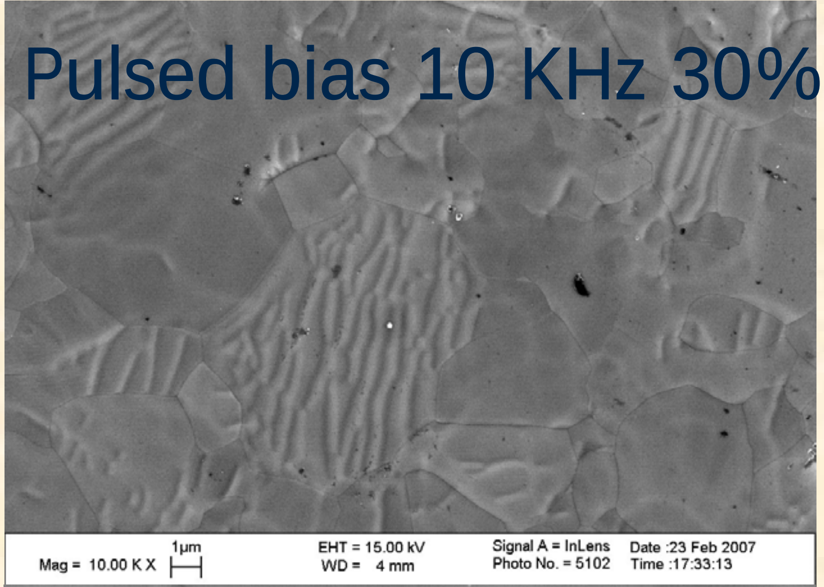


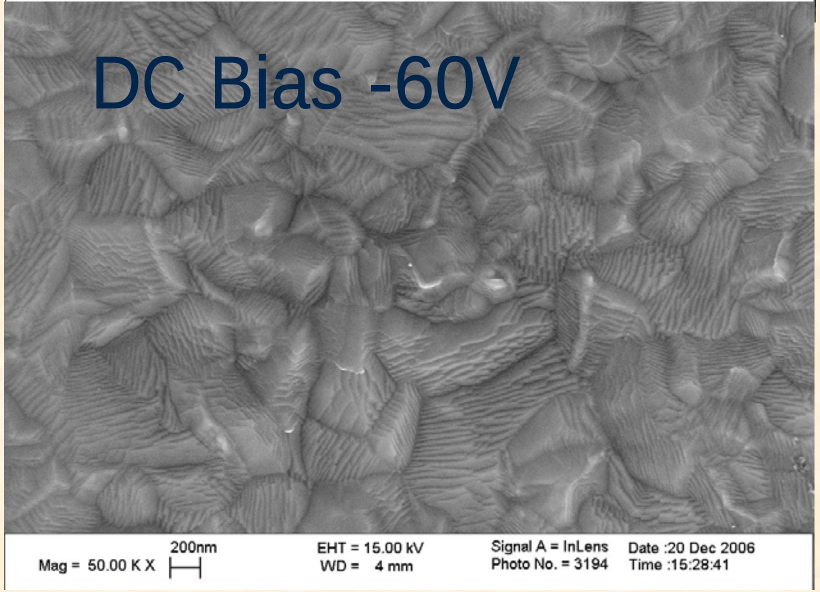
Pulsed bias can give superior results

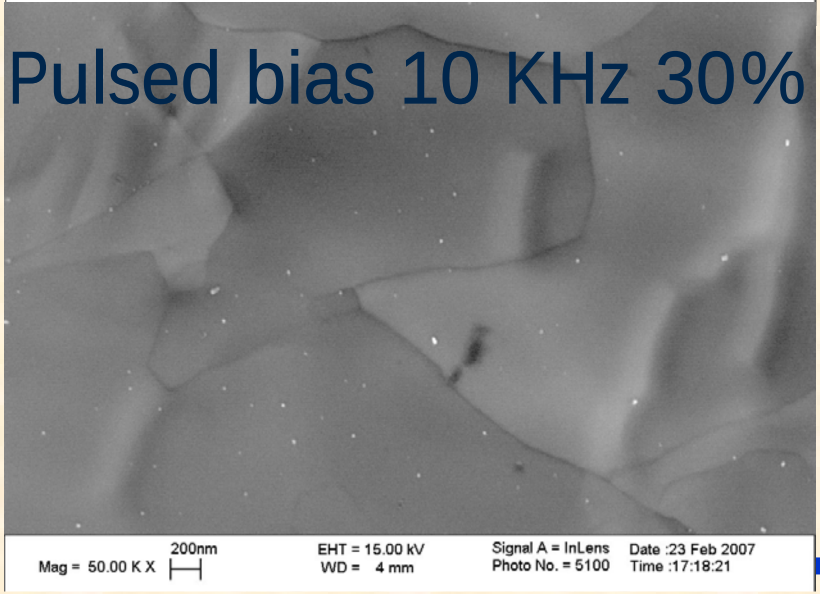


DC vs Pulsed Bias


DC Bias -60V


Pulsed bias 10 KHz 30%


DC Bias -60V


Pulsed bias 10 KHz 30%

Deposition of HTc Materials

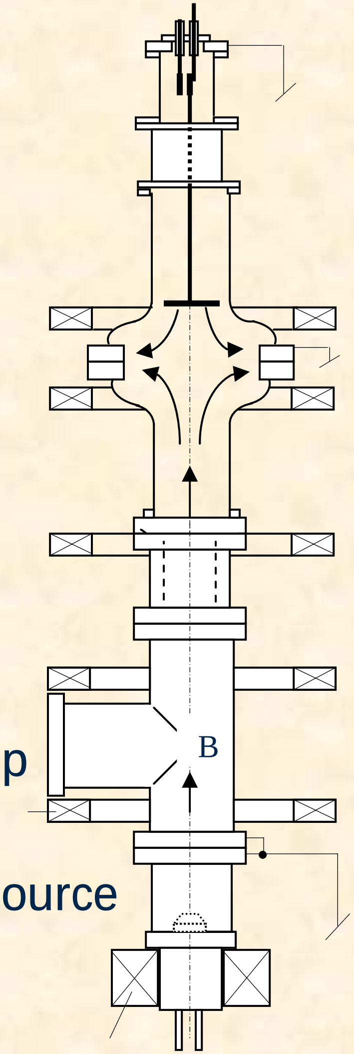
We have tried to deposit HTc materials (mainly NbN) but results were not satisfactory.

From literature the arc technique should be well suitable to deposit nitrides and deposition parameters should be less critical than in sputtering

In our experience and configuration we found that it was not easy at all to find the proper deposition parameters (we tried several N_2 pressures and voltage bias without success) and deposit nitrides could be as difficult as for the sputtering

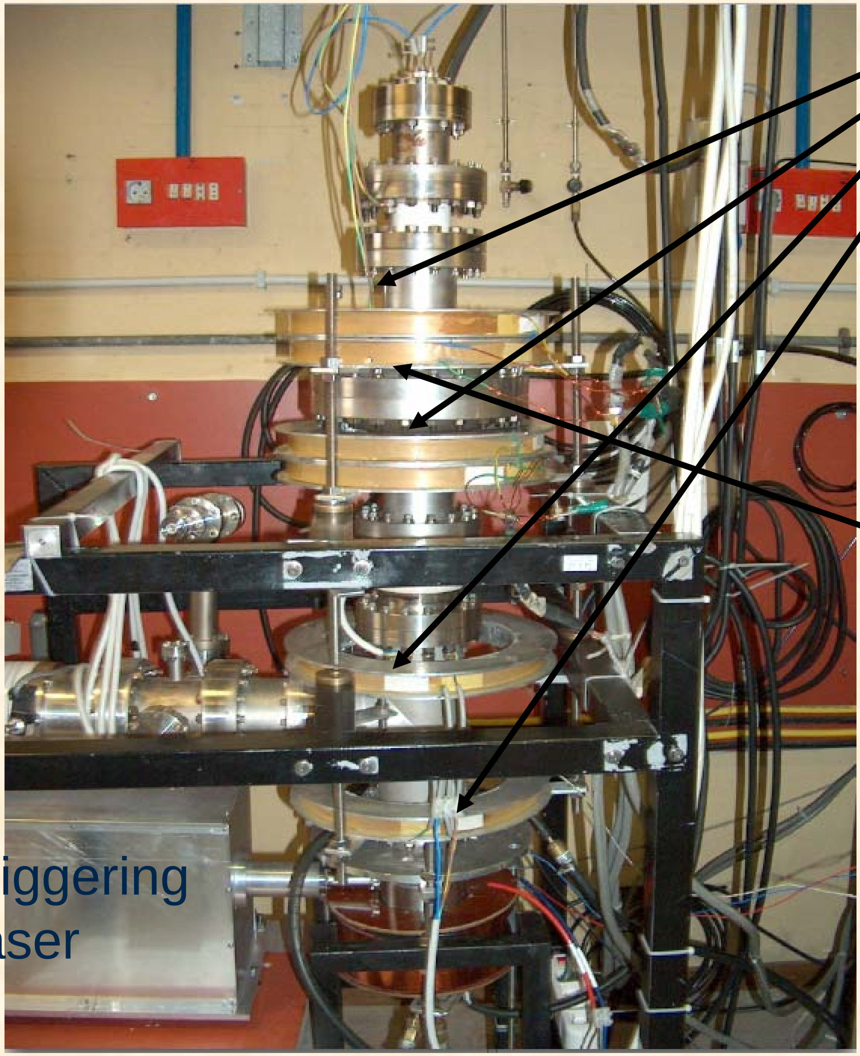
Therefore we concentrated our efforts on the single cell cavity coating

Unfiltered planar arc system for single-cell cavity coating



To pump

Arc source

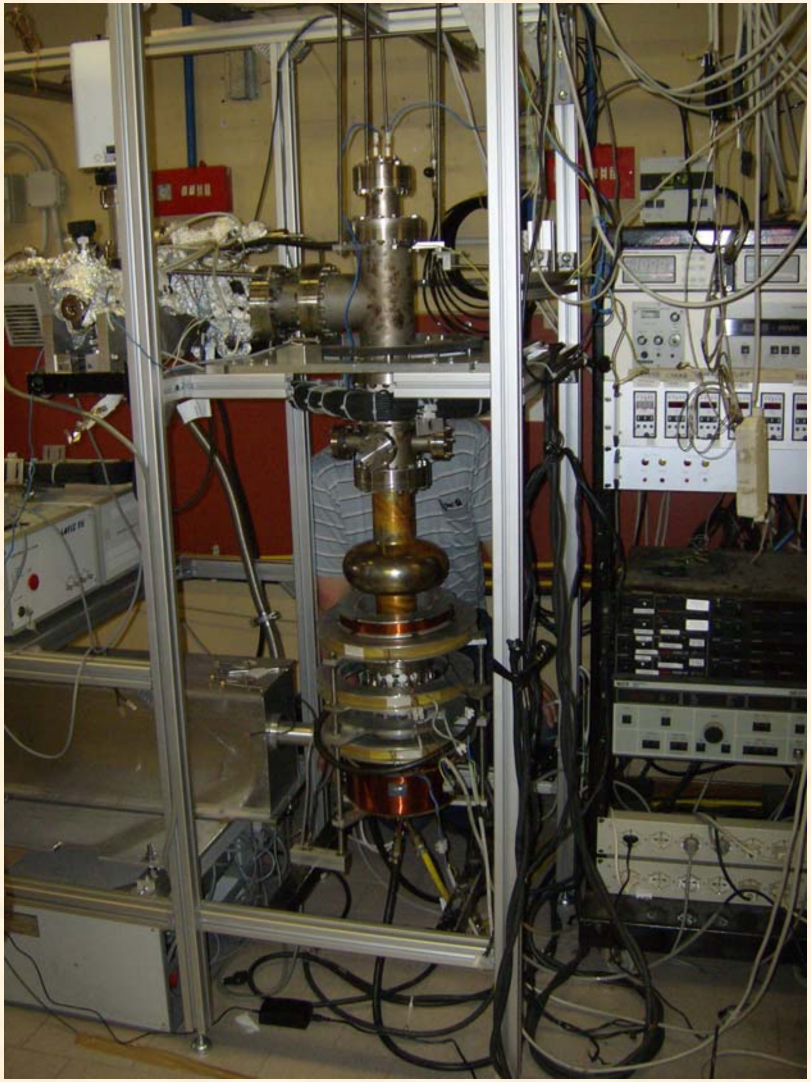


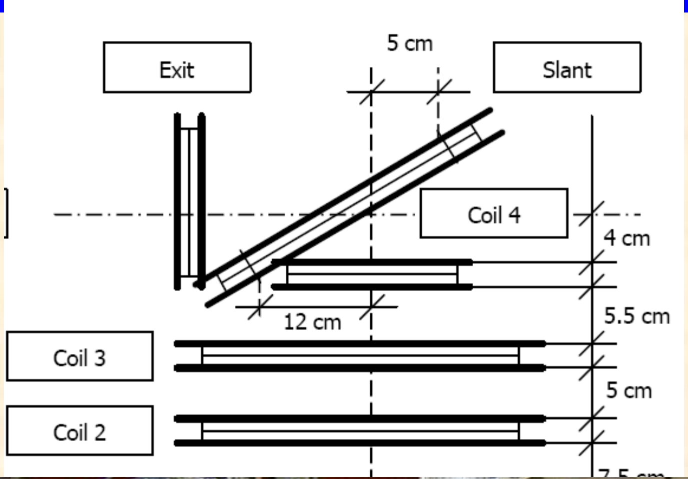
Guiding field coils

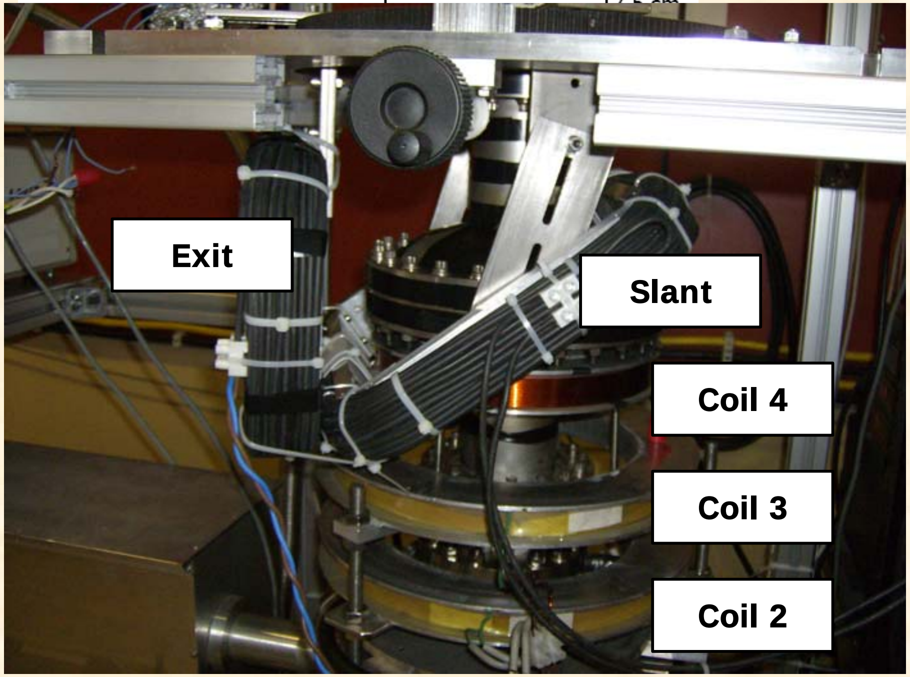
Split cavity-like SS chamber

Triggering laser

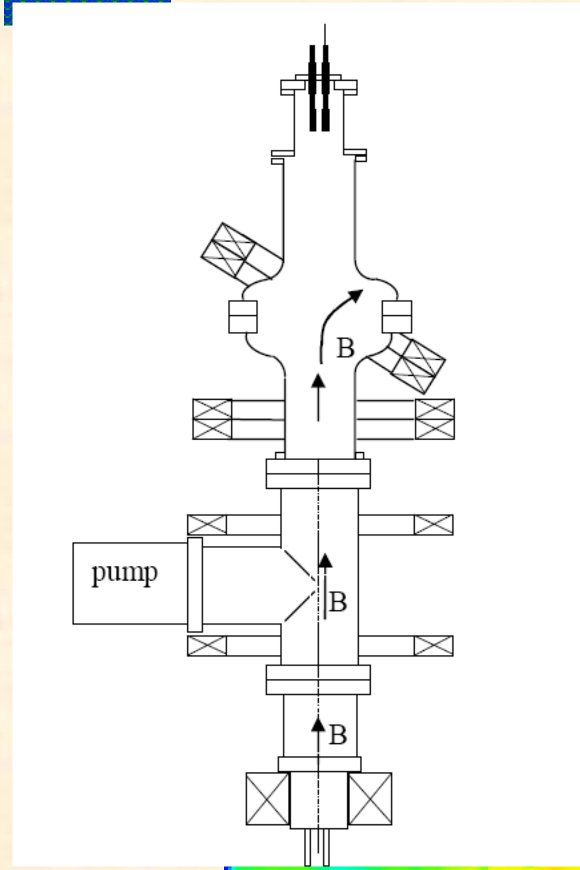
Cavity deposition system



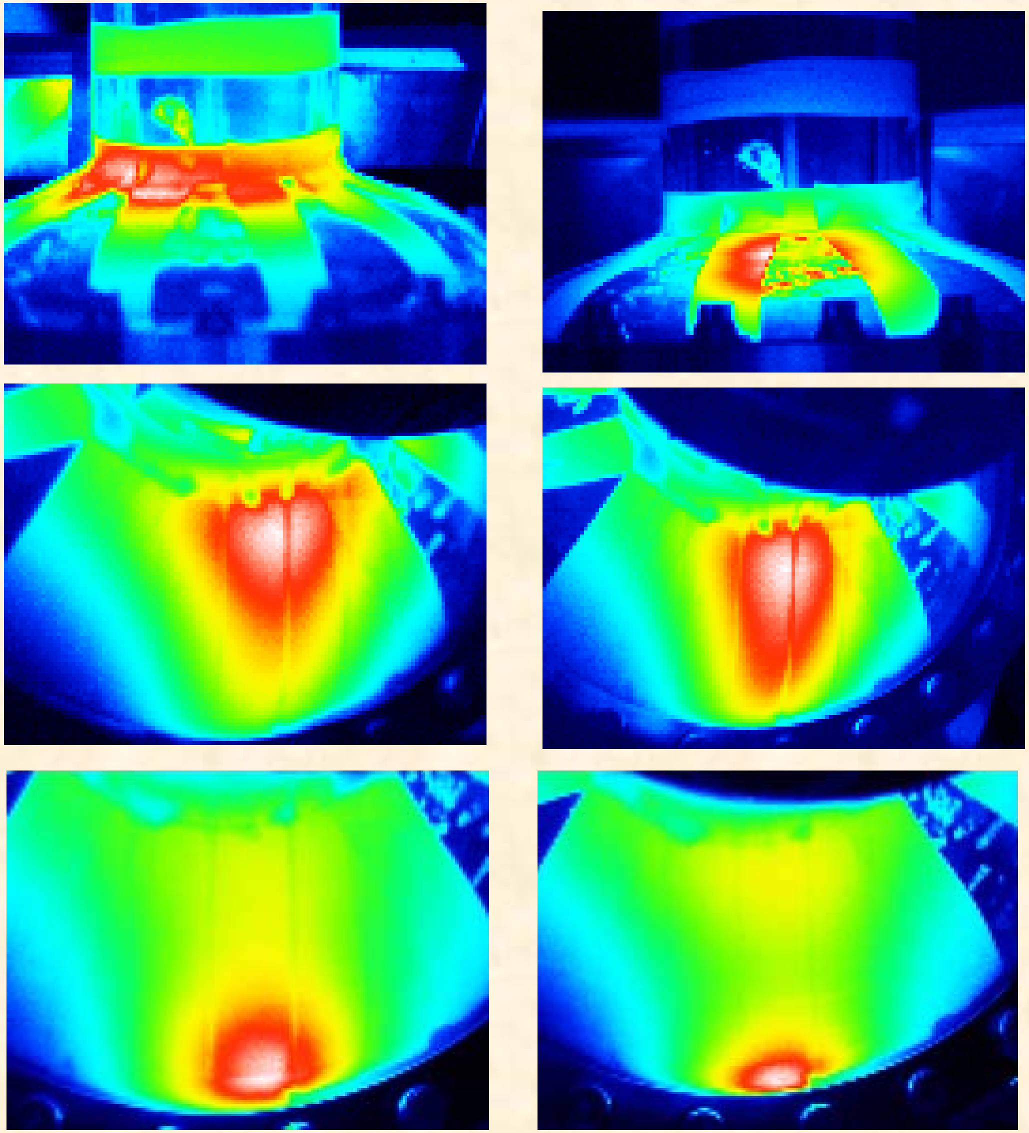


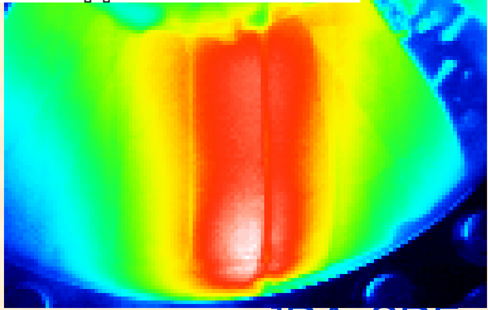


Cavity Thermal Map as function of current in the coils

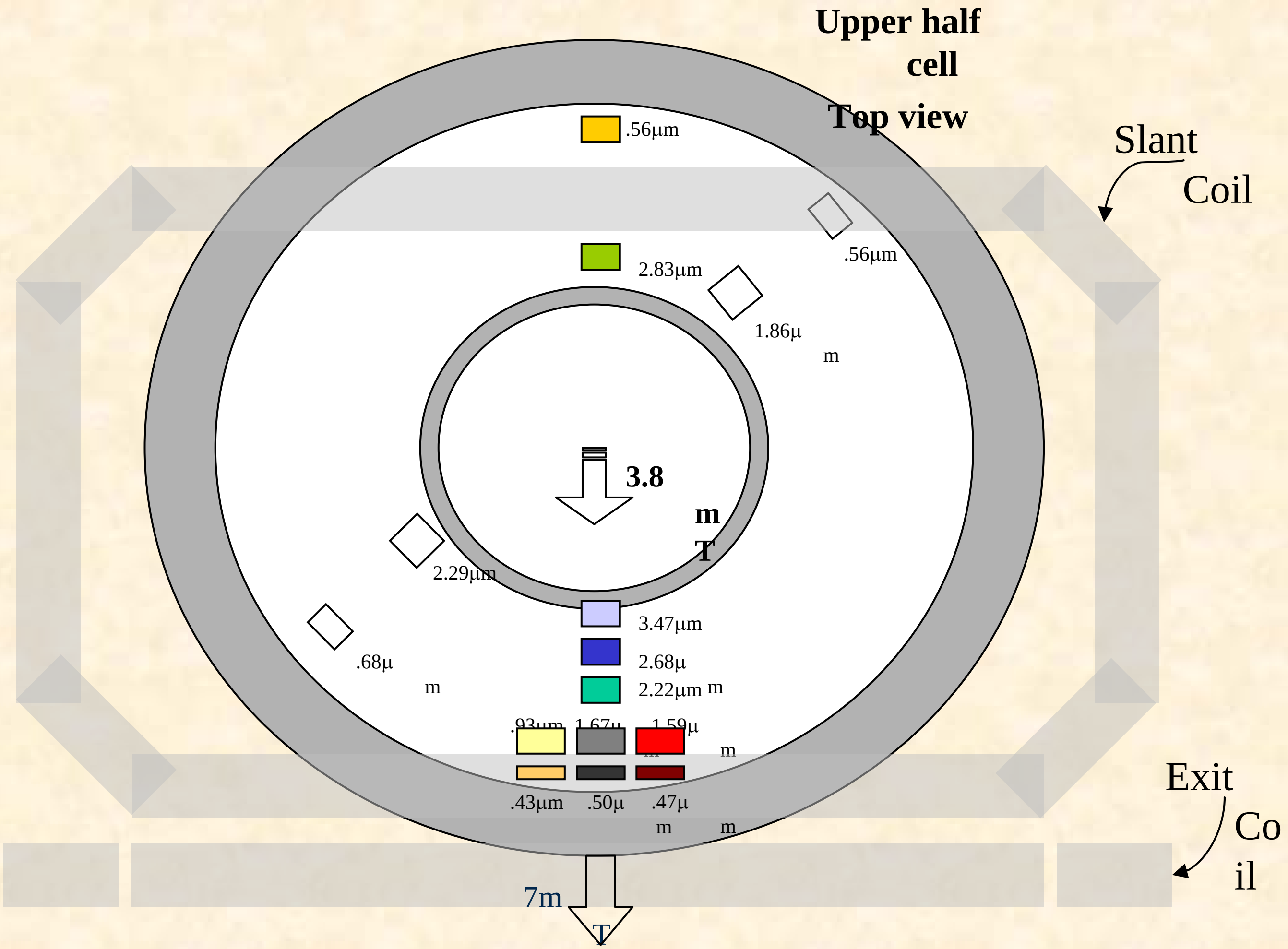


Slant
coil off

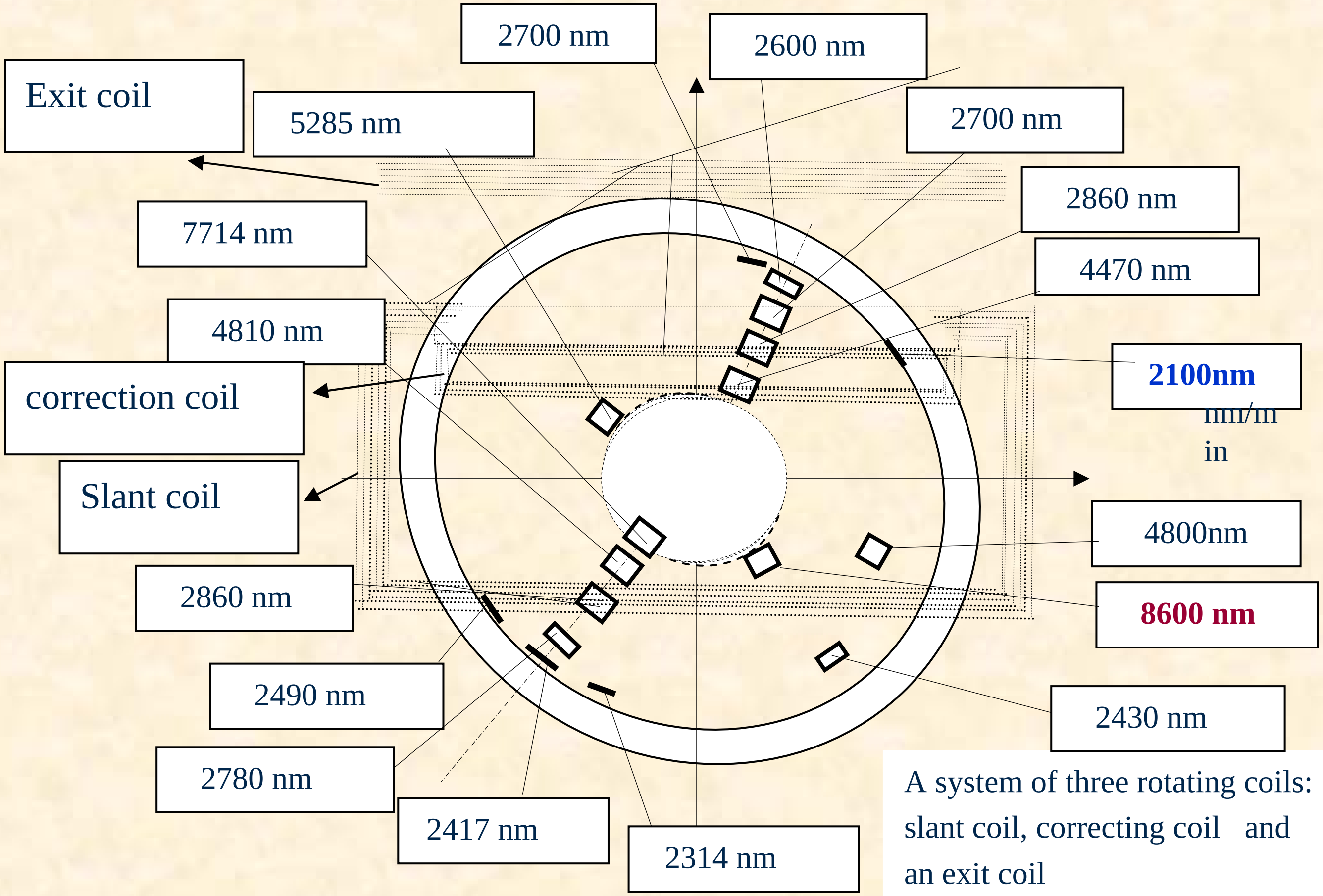




Thickness distribution in the cell: slant coil fixed



Thickness distribution in the cavity cell: two deposition with slant coil rotating



2700 nm

2600 nm

Exit coil

5285 nm

2700 nm

2860 nm

7714 nm

4470 nm

4810 nm

2100nm

correction coil

nm/m
in

Slant coil

4800nm

2860 nm

8600 nm

2490 nm

2430 nm

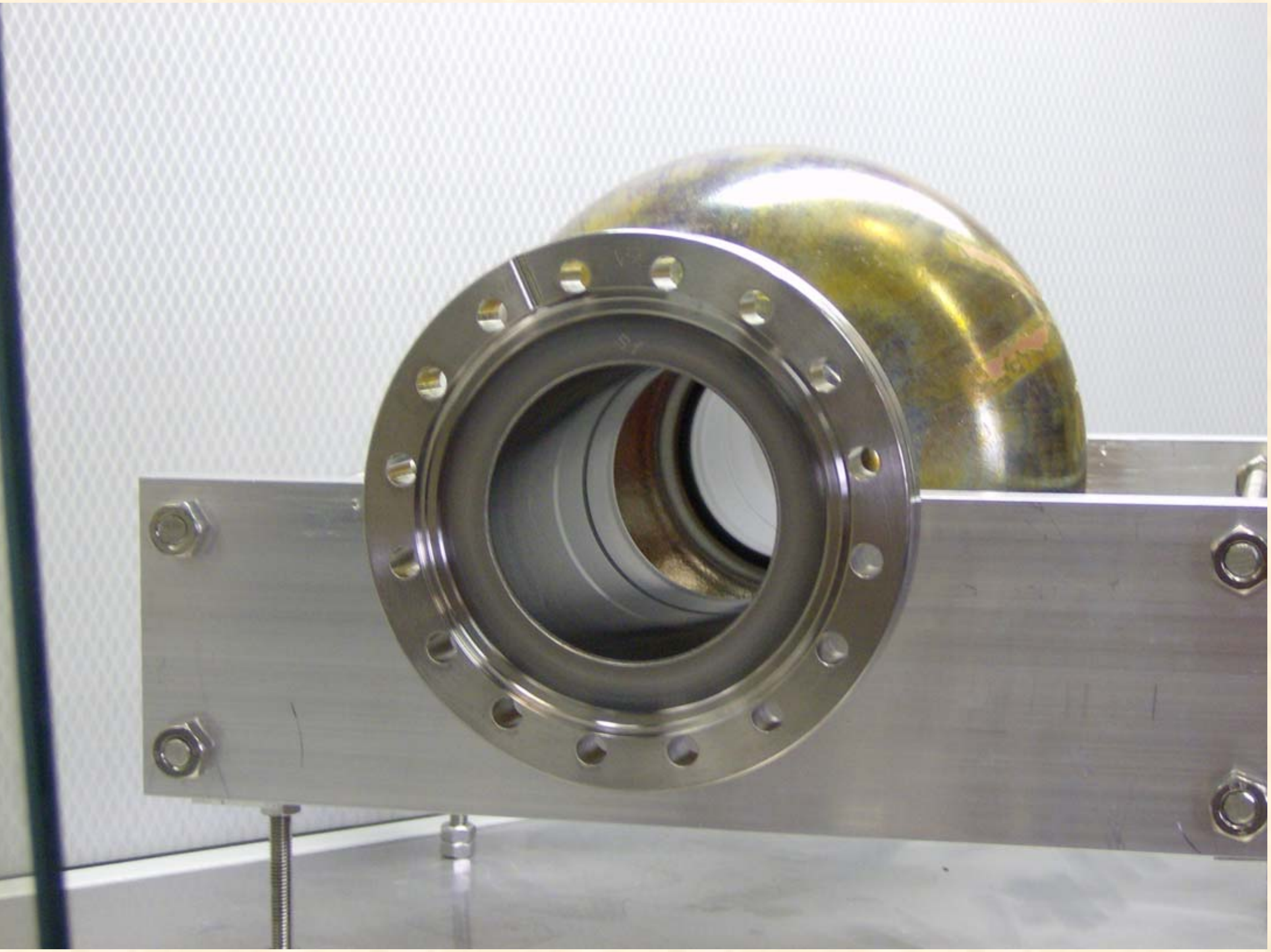
2780 nm

2417 nm

2314 nm

A system of three rotating coils:
slant coil, correcting coil and
an exit coil

First copper cavity coated by UHV planar arc (in collaboration with Saclay)



First cavity got peeling

At one iris-cut-off (second deposition)

Most likely due to pollution during the cavity turn after the first coating

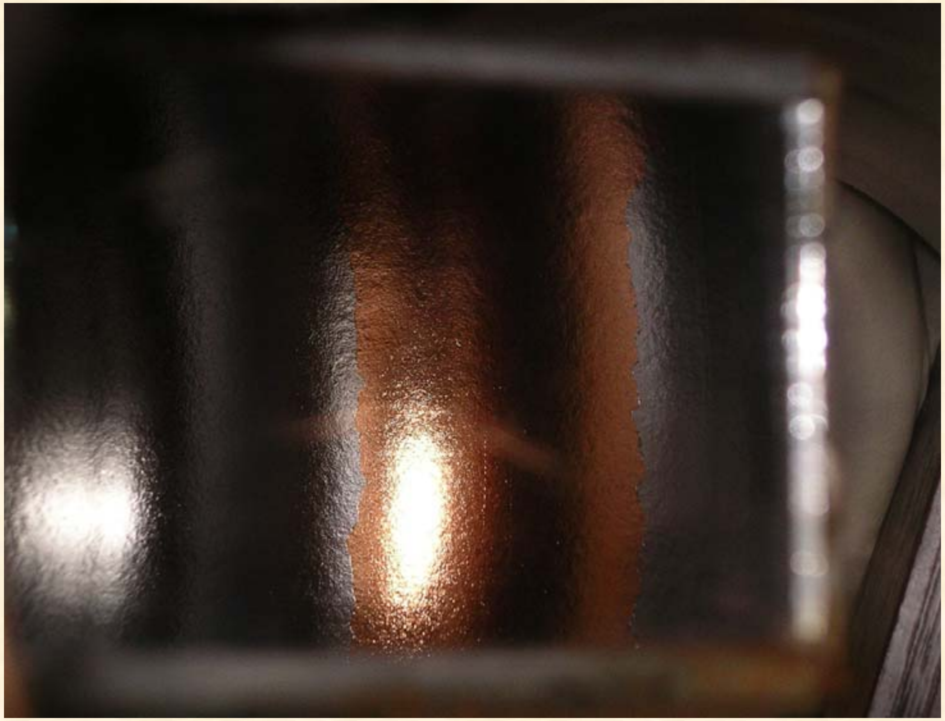
⇩

We have designed the two sources system to deposit the cavity in a single run

(Courtesy of B. Visentin and coworkers)

First cavity got peeling

Also At equator



Most likely due to electropolishing not optimized for copper cavity

(simple cilindrical cathode was used)

↓

A second cavity was prepared in Saclay by “standard” chemical polishing and it is now on the deposition system

(Courtesy of B. Visentin and coworkers)

Summary

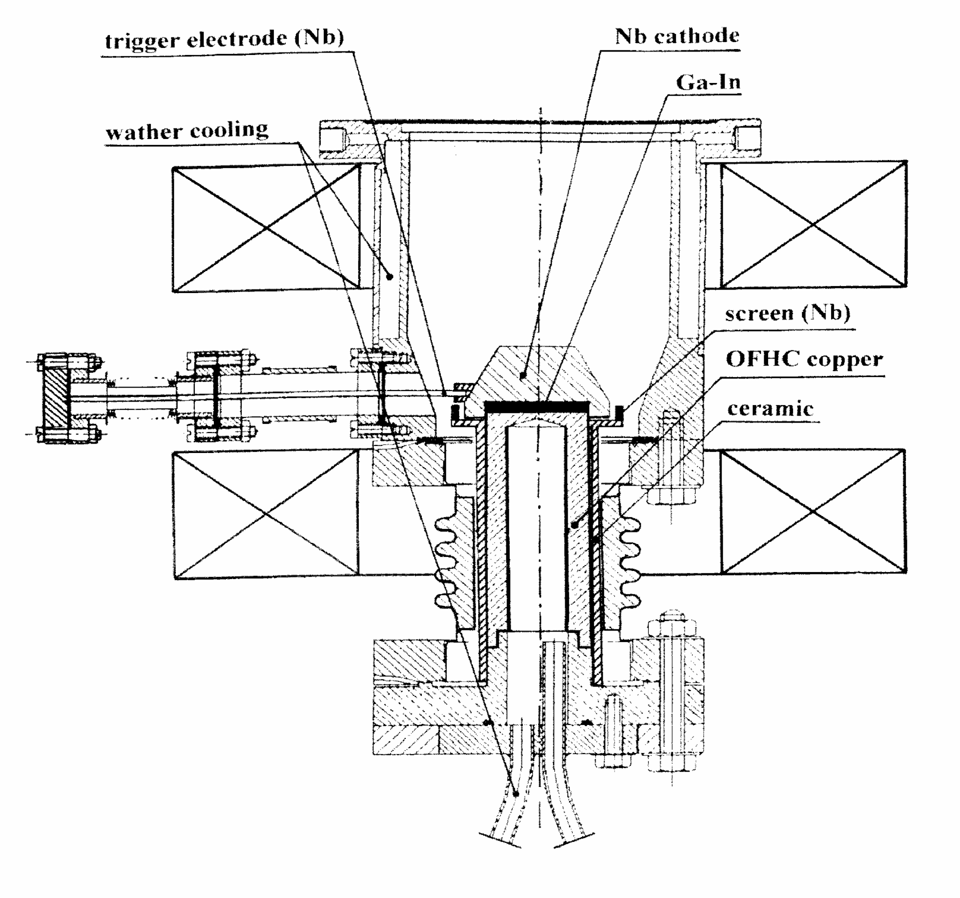
- UHV planar arc was designed, realized and characterized
- Superconducting thin Nb films with bulk material properties have been obtained (different grain sizes can be obtained using pulse dbias)
- Niobium films have a low impurity content (high RRR)
- the Niobium film structure can be controlled using a pulsed bias (still under investigation)
- The plasma transport in the cavity cell with the scanning coils has been studied and optimised
- First single cell cavities has been coated (some adhesion problems)
- Commissioning of the deposition system with two cathode sources will start soon (it will solve the adhesion problems)
- We can start the cavity production to study the RF film properties and the dependencies of RF properties from deposition parameter.
- We need more cavities to coat and RF measurements of produced cavities

Request for delivery of new cavities

- Copper cavities have to be electropolished (200μm the first time to remove the damaged cortical layer, 20μm after the first one)
- Be careful to cathode Shape! Copper it is not niobium and bath impedance cannot be neglected! (see paper from CERN at SRF workshop 2003)

  Chemical bath can also be a solution (not the best solution but a safe one)
- Rinse the cavity by HPWR and dry it
- After drying cavity have to be sealed with blank flanges and filled with inert gas (Ar or nitrogen) at a pressure slightly higher than atmospheric pressure.
- If possible the sealed cavity should be packed in a sealed plastic bag within the clean room (to keep also the external part clean and "dust free": not mandatory).
- To avoid damage during shipping the cavity must be fixed in the box from both cut-off tubes and/or end flanges
- Cavity will be unpacked in a laminar flow and mounted on the coating system
- After 2 weeks cavity will be coated and sent back for RF measurement
- Low pressure rinsing instead of HPR will be advisable until the adhesion problems (mainly related to the chemistry) are mastered.

UHV planar arc source



5 cm

We started in 2001 using a standard ceramic trigger to ingnite the discharge

Now we use a Nd Yag Laser Q-Swithed

DC Vacuum Arc discharge

Nb Cathode during arc

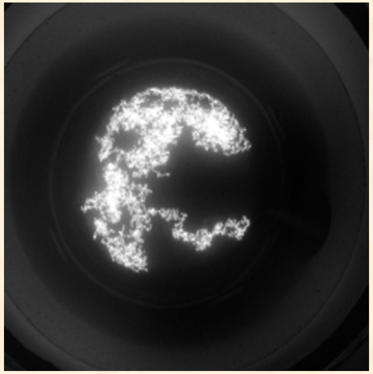


Photo t =0.1s

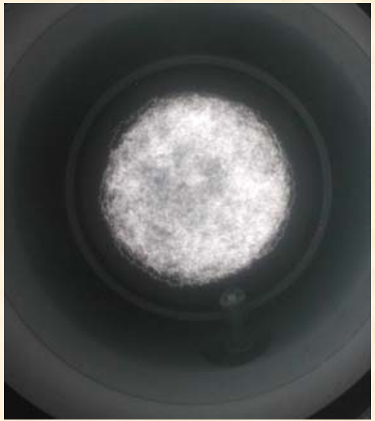


Photo t =1s

- Discharge (hot spot) sustained in the vapor of the cathode material (no working gas)
- Arc spot moves on the Nb cathode at 10m/s
- Niobium is fully ionized
- multiply charged ions +2,+3… (mean value for Nb +3)
- Average energy of Niobium ions about 130eV (tunable with bias)
- Minimum Arc Current is 60A
- Cathode voltage is ≈ 35V
- Base vacuum ≈ 10^{-10} mbar (10^{-8} Pa)
- Main gas during arc is Hydrogen (≈10^{-7} mbar)
- Voltage Bias on samples 20-100V

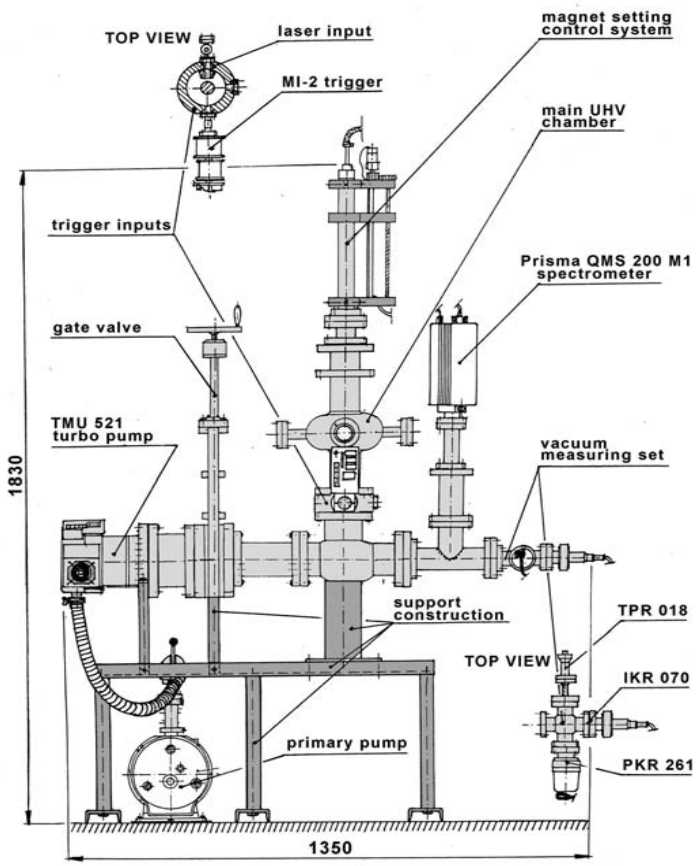
Possible approaches to cavity coating

1. Cylindrical cathode with arc generated in the cavity cell

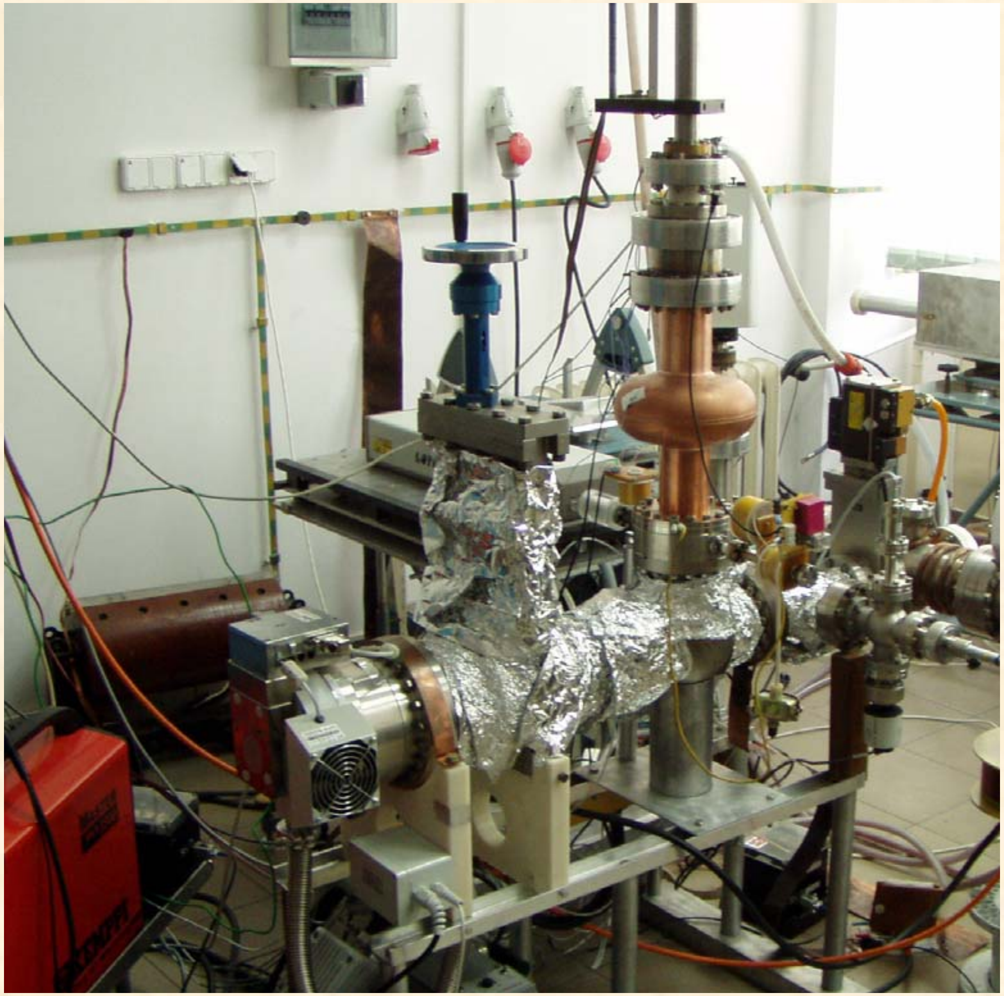
   (simplest but probably it needs a quite complicate macroparticle filter)

2. Using a magnetic field to introduce the plasma generated by a planar arc source in to the cavity cell
   - Without a magnetic filter
   - With a magnetic filter

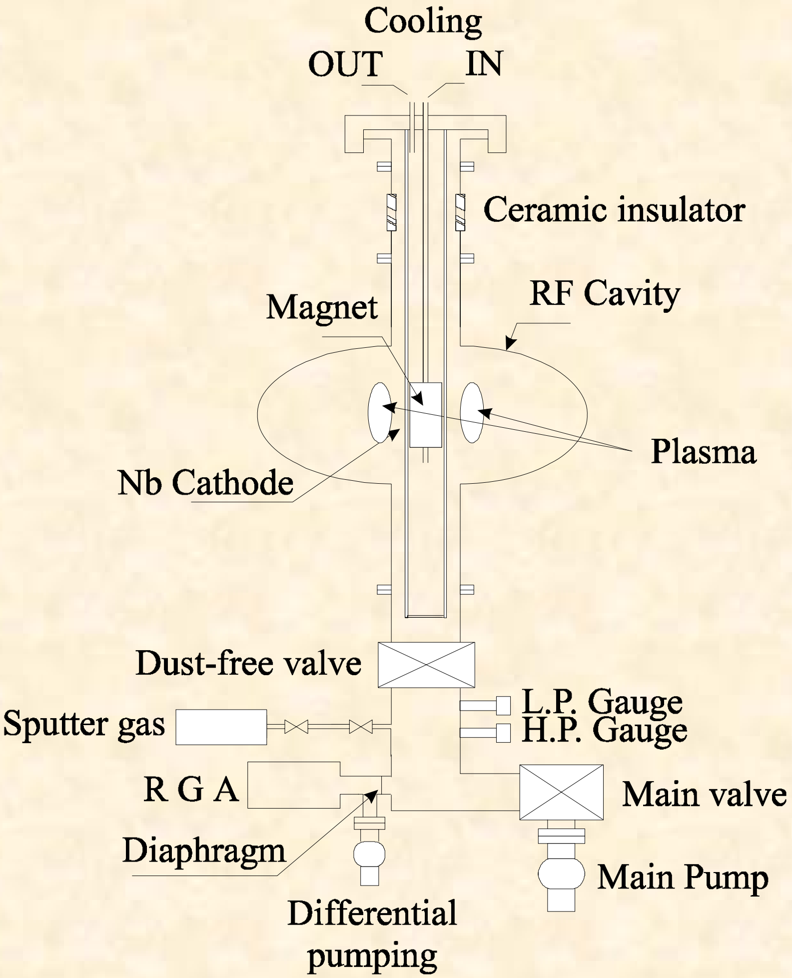
Cylindrical arc set-up in Swierk(PL)



General view of modified UHV set-up for deposition of superconducting Nb films



Cavity coating using Sputtering



Cooling

OUT

IN

Ceramic insulator

Magnet

RF Cavity

Plasma

Nb Cathode

Dust-free valve

Sputter gas

L.P. Gauge

H.P. Gauge

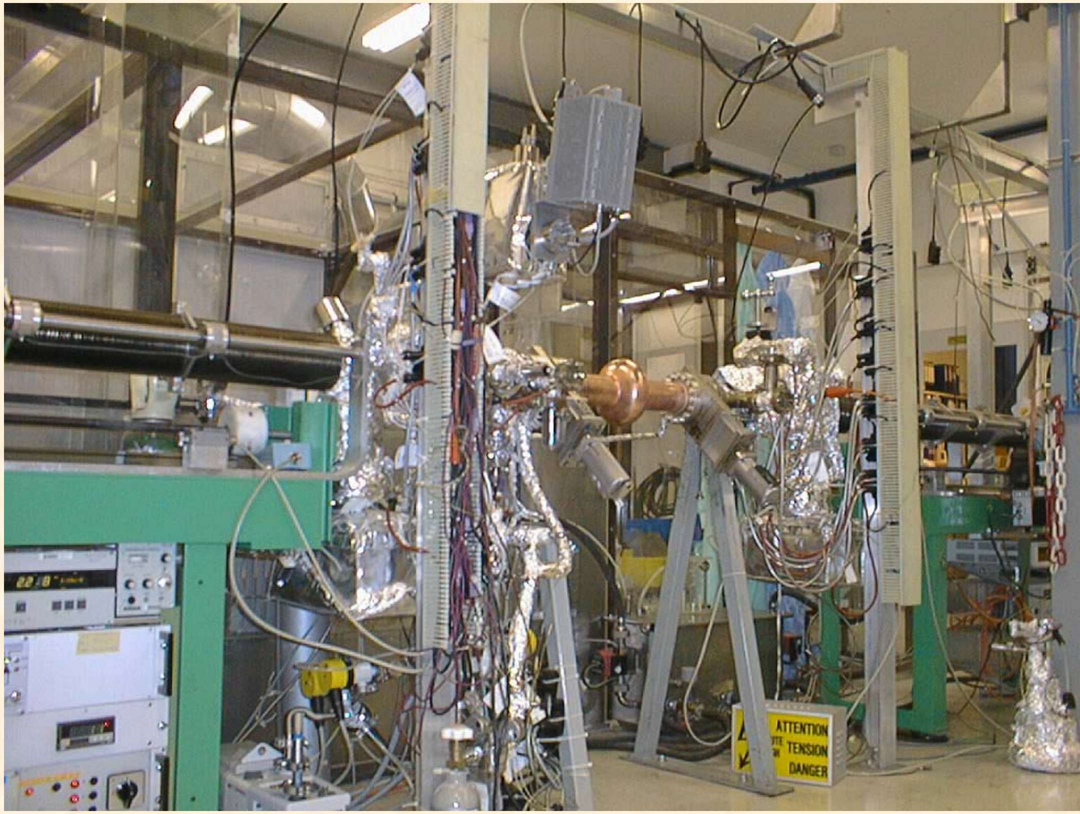
R G A

Main valve

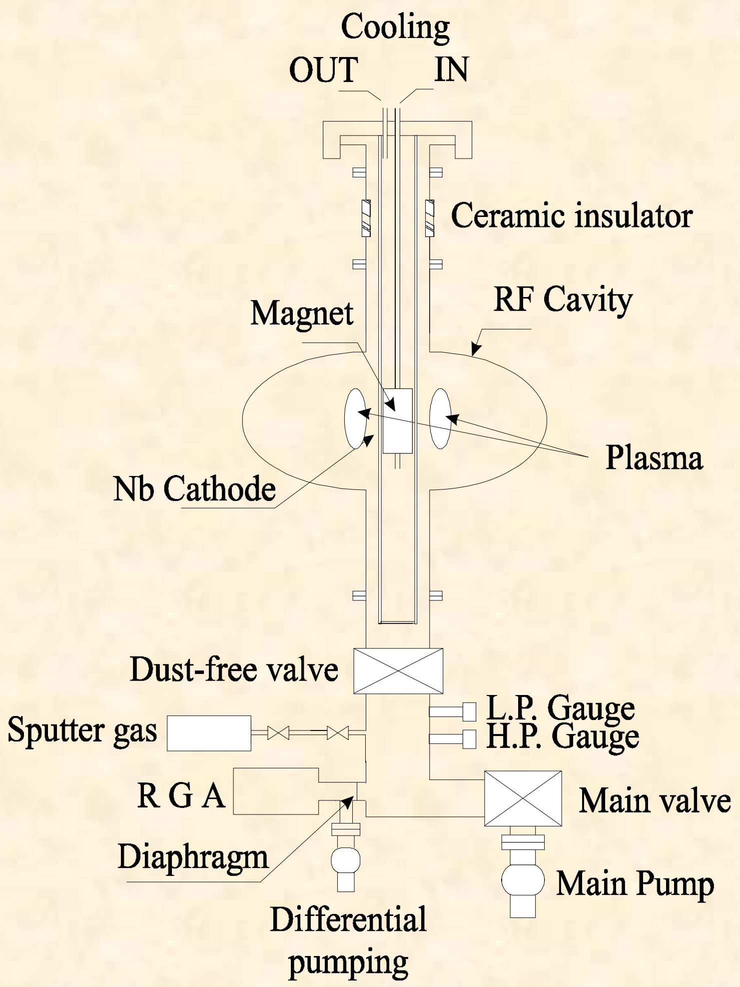
Diaphragm

Main Pump

Differential pumping



Magnetron sputtering have been used up to now



Sputtering parameters:

- Discharge current stabilized at 3A.
- Sputter gas pressure of 1.5×10^{-3} mbar, corresponding to ~ 360 V.
- Coating temperature is 150 °C.
- Thickness: 1.5 µm

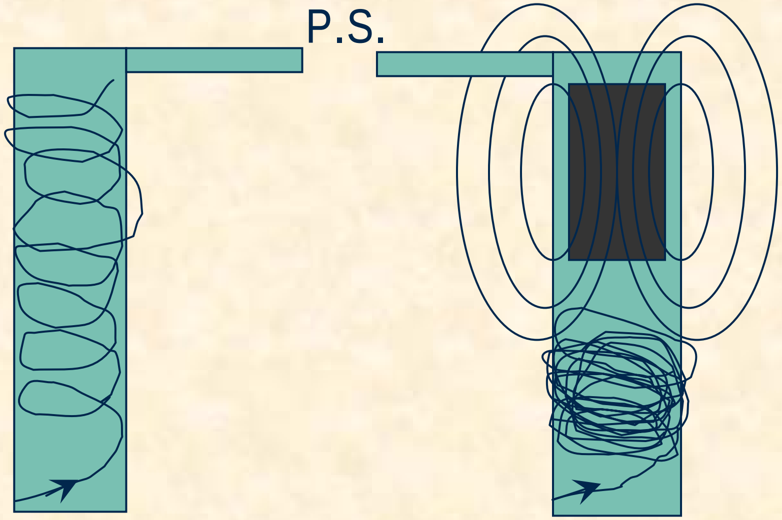
CERN "standard film" characteristics:

- RRR: 11.5 ± 0.1
- Argon content: 435 ± 70 ppm
- Grain size: 110 ± 20 nm
- Tc: 9.51 ± 0.01 K
- Strain: $\Delta a_{\perp}/a_{\perp} = 0.636 \pm 0.096$ %
- **Result: maximum about 15MV/m**

Cylindrical arc: working principle

In linear arc the arc current flowing along the cathode generates a magnetic field that interacts with the arc current. This interaction push the arc spot to move along a spiral around the cilindrical cathode in the up direction.

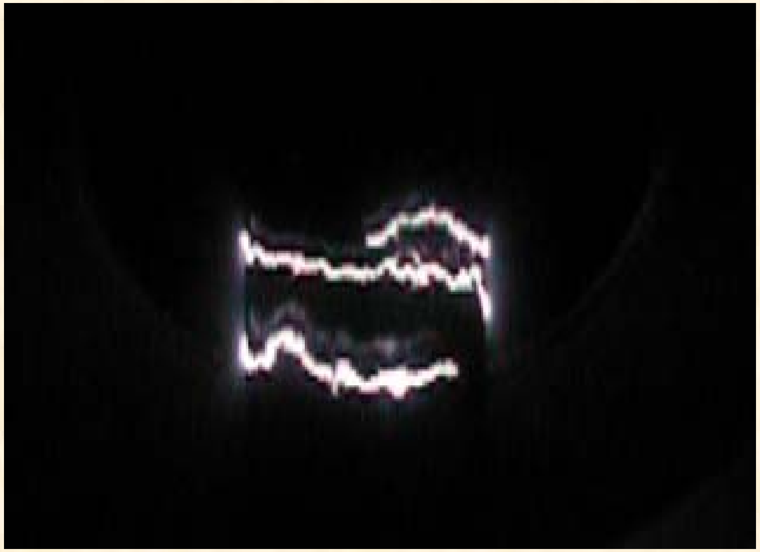
2 mode of operation are possible:

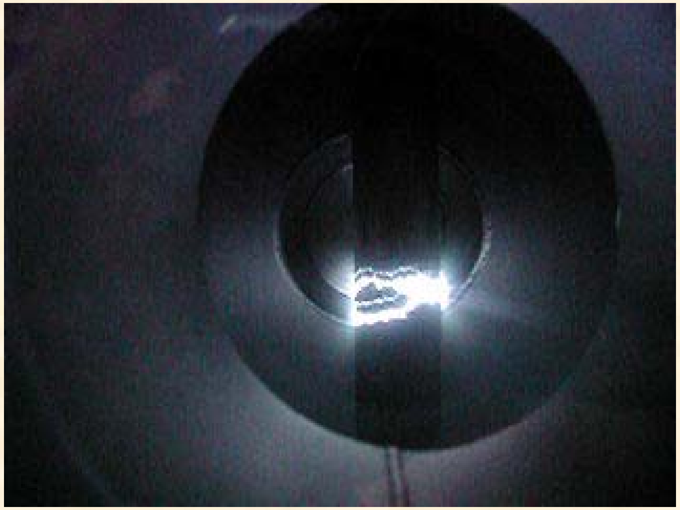


Arc is generated from the bottom part of the cathode and stops on the upper floating potential electrode**

A strong permanent magnet “reflects” the arc spot, confining the movement of the arc in the region below the magnet.

Arc spot motion on linear arc





In this configuration for a Iarc =100A the speed of the arc hot spot is about 3 m/s.

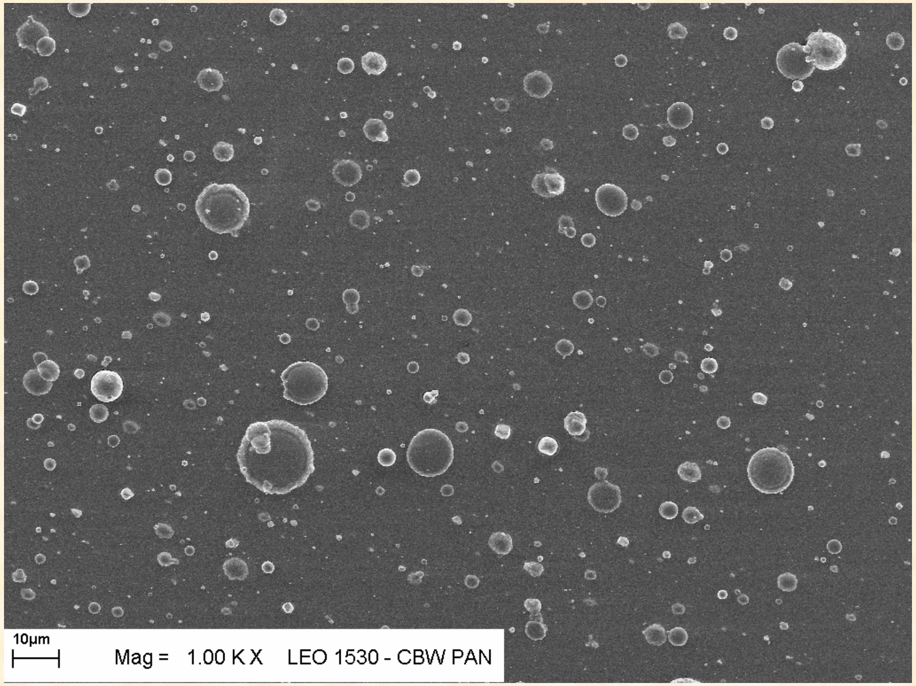
the arc is more stable even at I_{arc} about 50A Maybe due to no edges on the cathode surface

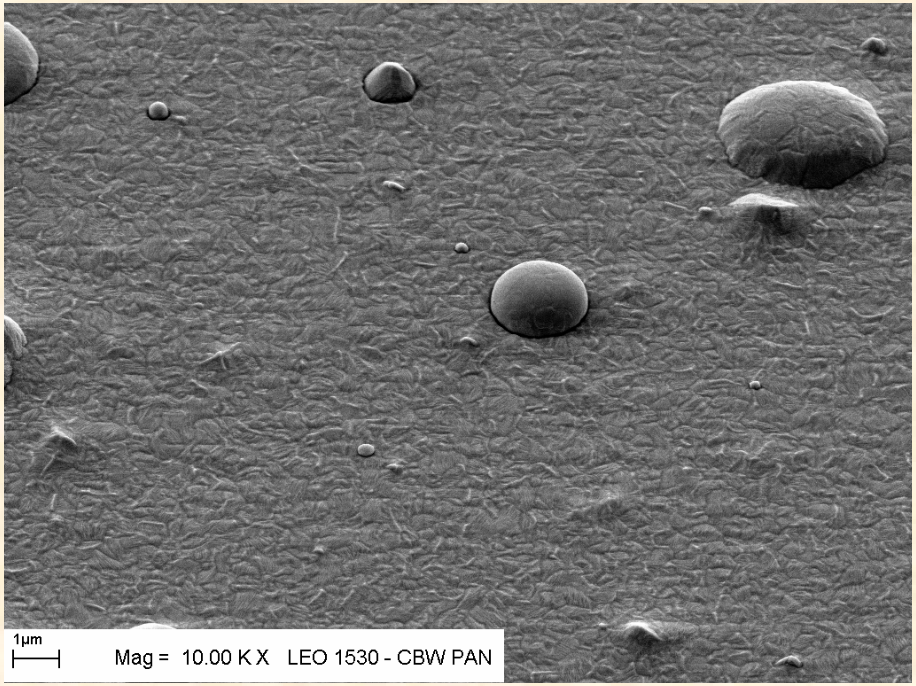
Preliminary test for cavity coating

The first single-cavity taken of the real accelerator unit, after its preparation, has been coated without micro-droplet filtering.

The coated single-cell has been cut along its symmetry axis in order to perform an analysis of the inner surfaces.

Analysis of the coated substrate

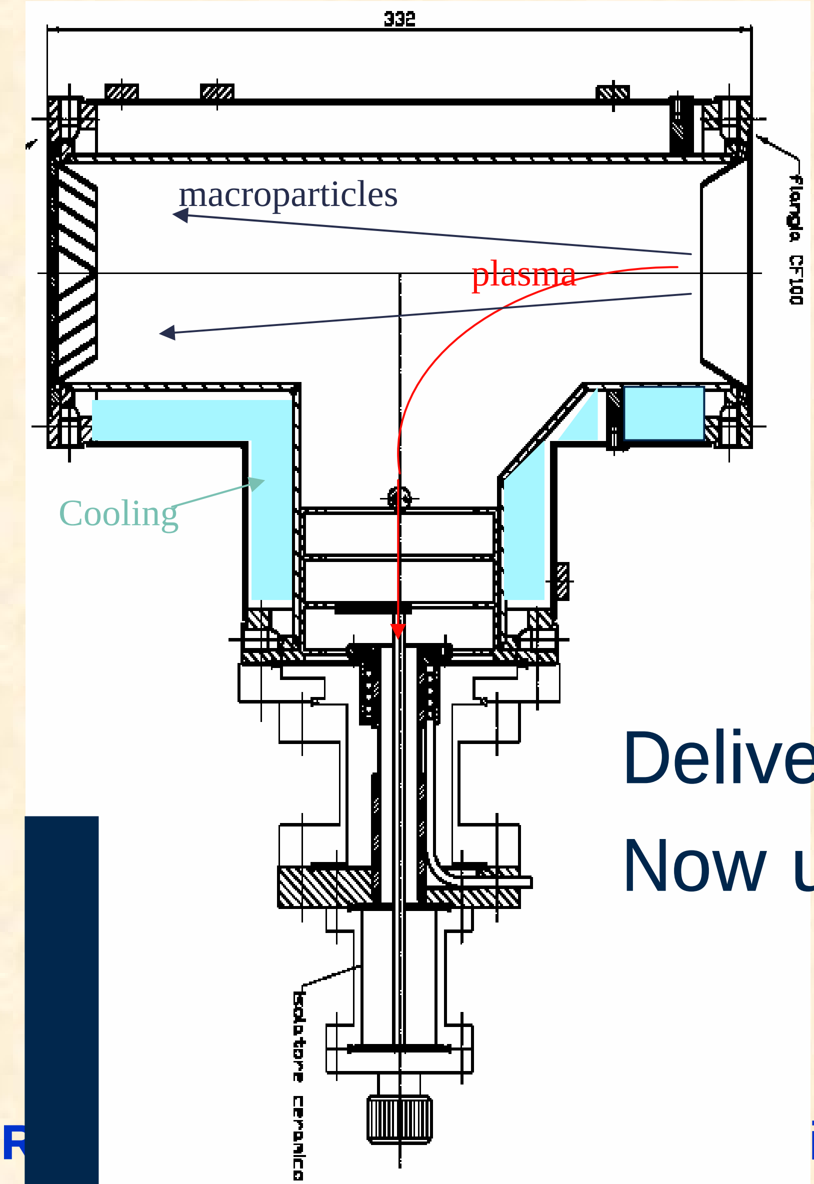




There is a larger number of macrodroplets respect to the planar arc (as aspected)

Most of the particles arrive on the substrate surface still in the liquid phase

New filter design



Access for UHV pumping

Higher efficiency

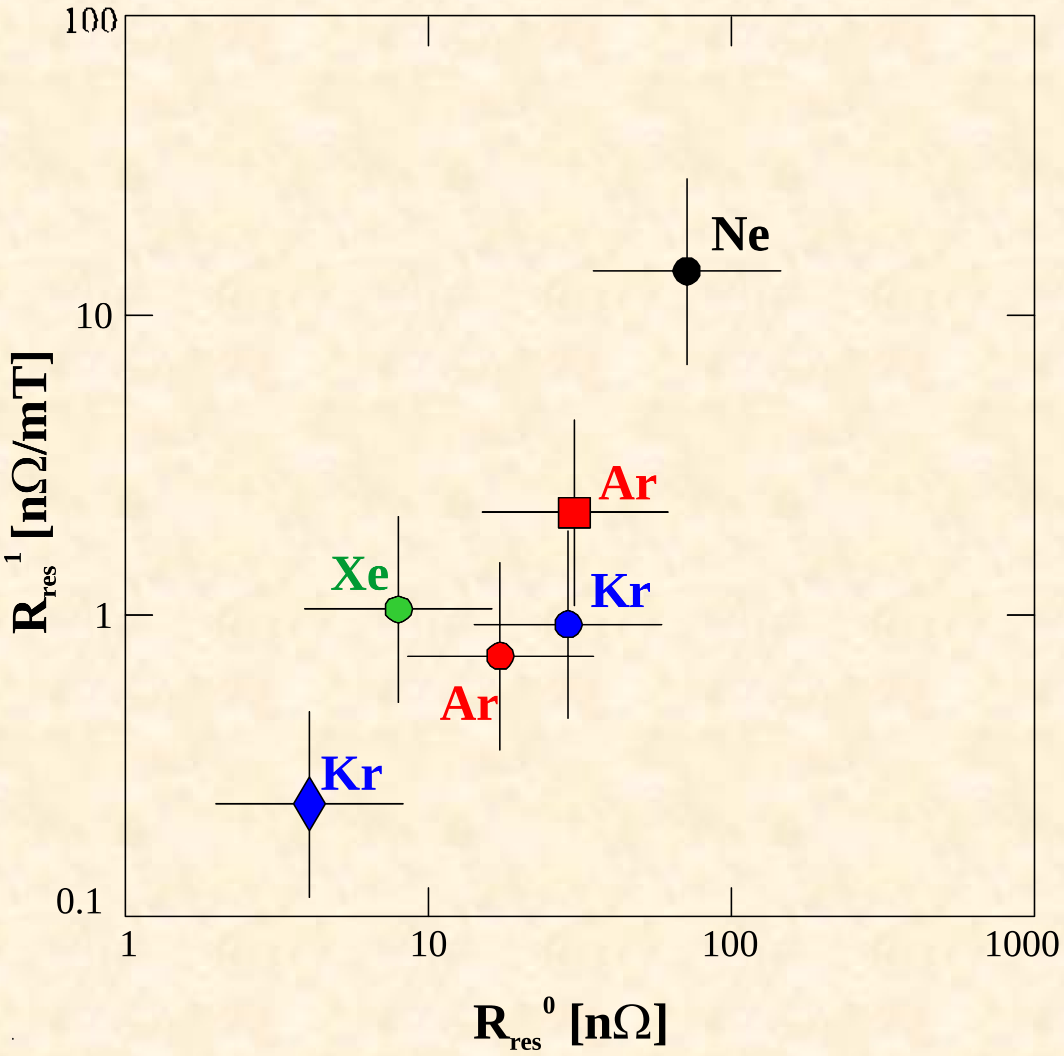
Easier to clean

Better cooling

Delivered to Rome march 2006

Now under study

Influence of the sputtering gas



Difference in Residual Surfarec Risistance due to different gases. The effect in very important in the case of neon

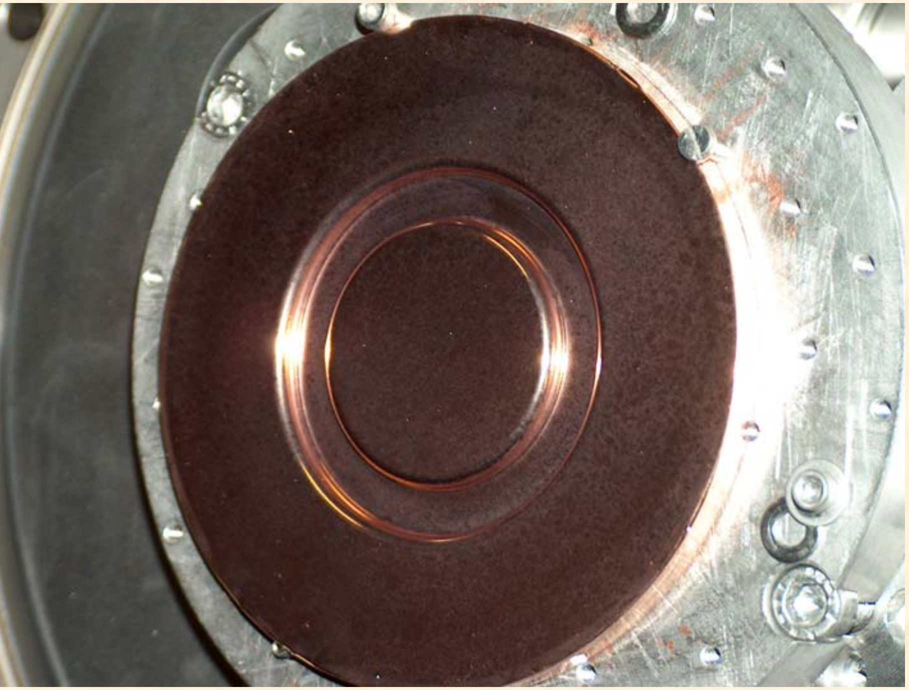
Noble gases implanted in metals are known to cluster in solid form small size: 3nm

Does it apply to Nb sputtered films? Pinning studies (R_{fl}) suggest that it does

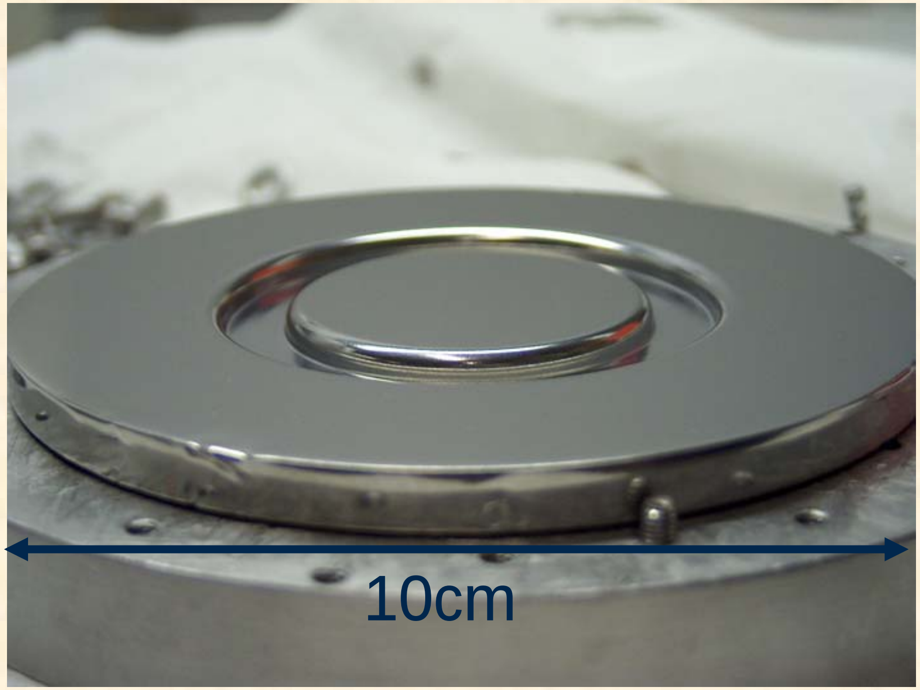
Thanks to S. Calatroni, CERN Geneva

RF Characterization of Nb film

First high field RF measurements, at 6 GHz were performed at Cornell of four niobium films deposited on large Cu-substrates using the filtered system

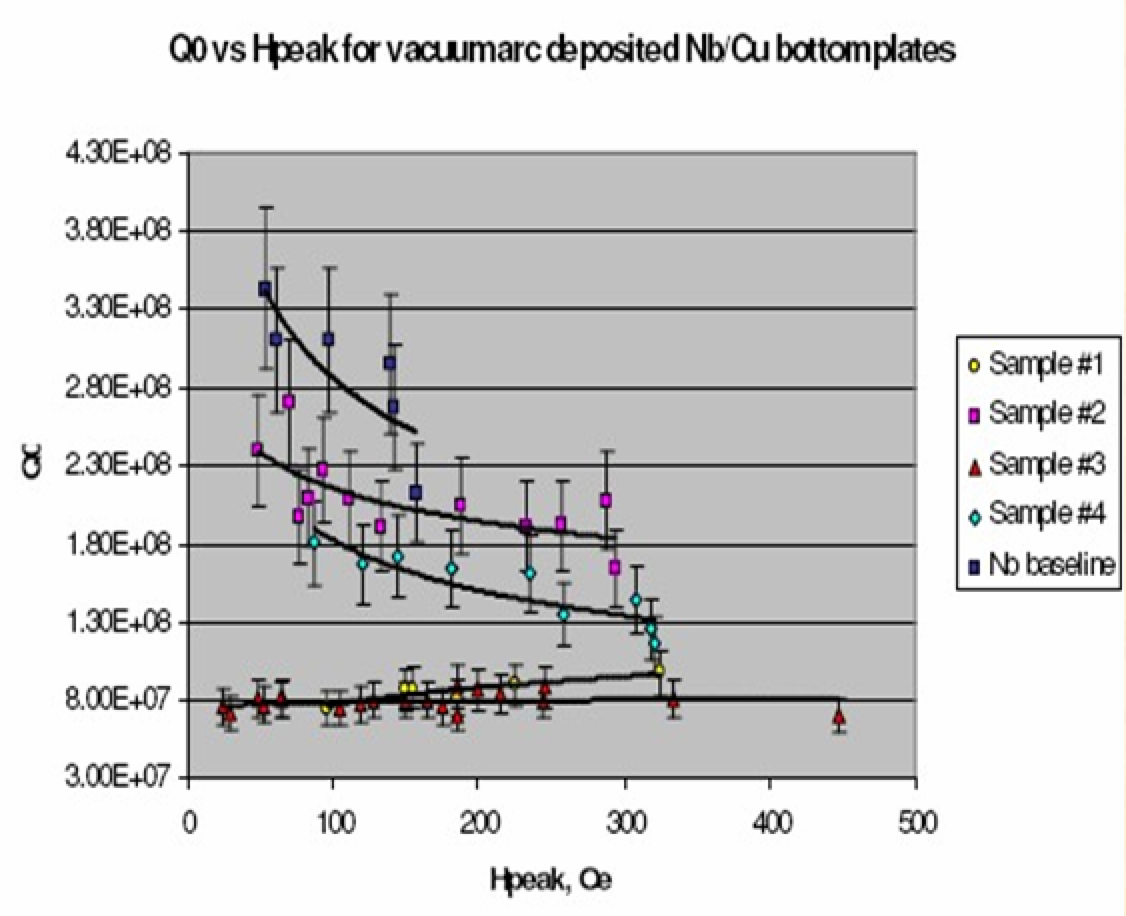


Copper disk before coating



Copper disk after Nb coating

First high field RF characterization of Nb-coated copper samples



Both Nb-bulk and Nb-film on Cu curves are limited by quench.

Nb-films show somewhat greater surface resistance than Nb bulk.

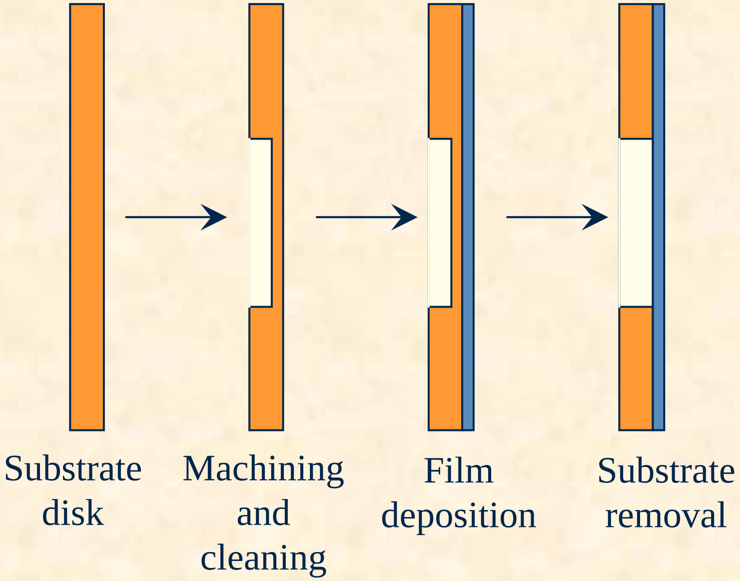
The surface resistance on two of the samples is constant up to 450 Oe.

The Cornell test system is being upgraded to higher Q.

A.Romanenko, H. Padamsee, *RF Properties at 6 GHz of Cathodic Arc Films up to 300 Oe,* Proc. SRF 2005, Cornell University (2005)- in print.

Surface defects: measurement of pinholes

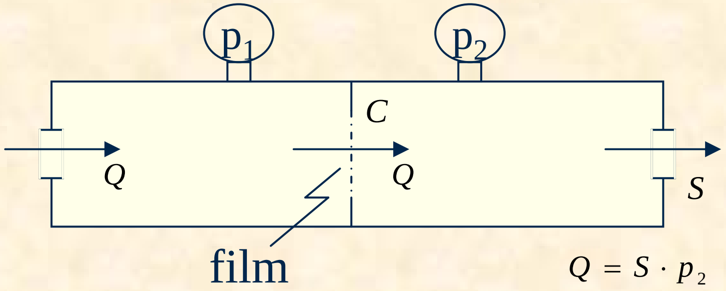
Irregularities on the substrate surface ➡ shadowing effect
film inhomogeneities



He leak rate experiment

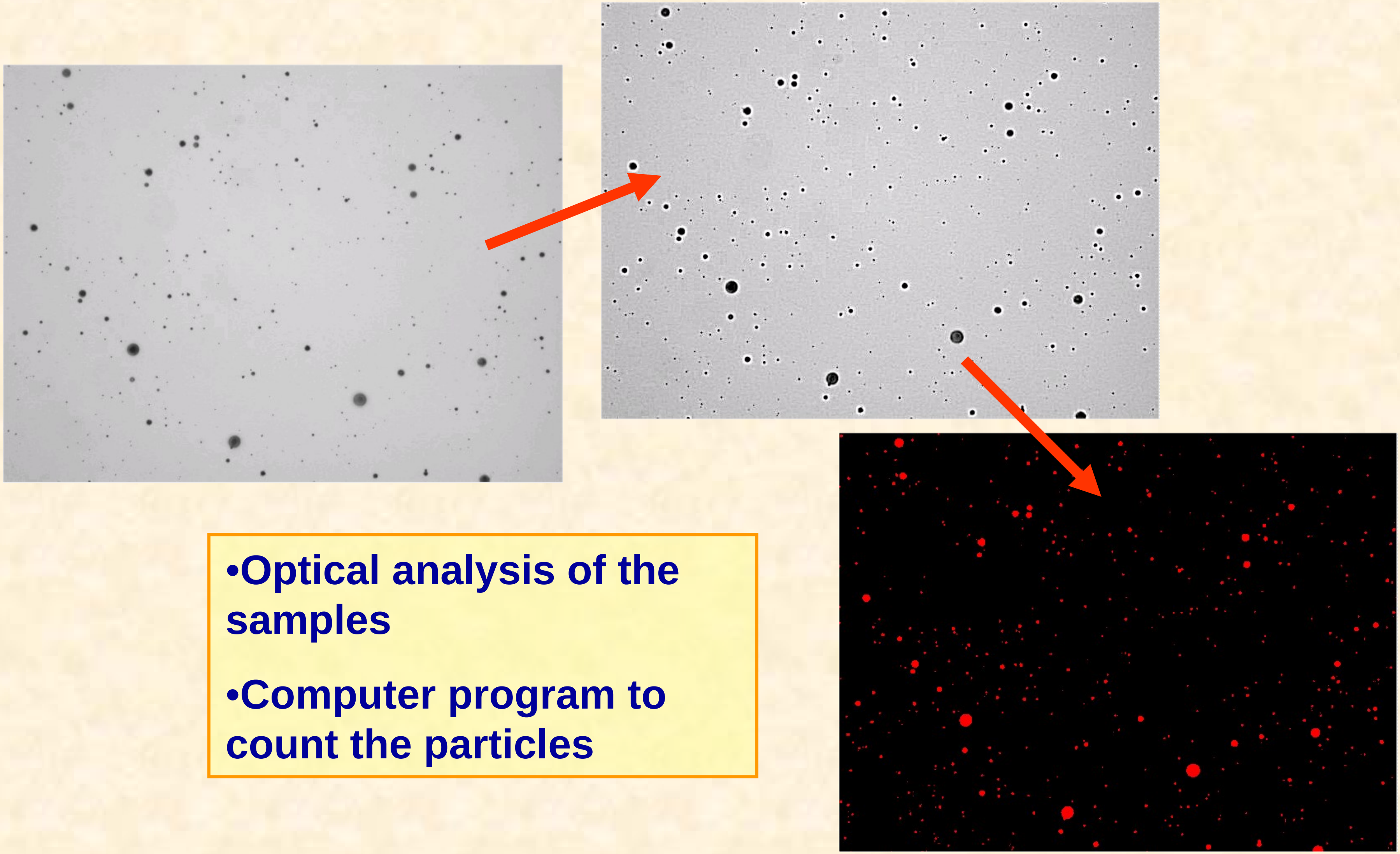
$\langle\Theta_{sp}\rangle$ fraction of leaky film surface

Chemical polishing	equator iris	9° 50°	4.4 ppm 25 ppm
Electro-polishing	equator	9°	0.1 ppm



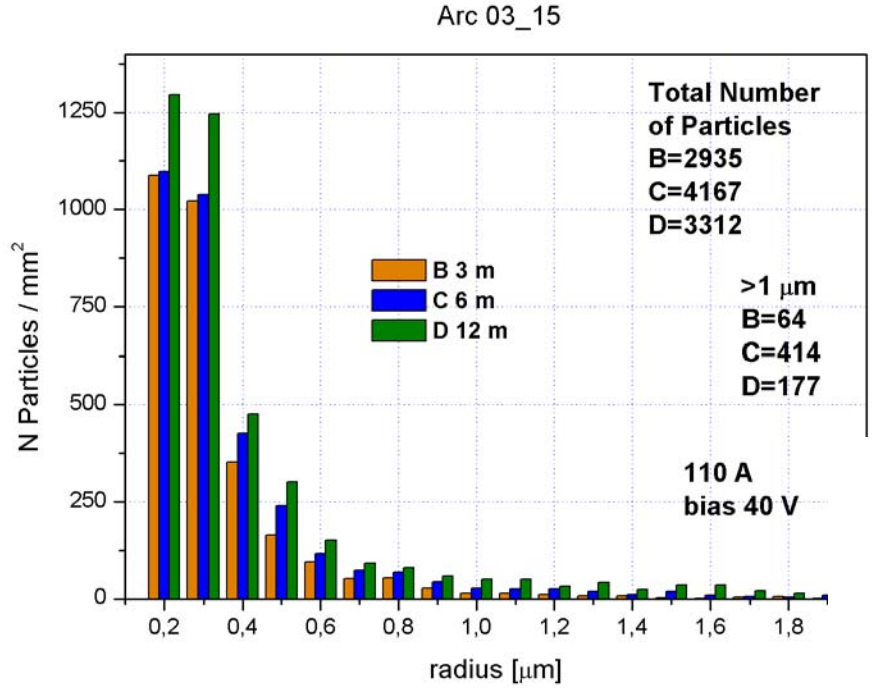
Thanks to S. Calatroni, CERN Geneva

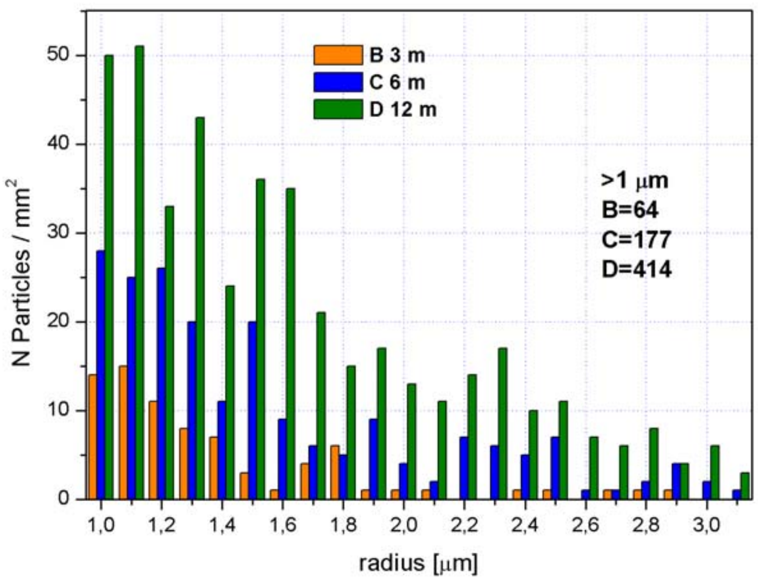
Droplets estimation



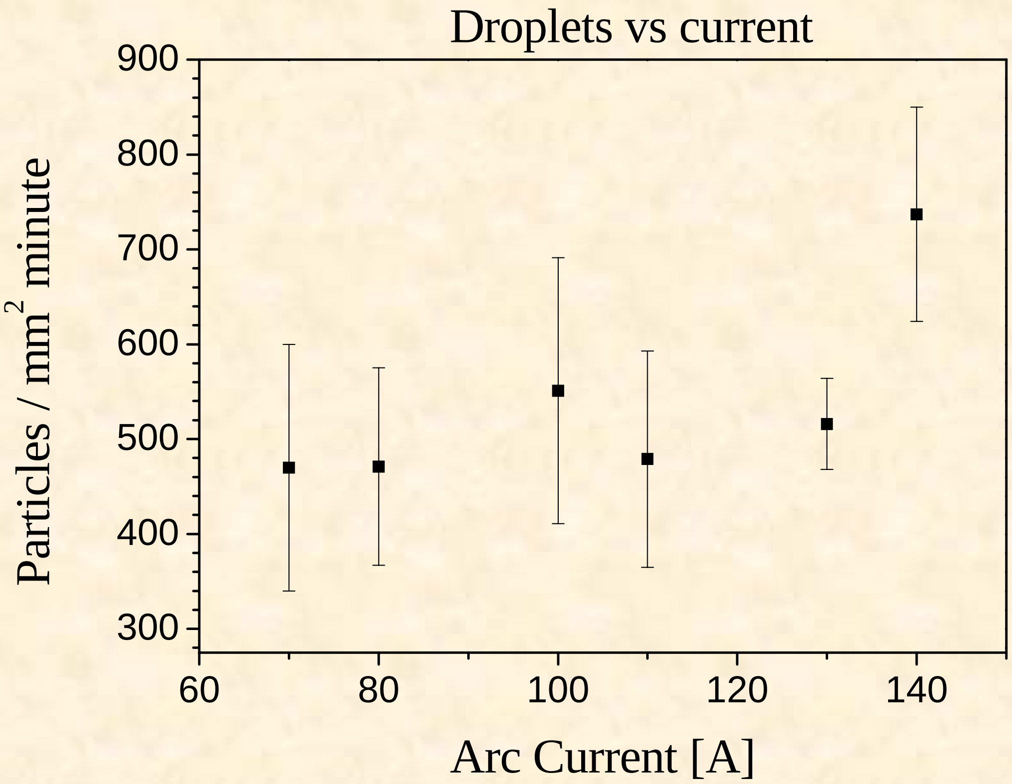
- •Optical analysis of the samples
- •Computer program to count the particles

Droplets results





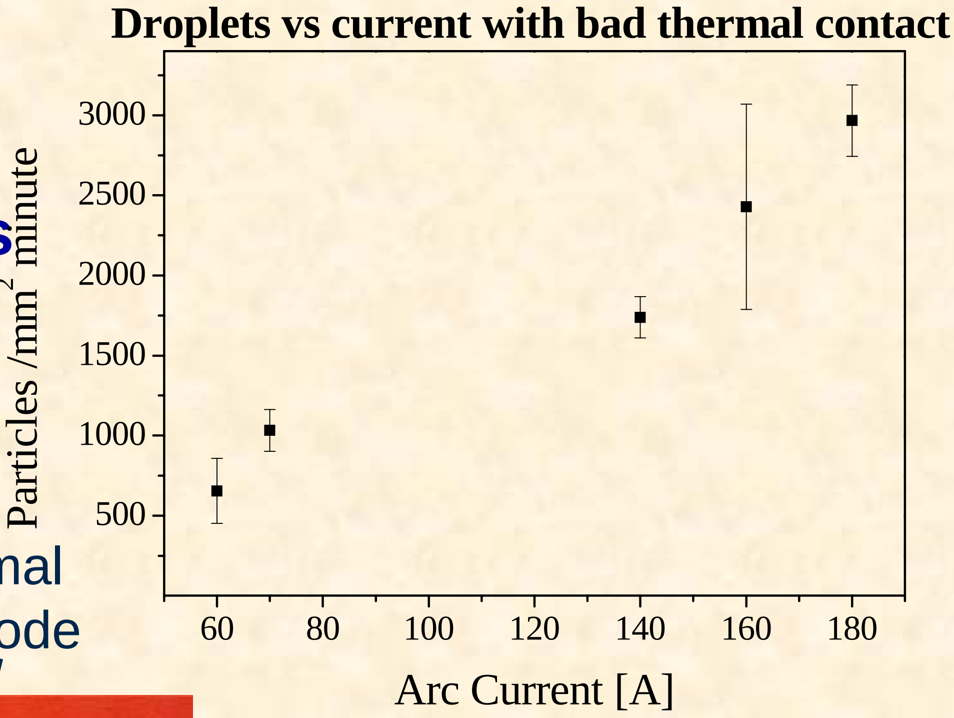
Macroparticles Vs Arc Current



- **No strong dependence from the current**
- **But with higher current there is greater deposition rate**

Bad thermal contact

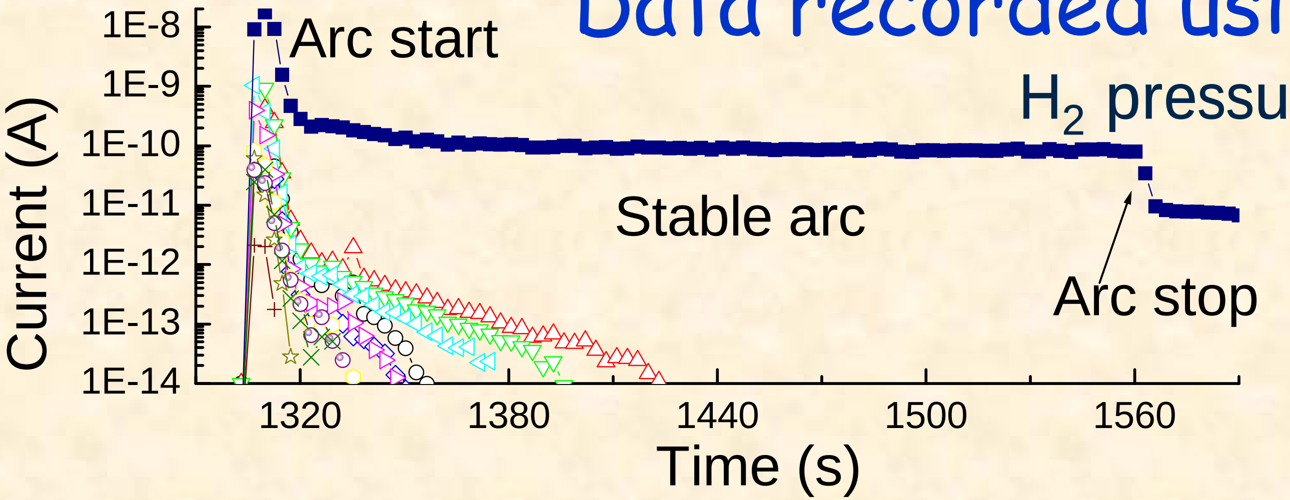
- In the case of bad thermal contact there is a dependence of the droplets from the current



Bottom view

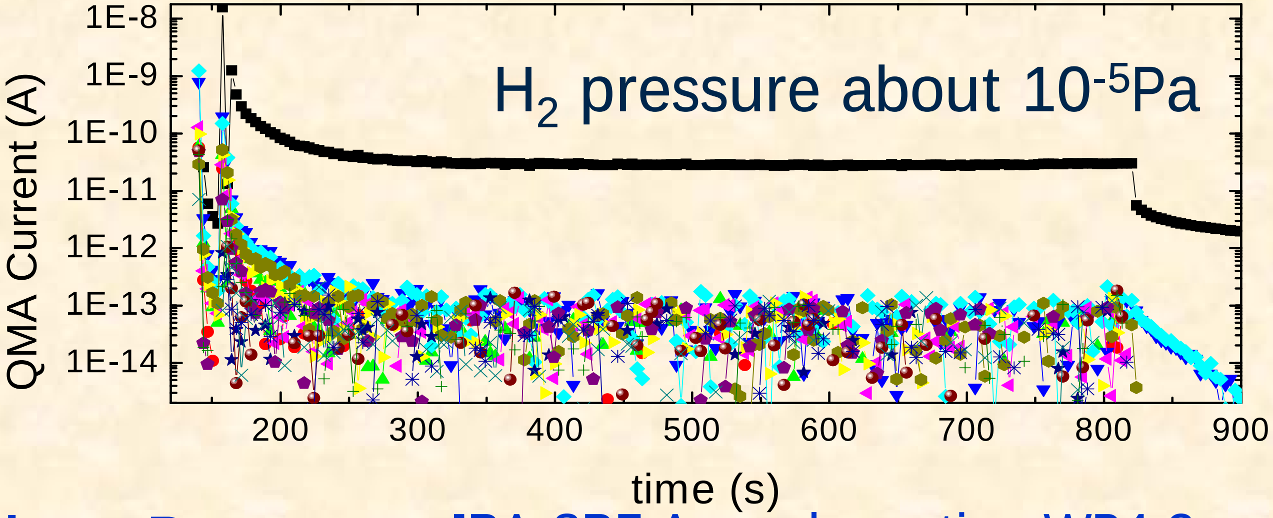
Vacuum condition during arc

Data recorded using a QMA



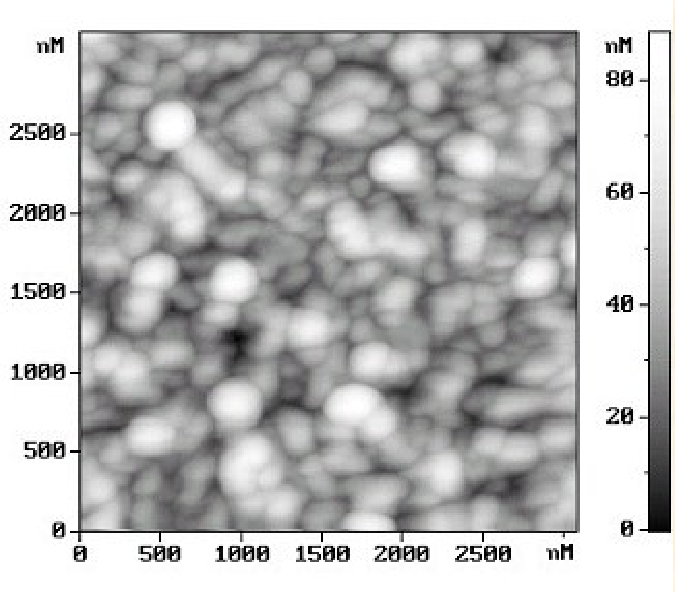
H_2 pressure about 10^{-5}Pa

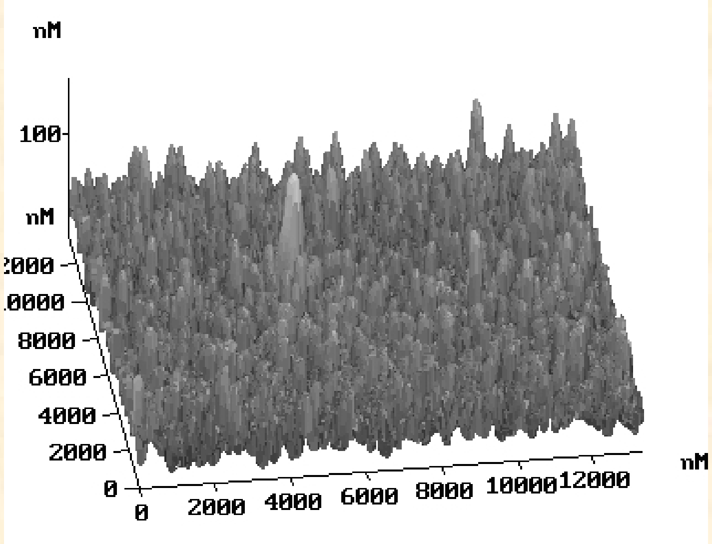
Clean condition



Presence of contaminants

AFM Analysis



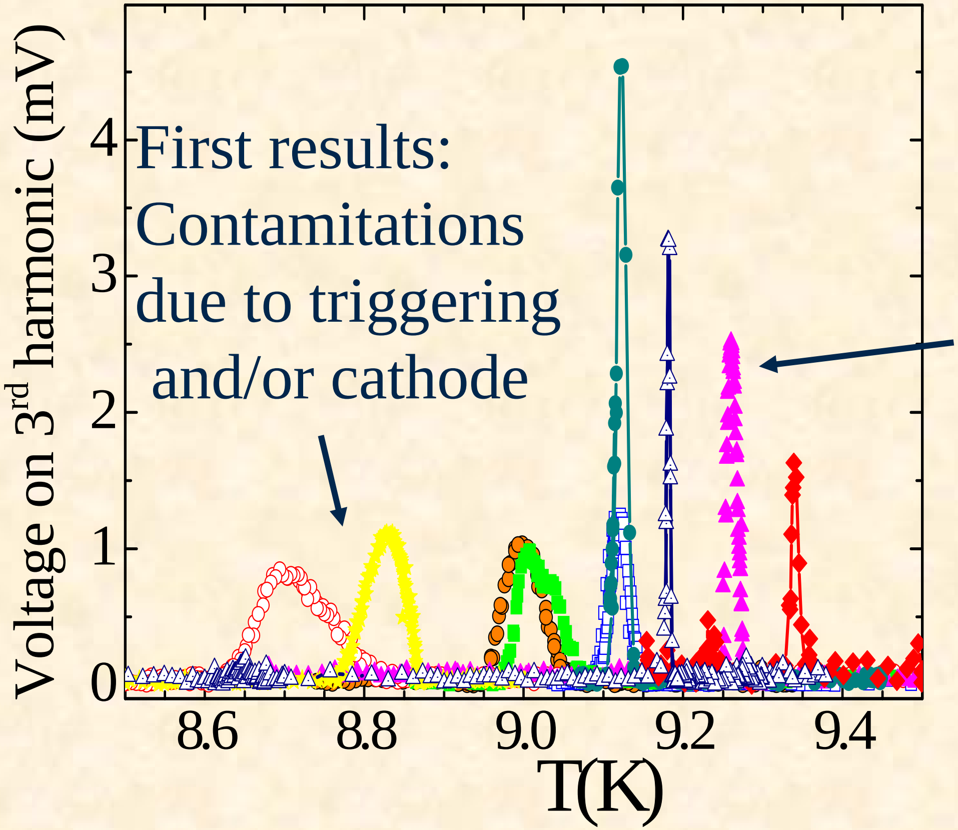


The grain size measured by AFM is about 30nm, a factor 3 larger then standard Nb sputtered films.

Dependence of grain size on voltage Bias are under study

Inductive T_c Measurement

Resistive Measurement of Critical temperature T_c doesn't provide information on uniformity. Inductive Measurement is an average on a large surface (several mm square)



First results: Contamitations due to triggering and/or cathode

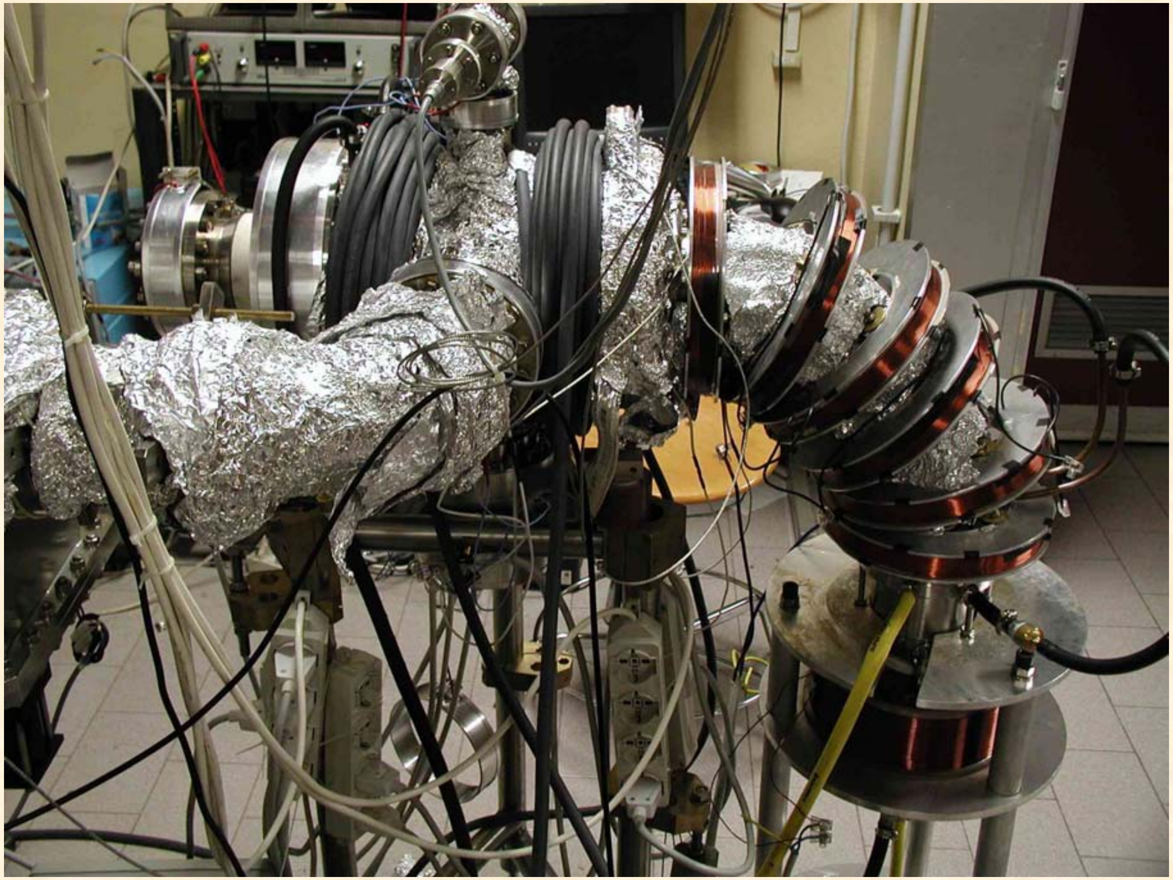
Nb film Thickness from 40 to 1000 nm

Our standard results after discharge stabilitation
$9.1K < T_c < 9.4K$
$\Delta Tc < 0.02K$
Very similar to bulk values

Nb Bulk has T_c=9.26K

The first UHV filtered system

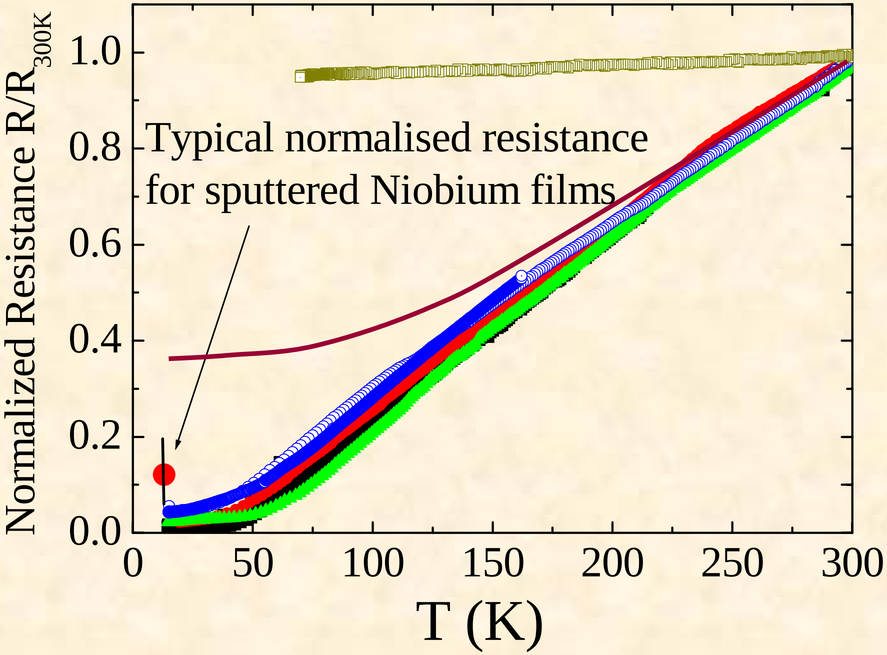
We commissioned a classic quarter torus type filter



- Main Vacuum Chamber and filter vacuum chamber are water cooled
- The electrons are magnetized and the magnetic field lines guides the electrons through the curved chamber.
- Ions mains follows electrons:
- Macroparticle stop on the chamber walls

R (T) measurements and RRR

$$\text{RRR} = \text{Residual Resistivity Ratio} = \frac{R_{300K}}{R_{4.2K}} \approx \frac{R_{300K}}{R_{10K}} \approx \frac{\rho_{ph} + \rho_{imp}}{\rho_{imp}}$$



RRR values from 10 to 100 were obtained

Deposition temperature below 100°C

Thickness 100-1000nm

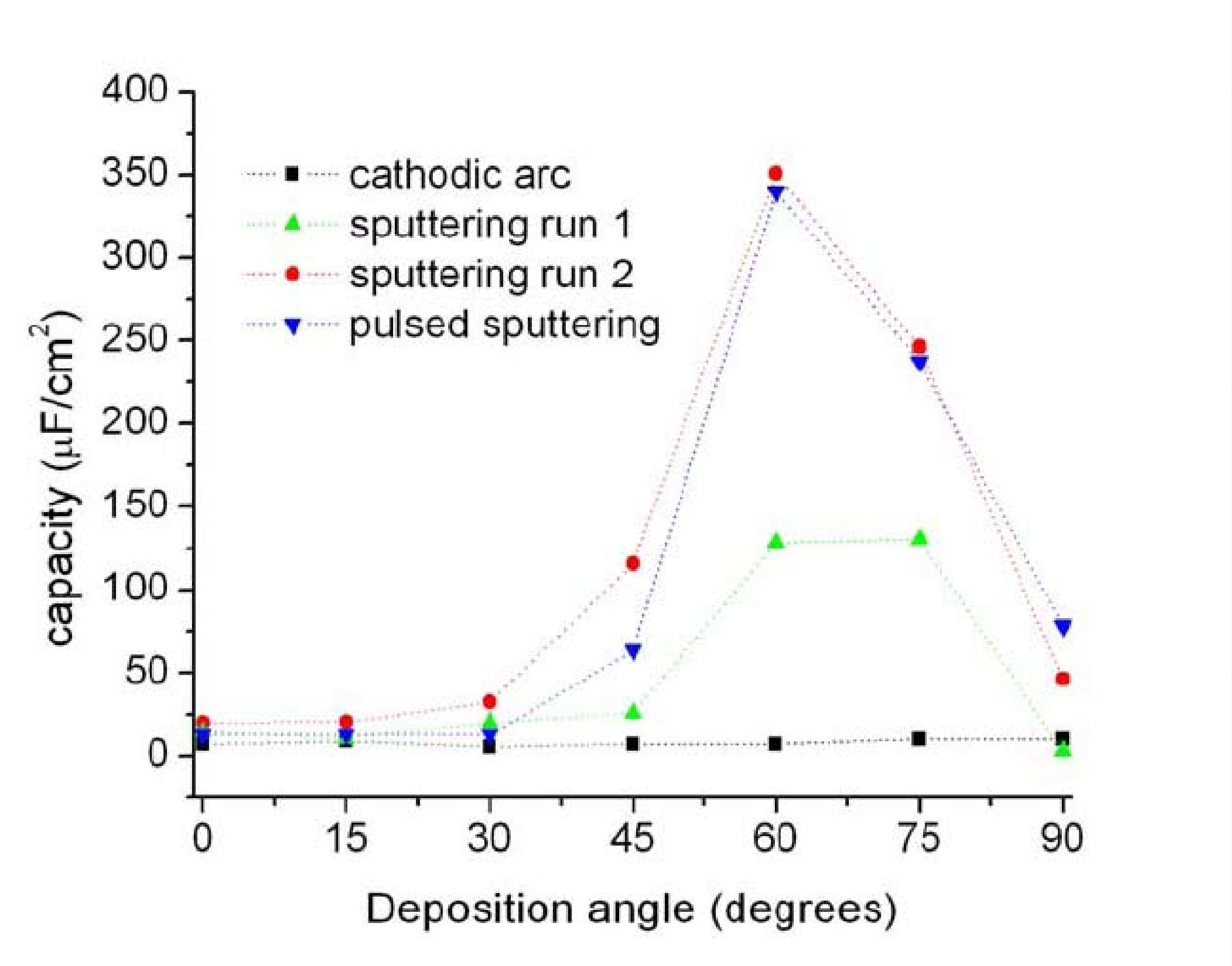
Main sources of impurities in vacuum arc can be:

the vacuum condition (we use UHV)

the ceramic trigger (replaced with a laser trigger)

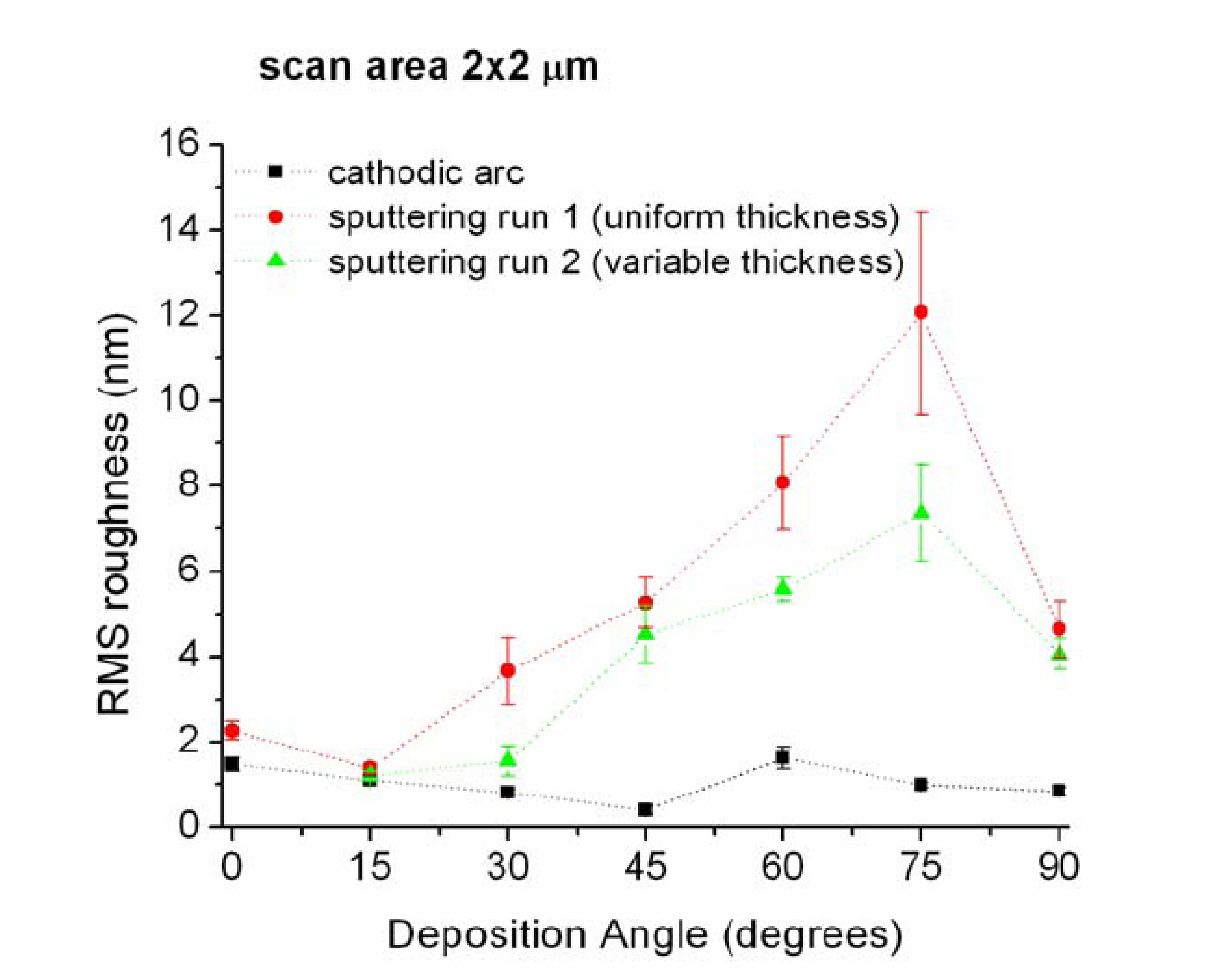
floating potential electrode (Nb floating electrode)

Results from G. Keppel, M. Musiani, N. Patron, V. Palmieri, D. Tonini, G. Torzo

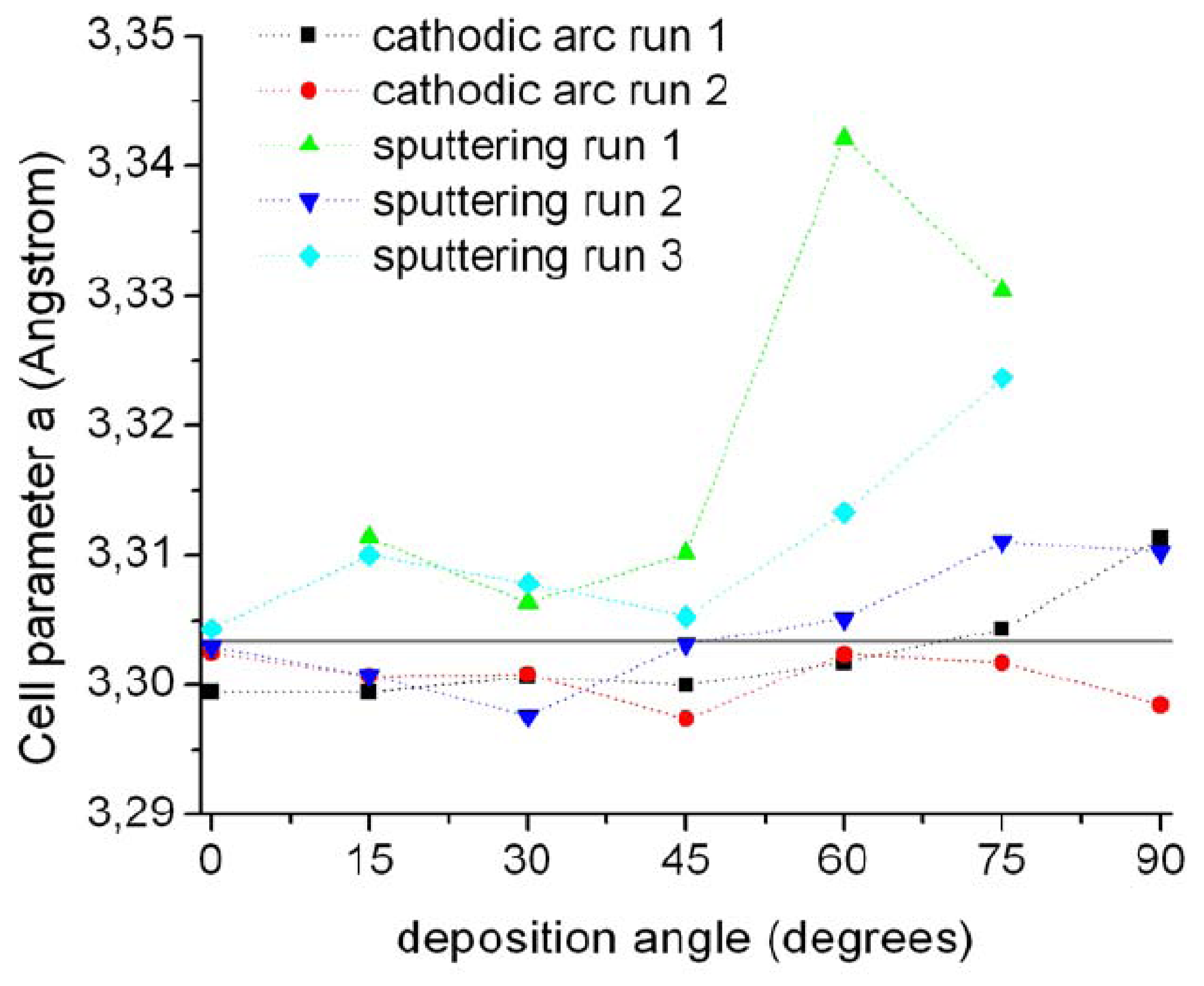


Results from G. Keppel, M. Musiani, N. Patron, V. Palmieri, D. Tonini, G. Torzo

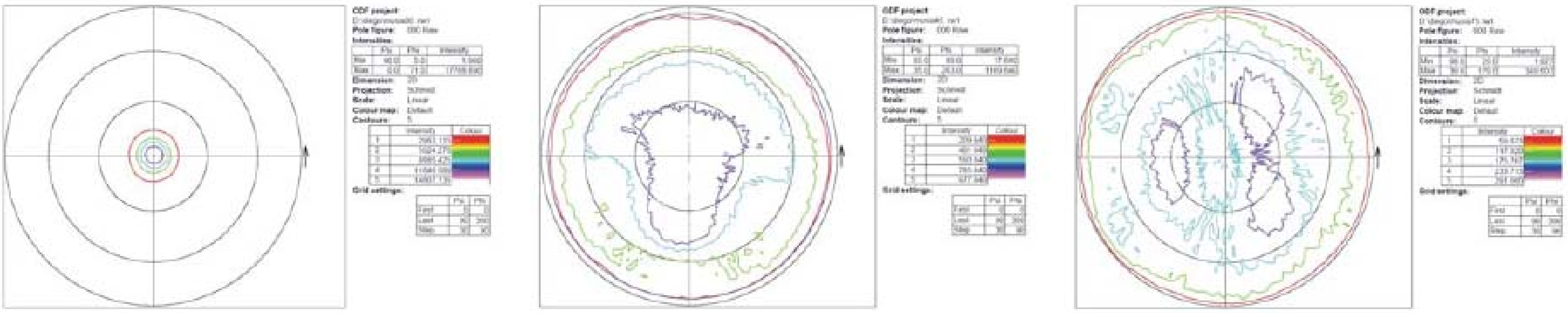
I



Results from G. Keppel, M. Musiani, N. Patron, V. Palmieri, D. Tonini, G. Torzo

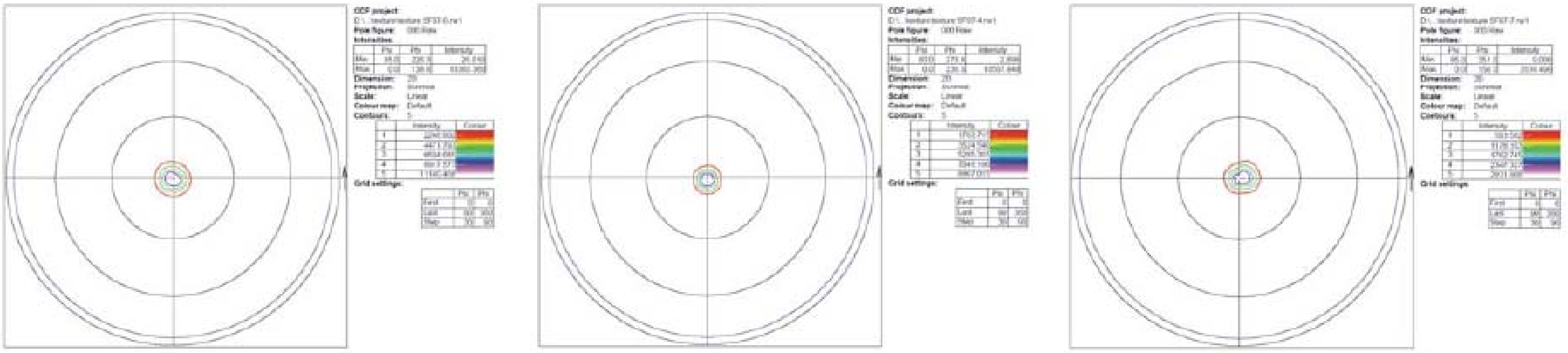


Results from G. Keppel, M. Musiani, N. Patron, V. Palmieri, D. Tonini, G. Torzo



0 degrees 45 degrees 75 degrees

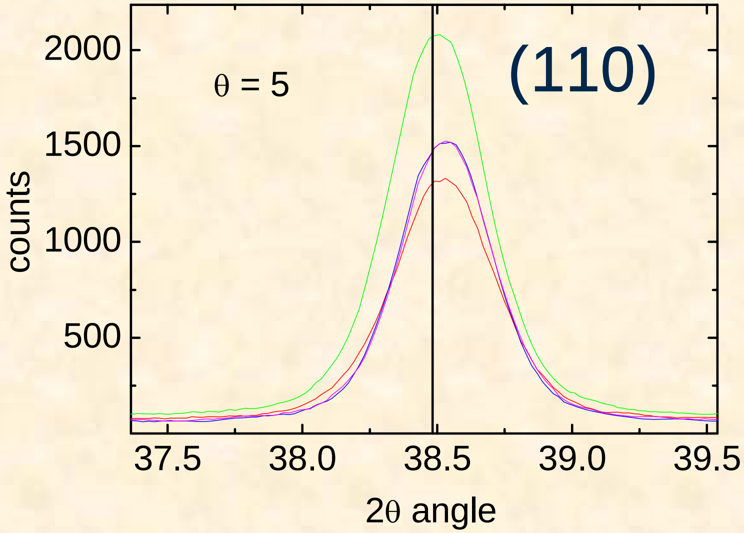
FIG. 9 Pole figure of texture respect to 110 XRD peak in sputtered films

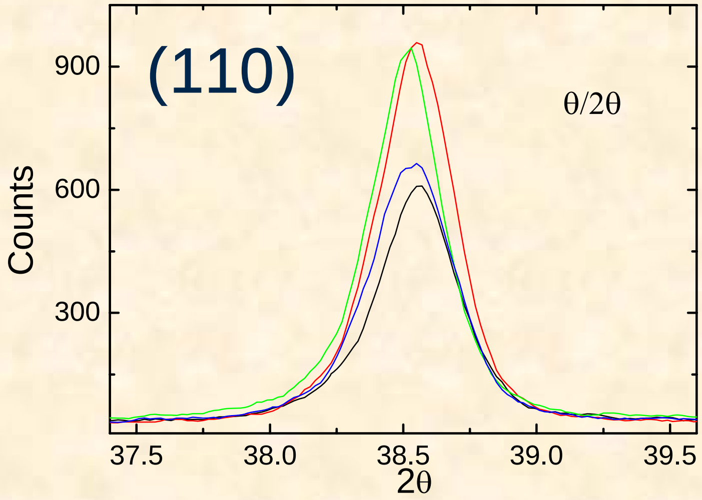


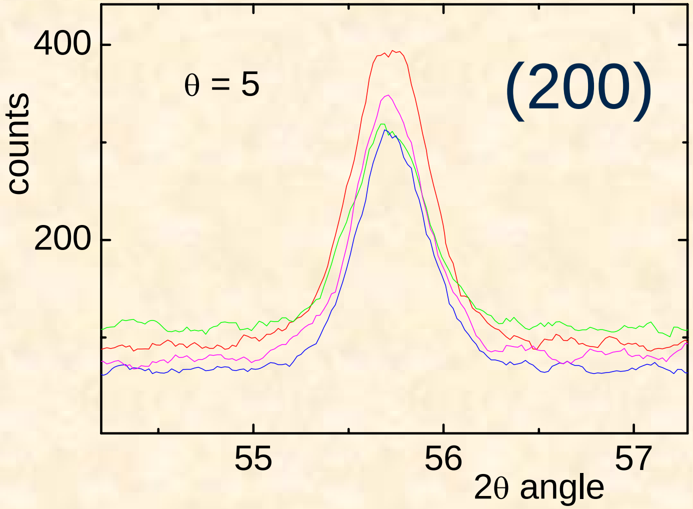
0 degrees 45 degrees 75 degrees

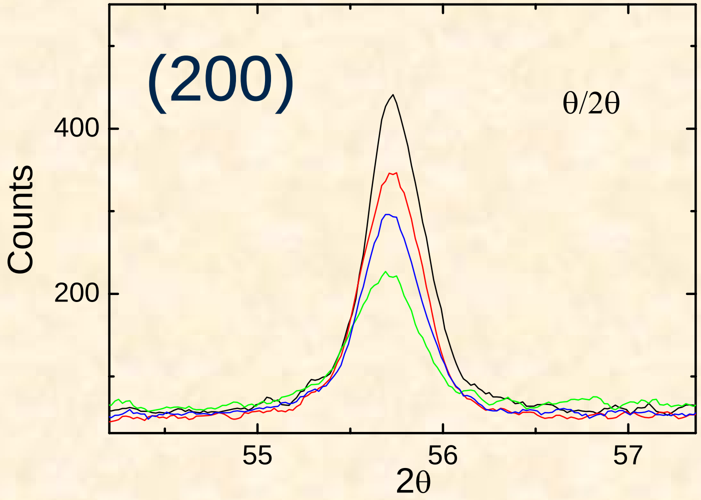
FIG. 10 Pole figure of texture respect to 110 XRD peak in arc deposited films

XRD on Nb/Cu samples vs Bias (I)



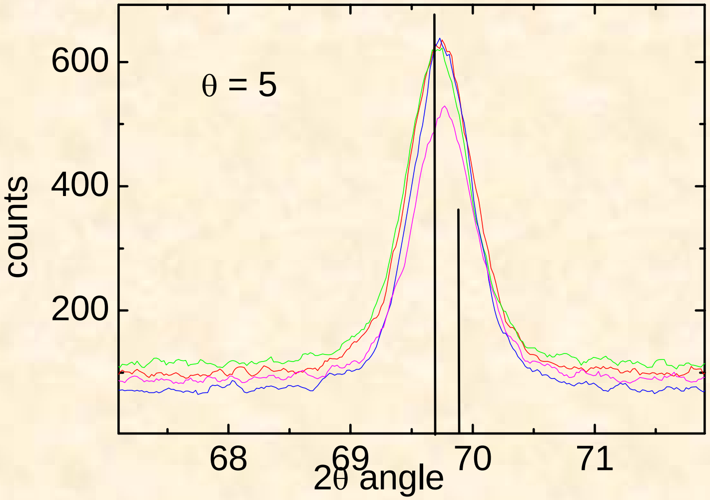


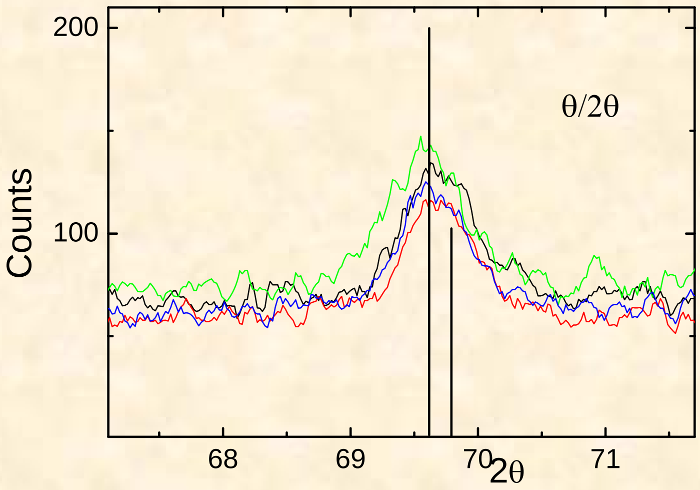




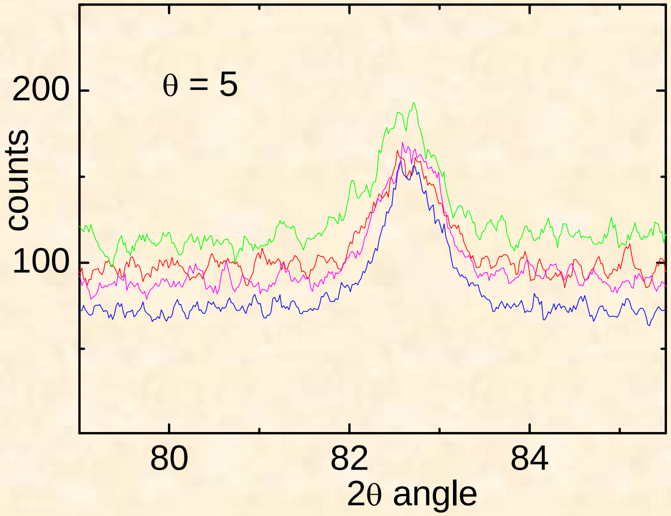
XRD on Nb/Cu samples vs Bias (II)

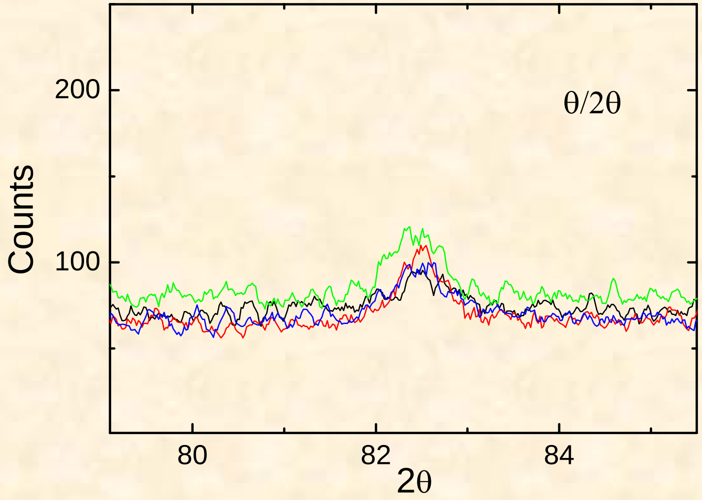
(211)



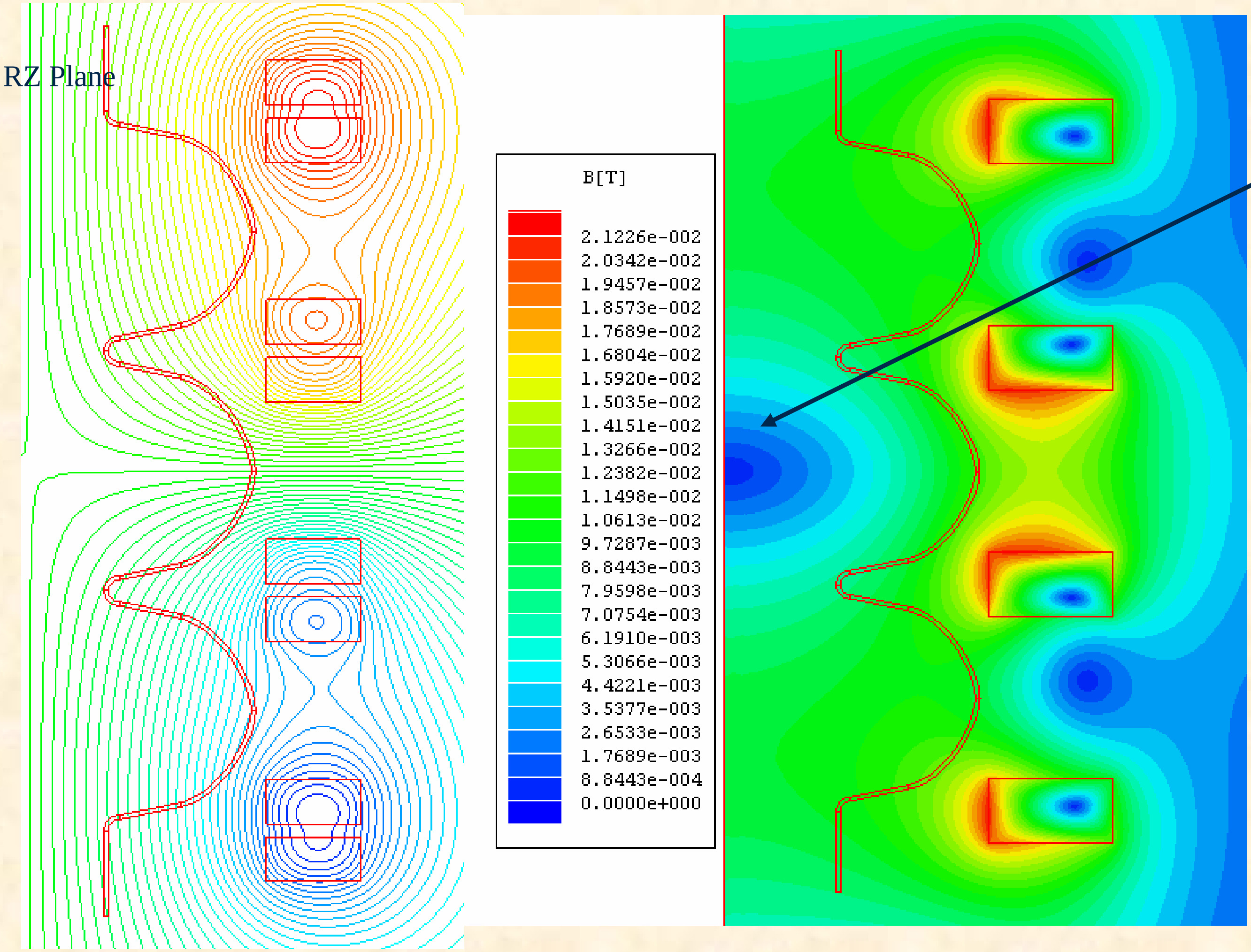


(220)





Magnetic field intensity



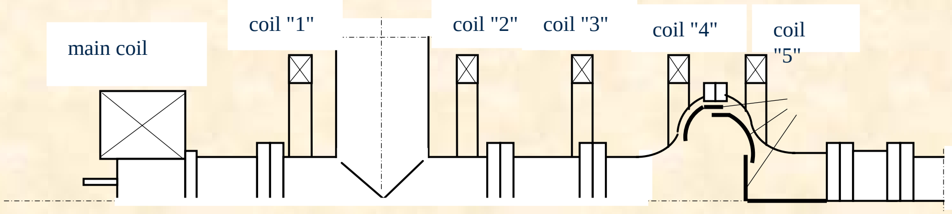
Magnetic field gradient

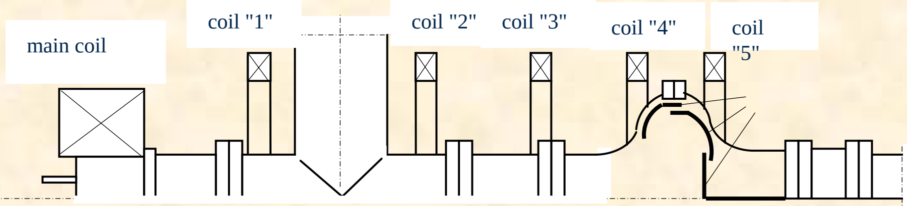
$$\mathbf{F}_{\|} = -\mu \frac{\partial B}{\partial \mathbf{s}},$$

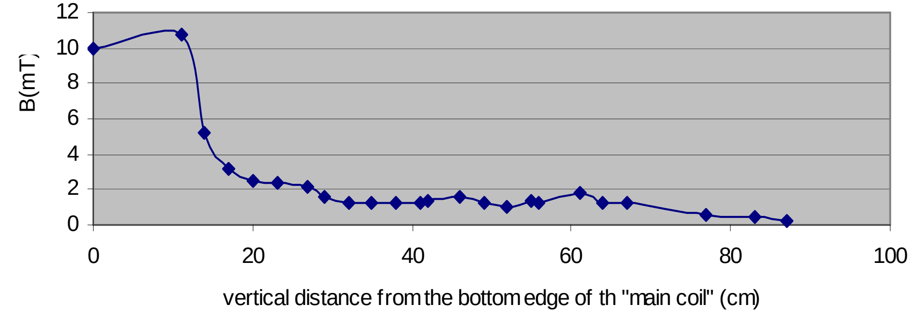
experimental
work will be needed
To check simulations

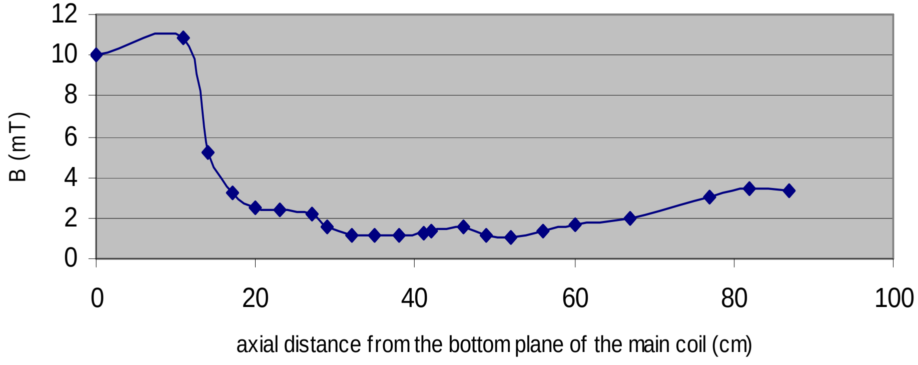
2x900A-t, 2x900, 2x900, 2x900

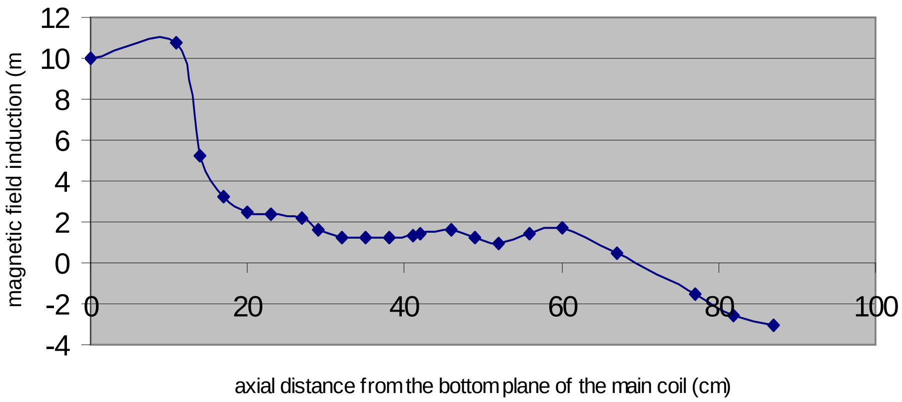
Magnetic field configuration for plasma transport and cavity coating





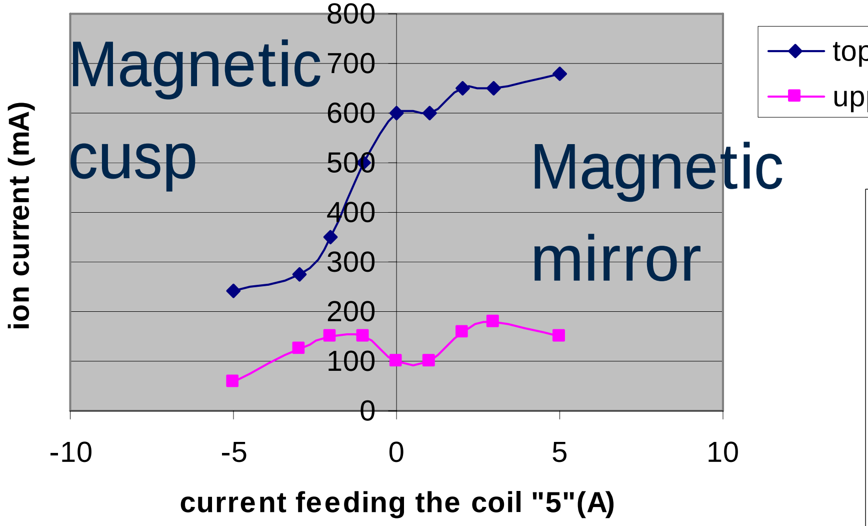

Axial distribution of a vertical component of magnetic field along the axis of the "cavity" system. The coil "5" above the cavity chamber is not excited.


Vertical component of magnetirc field along the axis of the "cavity" system. The coil "5",placed above the cavity chamber is fed with dc current of 3 A (magnetic mirror configuration)

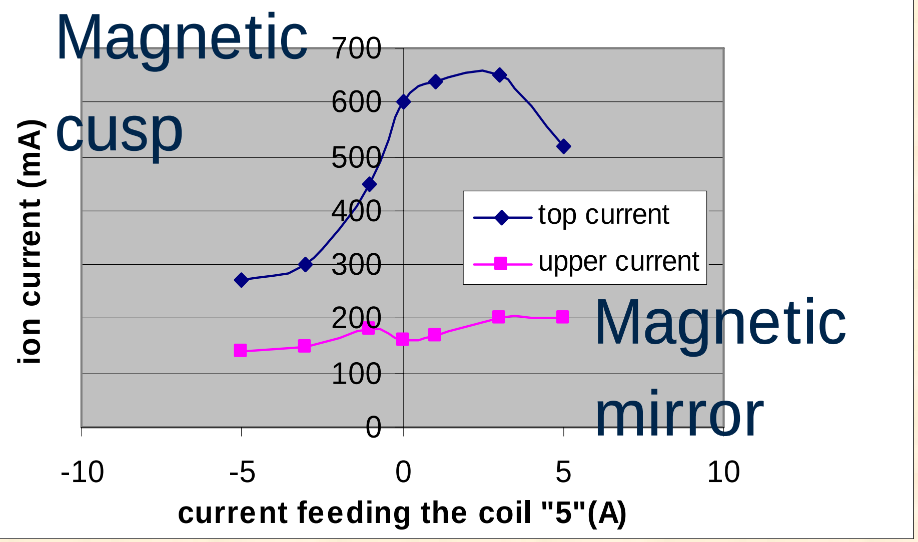

Axial component of magnetic fiel along the axis of the "cavity" systeml . The coil "5" above the cavity chamber is fede with the current of - 3A (cusp configuration).

Ion current Distribution

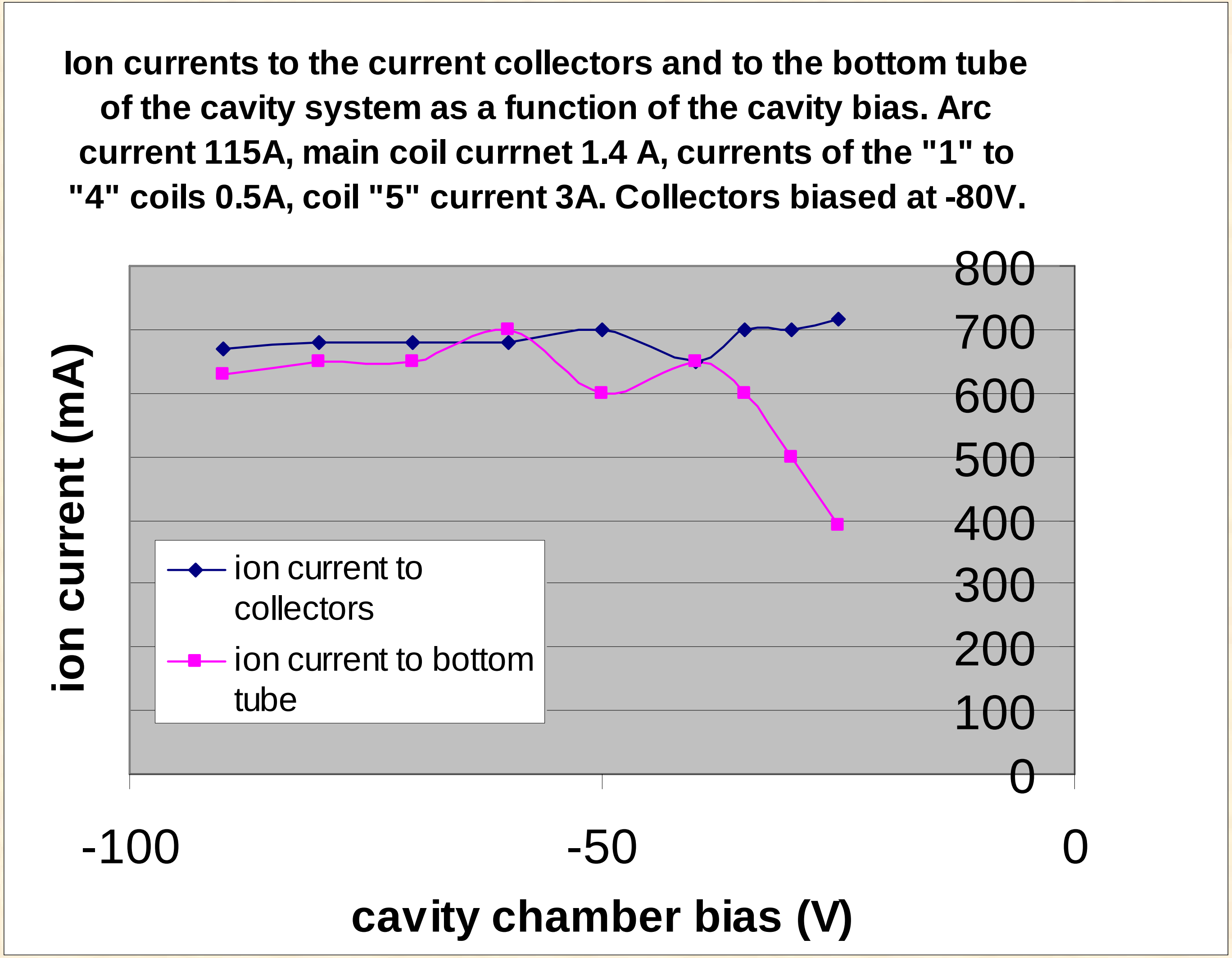
Ion currents to the "Top" and the "upper" collector in the cavity sytem. Collectors bias voltage -80 V,
cavity chamber - grounded

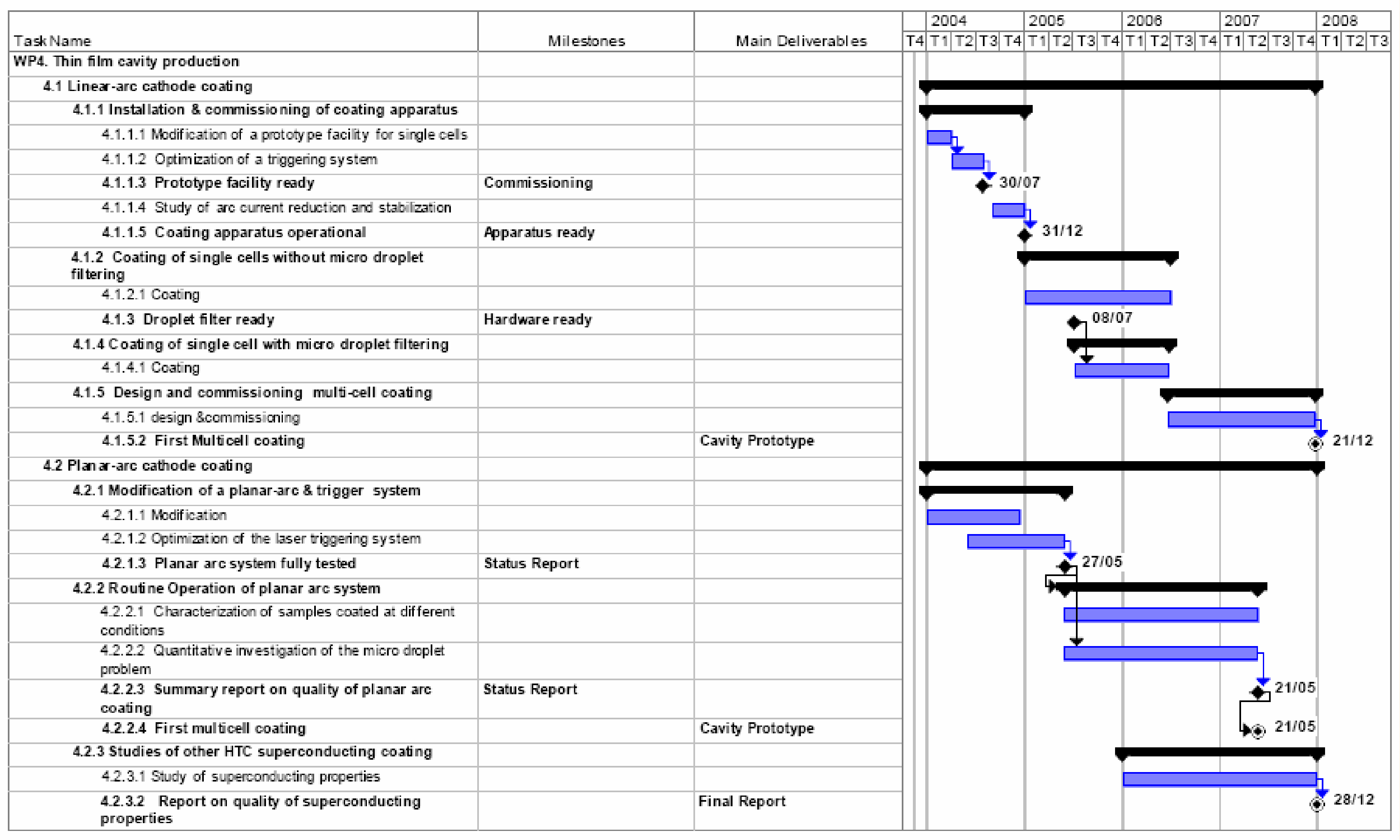


Ion current to the collectors "top" and "upper" vs the current feeding the coil "5"
Cavity Chamber Floting



Ion current distribution in the cut-off and in the cavity cell

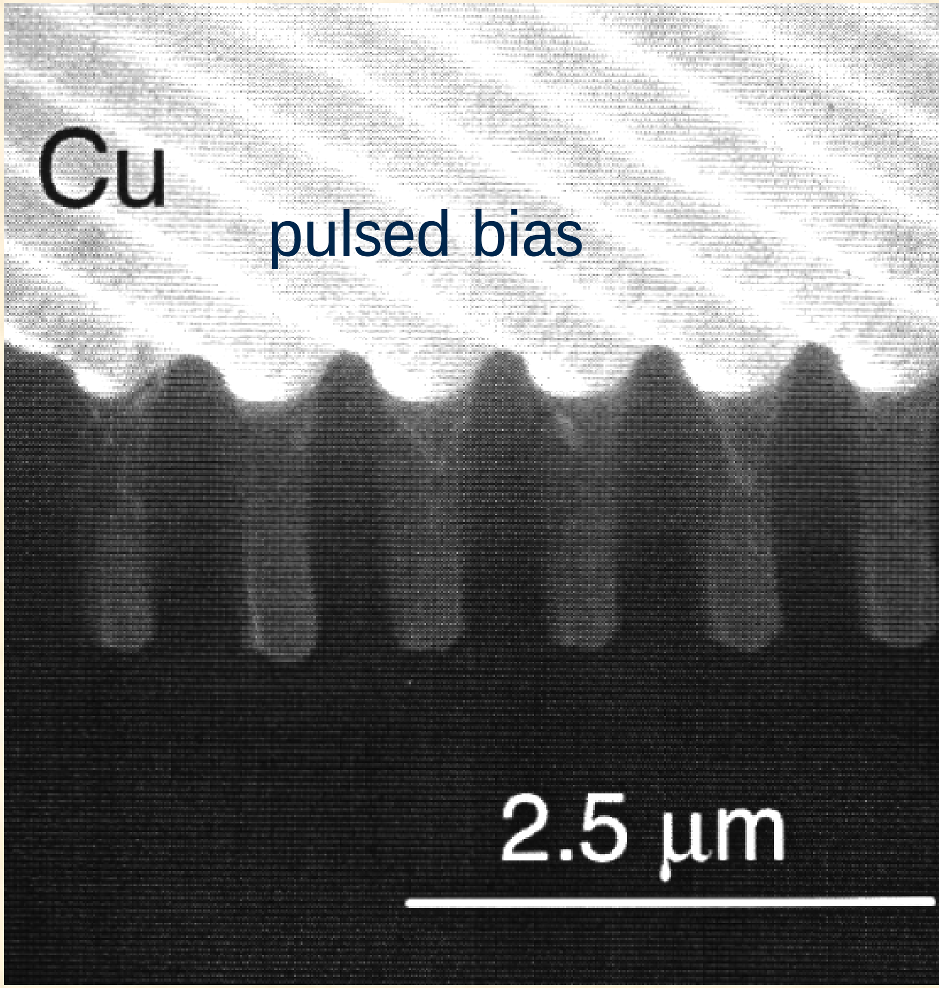




Task Name	Milestones	Main Deliverables
WP4. Thin film cavity production		
4.1 Linear-arc cathode coating		
4.1.1 Installation & commissioning of coating apparatus		
4.1.1.1 Modification of a prototype facility for single cells		
4.1.1.2 Optimization of a triggering system		
4.1.1.3 Prototype facility ready	Commissioning	
4.1.1.4 Study of arc current reduction and stabilization		
4.1.1.5 Coating apparatus operational	Apparatus ready	
4.1.2 Coating of single cells without micro droplet filtering		
4.1.2.1 Coating		
4.1.3 Droplet filter ready	Hardware ready	
4.1.4 Coating of single cell with micro droplet filtering		
4.1.4.1 Coating		
4.1.5 Design and commissioning multi-cell coating		
4.1.5.1 design &commissioning		
4.1.5.2 First Multicell coating		Cavity Prototype
4.2 Planar-arc cathode coating		
4.2.1 Modification of a planar-arc & trigger system		
4.2.1.1 Modification		
4.2.1.2 Optimization of the laser triggering system		
4.2.1.3 Planar arc system fully tested	Status Report	
4.2.2 Routine Operation of planar arc system		
4.2.2.1 Characterization of samples coated at different conditions		
4.2.2.2 Quantitative investigation of the micro droplet problem		
4.2.2.3 Summary report on quality of planar arc coating	Status Report	
4.2.2.4 First multicell coating		Cavity Prototype
4.2.3 Studies of other HTC superconducting coating		
4.2.3.1 Study of superconducting properties		
4.2.3.2 Report on quality of superconducting properties		Final Report

A new approach: UHV cathodic arc

- **no working gas (UHV)**
- Fully ionized niobium
- high ion energy (>100eV)
- possible to apply bias and magnetic field to guide the plasma
- Using pulsed bias it is possible to coat "no flat" surfaces



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Niobium films on Sapphire

Bias (V)	Thickness (μm)	RRR	a_{Nb} (nm)
-23	0.9-2.8	26	0.3308
-40	0.9-2.6	40	0.3316
-60	1.0-1.7	30	0.3313
-80	0.7-1.0	50	0.3310

Niobium films grown on sapphire present some residual stress, however there is no epitaxy due to low deposition temperature and high deposition rate.

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