

# International Workshop on **Thin films and New Ideas for Pushing the Limits of RF Superconductivity**

INFN - Legnaro National Laboratory - Padua University, October 9-12, 2006



**Enzo Palmieri:**

**what did we learn from this workshop?**

## *Some Figures ...*

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- 47 talks over 3 ½ days
- 68 participants
- International workshop → 3 regions represented

INFN / CNR / Univ. - DESY / Max Plank Inst. / Nurnberg Univ.

- CNRS / CEA – CERN - Andrzej Soltan Inst. - Tel Aviv Univ.

JLab – Los Alamos - Argonne - Fermi Lab - Cornell / California / Florida Univ.

- SLAC

KEK – China Institute of Atomic Energy

# ***Beijing 2004 - International Conference on High Energy Physics***



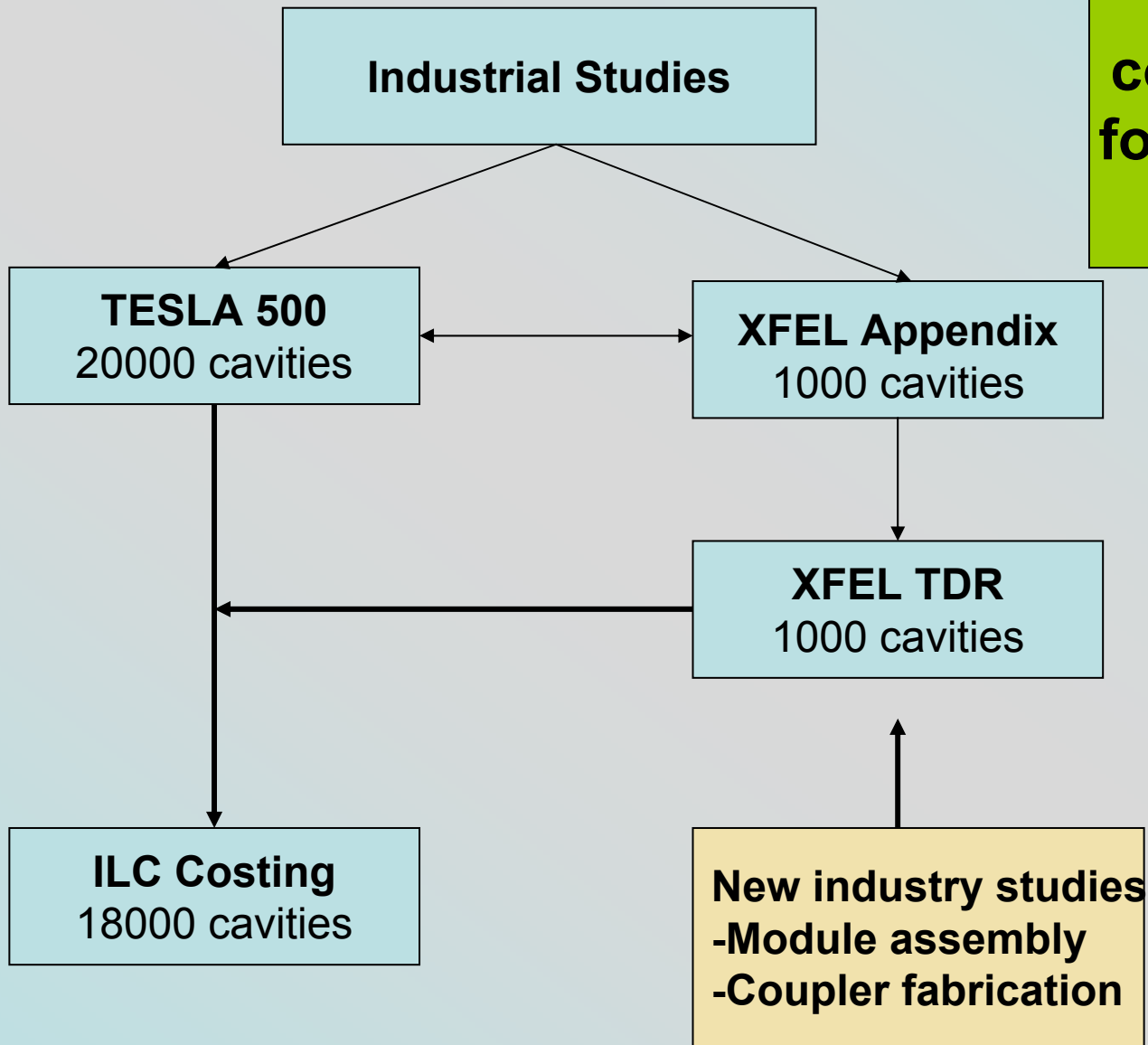
## ***International Technology Recommendation Panel of the International Committee for Future Accelerators (ICFA)***

Front line from left to right:

Akira Masaike, George Kalmus, Volker Soergel, **Barry Barish**, Giorgio Bellettini, Hirotaka Sugawara, Paul Grannis

Back line from left to right:

Gyung-Su Lee, Jean-Eude Augustin, David Plane, Jonathan Bagger, Norbert Holtkamp, Katsunobu Oide



**European  
cost approach  
for mass scale  
production**



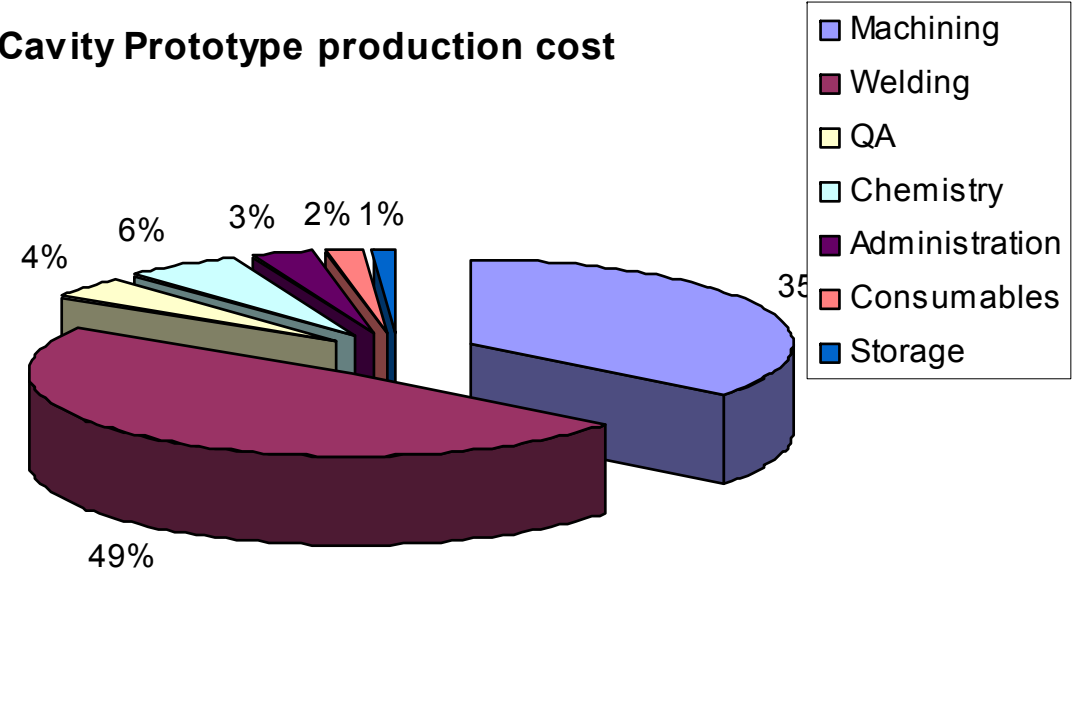
# Comments to cost evaluation

- **Nb Material** (high purity, RRR 300)
  - No shortage of raw Nb material (40.000 tons annual production, ILC needs around 500 tons)
  - But limited number of high purity melting facilities
    - Today there are 4 qualified companies, but only one is capable of producing full yield
  - Marginal savings in mass production (from industrial study)
    - Size of melting furnace is limited
    - But some saving can be realized by
      - Disc rather than rectangular sheet (scrap can be recovered)
      - Other material produced ready for fabrication, e.g. flange material
    - But: Latest developments in large/single crystal cavities promise cost reductions
- **The result is robust costing**

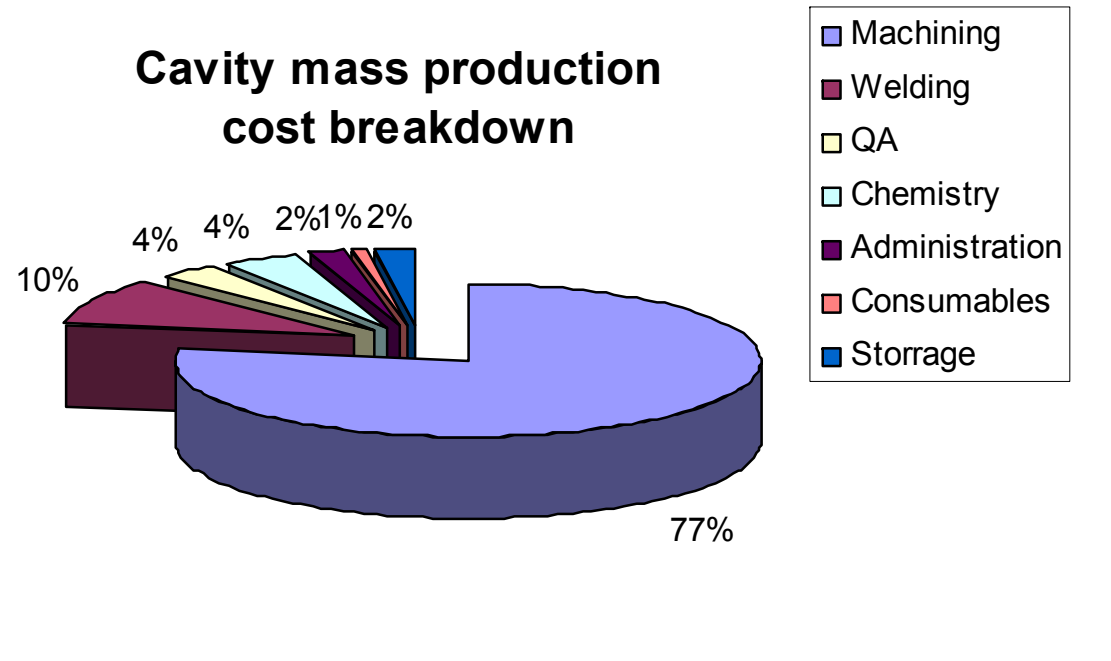


**500KW electron beam cold hearth furnace**

Cavity Prototype production cost

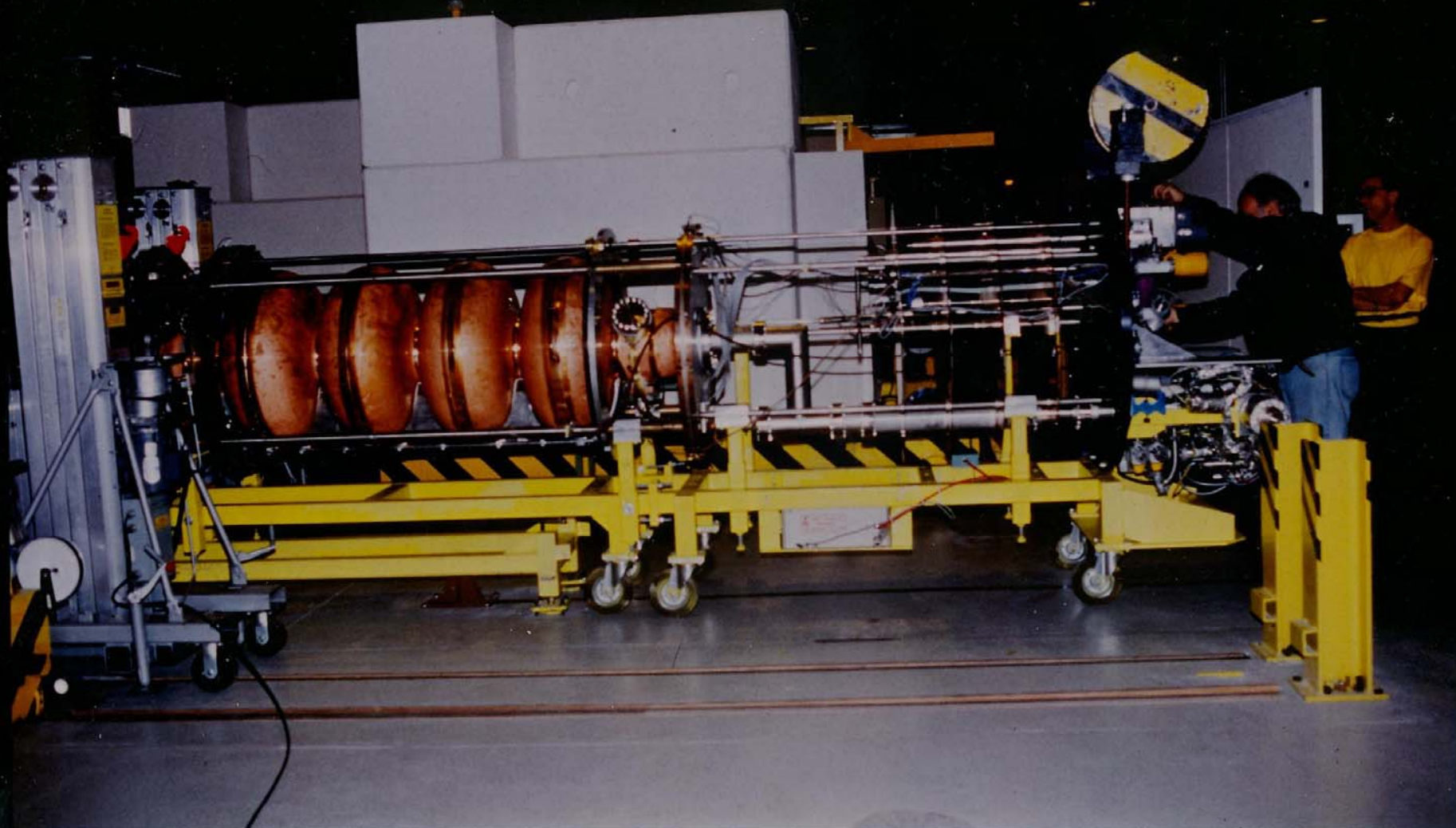


Cavity mass production cost breakdown





D. Bloess



IT 17-6.92/18



0739-7-88





AC 17-4-95/4

**Nb/Cu-cavities are of course cheaper, however, more difficult to produce:**

- EB-welding is delicate because of the high thermal conductivity more power than for bulk Nb is needed:

problems with thermal deformation,

holes in the weld,

porosities due to very fast cooldown of vortices in the liquid copper, and due to impurities on the surface

- Great care has to be taken to produce a smooth and clean surface without holes and inclusions:
  - Electrochemical polishing(EP) 60  $\mu\text{m}$
  - Inspection and careful grinding
  - EP 60  $\mu\text{m}$
  - Inspection and if necessary another correction
  - EB-welding
  - Inspection and grinding of welding beads
  - Chemical polishing (CP) 20-25  $\mu\text{m}$
  - Rinsing with high purity water in clean room
  - Drying with reagent grade ethanol in clean room

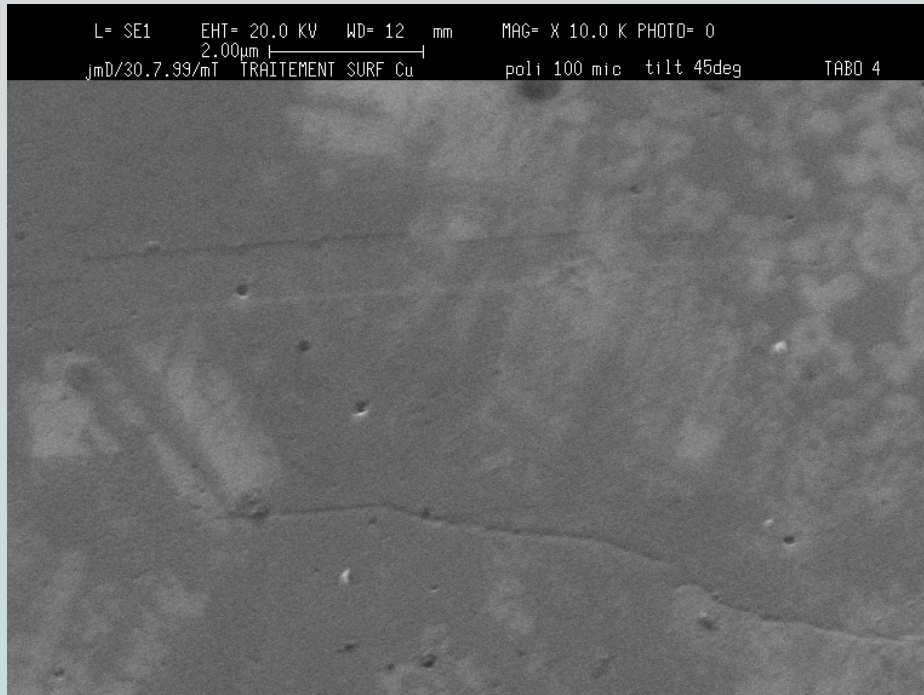
# Why films

S. Calatroni - CERN

- Advantages (primary objectives)
  - Thermal stability
  - Cost
  - Innovative materials
- 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)
  - Deposition of innovative materials is very difficult
  - Steep  $R_{res}$  increase with RF field (sputtered niobium films)

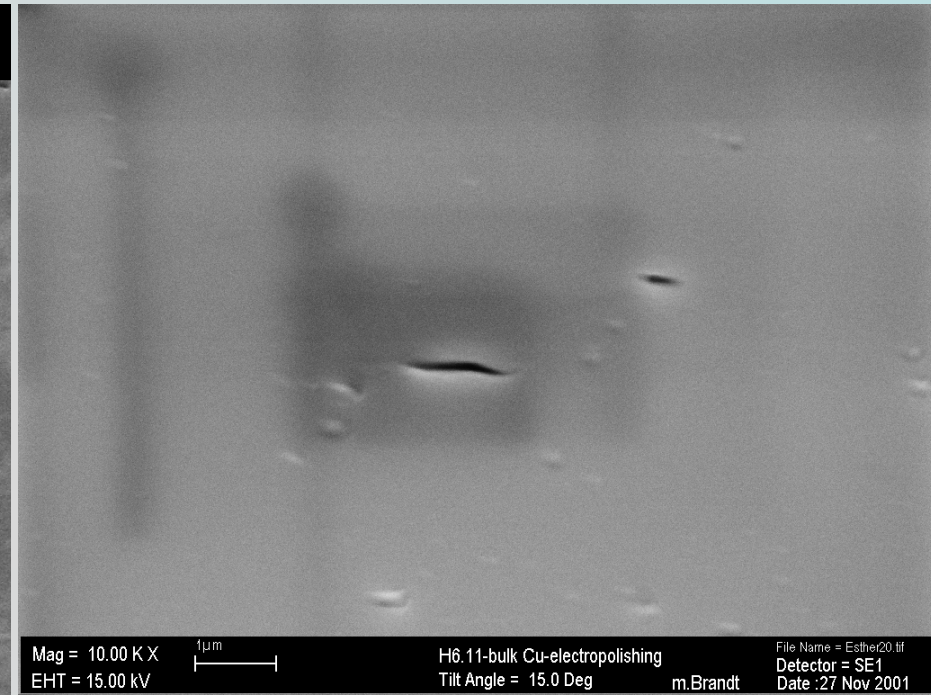


# Defects in Cu substrate



## Electropolished copper surface

- average roughness:  $0.02\text{ }\mu\text{m}$
- A few defects still appearing



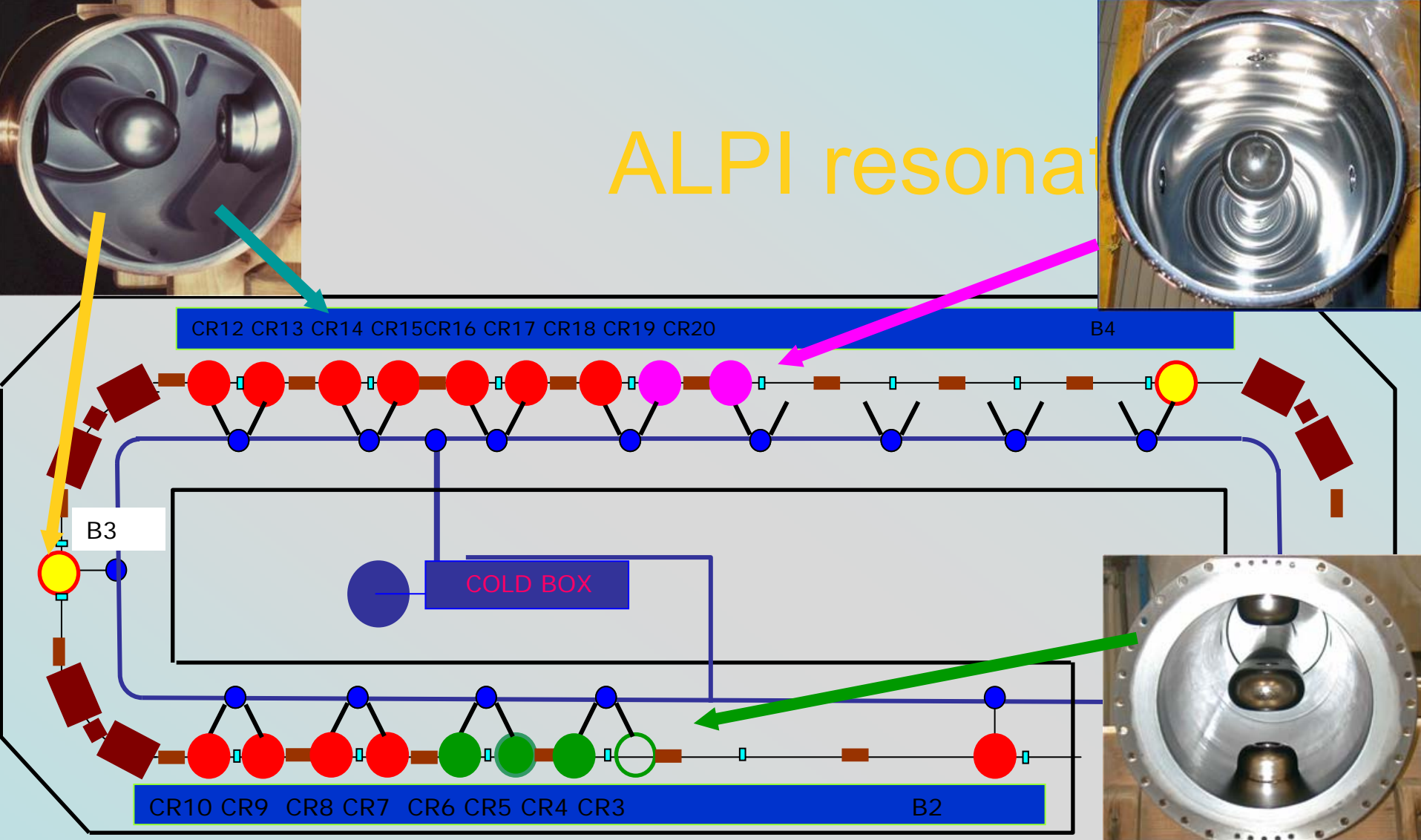
## Cross section of a copper cavity

- Defects are present inside !
- Not an artifact of the preparation

Thanks to: G. Arnau-Izquierdo



# ALPI resonator



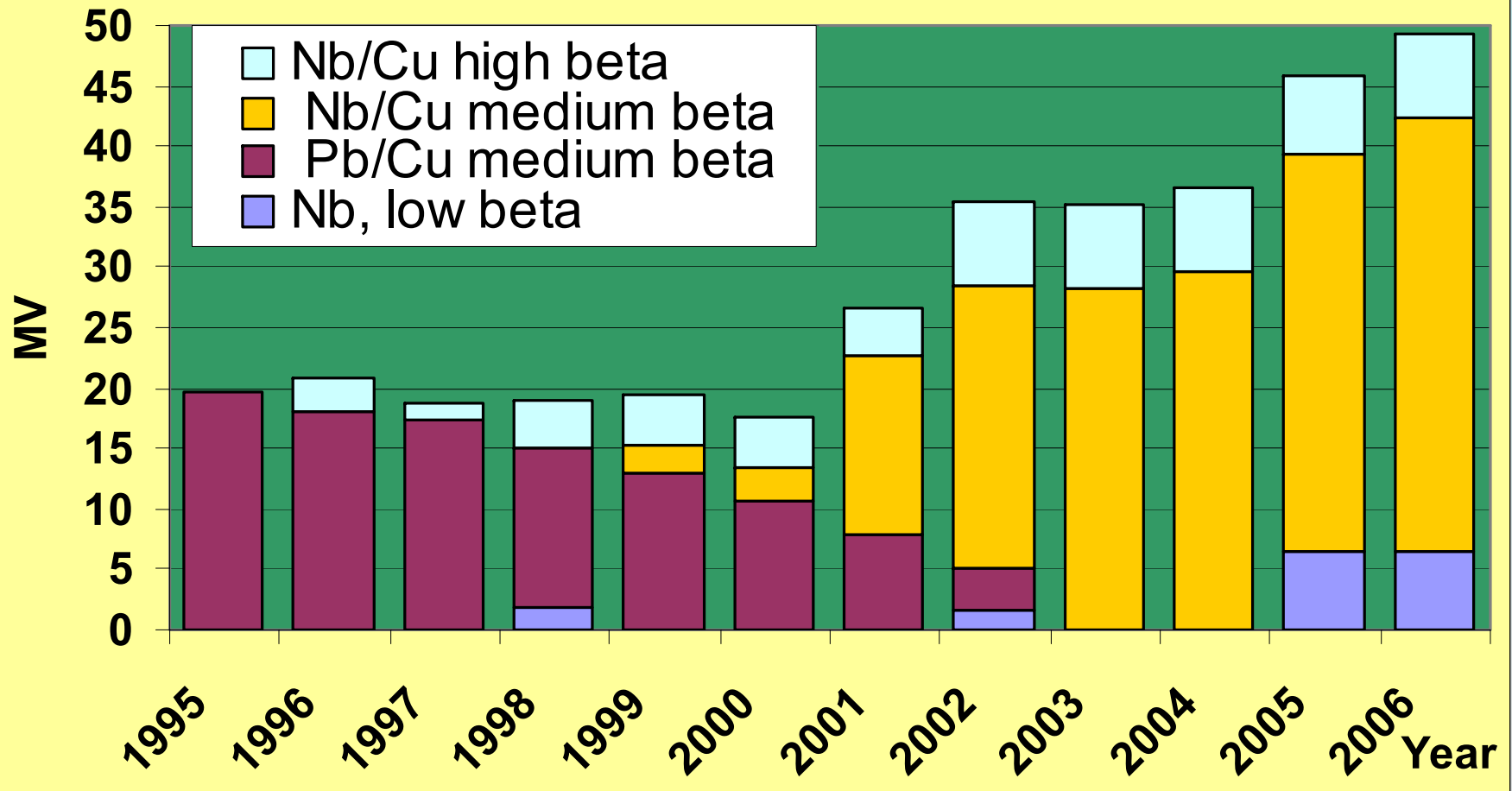
$\beta=0.056$ , 80 MHz, full Nb

$\beta=0.13$ , 160 MHz, Nb/Cu

$\beta=0.11$ , 160 MHz, Nb/Cu

$\beta=0.11$ , 160 MHz, Pb/Cu

## ALPI Equivalent voltage

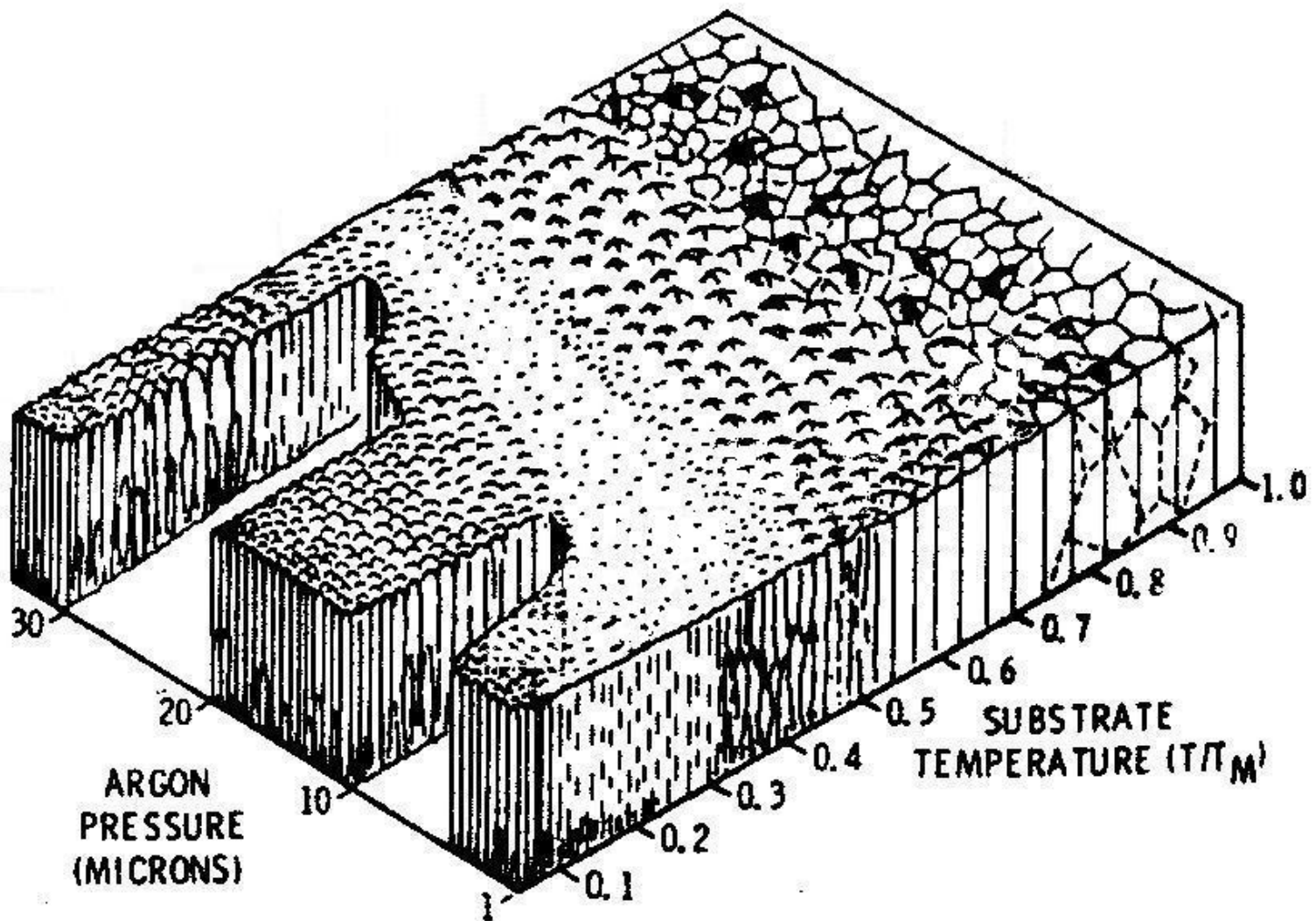


# QWR Sputtering development strategy

- ❑ 1987: Funding a research project on QWR Nb sputtering
- ❑ 1988-1999: DC biased sputtering choice and system set-up
- ❑ 1990: Obtaining good SC performance on samples
- ❑ 1991: Sputtering on a simplified prototype
- ❑ 1994: Design of a ALPI high  $\beta$  resonator suitable for sputtering production and compatible with existing cryostats
- ❑ 1995/1998 Production and installation of 4 high  $\beta$  cavities in ALPI

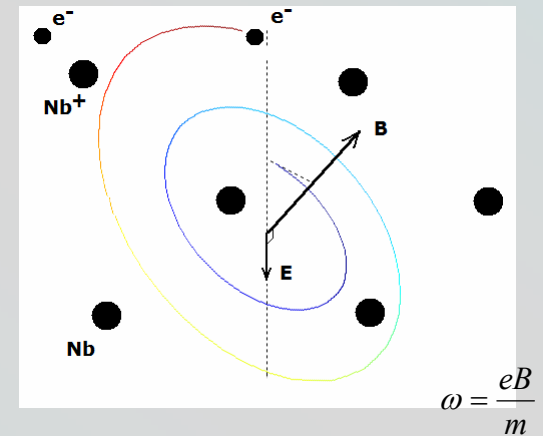
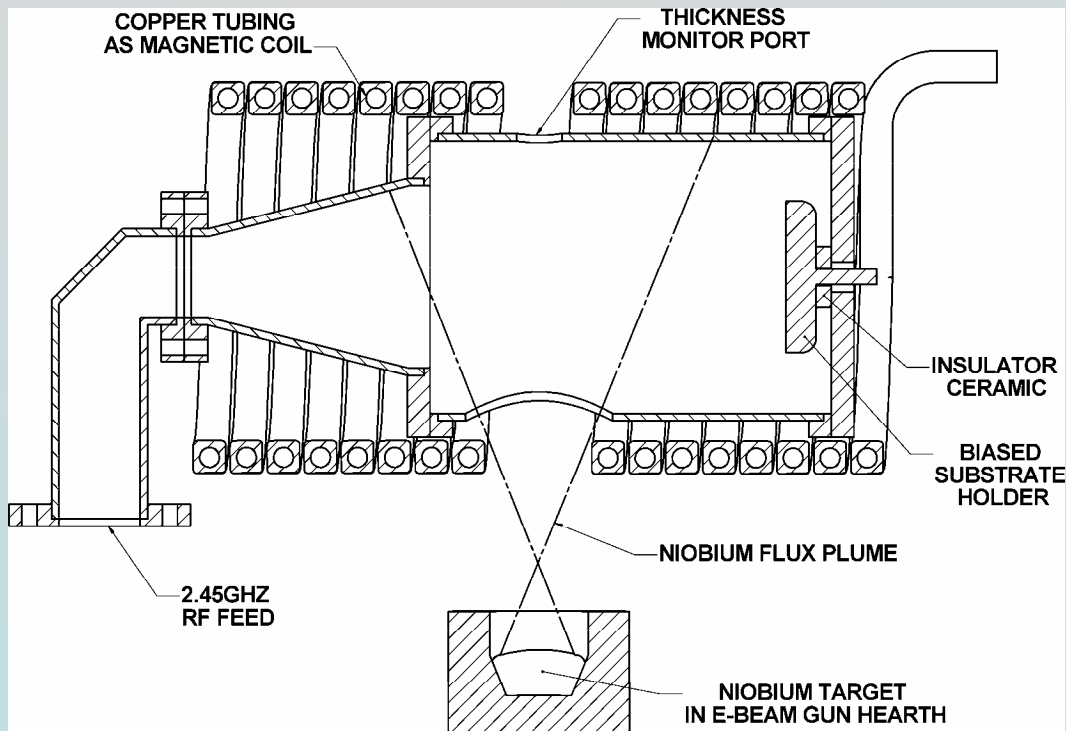
- ❑ 7W reached in laboratory
- ❑ 1998: Installation of four high  $\beta$  QWRs, operating at 6 MV/m @ 7W in ALPI
- ❑ 1998: First medium  $\beta$  ALPI QWR reaches 4 MV/m @ 7W
- ❑ 1999: The upgrading of medium  $\beta$  resonators begin
- ❑ 2003 : All the old 44 accelerating cavities have their Pb superconducting layer replaced by Nb

**The Thornton graph has in reality one more dimension:  
the energy of bombarding particles.**



Larry Phillips, Jlab

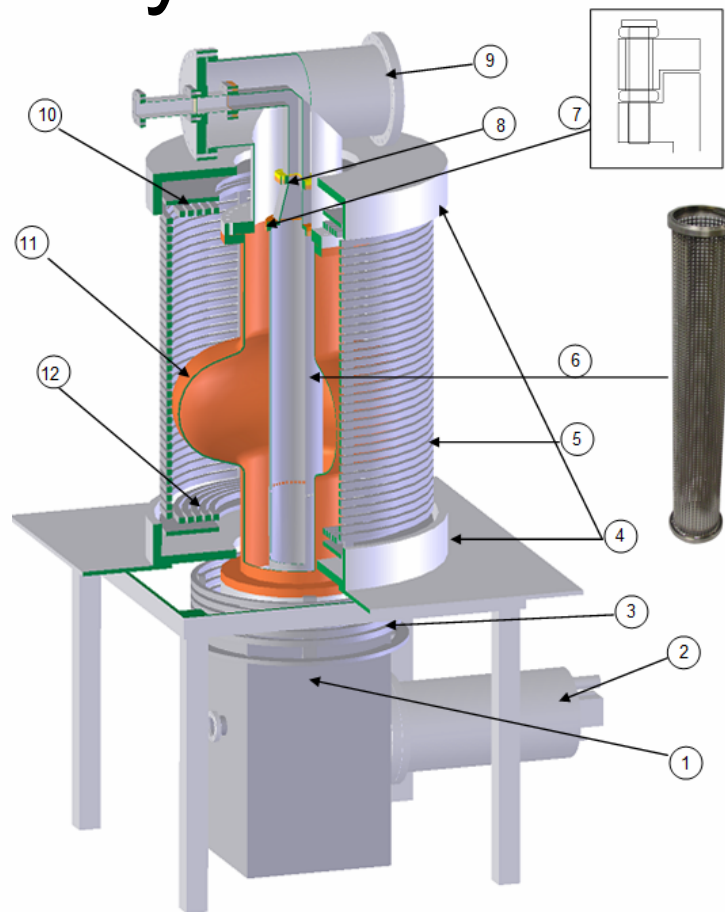
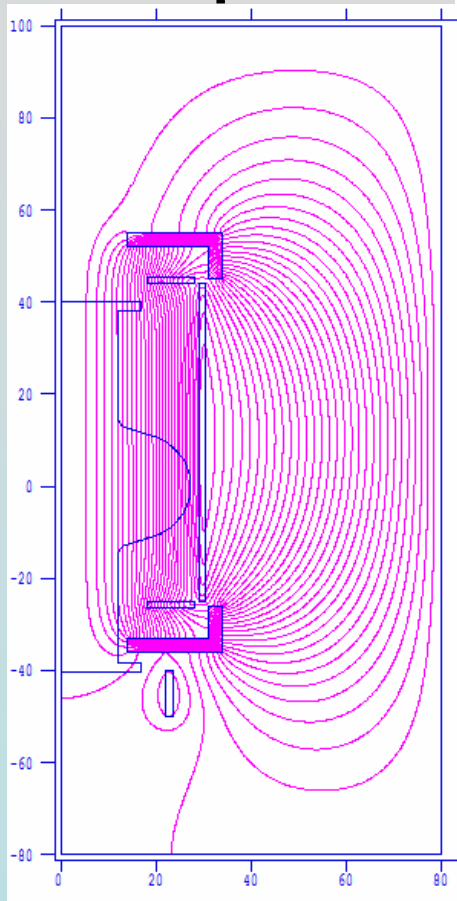
# Illustration of the energetic vacuum deposition by ECR plasma



Wu, G., et al., J. Vac. Sci. Technol. A Vol. 21, No. 4, (2003)



# ECR cavity deposition system



- (1) 14kW rod-fed E-gun
- (2) 9000 l/s cryopump system
- (3) bucking coil for E-gun
- (4) top and bottom iron yokes (outer iron shield is removed for illustration)
- (5) center coils
- (6) Nb grid tube
- (7) bias insulator
- (8) WR284 waveguide E-bend and horn to the grid tube
- (9) "T" vacuum chamber
- (10) top pancake coil
- (11) Cu cavity
- (12) bottom pancake coil.

Potential use for  
surface cleaning and  
 $\text{Nb}_3\text{Sn}$ , NbN coatings

**Genfa Wu**

Inside

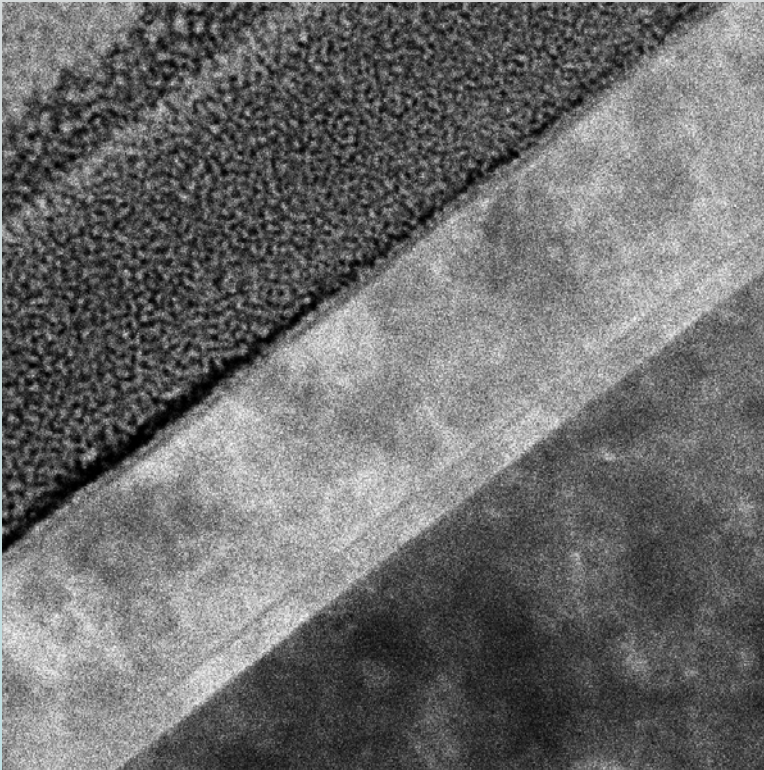


E-gun chamber



**Genfa Wu**

# A multilayer sample of Nb-NbO<sub>x</sub>-Nb

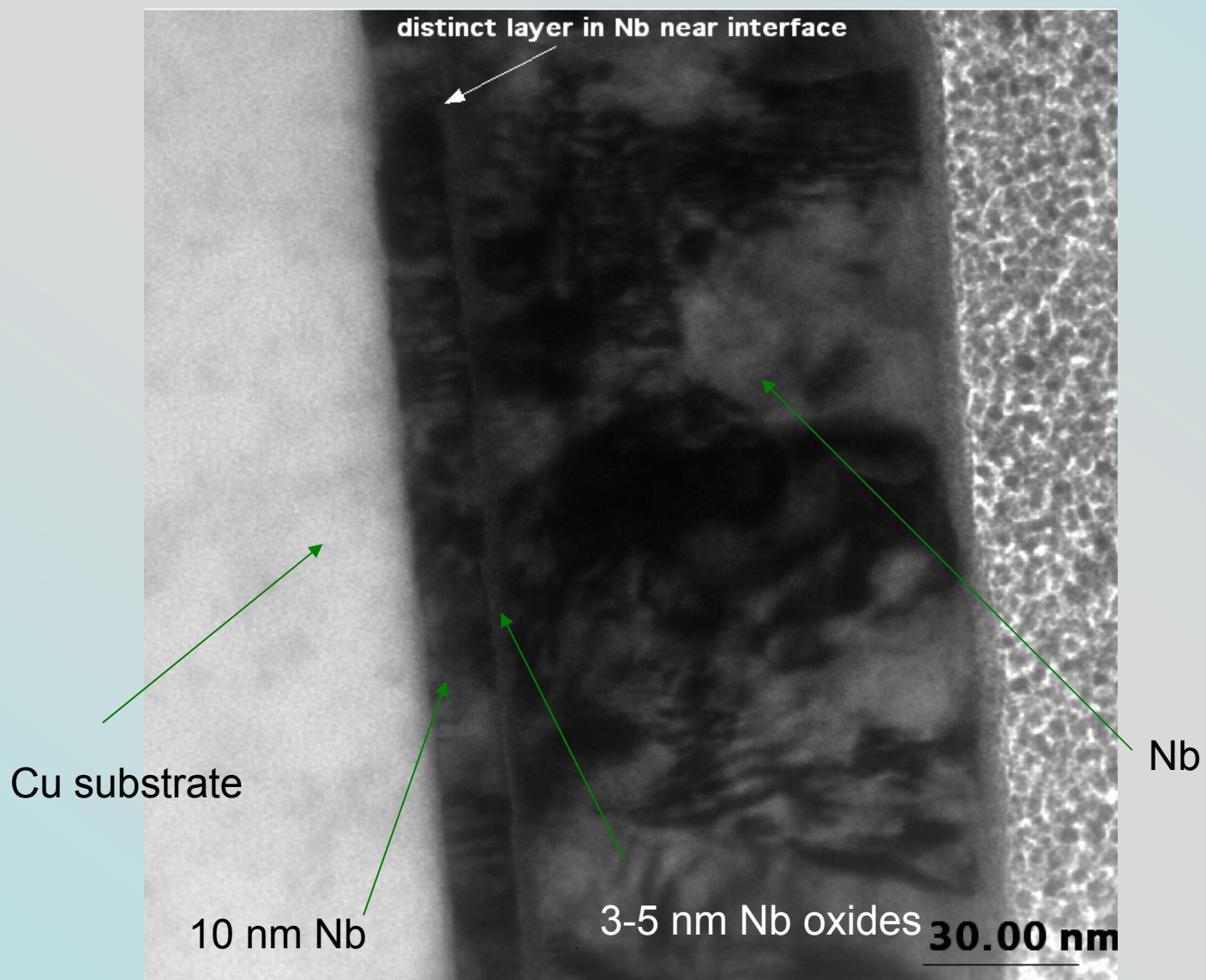


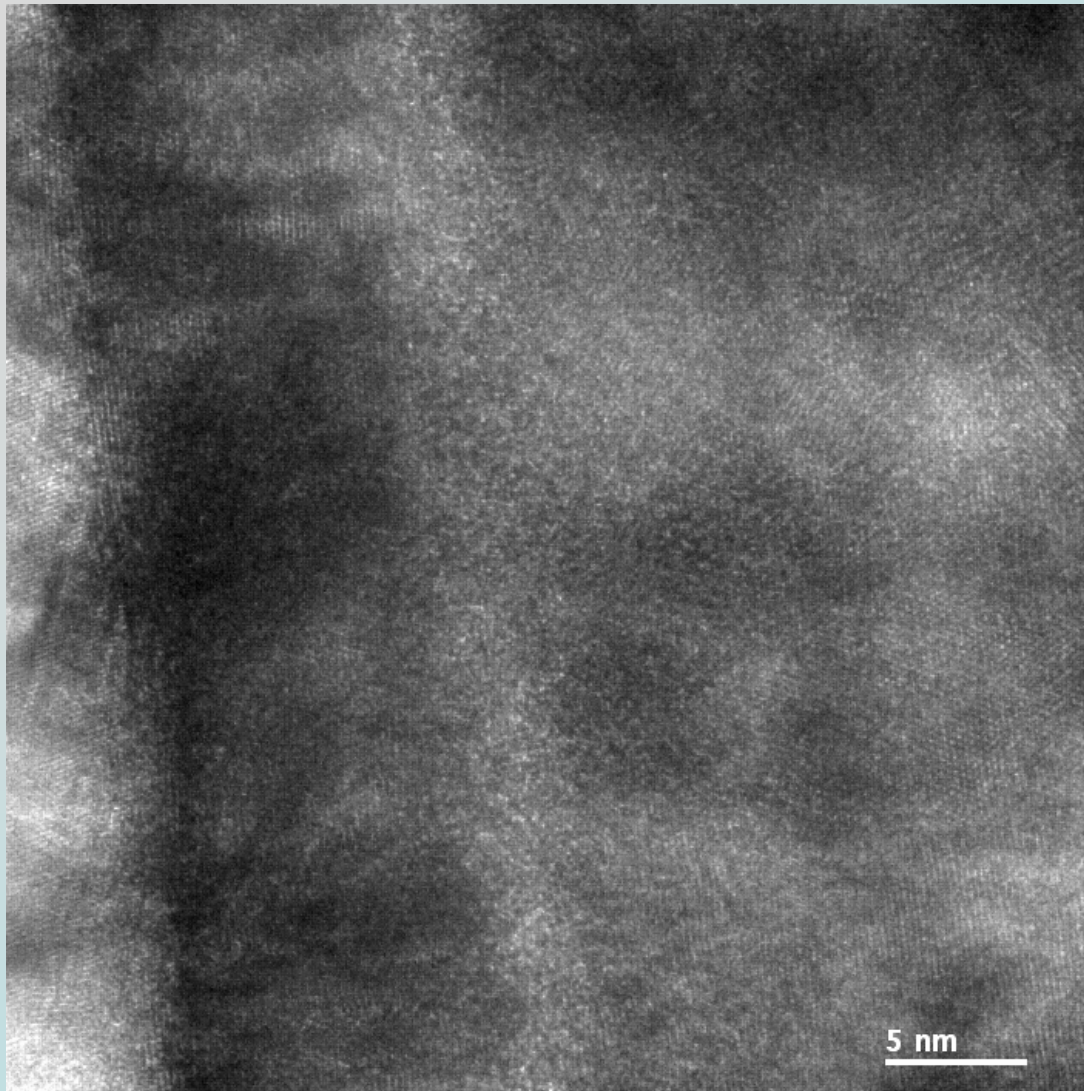
- Copper substrate was prepared by electropolishing.
- First layer of Nb was deposited at 500 V bias voltage.
- Exposed to air to form Nb oxides.
- Second layer (thicker) of Nb was deposited at 60 V bias voltage.
- TEM sample prepared (FIB) and analyzed at SANDIA

TEM by T. Renk, P. Provencio, SNL

**Genfa Wu**



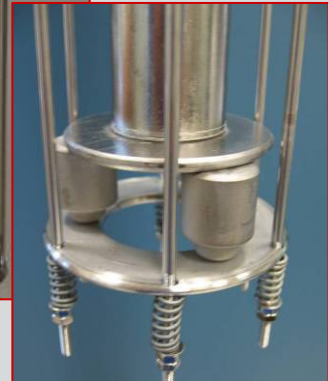




Zoomed view of the interface



# Biased Magnetron Sputtering: the construction



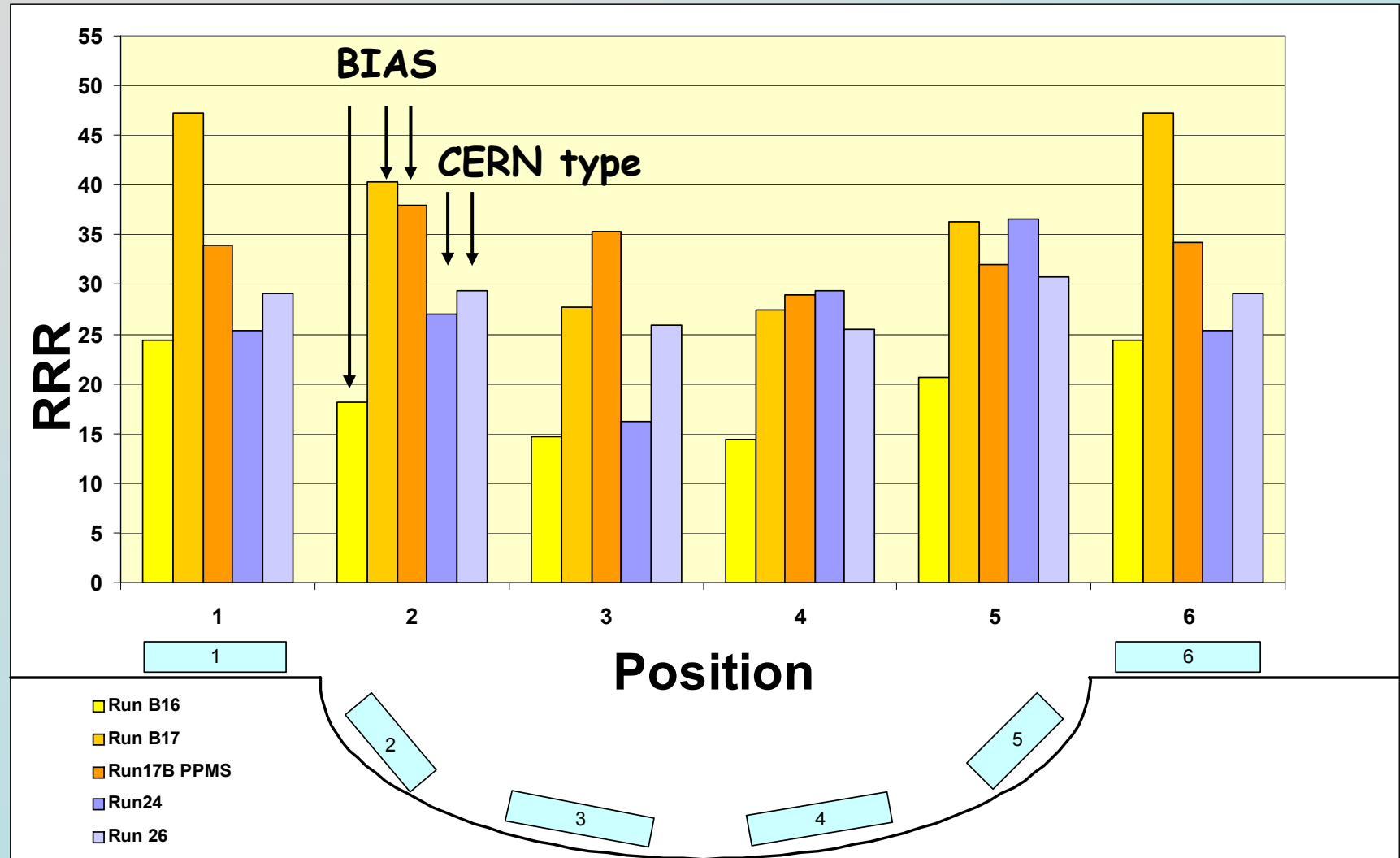
G. Lanza

# Biased Magnetron Sputtering: parameters

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	<b>CERN type</b>	<b>BIAS INFN-LNL</b>
<b>Cathode Current (A)</b>	<b>3</b>	<b>7</b>
<b>Cathode Power (kW)</b>	<b>1.38</b>	<b>1.86</b>
<b>Bias Voltage (V)</b>	<b>0</b>	<b>100</b>
<b>Pressure (mbar)</b>	<b><math>2 \times 10^{-3}</math></b>	<b><math>3 \times 10^{-3}</math></b>
<b>Time (min)</b>	<b>15</b>	<b>20</b>

# Biased Magnetron Sputtering: RRR results



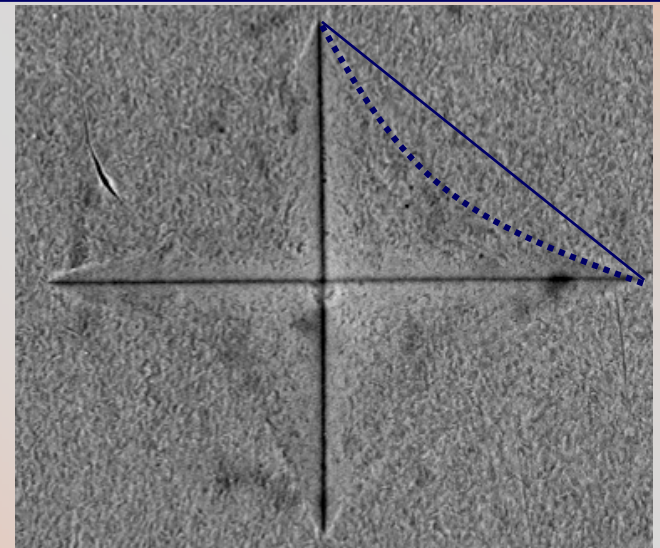
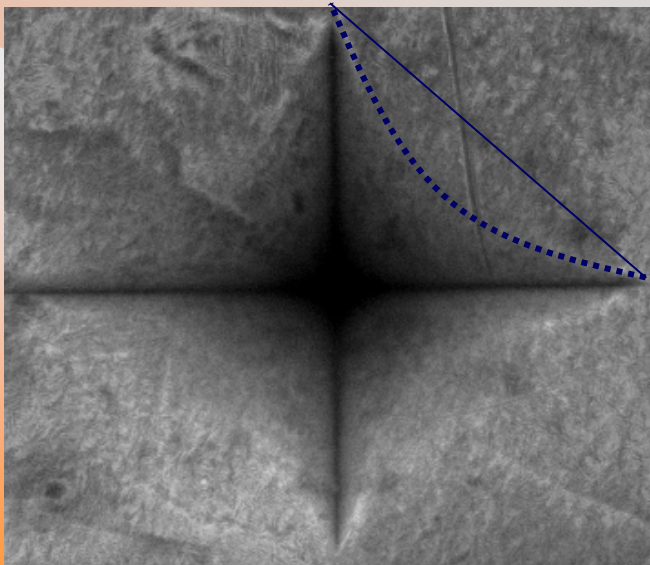
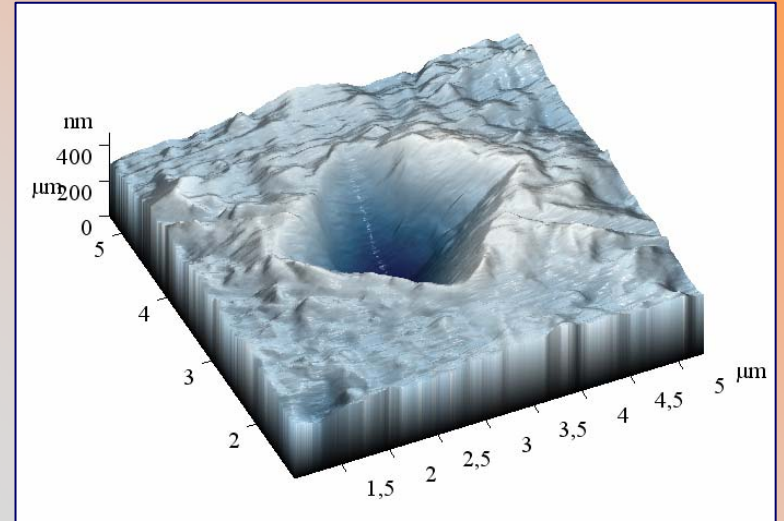
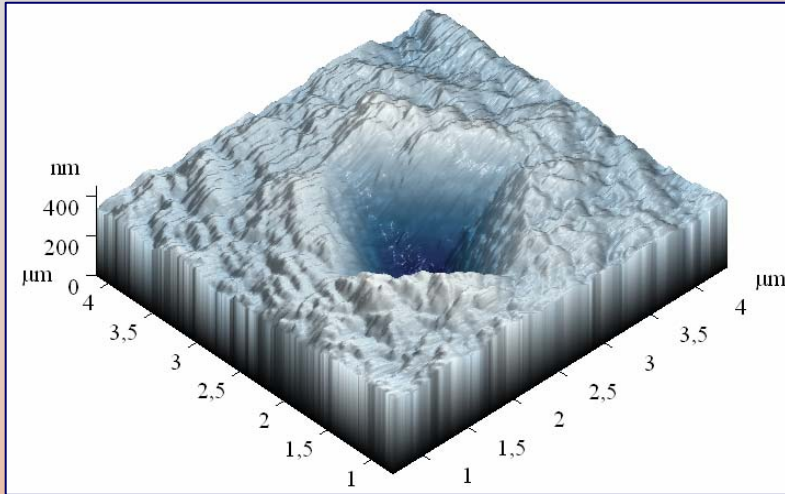
The grid still doesn't affect much the equator part



# Elastic modulus and ISE

Biased MS  $E = 88,95$  GPa

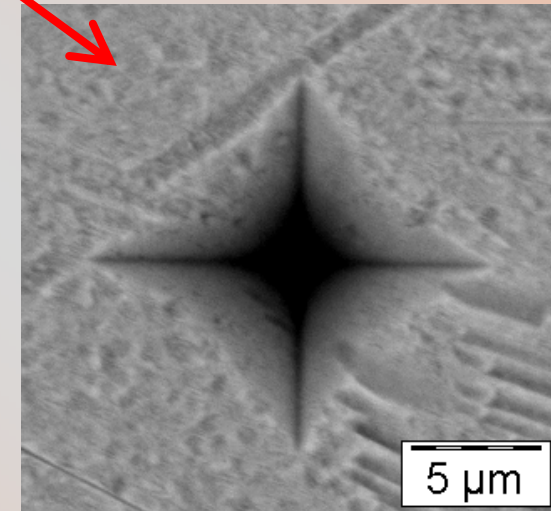
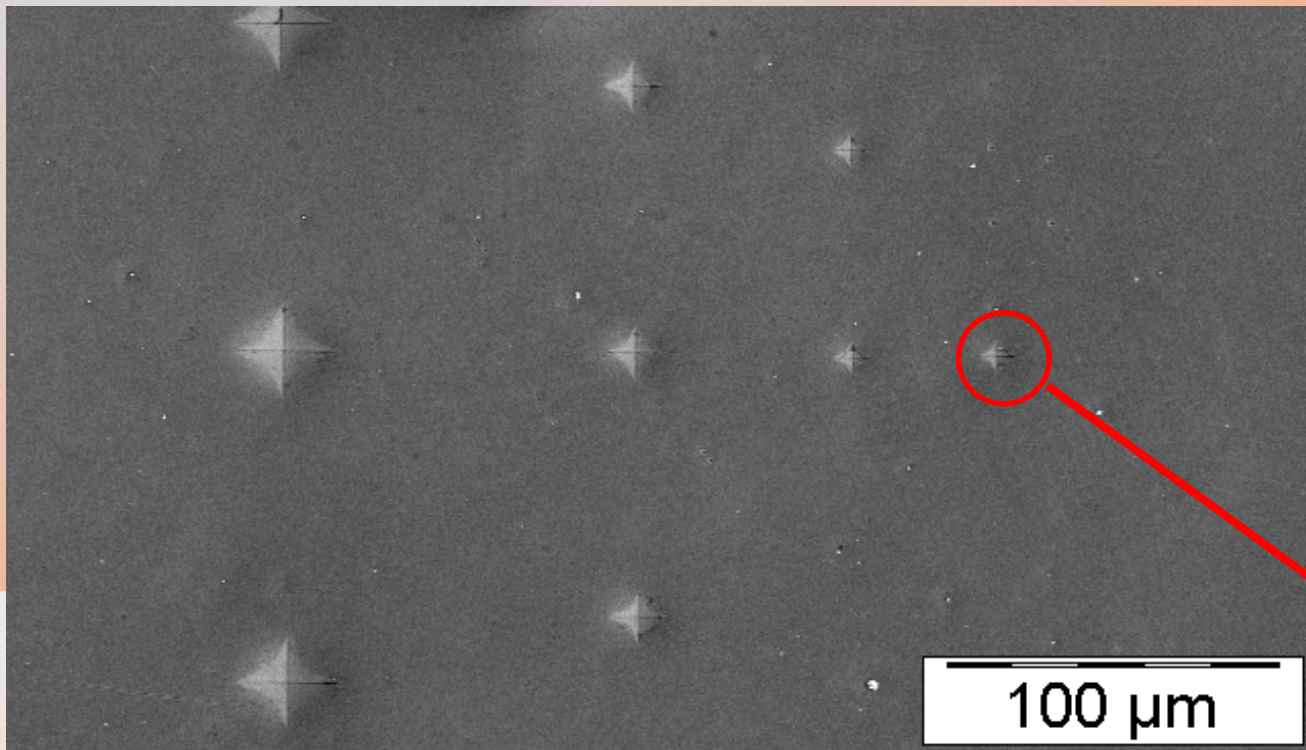
unbiased MS  $E = 54,33$  GPa







# Vickers indentation at different loads



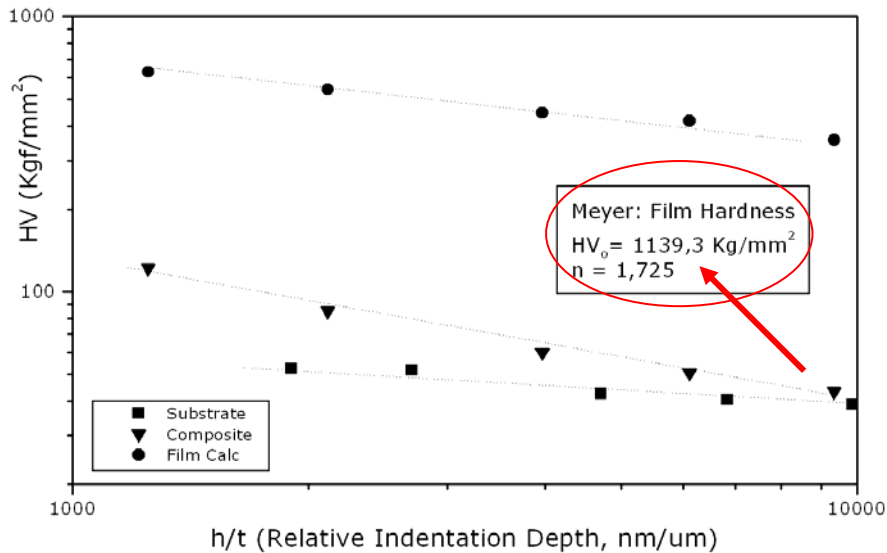




# Hardness comparison

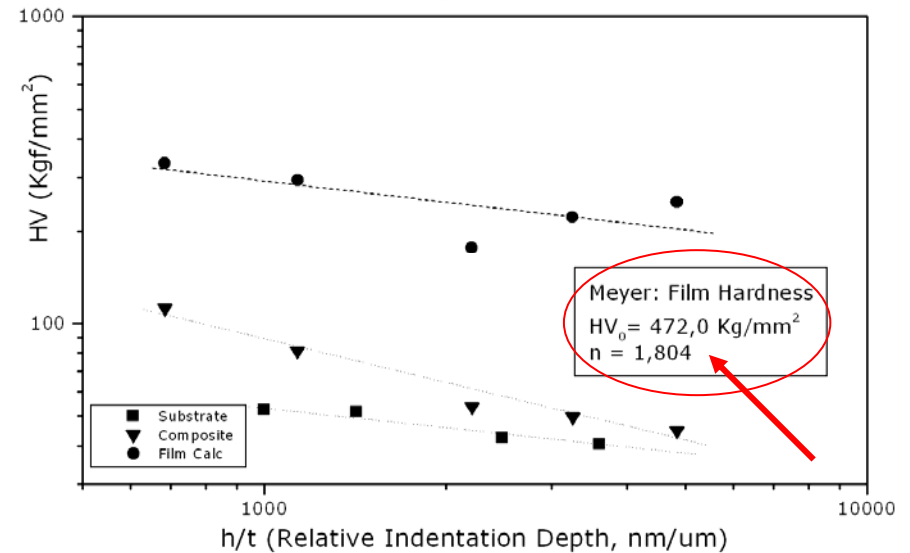
## biased MS (#YY-767)

Jonsson - Hogmark #767 ( $t = 1 \mu\text{m}$ )

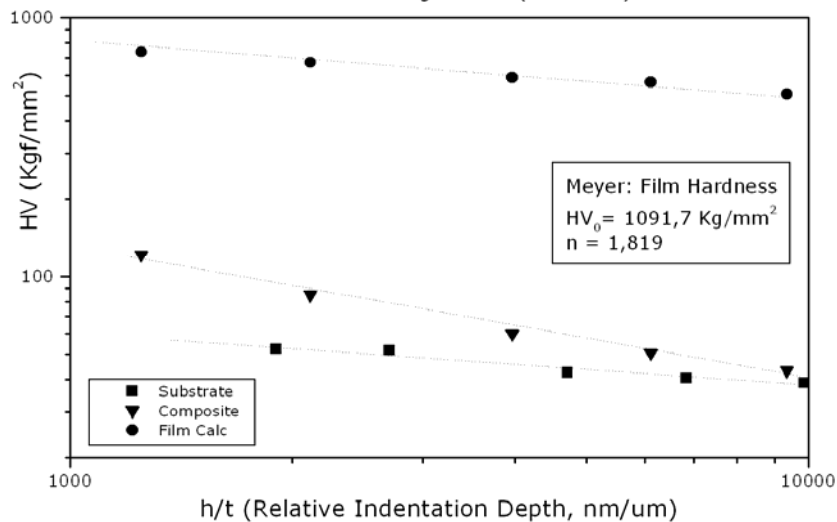


## unbiased MS (#12-766)

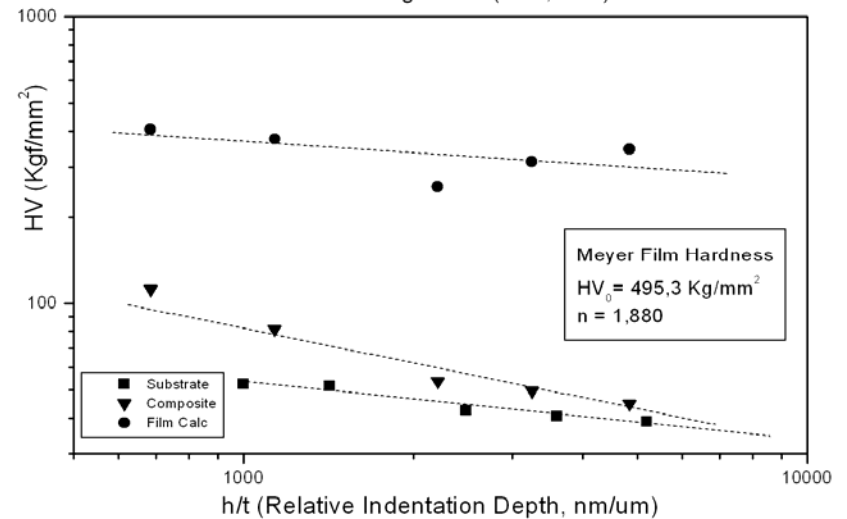
Jonsson-Hogmark #766 ( $t = 1,9 \mu\text{m}$ )



Chicot - Lesage #767 ( $t = 1 \mu\text{m}$ )

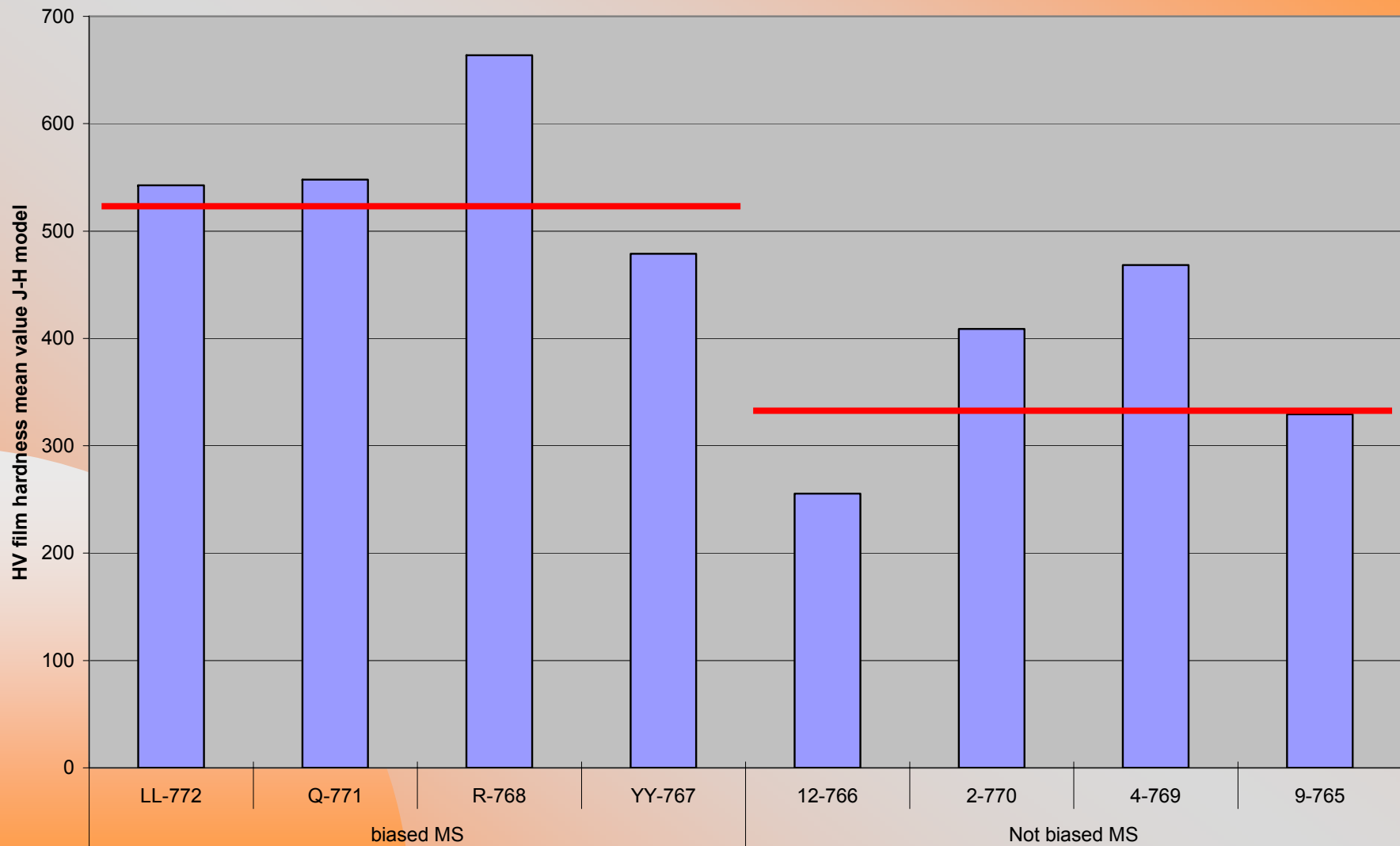


Chicot-Lesage #766 ( $t = 1,9 \mu\text{m}$ )



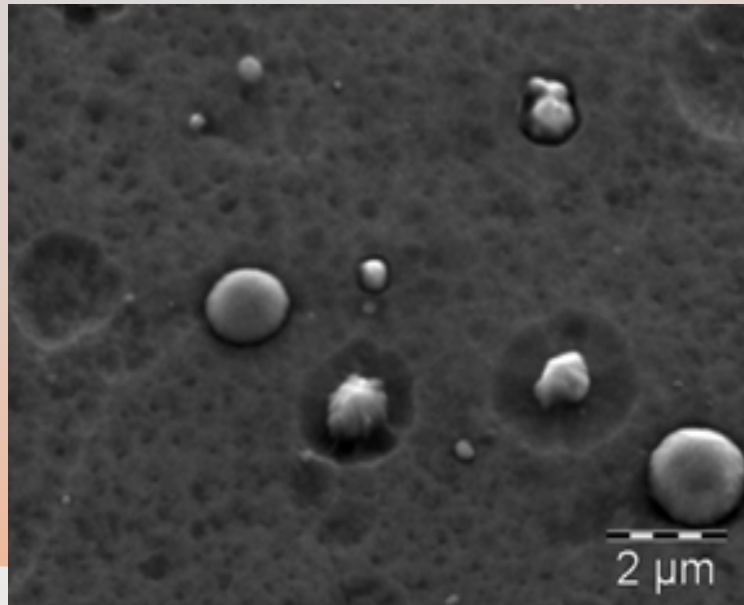


# Hardness comparison

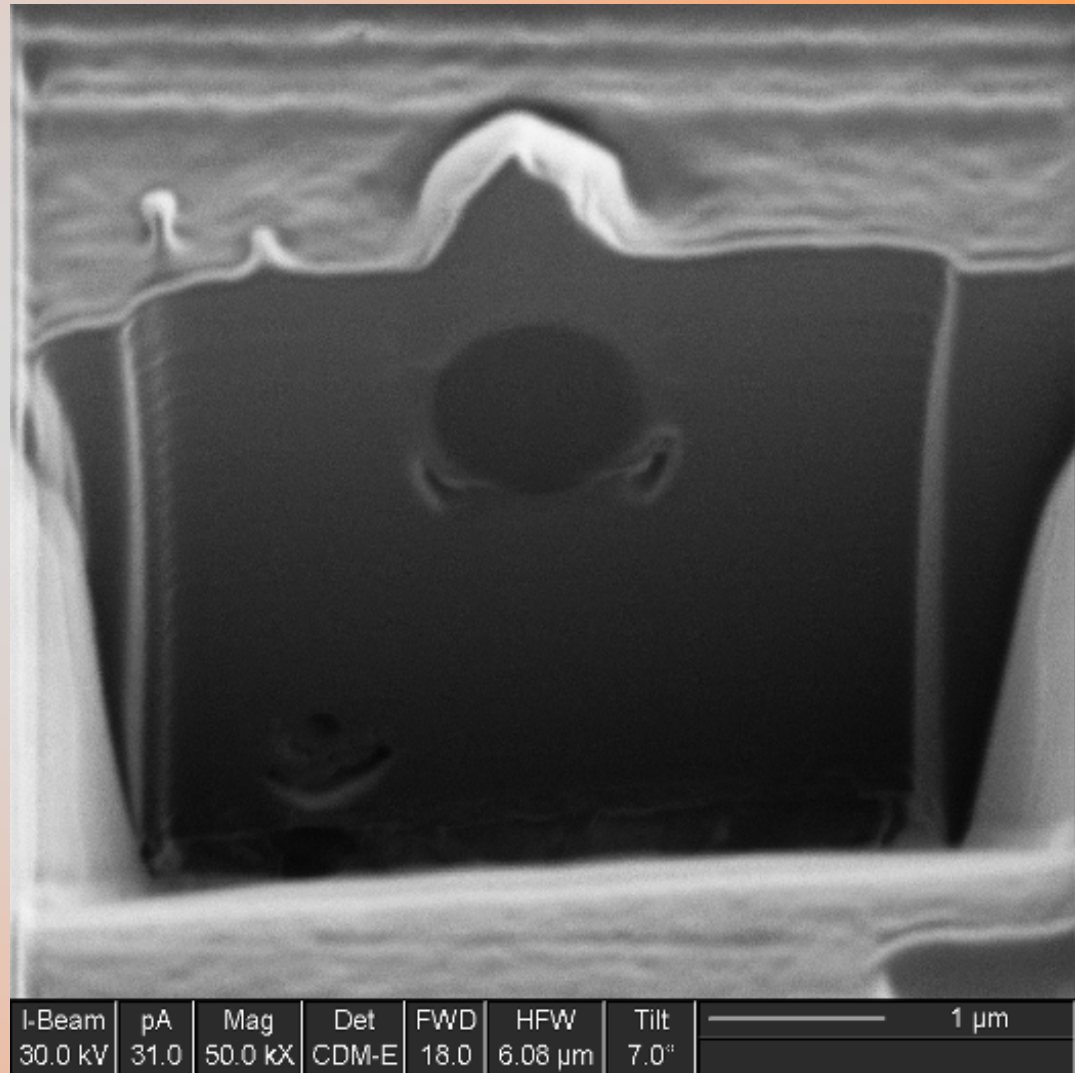




# Surface defects: FIB

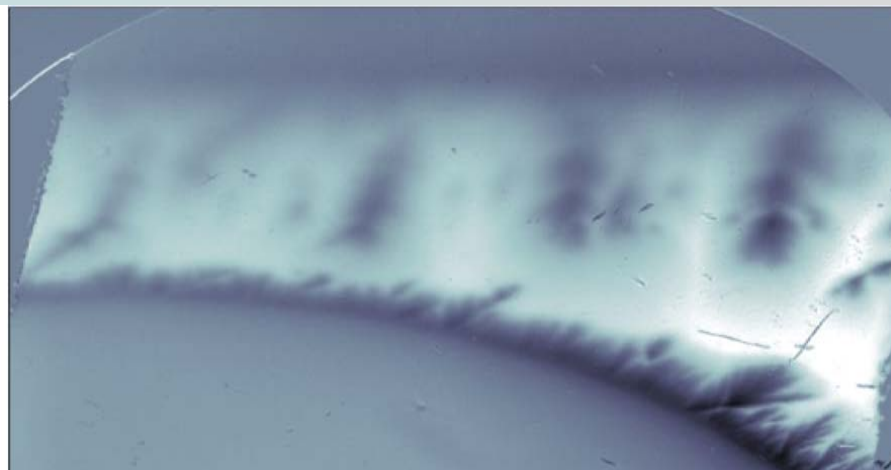
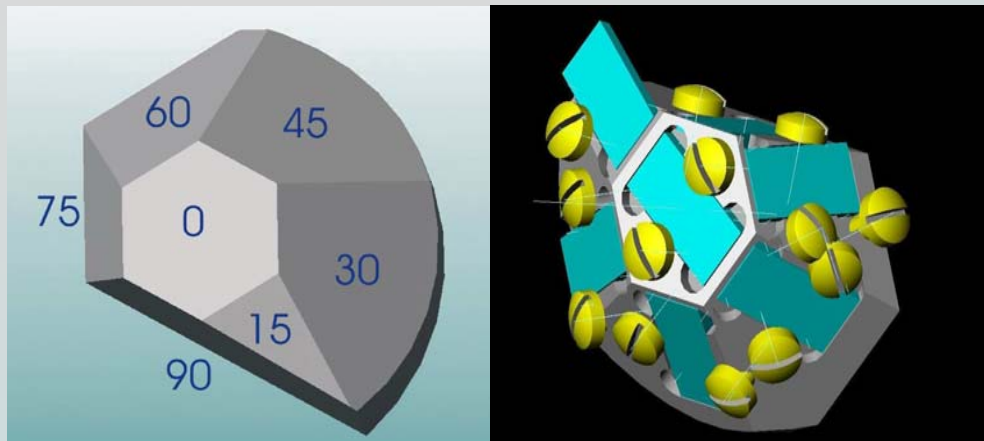


(25 kV, 8.000 x, SEM  
SE detector)

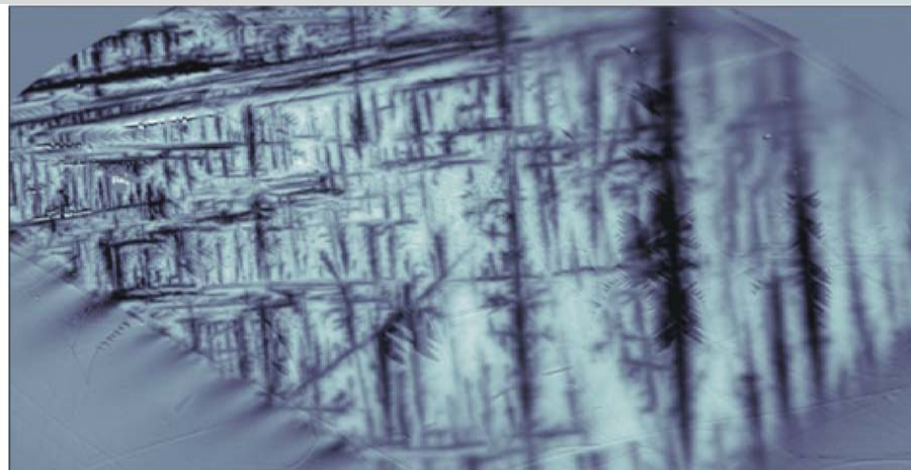


# MORPHOLOGY OF NIOBIUM FILMS SPUTTERED AT DIFFERENT TARGET – SUBSTRATE ANGLE

D. Tonini, C. Greggio, G. Keppel, F. Laviano, M. Musiani, G. Torzo, V. Palmieri



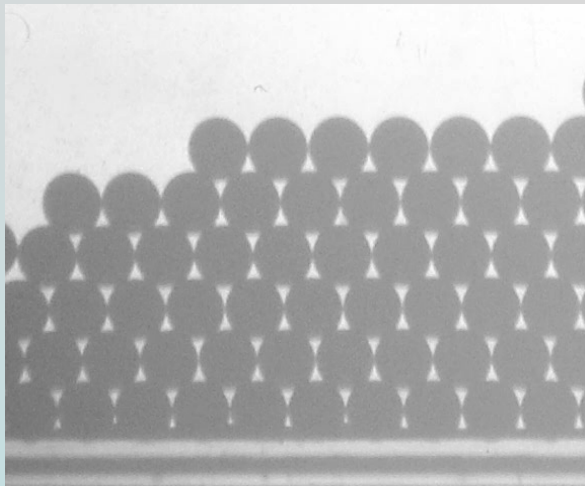
**Substrate PARALLEL to the target**



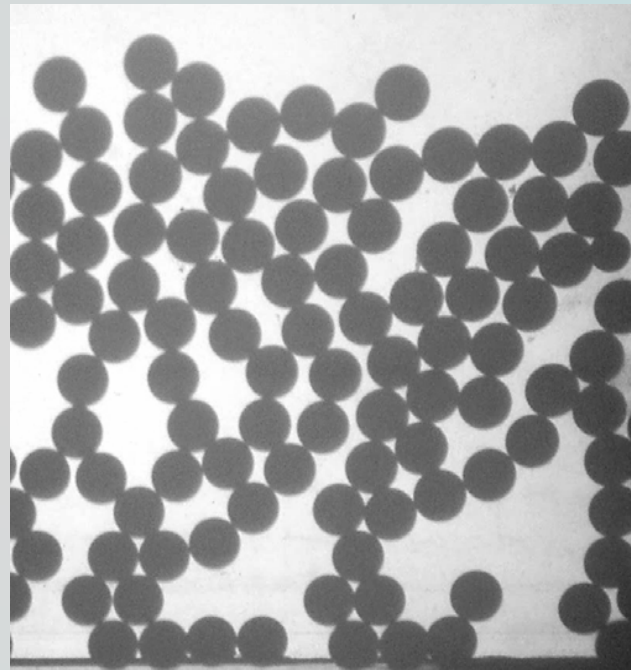
**Substrate at 45 degrees from the target**



## Simulation of film growth



0 degrees

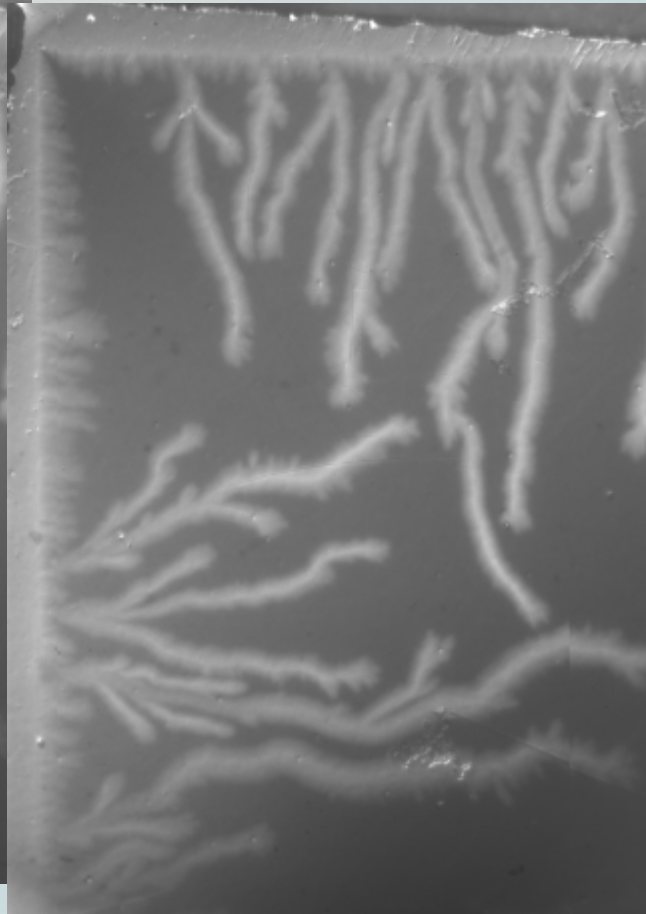


45 degrees

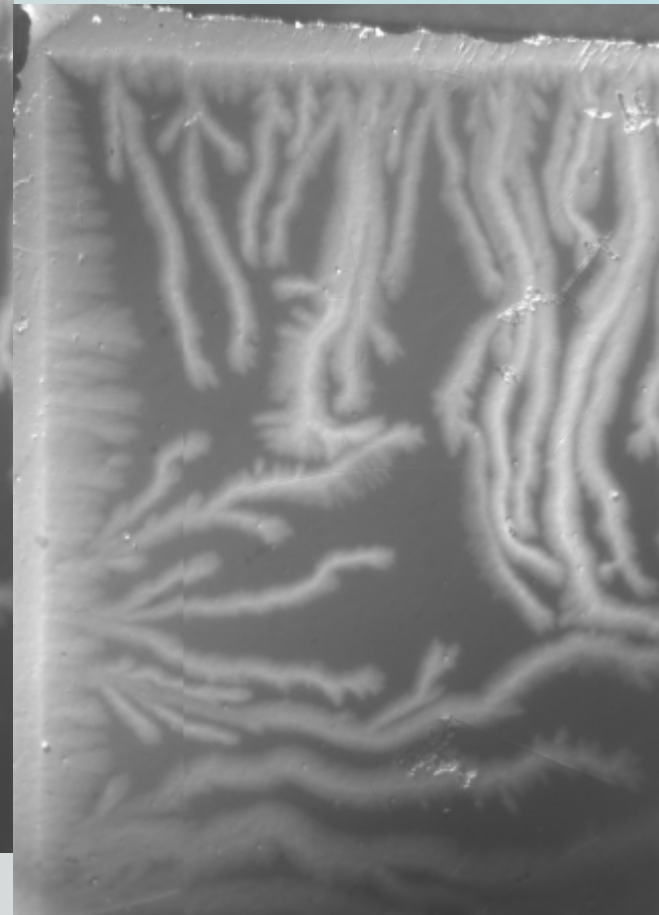
MgB<sub>2</sub> film ID # MO 6006 from Brian Moeckly. Alumina substrate. ZFC T=5.3K. Dendritic flux instability



#15 H=13.2 mT



#19 H=20 mT

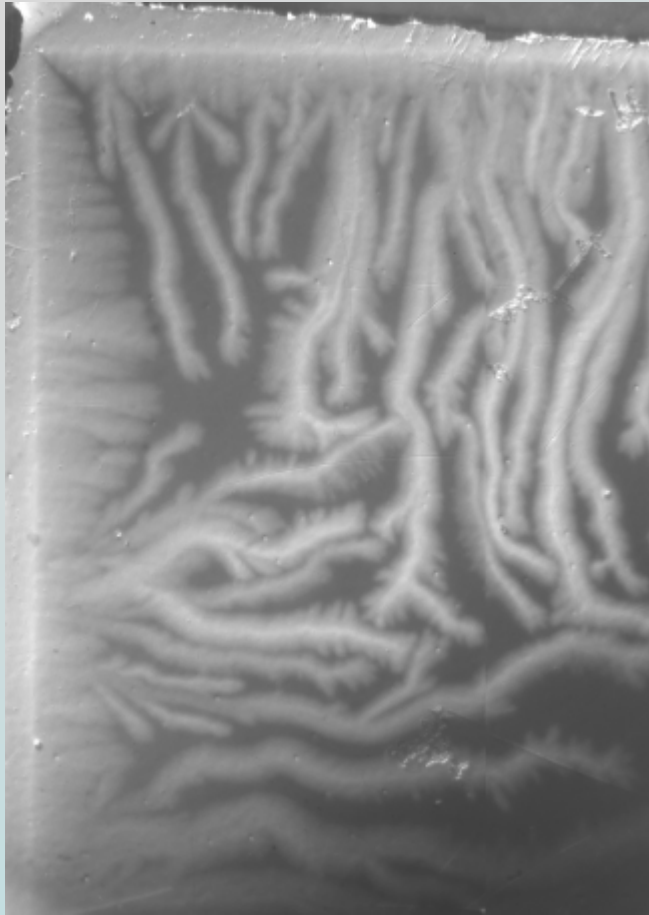


#23 H=32 mT

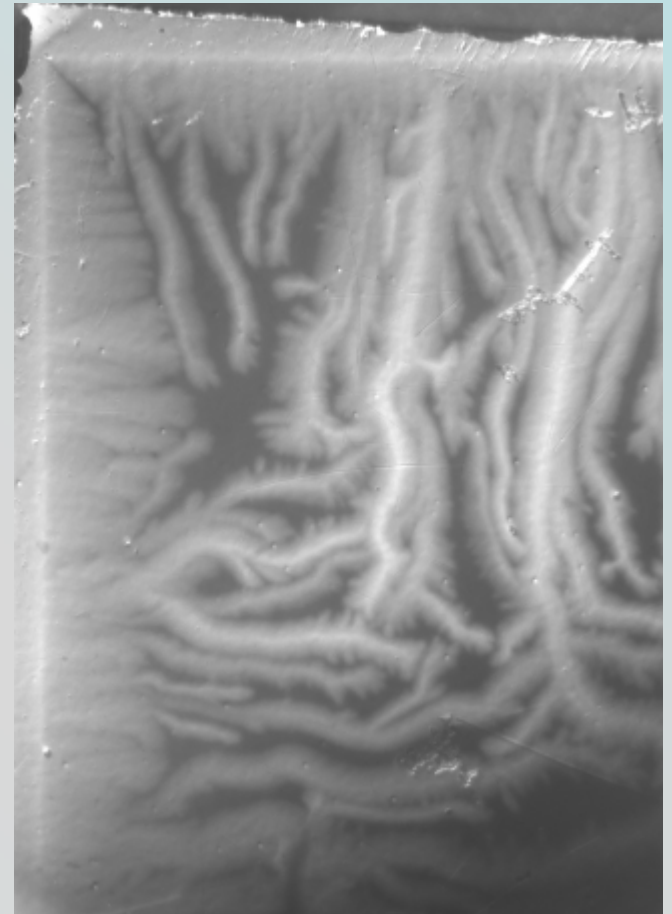
A. Polyanskii



MgB<sub>2</sub> film ID # MO 6006 from Brian Moeckly. Alumina substrate. ZFC T=5.3K Dendritic flux instability



#25 H=40 mT

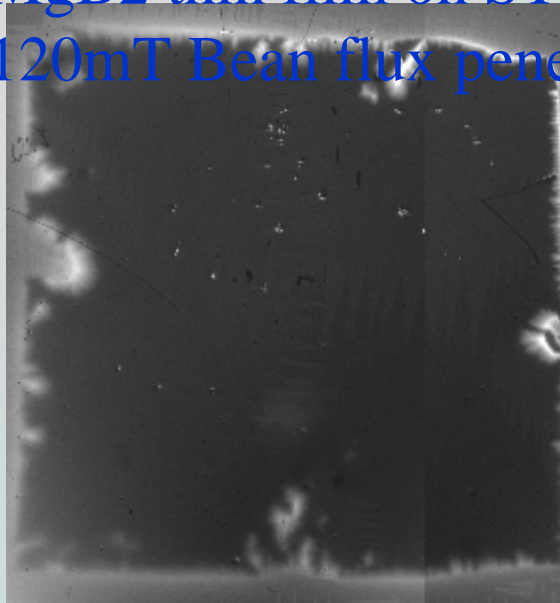


#27 H=48 mT

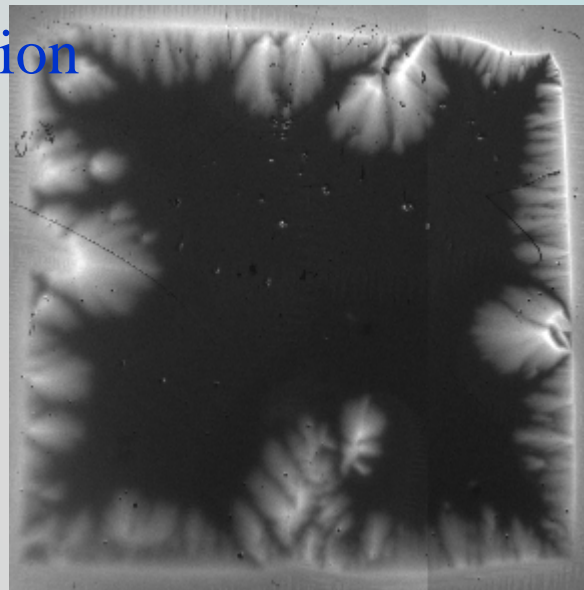
A. Polyanskii



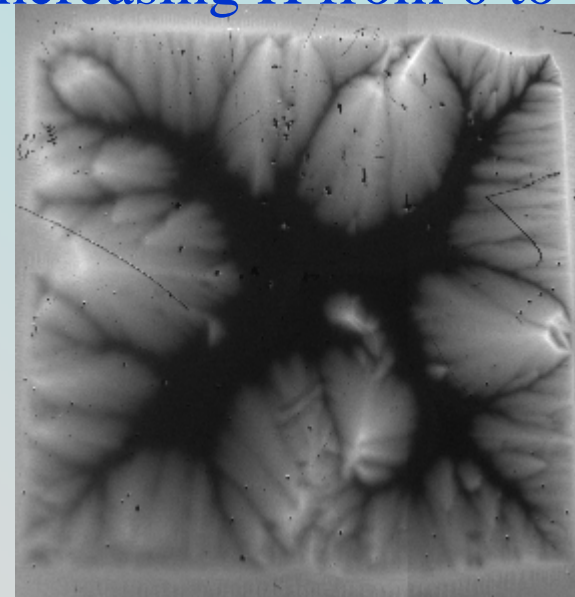
MgB2 thin film on STOCBS 33-1-5 ZFC T=16K increasing H from 0 to 120mT Bean flux penetration



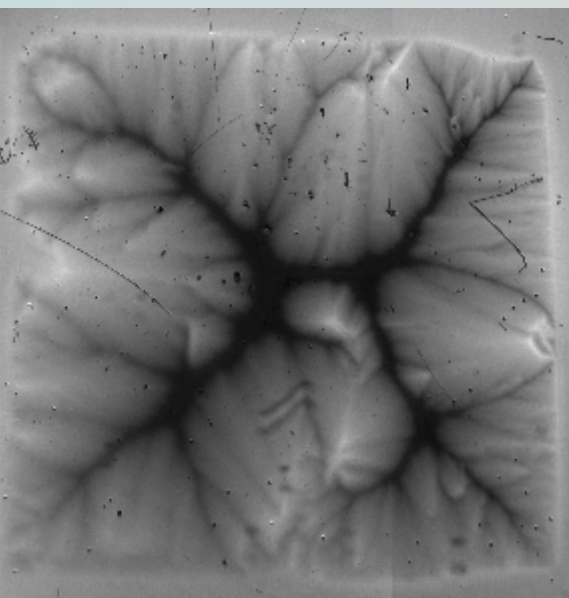
#1 H=8mT



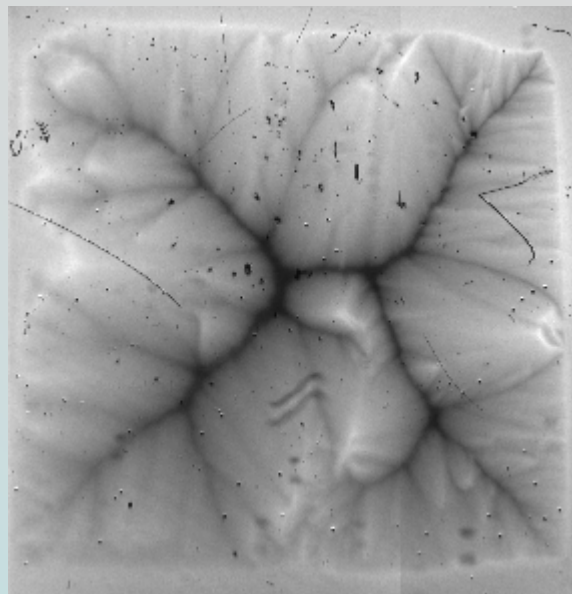
#5 H=20mT



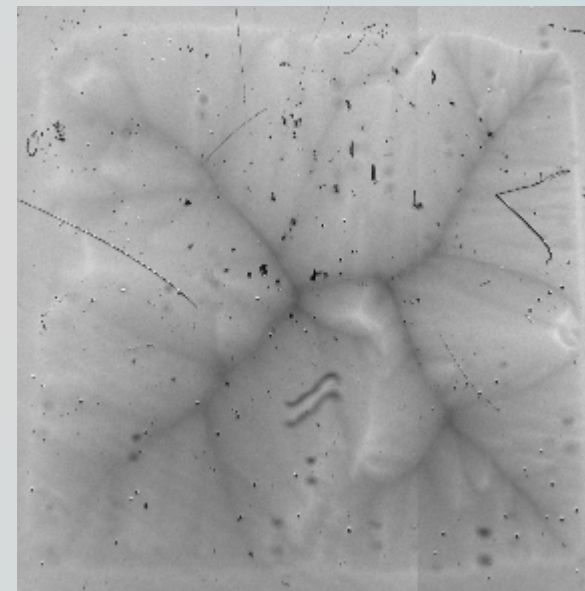
#9 H=40mT



#11 H=60mT



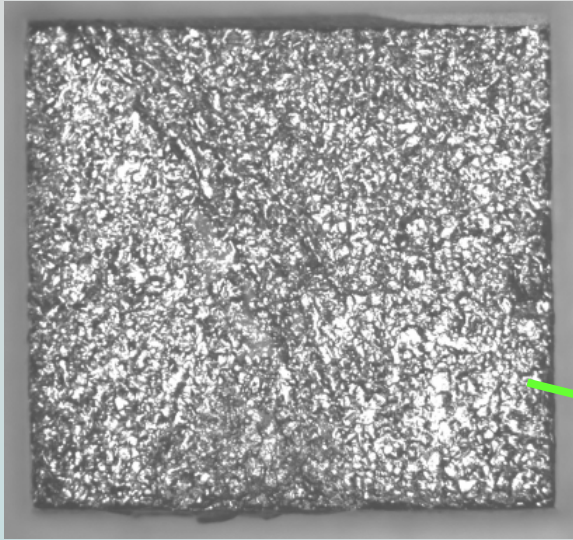
#13 H=80mT



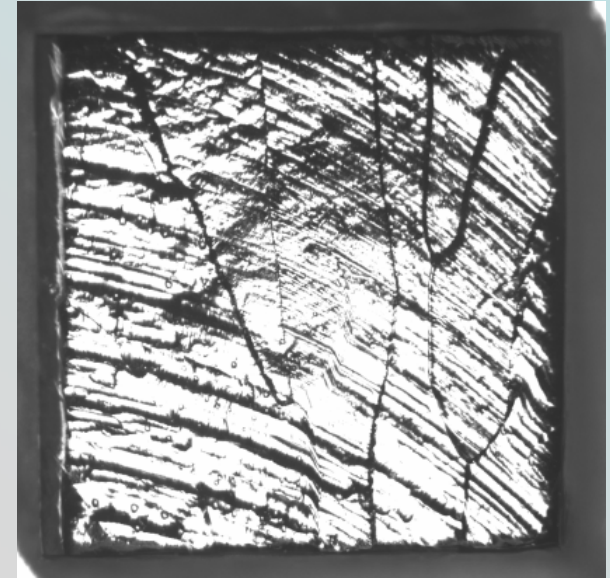
#17 H=120mT 1 mm



# OPTICAL IMAGES OF REGULAR AND WELD SAMPLES



REGULAR AREA: small grain size

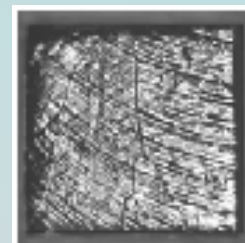
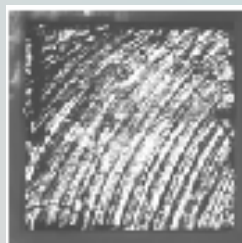
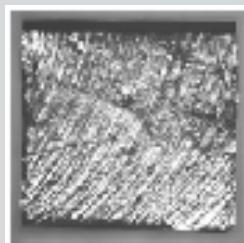
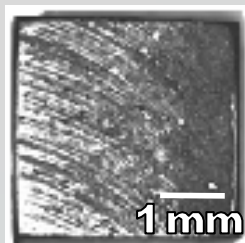


WELDED AREA: large grain size  
Machine marks (like grooves), large grains and height steps at GBs are well visible

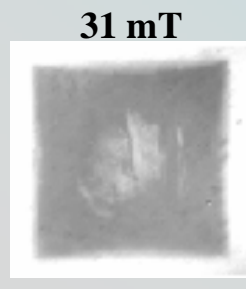
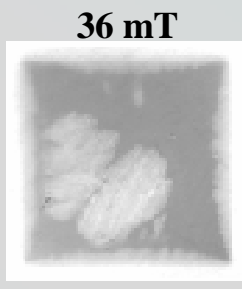
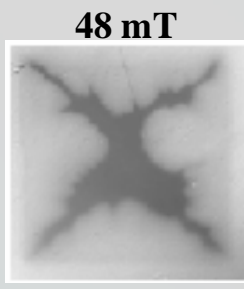
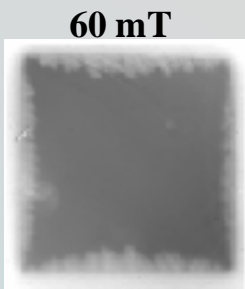
# MOI of Weld samples with big grains taken through a typical cavity optimization process: 5 steps

CW, degrease → 100  $\mu\text{m}$  etch → 5 hr/750°C → 20  $\mu\text{m}$  etch → 50 hr/120°C

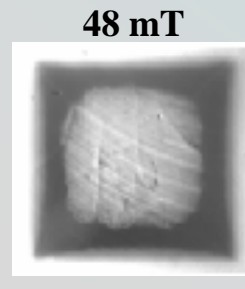
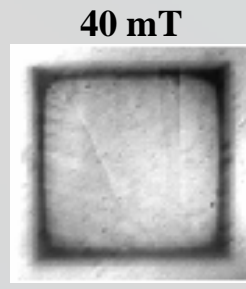
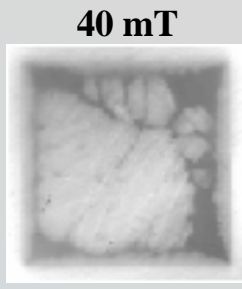
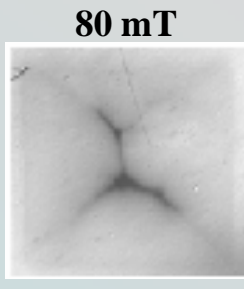
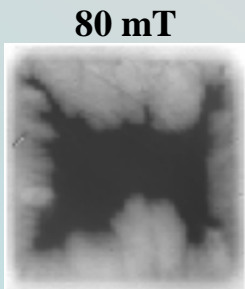
Optical



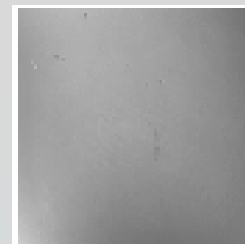
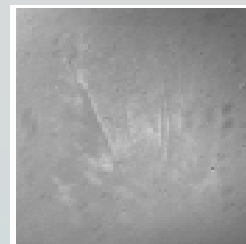
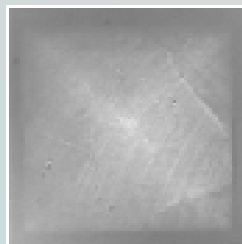
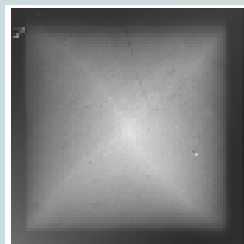
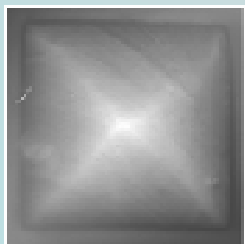
ZFC  
Then  $H_{\text{app}}$




ZFC  
Then  $H_{\text{app}}$

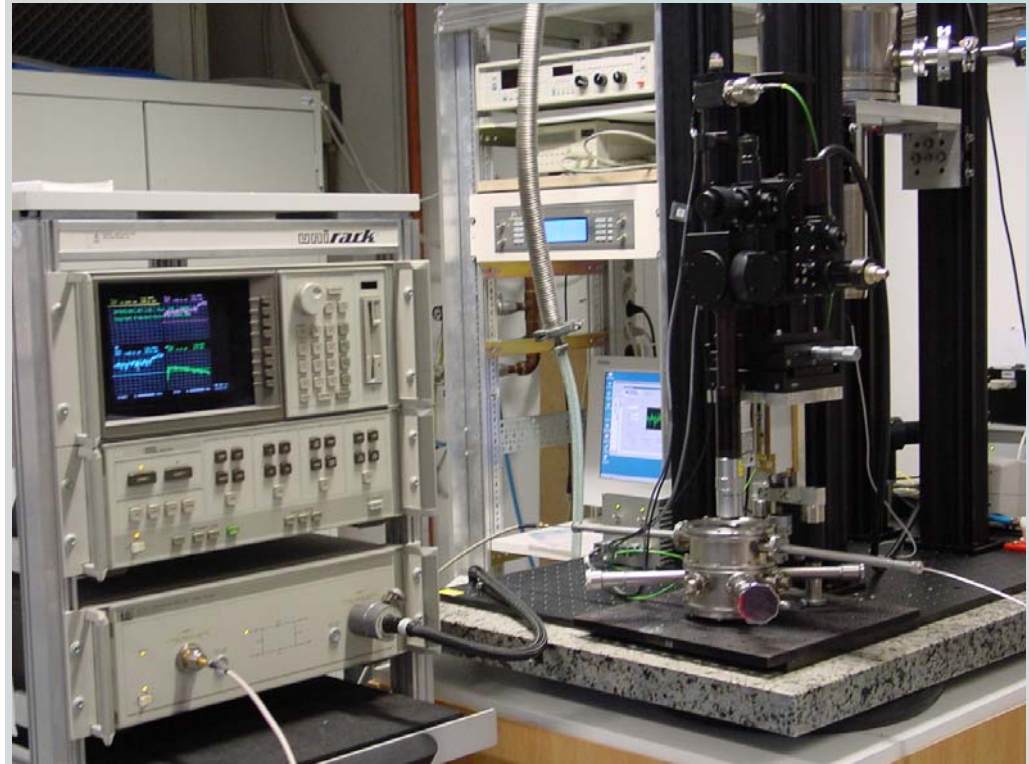
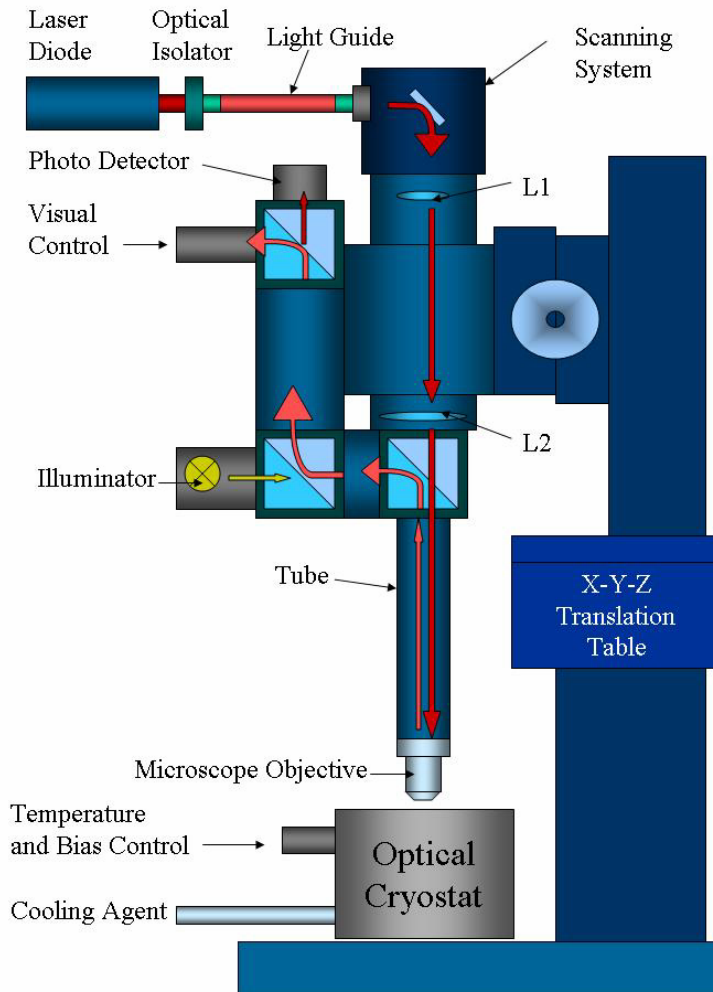


FC in  
110 mT,  
then  $H=0$



1 mm  


# Erlangen LSM setup



# Motivation / Goals

---

To **image rf currents** in operating superconducting microwave circuits and devices

To identify **sources of microwave nonlinearities** in superconductors

To investigate how rf currents are redistributed by  **$\mu\text{m}$ - and nm-scale defects**

To **develop new methods** to investigate microwave nonlinearities in superconductors



# Magnetic sensors & applications

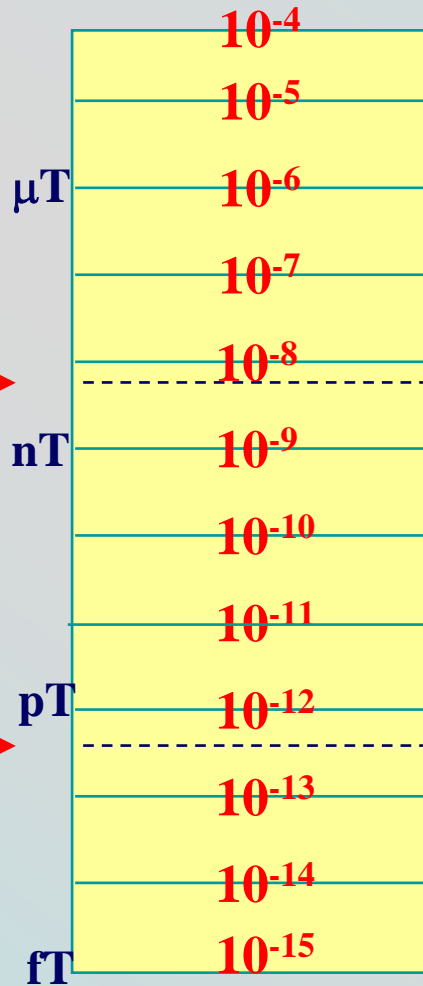
Magnetic field (Tesla)

Applications

NDE

Fatigue  
corrosion

Biomagnetic  
applications



Magnetometer field  
resolution

Hall

GMR

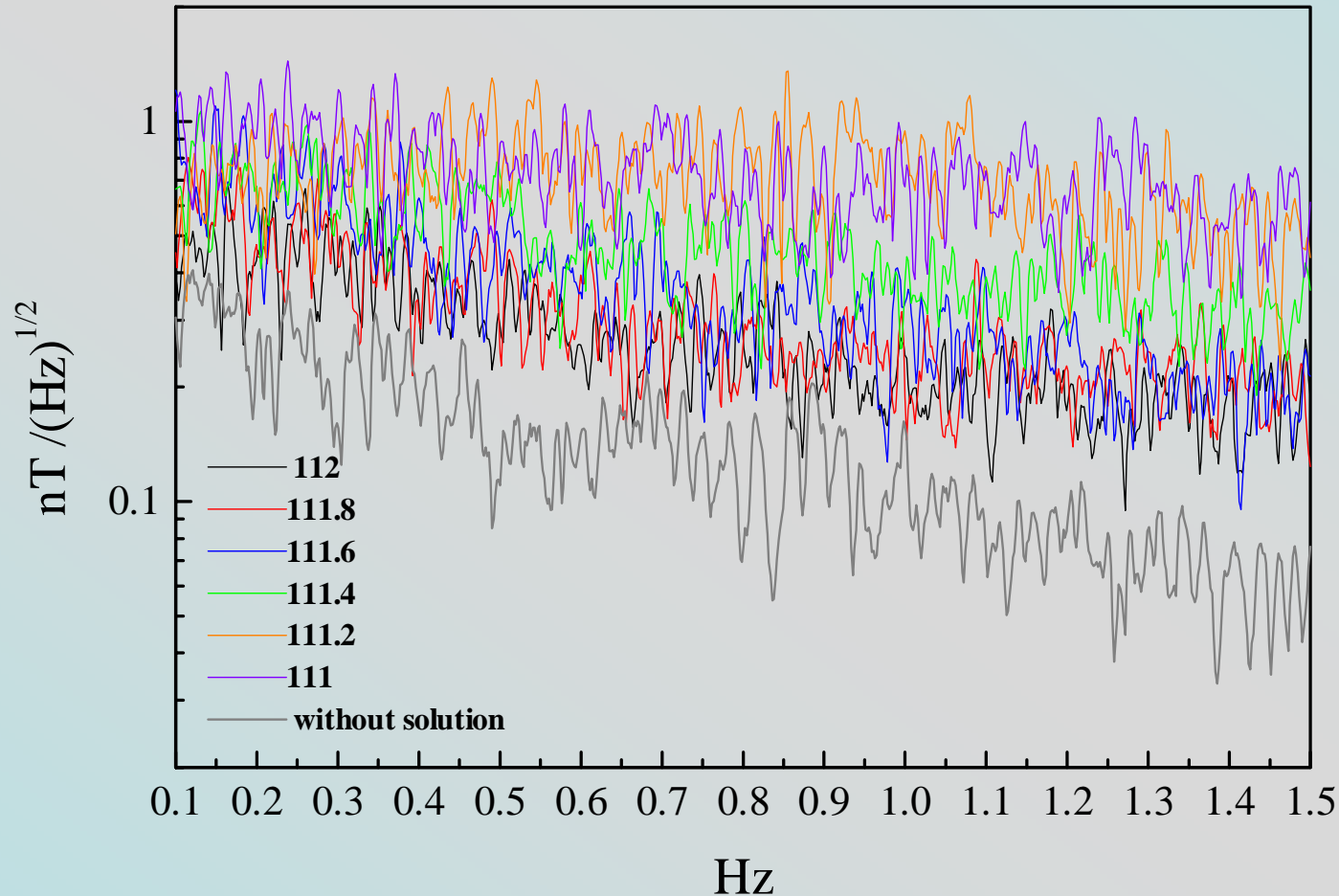
Flux-gate

HTS

LTS

# Buffered Chemical polishing of Niobium

BCP 1:1:1 to BCP 1:1:2 increasing the  $\text{H}_3\text{PO}_4$  percentage the magnetic signal intensity increase



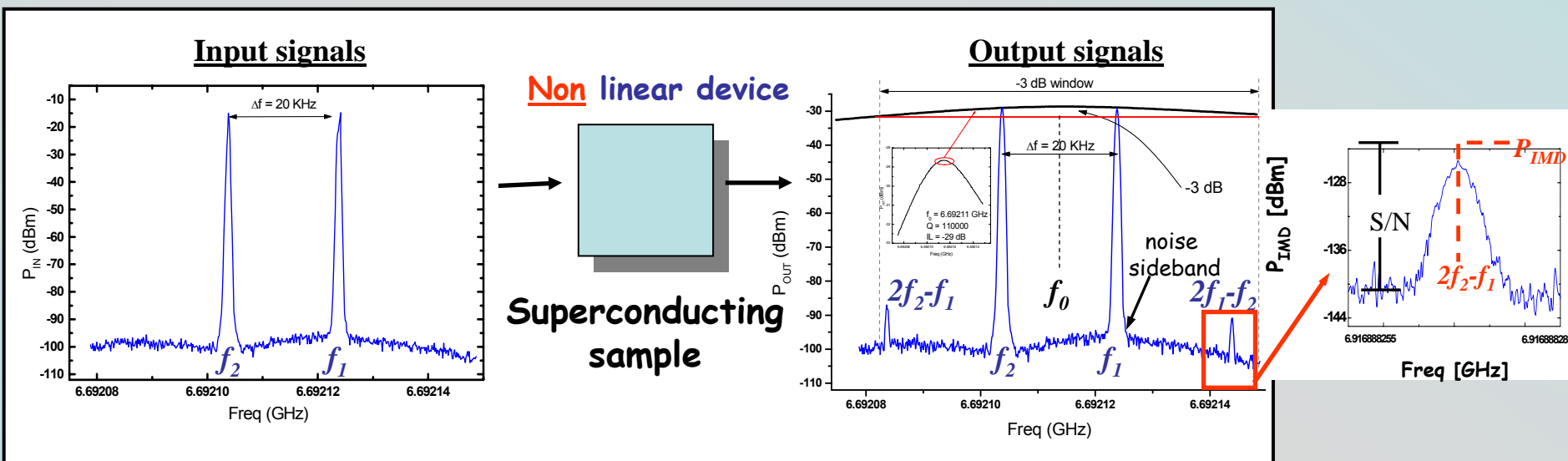


# The IMD technique

Input signal made up of two signals of equal magnitude but different frequencies:

$$V_{in} = V_1 \cos(2\pi f_1 t + a_1) + V_2 \cos(2\pi f_2 t + a_2)$$

$$|V_1 - V_2| < 0.1 \text{ dBm}, f_1 - f_2 = 20 \text{ kHz} \ll 3 \text{ dB bandwidth}$$



High purity CW mw sources and high sensitivity spectrum analyser set noise threshold  $\sim -140 \text{ dBm}$

**Dynamic range can be larger than 100 dB**

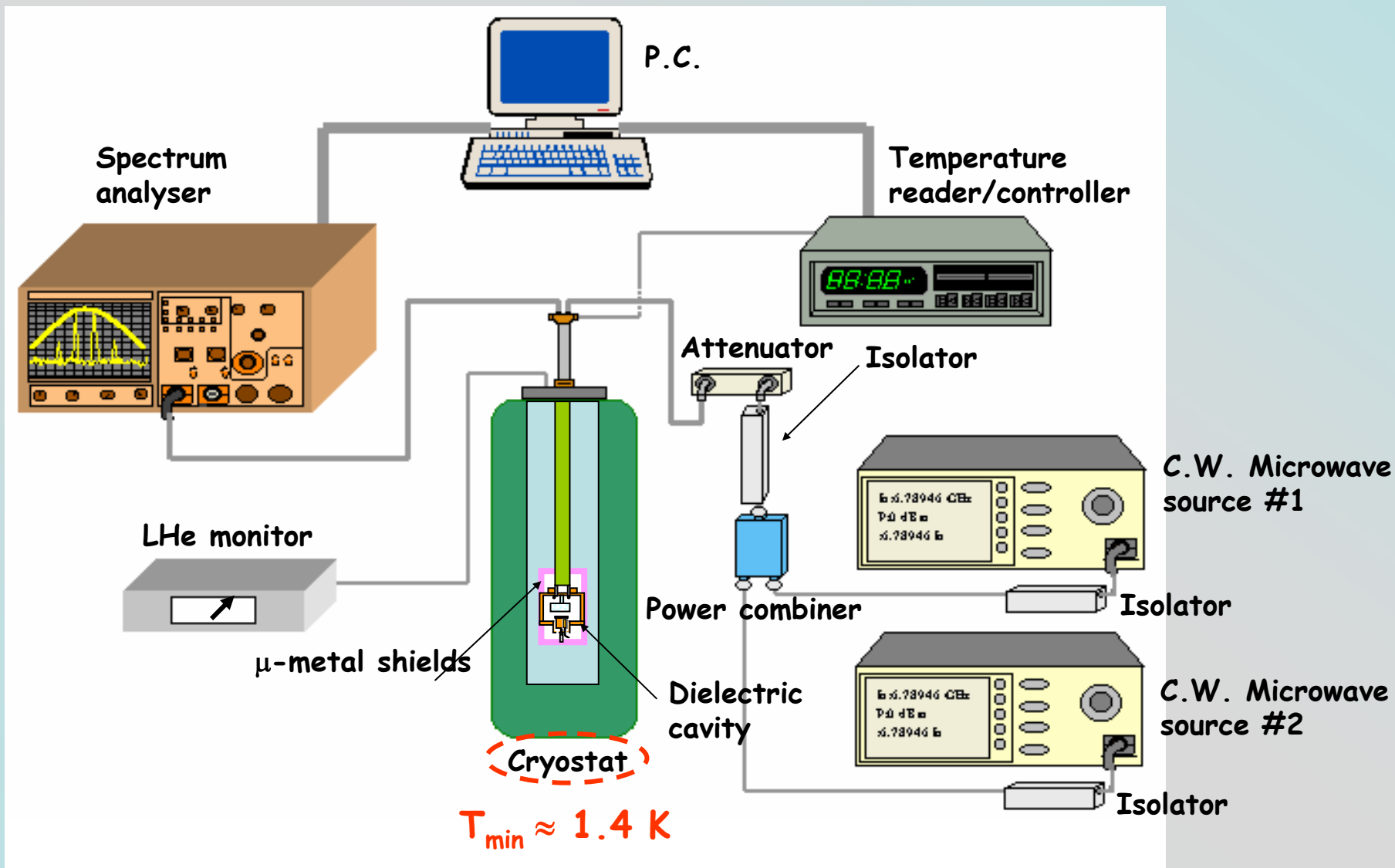
Higher order products are usually reduced in amplitude

**A. Andreone**



# Experimental set-up

A. Andreone

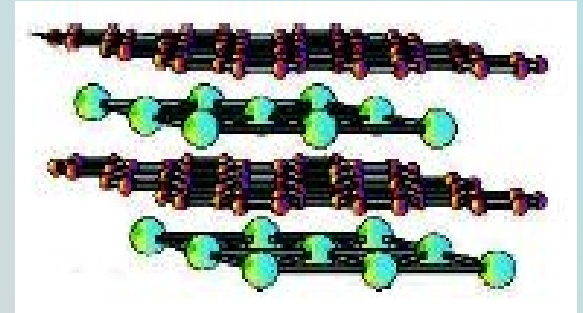




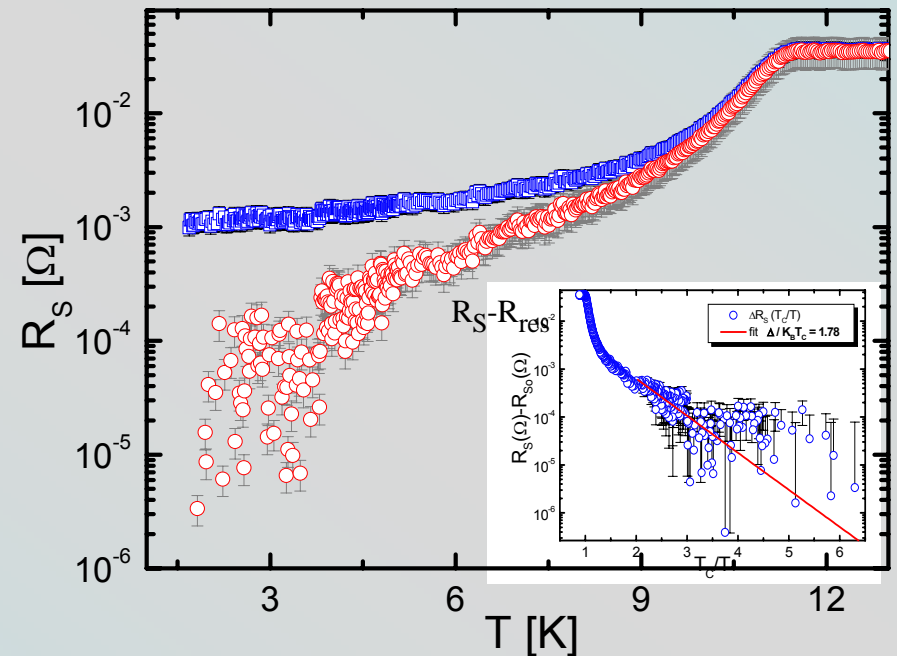
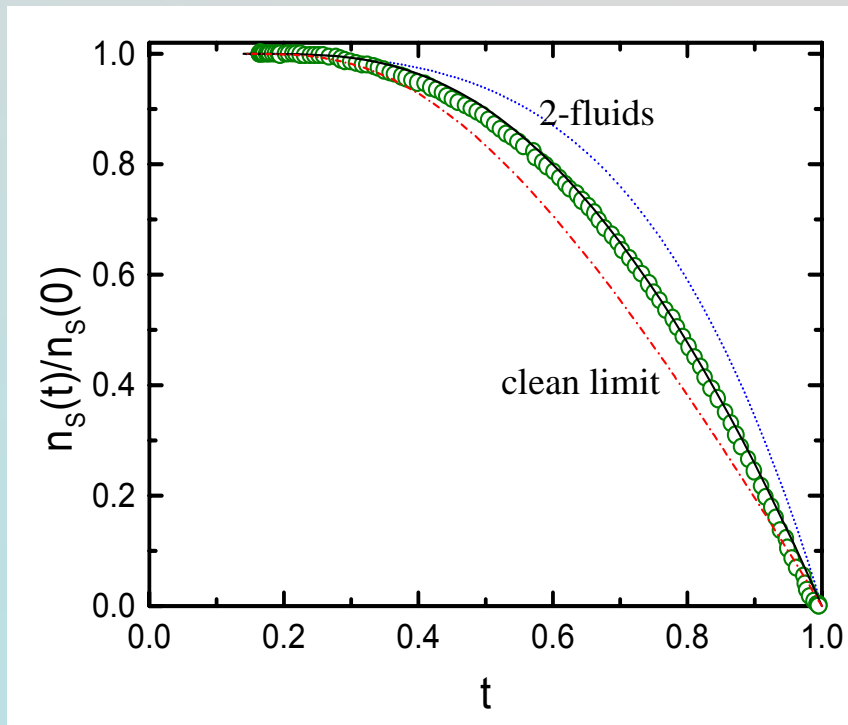


# CaC<sub>6</sub>: a graphite intercalated superconductor

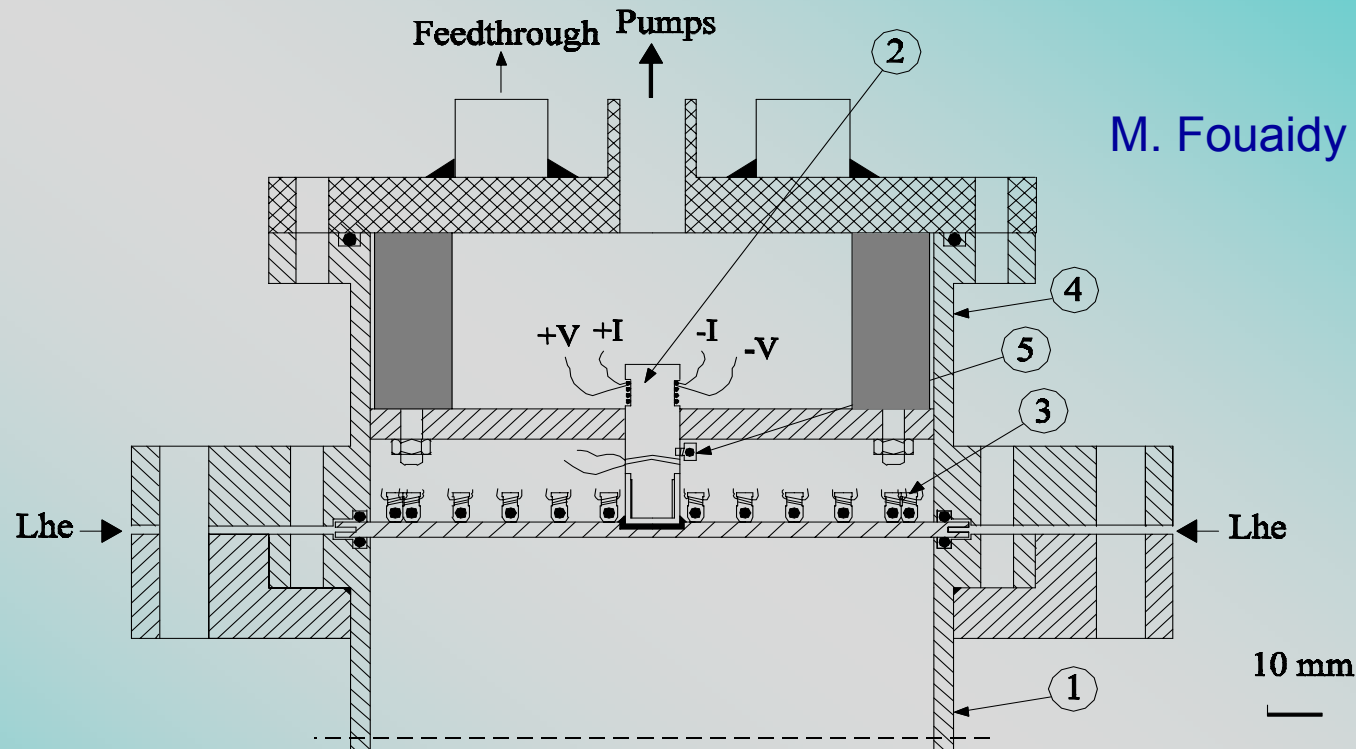
$$T_C = 11.2 \text{ K}$$



Both superfluid density and microwave losses give evidence of conventional, weak-coupled, superconductivity ( $2\Delta/K_B T_C \sim 3.6$ )



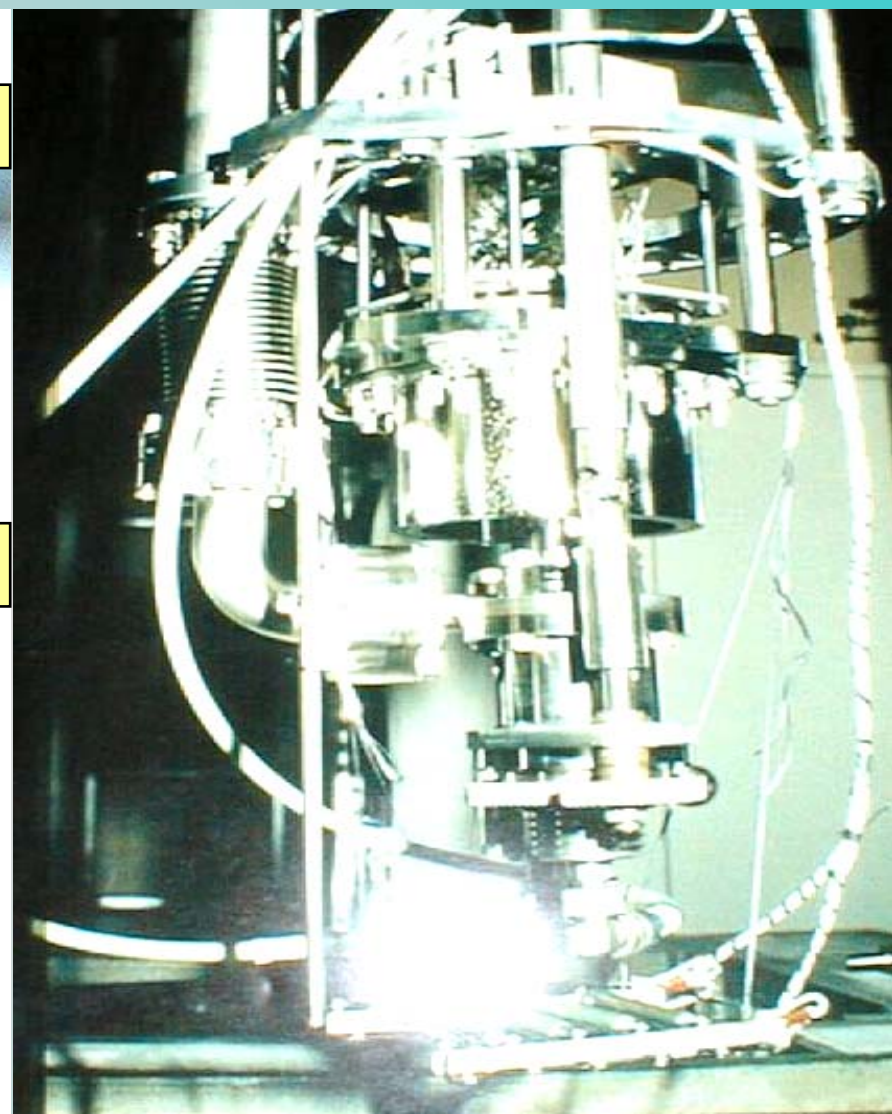
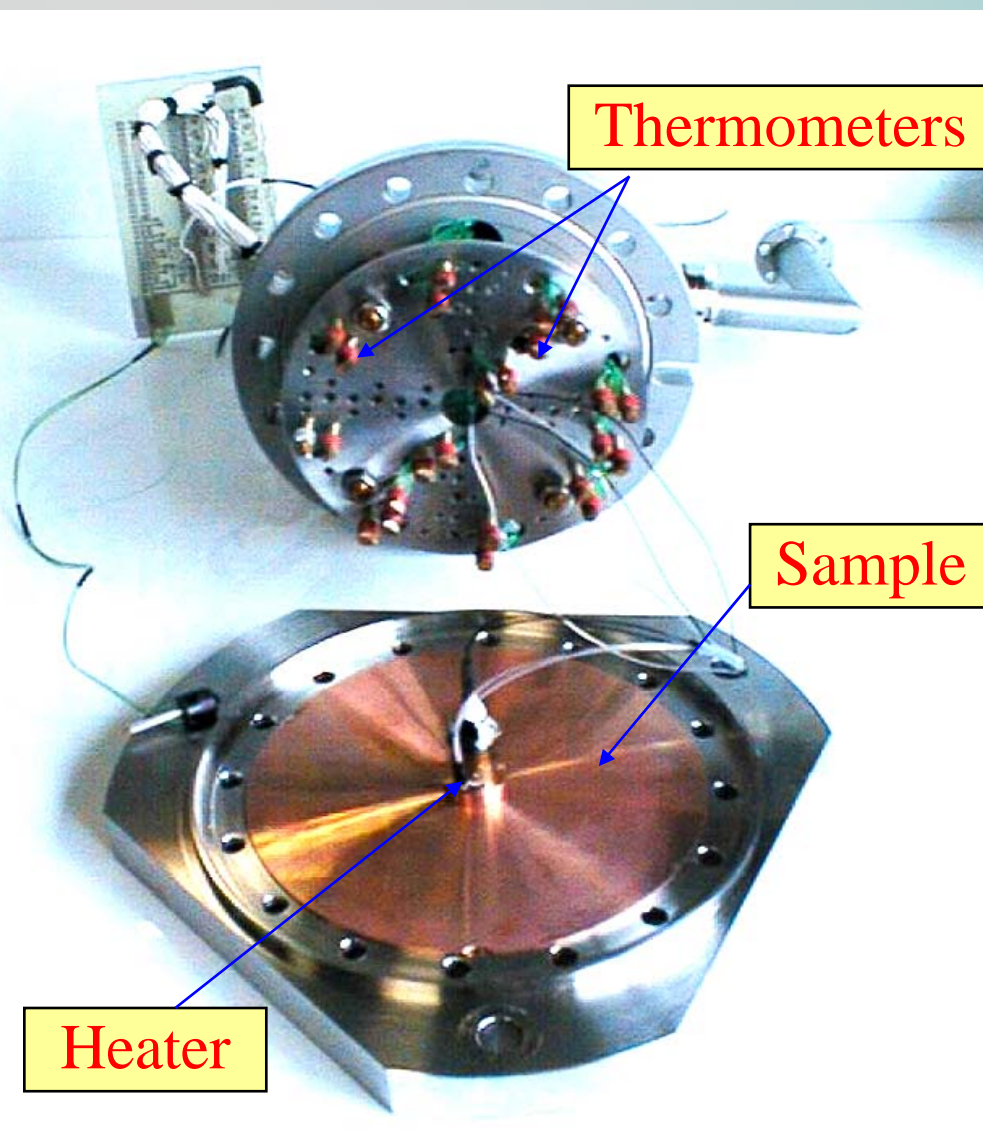
# Thermometric system (2)



M. Fouaidy

- ① Niobium cavity TE011 mode  $f=4\text{GHz}$  TE012 mode  $f=5.6\text{GHz}$
- ② Calibration heater
- ③ thermometers
- ④ Vacuum
- ⑤ Heater thermometer

# Thermometric system (3)

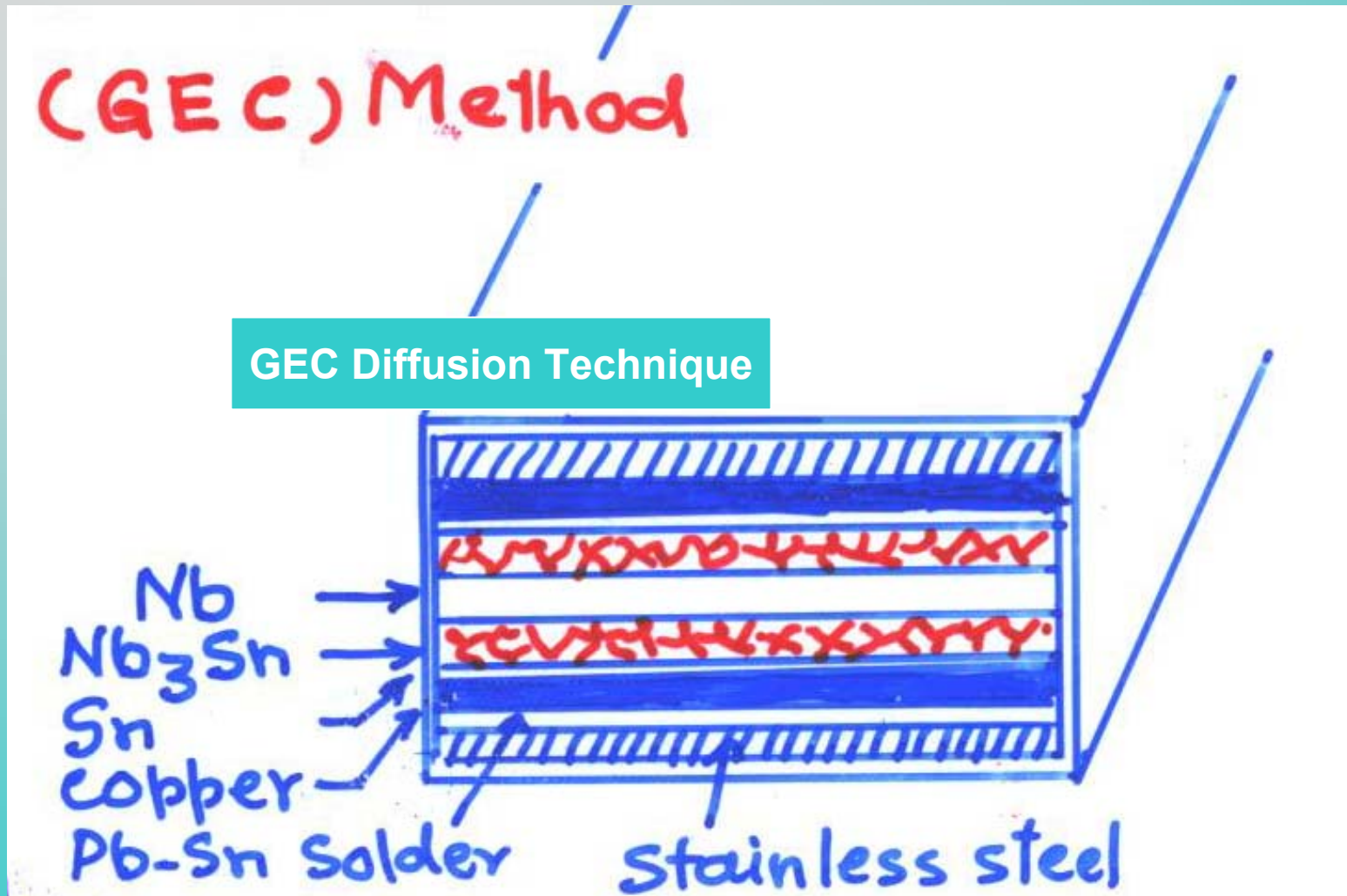


# High Field Superconductor With Technological Potential

Material	Structure Classification	Tc (K)	H <sub>c2</sub> (T) Obs. 4.2K	Jc (A.cm <sup>-2</sup> ) 4.2 K (T)
Nb-Ti	A-2	10.2	12	5 x 10 <sup>5</sup> (5 T)
Nb-Zr	A-2	10.8	11	
V <sub>3</sub> Ga	A-15	15.5	23.6	2 x 10 <sup>6</sup> (10T)
V <sub>3</sub> Si	A-15	17.0	23	
Nb <sub>3</sub> Sn	A-15	18.3	26	3 x 10 <sup>5</sup> (10T)
Nb <sub>3</sub> Al	A-15	18.9	29.5	10 <sup>5</sup> (22T)
Nb <sub>3</sub> (Al,B,Be)	A-15	20.0		3 x 10 <sup>4</sup> (8T)
Nb <sub>3</sub> Ga	A-15	20.3	33	
Nb <sub>3</sub> (Al,Ge)	A-15	20.5	41	1 x 10 <sup>4</sup> (12T)
Nb <sub>3</sub> Ge	A-15	23.2	37	
NbCN	B-1	17.8	12	1 x 10 <sup>3</sup> (7T)
V <sub>2</sub> (Hf,Zr)Lave	C-15	10.1	23	3 x 10 <sup>5</sup> (6T)
PbMo <sub>6</sub> S <sub>8</sub>	C-15	14.0	65	



- **Crosssection of Diffusion Nb<sub>3</sub>Sn Tapes**



# What happens with changing Sn content?



## Pure Nb

- ➔ *bcc* Nb spacing 0.286 nm
- ➔  $T_c = 9.2$  K

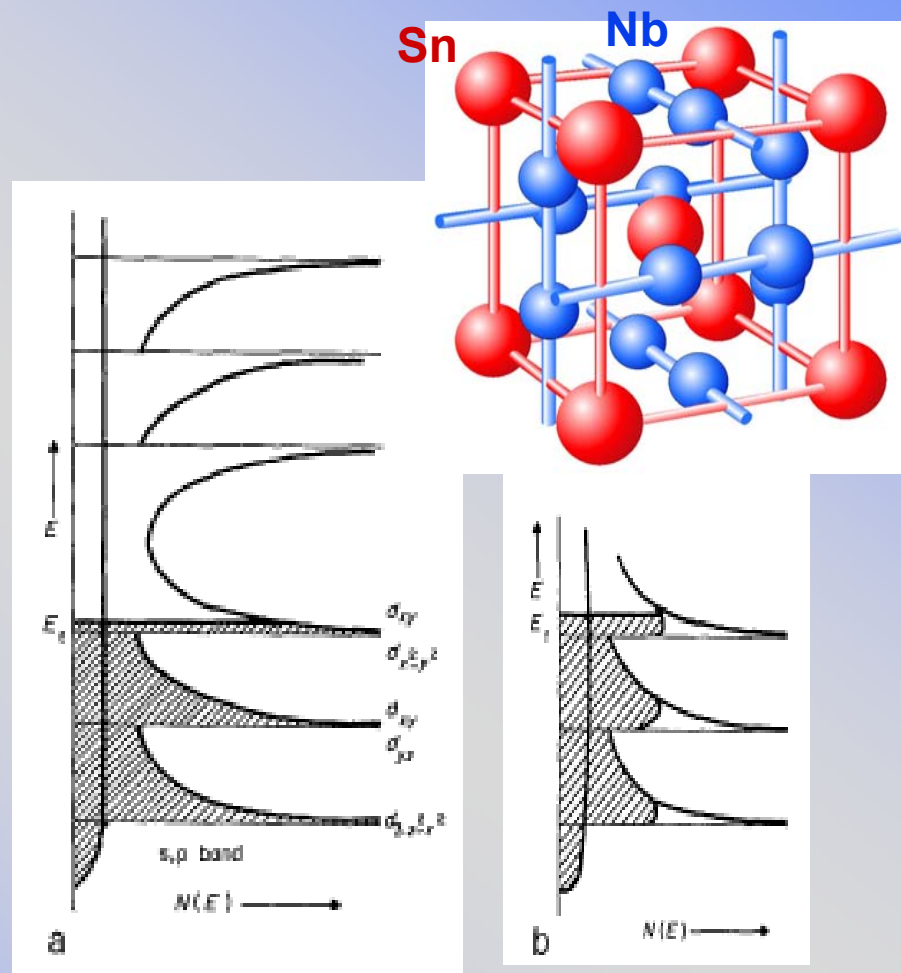
## $\text{Nb}_3\text{Sn} \rightarrow \text{A15 unit cell}$

- ➔ *bcc* Sn, orthogonal Nb chains
- ➔ Nb spacing 0.265 nm
- ➔ High peaks in d-band DOS
- ➔ Increased  $T_c = 18$  K

## Off-stoichiometry

- ➔ Sn vacancies unstable
- ➔ Excess Nb on Sn sites
  - Additional d-band
  - Less electrons for chains
  - Rounded off DOS peaks
  - Reduced  $T_c$

## A15 lattice and DOS

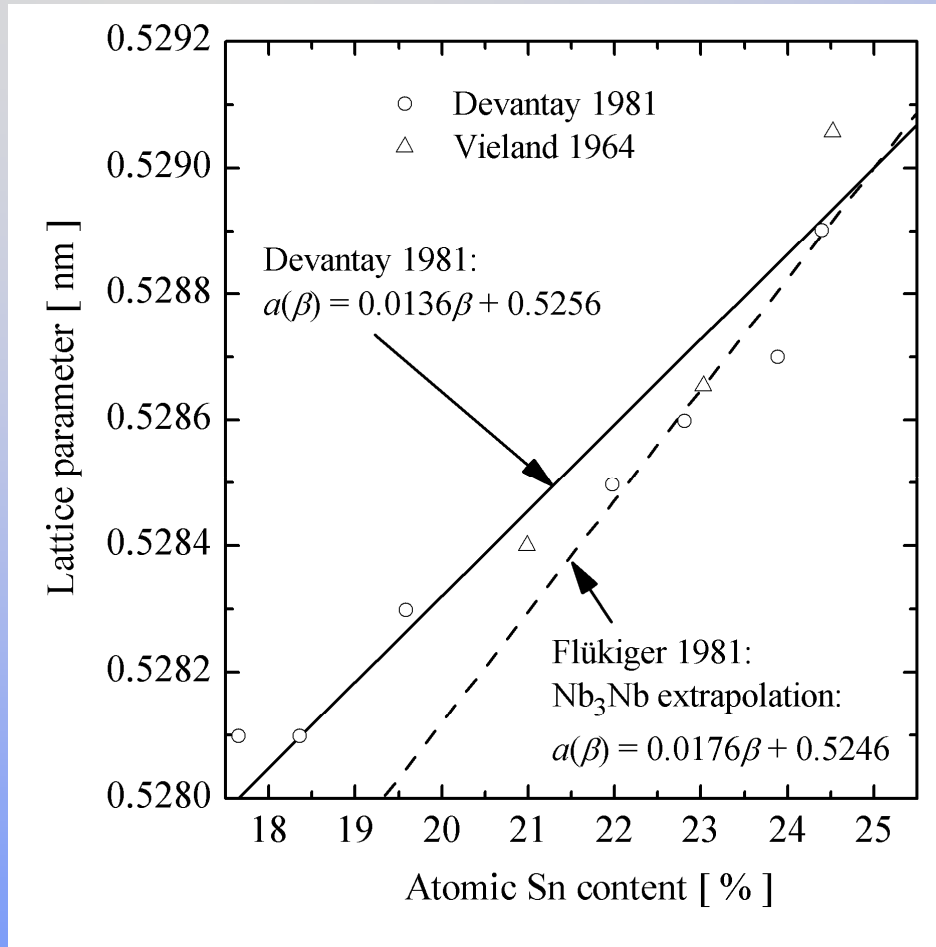


➔ Dew-Hughes, Cryogenics 1975

# Sn content: Lattice parameter



$a$  increases with Sn content (as does  $T_c$  (below))



Maximum  $T_c$

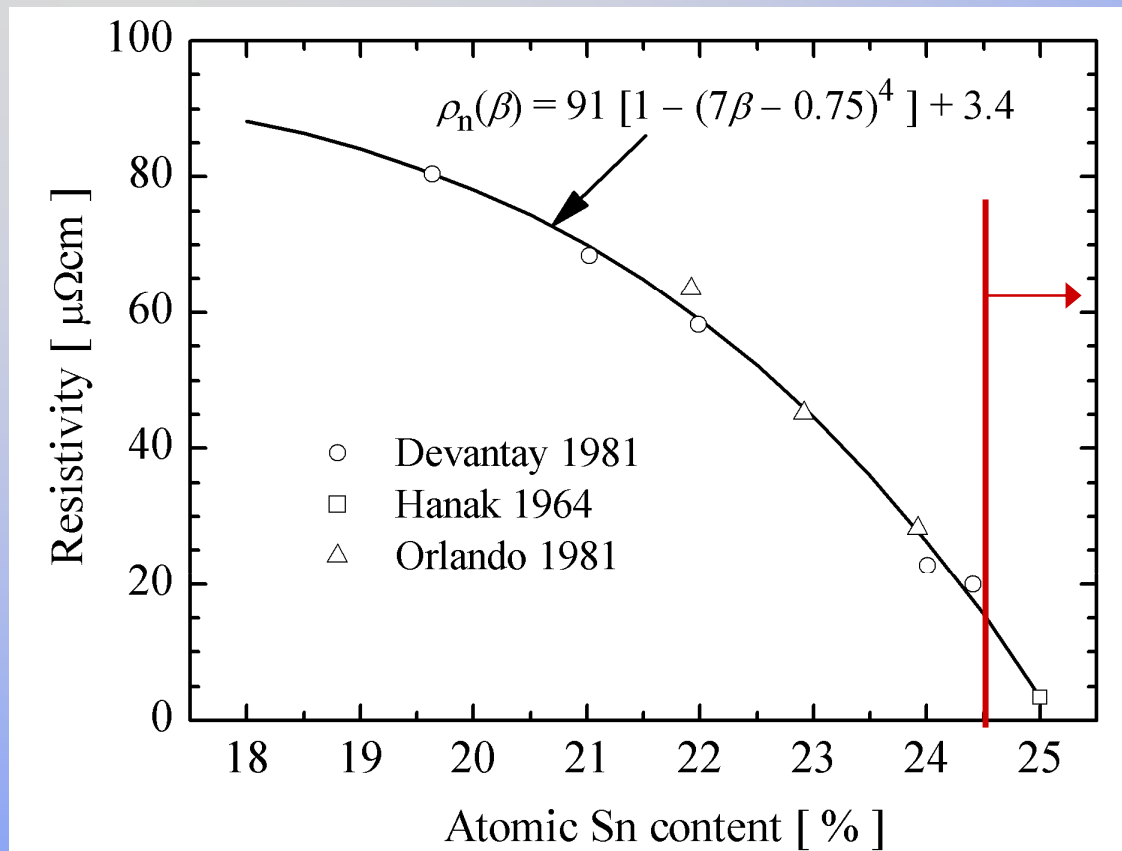
Reduced  $T_c$   
(from Sn deficiency  
though Nb spacing  
is smaller)

➡ Devantay, JMS 1981; Vieland, RCA Rev. 1964; Flükiger, 1981

# Sn content: Resistivity



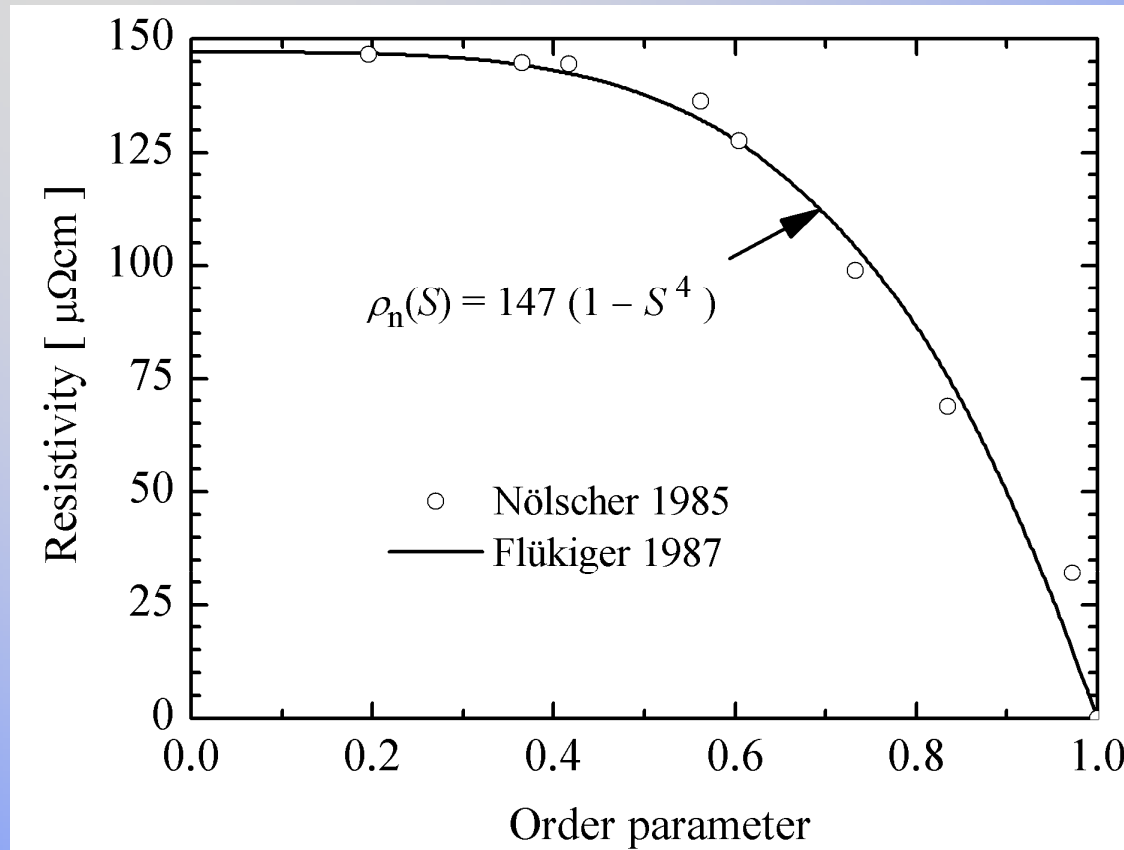
**Nb<sub>3</sub>Sn is cleanest at stoichiometry**



➡ Devantay, JMS 1981; Hanak, RCA Rev. 1964; Orlando, TM 1981



## Bragg-Williams Order Parameter varied through irradiation



- Effect on  $\rho_n$  similar as changing Sn content
- $a$ ,  $S$  and  $\rho_n$  can all be related to atomic Sn content

# Liquid Sn Diffusion?



Bulk Nb substrate dipping  
in a liquid Tin bath



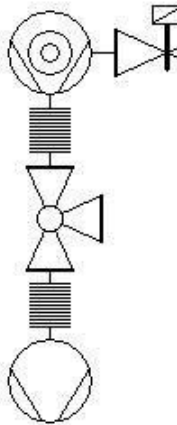
Sample Annealing

- No nucleation sites on Nb are required
- Fast growth of  $\text{Nb}_3\text{Sn}$  layer
- Desirable uniform thickness

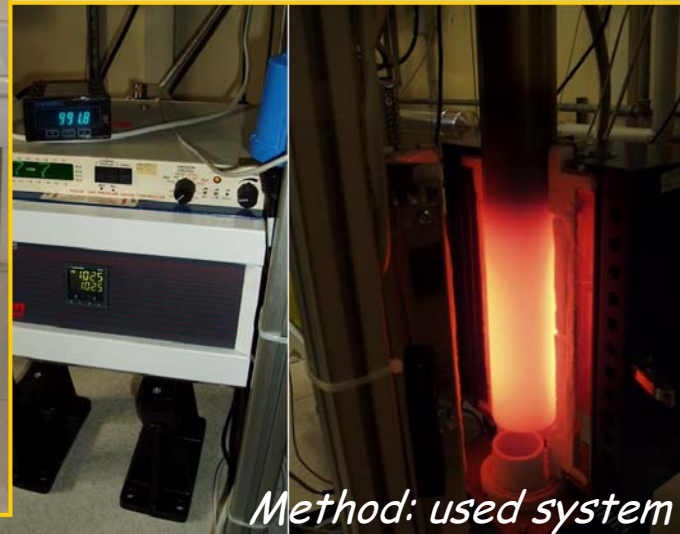
# Used System



Linear  
feedthrough

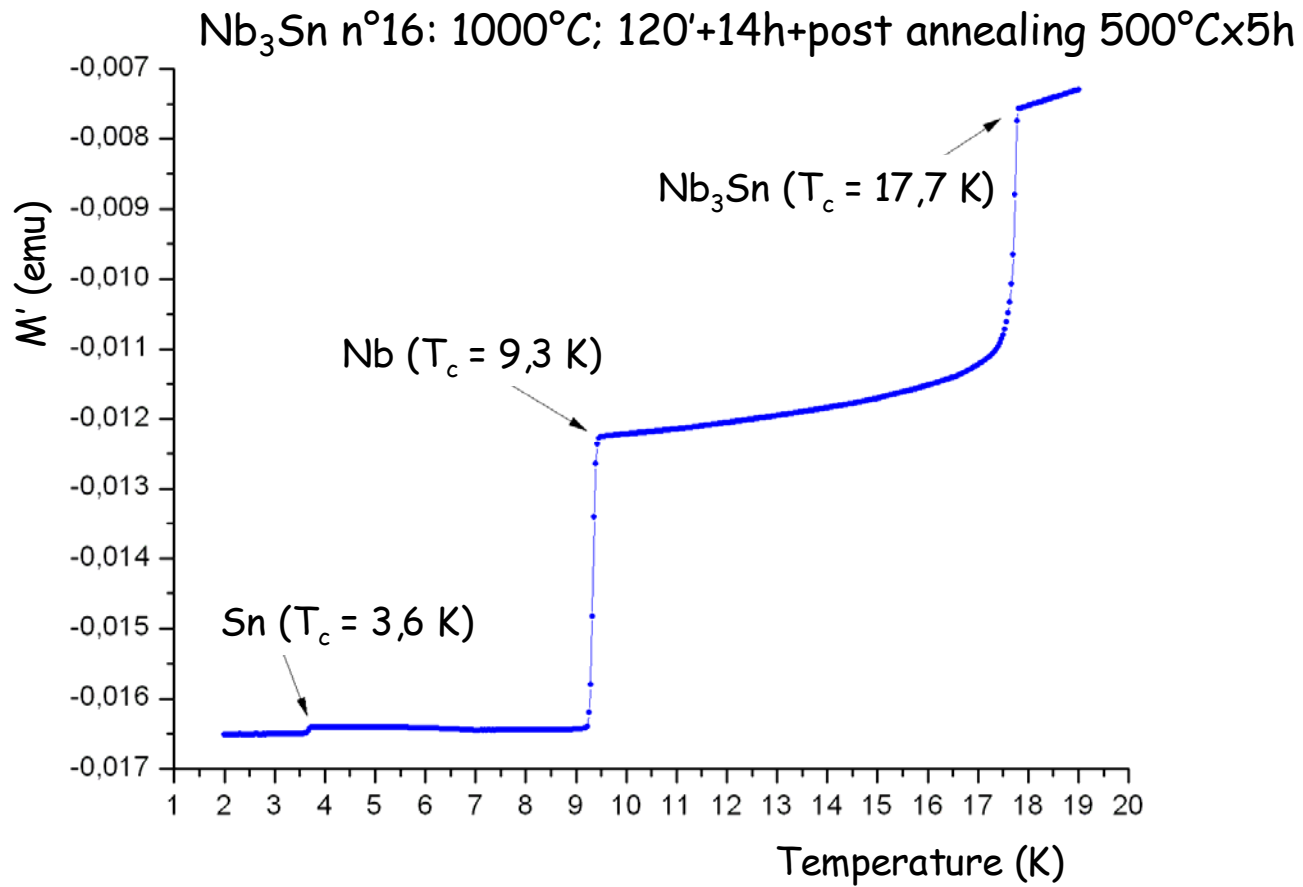


Liquid Sn



Method: used system

# S. Deambrosis et al



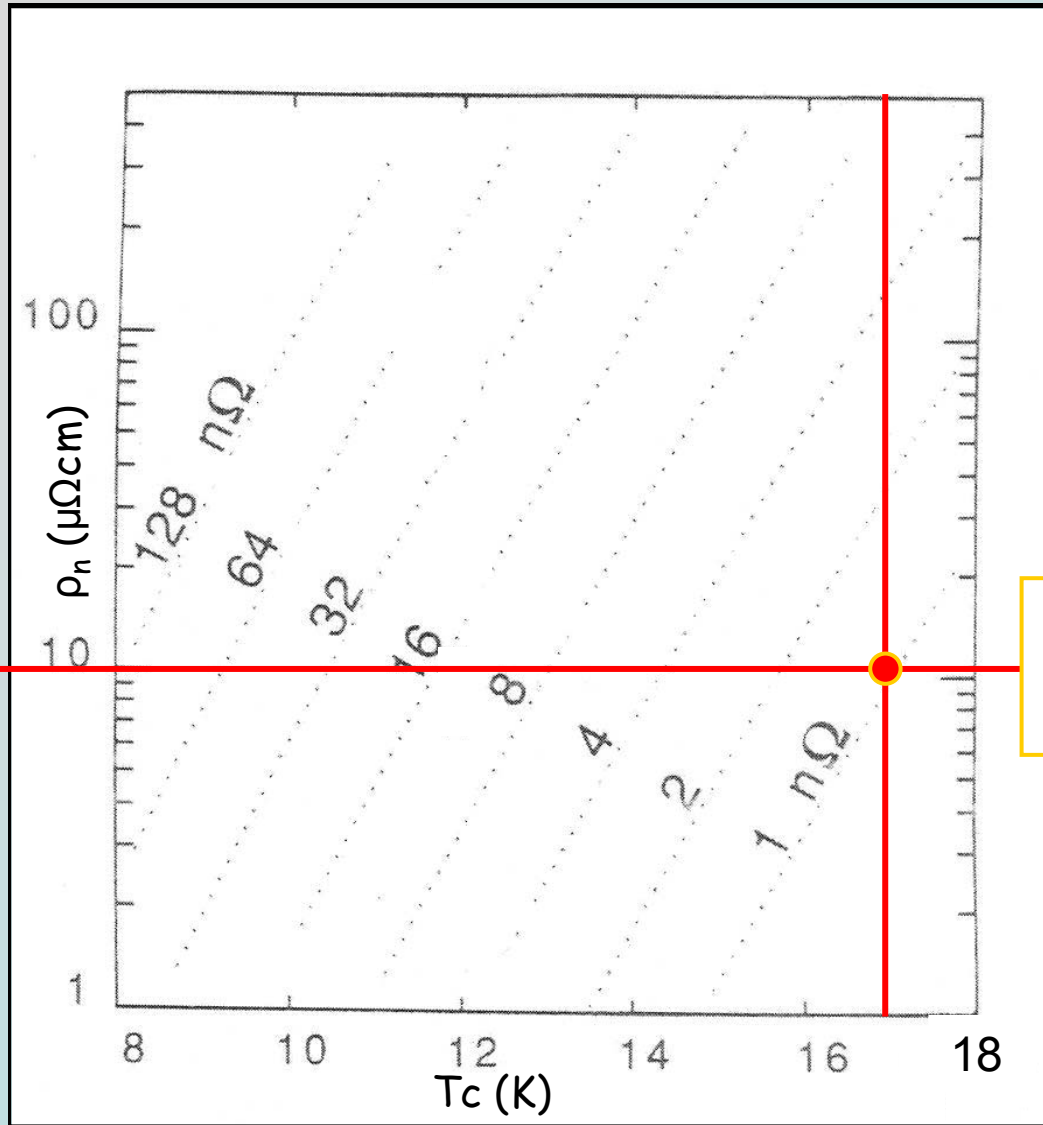


# V3Si



At  $T = 4.2$  K,  
 $f = 500$  MHz,  
 $s = 4$ ,  
 $R_{\text{BCS}}$  depends  
on  $\Delta$  and  $\rho_n$

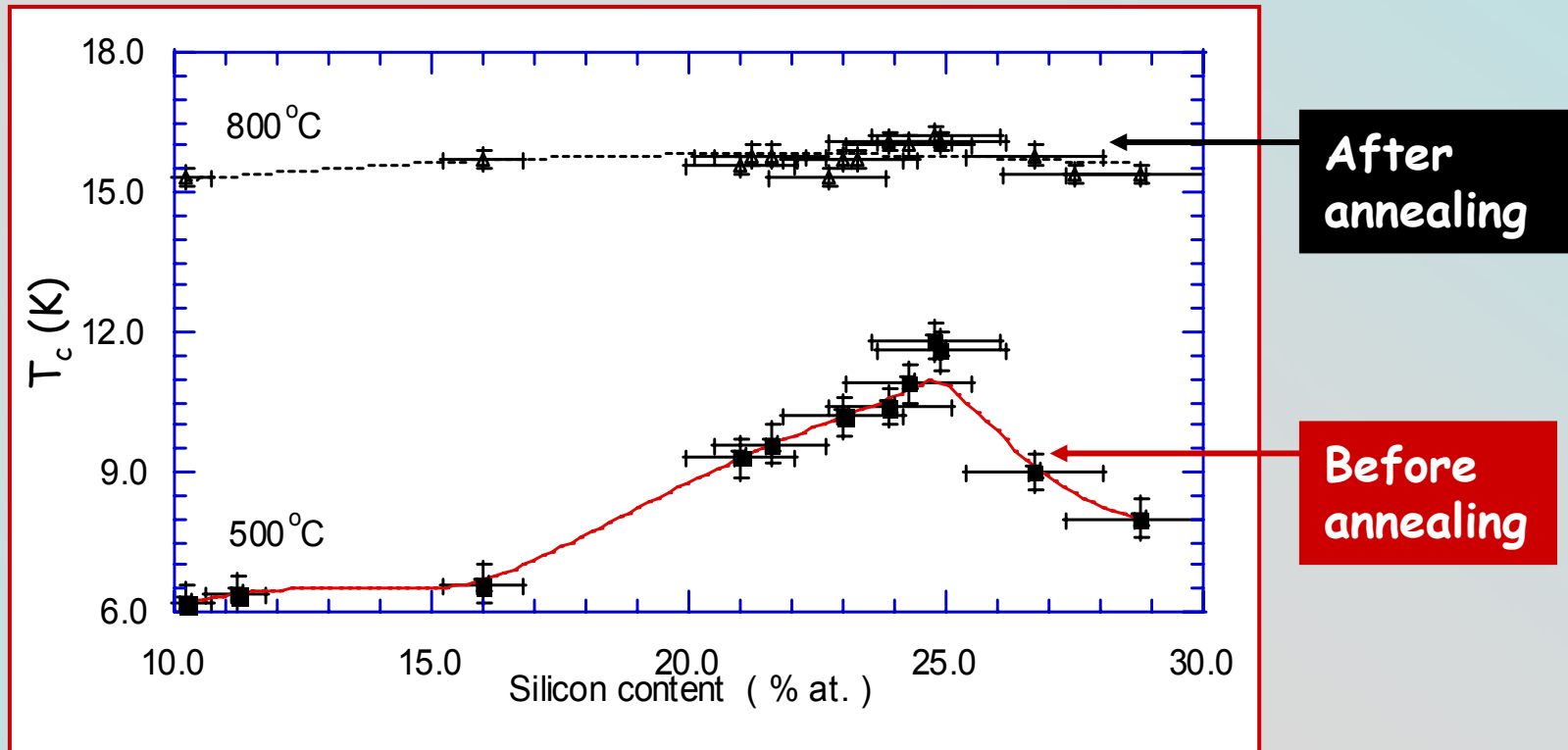
$\sim 10 \mu\Omega\text{cm}$



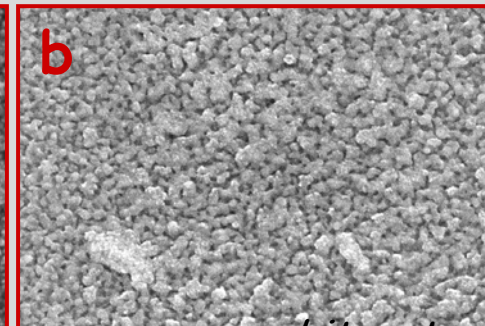
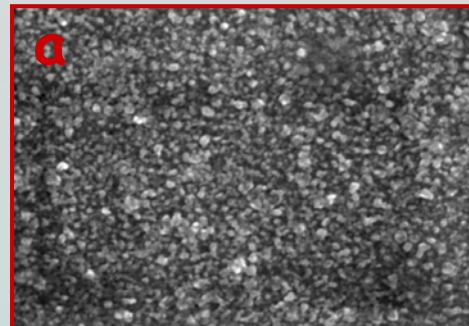
**Ideal**  
 $R_{\text{BCS}} \sim 1$  n $\Omega$

# Reactive sputtered $V_3Si$ films

Y. Zhang, V. Palmieri, W. Venturini, R. Preciso, Legnaro National Laboratory - INFN, Italy



Surface of two annealed samples under SEM: Grain size, (a)  $0.2\mu\text{m}$ , (b)  $0.5\mu\text{m}$



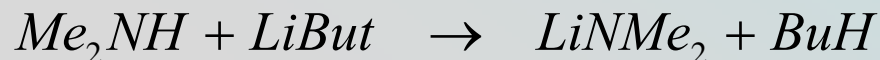
*Literature*

# 1- Pentakis(dimethylamide)niobium

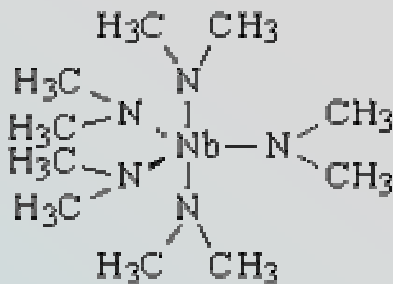
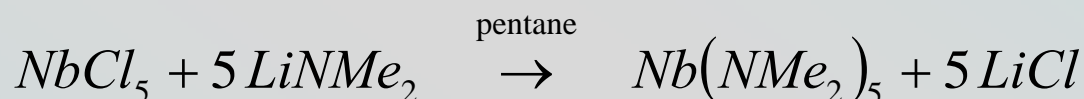
  

## $\text{Nb}(\text{NMe}_2)_5$

►  $\text{Me}_2\text{NH}$  gas bubbled into 50 mL  $\text{LiBut}$  1,6 M (80mmol) for 90 min;



added 4,3 g  $\text{NbCl}_5$  (16 mmol)



- after filtering  $\text{LiCl}$  the solution was dried under vacuum obtaining a dark brown solid;
- purification by sublimation at  $130^\circ\text{C}$  under vacuum;
- NMR  $^1\text{H}$  and  $^{13}\text{C}$  characterization: good product but with a low volatility.

This compound was not used for the MOCVD deposition.

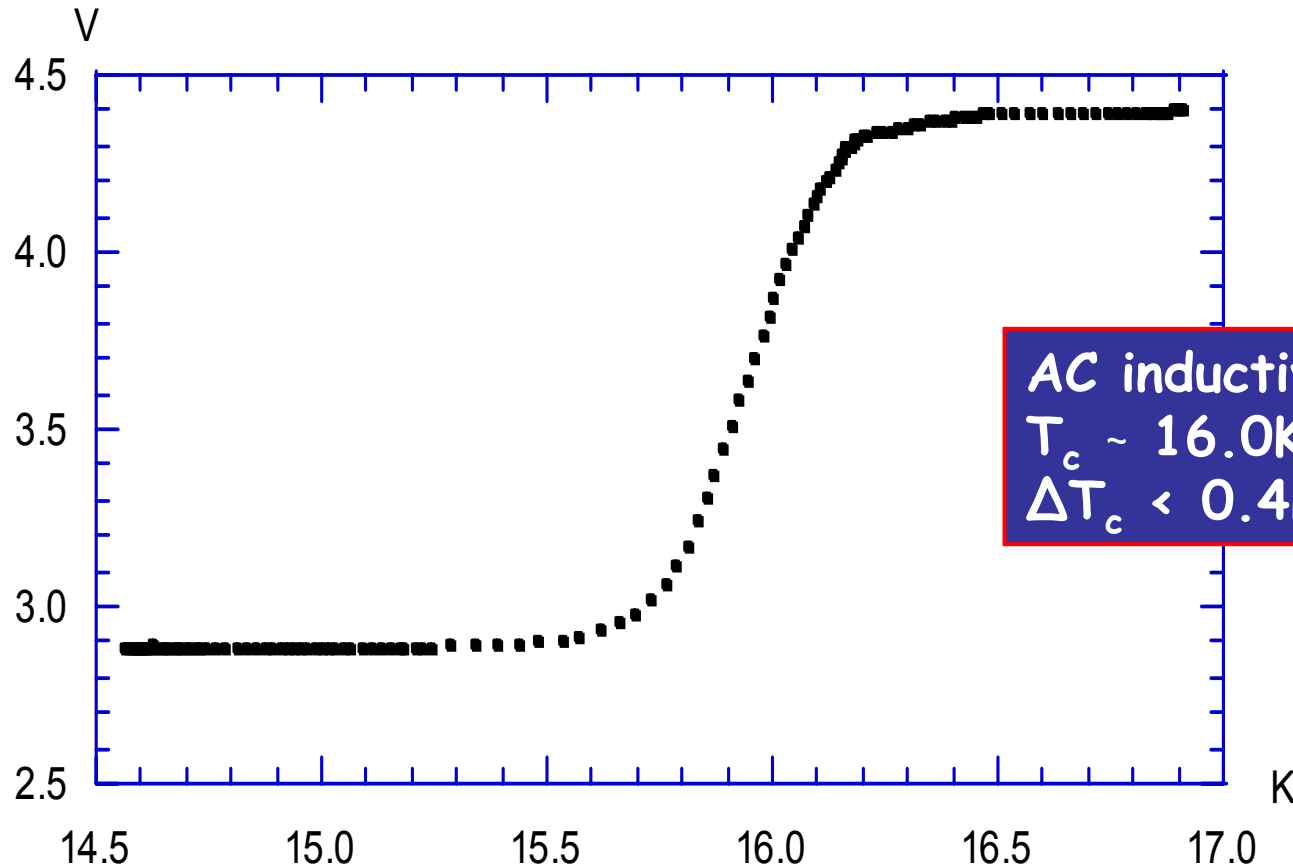
# Thermal diffusion of $V_3Si$ films

Y. Zhang, V. Palmieri, W. Venturini, F. Stivanello, R. Preciso, Legnaro National Laboratory, ITALY



Diffusion  
Parameters:

Silane pressure	Heat power	Temperature	Diffuse in silane	Anneal in vacuum
$1.2 \cdot 10^{-4}$ mbar	300W	900°C	20h	40h

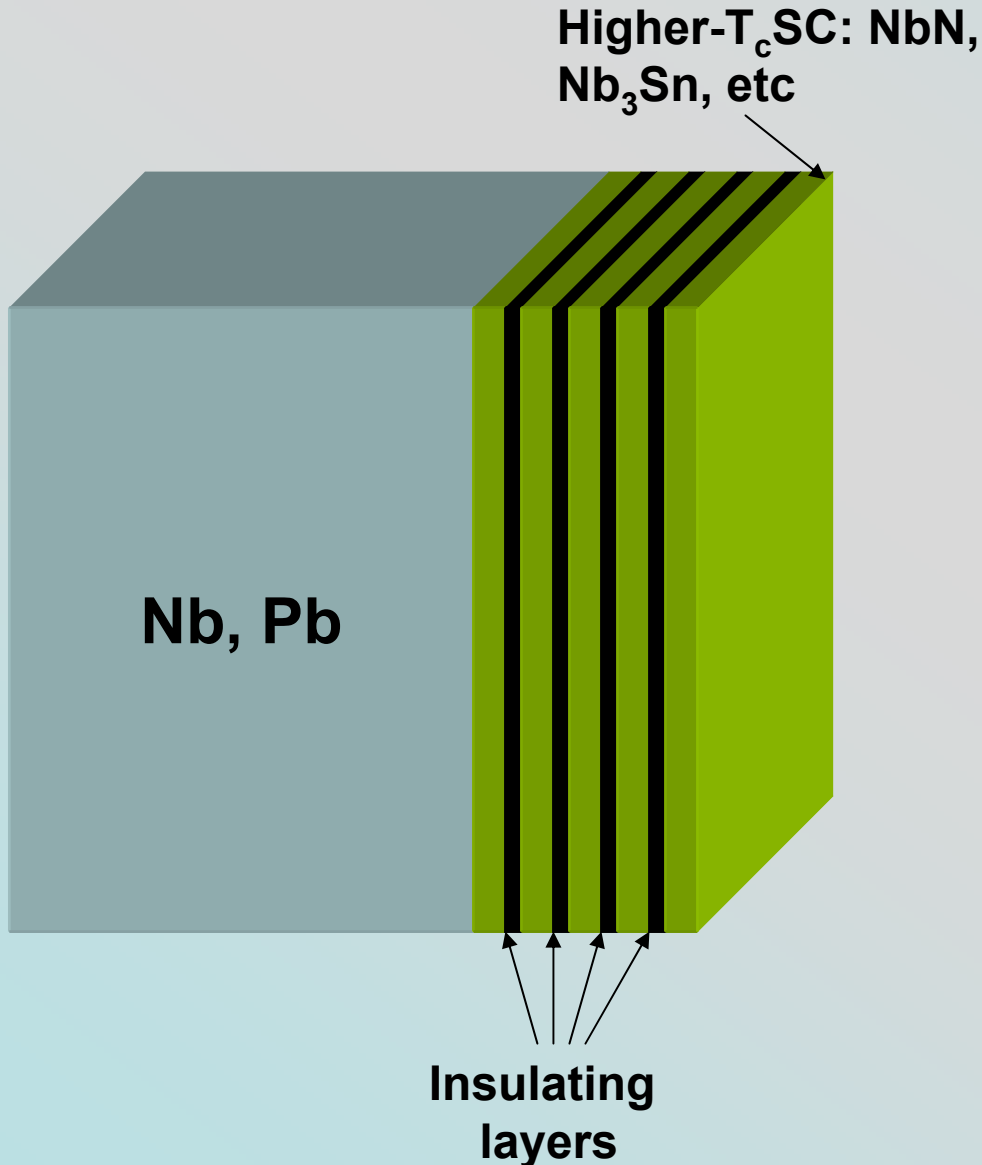


AC inductive measurement:  
 $T_c \sim 16.0\text{K}$   
 $\Delta T_c < 0.4\text{K}$

Literature



# How one can get around small $H_{c1}$ in SC cavities with $T_c > 9.2K$ ? AG, Appl. Phys. Lett. 88, 012511 (2006)



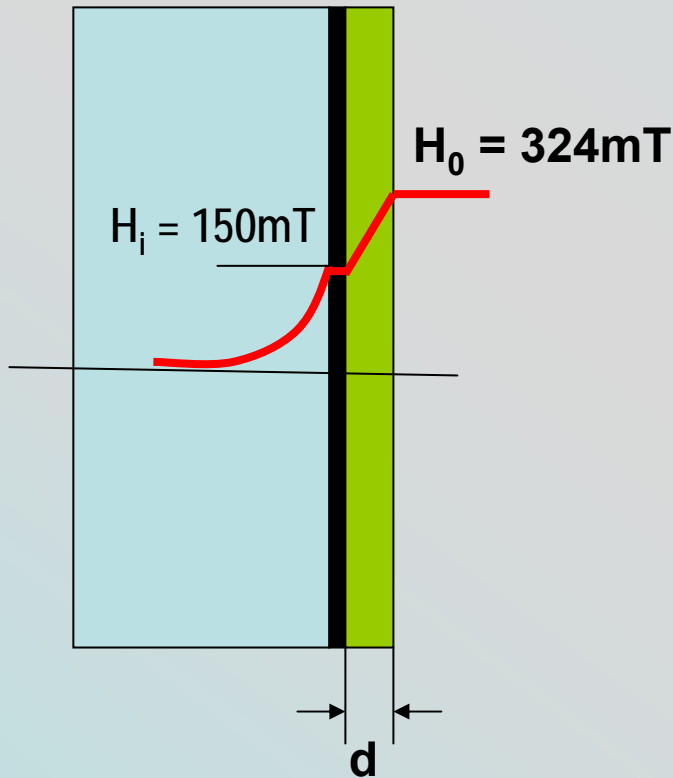
Multilayer coating of SC cavities: alternating SC and insulating layers with  $d < \lambda$

Higher  $T_c$  thin layers provide magnetic screening of the bulk SC cavity (Nb, Pb) without vortex penetration

For NbN films with  $d = 20$  nm, the rf field can be as high as 4.2 T !

No open ends for the cavity geometry to prevent flux leaks in the insulating layers

# A minimalistic solution



A Nb cavity coated by a single  $\text{Nb}_3\text{Sn}$  layer of thickness  $d = 50\text{nm}$  and an insulator layer in between

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

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

Lower critical field for the  $\text{Nb}_3\text{Sn}$  layer with  $d = 50\text{ nm}$  and  $\xi = 3\text{nm}$ :  $H_{c1} = 1.4\text{T}$  is much higher than  $H_0$

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

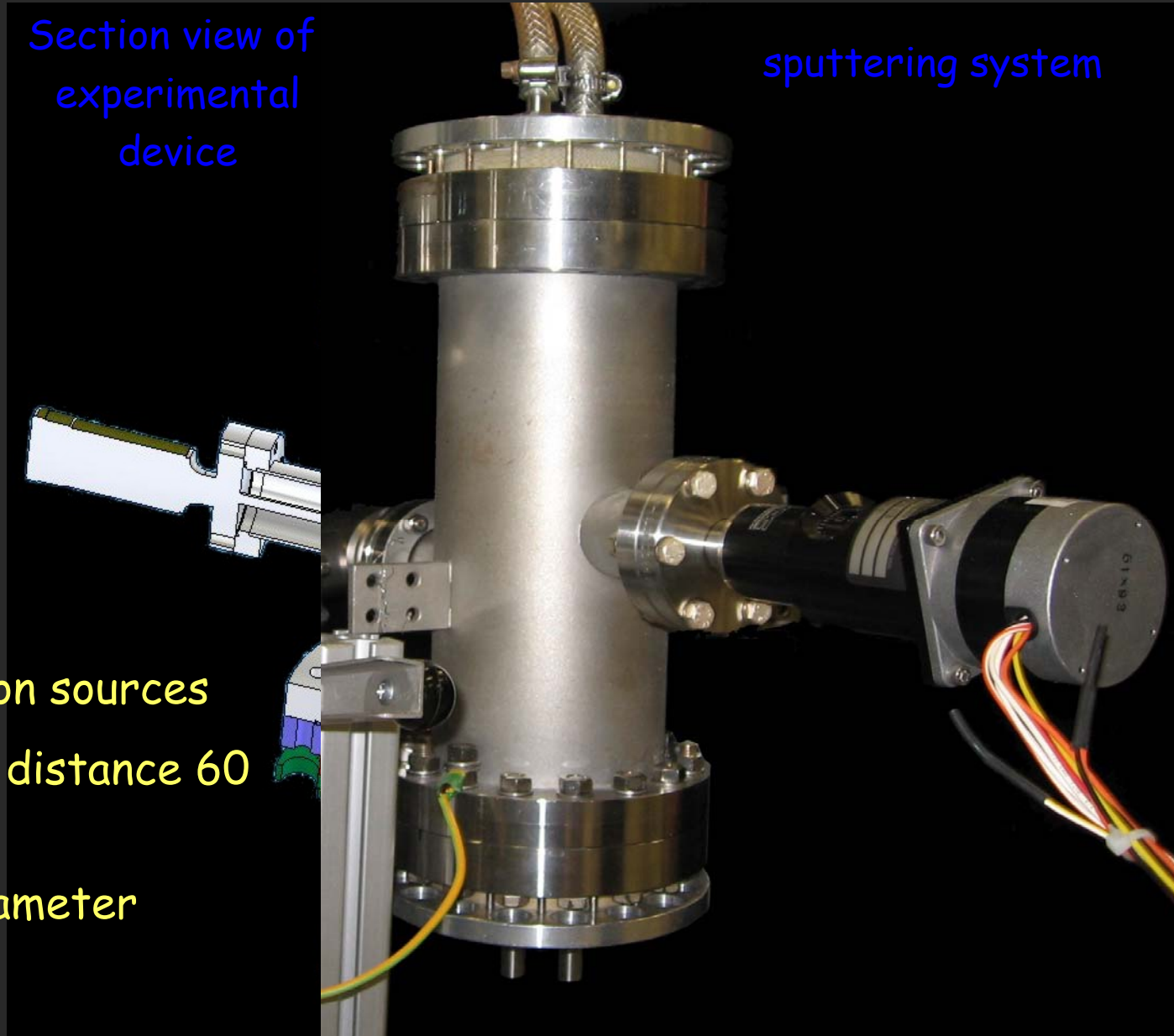
# Gurevitch idea:

- Multilayer S-I-S-I-S coating could make it possible to take advantage of superconductors with much higher  $H_c$ , than those for Nb without the penalty of lower  $H_{c1}$
- 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
- Strong reduction of BCS resistance because of using SC layers with higher  $\Delta$  ( $Nb_3Sn$ , NbN, etc)
- The significant performance gain may justify the extra cost.

# Experimental setup: sputtering

Section view of  
experimental  
device

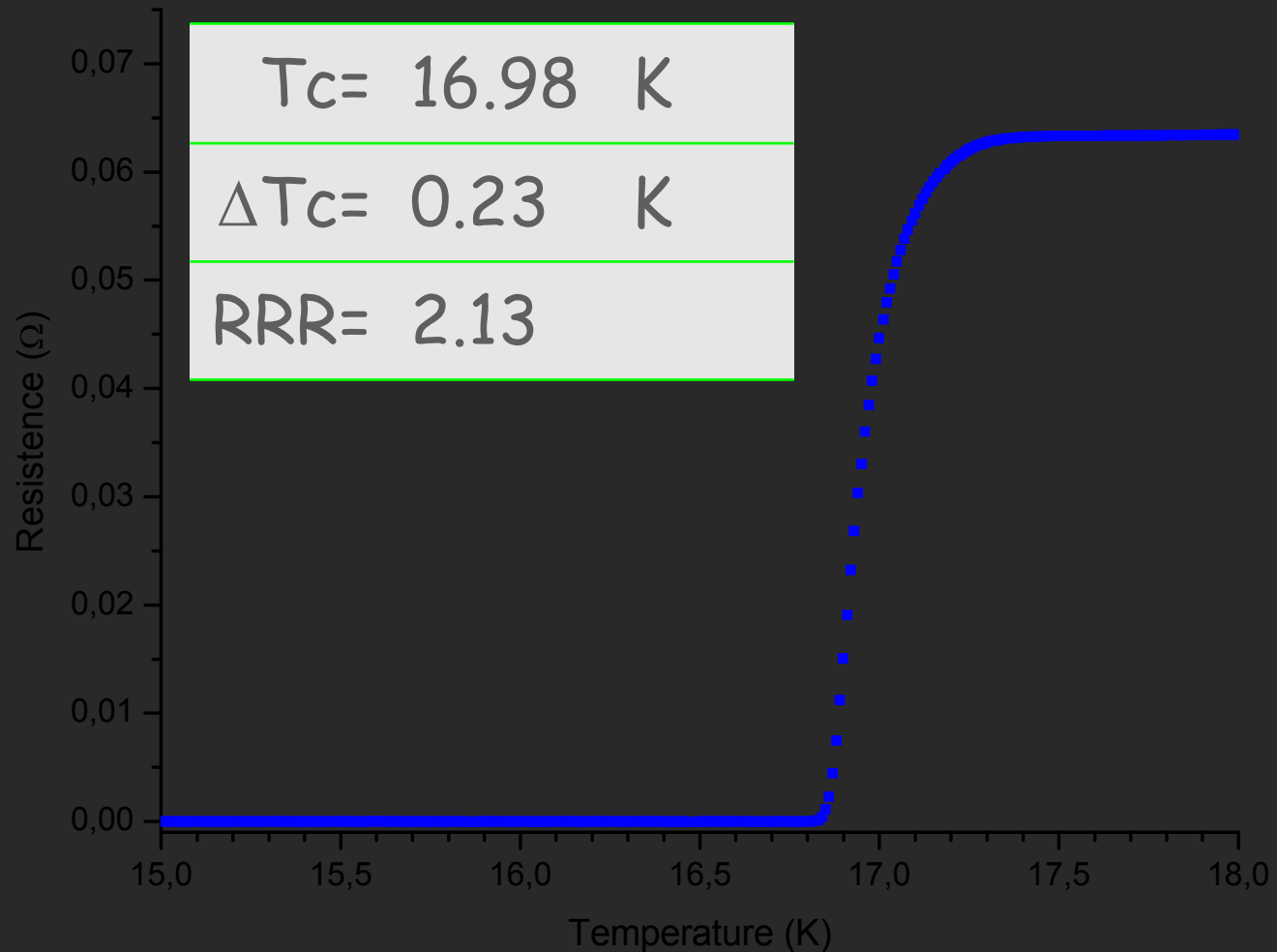
sputtering system



- ✓ Balanced Magnetron sources
- ✓ Target-Substrate distance 60 mm
- ✓ 2 inches target diameter

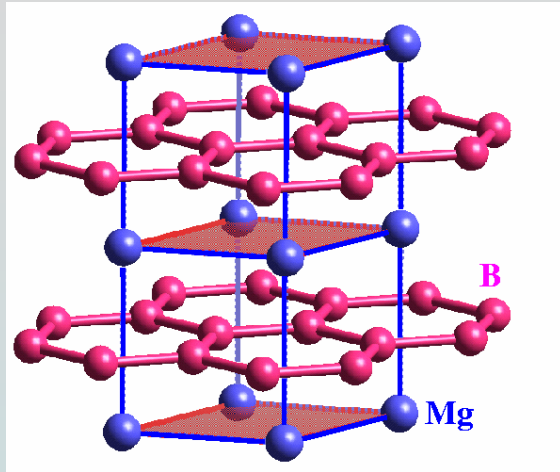


# Results: Superconductive characteristic



# MgB<sub>2</sub> properties-I

## Crystalline structure



$$T_c \approx 40 \text{ K}$$

J. Nagamatsu et al. Nature (2001) 410

3D  $\pi$  bands

2D  $\sigma$  bands

## Fermi surface

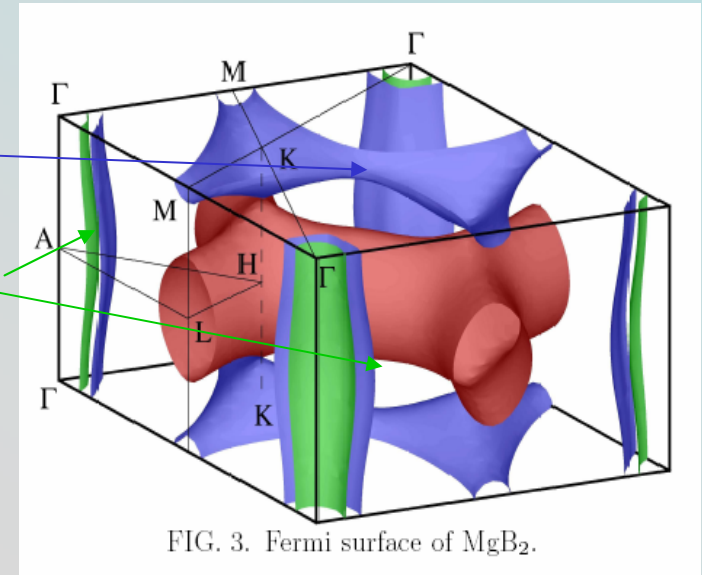


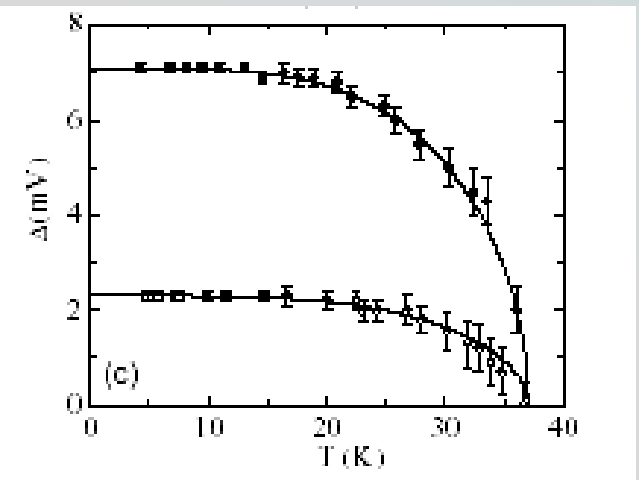
FIG. 3. Fermi surface of MgB<sub>2</sub>.

- Simple layered structure
- Covalent bonding between B atoms
- Conventional superconductivity (isotopic effect)
- Coupling with vibrational modes of B atoms ( $\sigma$  bands)

Weak interband scattering  
due to different symmetry of the  
two bands



The two bands are two conducting channels in parallel:  
crucial role of disorder in coupling them



# A comparison with conventional SC for RF applications

	MgB <sub>2</sub>	Nb
T <sub>c</sub> (K)	39	9.2
ρ <sub>0</sub> (μΩcm)	0.1-10	0.05
RRR	3-30	300
Δ <sub>π,σ</sub> (meV)	2, 7	1.2
2 Δ <sub>π,σ</sub> /K <sub>B</sub> T <sub>c</sub> (meV)	1.6, 4	3.9
ξ <sub>π,σ</sub> (nm)	50, 12	40
λ (nm)	85	80
μ <sub>0</sub> H <sub>c2</sub> (T)	6-50	0.2
R <sup>BCS</sup> <sub>s</sub> @ 4K, 500MHz (nΩ)	2.5/2.3x 10 <sup>-5</sup>	69



$$\text{from } R_s^{BCS} (n\Omega) = \left( \frac{1}{T} \right) 10^5 v_{GHz}^2 e^{(-\Delta/KT_c)}$$

# Pressure-Composition Phase Diagram

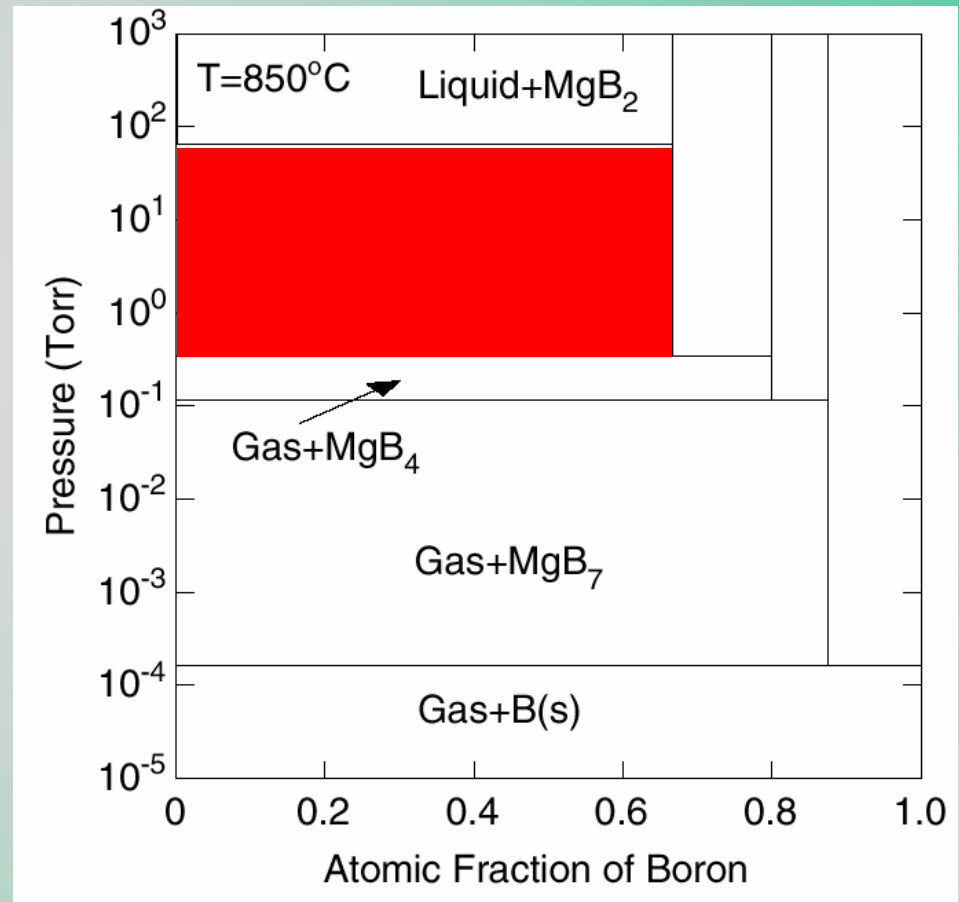
**Process window:** where the thermodynamically stable phases are **Gas+MgB<sub>2</sub>**.

If deposition is to take place at **850°C**, Mg partial pressure has to be above **340 mTorr** to keep the MgB<sub>2</sub> phase stable.

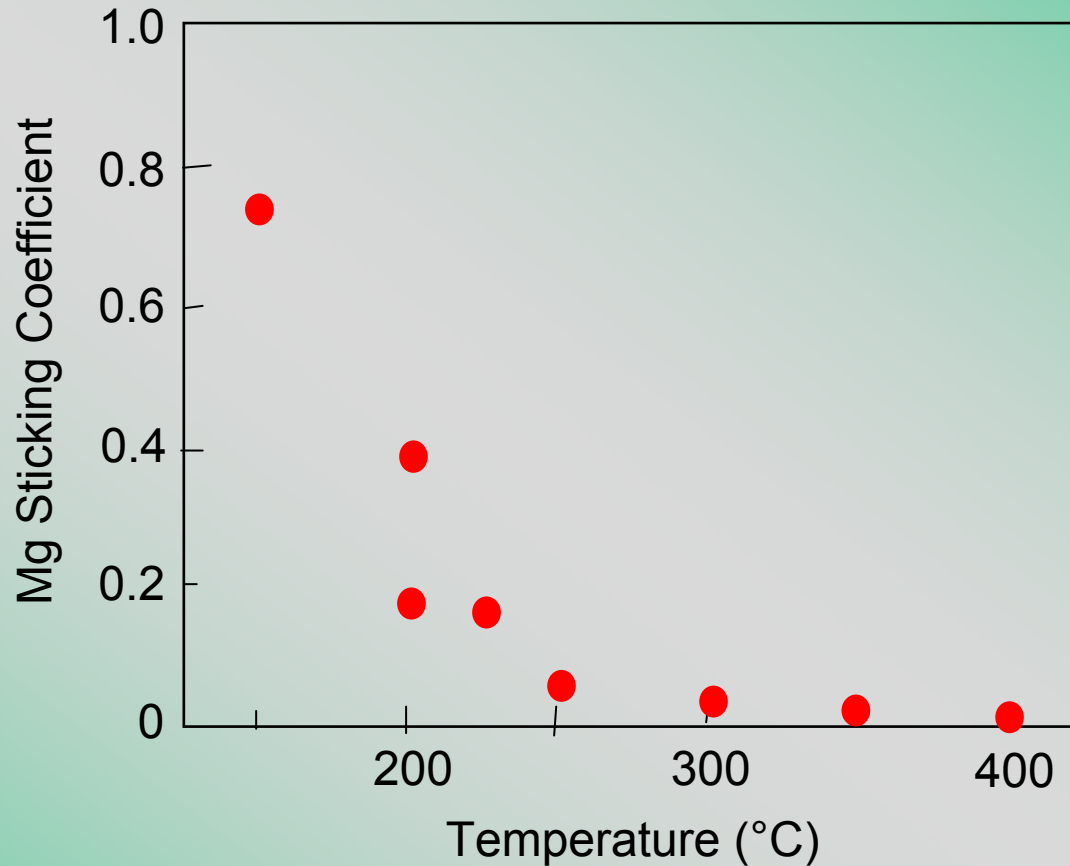
Adsorption-controlled growth: automatic composition control if Mg:B ratio is above 1:2.

**You can provide as much Mg as you want above stoichiometry without affecting the MgB<sub>2</sub> composition.**

**P-x Phase Diagram at 850°C**



# Sticking Coefficient of Mg



Mg sticking coefficient **drops to near zero above 300°C.**

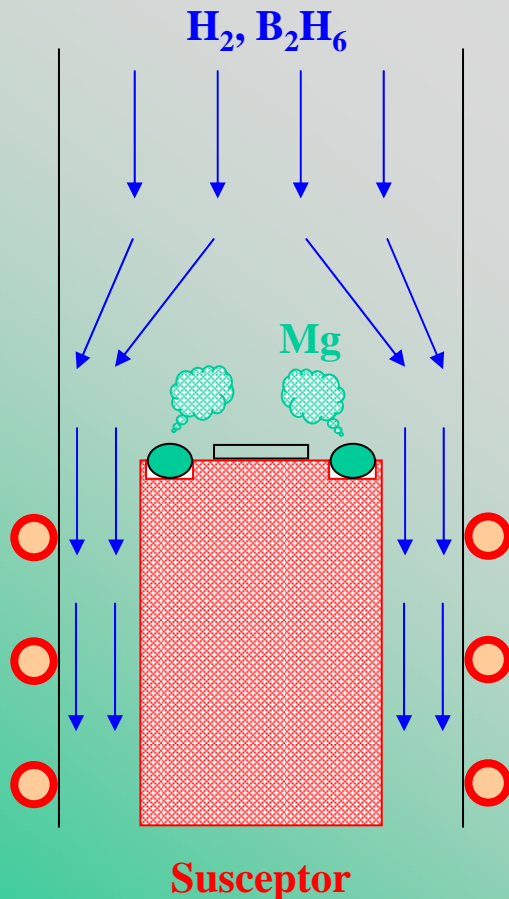
**Not much Mg available to react with B.**

Kim *et al*, IEEE Trans. Appl. Supercond. 13, 3238 (2003)



# Hybrid Physical-Chemical Vapor Deposition

## Schematic View



## Deposition procedure and parameters:

- Purge with N<sub>2</sub>, H<sub>2</sub>
- Carrier gas: H<sub>2</sub>
- $P_{total} = 100$  Torr.
- Inductively heating susceptor, AND Mg, to 550–760 °C.  $P_{Mg} = ?$  (44 mTorr is needed at 750 °C according to thermodynamics)
- Start flow of B<sub>2</sub>H<sub>6</sub> mixture (1000 ppm in H<sub>2</sub>): 25 - 250 sccm. Film starts to grow.
- Total flow: 400 sccm - 1 slm
- Deposition rate: 3 - 57 Å/sec
- Switch off B<sub>2</sub>H<sub>6</sub> flow, turn off heater.

rid of oxygen  
prevent oxidation

make high Mg  
pressure possible

generate high  
Mg pressure

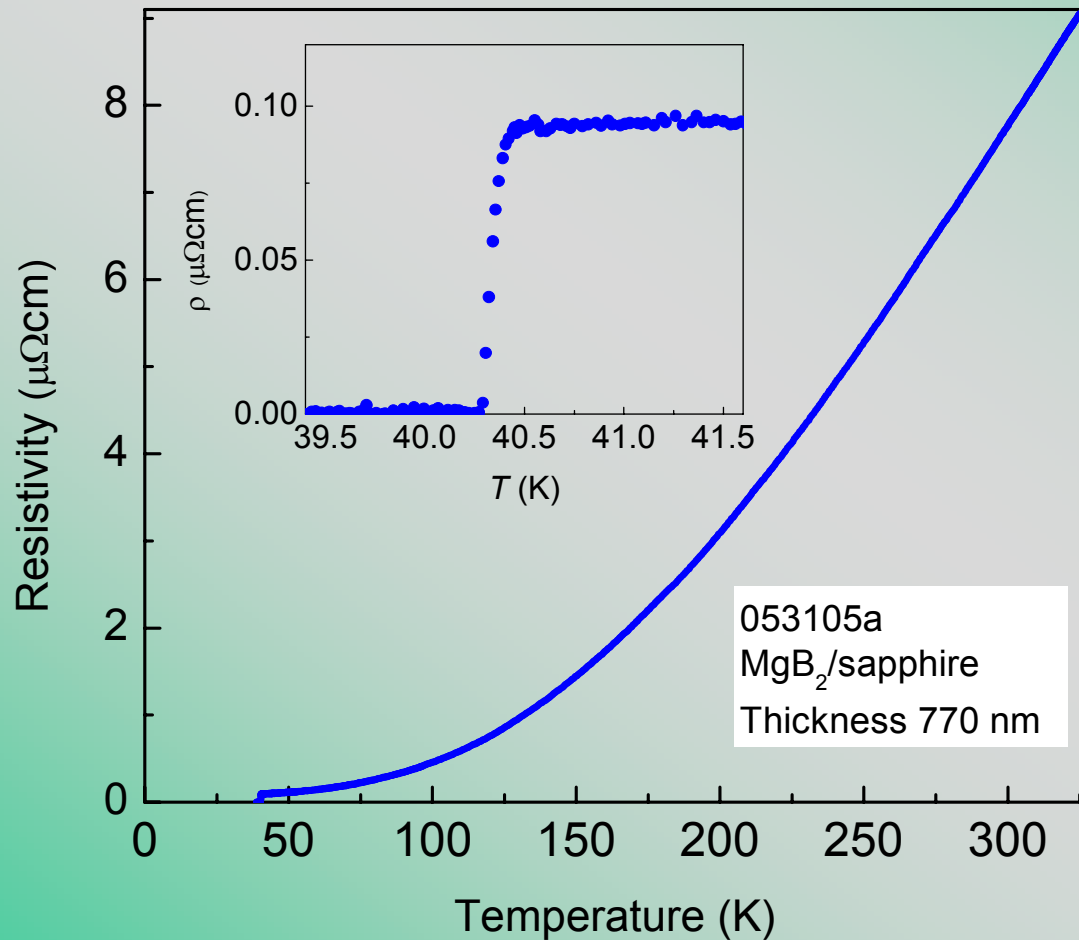
high enough  $T$   
For epitaxy

pure source of B

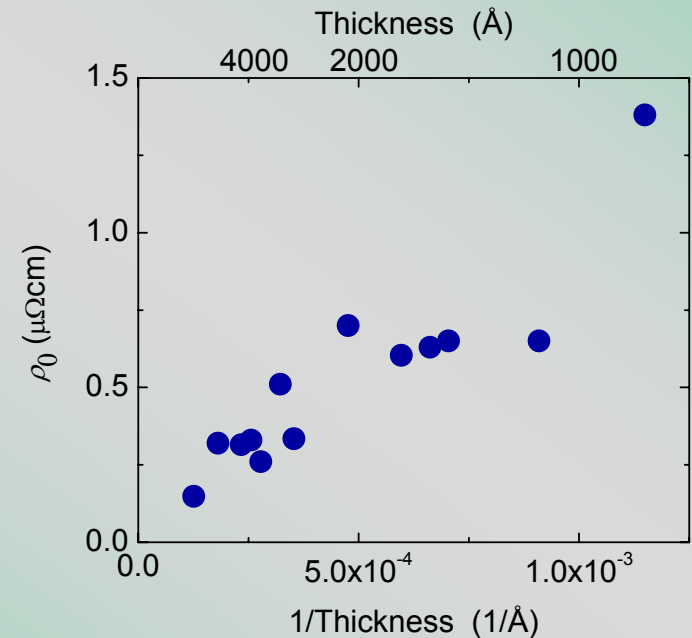
control growth  
rate

low Mg sticking  
no Mg deposit

# Very Clean HPCVD $\text{MgB}_2$ Films: $RRR > 80$



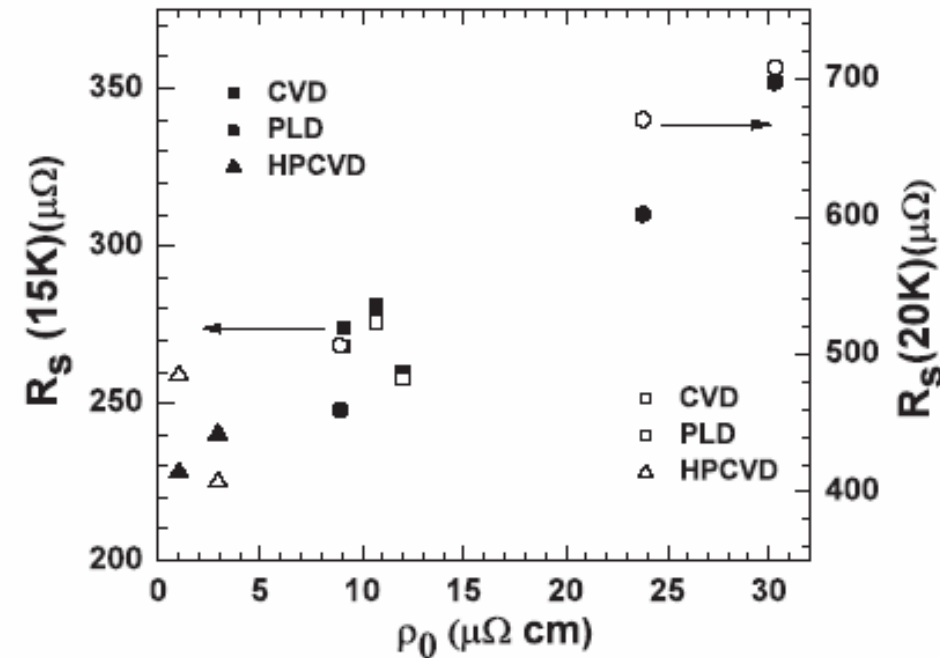
Mean free length is limited by the film thickness.



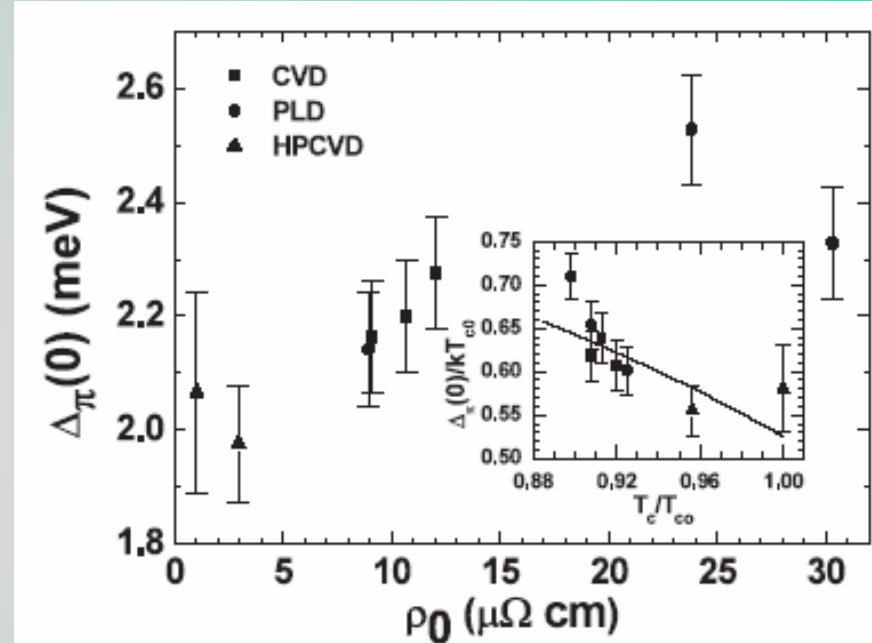
# Good Microwave Properties in Clean Films

Microwave measurement: sapphire resonator technique at 18 GHz.

Surface Resistance @ 18 GHz



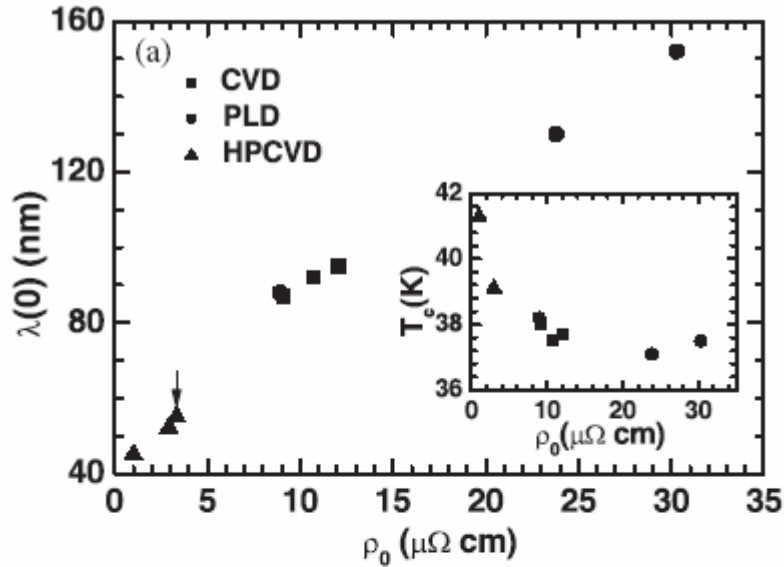
$\pi$ -Band Gap



— Surface resistance decreases with residual resistivity. Clean HPCVD films show low surface resistance.

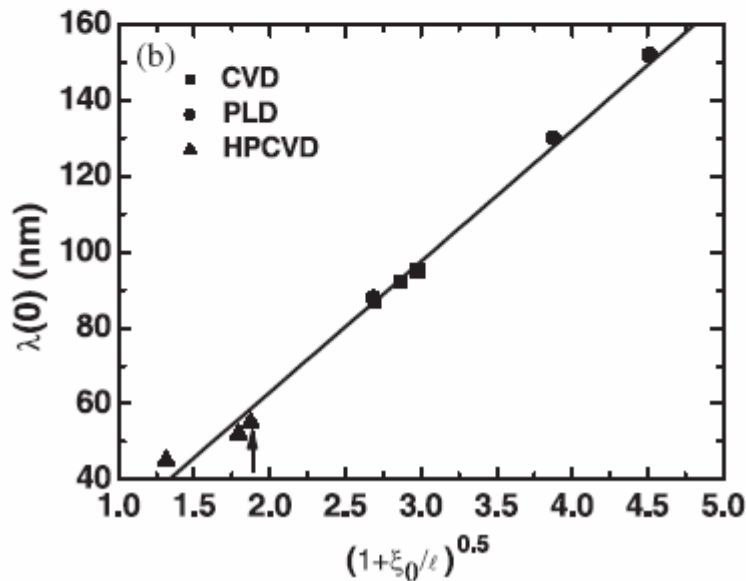
— Interband scattering makes  $\pi$  band gap larger.

# Short Penetration Depth in Clean Films

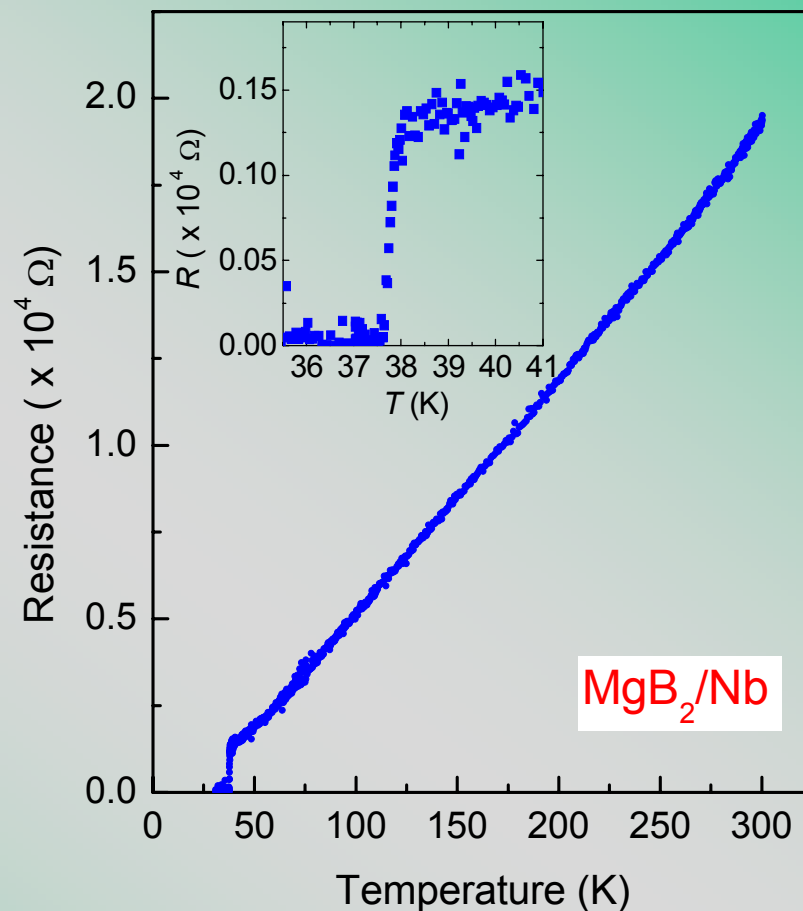
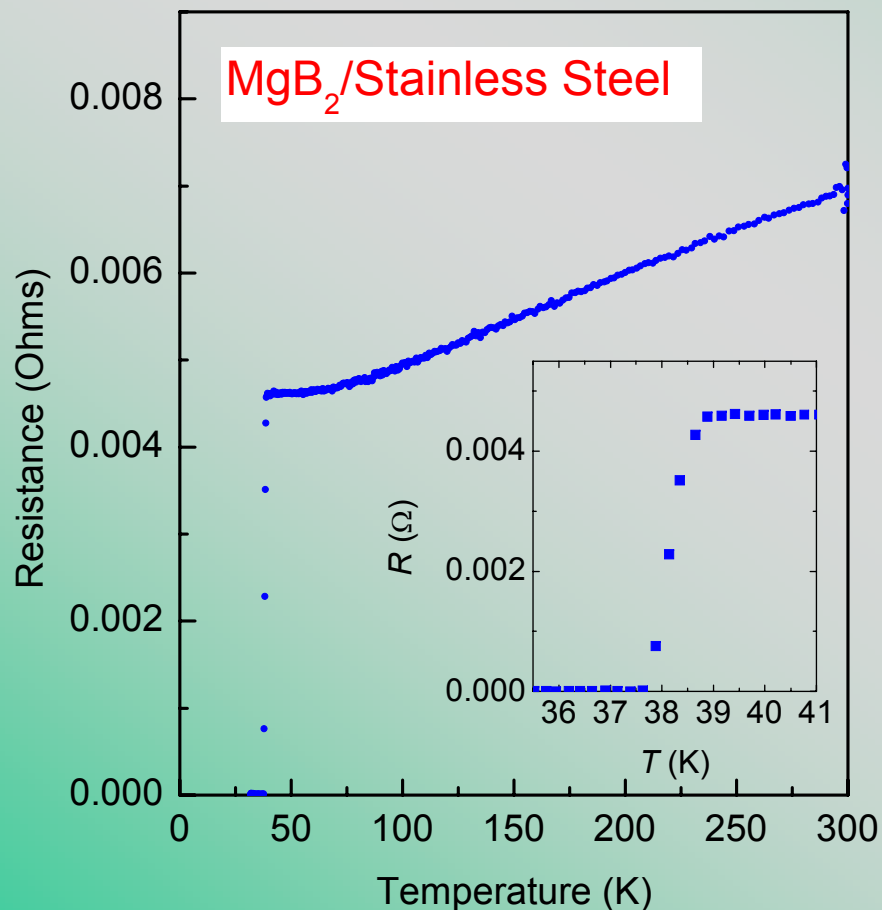


— Penetration depth decrease with residual resistivity.

— London penetration depth  $\lambda_L$ : 34.5 nm



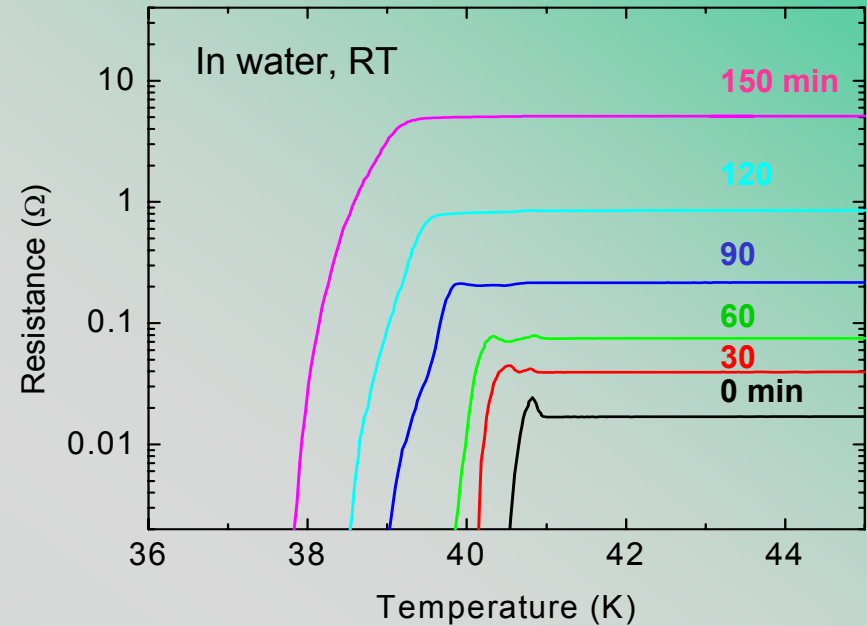
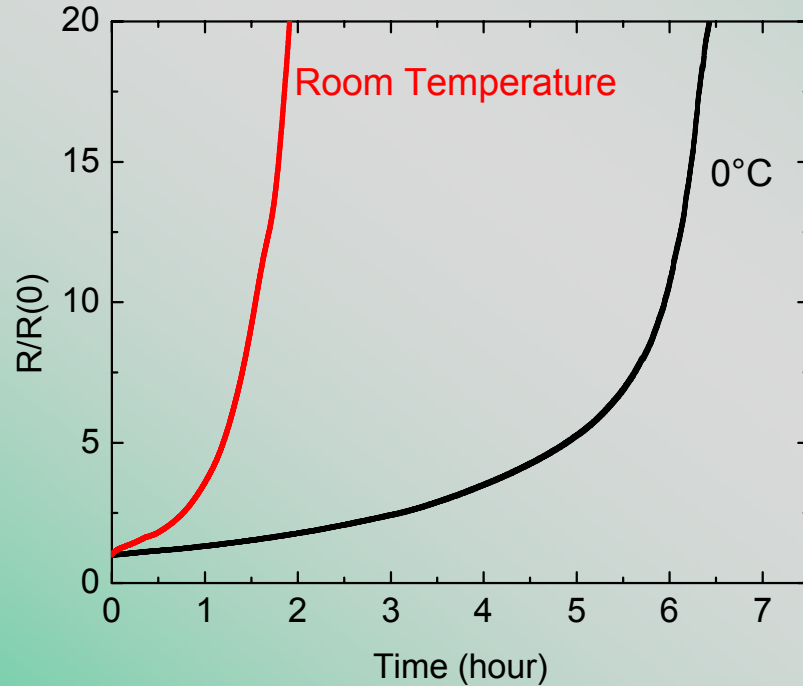
# HPCVD $\text{MgB}_2$ Films on Metal Substrates



High  $T_c$  has been obtained in polycrystalline  $\text{MgB}_2$  films on stainless steel, Nb, TiN, and other substrates.



# Degradation of HPCVD $\text{MgB}_2$ Films in Water

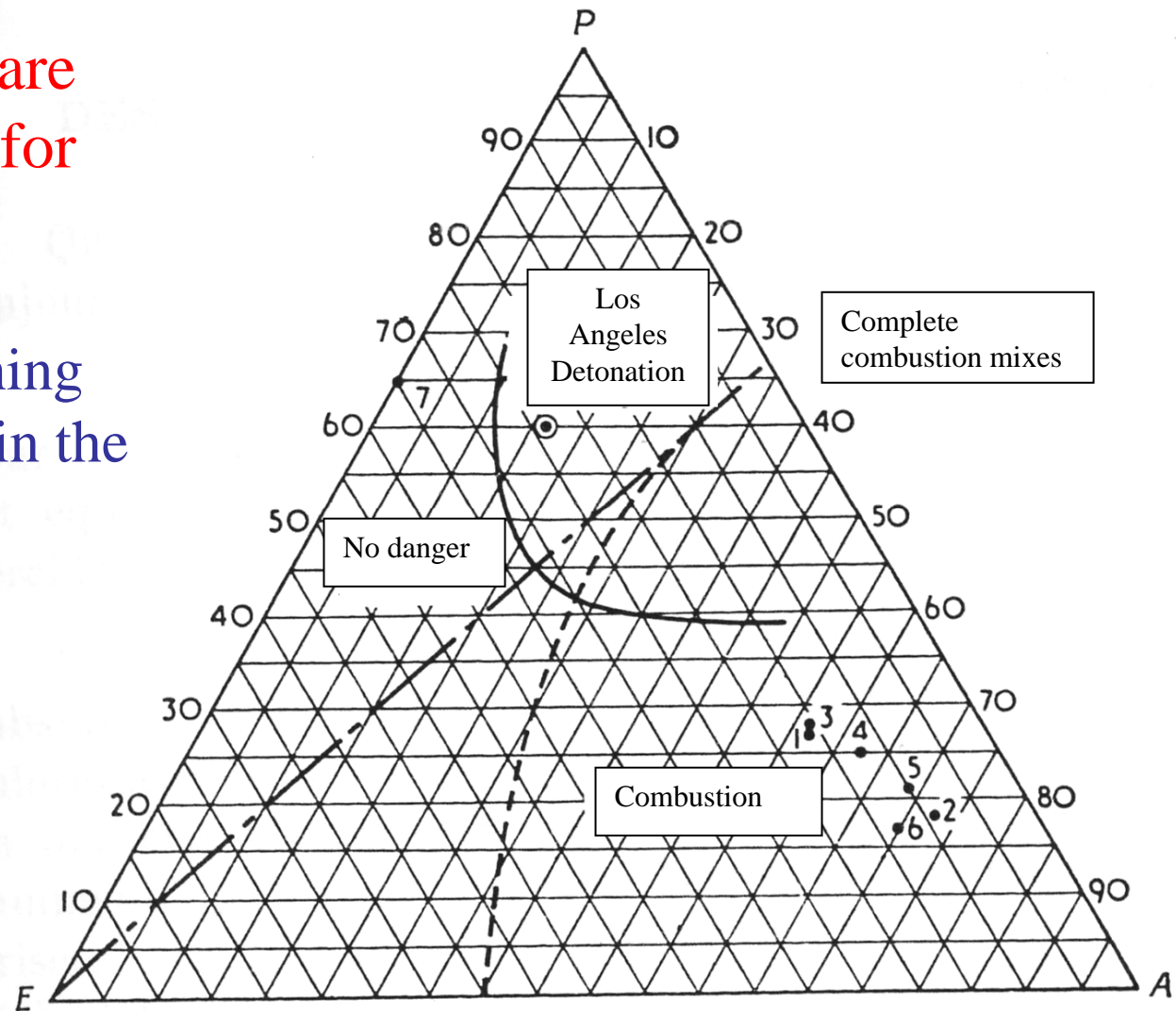


- Film properties degrade with exposure to air/moisture: resistance goes up,  $T_c$  goes down
- Experiments show that  $\text{MgB}_2$  degrades quickly in water, and is sensitive to temperature.

Mixes containing less than 55% in weight of perchloric acid cannot explode

Unfortunately they are  
the less interesting for  
the EP use

The convenient polishing  
baths are just located in the  
flammable field



## Case 3

A woman etching computer chips developed a pin-hole in her glove during the four hours that she was working in a dip tank with 5% hydrogen fluoride. She went to a doctor's office where **a non-specific burn ointment was applied** (no calcium gluconate was applied). She continued to have pain during the next four days. At that time she had severe pain under the finger nail and the subungual tissues were black. There was mild erythema around the proximal cuticle. Upon removal of the finger nail at a burn treatment center where she was referred, exposed and necrotic bone was identified. The distal phalanx was demineralized and the patient required distal amputation of the finger (Edelman, 1986).

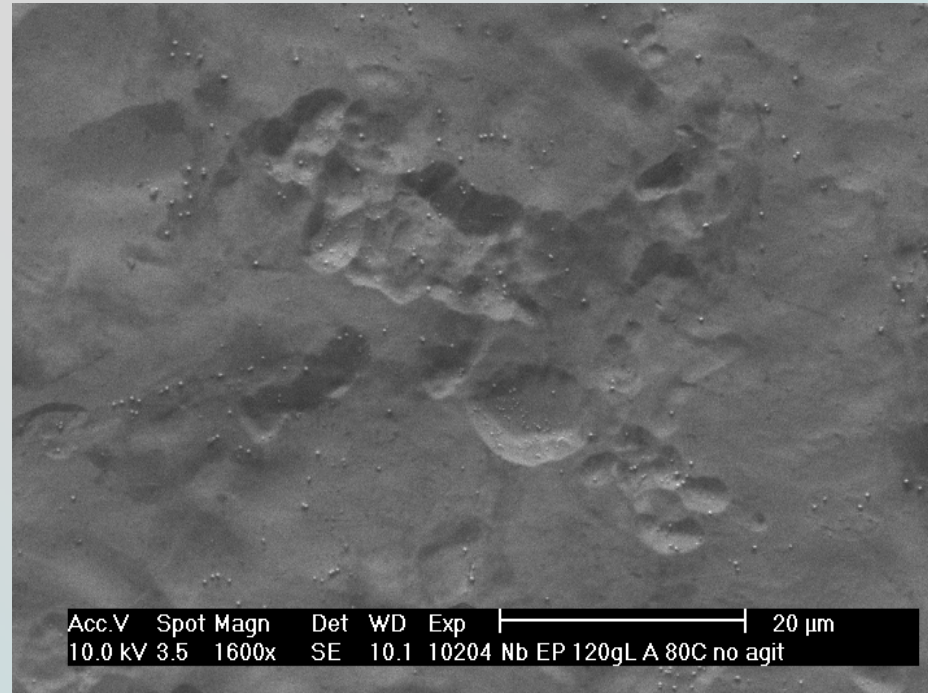
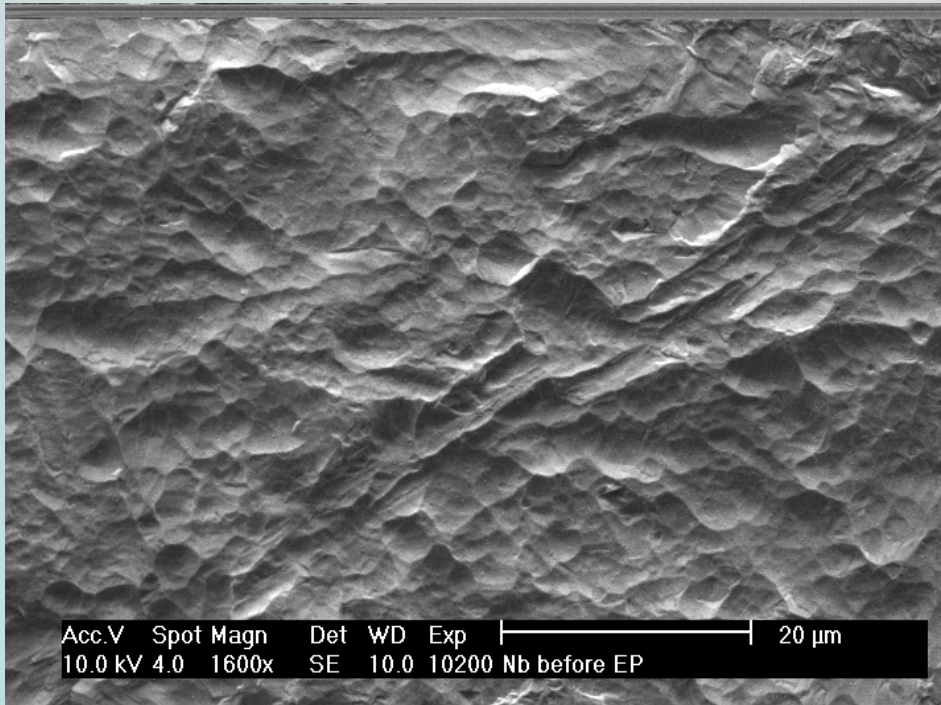
Hydrofluoric acid (HF) differs from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers, including bone.

**Pain associated with exposure to solutions of HF (1-50%) may be delayed for 1-24 hours. If HF is not rapidly neutralized and the fluoride ion bound, tissue destruction may continue for days and result in limb loss or death.**

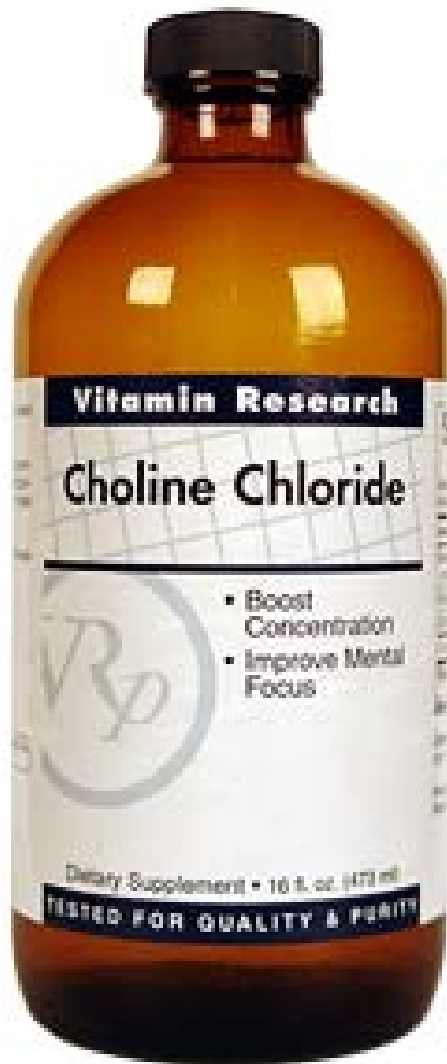
*... Do you really want I continue with case 4?*

On the basis of Abbott Patent,

We succeeded in electropolishing Nb!  
by a mixture of Choline Chloride,  
Urea,  $\text{NH}_4\text{F}$  at  $80^\circ\text{C}$



# Choline Chloride Drink

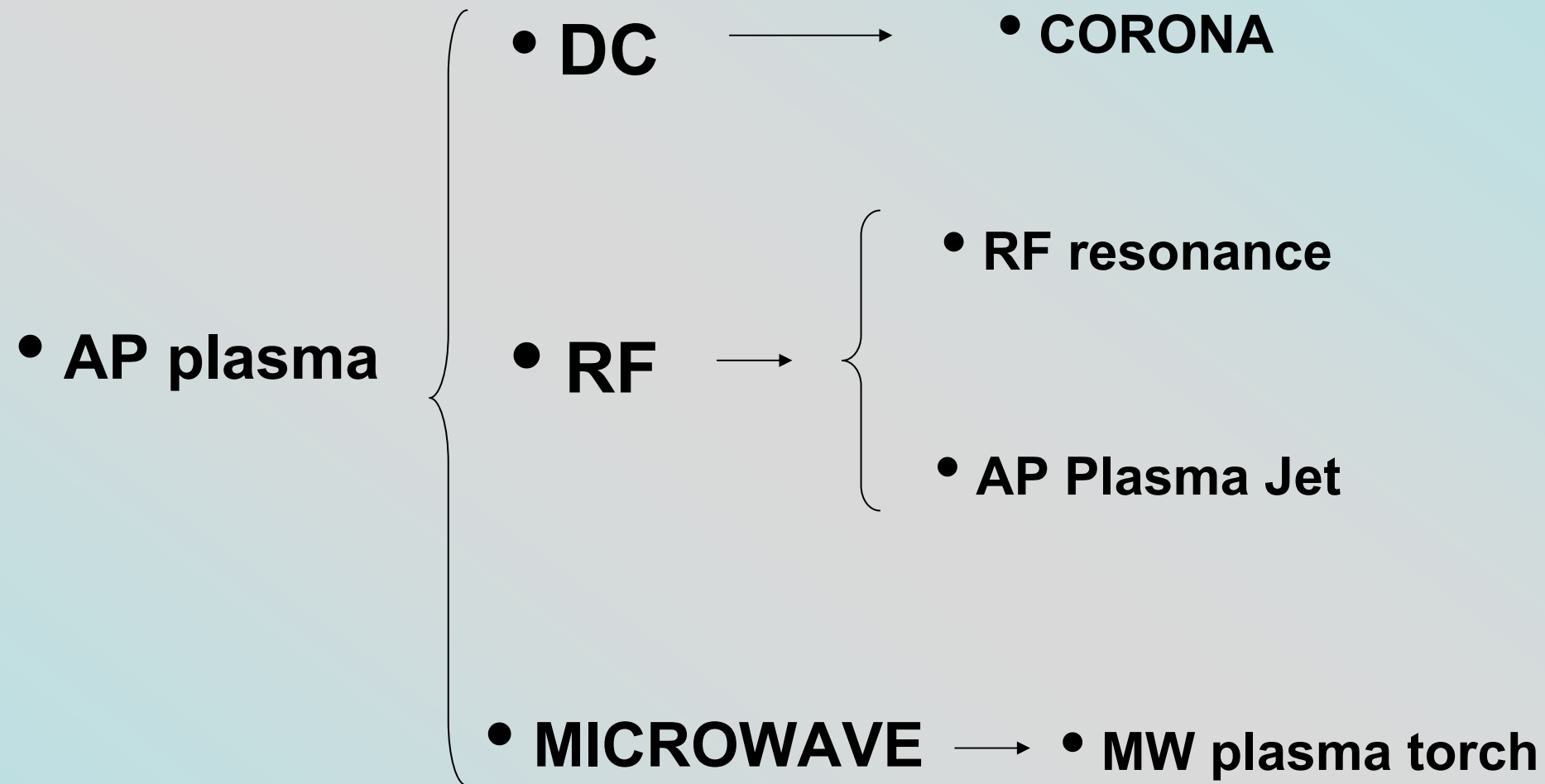


*The brain has a voracious appetite for choline. There are two main reasons for the brain's huge need for this nutrient: Choline is required for synthesis of the key neurotransmitter acetylcholine, and it is used for the building and maintenance of brain cell membranes. Acetylcholine is vital for thought, memory and sleep, and is also involved in the control of movements*

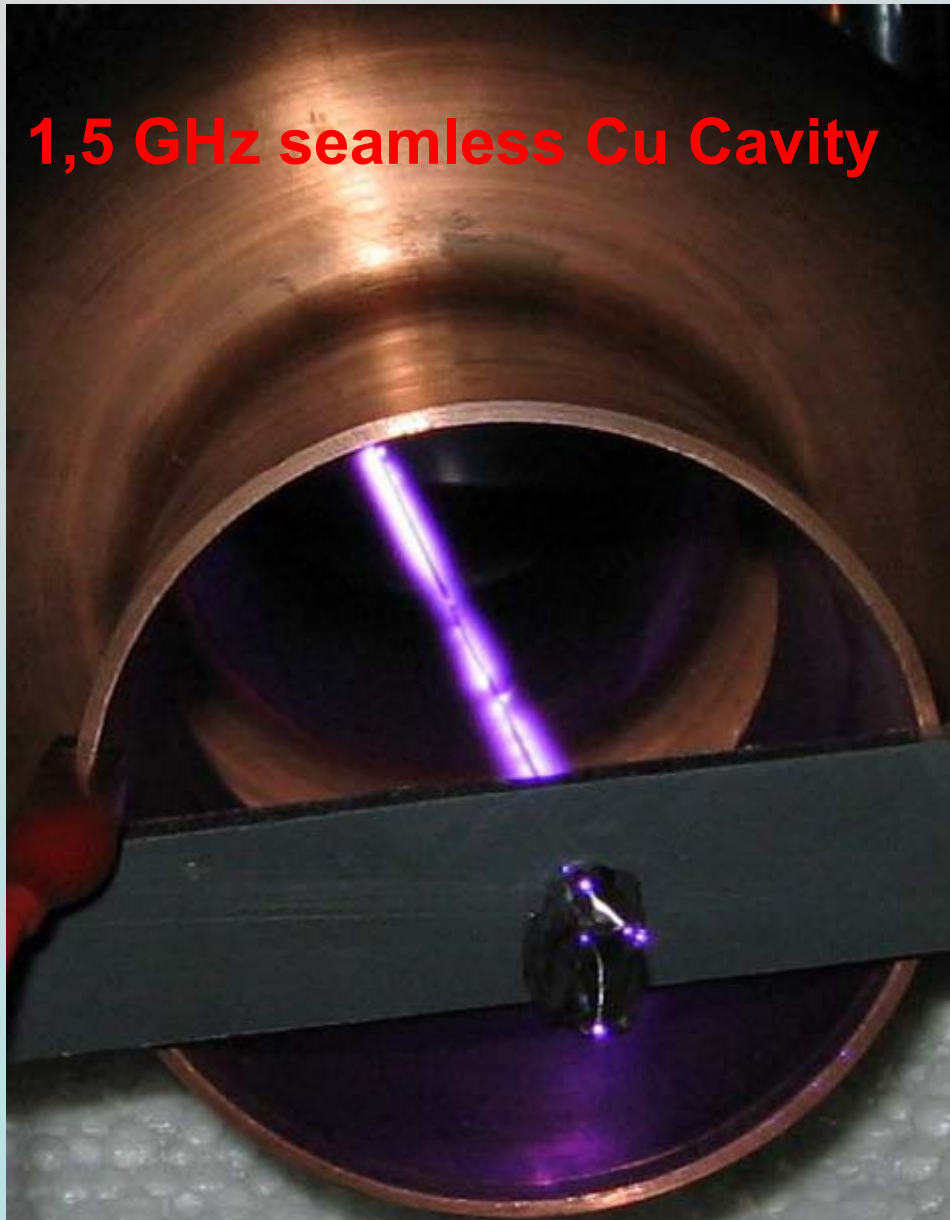
## **Dosage and Use**

Take 1 to 3 teaspoons daily.  
It is best mixed with approximately  
2 oz. of juice per teaspoon.





# The early stage of our studies



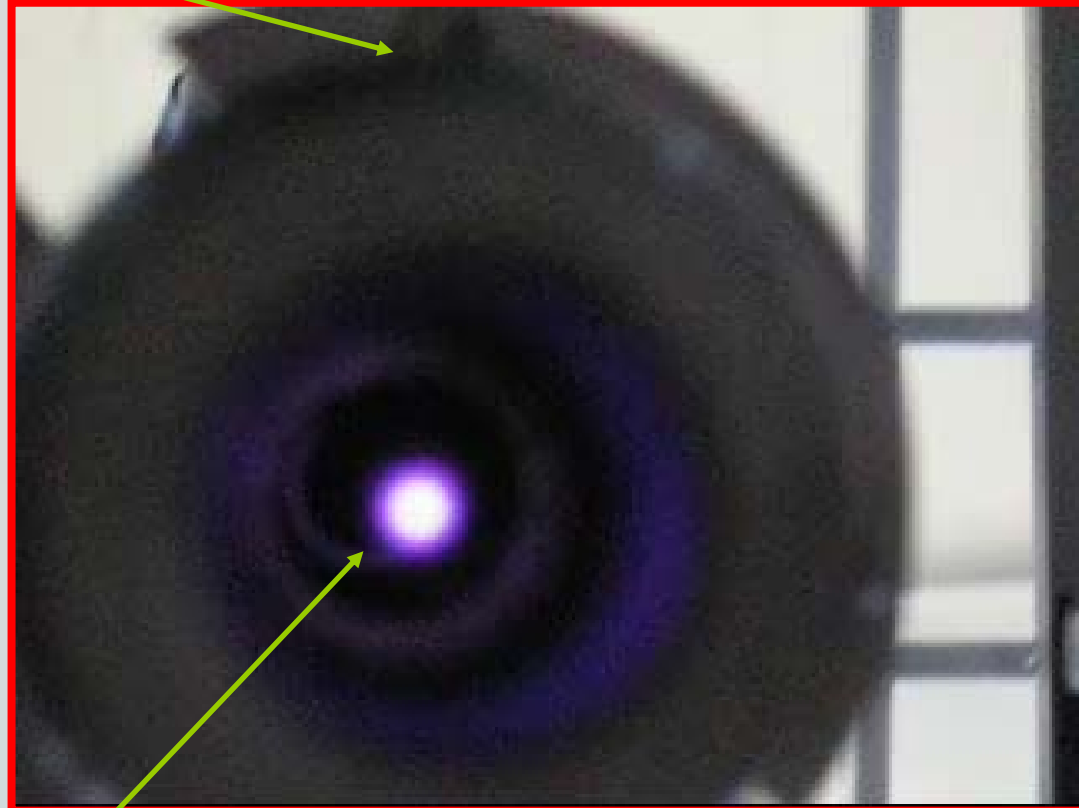
1,5 GHz seamless Cu Cavity

- Negative Corona inside a 1,5 GHz cavity
- Discharge voltage 30kV
- Strong production of  $O_3$

# 6 GHz cavity

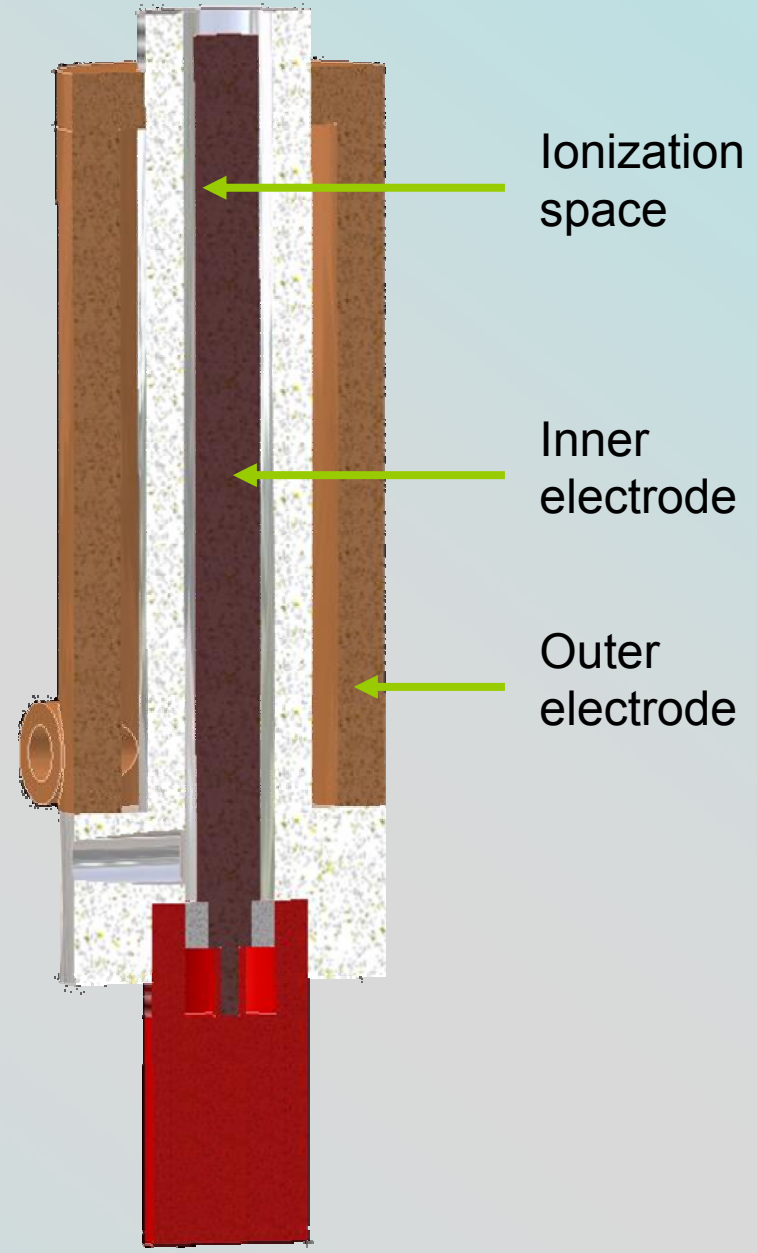
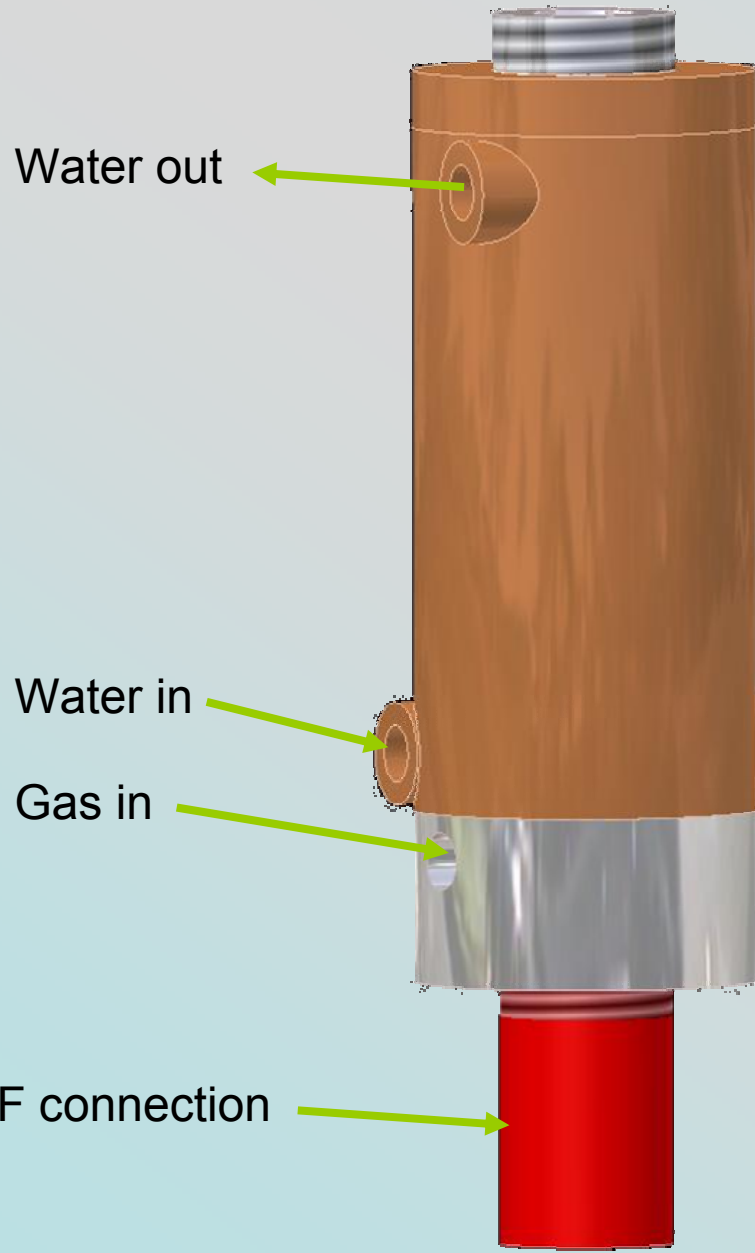


Cavity

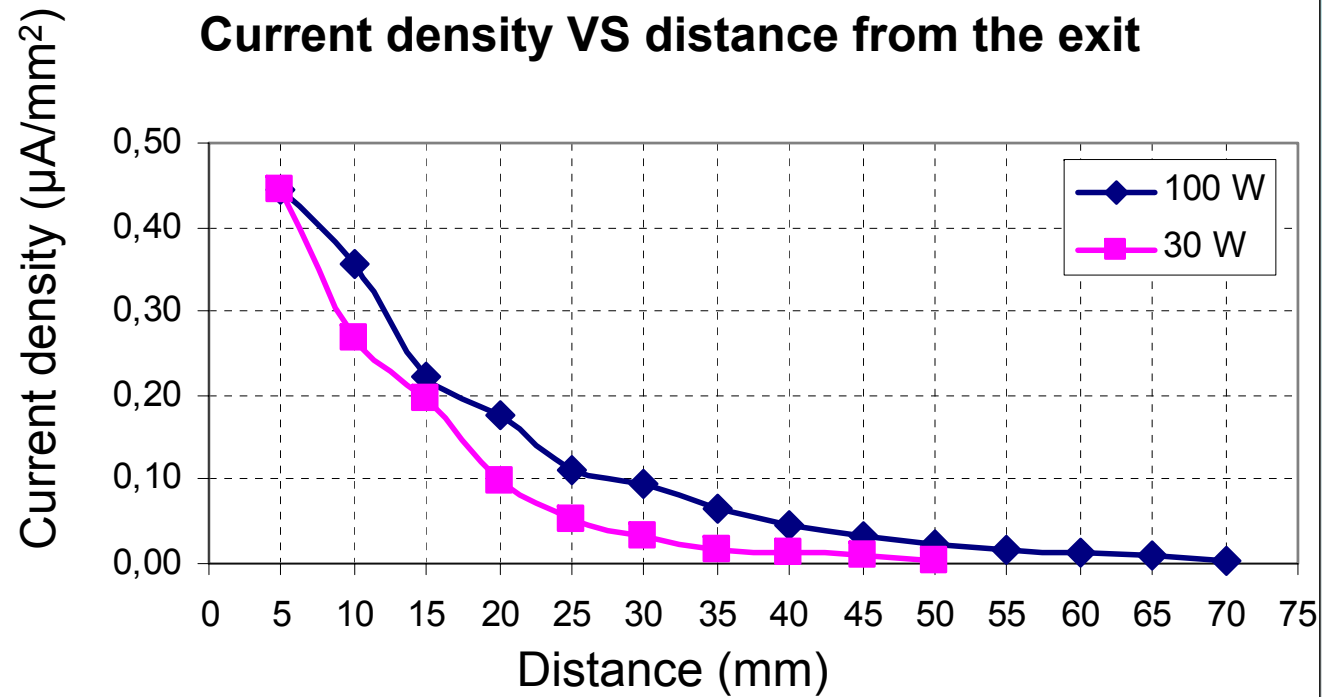


TM010  
plasma at  
a power of  
50 W

# 13,56 MHz / 2,45 GHz APPJ Device

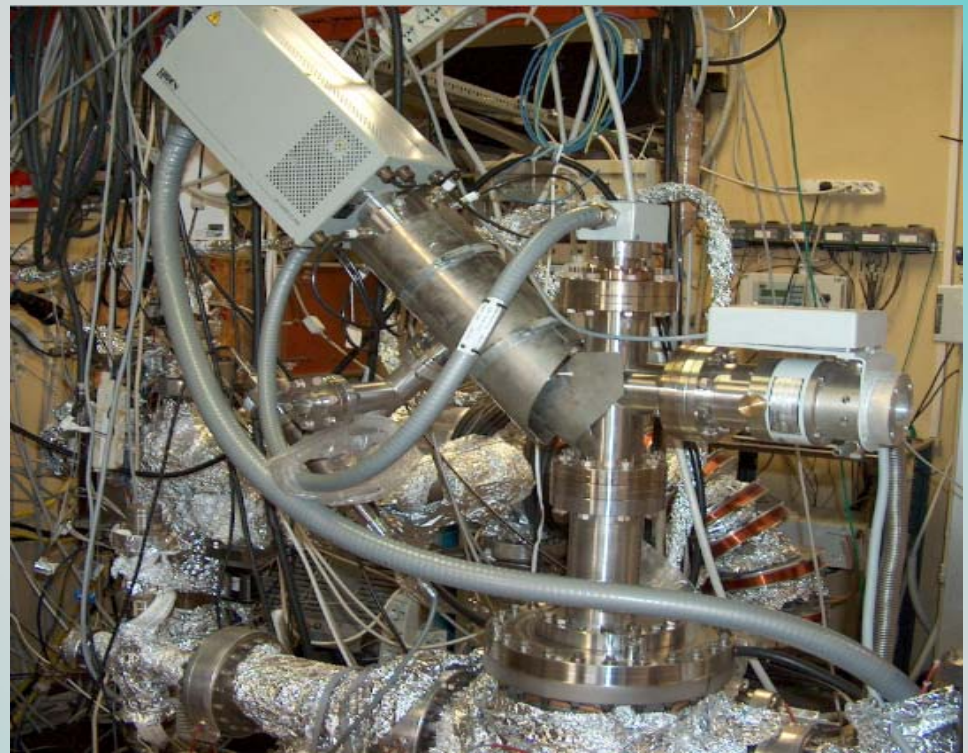
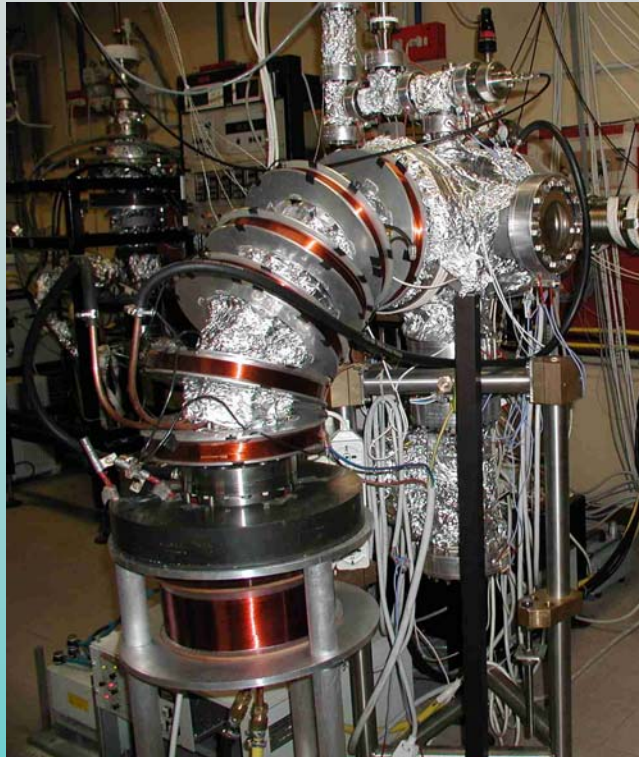


# 13,56 MHz





**To reduce an amount of micro-droplets one can use a magnetic filter deflecting a plasma-ion stream and separating micro-droplets.**



**Magnetic filter (on the left) and ion energy analyzer (on the right), as installed upon the second planar-arc facility in Tor Vergata University in 2005.**

# **UHV planar-arc sources constructed at IPJ (Poland)**



A new filtered planar-arc system operated  
at the Dept. of Plasma Physics &  
Technology.

The detailed description  
of the UHV arc facilities and  
their operational  
characteristics can be found  
in our previous papers.

J. Langner, M.J. Sadowski, et al.,  
*Czech. J. Phys.* 54 (2004) C914.

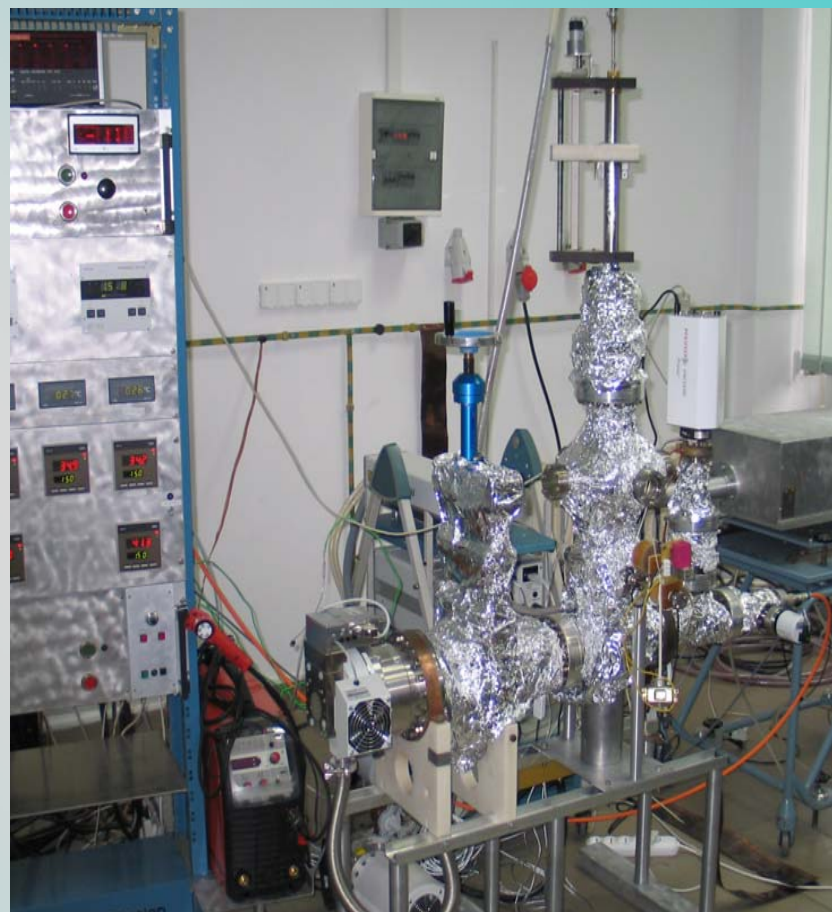
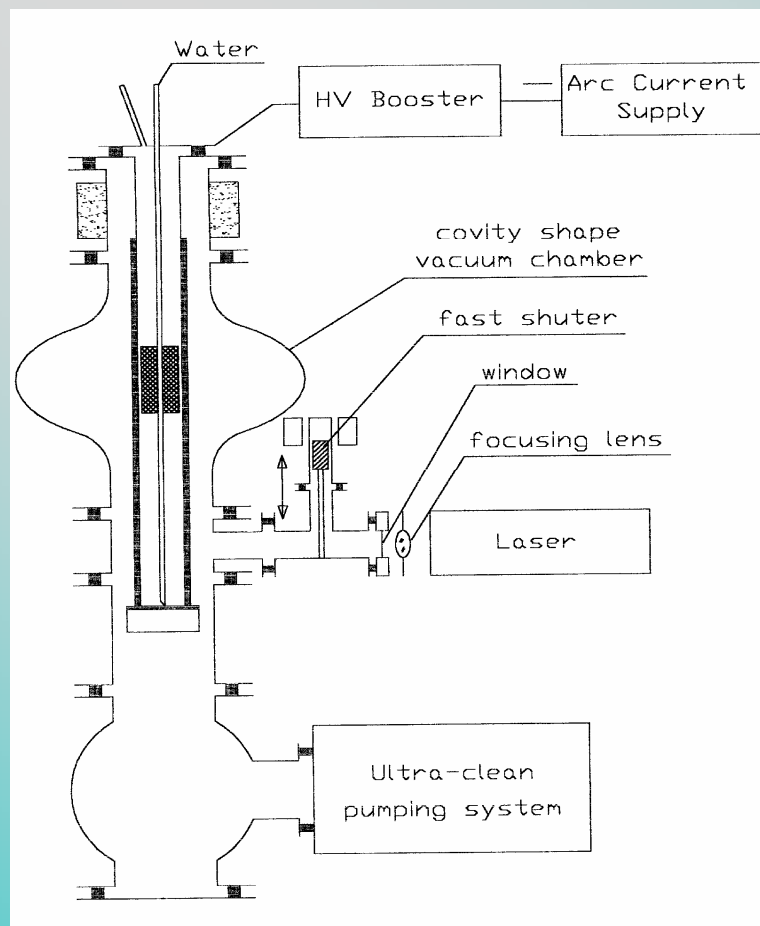
R. Russo, L. Catani, et al.,  
*Supercond. Sci. Tech.* 18 (2005) L41.

**P. Strzyzewski, J. Langner, et al.,  
*Physica Scripta* T123 (2006) 135.**

**P. Strzyzewski, L. Catani, et al.,  
*AIP CP* 812 (2006) 485.**



# UHV arc systems with a linear cathode



Scheme of the UHV system with a linear (cylindrical) arc and the first linear-arc facility constructed at IPJ in Swierk, Poland, in 2005.

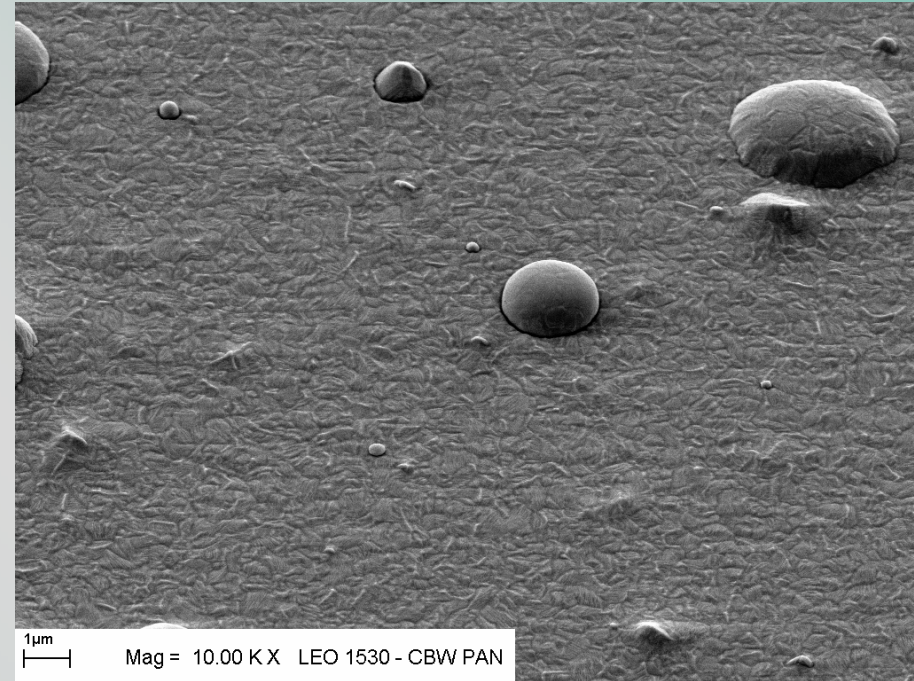
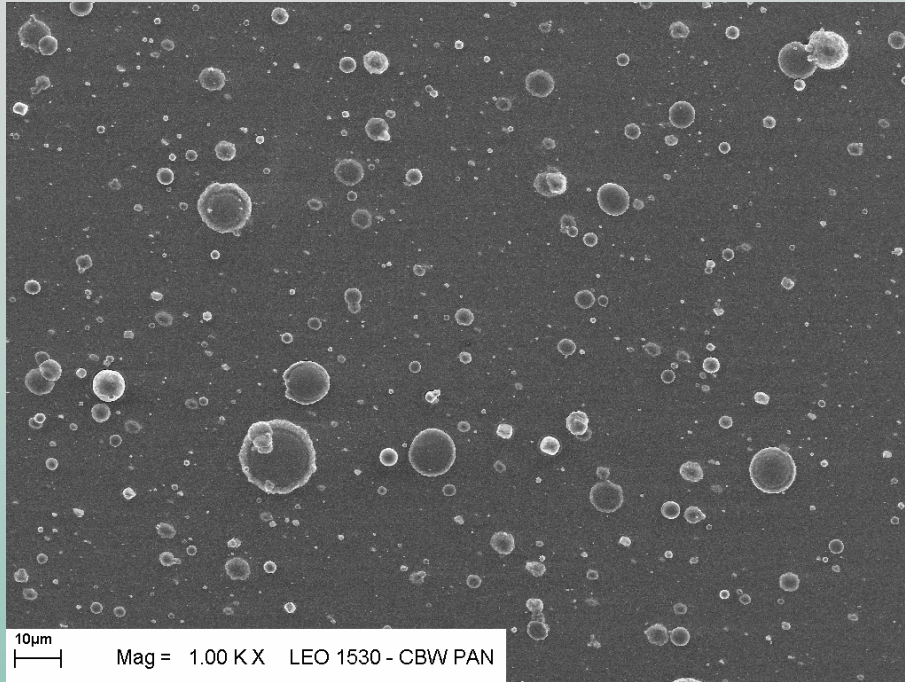
# Preliminary test for cavity coating

**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



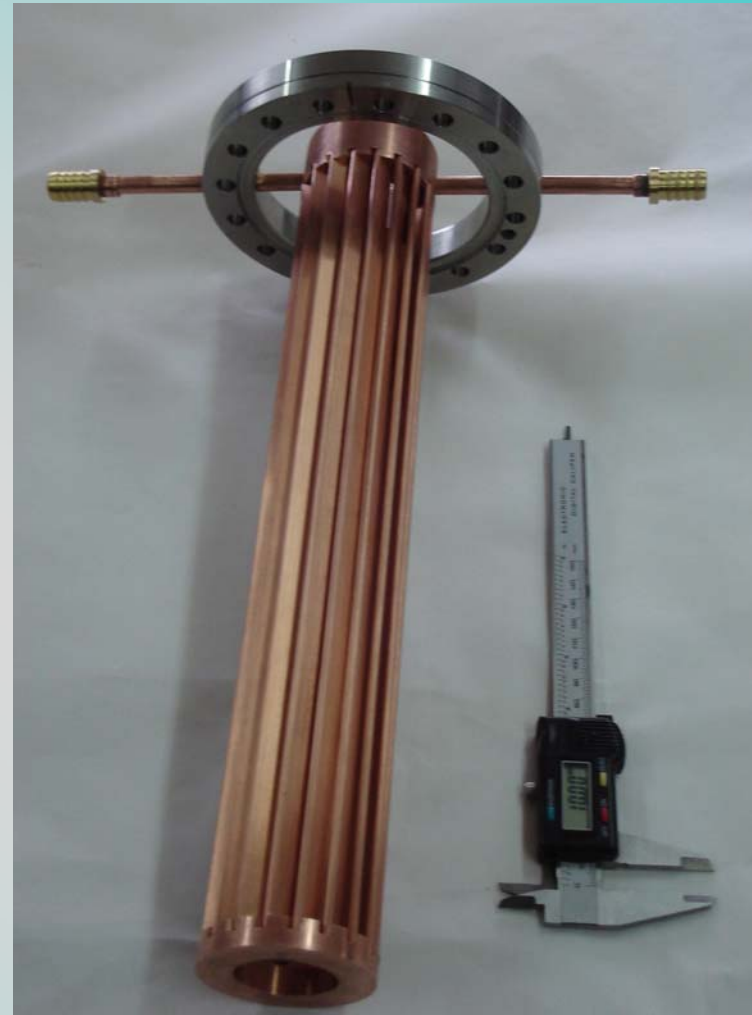
There is a larger number of macrodroplets respect to the planar arc (as aspected)

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



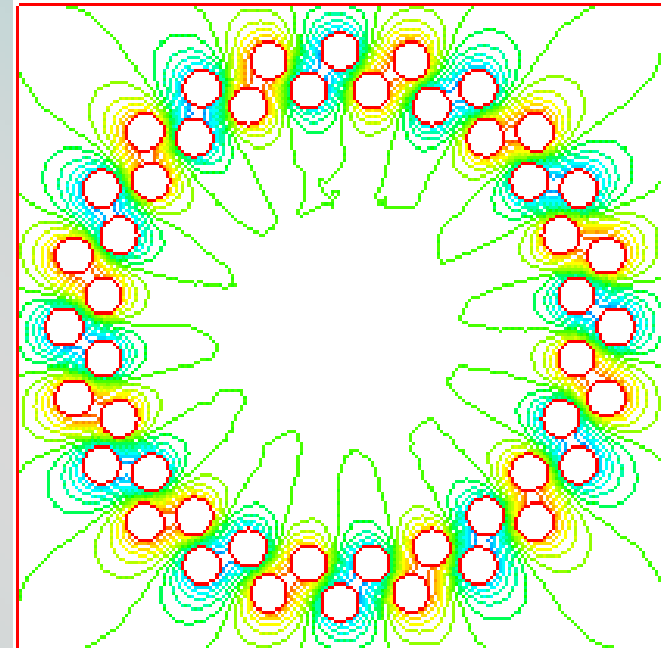
**To eliminate micro-droplets (macro-particles) or at least to reduce their amount in the UHV linear-arc facility - special cylindrical filters have been considered.**

Two different versions of such filters have been designed: a passive Venetian-type filter and an active cylindrical filter (supplied by a magnetizing current and producing an additional magnetic field).



Prototype of a cylindrical Venetian-blind filter.

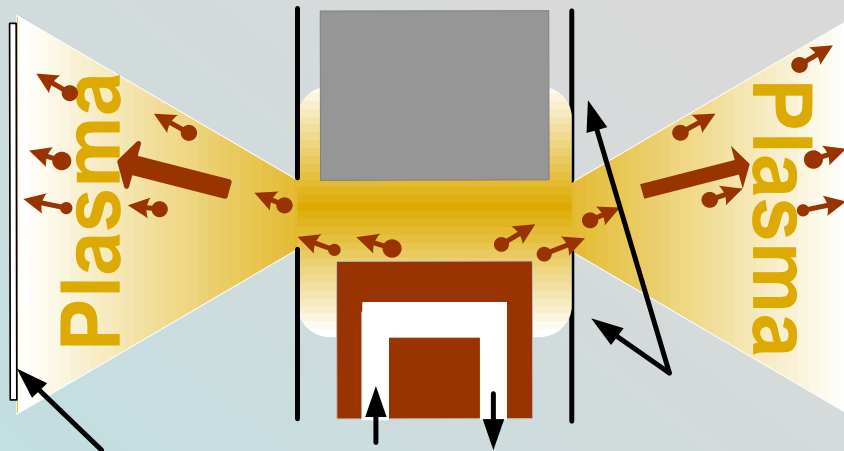
# New cylindrical filters for elimination of micro-droplets

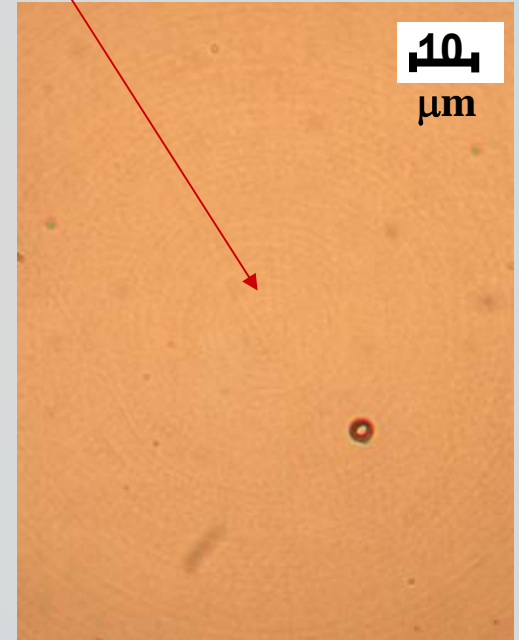
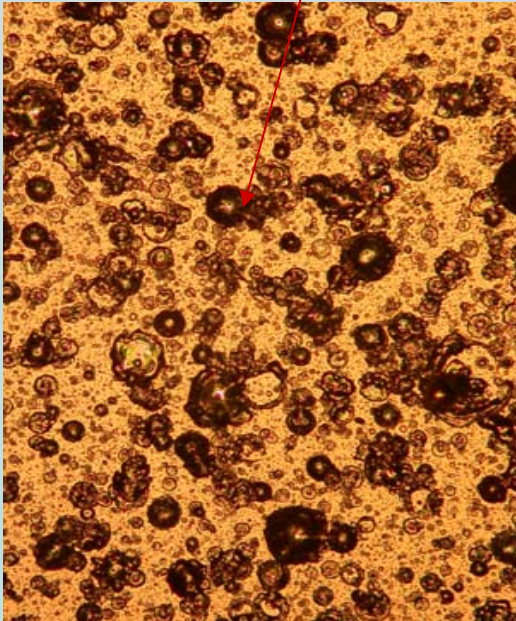
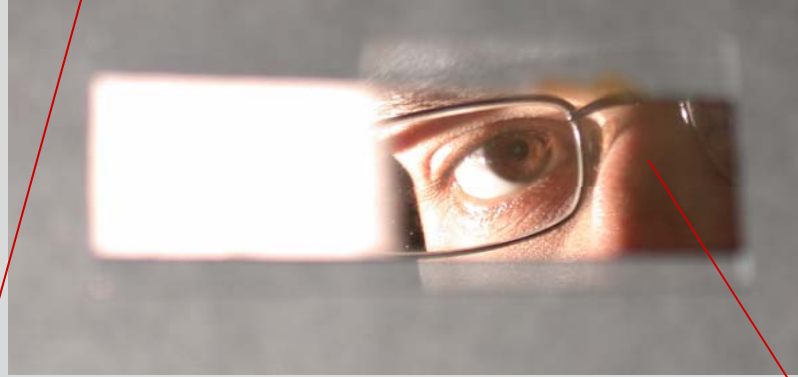


A cylindrical magnetic filter consisting of current-carrying tubes (left) and the distribution of magnetic field lines in its cross-section (right).

## Other Arc Modes proposed by Ray Boxman

- Hot Anode Vacuum Arc
  - Crucible anode
- Hot Refractory Anode Vacuum



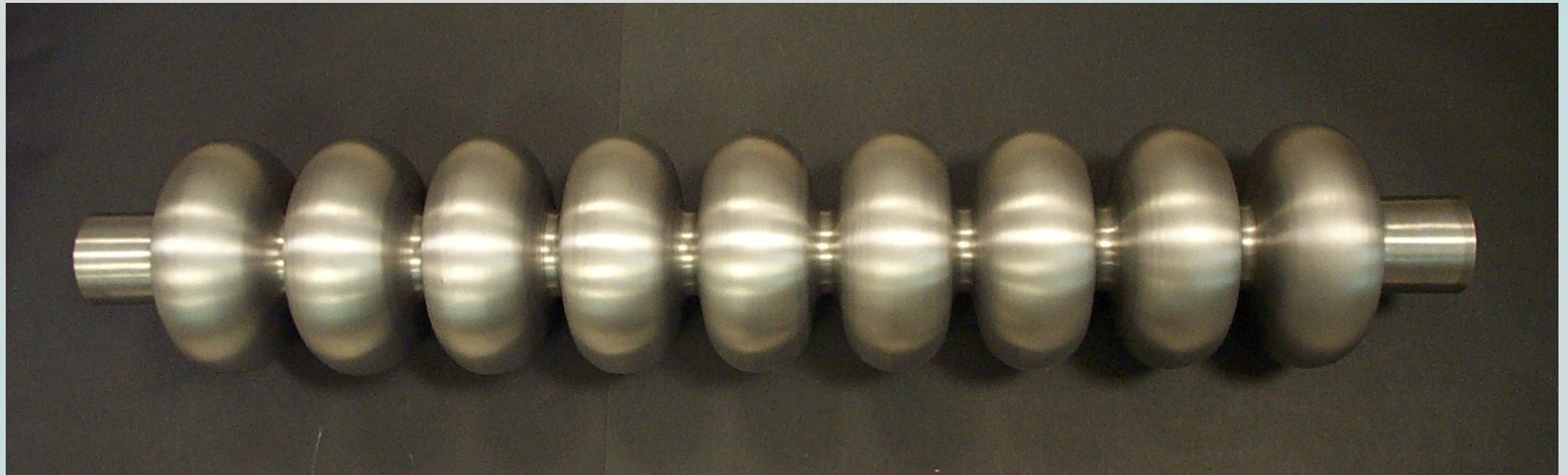


# Ray Boxman

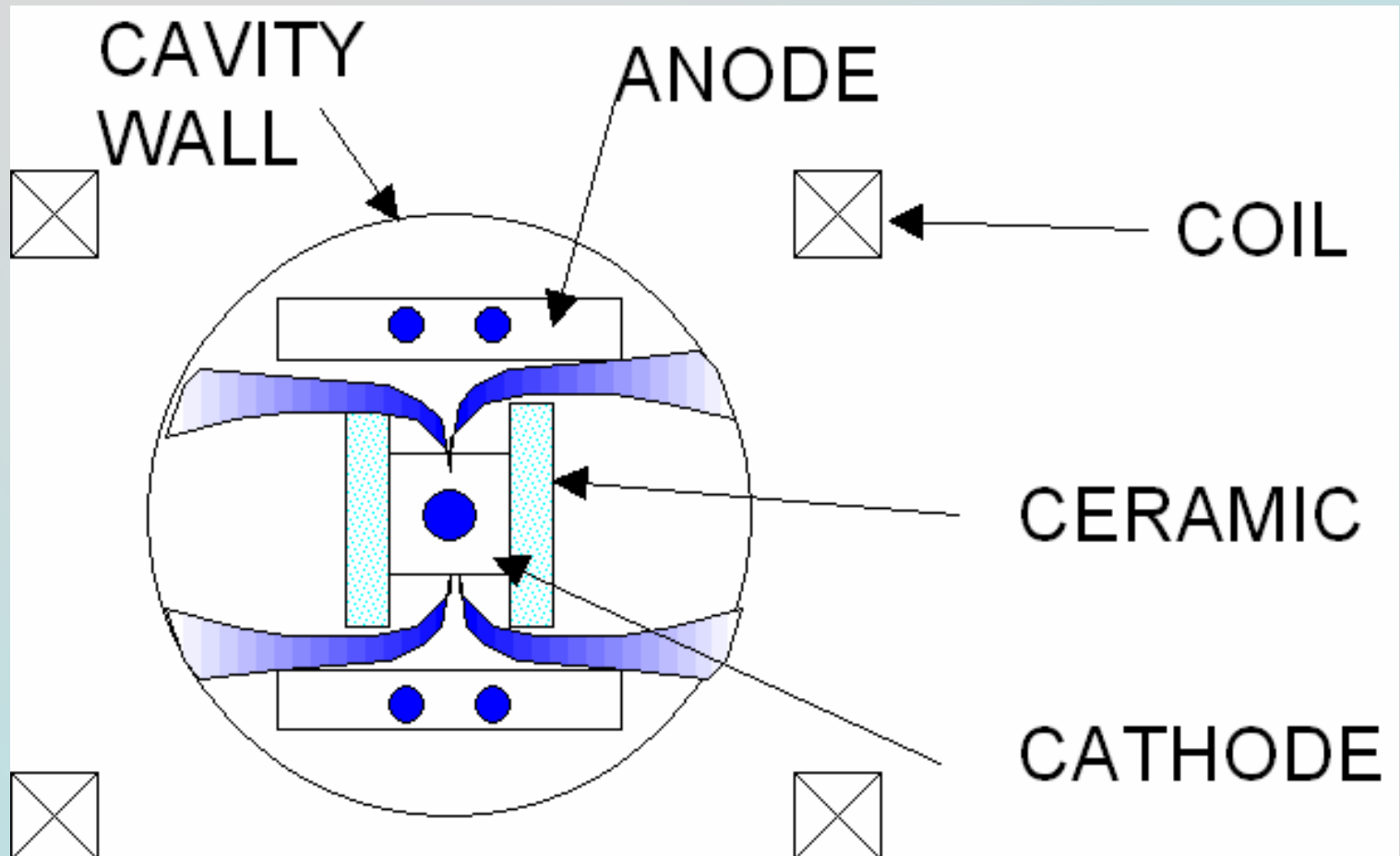
- Vacuum Arc Deposition
  - (a.k.a. cathode arc deposition, arc evaporation)
  - Most popular method for applying hard coatings in tool industry
  - ...but less well known than other PVD (e.g. sputtering, e-beam evaporation) and CVD methods
- Objectives of this lecture:
  - Review:
    - Physics of vacuum arc
    - Engineering issues in vacuum arc deposition
  - Suggest implementations with interior cavity



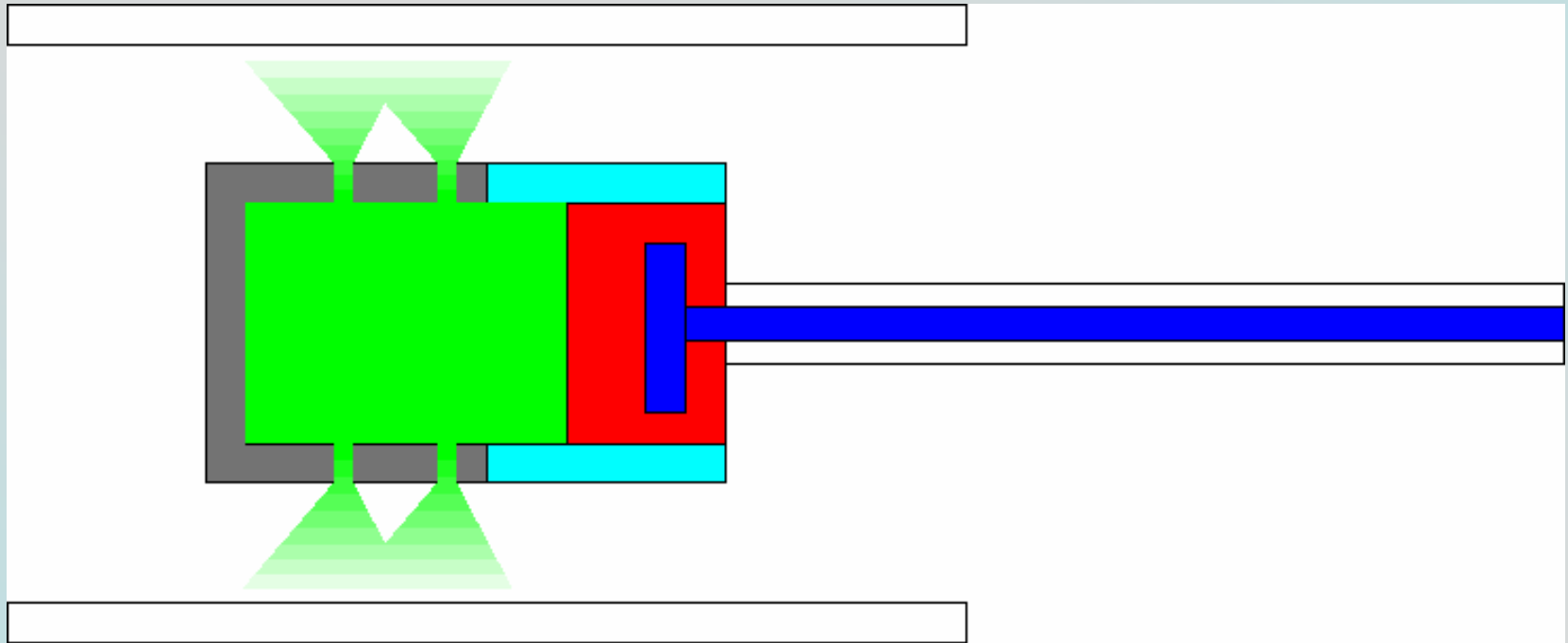
### III. How can we coat the inside of:



# Approach 2: Miniature Filter: Example – Welty Rect. Filter



# Approach III. Beilis “black-body” HRAVA deposition device



## JUST AN EXAMPLE OF HOW TO WORK:

Xiaoxing Xi group (Physics and Materials Sci & Eng); Ke Chen, Derek Wilke, Yi Cui, Chenggang Zhuang (Beijing), Arsen Soukiassian, Valeria Ferrando (Genoa); Pasquale Orgiani (Naples); Alexej Pogrebnyakov, Dmitri Tenne, Xianghui Zeng, Baoting Liu: CVD growth, electrical characterization, junctions

Joan Redwing Group (Materials Sci & Eng): HPCVD growth, modeling

Qi Li Group (Physics): Junctions, transport and magnetic measurements

Darrell Schlom Group (Materials Sci & Eng): structural analysis

Zi-Kui Liu Group (Materials Sci & Eng): Thermodynamics

Xiaoqing Pan Group (U. Michigan): Cross-Section TEM

John Spence Group (ASU): TEM

N. Klein Group (Jülich): Microwave measurement

A. Findikoglu (LANL): Microwave measurement

Qiang Li Group (Brookhaven National Lab): Magneto-optic measurement

Tom Johansen Group (U Oslo): Magneto-optic measurement

Qing-Rong Feng Group (Peking University): SiC fiber

Chang-Beom Eom Group (U Wisconsin): Structural analysis

J. B. Betts and C. H. Mielke (LANL): High field measurement



