



18<sup>th</sup> International Conference on  
RF Superconductivity  
Lanzhou China  
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# Alternative coating techniques and materials for SRF cavities

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1: CERN: TE-VSC/SCC

2: CERN: BE-RF/SRF

3: CERN: TE-VSC/VSM

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18/07/17

# Outline

1. Context

2. Overview

3. @ CERN – HiPIMS and A15

4. Summary - Perspectives

# 1. Context



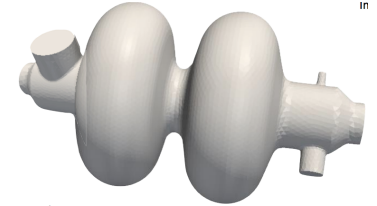
# 1.1 Coated SRF Cavities



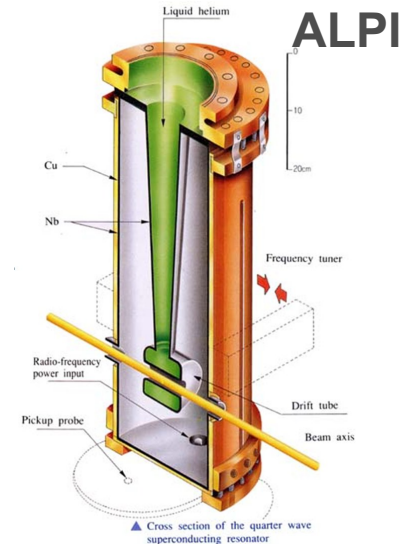
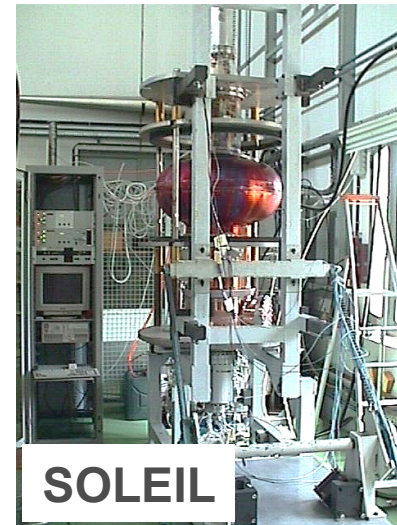
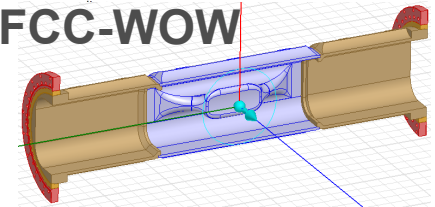
- Thermal stability
  - No quench
  - Higher working temperature
- Low magnetic field sensitivity
- Low fabrication costs



**FCC**



**FCC-WOW**



# 1.2 Coating Techniques

## Direct Current Magnetron Sputtering

Adapted to elliptical cavities

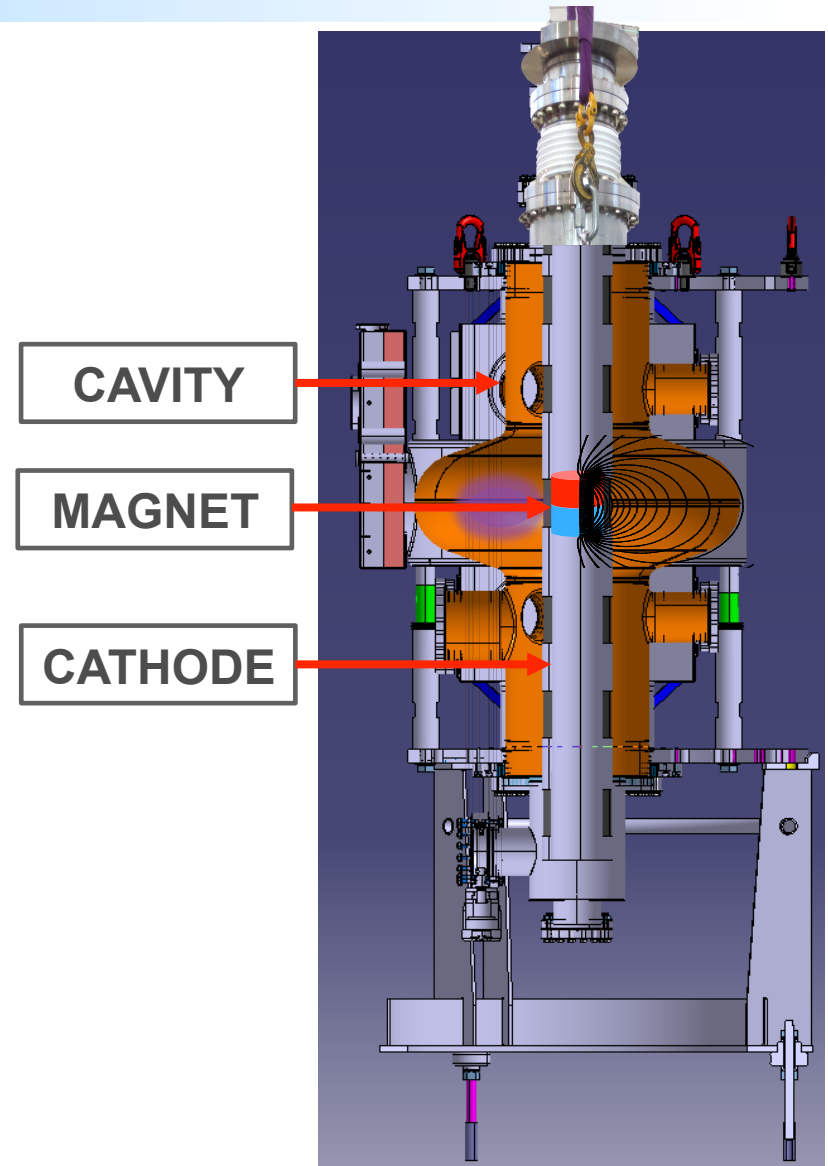
Low working pressure ( $10^{-4}$  up to  $10^{-2}$  mbar)

Kr or Ar as sputtering gas  
 $10$ 's  $\text{W}\cdot\text{cm}^{-2}$

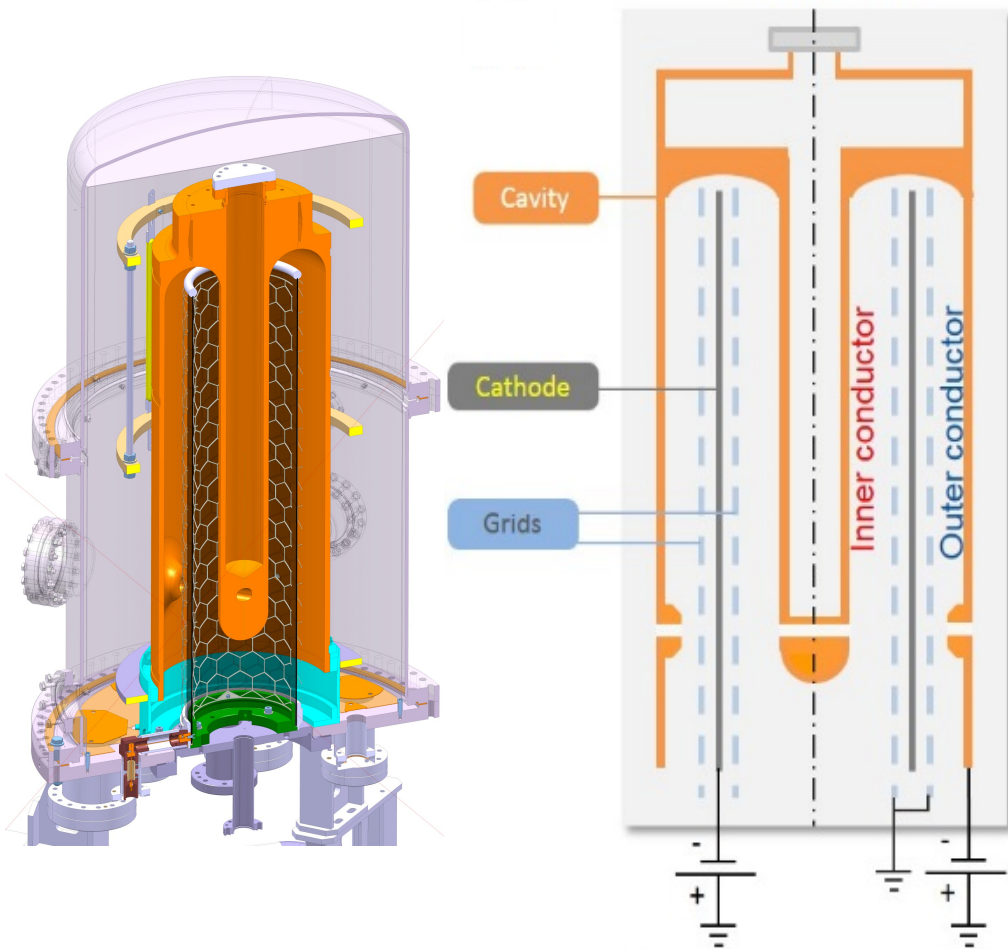
High coating rate (up to  $100$ 's  $\text{nm}/\text{min}$ )

“low temperature” coatings

Cavity used as vacuum chamber



# 1.2 Coating Techniques



## Biased Diode Sputtering

QWR type

Limited available space

Higher working pressures ( $10^{-2} - 10^{-1}$  mbar)

Ar or Kr as sputtering gas

$\sim 1 \text{ W} \cdot \text{cm}^{-2}$

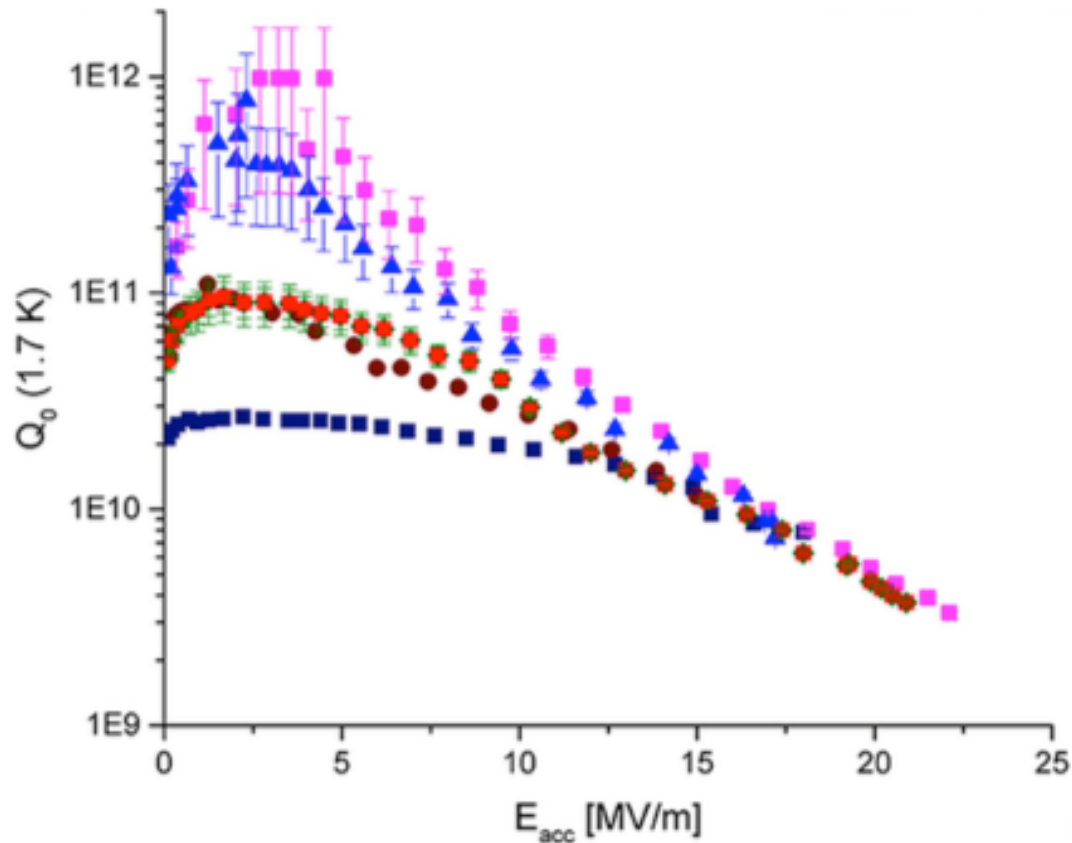
Cavity under UHV

→ Not UHV leak tight cavity

High temperature reachable ( $\sim 650^\circ\text{C}$ )  
(outgassing, Nb mobility, adhesion)

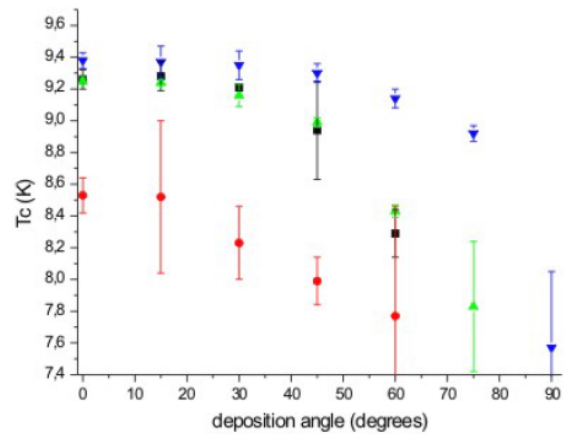
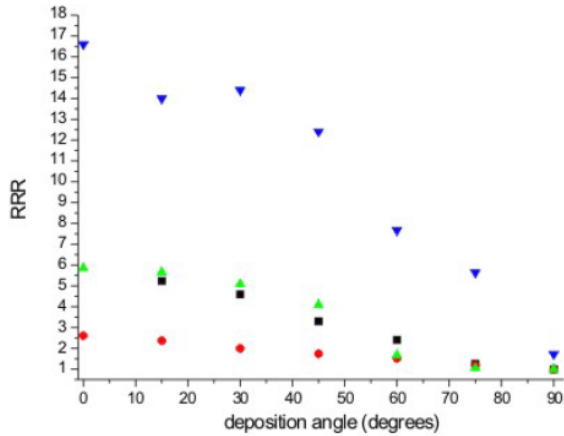
# 1.2 Coating Techniques

**Proven to be good at low field**

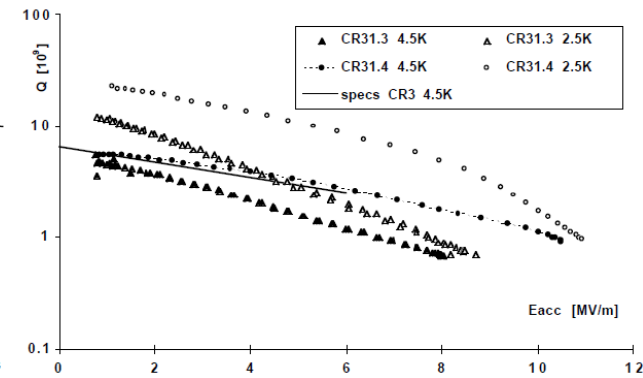
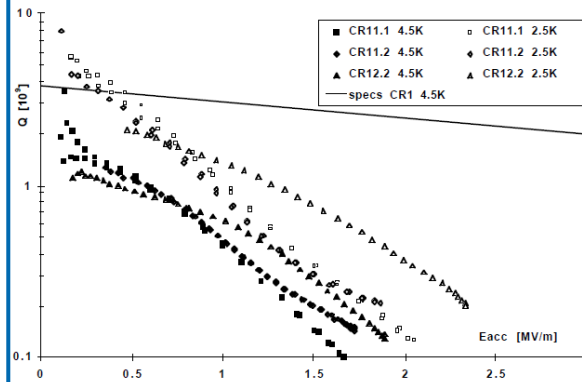
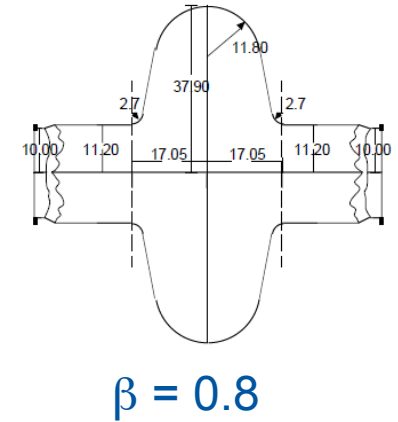
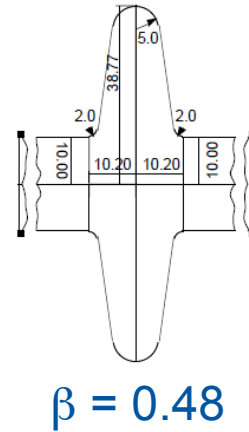


# 1.3 Limitations

## Samples [1]



## 352 MHz Cavities [2]



[1] D. Tonini et al, Morphology of niobium films sputtered at different target-substrate angle, 11<sup>th</sup> workshop on RF superconductivity, THP11

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

# Which Solutions?

# 2. Overview

# 2.1 Energetic condensation

**PROPOSAL:** Use ionized Nb to coat cavities instead of neutrals.  
→ Coating conformality, density

HOW?

**E**lectron **C**yclotron  
**R**esonance

Jlab, Fermilab

**UHV** **C**athodic **A**rc

Alameda, NCBJ, INFN

**H**igh **P**ower **I**mpulse  
**M**agnetron **S**puttering

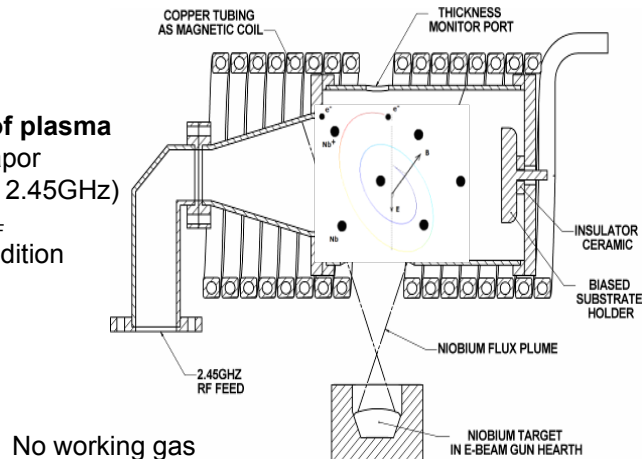
CERN, Jlab, Fermilab,  
STFC



# 2.2 ECR @ Jlab (courtesy of A.M. Valente-Feliciano)

## Generation of plasma

Neutral Nb vapor  
RF power (@ 2.45GHz)  
Static  $B \perp E_{RF}$   
with ECR condition

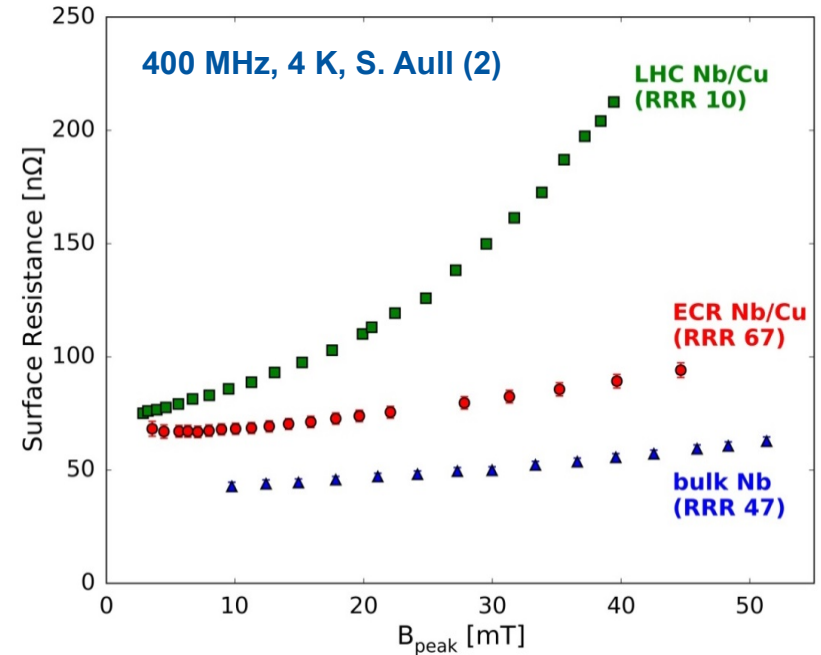


No working gas

**Singly charged ions (64eV) produced in vacuum**

**Controllable deposition energy with Bias voltage**

Excellent bonding , No macro particles



QPR results are more than promising:

- Low  $R_{res}$  combined to mitigated Q-slope
- Good adhesion
- What else has to be done?

**Pros**

- Gasless
- No macroparticles

**Cons**

- Scalability?

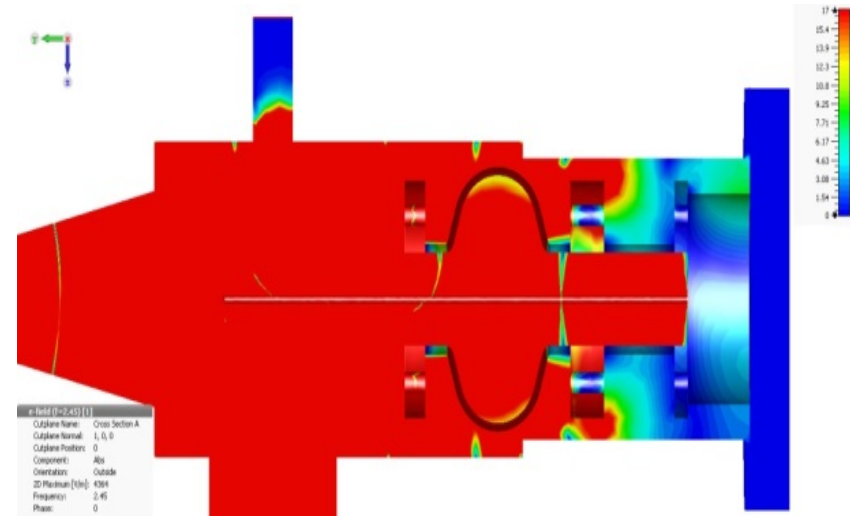
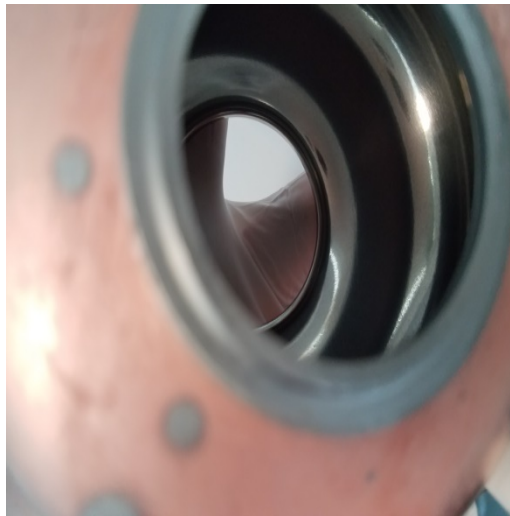
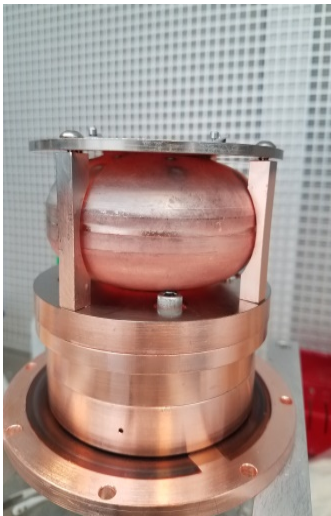


1: A. M. Valente-Feliciano, Supercond. Sci. Technol. 29 (2016) 113002

2: Aull S et al 2015 Proc. 17th Int. Conf. on RF Superconductivity (Whistler, BC, Canada, 13–18 September) TUBA03 494

# 2.2 ECR – Next steps

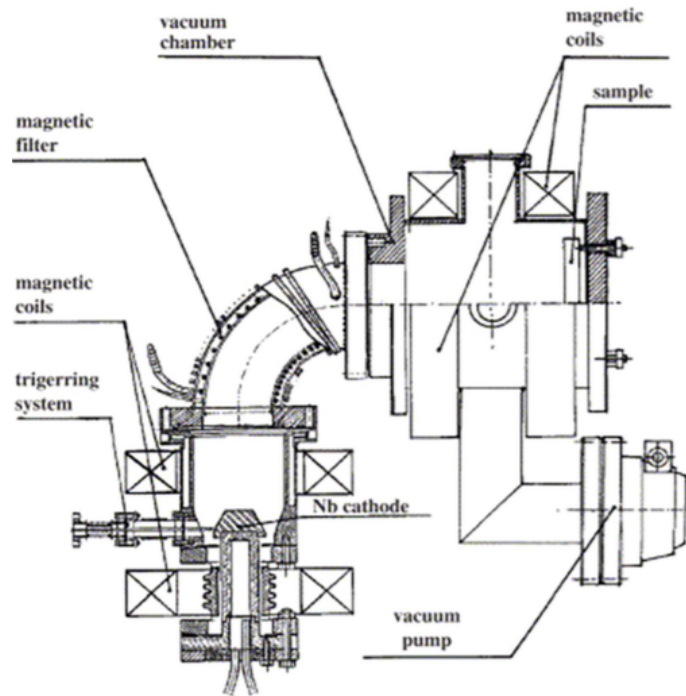
## Scale-up to cavities



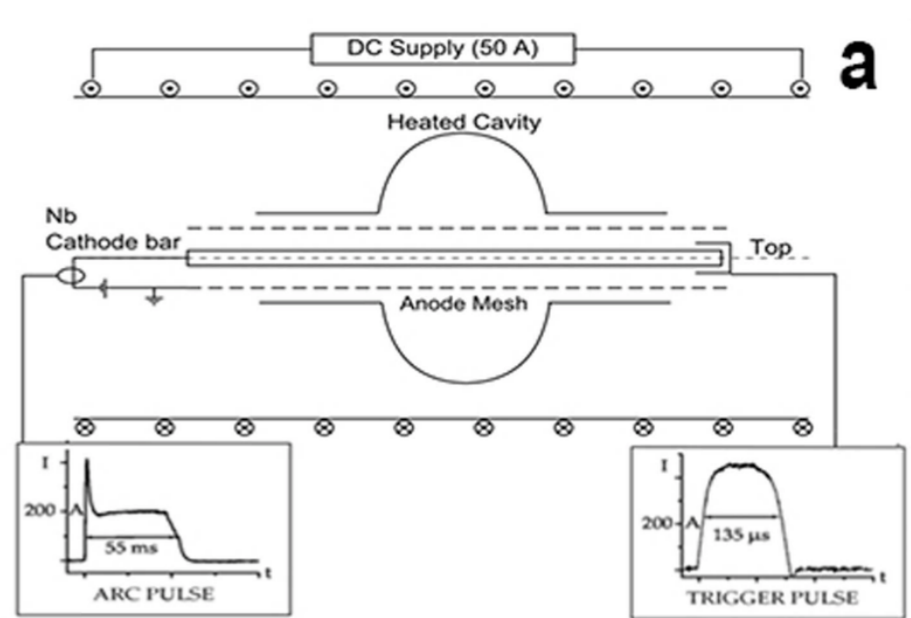
3 GHz Cavity with beam tubes: 5GHz frequency cut-off

Adjust coating geometry to allow RF fields to penetrate the cell and get adequate plasma conditions

# 2.3 – UHV Cathodic Arc



Langner J et al 2006 Vacuum 80 1288–93



Krishnan M et al 2012 Phys. Rev. Spec. Top. Accel. Beams 15 032001

## Pros

- High coating rates
- High ionization degree (+3)

## Cons

- Macroparticles formation
- Delamination
- Not yet satisfactory on cavities
- Not technique related

# 3. @ CERN: HiPIMS

# 3.1 HiPIMS Setup

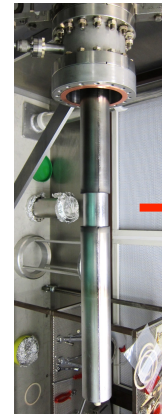
DCMS System already existing  
 Pulsed power supplies (Huttinger-TRUMPF)  
 DC Bias

## Modification:

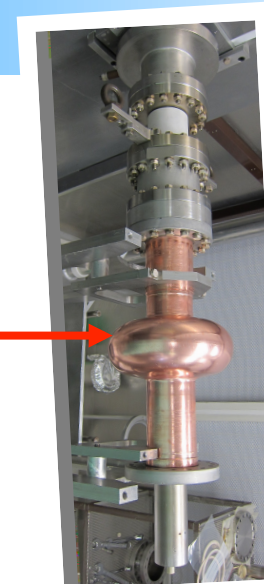
- Anodes implementation
  - Enables bias configuration

## Typical parameters

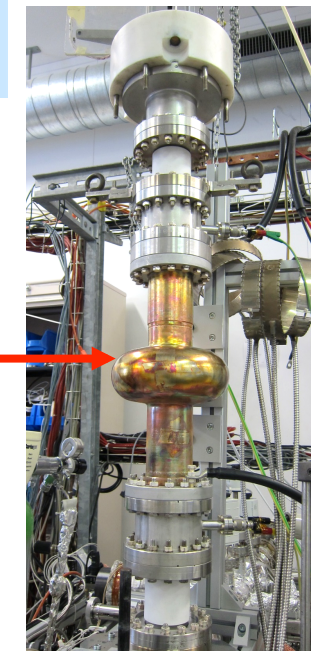
Parameter	Typical value/range
Gas	Kr
Pressure	$2 \cdot 10^{-3} - 1 \cdot 10^{-2}$ mbar
Power (Avg)	1 kW
Peak Power	80 kW ( $1 \text{ kW} \cdot \text{cm}^{-2}$ )
Peak Current	150 - 250 A ( $2-3 \text{ A} \cdot \text{cm}^{-2}$ )
Pulse duration	50 - 200 $\mu\text{s}$
Pulse Frequency	20 - 500 Hz
Temperature	150°C



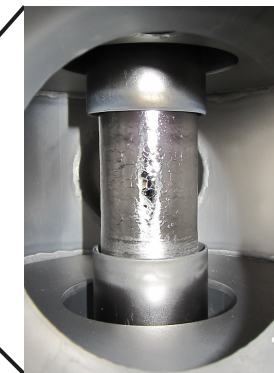
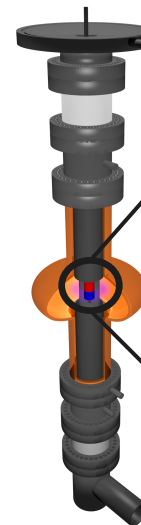
Nb cathode



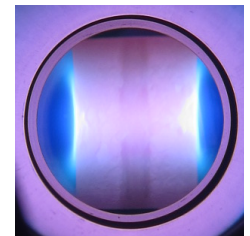
1.3 GHz cavity



1.3 GHz cavity coating setup



Nb cathode with permanent magnets inside and Nb anodes

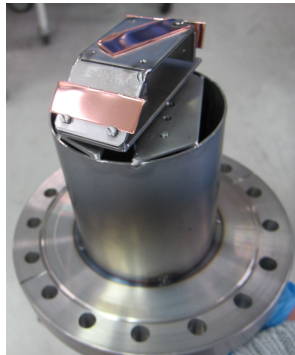


HiPIMS discharge



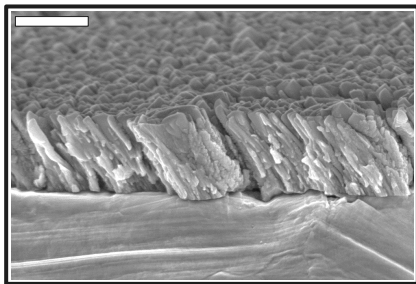
# 3.2 HiPIMS results : Morphology

- Samples - Coupons

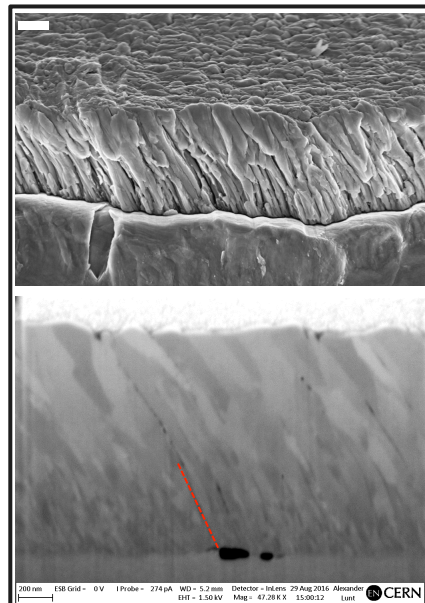


Grounded HiPIMS = Grounded DCMS  
Bias compulsory to densify the layer

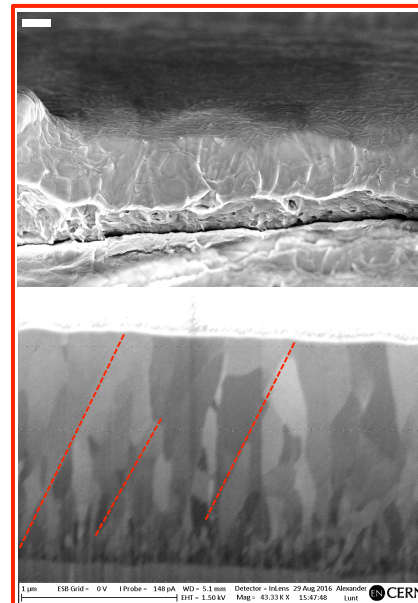
0V DCMS



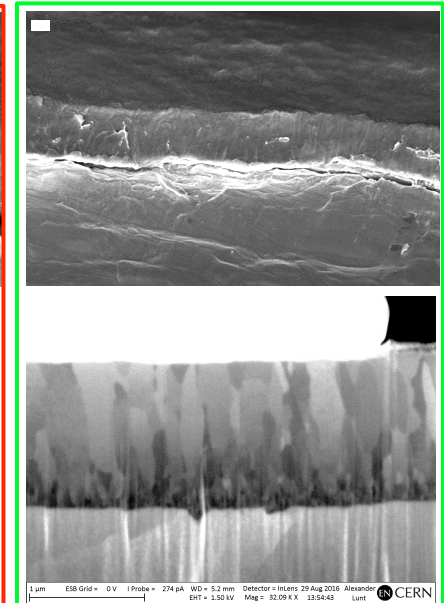
0V HiPIMS



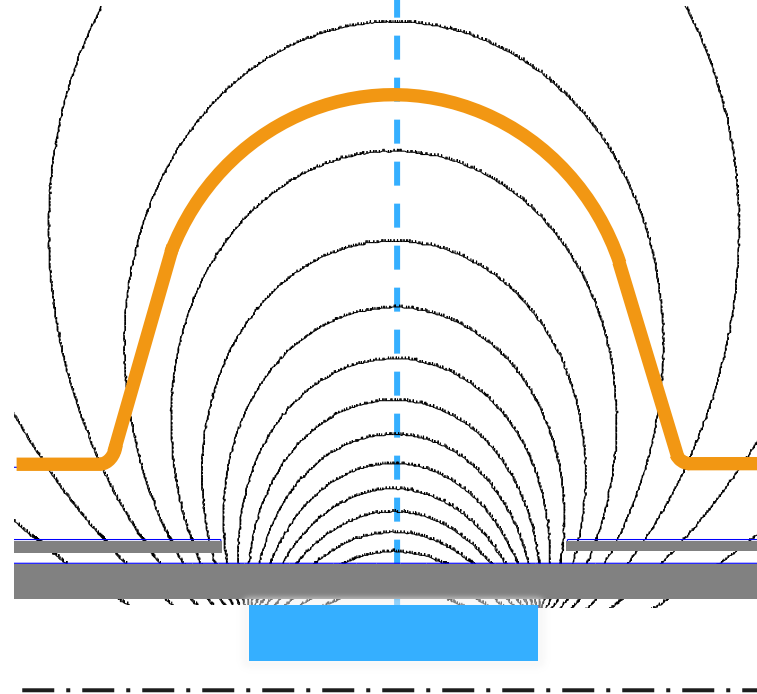
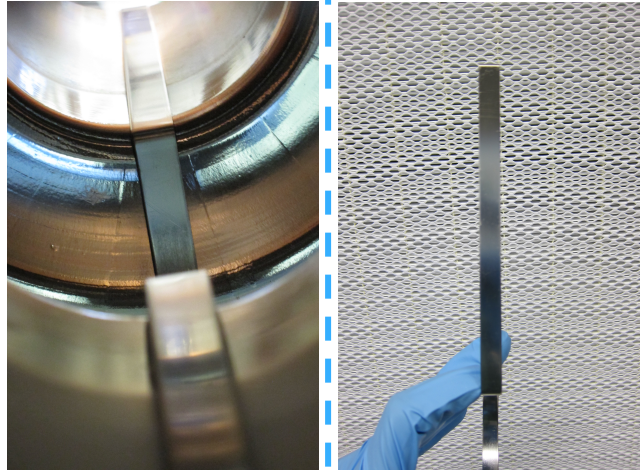
-50V



-100V

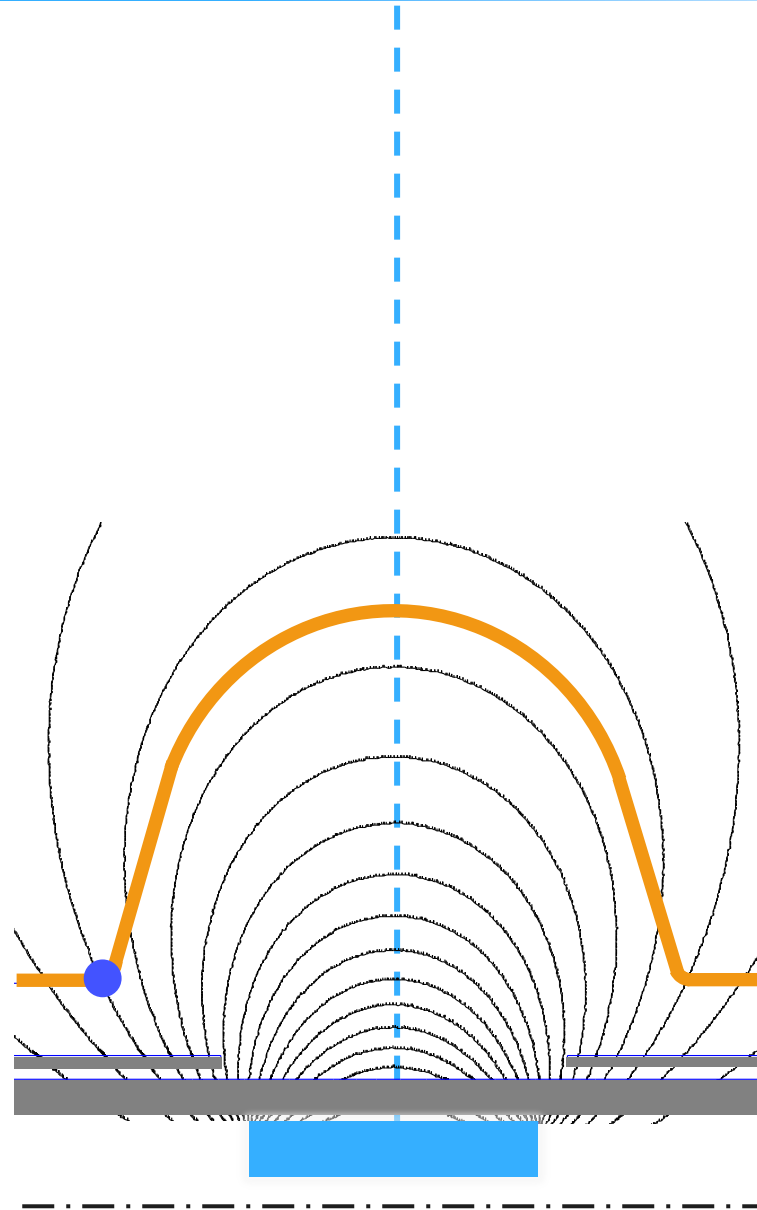


# 3.4 HiPIMS results : Morphology

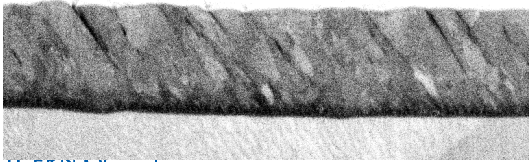


# 3.4 HiPIMS results : Morphology

DCMS



1  $\mu\text{m}$

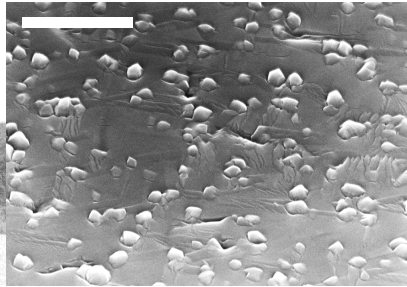




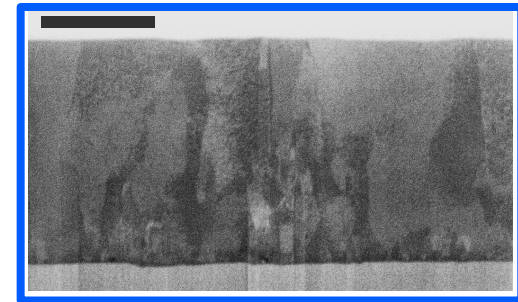
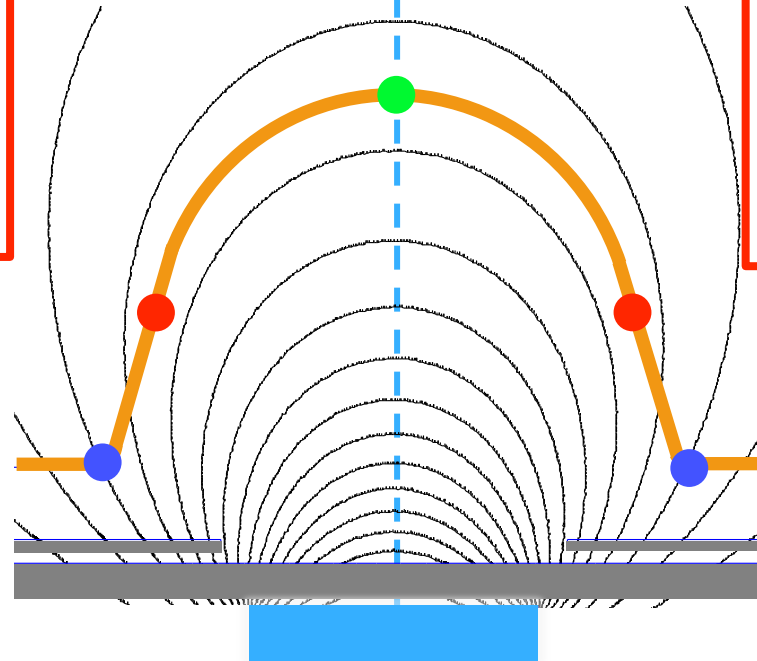
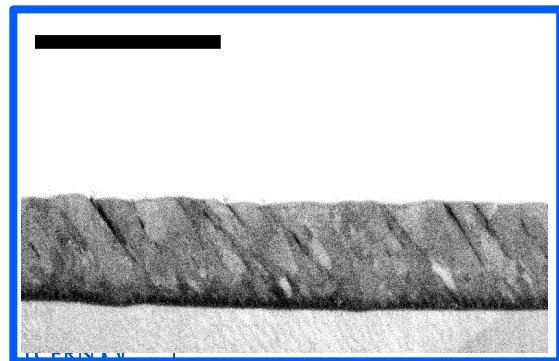
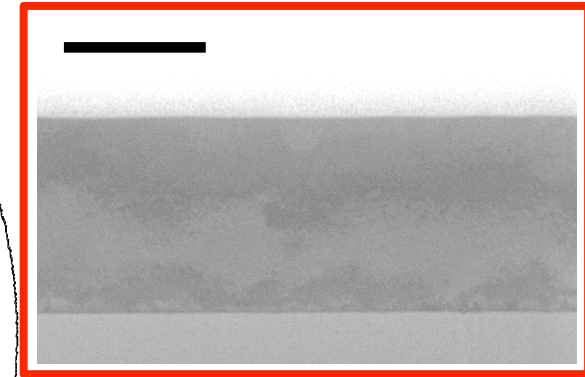
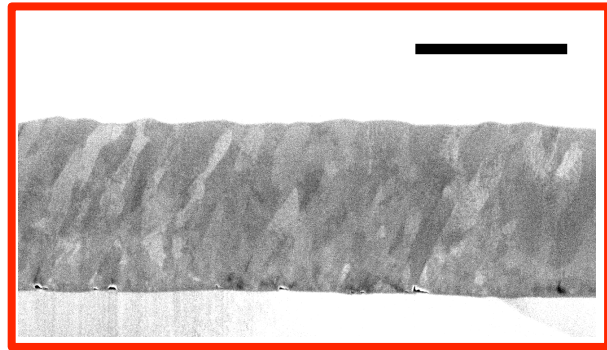
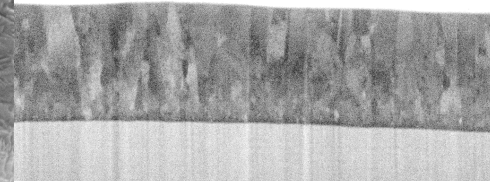
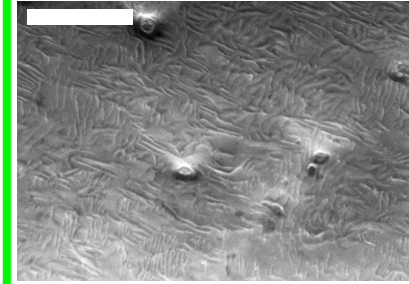
# 3.4 HiPIMS results : Morphology

DCMS

1  $\mu\text{m}$



Biased (-100V) HiPIMS



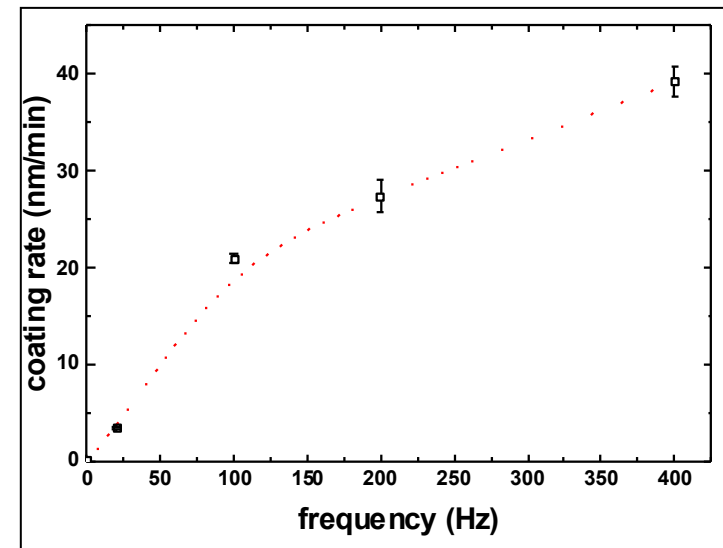
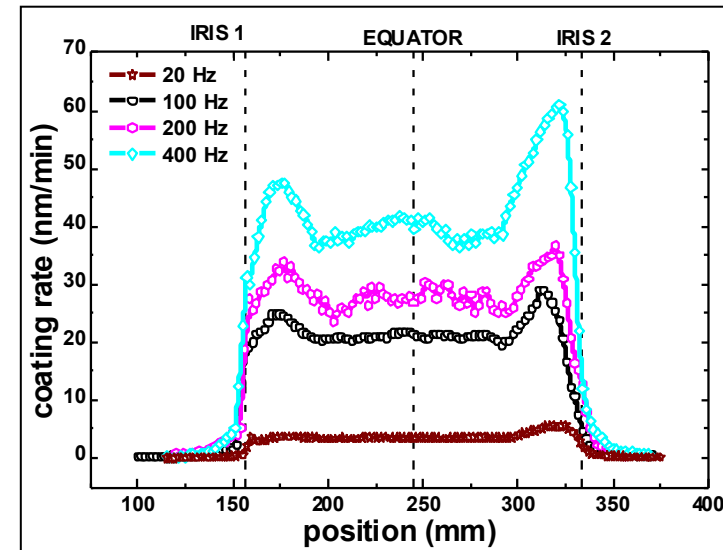
# 3.3 HiPIMS results : Frequency

- Samples – cavity replicates

Thickness profile obtained by XRF

Lower frequencies lead to

- Lower coating rates
- Modified coating profile



# 3.3 HiPIMS results: Magnet

- Samples – cavity replicates

Thickness profile obtained by XRF

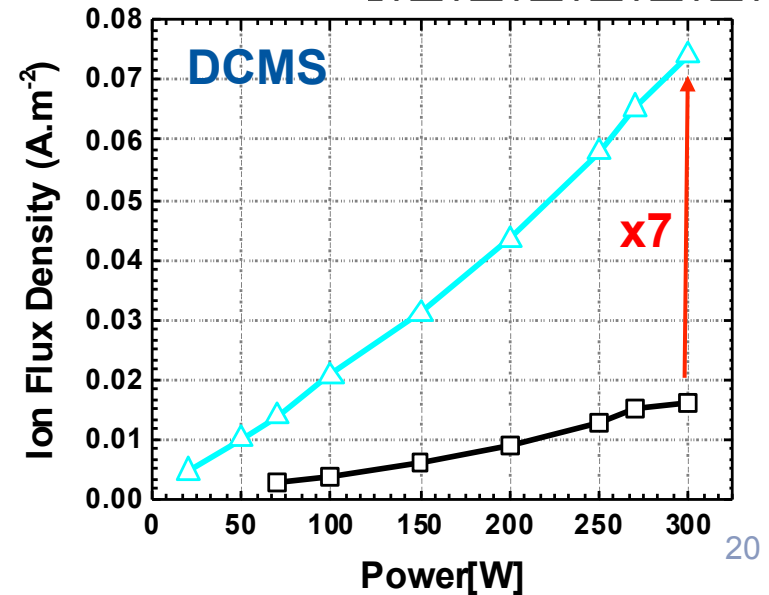
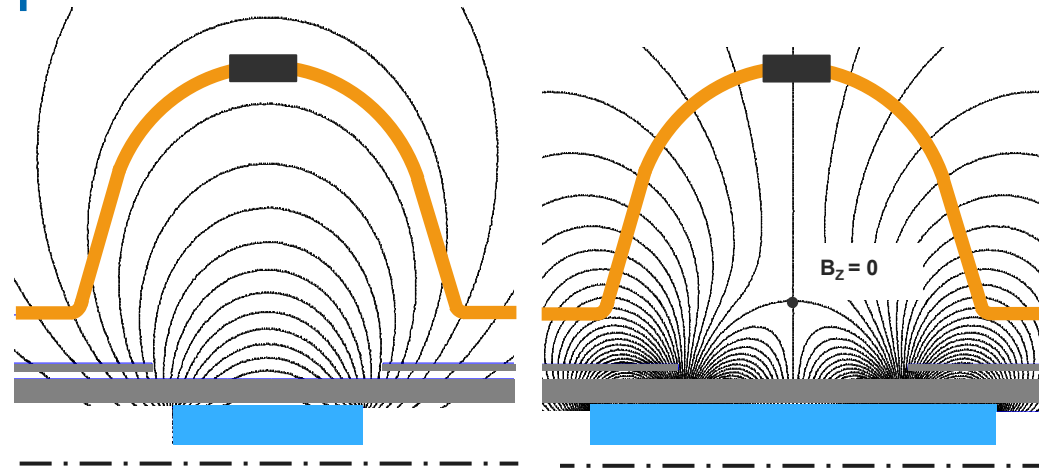
Lower frequencies lead to

- Lower coating rates
- Modified coating profile

Possibility to tune the ion flux density

Impact on the layer to be assessed

- Samples
- Cavity



# 3.3 HiPIMS results: Magnet

- Samples – cavity replicates

Thickness profile obtained by XRF

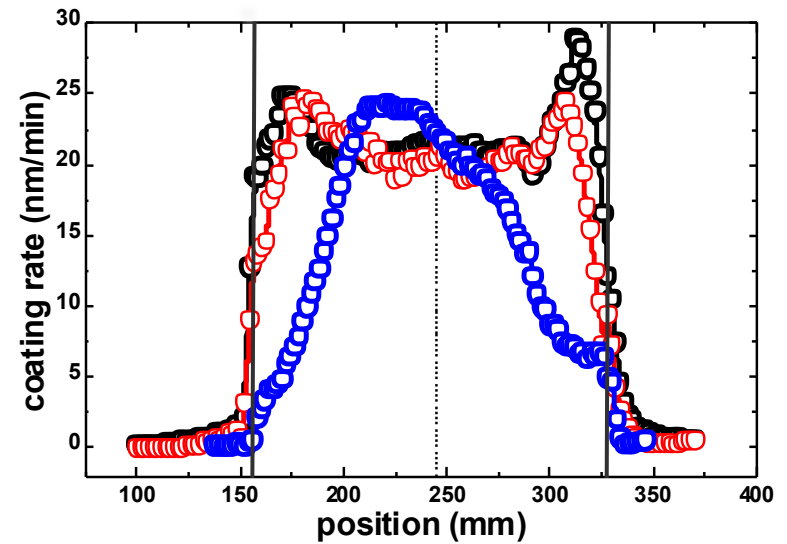
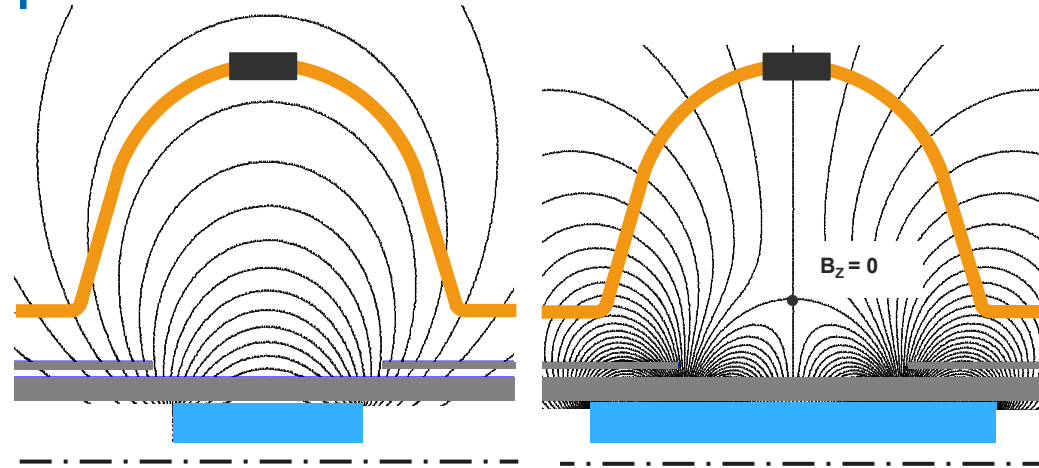
Lower frequencies lead to

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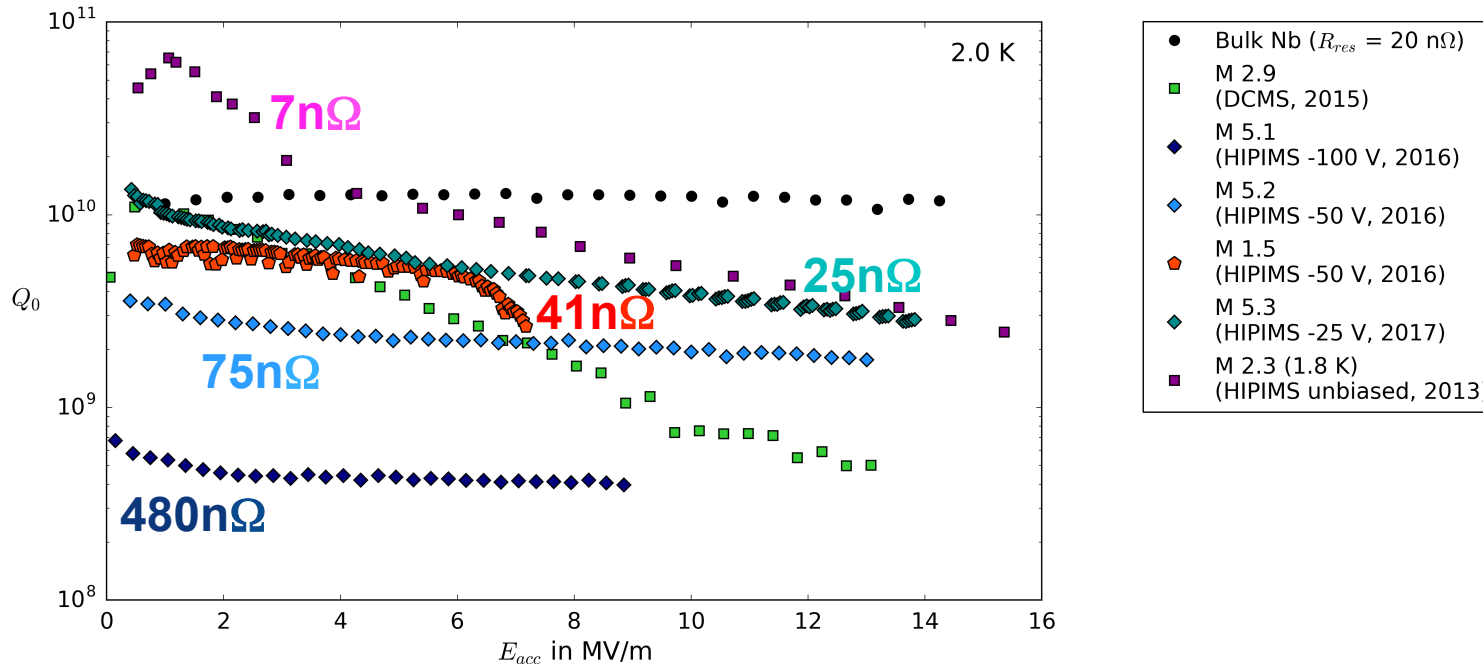
- Samples
- Cavity



**IMPACT ON LAYER AND RF PERFORMANCES?  
COATINGS ON CAVITIES NEEDED**

# 3.5 HiPIMS results: Cavities

## • 1.3 GHz Cu Cavities



High Bias does not give good results (gas implantation , stress...)

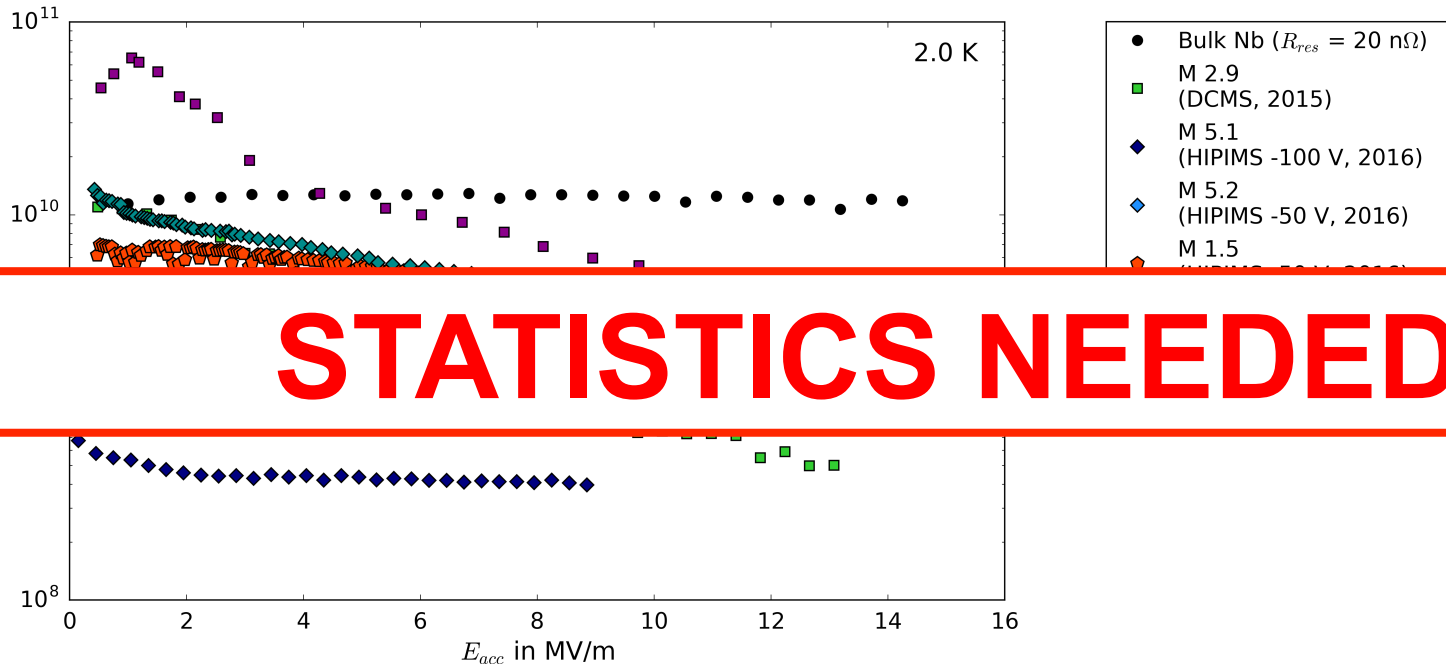
Lower pressure tends to better performances (contamination, stress...)

Q-slope looks mitigated vs DCMS coating

Best HiPIMS :  $R_{res} = 5.2 \text{ n}\Omega$

# 3.5 HiPIMS results: Cavities

- 1.3 GHz Cu Cavities



High Bias does not give good results (gas implantation , stress...)

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Best HiPIMS :  $R_{res} = 5.2 \text{ n}\Omega$

# 3. @ CERN: A15

K. Ilyina-Brunner



# 3.6 A15 Setup

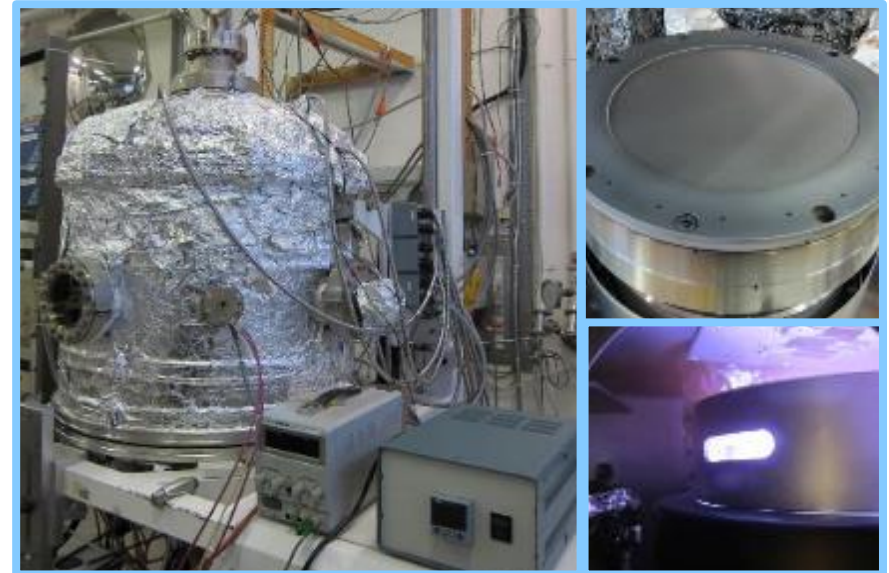
## DCMS System

- Heater integrated
- Single target Nb:Sn 3:1

## UHV furnace

- Post coating annealing

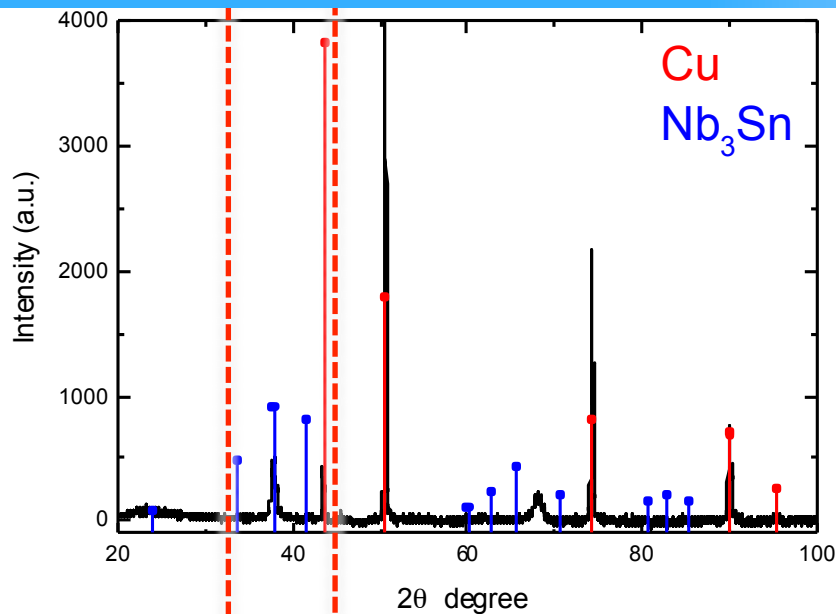
Parameter	Typical value/range
Gas	Kr/Ar
Pressure	$5 \cdot 10^{-4} - 5 \cdot 10^{-2}$ mbar
Power (Avg)	200 W
Temperature	From 150°C to 700°C in-situ



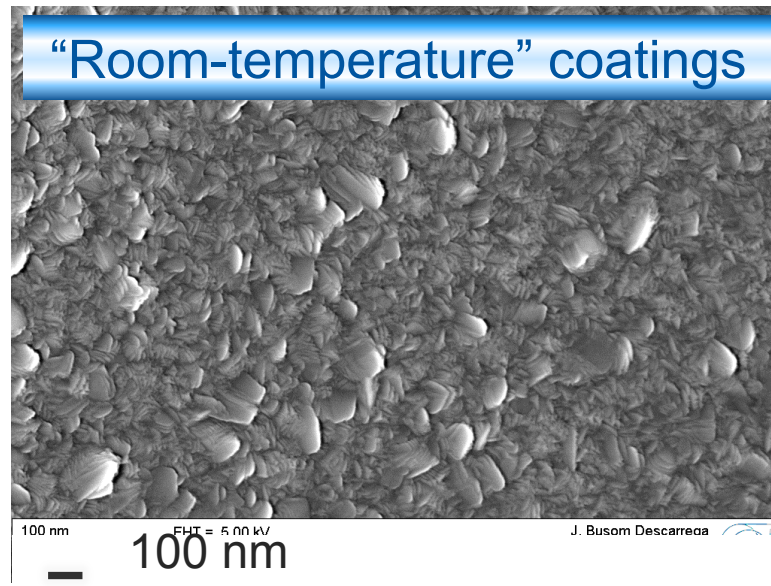


# 3.6 XRD - Morphology

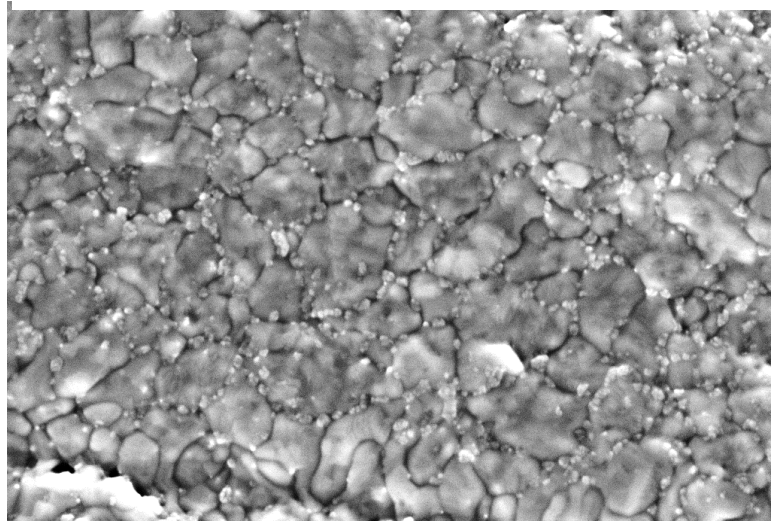
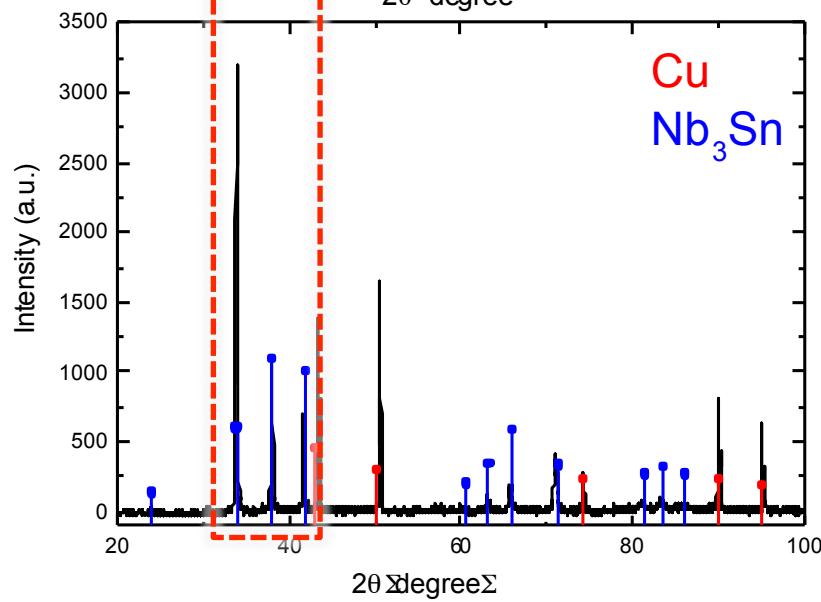
as-deposited



“Room-temperature” coatings



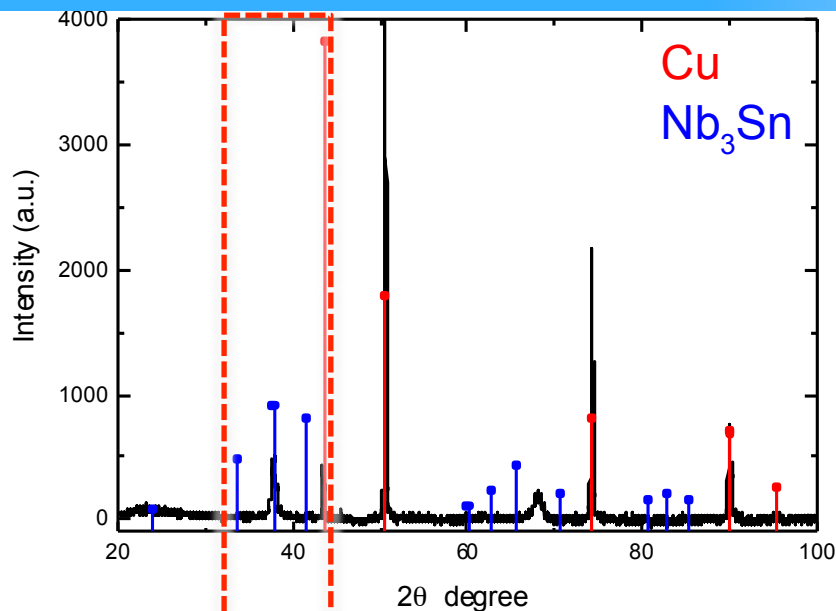
after annealing



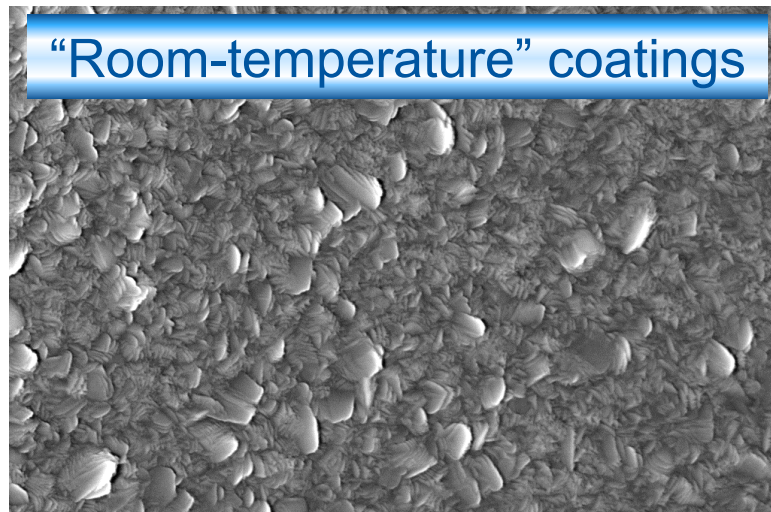
— 100 nm

# 3.6 XRD - Morphology

as-deposited

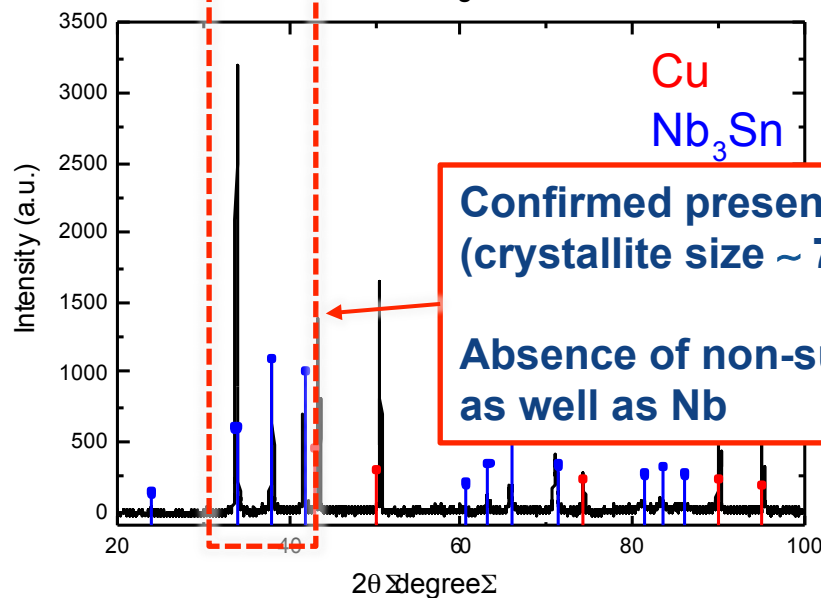


“Room-temperature” coatings



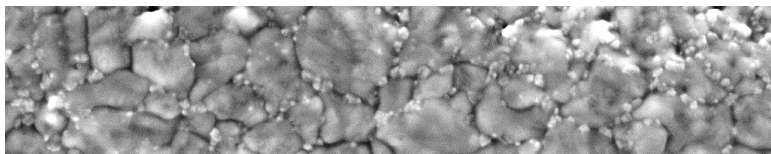
— 100 nm

after annealing



Confirmed presence of superconducting A15 phase  
(crystallite size  $\sim 70 - 200$  nm)

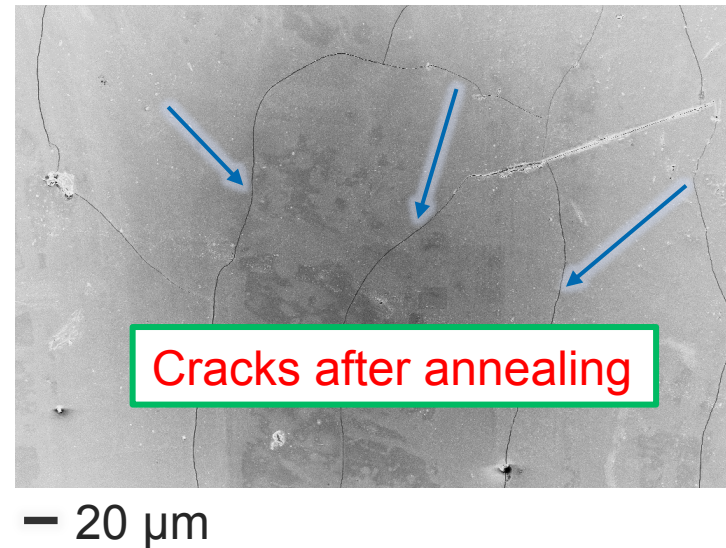
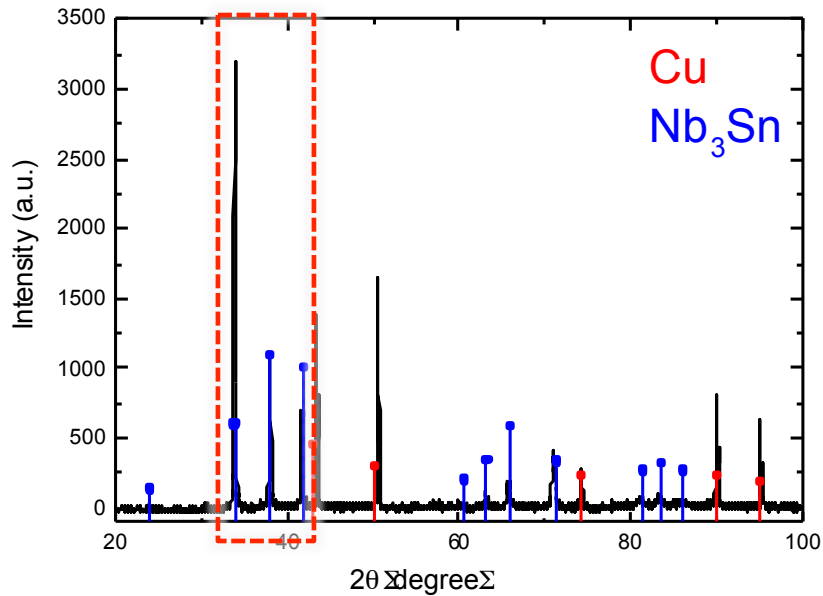
Absence of non-superconductive phases -  $NbSn_2$ ,  $Nb_6Sn_5$ ,  
as well as Nb



— 100 nm

# 3.6 XRD - Morphology

**after annealing**

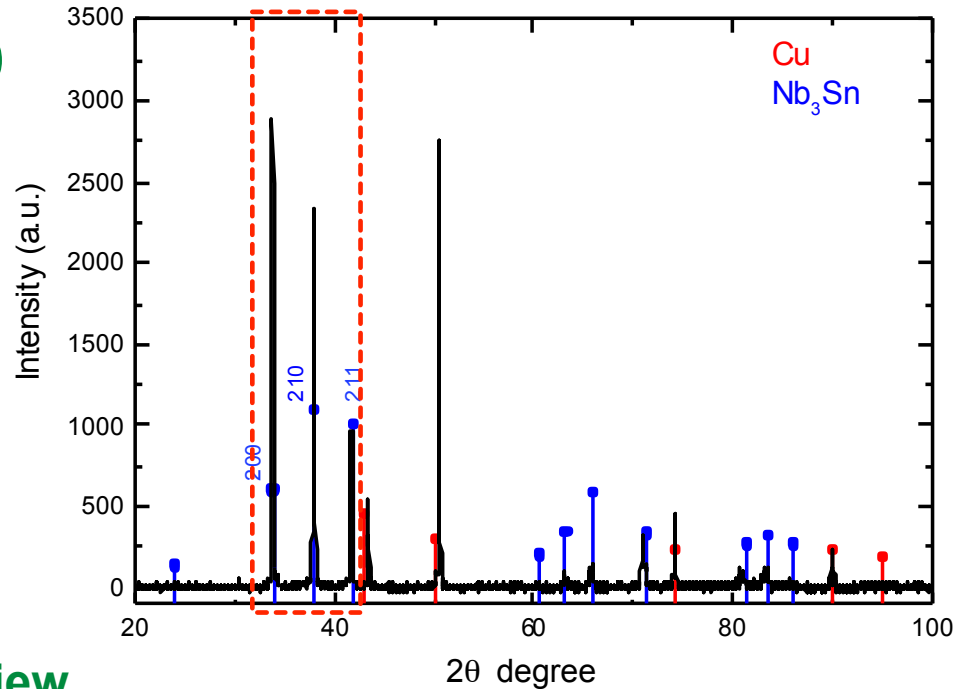


**All “room temperature” samples are evincing cracks after annealing.**

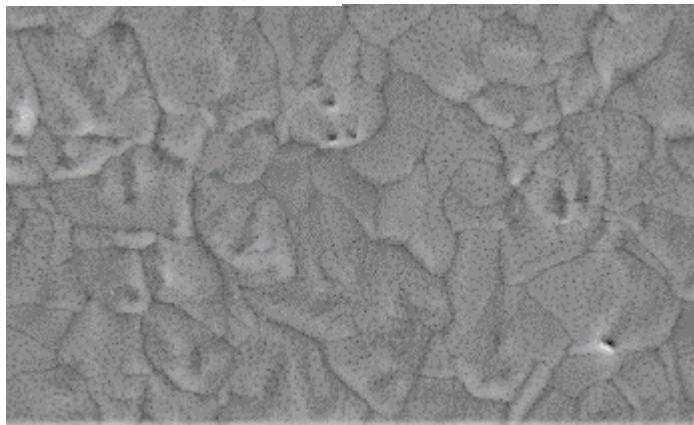
**Now coating recipe to overcome this problem has been found !**

# 3.6 High temperature coatings

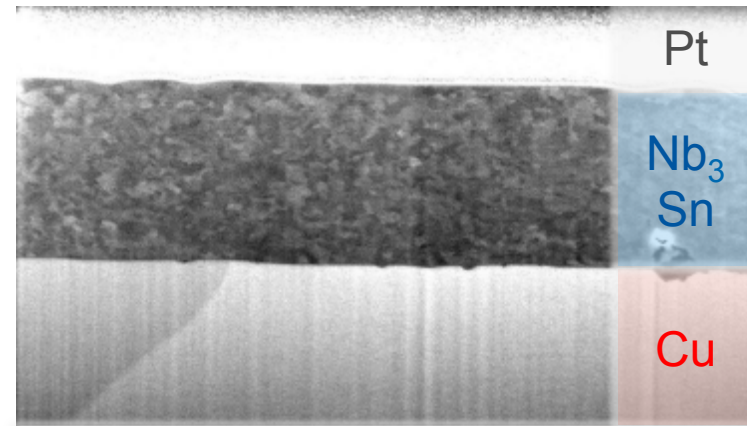
XRD



SEM top view



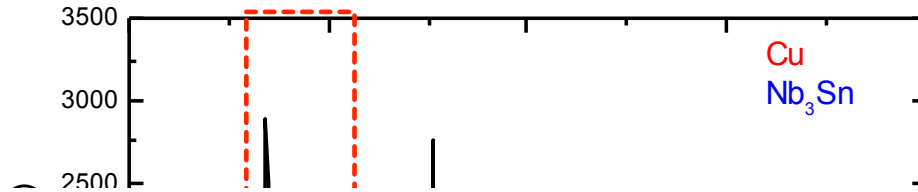
FIB cross section



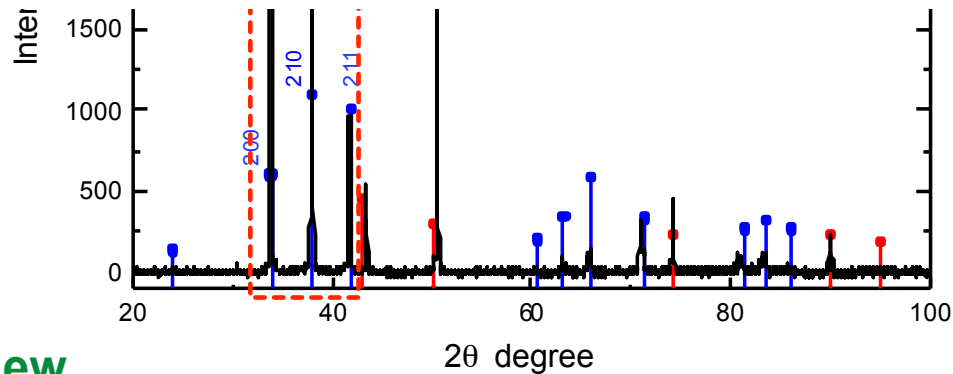


# 3.6 High temperature coatings

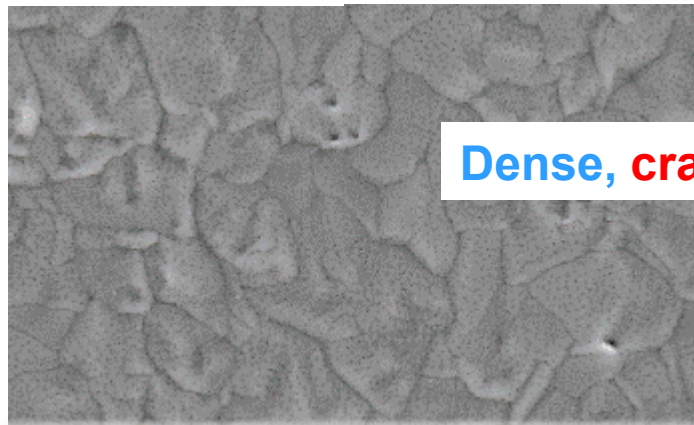
**XRD**



**Confirmed presence of superconducting A15 phase  
With crystallite size ~ 150 - 400 nm**

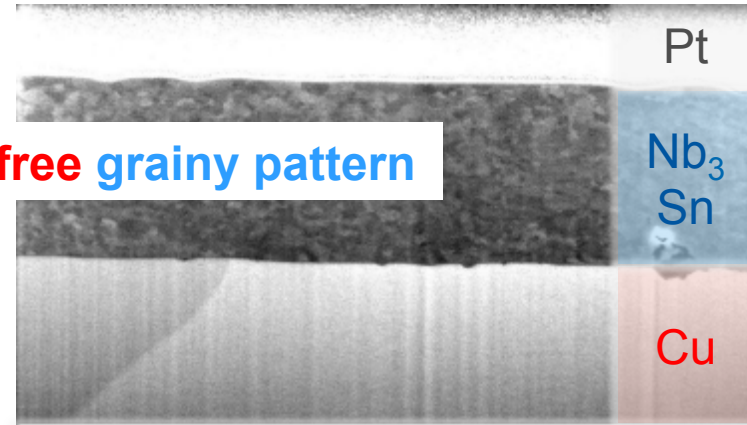


**SEM top view**



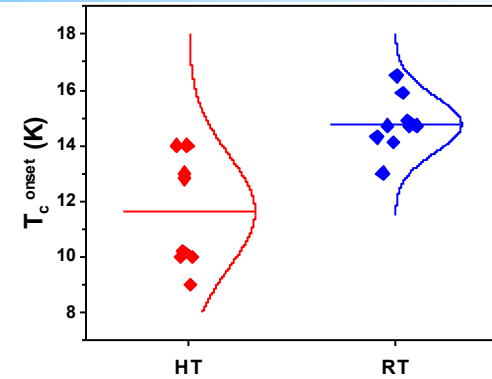
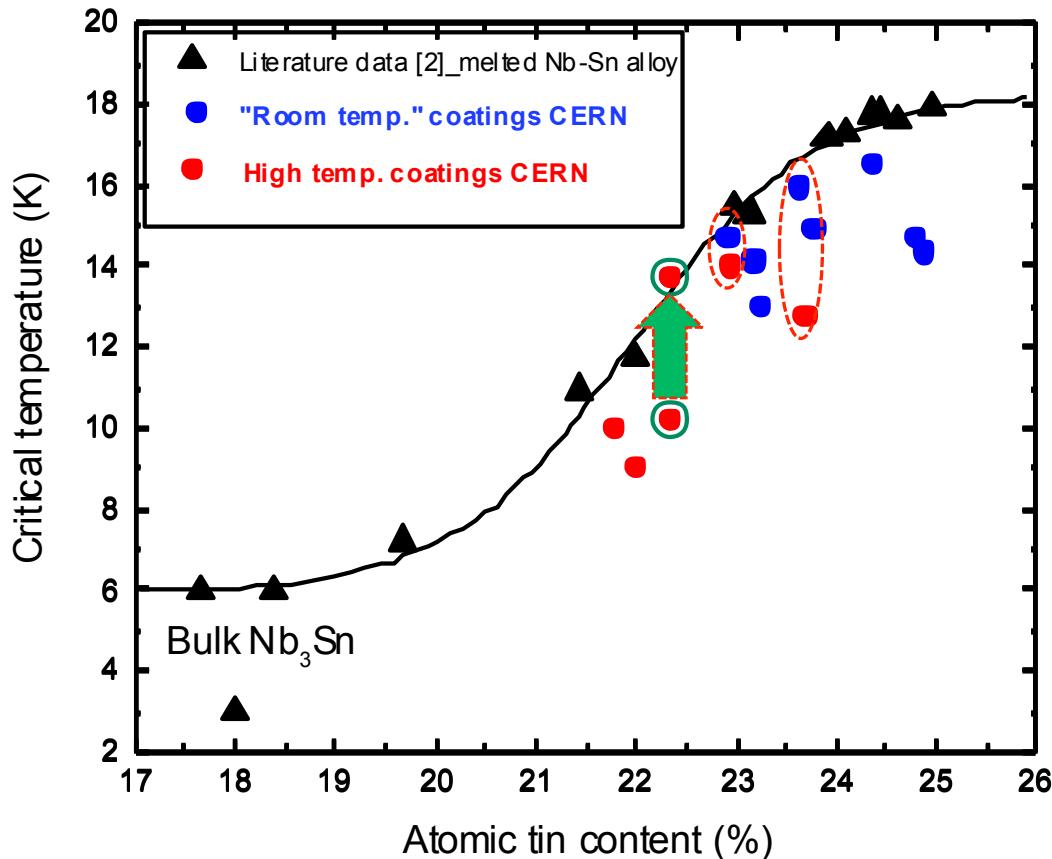
**Dense, crack free grainy pattern**

**FIB cross section**



# 3.6 $T_C$ vs composition

[2] A. Godeke. *Supercond. Sci. Technol.*, 19 (2006) R68-R80



Room temperature coatings process lead to higher critical temperature values no matter coating parameters

High temperature coating + annealing post coating

Could be a good combination by modulating temperature and duration

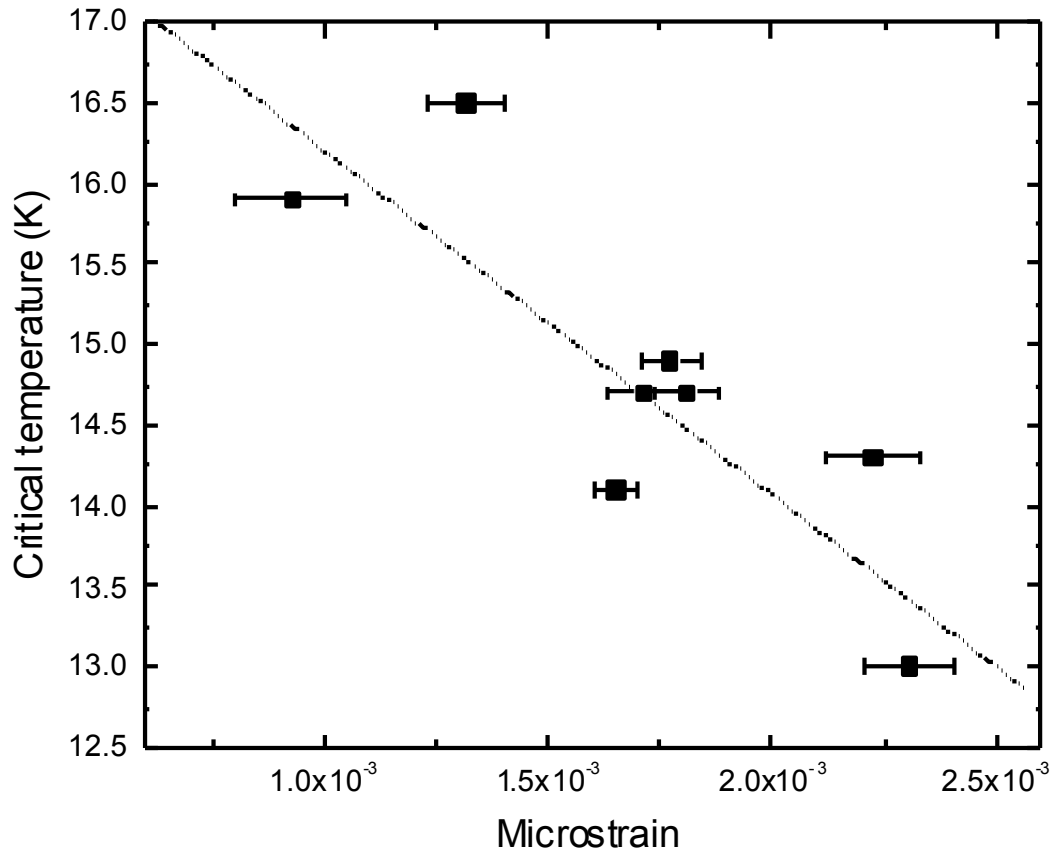
$T_C$  values constantly lower for the films coated on copper substrates with respect to the bulk Nb<sub>3</sub>Sn values

Copper substrate influence? Stresses in the film??

# 3.7 $T_C$ vs microstrain

- XRD rietveld analysis

“Room-temperature” coatings+annealing



Micro-strain mitigation seems to be critical to ensure highest possible  $T_c$

- small-range order matters too
- Diffusion driven process

# 4. Summary / Perspectives



1. ECR and HiPIMS have both shown promising SRF results
2. Work needed to stabilize / scale-up the processes
3. A15 onto copper: challenging but converging to promising recipes. Next step: RF
4. A15 scale-up promises new challenges

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