

# HIPIMS Nb coatings, from 1.3 GHz to 400 MHz

C. P. A. Carlos, S. Leith, G. Rosaz, M. Taborelli, M. Bonura, C. Senatore, S. Pfeiffer, A. -T. P. Fontenla, G. Bellini, L. Ferreira, L. Vega-Cid, A. Bianchi, W. Venturini-Delsolaro, T. Proslier, and Y. Kalboussi

FCC Week 2023

8<sup>th</sup> of June 2023

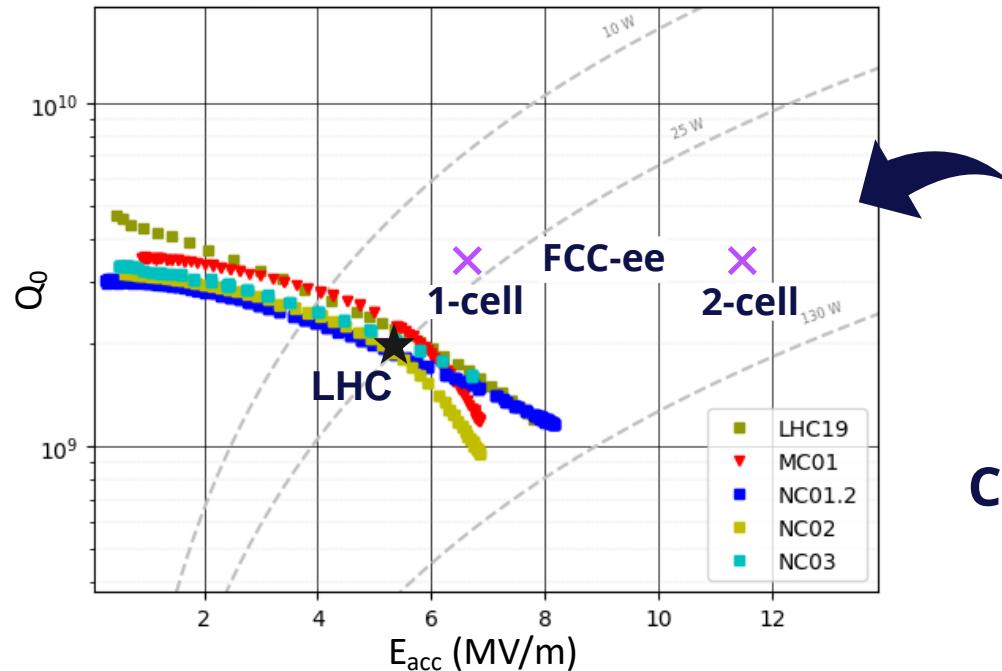


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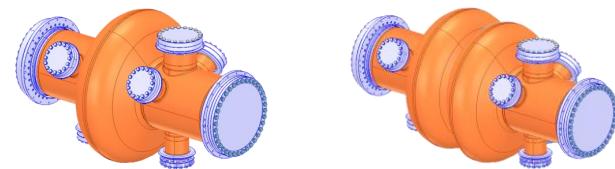
# Motivation

LHC cavities Q vs  $E_{acc}$  @4.5 K



Degradation of  $Q_0$  with increasing  $E_{acc}$ : Q-slope

FCC-ee 400 MHz Nb/Cu



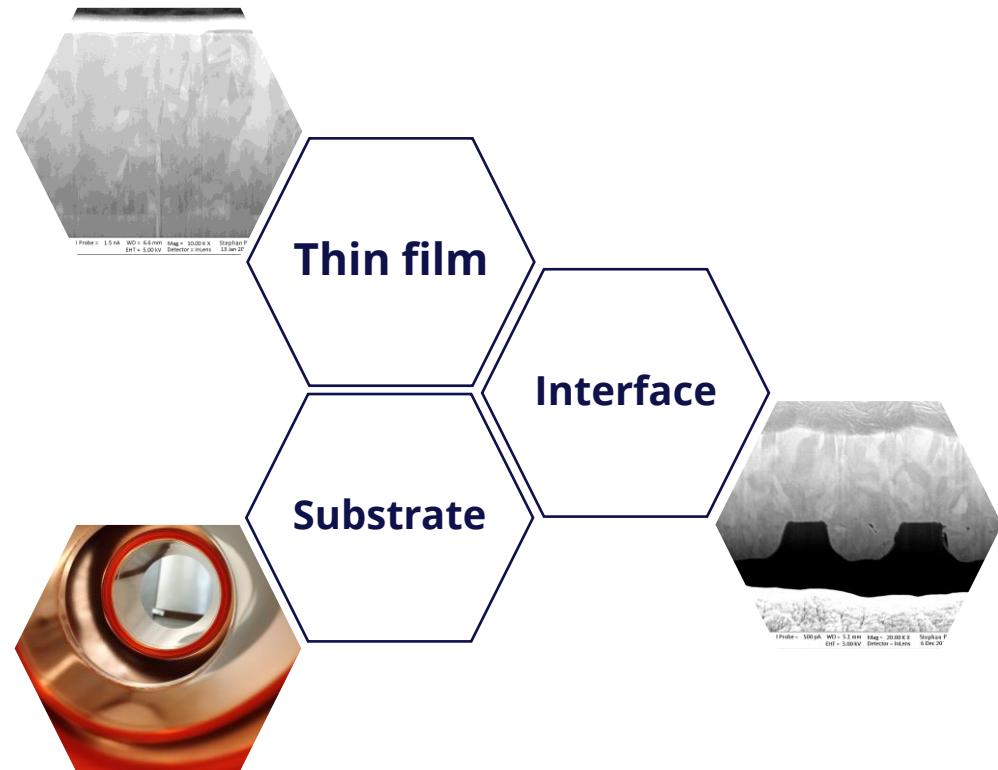
Cure Q-slope & extend field reach!

# SRF cavities R&D

Performance optimization



Defect density minimization



# Previous results

See C. P. Carlos et al., Nb/Cu thin film HiPIMS coatings optimization for SRF applications, FCC 2022  
<https://indico.cern.ch/event/1064327/timetable/>



## HiPIMS

performance boost to achieve  
FCC-ee target

1 μm

## DCMS

## HiPIMS

## Energetic condensation

DCMS – Direct Current Magnetron Sputtering

HiPIMS – High Power Impulse Magnetron Sputtering



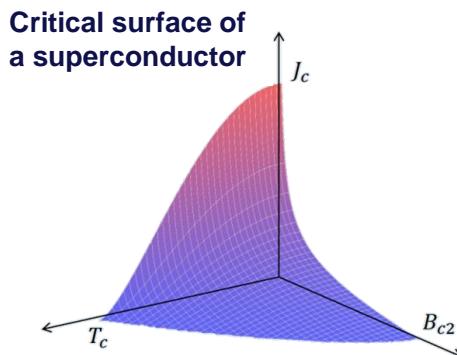
## HiPIMS

$J_c$

performance boost achieve  
FCC-ee target

### Critical current density

used as metric for qualifying thin film quality for SRF applications



Depends on pinning centres (structural defects)

The higher the defect density, the higher the  $J_c$

Defect density minimization  $\longleftrightarrow$   $J_c$  minimization

See C. P. Carlos et al., Nb/Cu thin film HiPIMS coatings optimization for SRF applications, FCC 2022  
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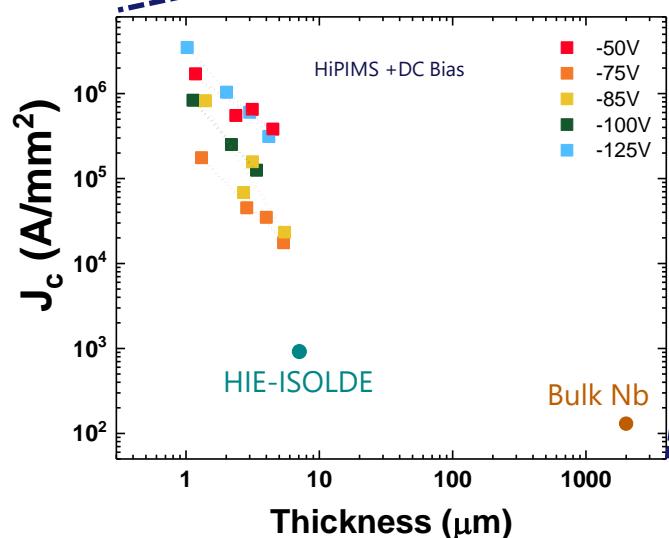


See C. P. Carlos et al., Nb/Cu thin film HiPIMS coatings optimization for SRF applications, FCC 2022  
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HiPIMS

$J_c$

performance boost achieve  
FCC-ee target



**$J_c$  decreases with increasing film thickness**

Independently of bias

**optimum  $J_c$  with -75 V applied DC bias**

(Other parameters under study)

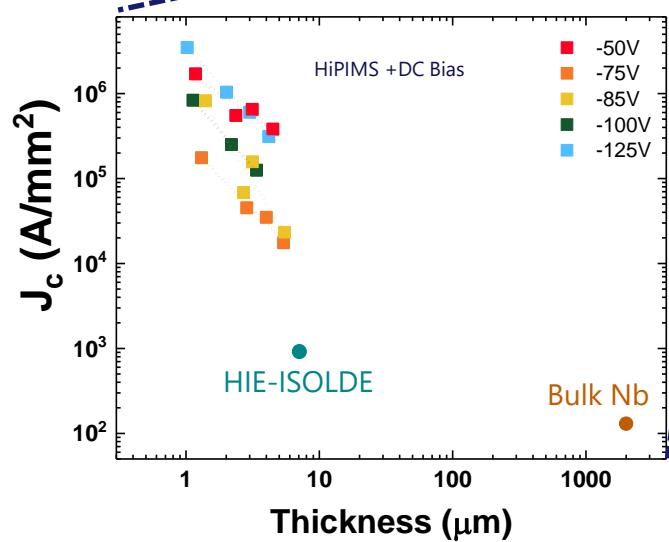
See C. P. Carlos et al., Nb/Cu thin film HiPIMS coatings optimization for SRF applications, FCC 2022  
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HiPIMS

$J_c$

performance boost achieve  
FCC-ee target

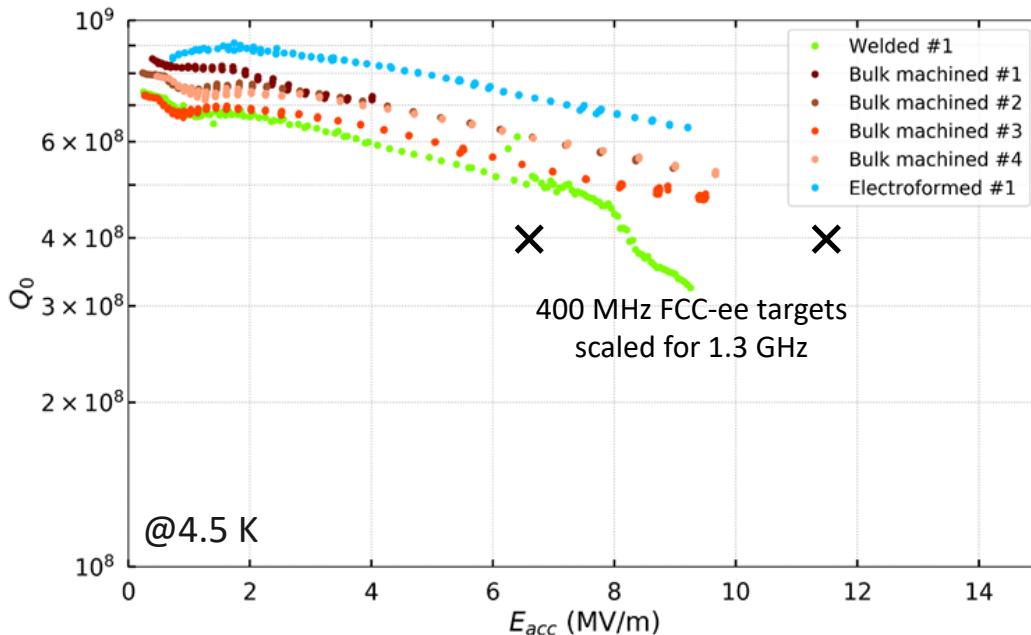


Optimized coating  
parameters:

-75 V DC bias and 6  $\mu\text{m}$  thickness  
(cell)

# 1.3 GHz test cavities

## Results 2020-2023



- HiPIMS coating w/ optimized coating recipe + seamless substrates
- bulk machined & electroformed cavities (no weld at equator) +
- chemistry on Cu substrate : electropolishing (instead of SUBU) + passivation (improve adhesion)

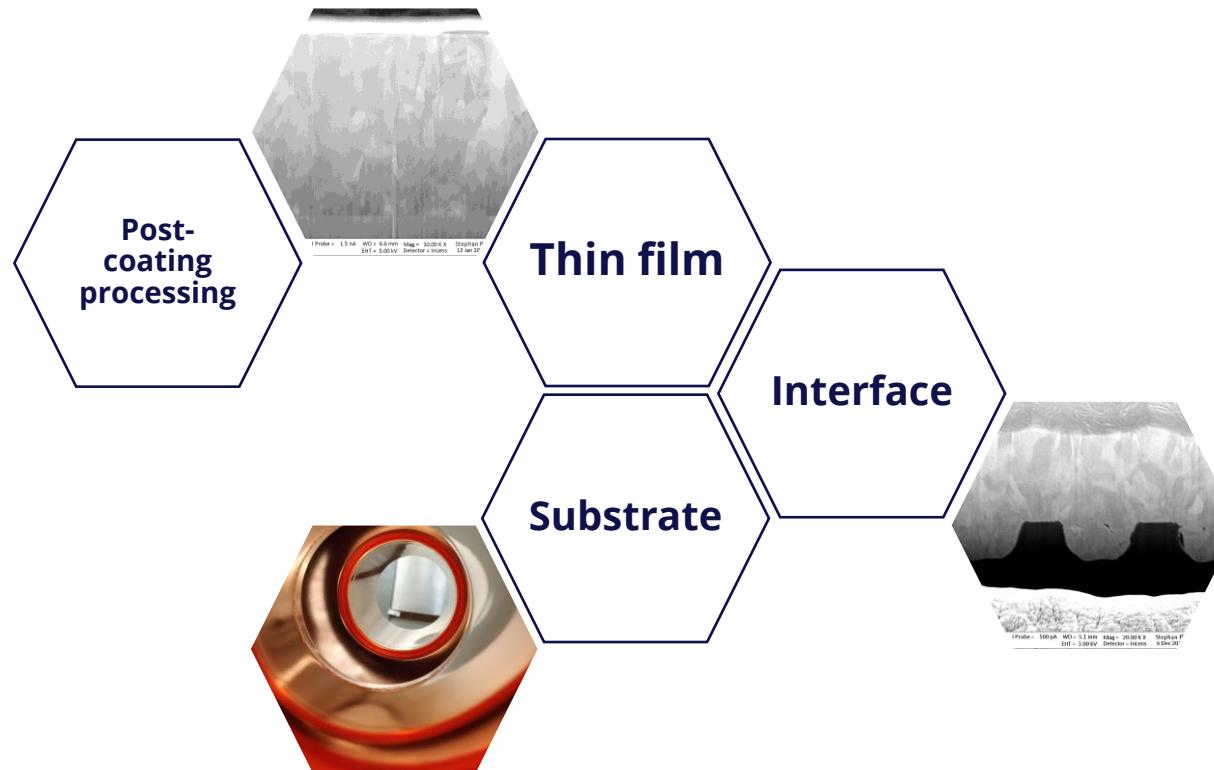


**Improved performance with mitigated Q-slope**

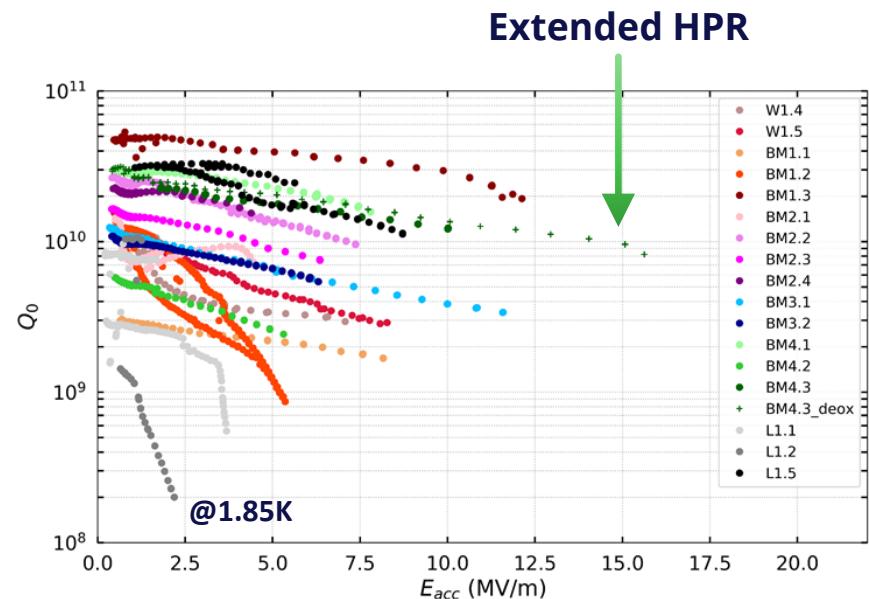
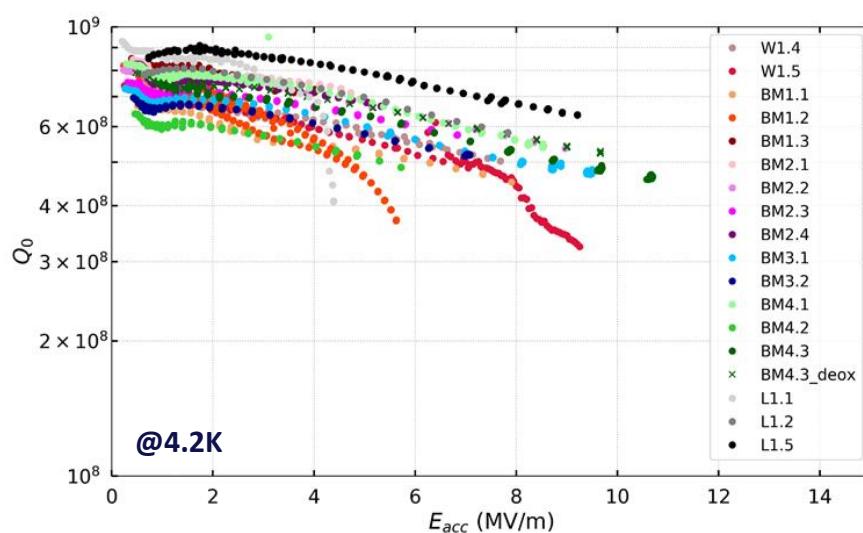
# Ongoing studies

1.3 GHz and small samples

# SRF cavities R&D



# High Pressure Water Rinsing

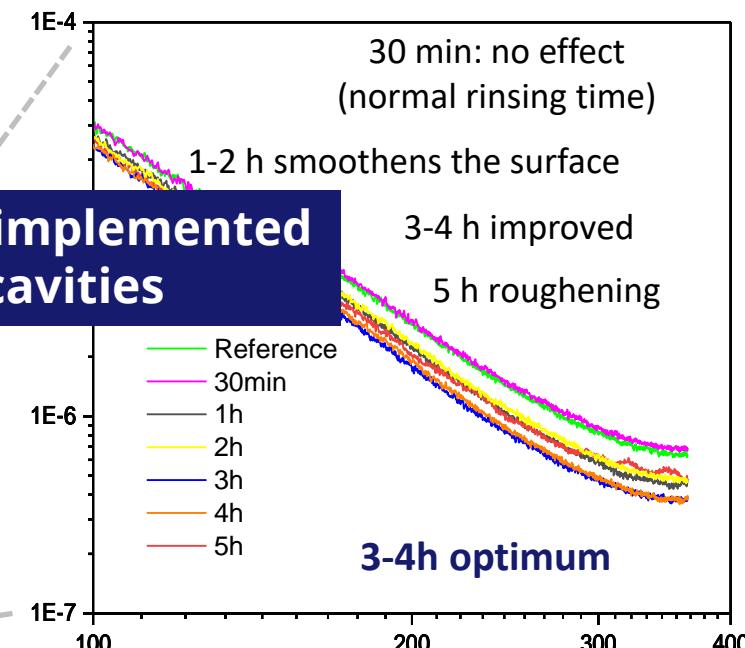
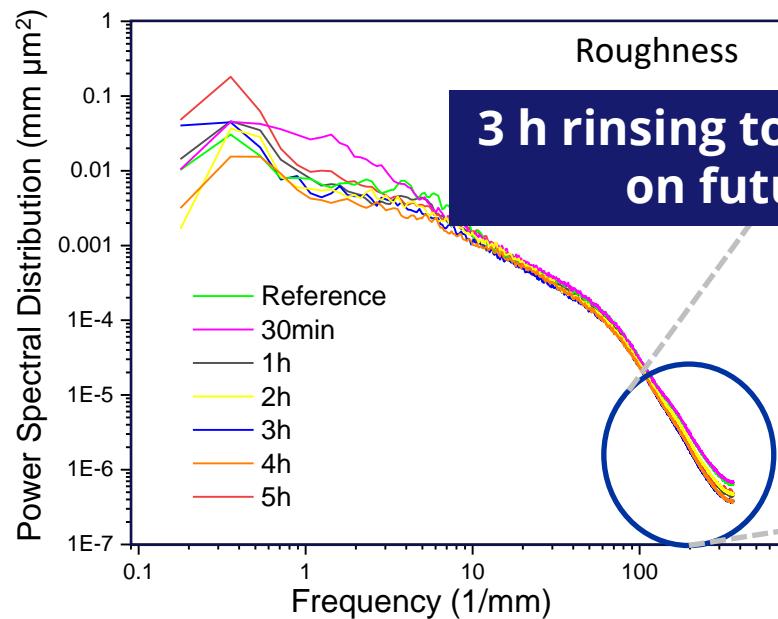


Improved cleanliness, reached higher  $E_{acc}$

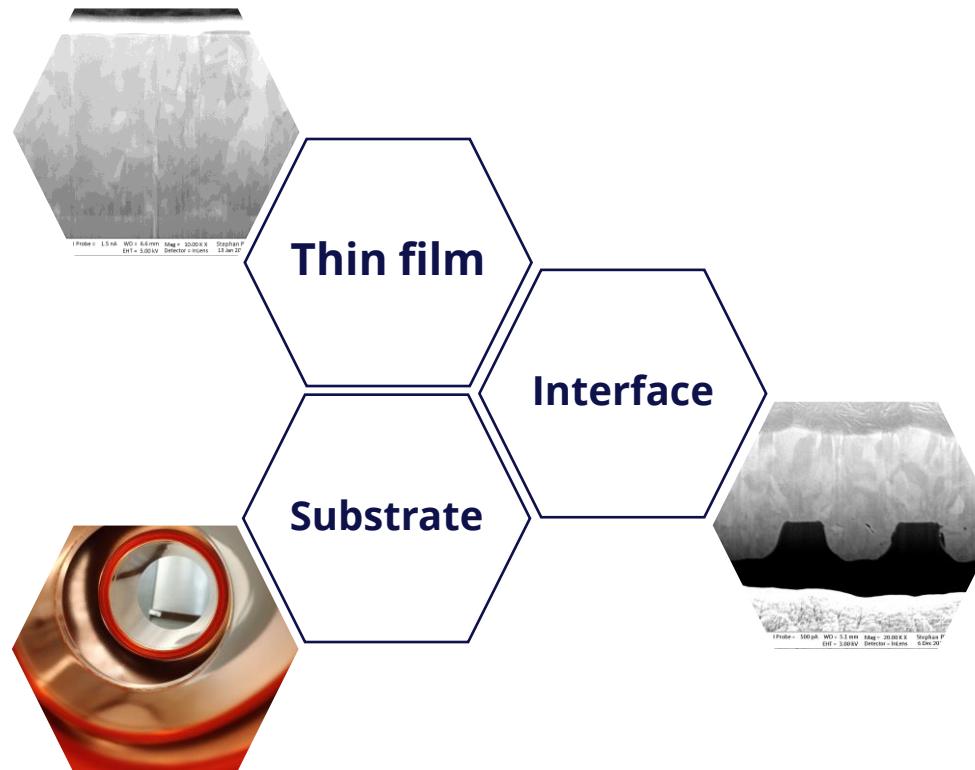
# High Pressure Water Rinsing

Effect of HWPR duration on film surface?

(smoothening, hardening...)



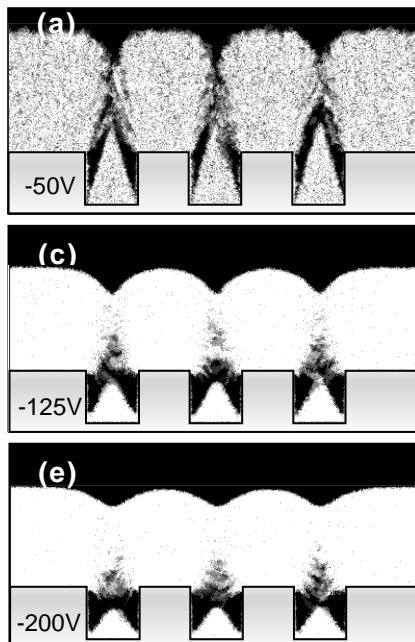
# SRF cavities R&D



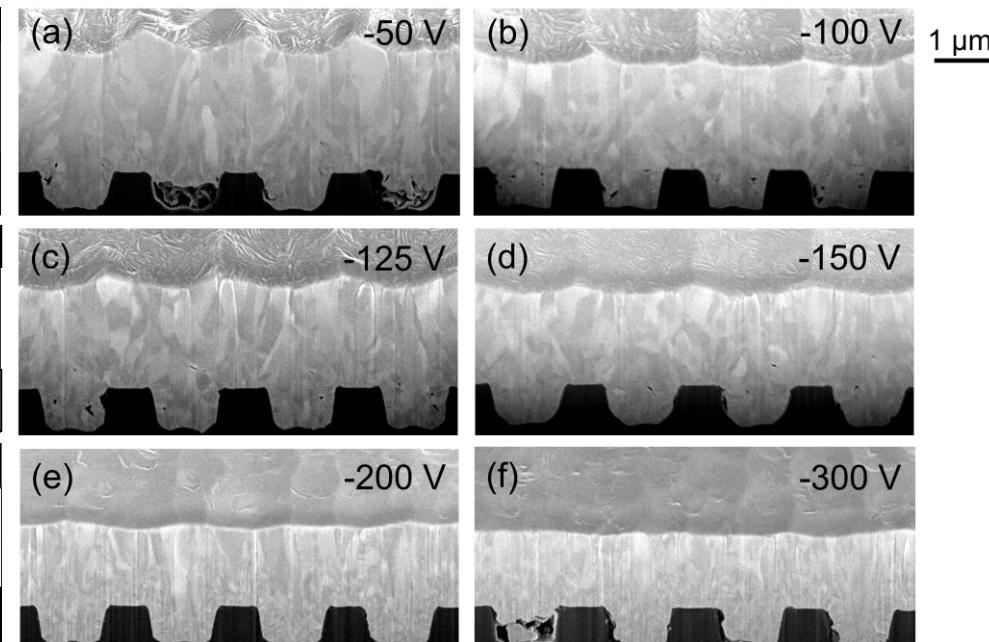
# Film planarization

Suppress surface defects “propagation”

Produce flat layer independently of substrate's roughness



Simulations (SRIM, SIMTRA + NASCAM, 70% ionization)



Experimental results, Nb on trenched Si samples

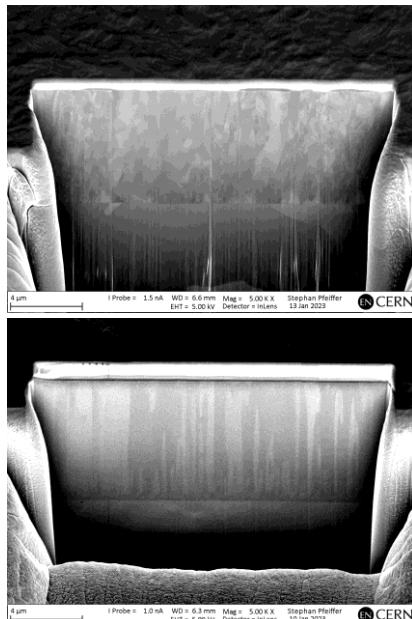
Focus Ion Beam (FIB) measurements

# Film planarization

## Nb/Cu samples

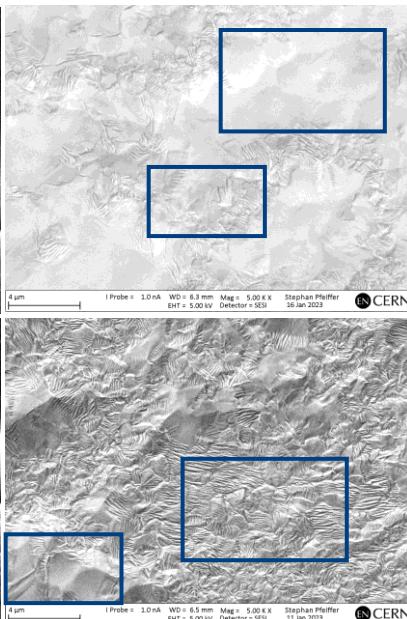
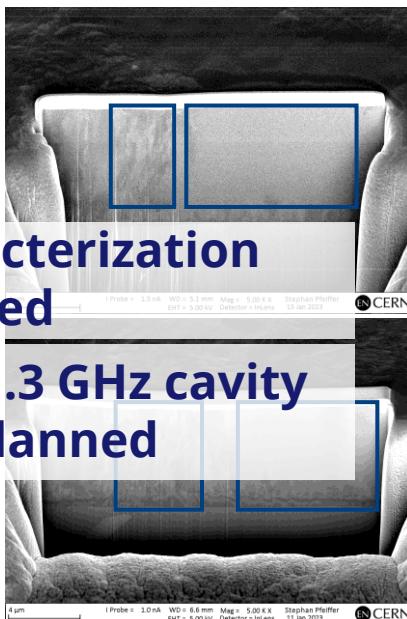
Focus Ion Beam (FIB) measurements

Reference (6 µm @ -75 V)



Further characterization  
needed

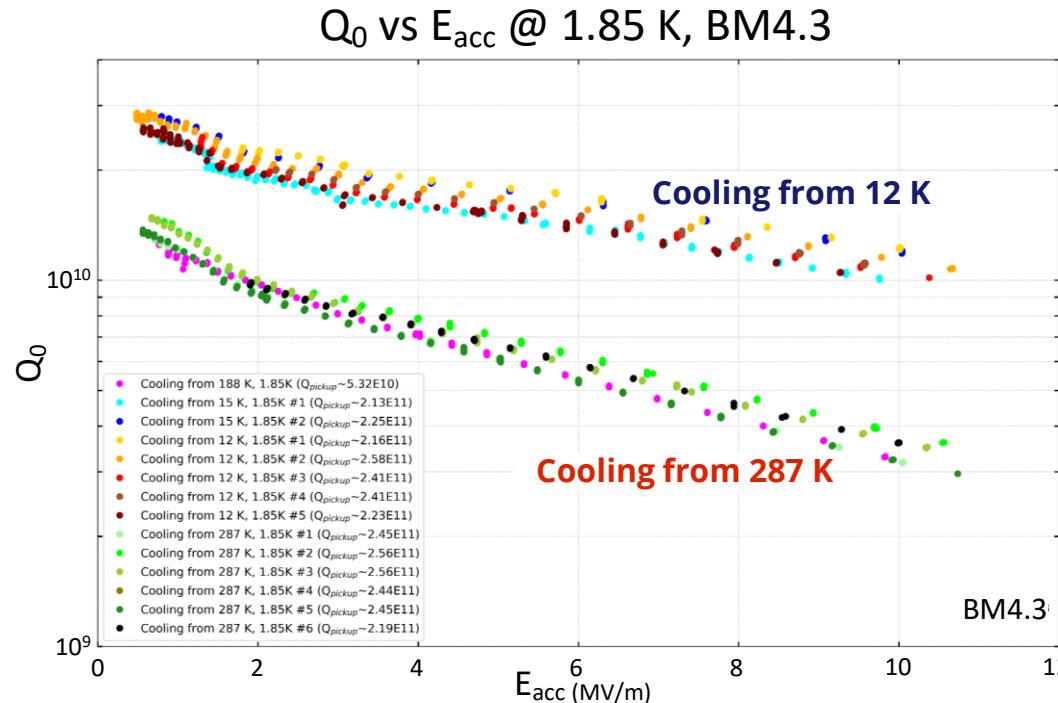
RF evaluation: 1.3 GHz  
coating planned



2 layers, 1µm @ -200 V + 5 µm @ -75 V

2 layers, 1µm @ -400 V + 5 µm @-75V \

# Thermal currents effect



Improved performance  
when thermal gradients  
are smaller



thermal currents due to metal/metal  
interface?

# Interfacial oxide

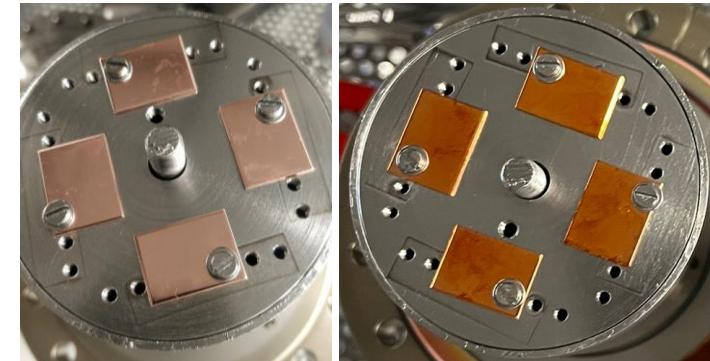
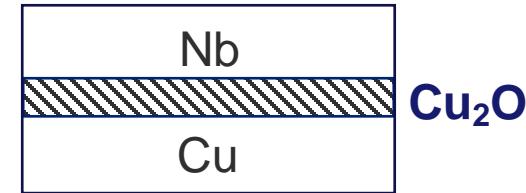
Objective: Mitigate thermal currents

Strategy: Build-up an interfacial oxide between the Nb film and the Cu cavity

Approaches:

- **In-situ thermal oxidation of Cu**

- Validated on samples (200°C, 10mbar N<sub>2</sub>/O<sub>2</sub>, 3h) Cu<sub>2</sub>O formed
- Thermal stability validated (can withstand bake out procedure)
- Cu<sub>2</sub>O survives HiPIMS coating @ -75 V of bias (confirmed by XPS)
- **1<sup>st</sup> Nb HiPIMS coating on 1.3GHz oxidized cavity 02/06/2023**



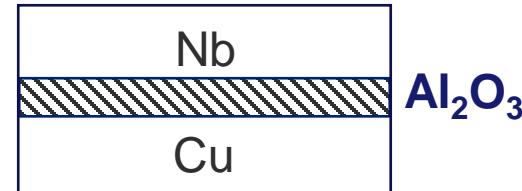
# Interfacial oxide

Objective: Mitigate thermal currents

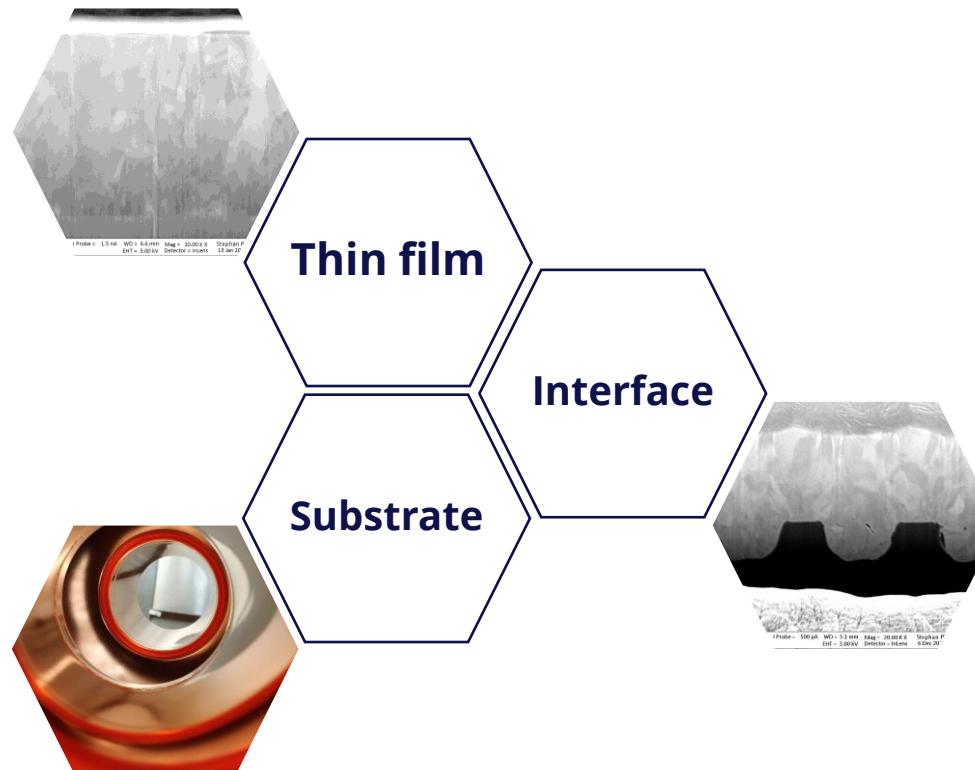
Strategy: Build-up an interfacial oxide between the Nb film and the Cu cavity

Approaches:

- **In-situ thermal oxidation of Cu**
- **Atomic Layer Deposition coating of  $\text{Al}_2\text{O}_3$** 
  - Samples provided by CEA/Saclay
  - Nb/ $\text{Al}_2\text{O}_3$  done
  - To be assessed:
    - Adhesion
    - Defect density
    - Possibility to scale up to cavities



# SRF cavities R&D

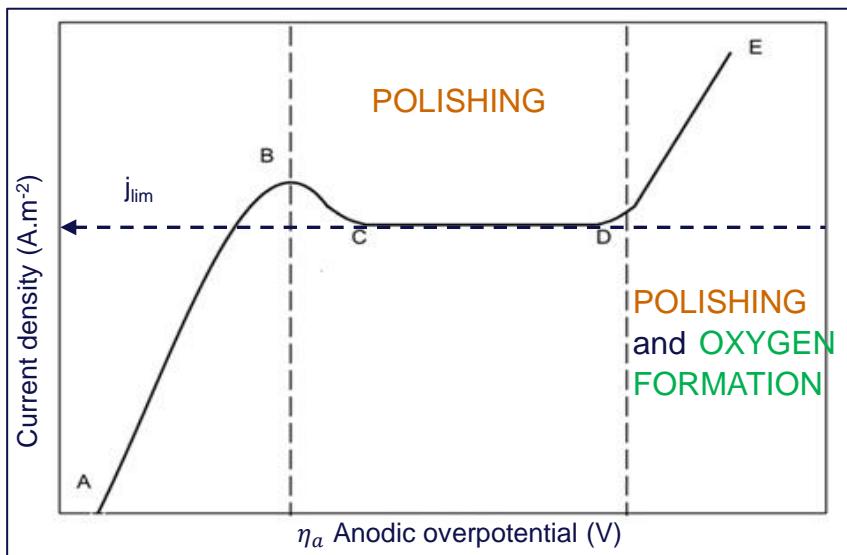


# Electropolishing

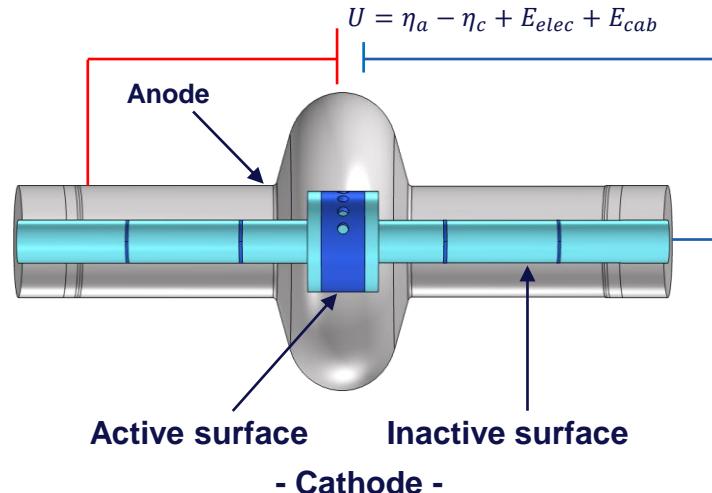
Gloria Bellini, Leonel Ferreira  
FCC Week 2023

[gloria.bellini@cern.ch](mailto:gloria.bellini@cern.ch)  
[leonel.ferreira@cern.ch](mailto:leonel.ferreira@cern.ch)

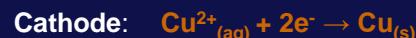
# Electropolishing - theoretical principle



Example of an anodic polarization curve



Electrolyte:  $\text{H}_3\text{PO}_4$  and n-butanol

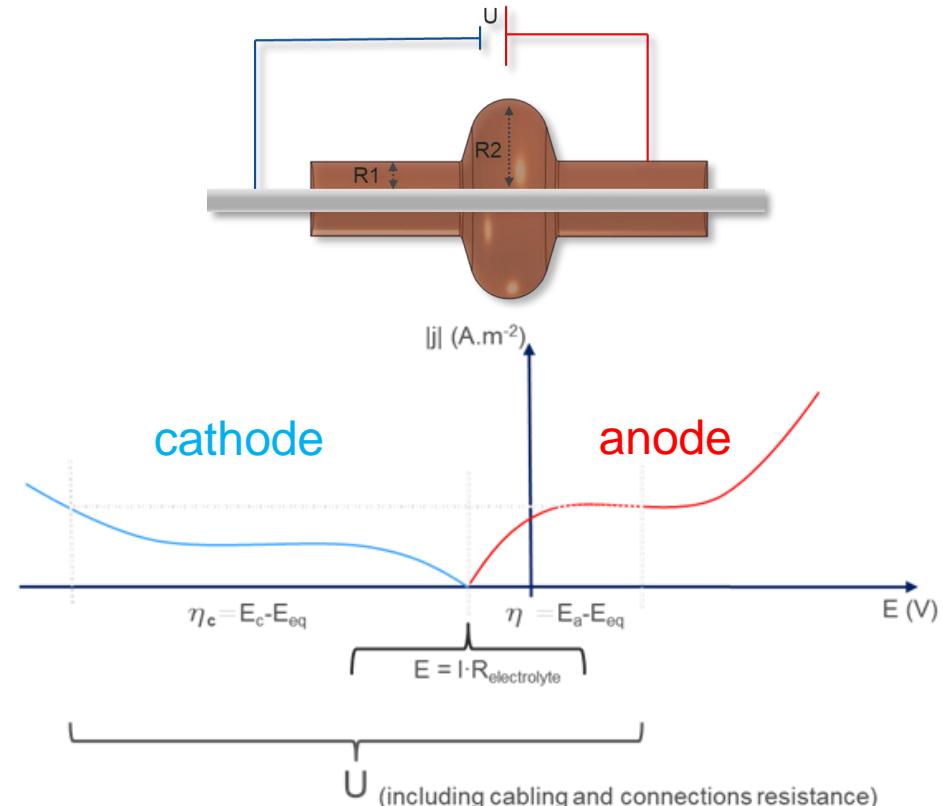


# Electropolishing modelling and optimisation

# working parameters

$$j = f(U, T, v_b, S_c/S_a, \sigma_l, [b])$$

- $j$ , Current density
- $U$ , Overall applied tension
- $T_b$ , Bath temperature
- $v_b$ , Bath fluid dynamics
- $S_c/S_a$ , Cathode geometry & Cathodic/Anodic surface ratio
- $\sigma_l$ , Bath conductivity
- $[b]$ , Bath composition



# Electropolishing modelling benchmarking



1.3GHz

Mass flow rate: 10 L/min

Temperature: 10 °C

0.5 rotations per minute

Overall applied tension: 12 V

Measured local thickness variation

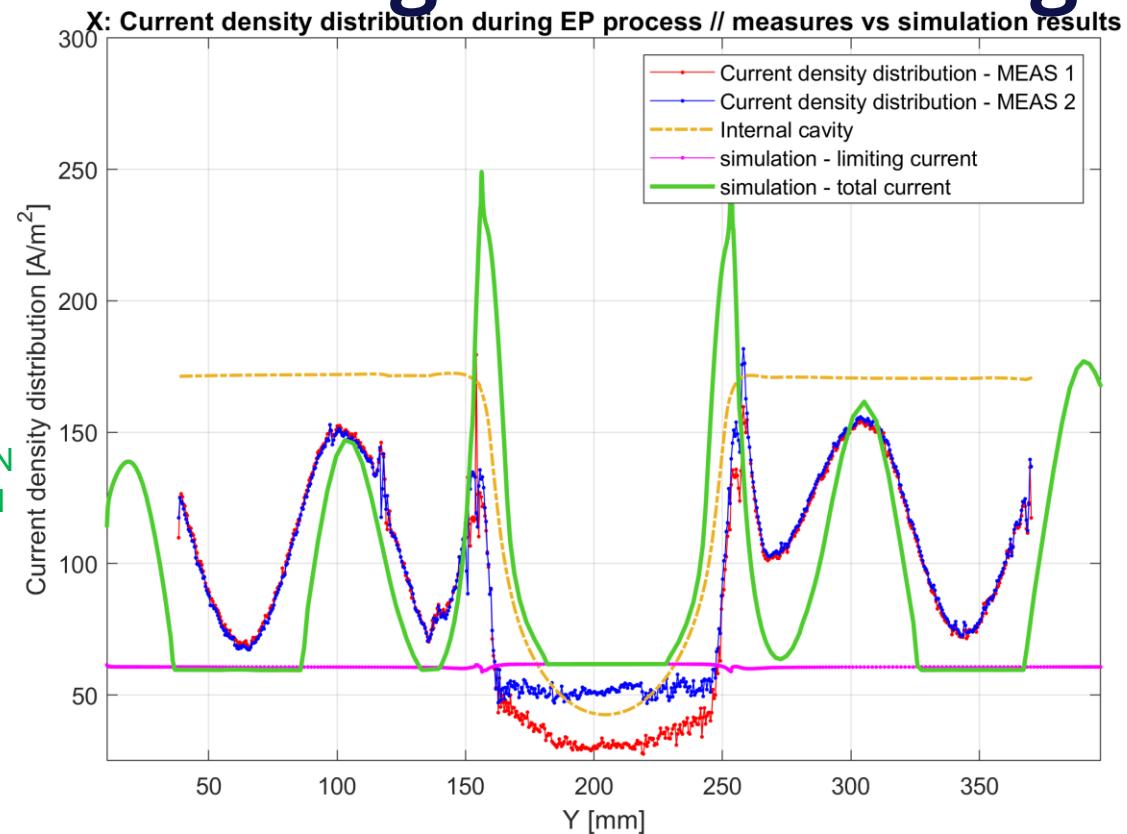
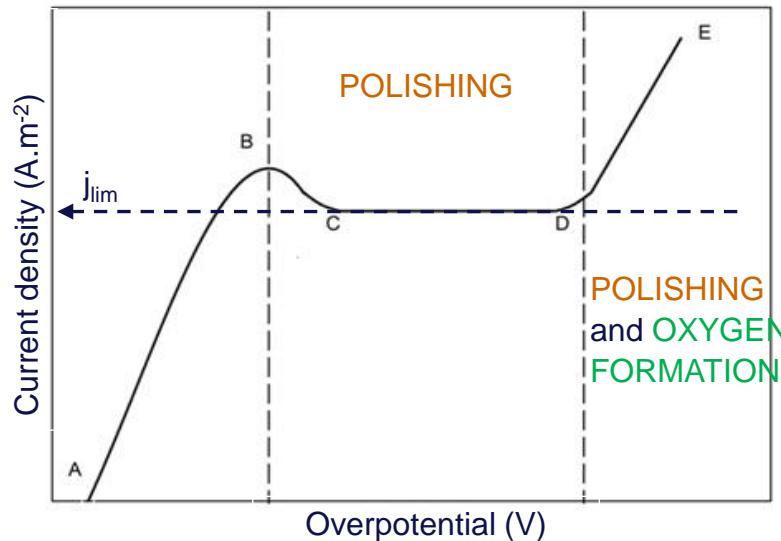
Local mass variation

$$\frac{\Delta m}{A} = \rho \cdot \Delta \text{thickness}$$

Current density distribution

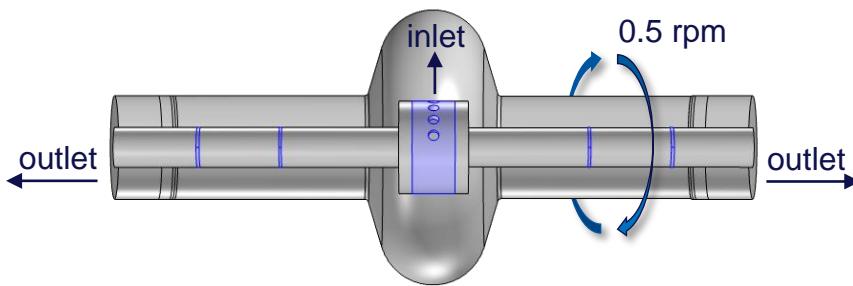
$$J = \frac{n \cdot F \cdot \Delta m}{\Delta t \cdot M \cdot A}$$

# Electropolishing modelling benchmarking

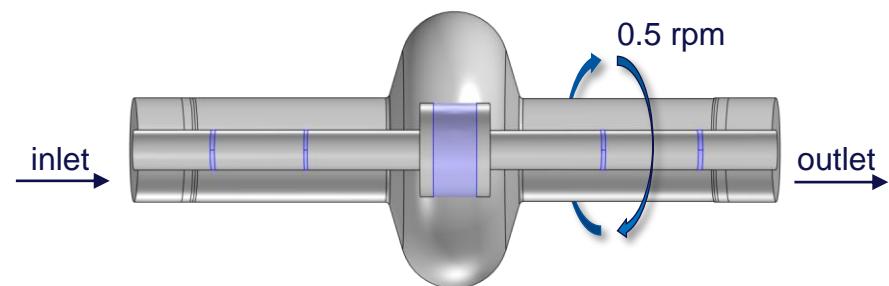


# Cathode optimisation 1.3GHz RF cavity

Before benchmarking\*



After benchmarking



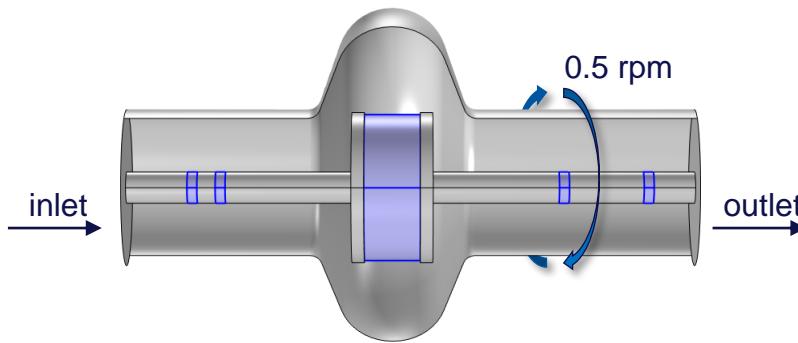
Mass flow rate	10 L/min
$T_{\text{inlet}}$	15 °C
Overall applied tension	10.6 V
Power input	75.5 W
$T_{\text{inlet}} - T_{\text{outlet}}$	0.17 °C

Mass flow rate	10 L/min
$T_{\text{inlet}}$	10 °C
Overall applied tension	9 V
Power input	<b>60.4 W</b>
$T_{\text{inlet}} - T_{\text{outlet}}$	0.14 °C

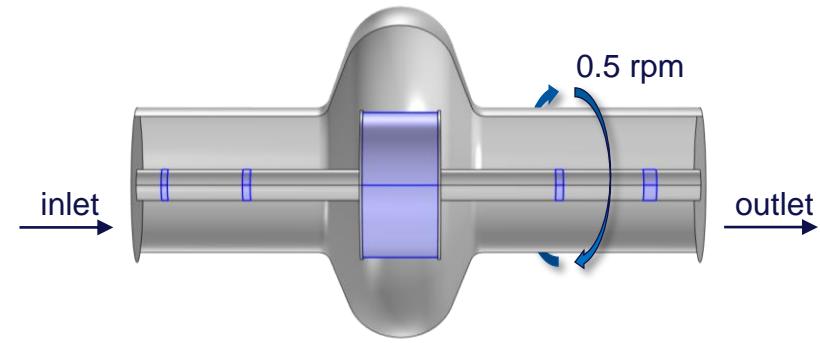
\*Electropolishing 1300 & 400 MHz SRF copper FCC week 2022 .pdf (cern.ch)

# Cathode optimisation 400MHz RF simplified cavity

Before benchmarking\*



After benchmarking

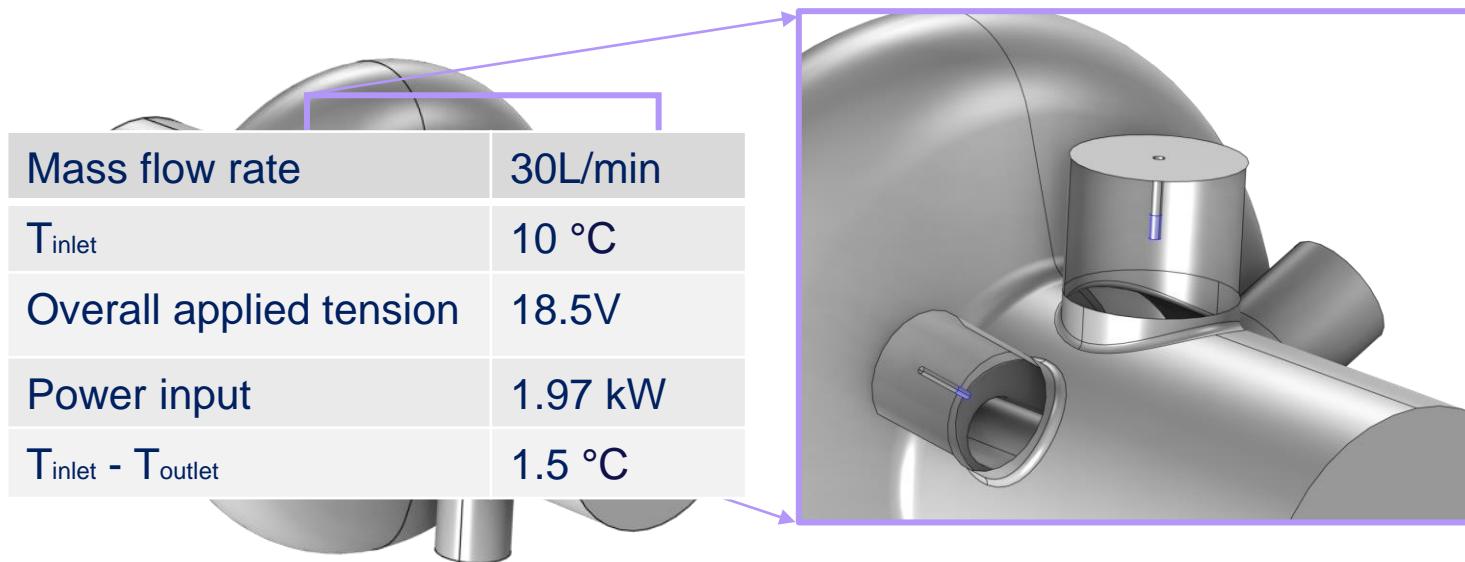


Mass flow rate	30 L/min
$T_{inlet}$	15 °C
Overall applied tension	20.5 V
Power input	2.2 kW
$T_{inlet} - T_{outlet}$	1.7 °C

Mass flow rate	30 L/min
$T_{inlet}$	10 °C
Overall applied tension	18.5 V
Power input	<b>1.97 kW</b>
$T_{inlet} - T_{outlet}$	1.5 °C

\*Electropolishing 1300 & 400 MHz SRF copper FCC week 2022 .pdf (cern.ch)

# Cathode optimisation 400MHz RF FCC cavity

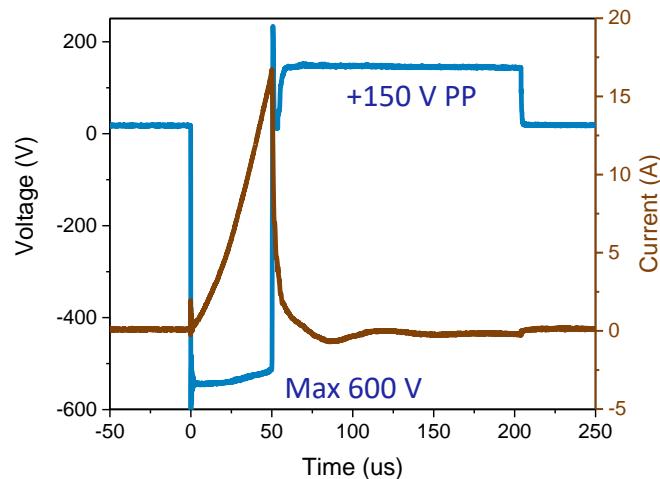




400 MHz cavities

# 400 MHz HiPIMS

Bipolar HiPIMS coating of PC04 on 05/2022  
(simplified cavity, no HOM ports)



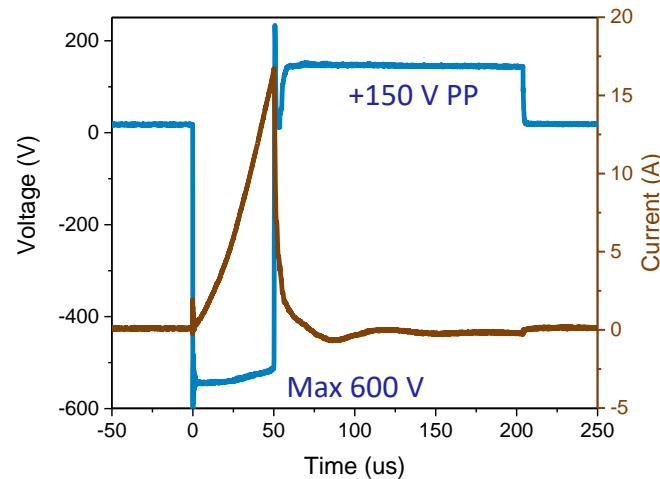
HiPIMS hardware limitations

Limited to ~1kW average power



# 400 MHz HiPIMS

Bipolar HiPIMS coating of PC04 on 05/2022  
(simplified cavity, no HOM ports)



HiPIMS hardware limitations

Limited to ~1kW average power

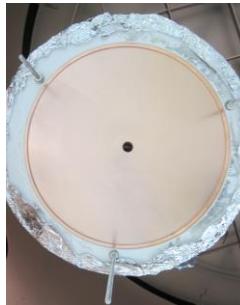
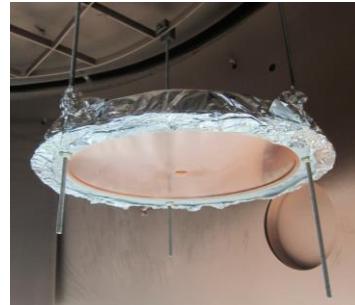


# 400 MHz HiPIMS

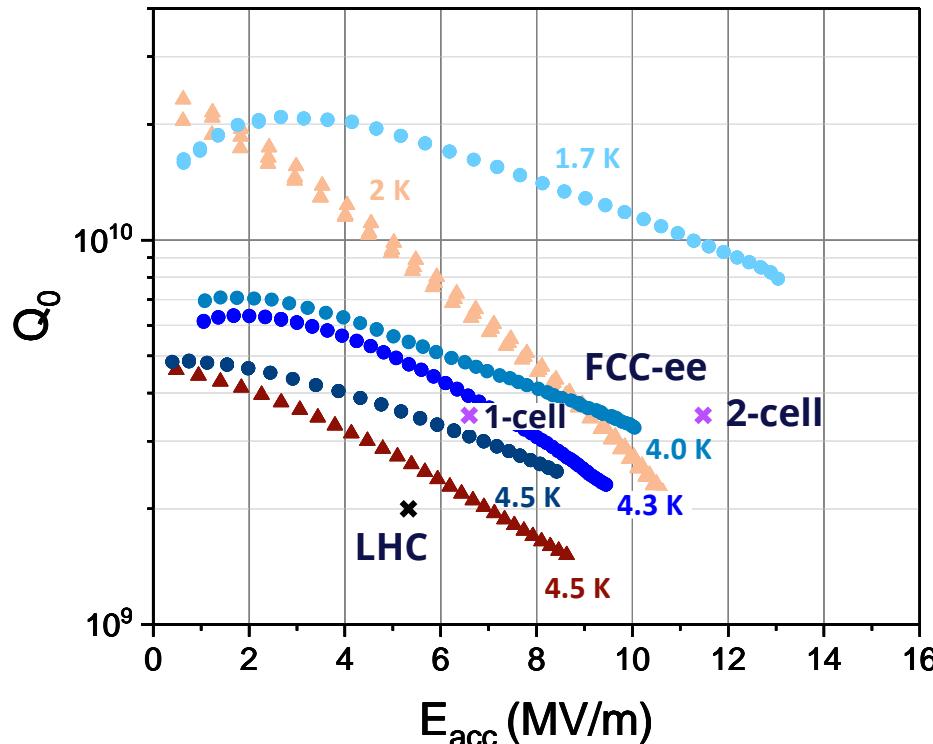
SUBU + HiPIMS coating + LPWR + RF testing (2022)



**HPWR + Nb HiPIMS coated flanges + RF testing  
(2023)**



# 400 MHz HiPIMS: results 2023

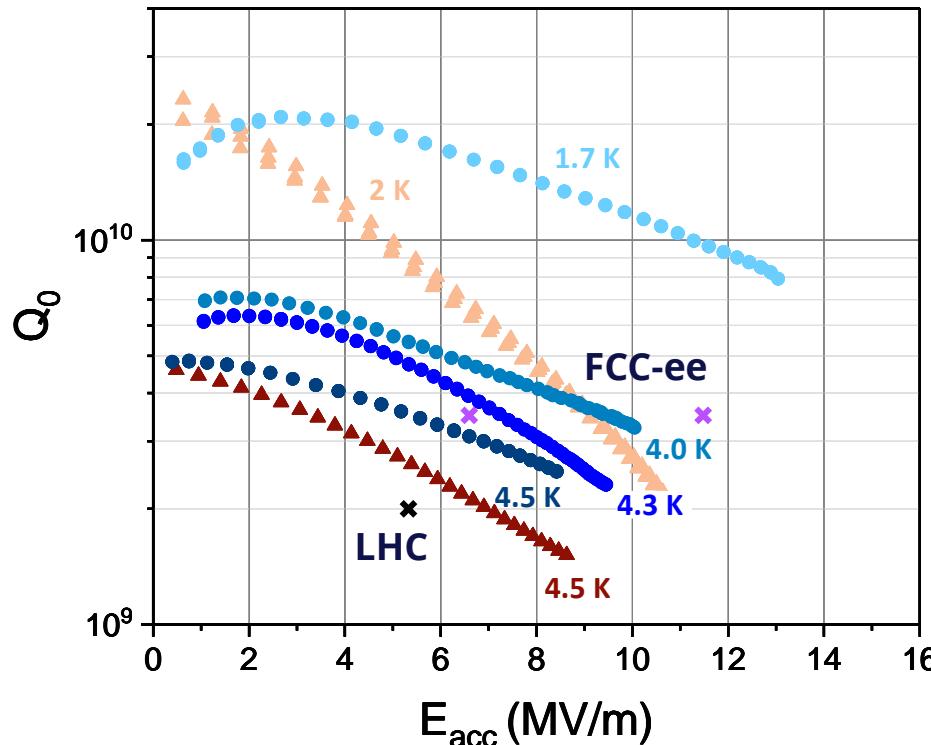


- ▲ "LHC type" (DCMS)
- HiPIMS
- HiPIMS after processing at 2K

**Higher  $Q_{0s}$  and mitigated Q-slope compared with DCMS**

**Crossing of HiPIMS' 4 K Q-curve with the 2 K of DCMS  
~9MV/m**

# 400 MHz HiPIMS: results 2023



Very promising result:

- **No electropolishing** and this surface treatment has demonstrated its strong impact on 1.3 GHz cavity performance.
- **coating was not performed in optimal conditions**
- **cavity was not been internally e-beam welded, nor it is a seamless substrate**, which would lead to an improved surface state

# 400 MHz HiPIMS: future work



- Optimize Bipolar HiPIMS 400 MHz system to fix hardware limitations
- Cu Electropolishing
  - EP of PC04 (simplified cavity, no HOM ports) + coating
  - EP of NCO2 cavity + coating (simplified EP, not on HOM ports)
  - EP of NCO2 cavity + HOM ports + coating
- Thicker layer (6 µm against currently 1.5 µm) as optimized on 1.3GHz cavities
- Biased HiPIMS system to be tested (under manufacturing)
- Coating on 400 MHz seamless substrates

# Summary

## 1.3 GHz cavities previous results:

**Improved performance + mitigated Q-slope, promising for FCC-ee target performance**

Seamless substrates + electropolishing & passivation + optimized (low  $J_c$ ) parameters of Nb biased-HiPIMS

## Additional ongoing studies on samples and 1.3 GHz cavities to further improve cavity performance

$J_c$  study + HPWR scheme optimization + film planarization + creation of an interfacial oxide to mitigate thermal currents + RF ready surfaces with no chemistry + Cu electropolishing optimization

## 1<sup>st</sup> HiPIMS coated 400 MHz SRF cavity

**Improved RF performance with lots of space for further progress** due to coating, substrate and substrate treatment optimization



Thank you  
for your attention!

# EP: conclusions

The built model foresees the impact of fluid dynamics on EP.

Regarding the electrochemical process, the latest work allowed to get still some improvements.

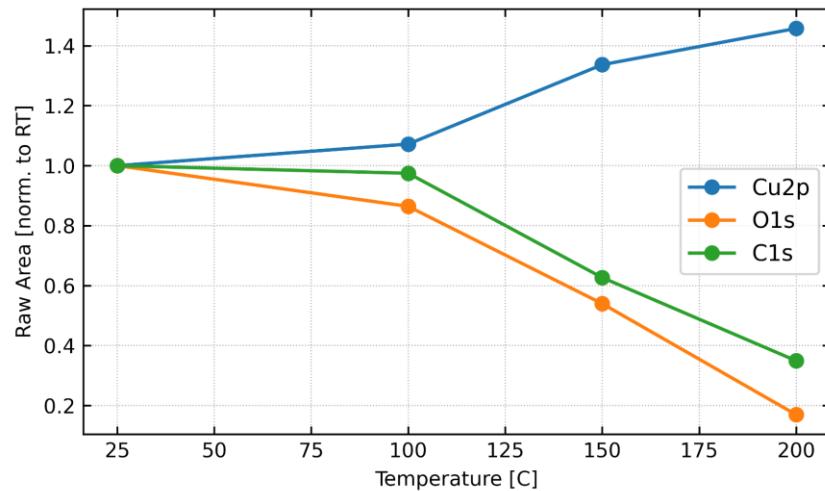
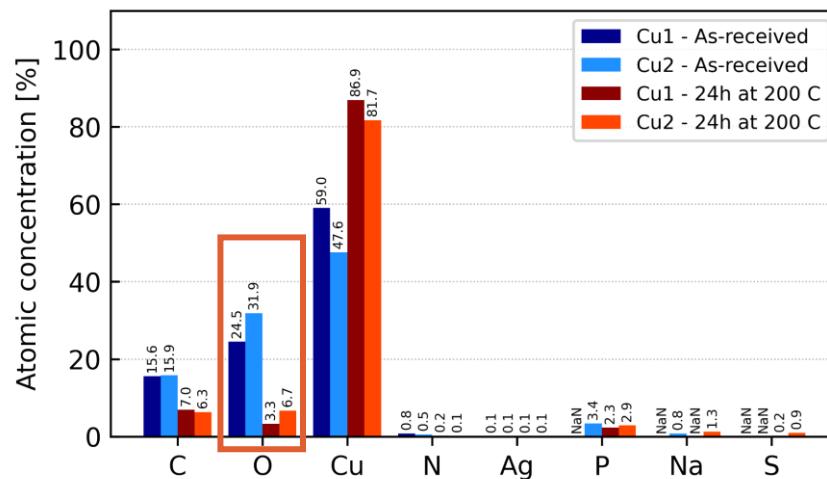
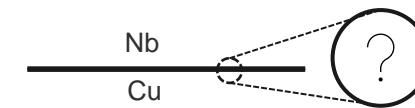
Still some features to be understood:

- Ongoing analysis on oxygen bubble formation and impact of EP
- Diffusion layer build up by natural convection

# Interfacial oxide

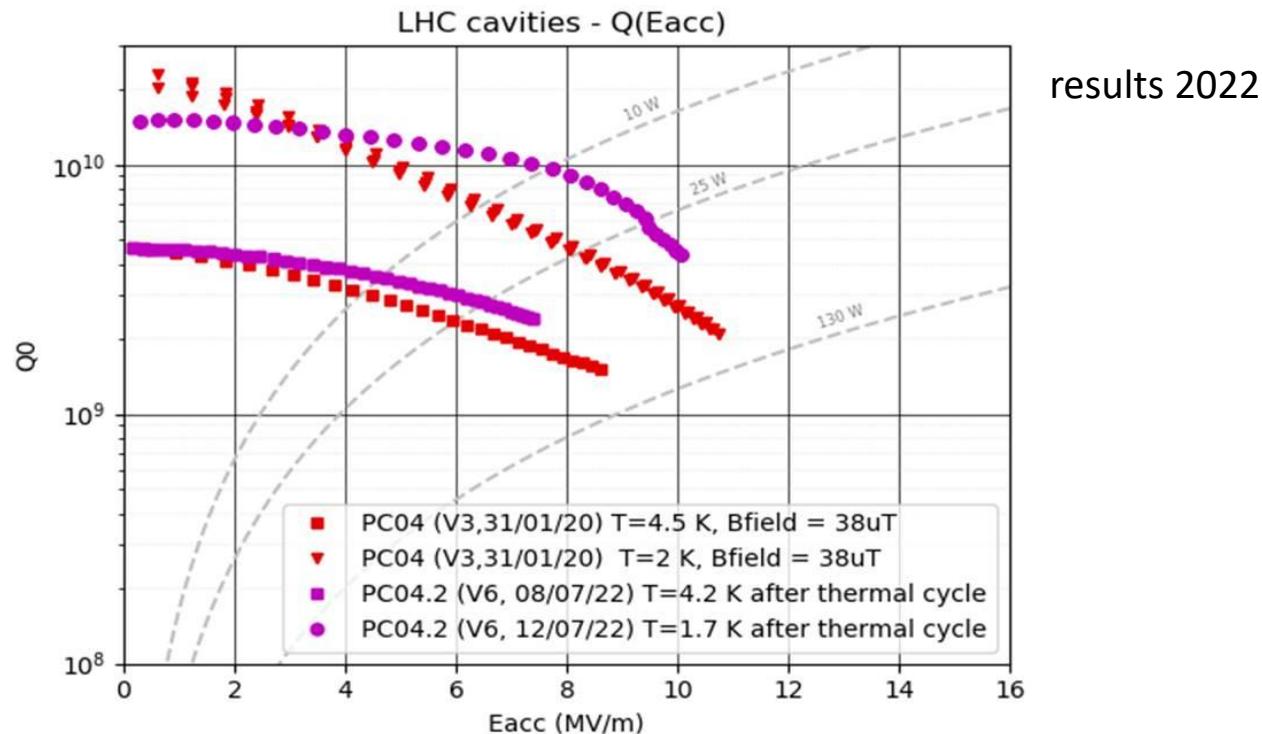
Cu samples, EP + passivation

XPS *in situ*, heat treated to replicate BO process

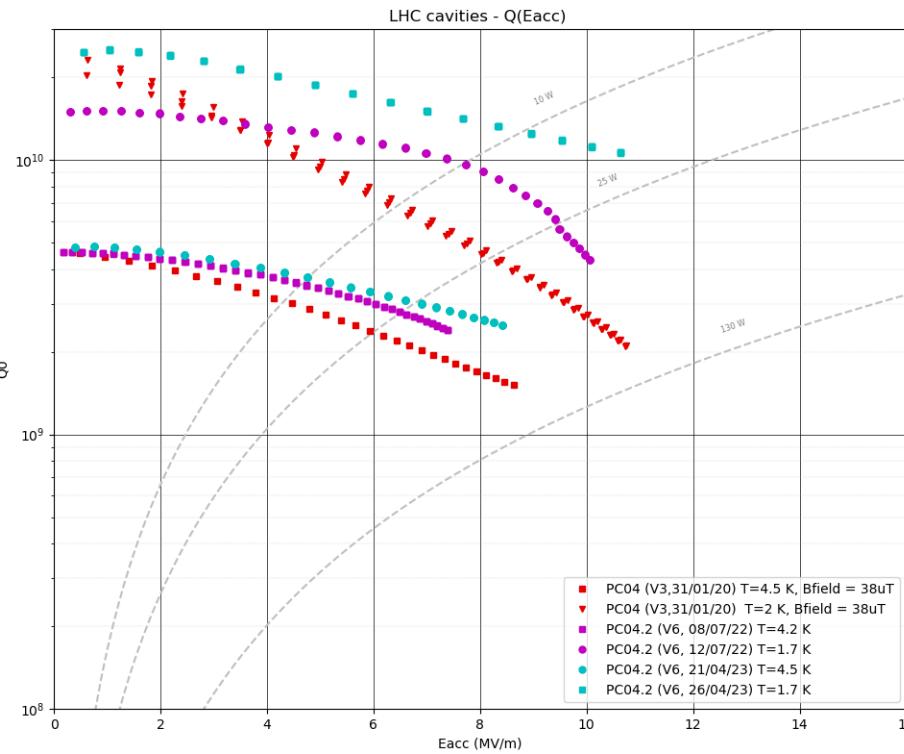


**No interfacial oxide layer**, also confirmed by XPS depth profile analysis

# 1<sup>st</sup> HiPIMS-coated 400 MHz cavity



# 1<sup>st</sup> HiPIMS-coated 400 MHz cavity



results 2023 vs 2022: effect of Nb  
flanges + HPWR