

HiPIMS Nb/Cu at 1.3 GHz

A-M VALENTE-FELICIANO

S. Bira, O. Hryhorenko, M. Ge, E. Lechner, U. Pudasaini, O. Trofimova

Outline

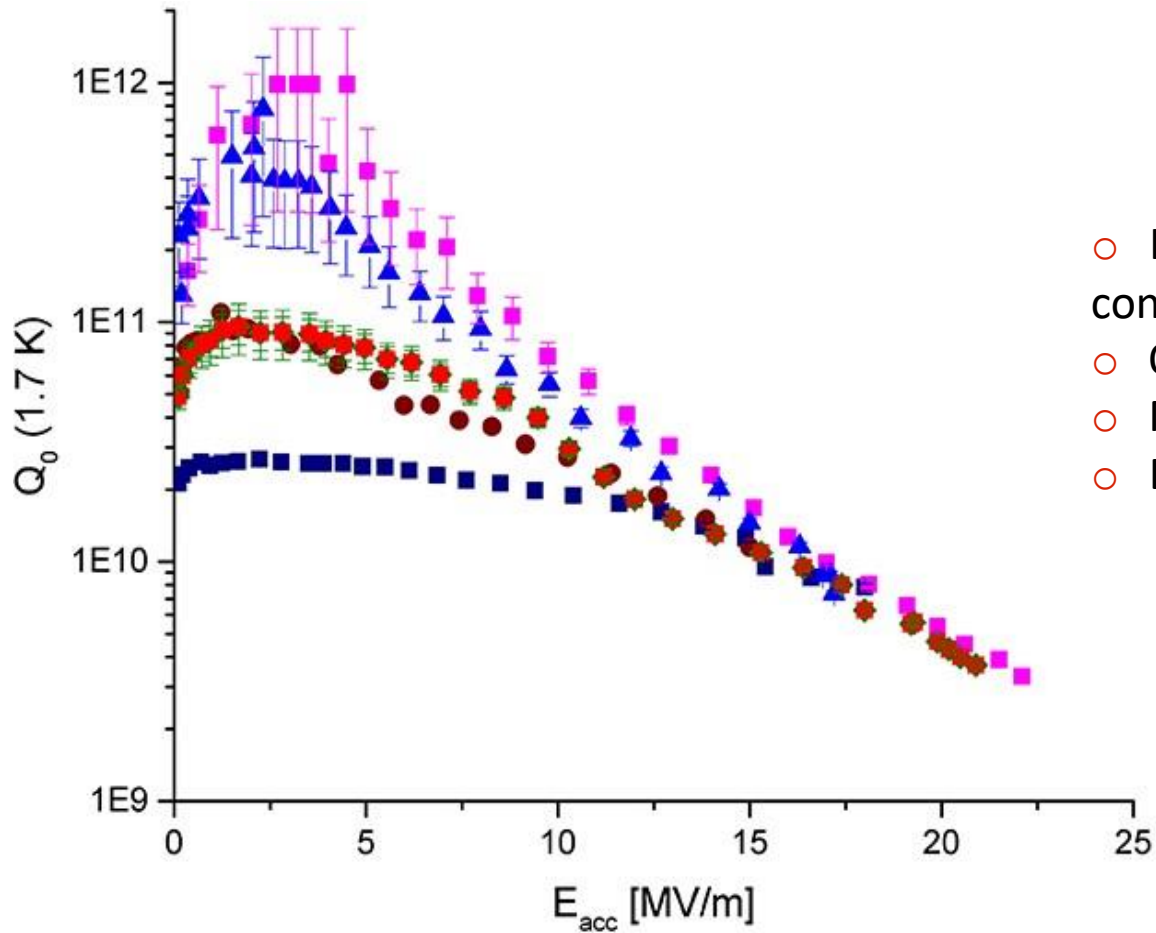
- Approach
- Substrates
- Nb on Cu
- Nb on Nb
- Summary & Remarks

Background

Nb/Cu Technology proof of principle with LEP2, LHC, ALPI machines

Great potential for cost savings and operational advantages for machines operating at lower frequency and relatively modest gradients

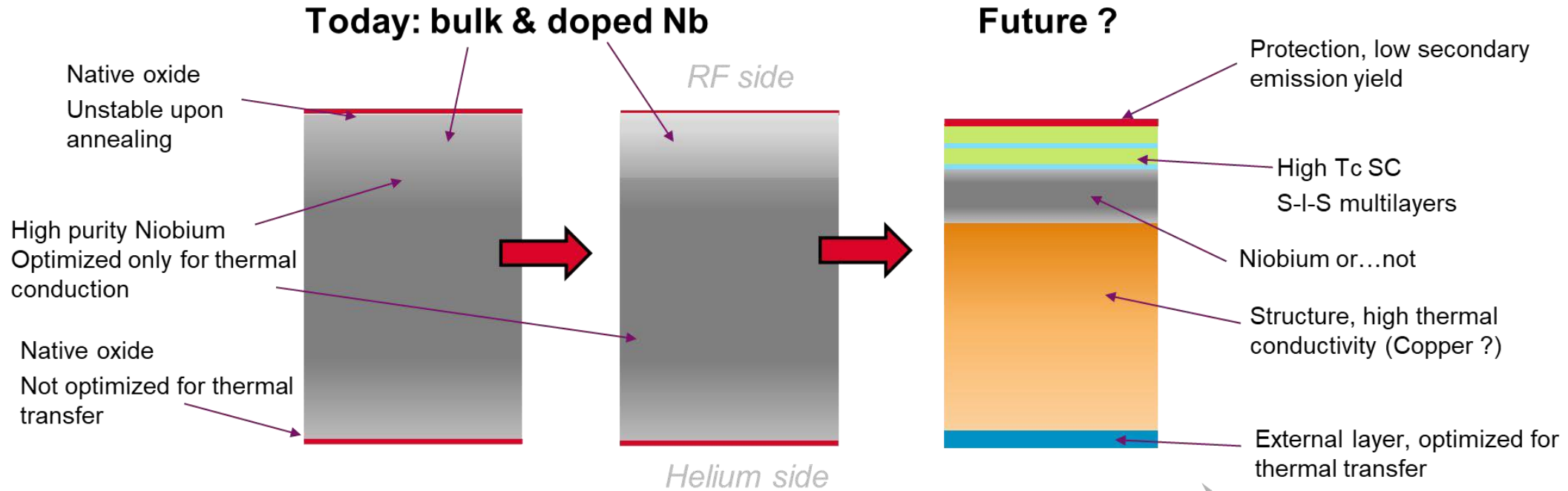
high current storage ring colliders : FCC, EIC and CEPC



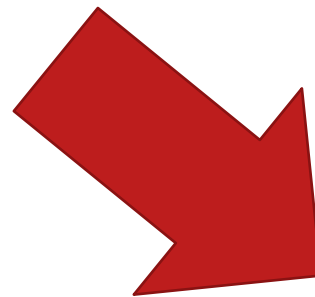
- Increased temperature stability due to Cu substrate higher thermal conductivity
- Operation at 4.5 K, generating capital and operational cost savings
- Material cost saving, particularly for low frequency structures
- Easily machinable and castable structures

Perspectives for significant cryomodule simplification.

SRF Thin Films for Next-Generation Cavities



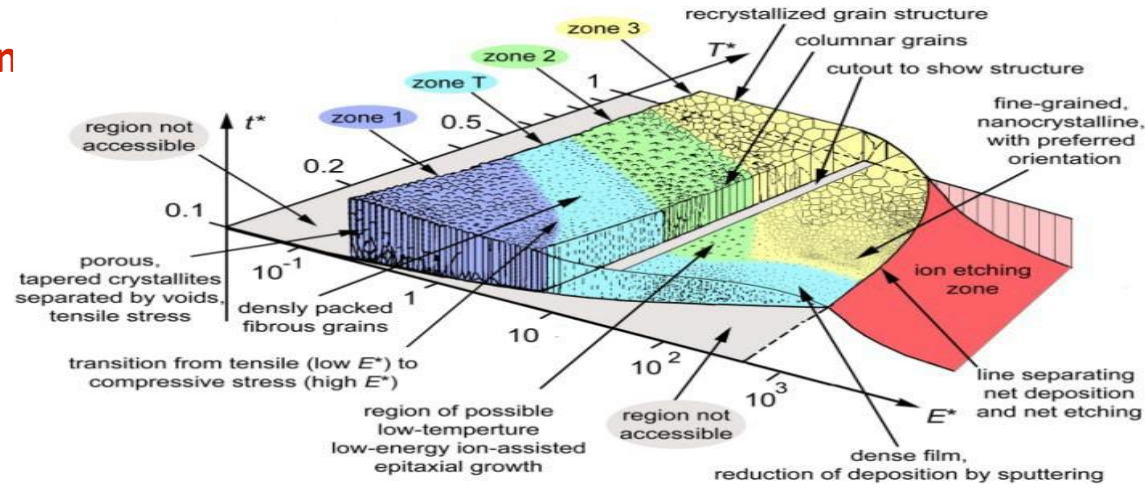
From C. Antoine



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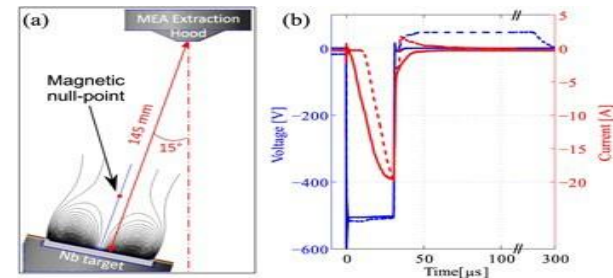
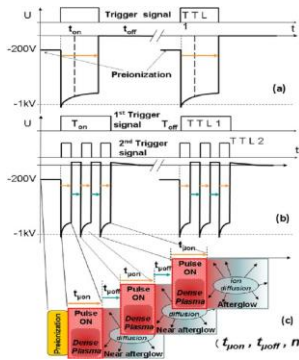
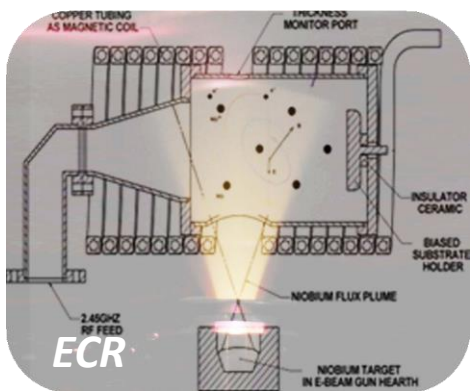
Advancing Thin Film Nb on Cu Technology

Energetic Condensation



Anders, André. "A structure zone diagram including plasma-based deposition and ion etching." *Thin Solid Films* 518.15 (2010): 4087-4090.

Novel deposition techniques exploiting species energetics offer opportunities to improve & manipulate film structure & performance



F Avino et al 2019 *Plasma Sources Sci. Technol.* 28 01LT03

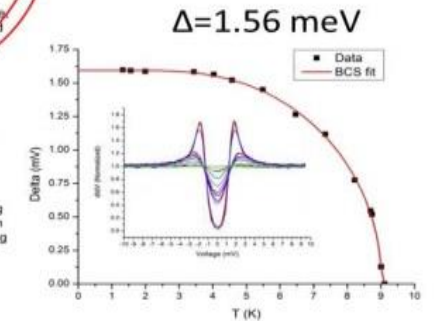
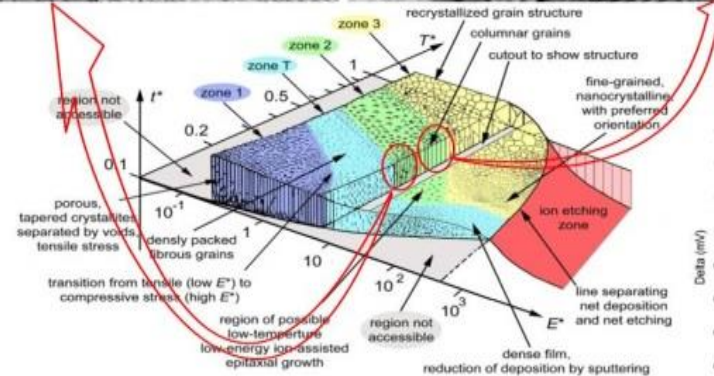
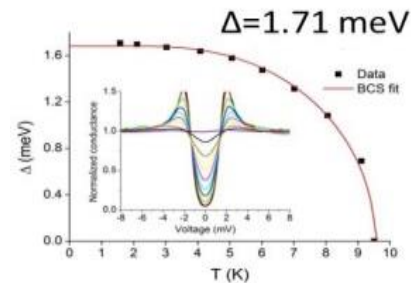
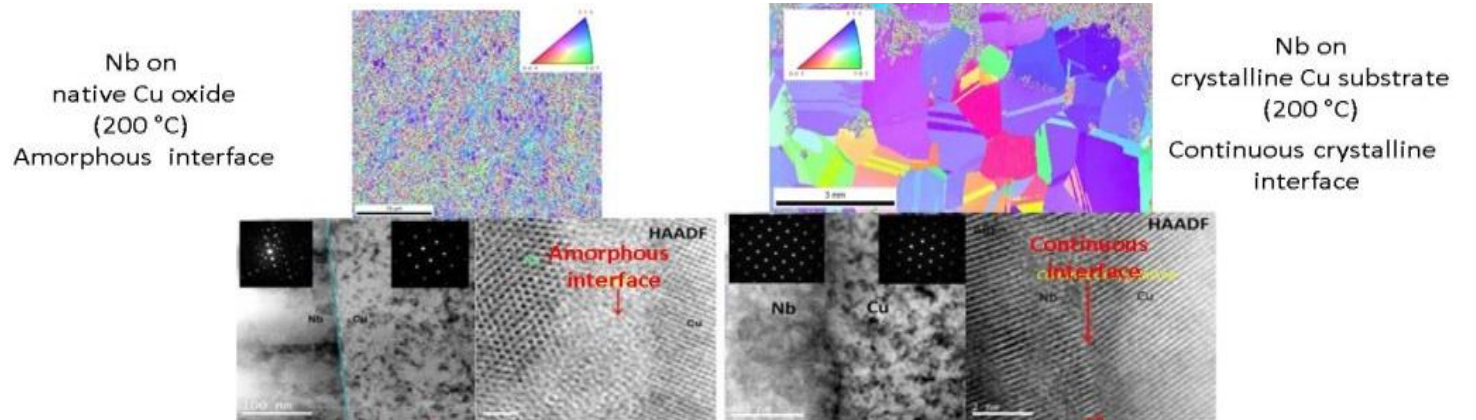
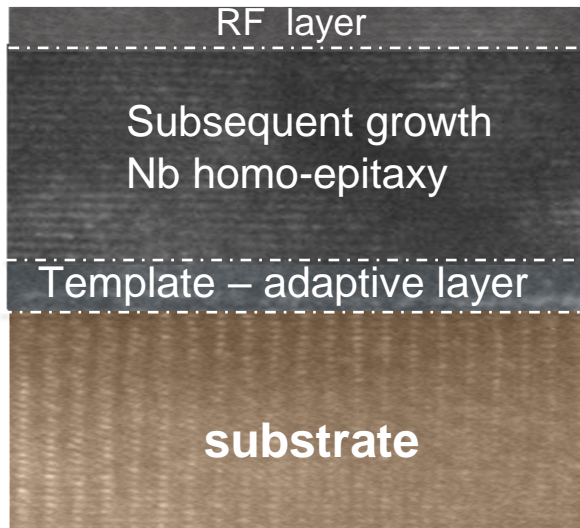
HiPIMS + kick pulse

Nb Film Development Approach

taking advantage of energetic condensation

Control over nucleation & subsequent growth, thus structure

Sequential phases for film growth



- ❑ Interface
- ❑ Film nucleation
- ❑ Growth of appropriate template for subsequent deposition
- ❑ Deposition of final surface optimized for minimum defect density.

Full control over final SRF performance with strict process protocols

Control over thin film structure, quality (RRR values), properties, impurity (H) content, adhesion to the substrate

SUBSTRATES

HiPIMS Nb/Cu at 1.3 GHz

Standard Cu Substrates

Fabrication via deep-drawing & E-beam welding

Surface preparation with SUBU & Sulfamic rinse



Beam pipe cuff weld



Equator weld



Bulk machined 1.3 GHz Cu substrates

Machined-in-bulk 1.3 GHz Cavity

Specific process definition for bulk machining

- Diamond finishing
- Roughness $R_a \sim 0.15 \mu\text{m}$ (diamond machining)
- Max. internal shape deviation $\sim 20 \mu\text{m}$
- Wall thickness variations $< 20 \mu\text{m}$

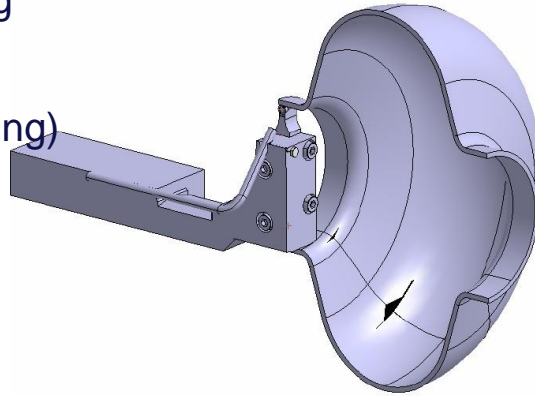
Advantages:

- No equator weld
- No mechanical stress from deformation due to deep drawing

“Ideal Substrate” to Best Performance in terms of coating & RF results !

For coating Studies only

- Alternative process for suitable large scale production yet to be developed



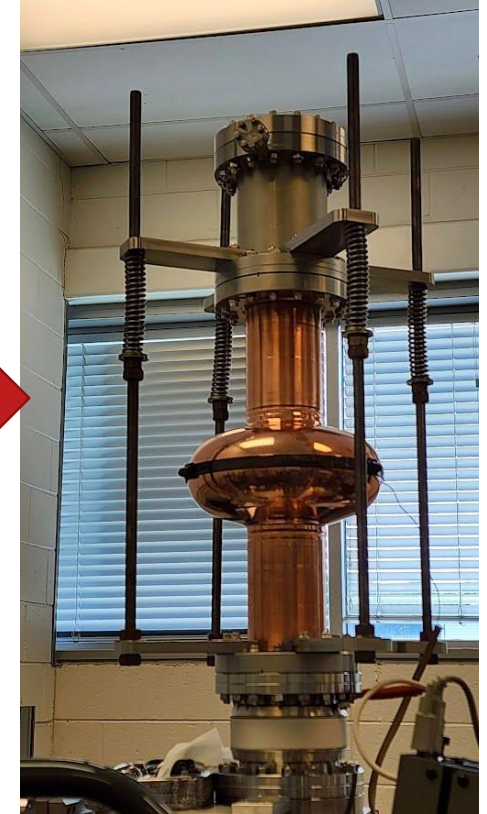
CERN Ref. : Karol SCIBOR

Order of 2 initiated June 2021

Ordered 2 more

1st two cavities received 05/23/23

2 last received 03 & 04/2024



4 cavities received

3 sent back for re-processing

Hydroformed 1.3 GHz Cu substrates

HYDROFORMING STRATEGIES

Baseline

two-step Hydroforming

ONE intermediate heat treatment

- Studied and optimized through simulations
- Based on detailed material characterization
- Verified against failure model

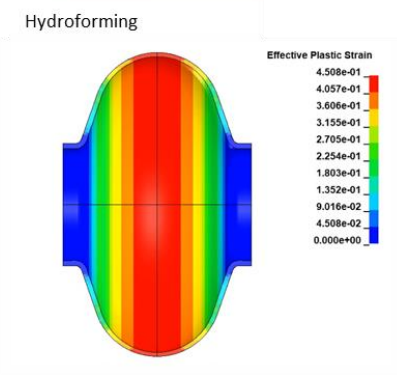
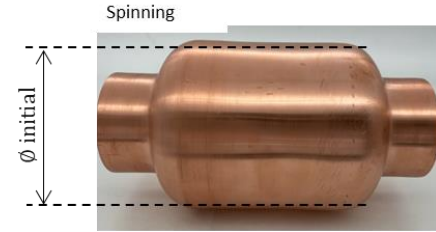
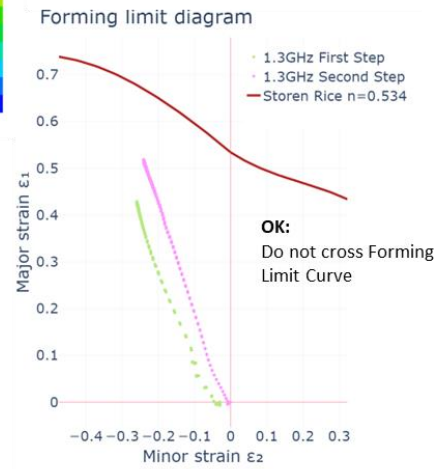
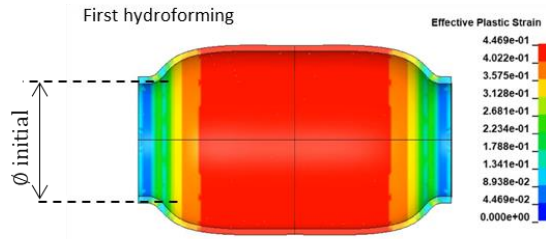
Both strategies are feasible with the material properties of tested Cu-OFE

Necking + Hydroforming

NO intermediate heat treatment

KEK: *A. Yamamoto, M. Yamanaka*

- Raw material procurement
- Tools design and manufacturing
- Hydroforming trials



On hand:

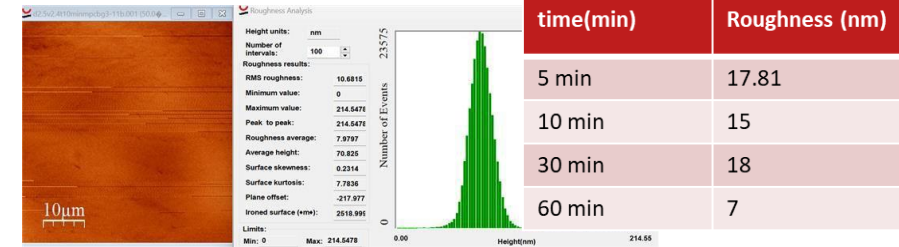
- ✓ 2 cavities from KEK
- ✓ 6 cavities from previously Bailey Tools (Texas)

Cu Substrate Preparation

Cu Electropolishing

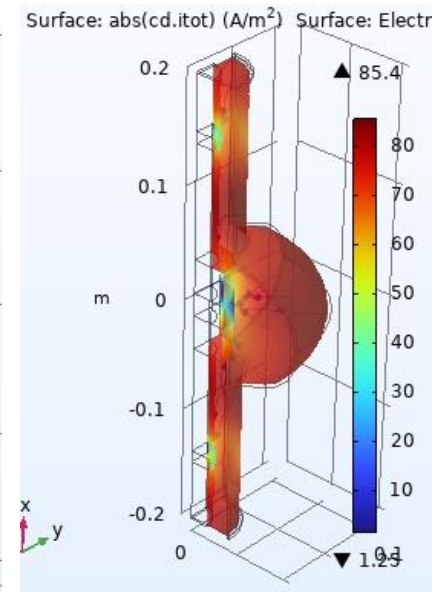
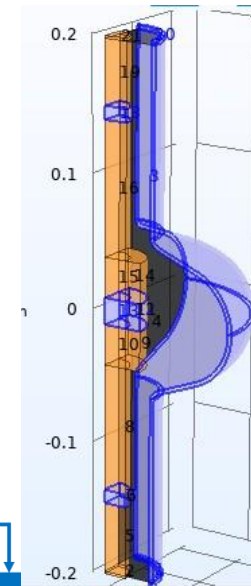
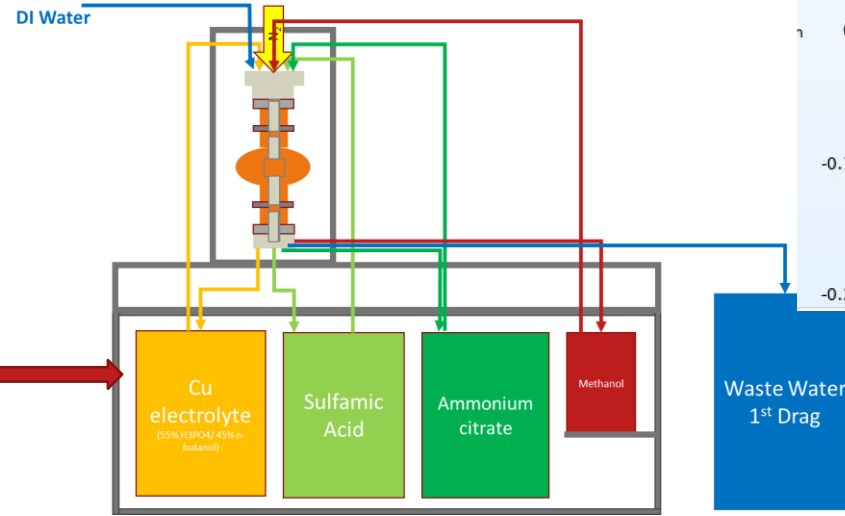
S. Bira, N. Brock

- Use analytical electrochemistry and surface characterization to develop Cu electropolishing method for Cu substrates
- System being design for operation in a walk-in fume hood (R&D Chemroom)
- Interaction with CERN
- **Homogeneous material removal from each part of a elliptical cavity resulting in uniformly smooth surface**
 - ✓ - Measure electrolyte properties (viscosity vs. T, I-V curve as a function of T)
 - ✓ - Sample EP experiments to study the roughness as a function of distance from the cathode, temperature, and time
 - ✓ - COMSOL multi-physics simulation to identify several set of (optimized)parameters
 - - Electropolishing experiment
 - Samples EP using a profile cavity to validate simulation results and identify optimized parameters for actual cavity
 - Cavity EP studies



Cavity Setup

Cu electropolishing integrating the developed cathode design
 DI water rinse
 Sulfamic rinse
 DI water rinse
 Passivation with Ammonium Citrate DI water rinse to 18 MΩ resistivity
 Methanol Fill 9< 1 gal.
 N2 dry with methanol gravity



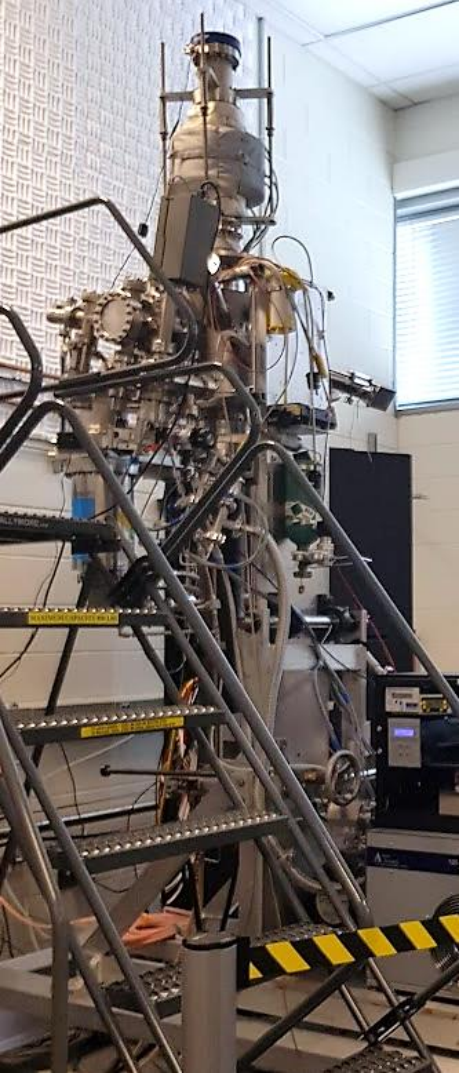
Cu Plasma Electropolishing – evaluation on QPR substrates

Collaboration with INFN-LNL

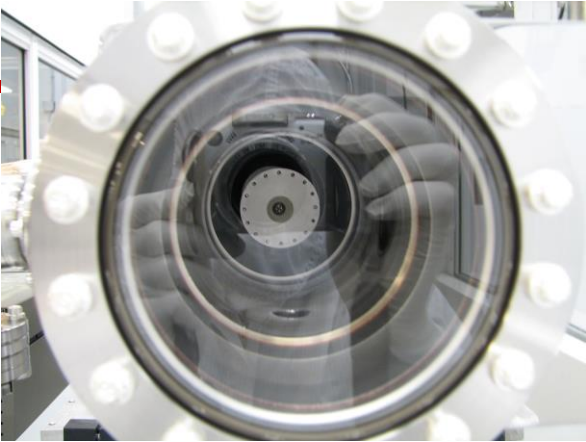
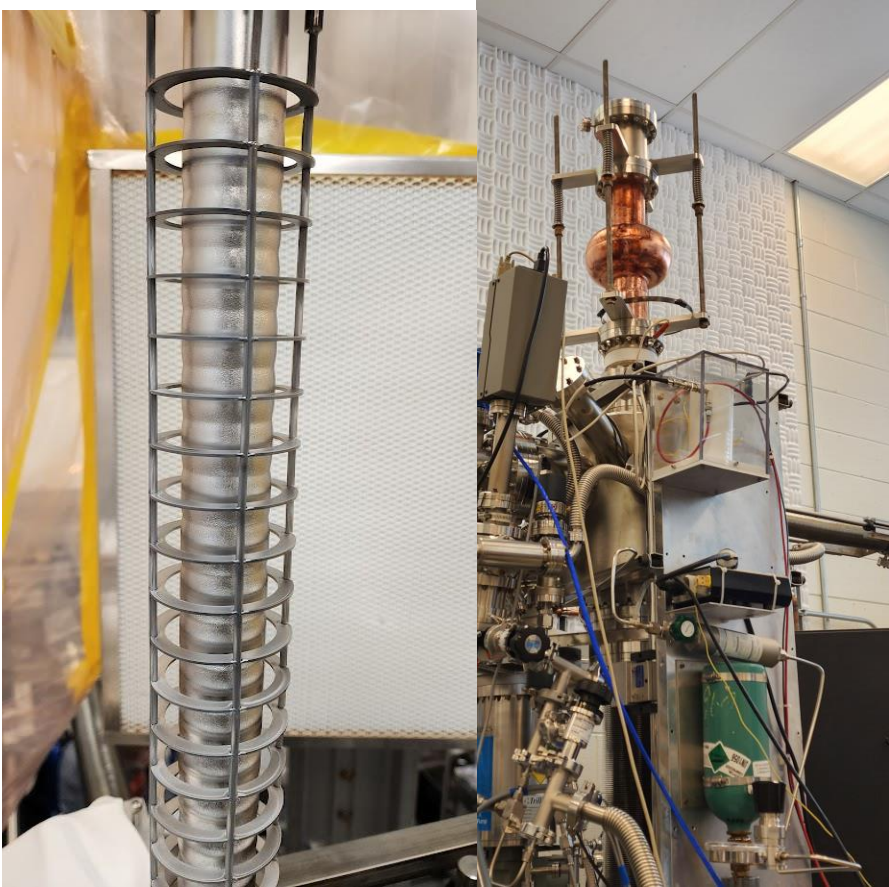
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HiPIMS Nb on Cu

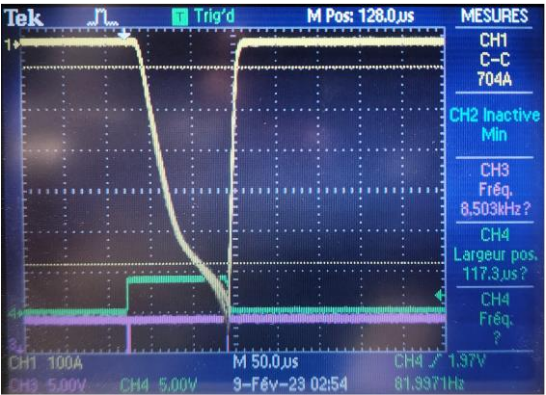
Cylindrical HiPIMS Cavity Deposition System



- Upgrade for serviceability & lower frequency cavities
Deposition ramped up to 1 cavity/week if substrate available and no outside interference (safety stand-downs, other facilities downs...)



16 magnetrons
Cavity profile
coated all at once



Surface Preparation

- SUBU 5
- 15 to 30 μm removal
- HPWR
- Drying with methanol sheeting
- Assembly in ISO-4 clean room
- Slow pump down
- Bake-out 24 h

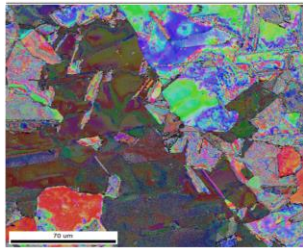
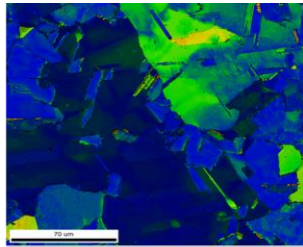
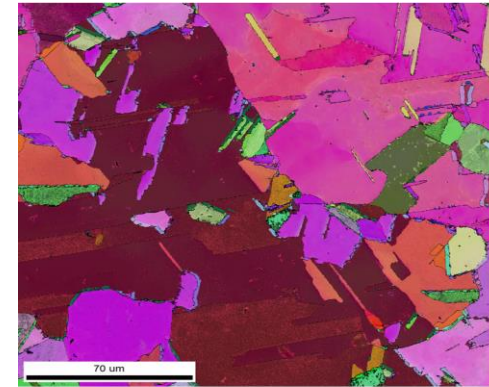
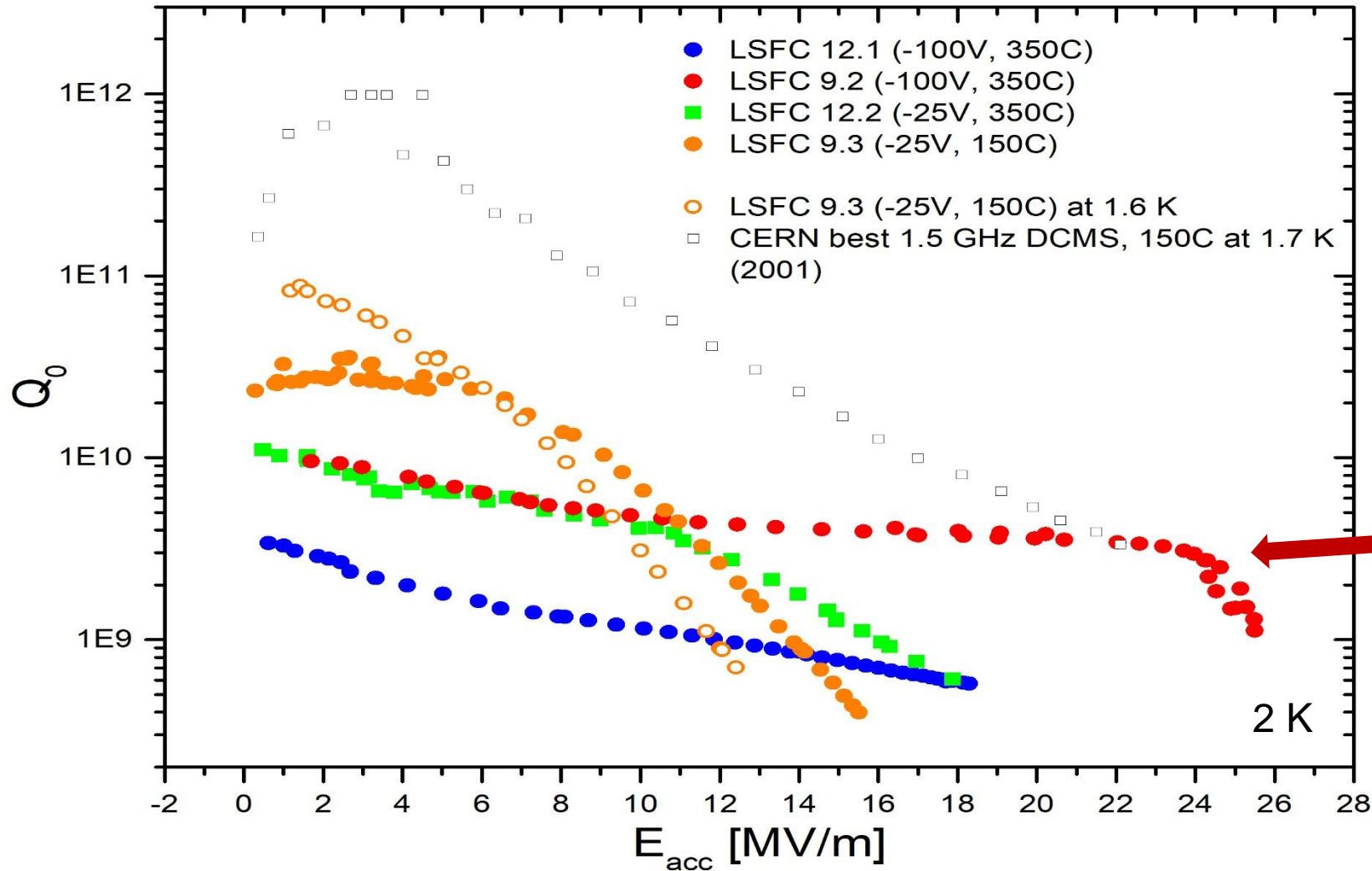
Coating

- $T_{\text{bake/coating}} = 350$ or 150 $^{\circ}\text{C}$
- Target-substrate distance = 10 cm
- [samples & equator]
- Film thickness $\sim 1\text{-}3$ μm
- $f = 83$ Hz
- Pulse width = 110 μs
- $P_{\text{Kr}} = 4.2$ mTorr
- Bias = -25 to -120 V
- Peak power = 220 kW

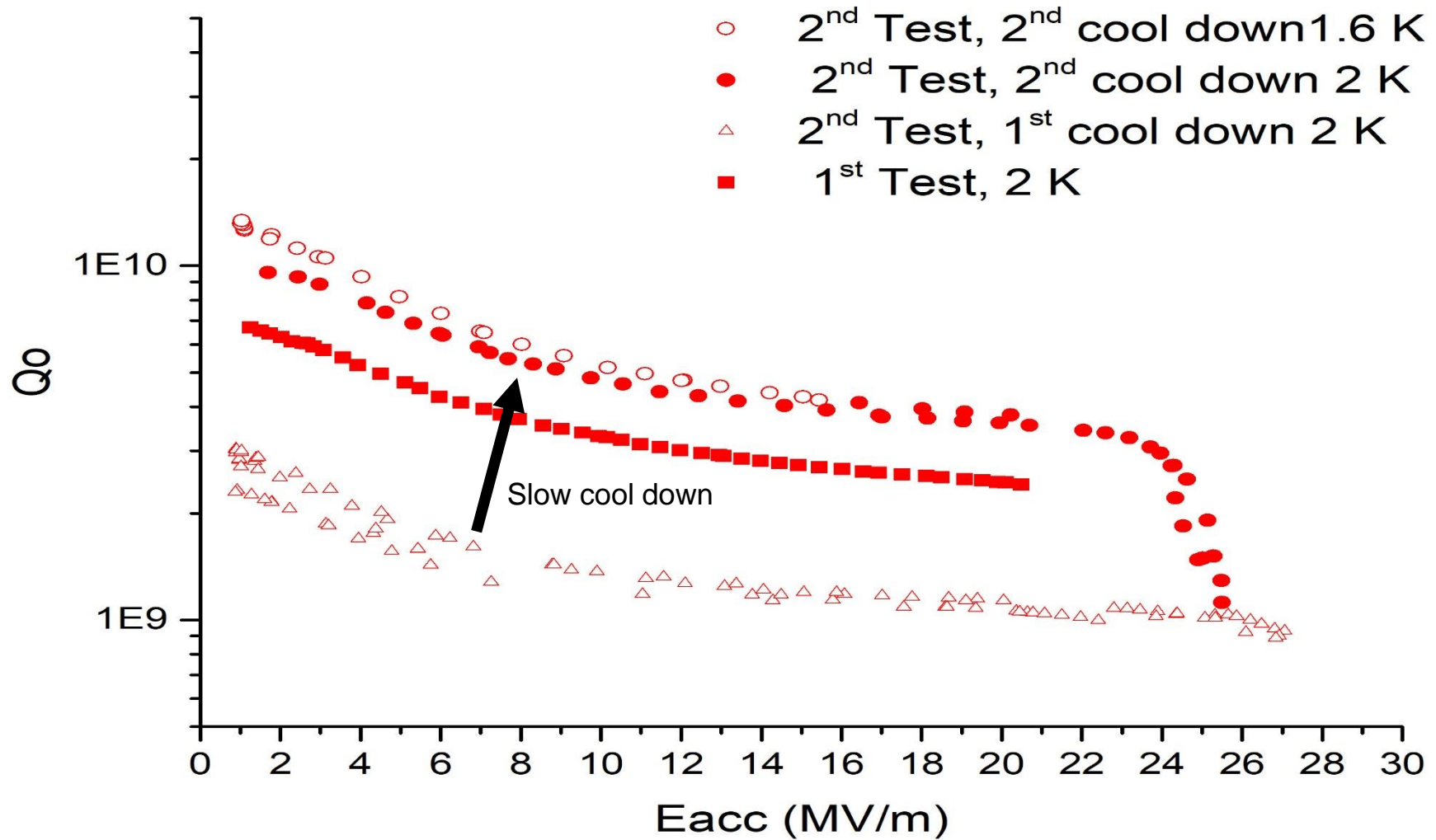
Preparation for RF test

- HPWR
- Drying in ISO-4 cleanroom
- Assembly in ISO-4 clean room
- Assembly on test stand
- Slow pump down
- Slow cool down /RF test

1.3 GHz HiPIMS Nb/Cu Cavities - Welded Substrates & SUBU

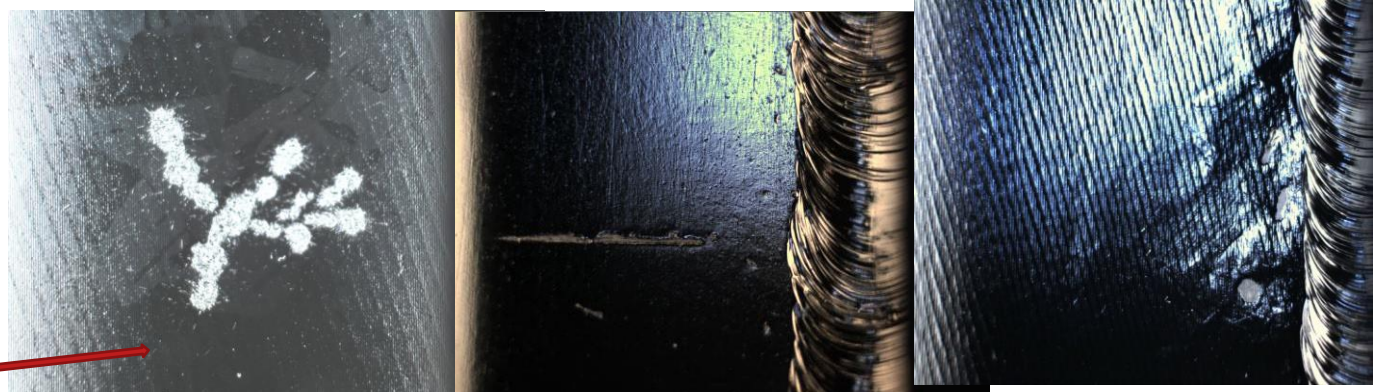
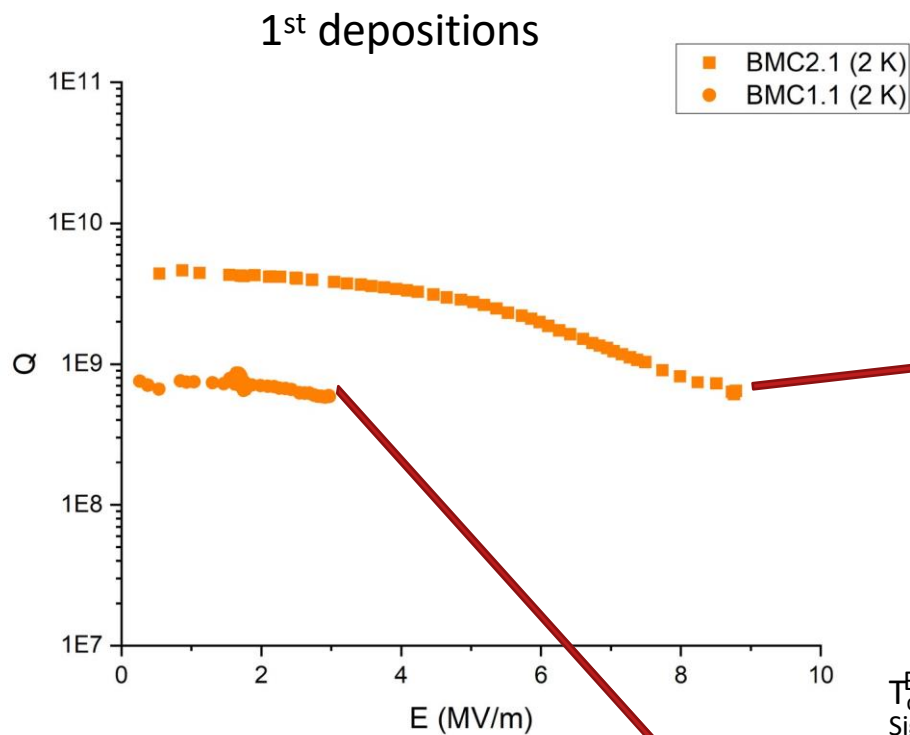


Effect of Cool-down rate

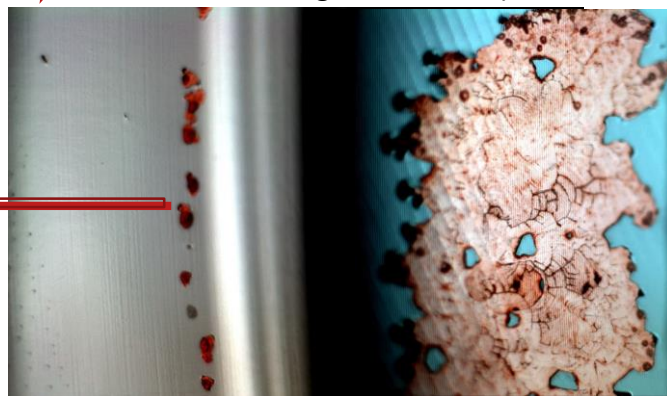
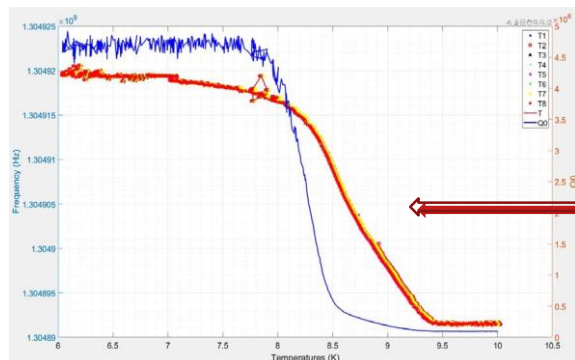
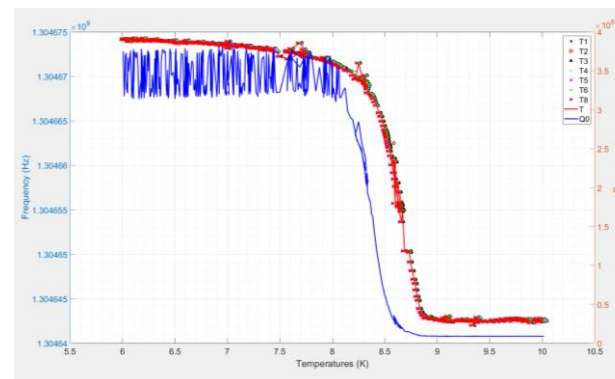


Cool down has significant influence on losses – flux pinning

HiPIMS Nb on bulk-machined Cu substrates - 1st deposition cycles



$T_c^{BMC1.1} < 9\text{ K}$
 Significant peel-offs
 throughout the cavity



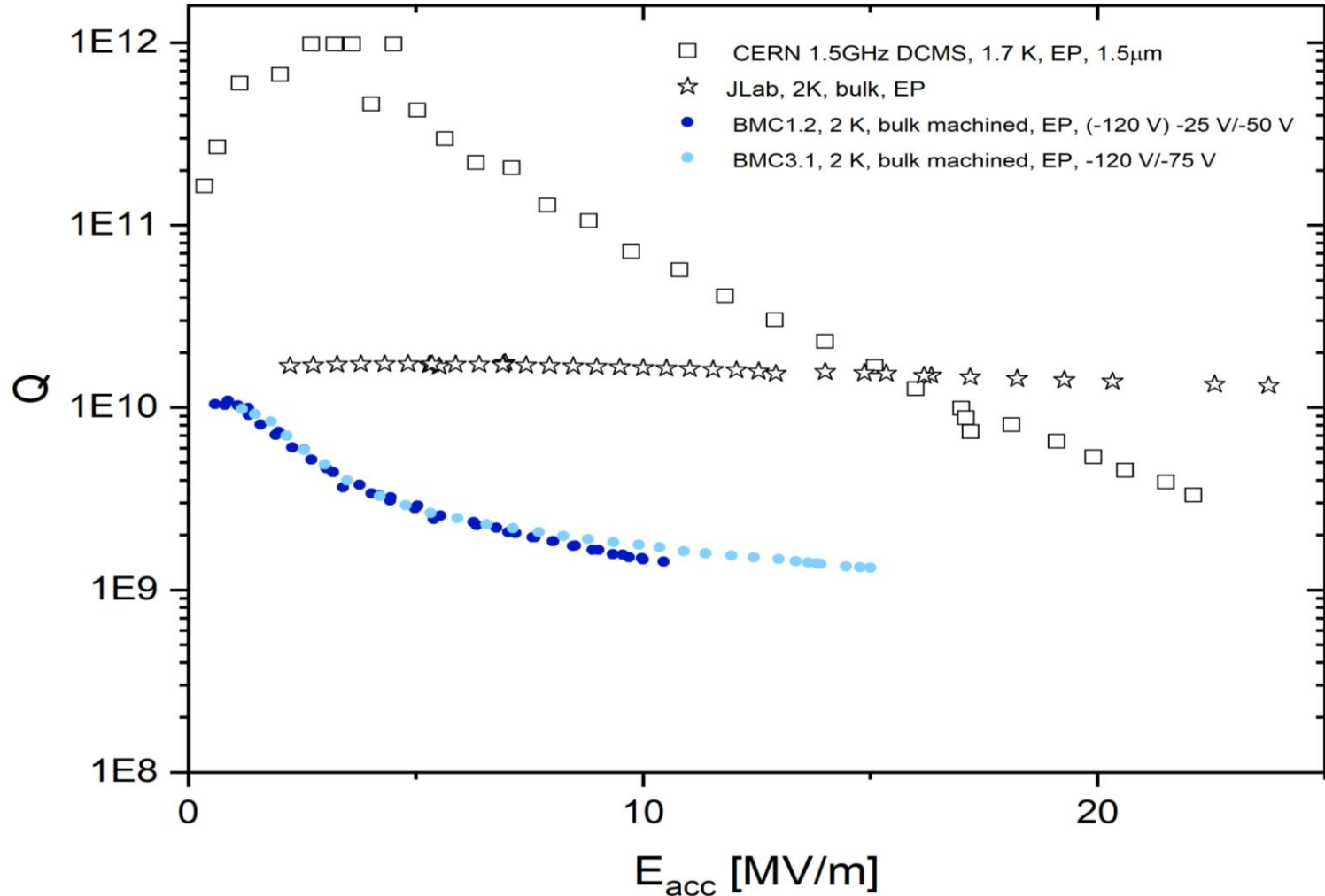
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Issues with the parameters used
 for Cu electropolishing process @
 CERN Corrected for BMC1.2 and
 BMC 2.2, BMC3&4

HiPIMS Nb/Cu on bulk-machined Cu substrates

Coating Temperature: 150 °C

Nucleation at -120 V/-25 V, subsequent growth -50 V



Very different energetics during film growth but similar (deceiving) results

Potential issues:

- Not enough material removal after diamond machining??— similar behavior seen for some depositions at CERN
- Weld defects (even after re-machining)?
- Space between cathode & grid too wide, triggering increased arcing

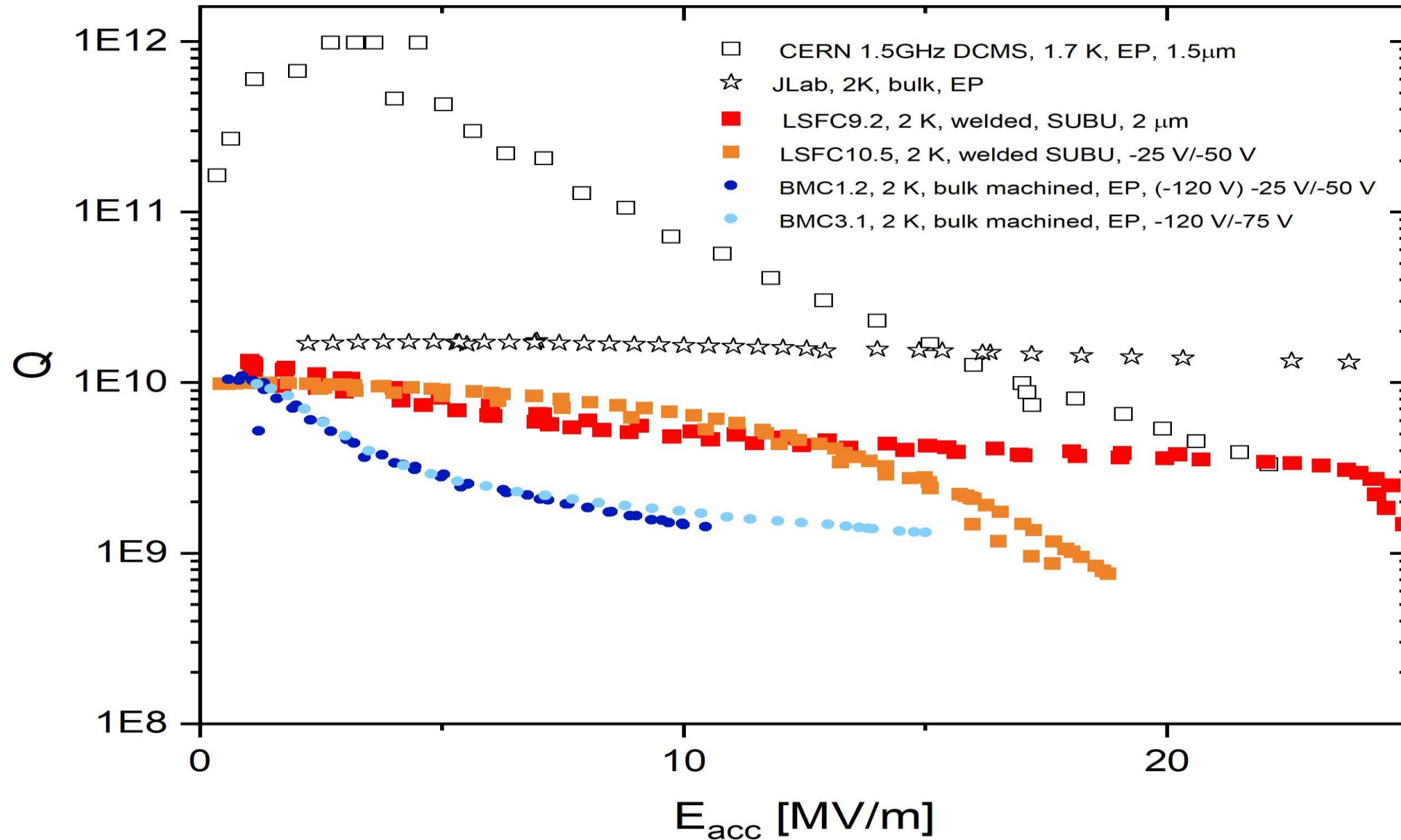


T_c yet suppressed (9.15-9.2 K) compared to nominal Nb/Cu (>9.3 K)

Found issues:

- Air leak at the level of the compressed gas regulator on Kr gas injection line

HiPIMS Nb/Cu



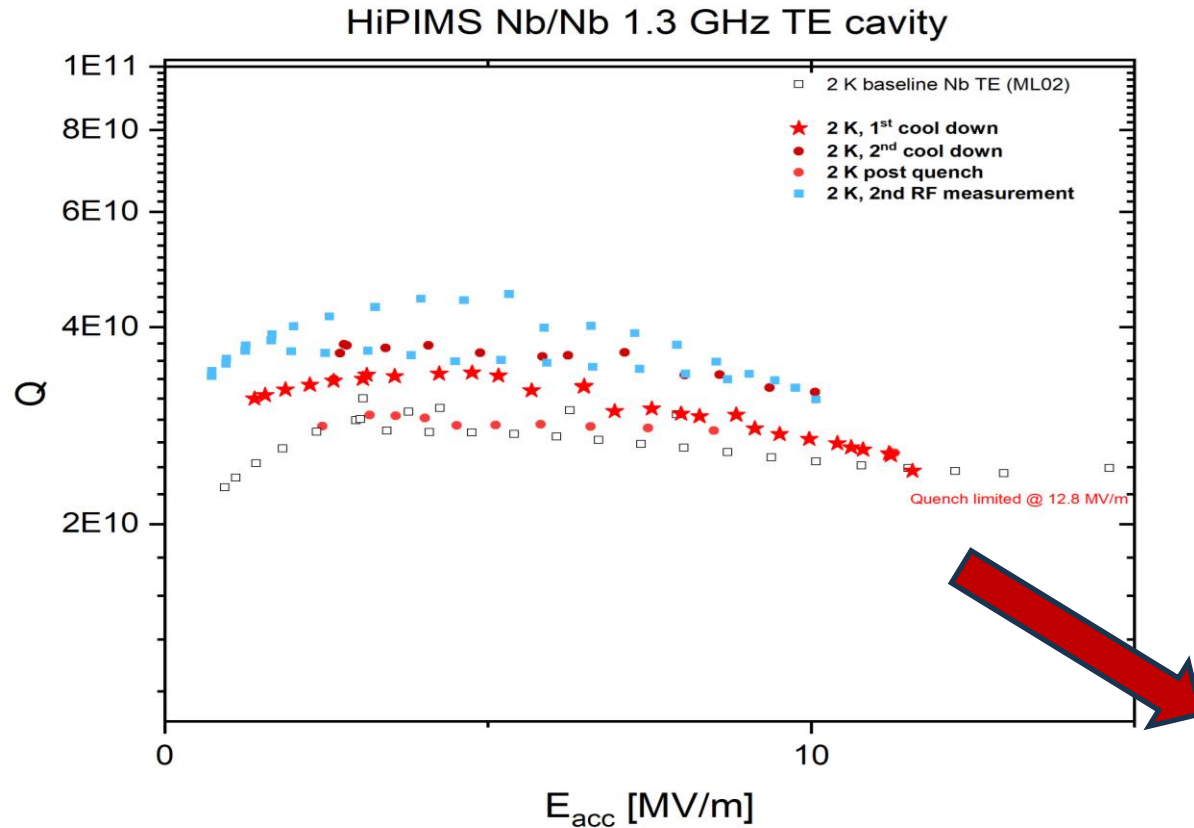
HiPIMS Nb/Cu at 1.3 GHz

HiPIMS Nb on Nb

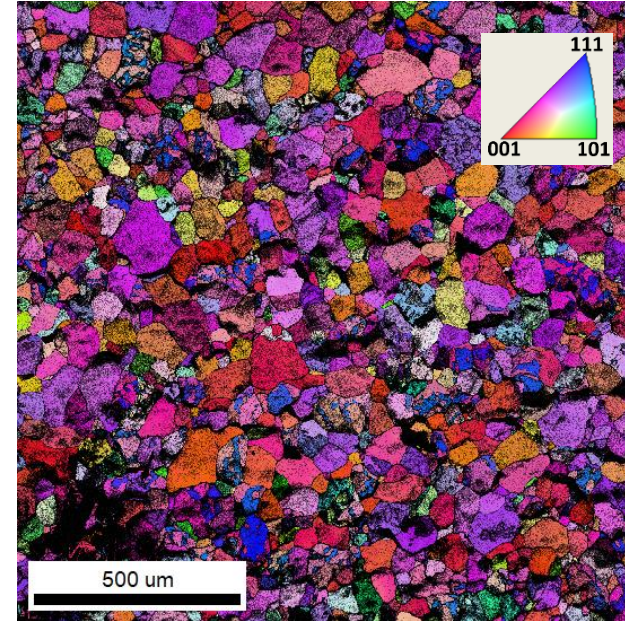
HiPIMS Nb film/Nb results

Coating Temperature: 150 °C

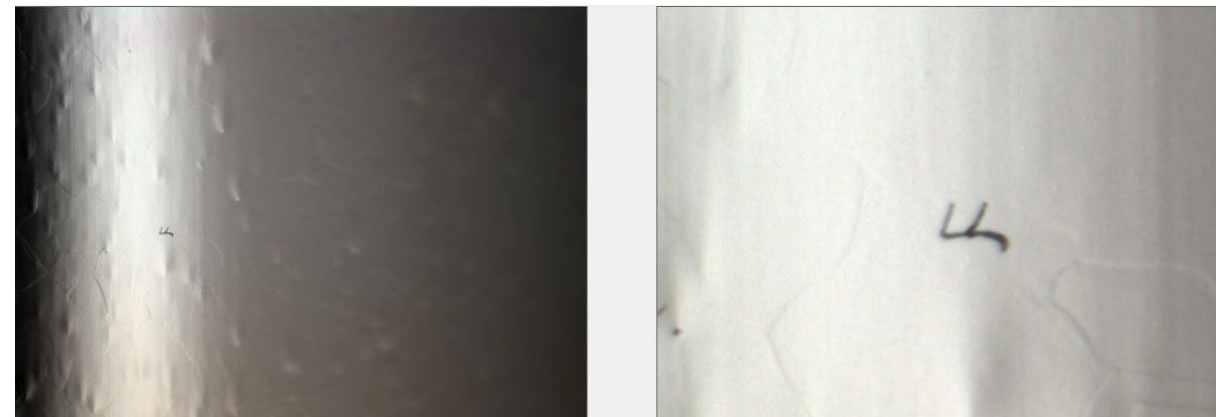
Nucleation at -25 V, subsequent growth -50 V



Several RF measurement cycles, without/with thermometry, each preceded with 100 bar HPR

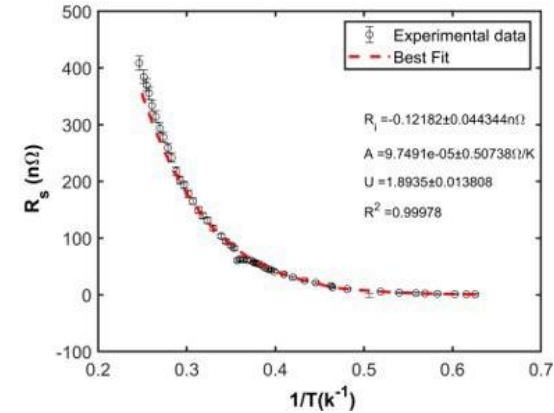
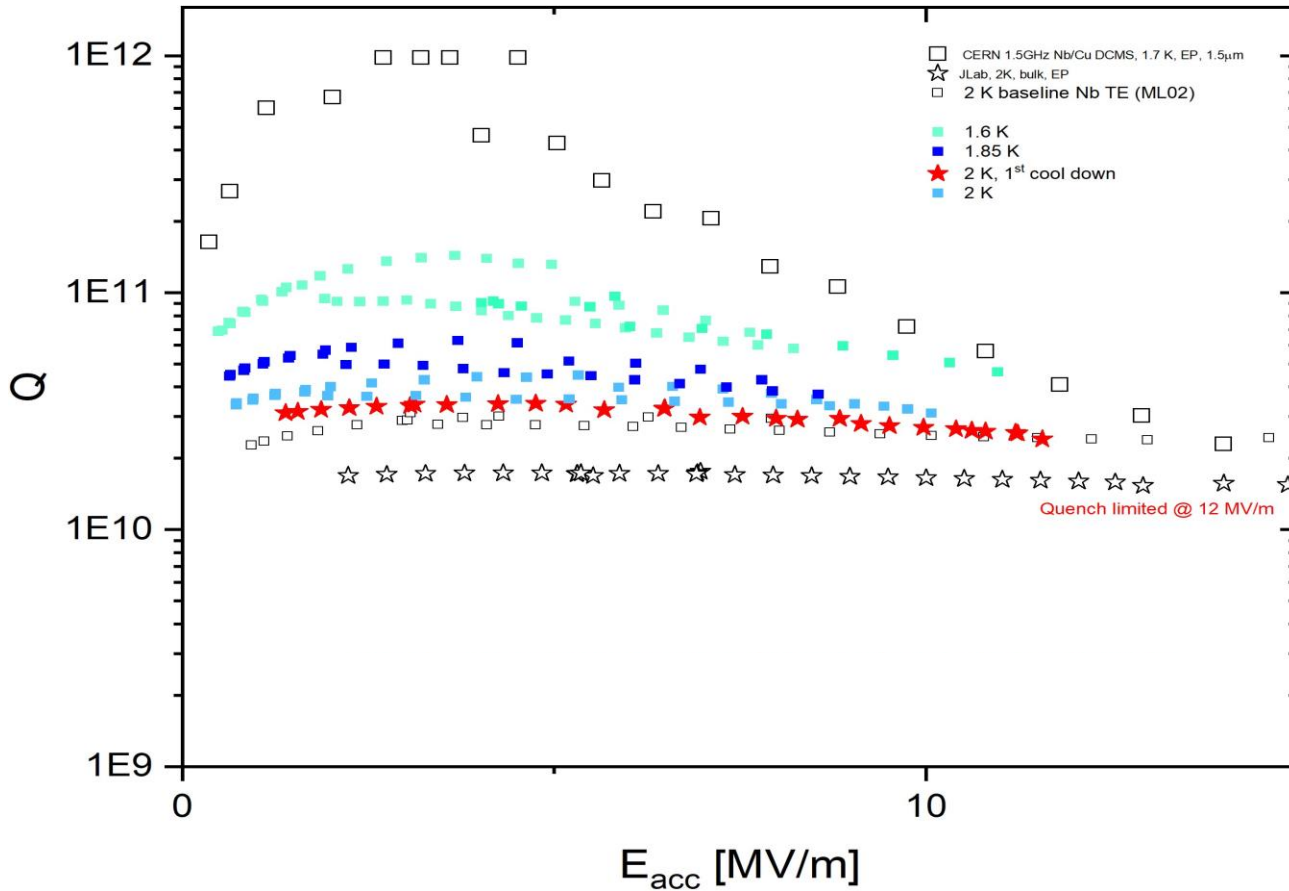


EBSD
IPF for HiPIMS Nb/Nb



Imaging at quench location

HiPIMS Nb film/Nb results



- $\Delta = 1.8935$ meV
- $R_{res} = 0.1$ n Ω
- mfp = 57.2 nm

No degradation in Q compared to bulk substrate
 Quench limitation early at 12 MV/m



No degradation in intrinsic limitation for Nb films
 Determine defect types generating performance degradation

Summary

- ✓ Mitigation of the Q-slope has been observed
- ✓ No intrinsic limitation in the Nb film itself as shown from Nb/Nb results
- ✓ Interface with substrate has critical importance. Substrate quality and preparation is paramount

➤ Investigate & solve current limitations observed in Q for Nb/Cu

Progress significantly hindered in the past 1.5year from

- Successive safety pauses Lab-wide
- He cryo-plant refurbishment ie lack of He from May '24 to now Dec'24

Verify limitation due to leak on Kr line

- Deposition on 952.6 MHz Cu cavity – Single cell undergoing CBP.
- Comparison study between ECR & HiPIMS energetics (nucleation, subsequent growth, final RF surface growth (energy threshold) and influence on intrinsic stress (minimize j_c), layer quality.
- Continue interface studies to erase the influence of the substrate