60 YEARS / ANS CERN

Nb-coated HIE-ISOLDE QWR SC Accelerating Cavities: coating process and film characterization

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Outline

- QWR coating process
  - HIE-ISOLDE upgrade
  - Production workflow
  - Coating process
  - Cavities performances
- Nb layer characterization
  - Thickness
  - RRR
  - SEM surface morphology
  - FIB-SEM cross section imaging
  - TEM composition and orientation mappings

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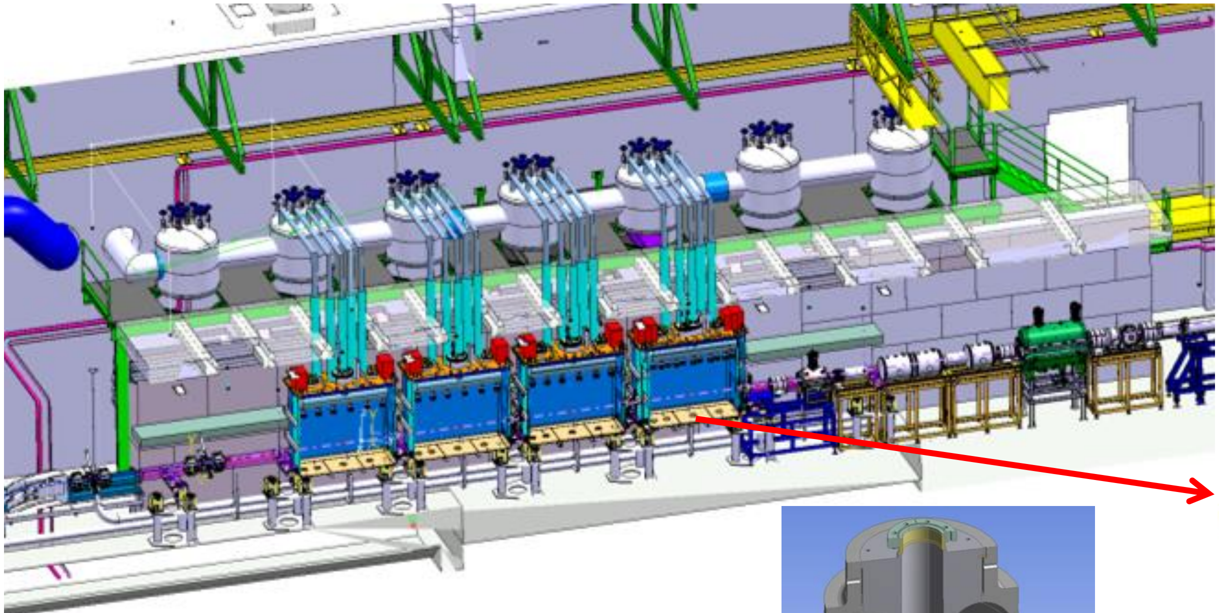
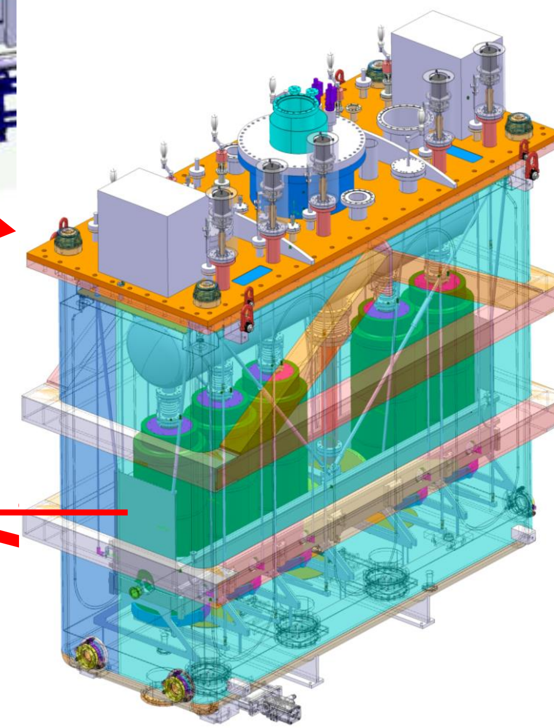
HIE-ISOLDE upgrade project

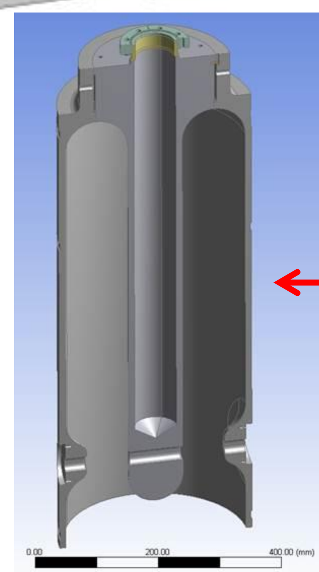
→ Boost the radioactive beam energy from 3MeV/u to 10MeV/u by using SC linac.

High Energy and Intensity – Isotope Separator On Line DEtector

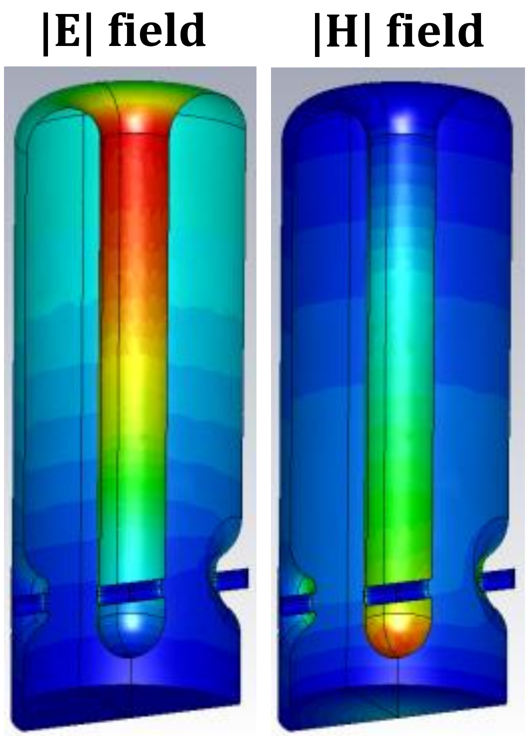




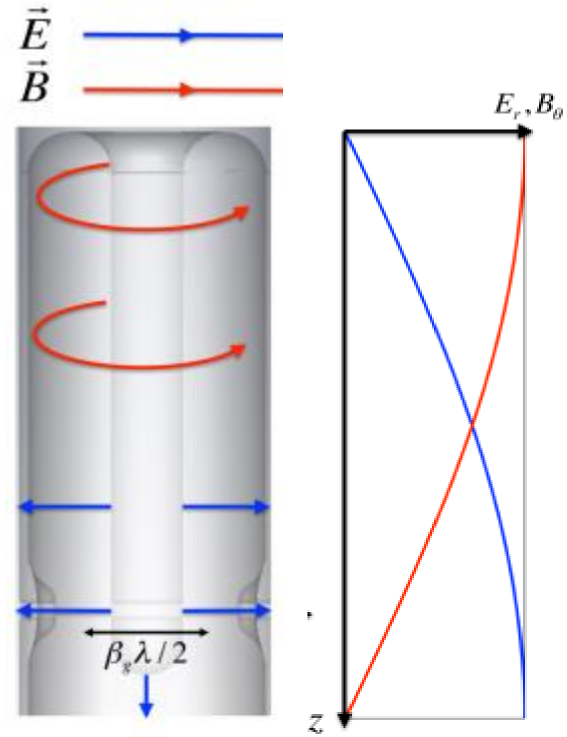
Quarter-wave resonator (QWR):
Nb thin film sputtered
on 3D forged OFE Cu substrate



High-β QWR



Frequency	**101.28 MHz**
E_{acc}	**6 MV/m**
$\beta_{optimum}$	**10.9%**
R/Q	553 Ω
E_{peak}/E_{acc}	5.0
B_{peak}/E_{acc}	95.6 G/(MV/m)
$G=R_sQ$	30.7 Ω
U/E_{acc}^2	0.207 J/(MV/m)^2
P_c at 6MV/m	**10W**



Courtesy M. Fraser

→ Total of 20 cavities to be produced

→ 5 cavities ready for assembly in first cryomodule by the end of 2014

Production workflow

1 month per cavity

Cavity reception

↓

Frequency tuning

↓

Surface treatment

↓

Niobium coating

↓

Cryostat preparation

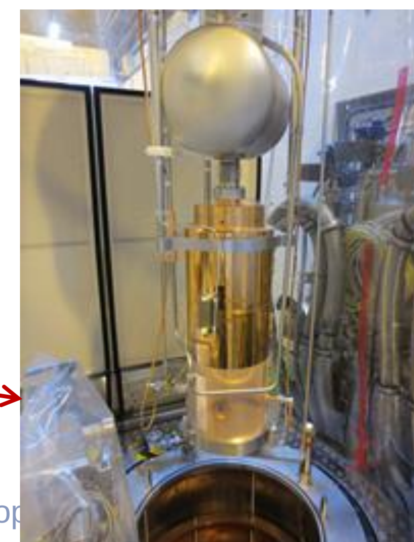
↓

RF cold test

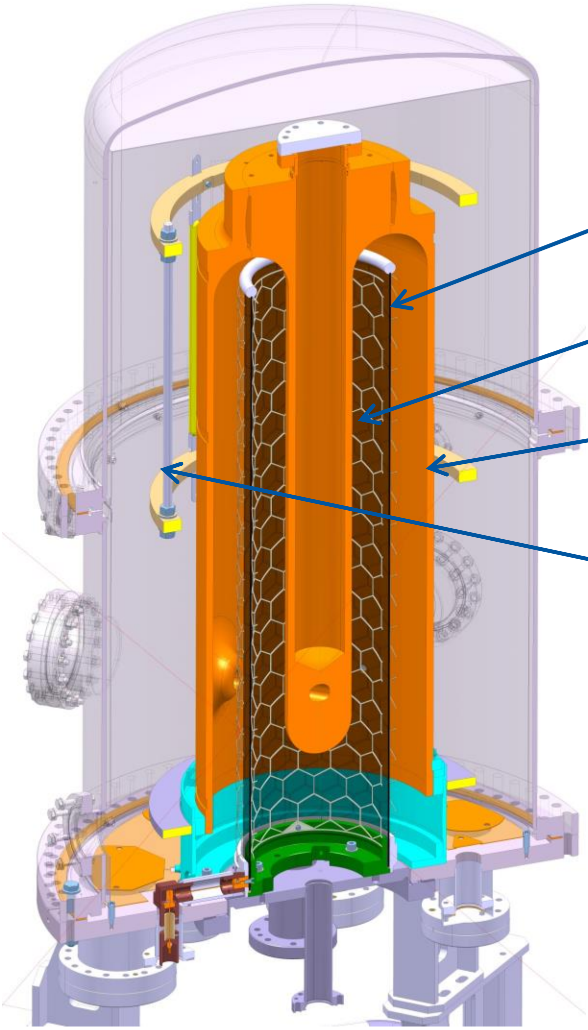
↓

Cavity storage

Niobium Stripping n+1 cycle

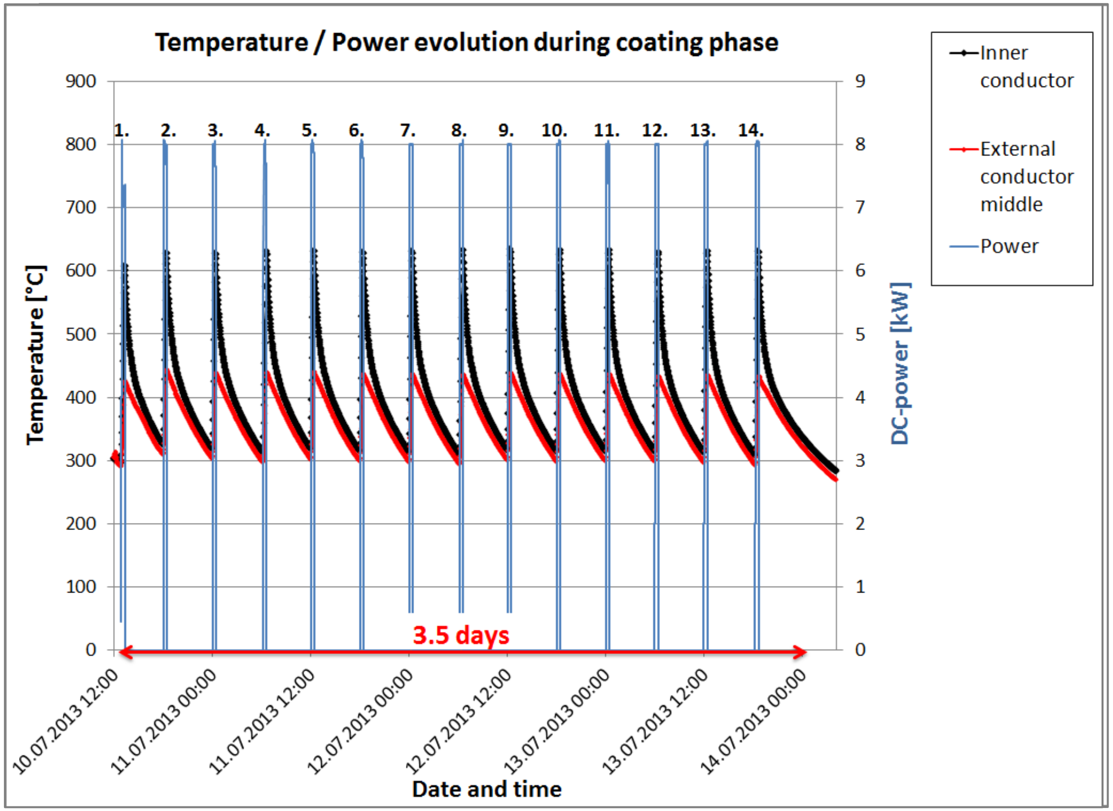


Coating hardware



- Nb cylindrical cathode at -1000V
- Grids grounded for plasma polarization
- Adjustable cavity bias: ions densify & smooth the Nb layer
- Coating with hot substrate (300-620°C) bakeout with IR lamp prior to coating
- Thermocouples along cavity to monitor temperature and RGA monitoring
- Viton O-ring or Cu-gasket
- 2 coating benches functional at CERN

Coating process



Baseline recipe DC-bias diode:

Pressure: 0.2mbar

Sputtering gas: Ar

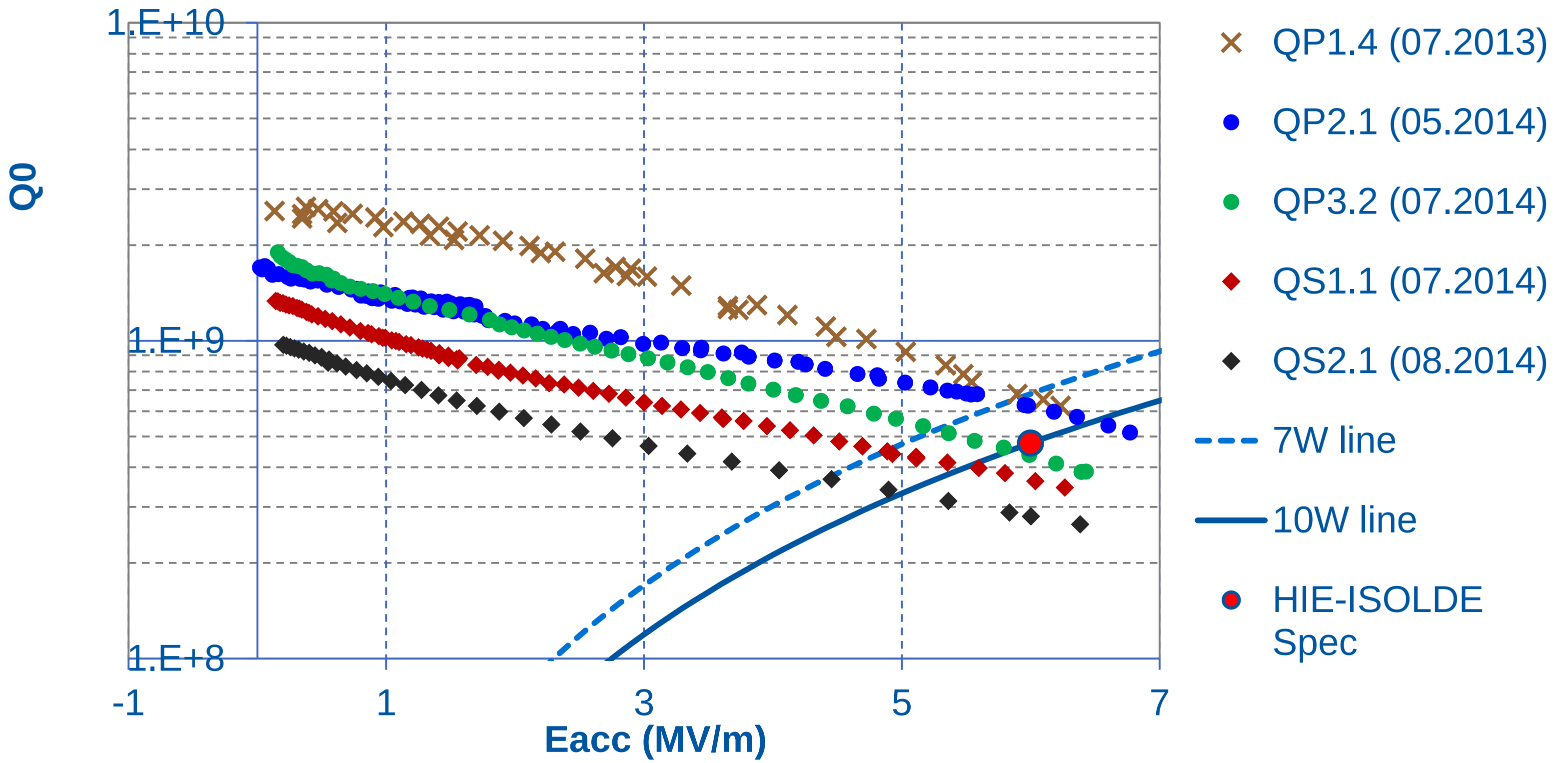
Nb-cathode power: 8kW

Cavity bias: -80V

14 runs: 25' coating + 5h35' cool down to 300°C each, total coating time = 6h

Temperature:
Inner: from 315°C to 620°C
Outer: from 300°C to 430°C

Cavities performances



Eacc=6MV/m	HIE-ISOLDE specifications	QP1.4 prototype 1	QP2.1 prototype 2	QP3.2 prototype 3	QS1.1 pre-serie	QS2.1 pre-serie
Q0	4.7E+08	6.51E+08	6.23E+08	4.37E+08	3.61E+08	2.80E+08
Pcav(W)	10	7.5	7.6	10.8	12.5	17.0

for cryomodule, avg = 12 W

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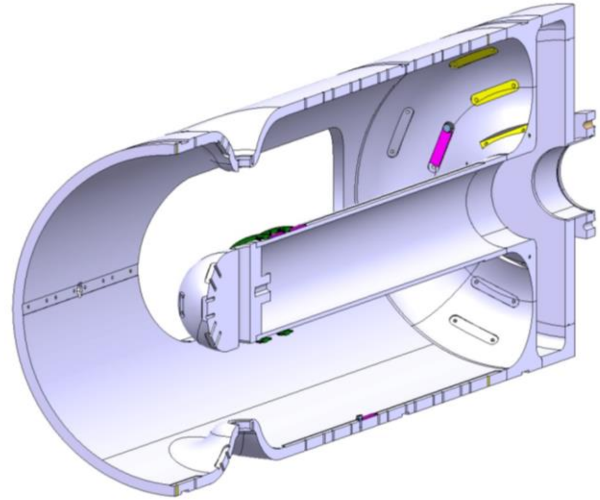
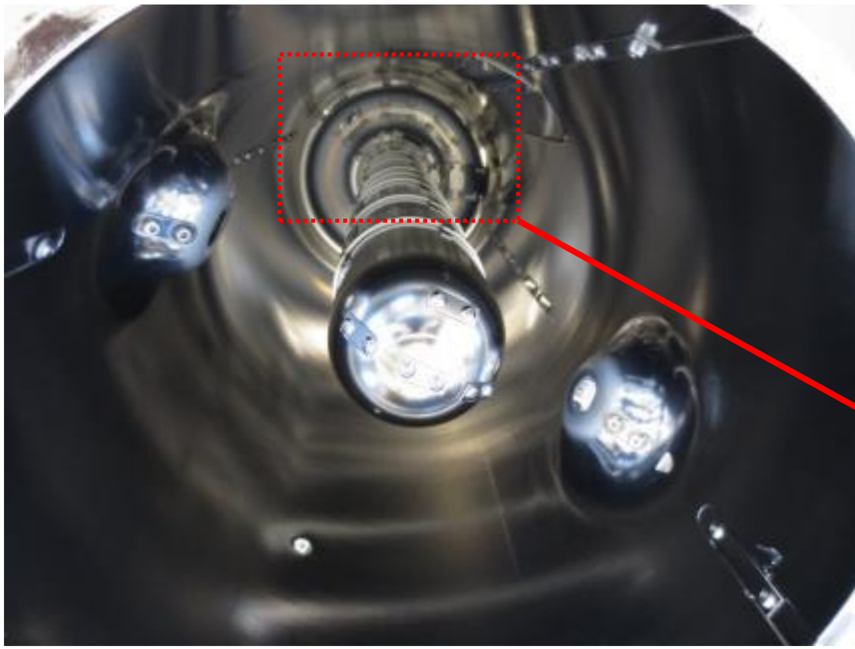
Test cavity Q4

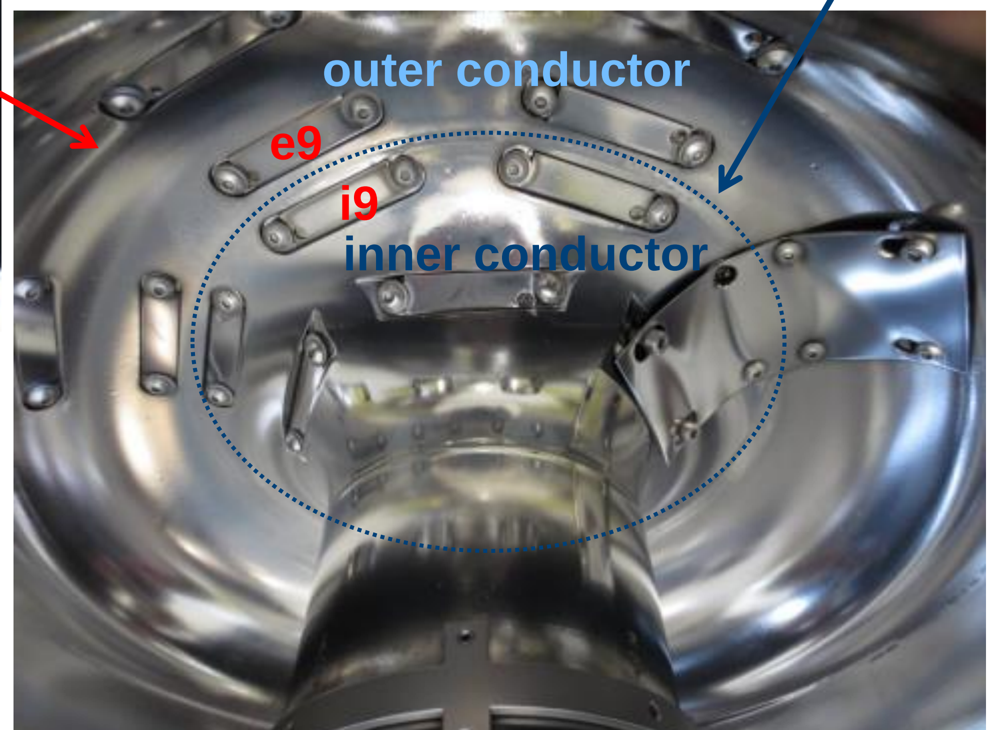




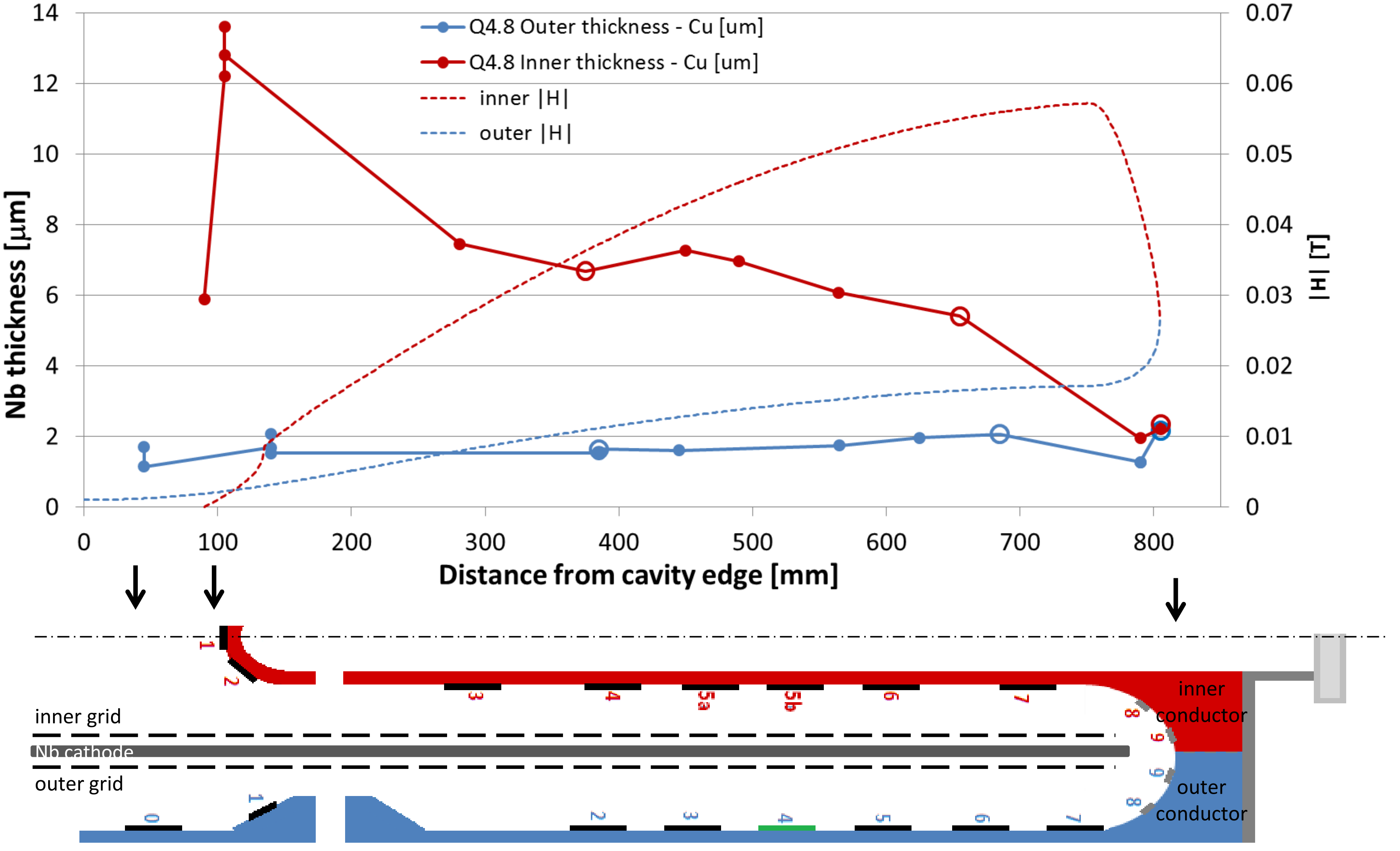
Baseline coated cavity

cavity top zoom with samples

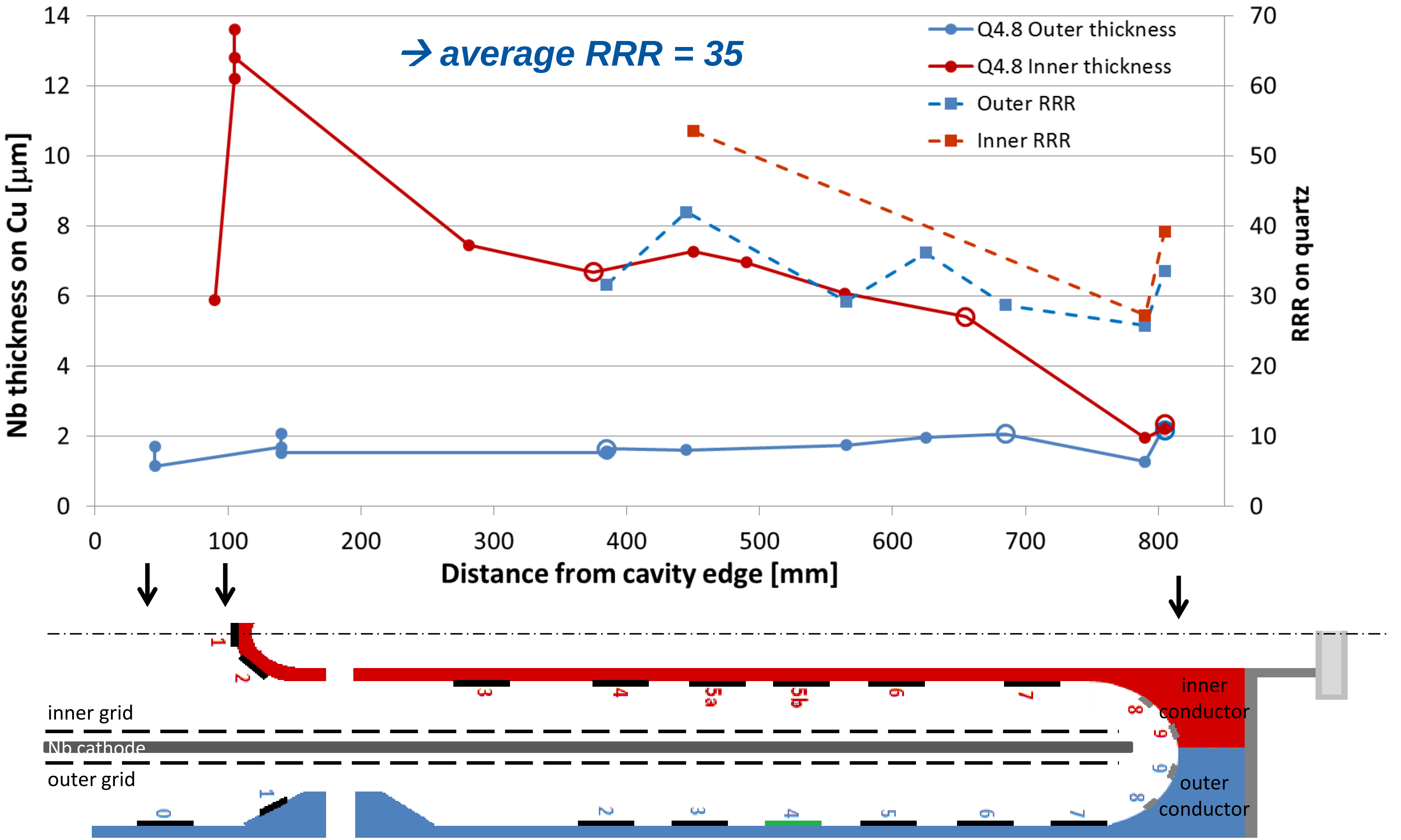
welding



Film thickness (XRF) and |H| profile

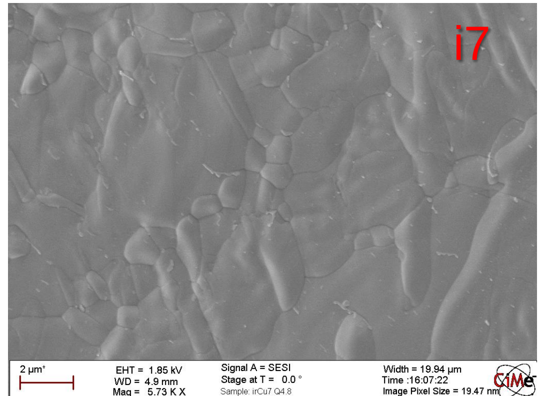


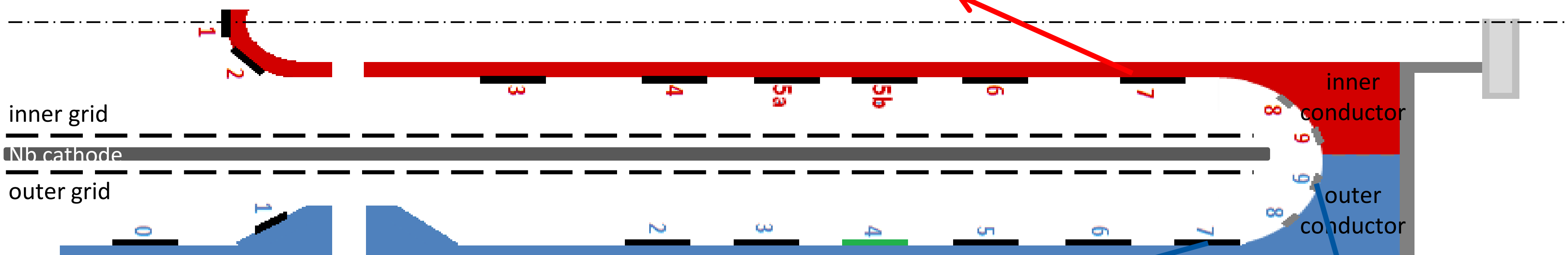
Film thickness (XRF) and RRR profile



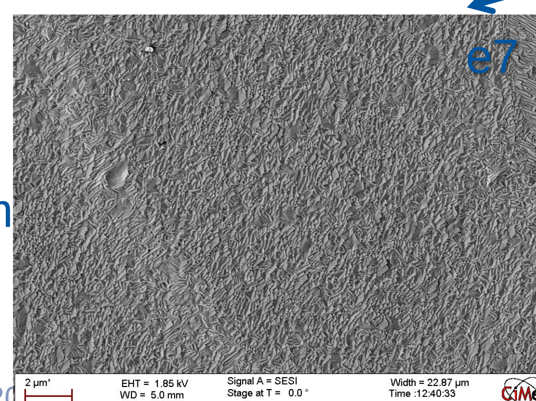
FIB-SEM surface morphology (SESI)

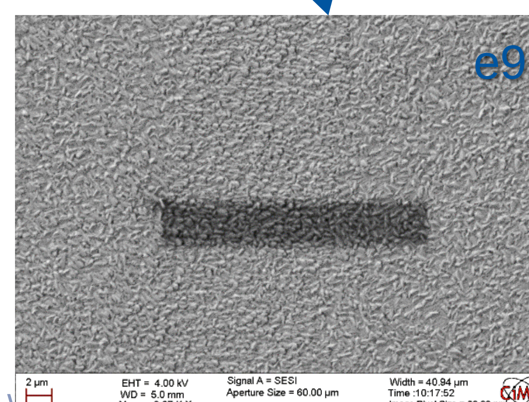
Inner conductor:
→ flat grains
→ apparent grain boundaries
→ grain size ~ 1 to few μm



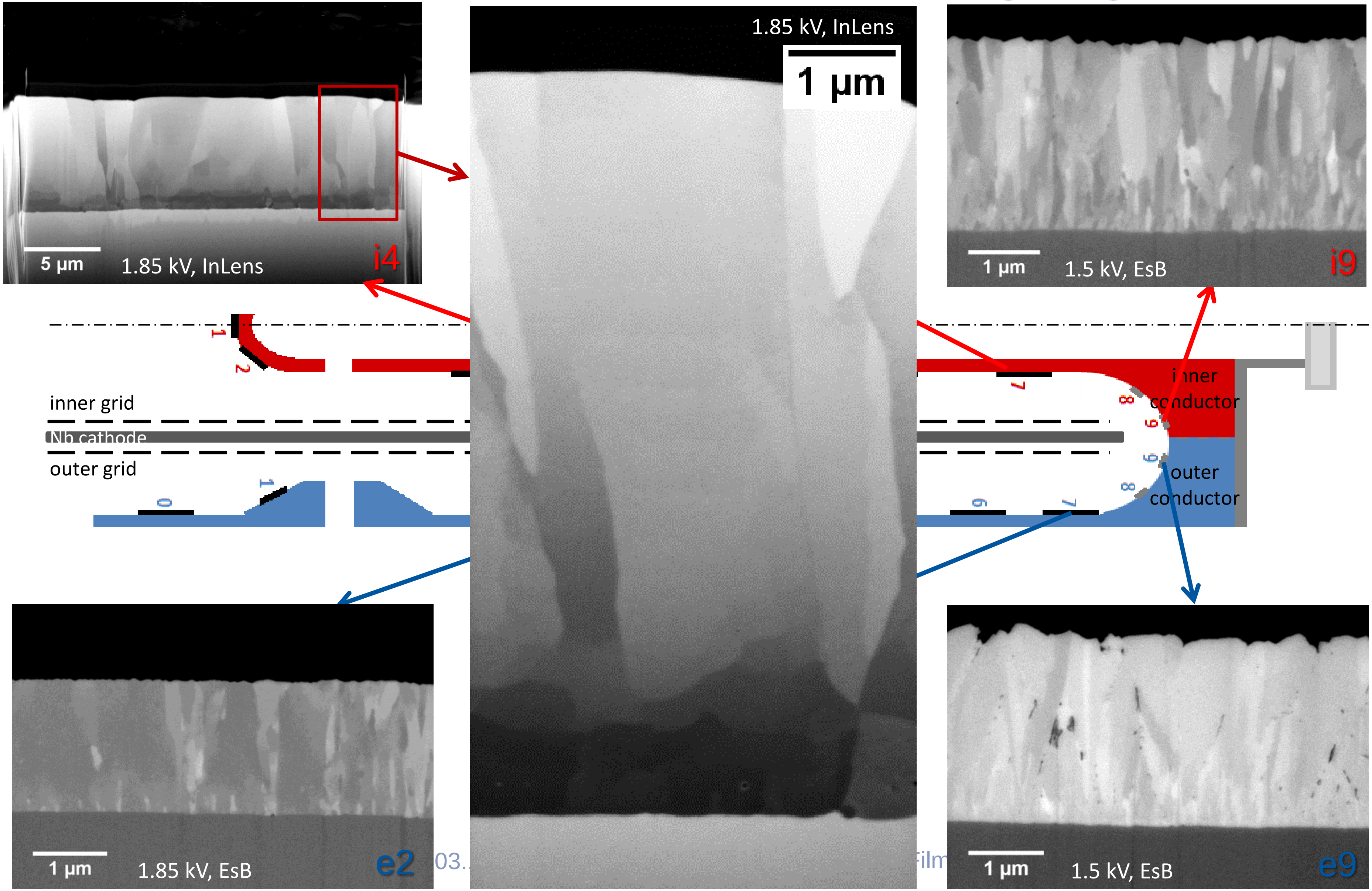


Outer conductor:
→ very fine plate-like structure
→ grain size ~ 100 to few 100nm

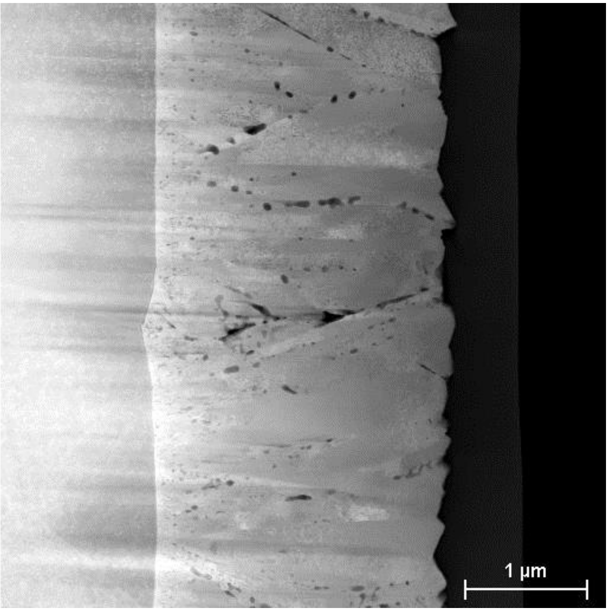


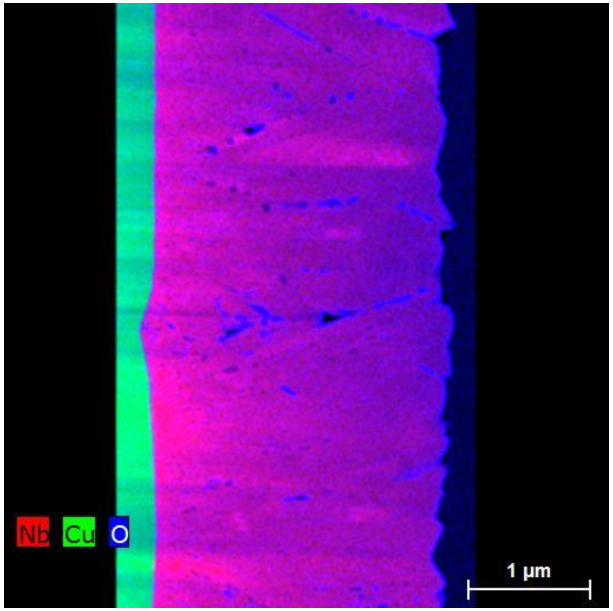


FIB-SEM cross section imaging



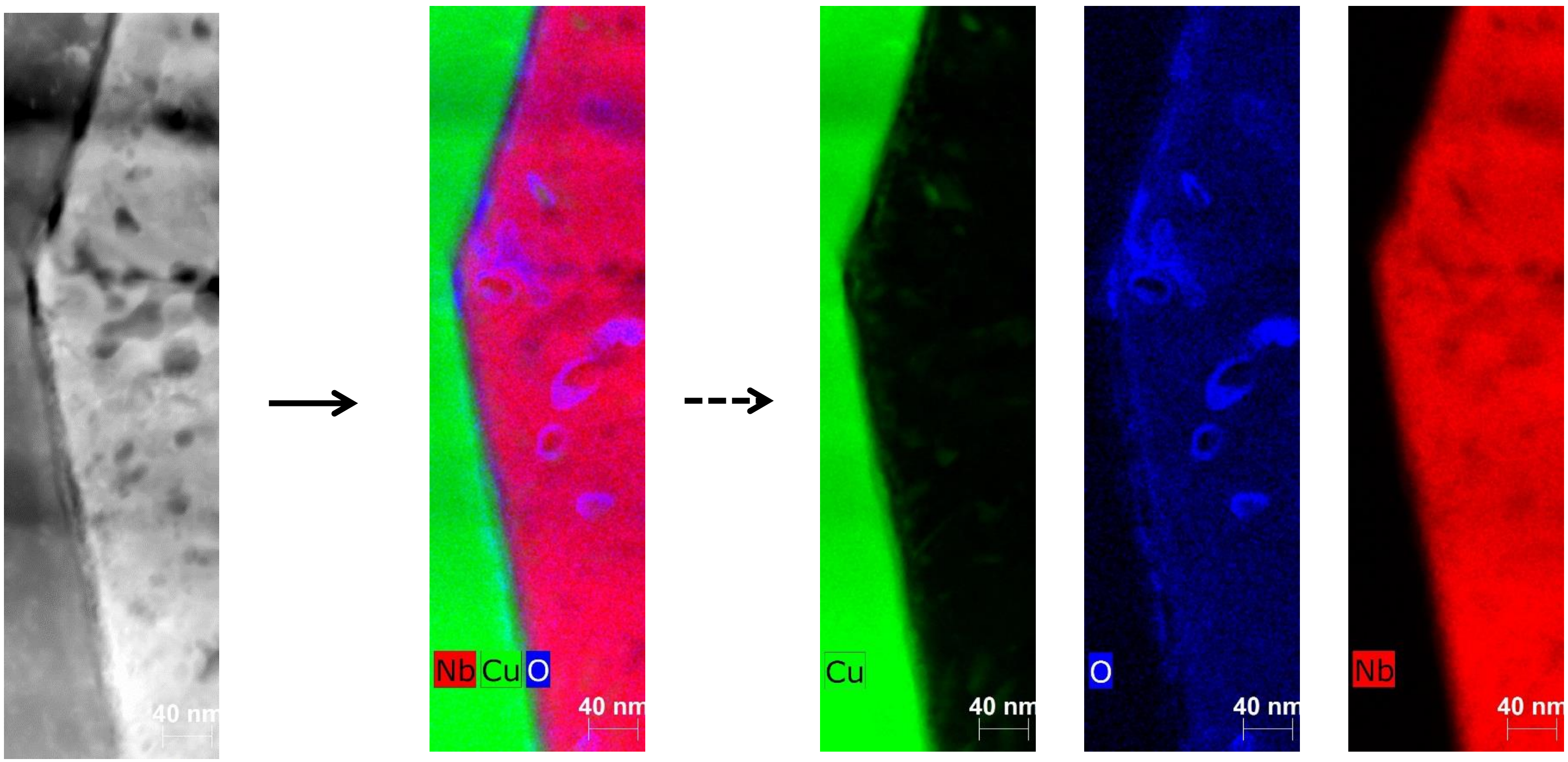
e9 TEM imaging and EDS analysis





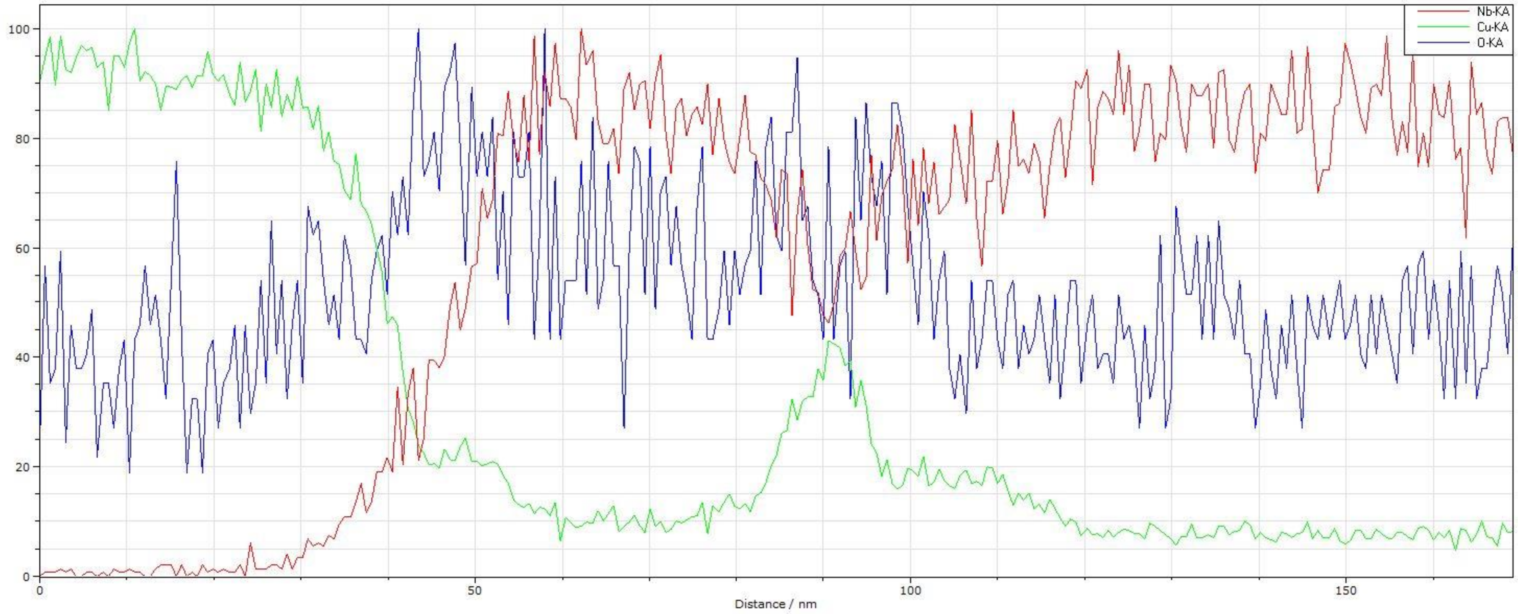
- HAADF STEM image with corresponding mapping of the Nb Coating.
- Oxygen layer at the top as well as around pores was revealed.

e9 EDS at Cu/Nb interface



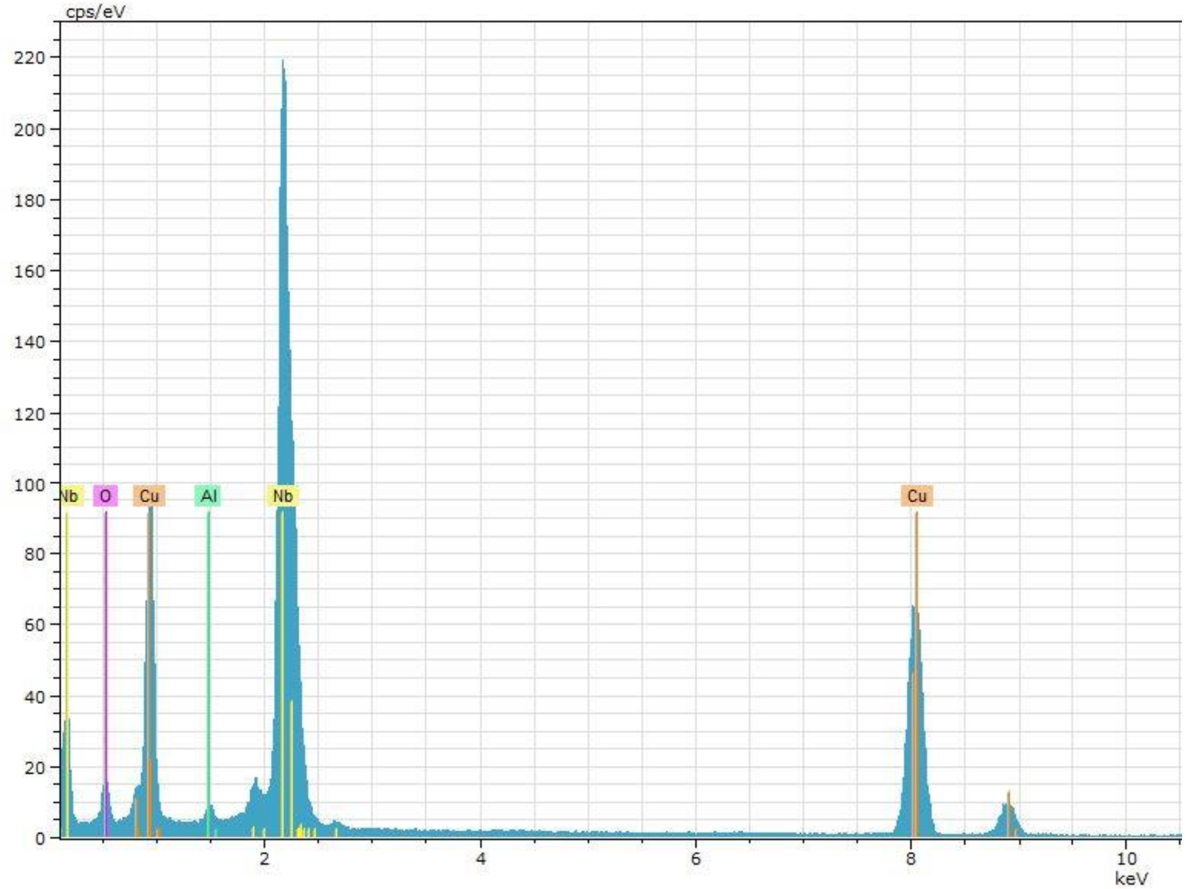
- Detailed mapping at the interface revealed presence of max 20 nm sized Cu precipitates.
- The precipitates are randomly scattered along the Cu/Nb interface and were found up to 200 nm far from the interface.
- Oxygen enrichment at the interface and around the porosity is detected.

Line scan along the interface



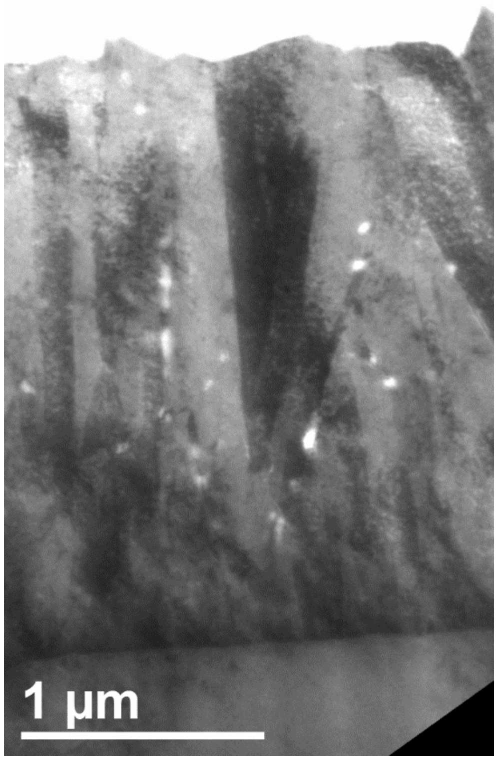
- Line scan shows the O enrichment at the interface and around the precipitate.
- Presence of Cu precipitate is confirmed

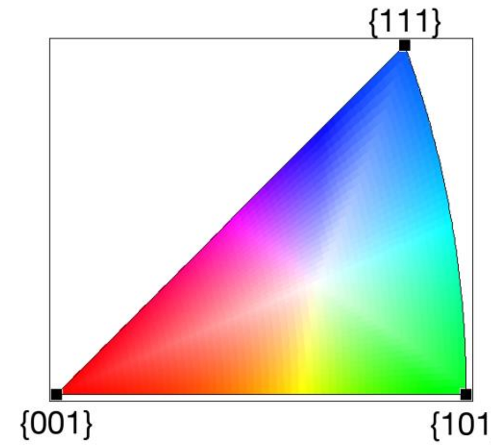
Precipitate EDS spectra



- The spectra was taken from the area marked with the red circle, the composition corresponds to Nb70Cu28Al2 in at.%. The peak of Al comes from TEM holder.

e9 Orientation mapping





- Technique based on collection of precession electron diffraction patterns and cross-correlation with the simulated template.
- Grains in the coating show no preferential orientation
- The very small grains close to the interface cannot be index because of grain overlap.
- For the grain size characterization plain view sample at well defined height of the coating is needed.
- XRD data measurement are needed for comparison of results.

Conclusions

- Workflow and reproducible coating baseline recipe are operational to achieve production of 1 cavity/month
- Production cavities RF performances good on average, need two more cavities in specification by the end of the year for the first cryomodule

- Nb layer of 2-10μm from outer to inner conductor with average RRR = 35
- Grain size ranging from 200nm on outer conductor up to few μm on inner conductor
- Large porosities at e9 position beside the weld, only very few nm-size elsewhere
- Presence of O-enrichment close to the interface and porosity
- Cu precipitates identified by TEM EDS measurements close to the interface
- FIB-SEM and TEM analysis are very powerful tools to characterize sputtered Nb-layer morphology and composition

Perspectives

- Further TEM investigation of top part of the Nb-layer will follow
- TEM analysis of thick layer to investigate the nature of observed layers
- In parallel PCT measurements
- and mu-SR measurements are on the way on same samples

→ This systematic characterization of the film efficiently highlight the regions to be improved and help to target the hardware/process parameters to adjust (bias voltage to densify the film, cathode geometry, etc.)

→ Combined together these material science and SC/RF approaches will help to tune the key parameters to achieve the best performances of Nb-coated SRF cavities

→ Optimization of Nb-film to match best RF properties

Thank you for your attention

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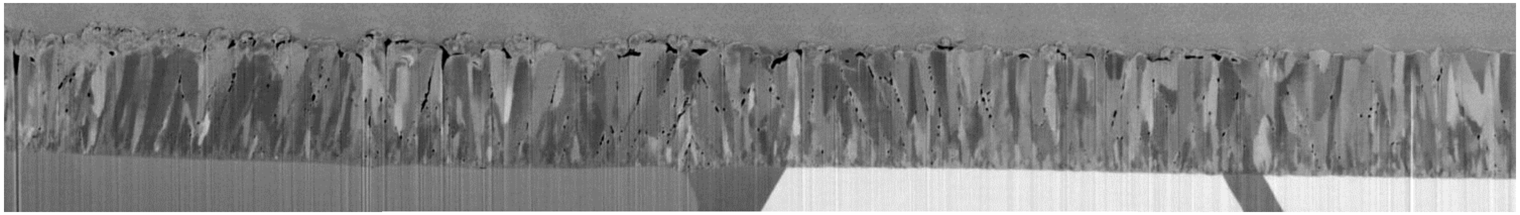
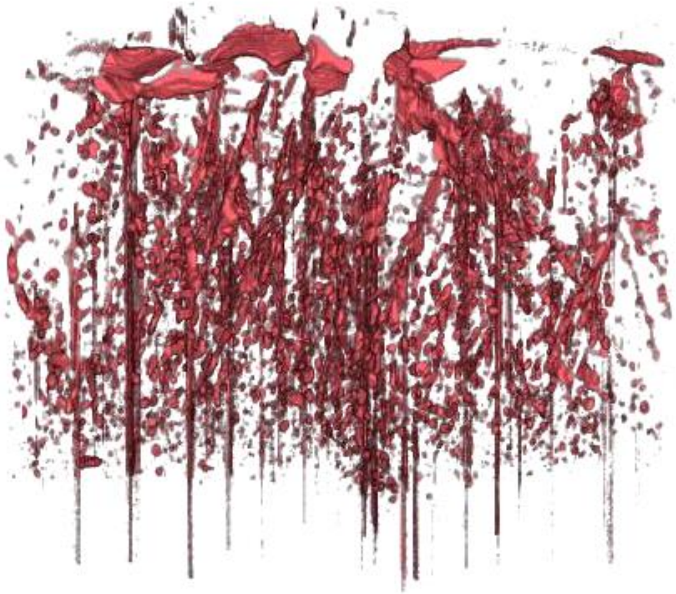
Brian Aebersold, Duncan Alexander and Marco Cantoni, EPFL/CIME

e9 FIB tomography

SESI detector 1.85 kV, M. Cantoni and B. Bartova



Pore reconstruction in a section (pixel size) of Nb coating



→ About 1% pore volume fraction

Unexpected presence of pores motivate a FIB tomography in order to visualize distribution of the pores and quantify their volume.
Accelerating voltage during acquisition was increased to 1.85 kV to improve quality of the images.
3D ATLAS software was used during the **16 hours experiment and 788 images** was acquired.