Electrodeposition of copper applied to the manufacture of seamless SRF cavities and other accelerator components

Lucia Lain Amador (CERN)

Sergio Calatroni, Paolo Chiggiato, Leonel M. A. Ferreira, D. Fonnesu, Guillaume Rosaz, Mauro Taborelli

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Outline

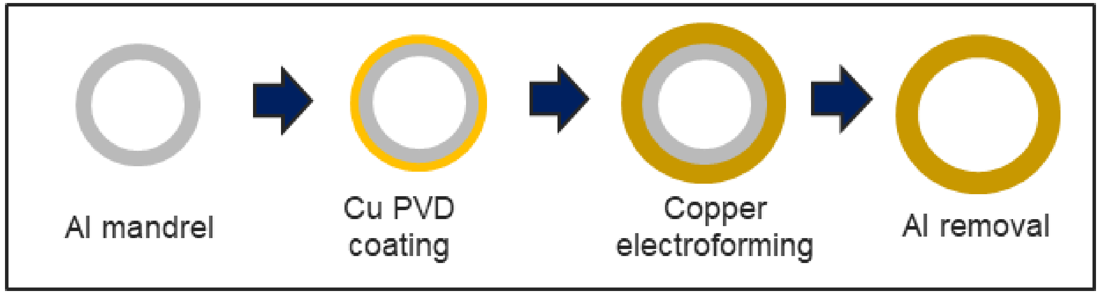
1. Electroforming process and copper properties

2. Electrodeposition of copper applied to the manufacture of seamless SRF cavities

3. Reverse thin film coatings for SRF cavities

4. Development of thin-walled copper electroformed vacuum chambers for undulators

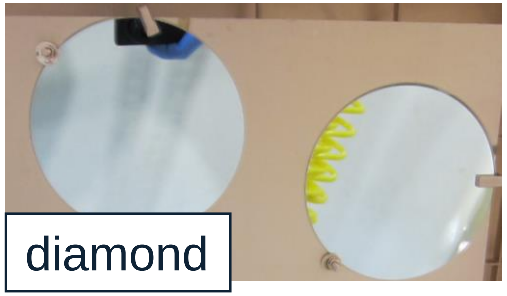
Electroforming process

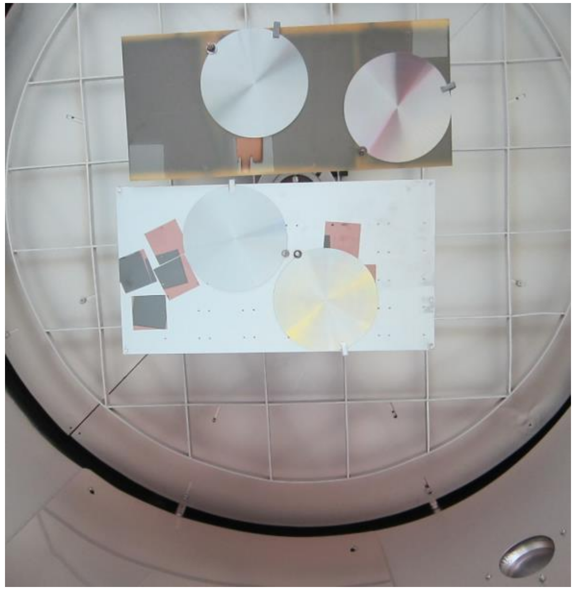


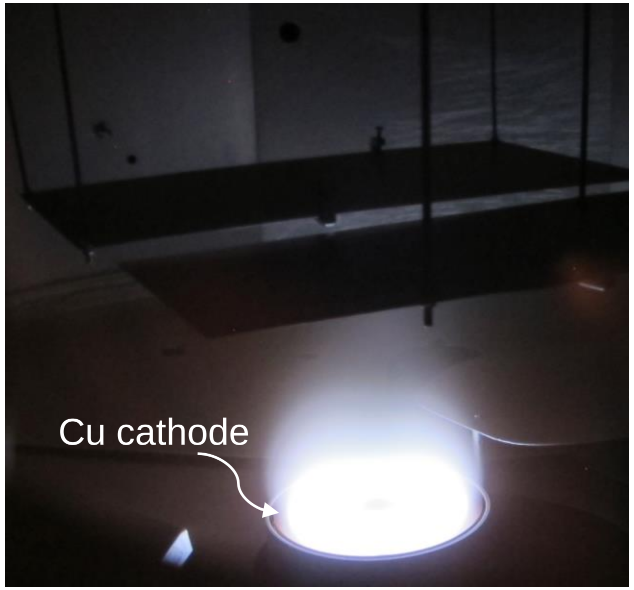
Process: copper electroforming around a sacrificial aluminium mandrel which is pre-coated with a copper thin film.

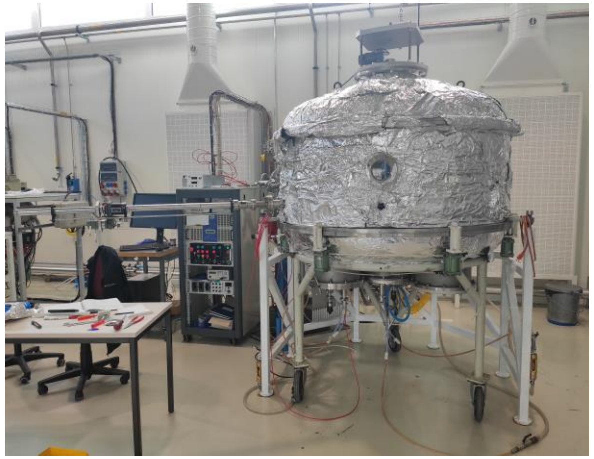
Electroformed copper properties on flat samples.

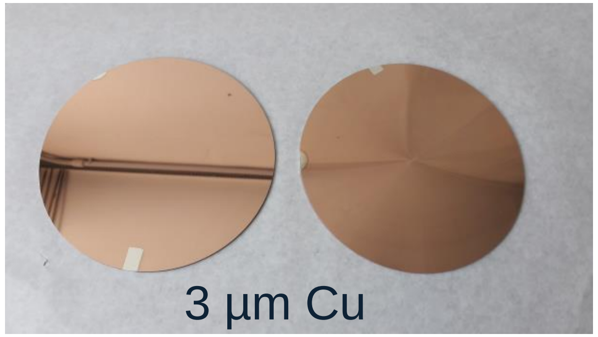
Cu PVD coating











Cu coating by plannar magnetron sputtering

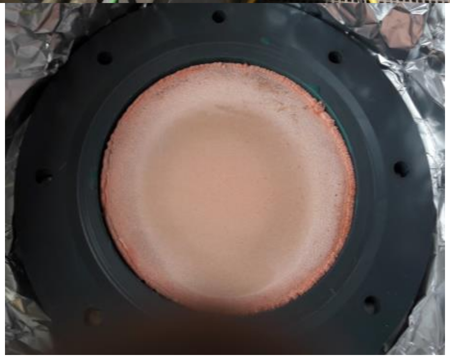
Electroforming process

Cu electroforming

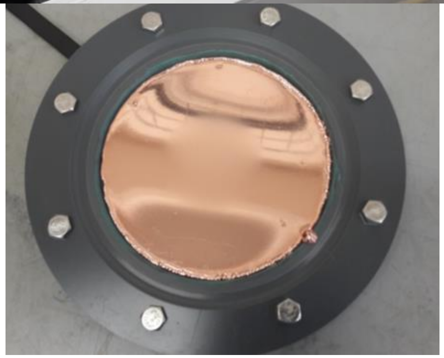
Two copper sulphate-sulphuric acid baths

Bath without additives

Bath with brightener

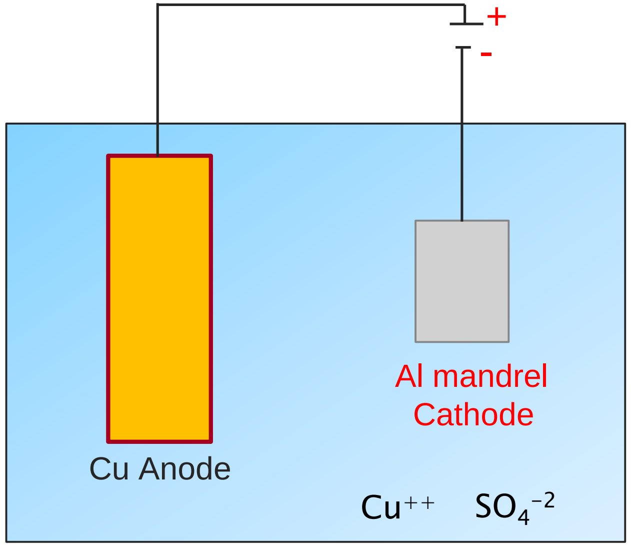


Pulse plating



DC plating

Setup Schematic



Chemistry

Cathode (reduction):

$$Cu^{2+} + 2e^- \rightarrow Cu$$

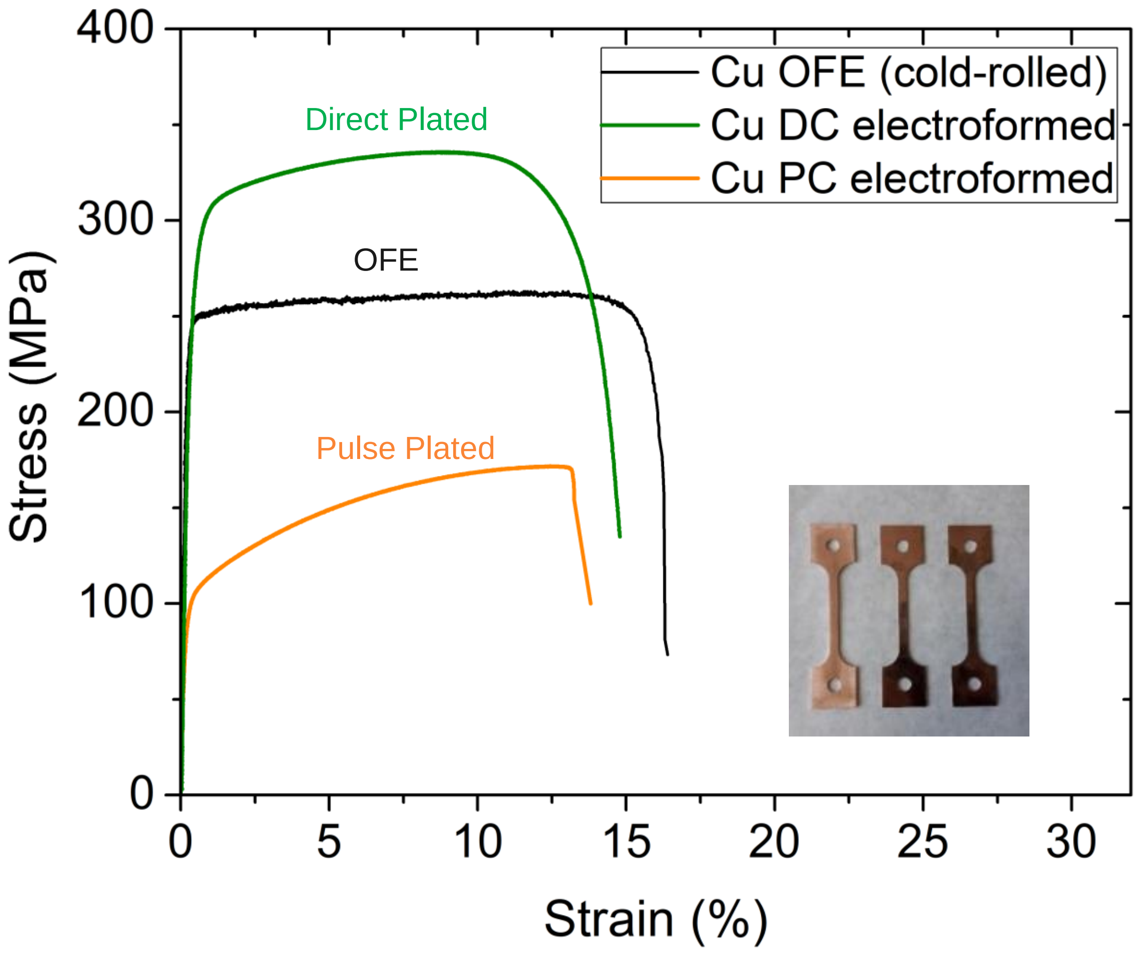
Anode (oxidation):

$$Cu \rightarrow Cu^{2+} + 2e^-$$

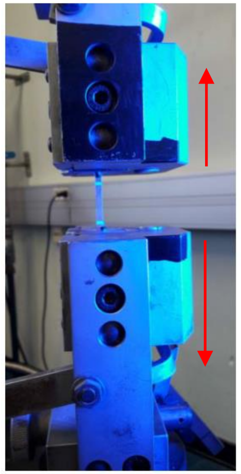
- Electrodeposition of Cu, 2 A/dm^2
  96 hours, 1.5 mm electroformed layer
- Aluminium removal dissolution

Electroformed copper properties

UTS/ Young modulus



- DC electroforming stronger than copper OFE cold-worked
- PC electroforming similar to copper OFE annealed



Ultimate tensile strength (UTS)

DC	PP
352 ± 41 MPa	174 ± 6 MPa

E modulus – impact excitation

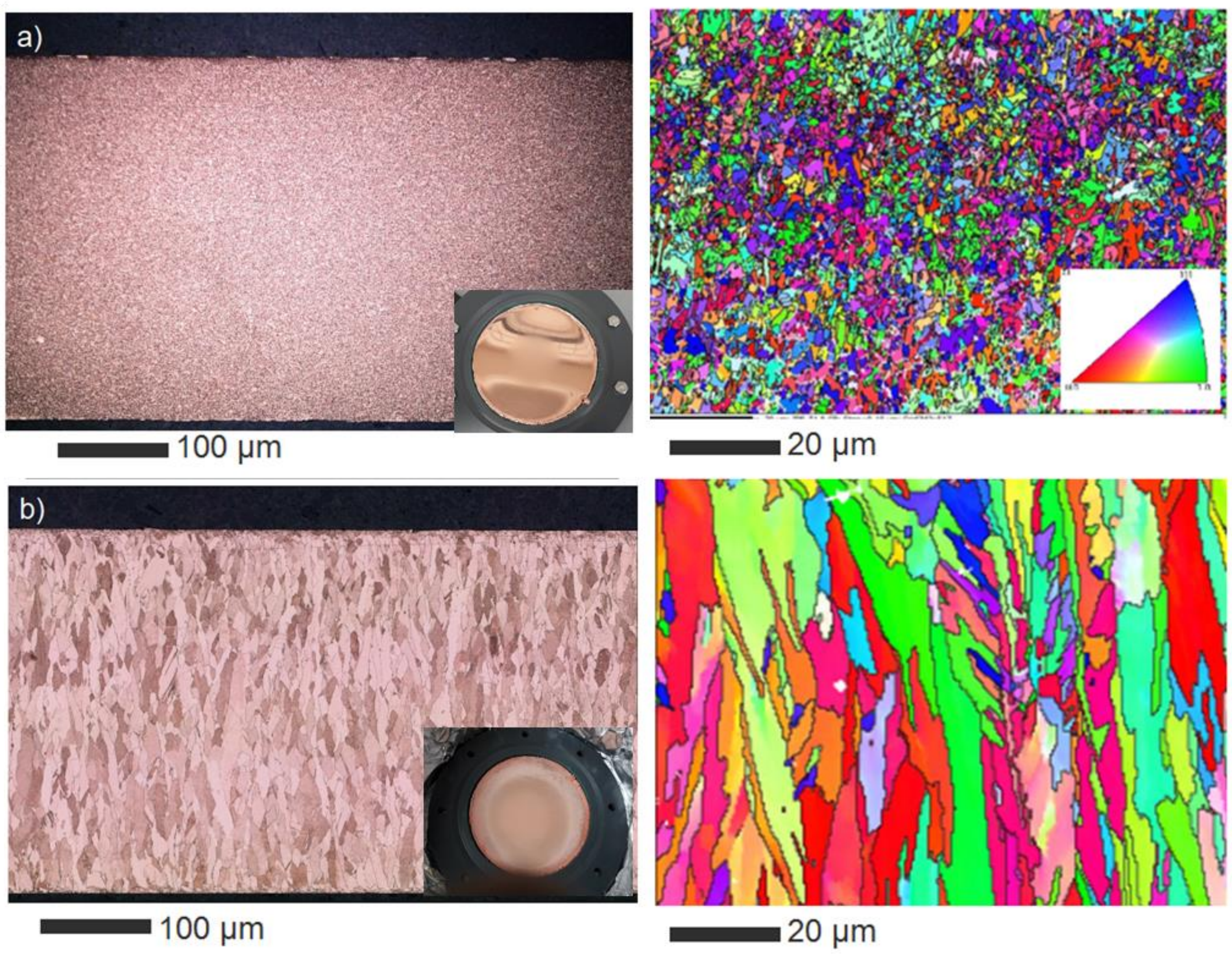
DC	PP
124 ± 15 GPa	131± 15 GPa

Electroformed copper properties

Microstructure and EBSD

DC plated
with additive

Pulse plated
w/o additives



- **Tensile strength:**

  DC>PP : grains morphology

- **Grain size:**

  DC plating = 1-3 µm

  Pulsed plating =30-70 µm

  Cu OFE = 13-17µm

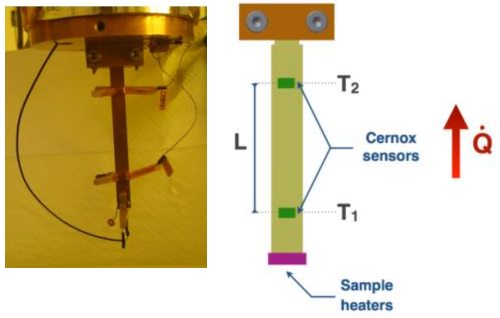
- **Different grain growth**

- **EBSD** shows no preferential grain orientation.

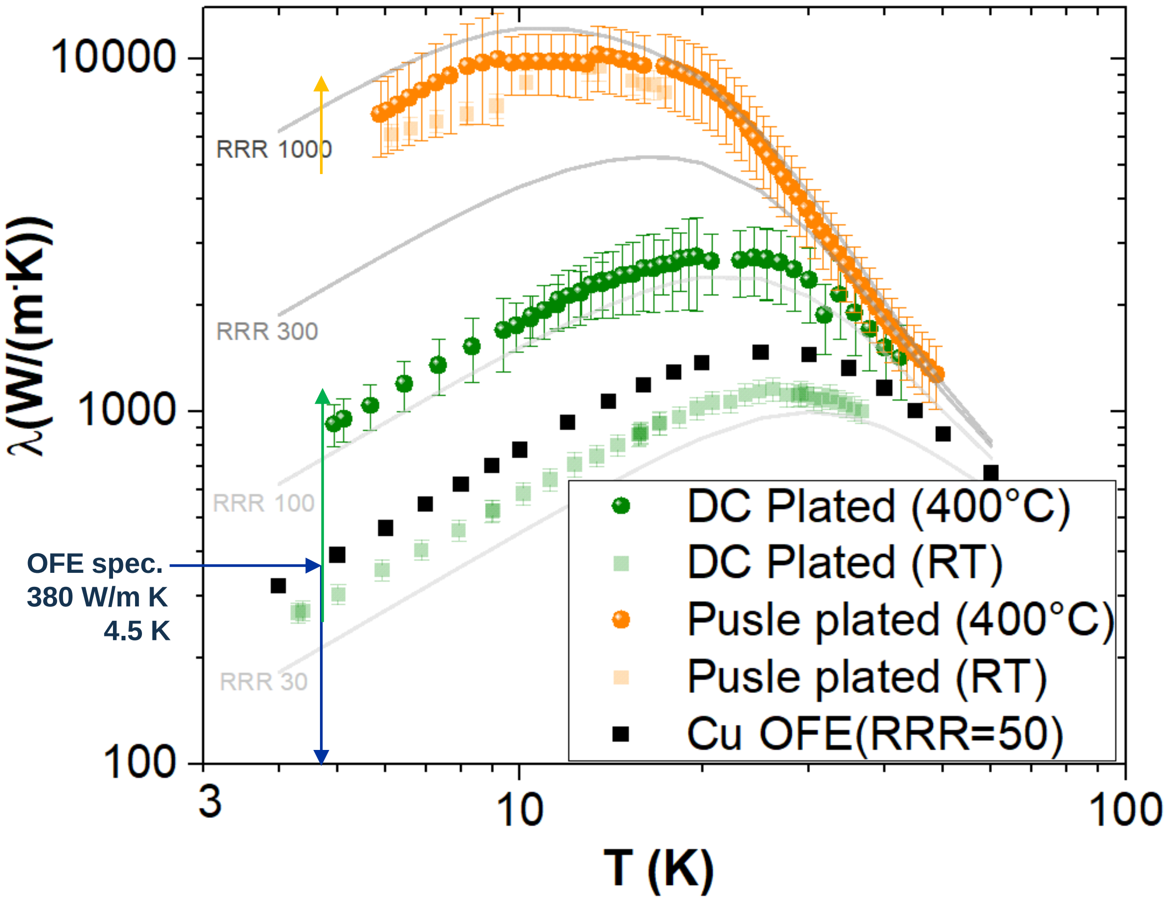
Electroformed copper properties

Thermal conductivity

Steady-state absolute measurements of thermal conductivity from 3 K - 40 K.



$$\lambda = \frac{\dot{Q}L}{A\Delta T}$$

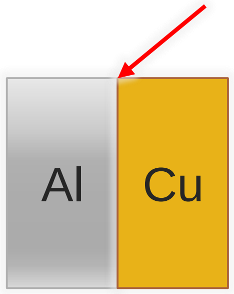


- *Samples after deposition:* Pulse plated sample conductivity 5 times larger than OFE spec.
- *After 2h at 400°C:* Triplicated conductivity for DC plated after thermal treatment

- Pulse plated layer is very pure (less than 2 ppm of Oxygen measured by IGA) in comparison with OFE copper (5 ppm) and DC plated copper (6.2 ppm)

Electroformed copper properties

Roughness of internal layer

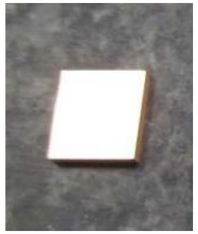


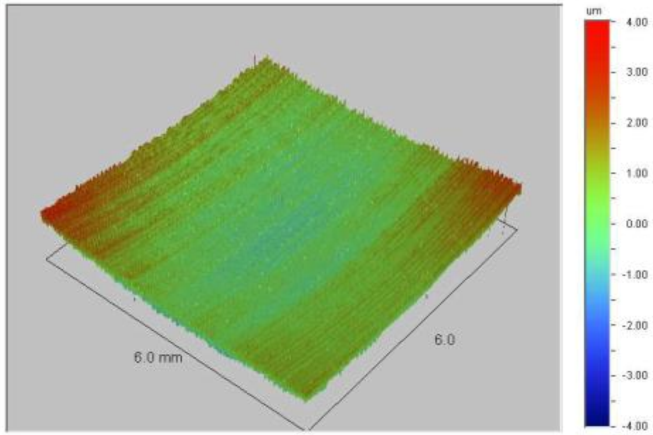
Standard mandrel machining
(Ra 0.49 µm)

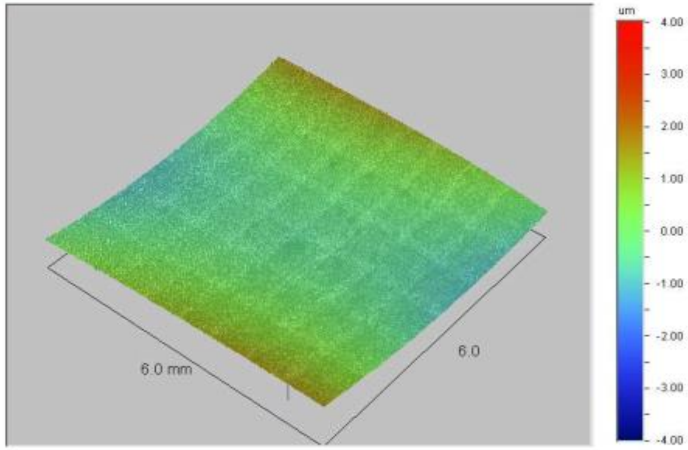
Diamond mandrel machining
(Ra 0.002 µm)

Ra (µm)	DC plated	Pulse Plated	DC plated	Pulse Plated
Cu	0.39	0.65	0.023	0.028







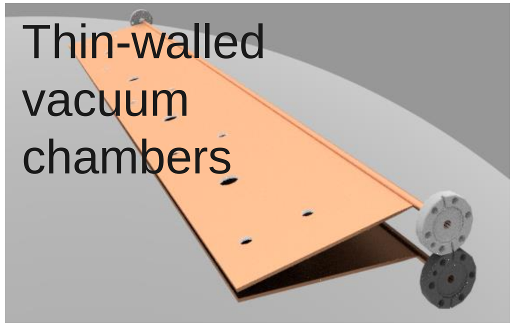
Cu layer reproduces mandrel topography

Electroformed copper properties

More suited for

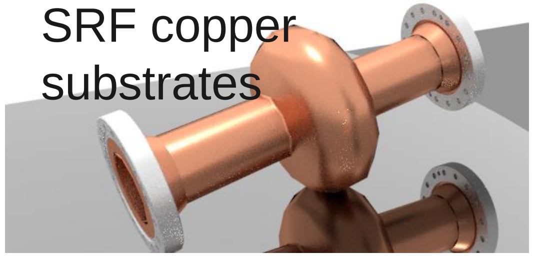
DC plated with additive

- High mechanical strength
- Very small grain size



Pulse plated w/o additives

- High thermal conductivity
- Very pure layer



Both

- Replicates surface mandrel state

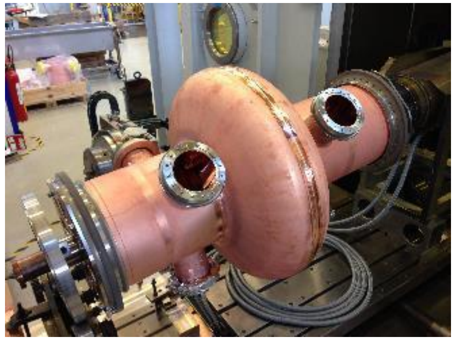
2. Electrodeposition of copper applied to the manufacture of seamless SRF cavities

In the framework of **Superconducting radio frequency niobium coated cavities**

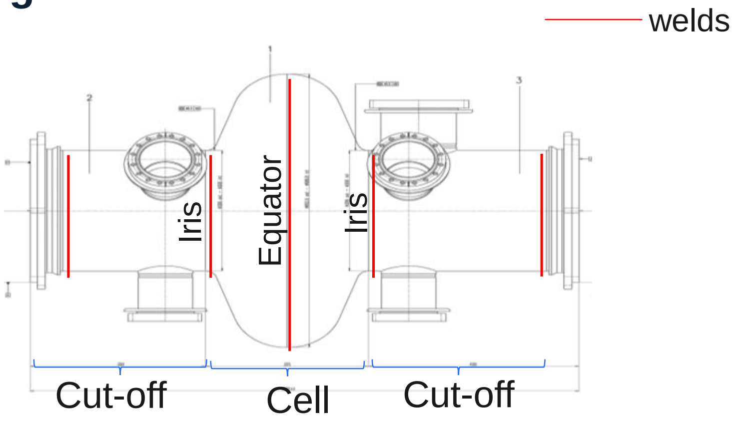
Production of copper SRF substrates

STANDARD METHOD - Half cell spinning and welding

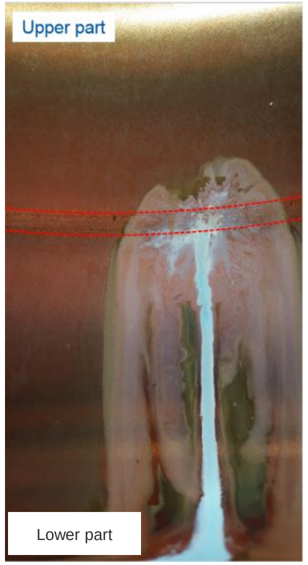
Half cell spinning

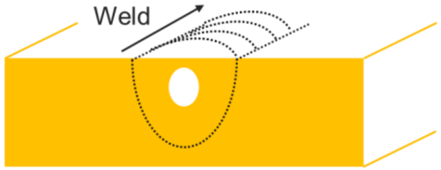


Welding



Possible defects



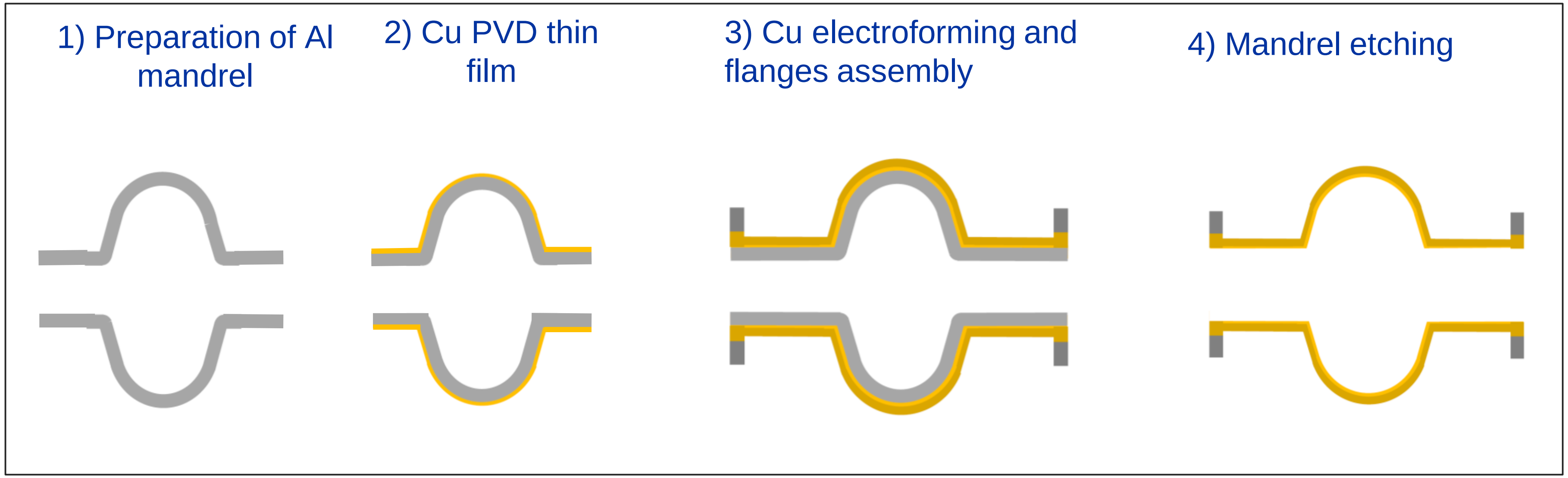


Weld porosities

- Presence of porosities along the junction caused by the welding process
- Welding grooves are localized in critical regions which are very important for RF performance.
- Copper sheets can contain defects.

Cu electroforming - approach

The cavity is produced by copper electroforming around a sacrificial aluminium mandrel which is pre-coated with a copper thin film.



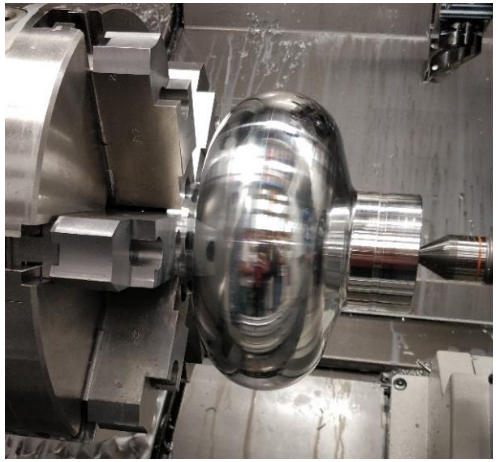
- Seamless cavities (**No EB welding**)
- Stainless steel flanges assembled during electroforming

Use this process to produce 1.3 GHz elliptical copper cavities

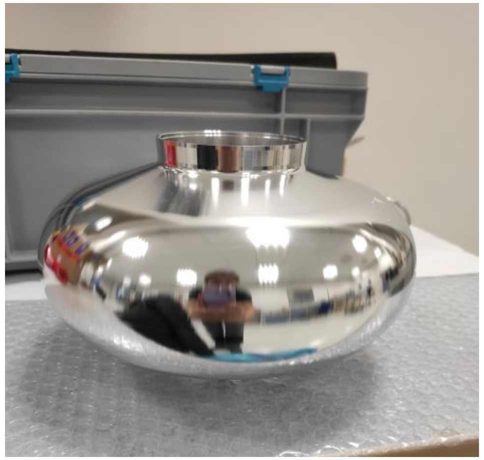
1.3 GHz Mandrel production

How to produce such an aluminium mandrel?

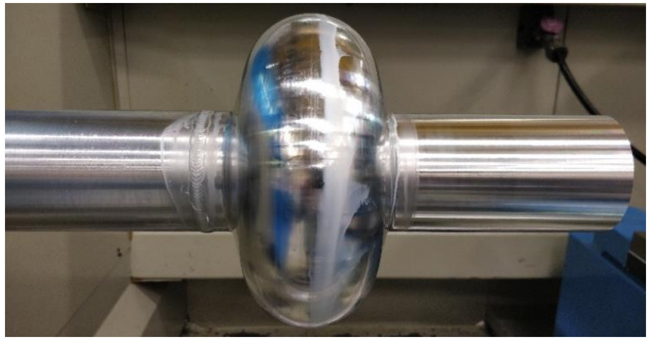
Machined from bulk aluminium



Mandrel cell turning



Mechanical finishing

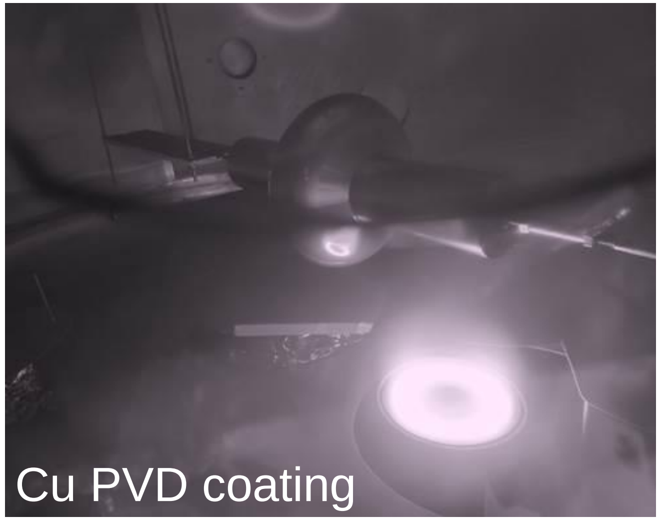


Tubes welding/machining

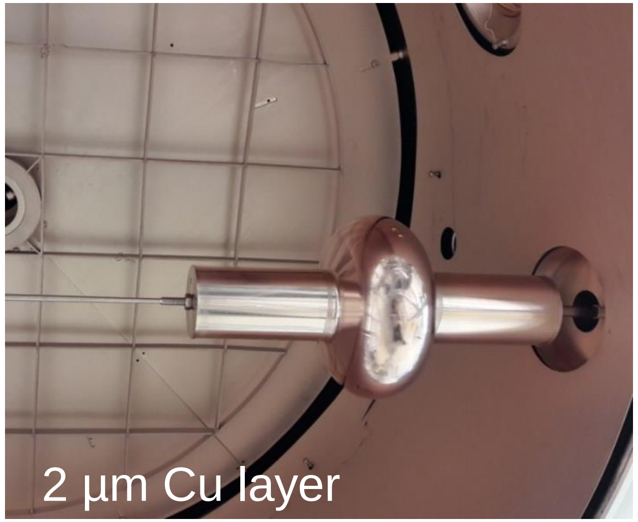
Final Mandrel

For the moment: Standard machining finishing

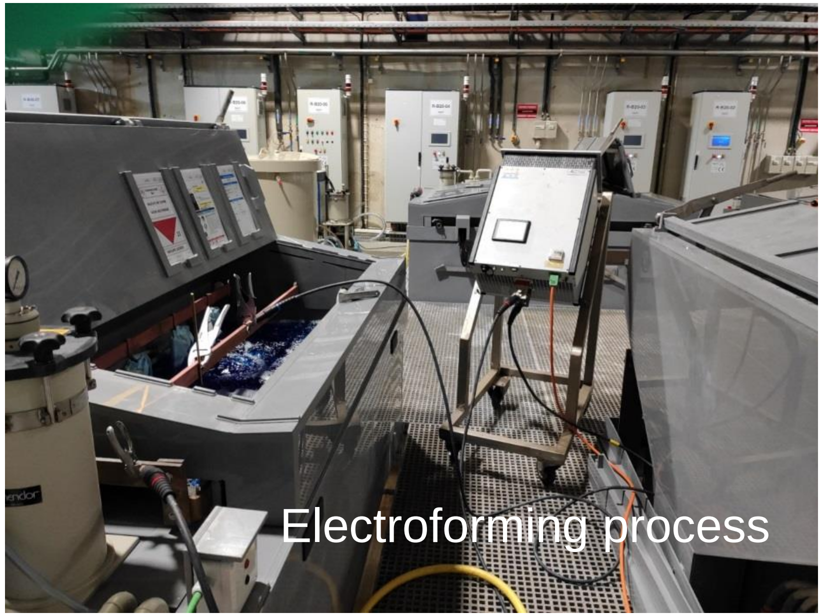
1.3 GHz cavity production



Cu PVD coating



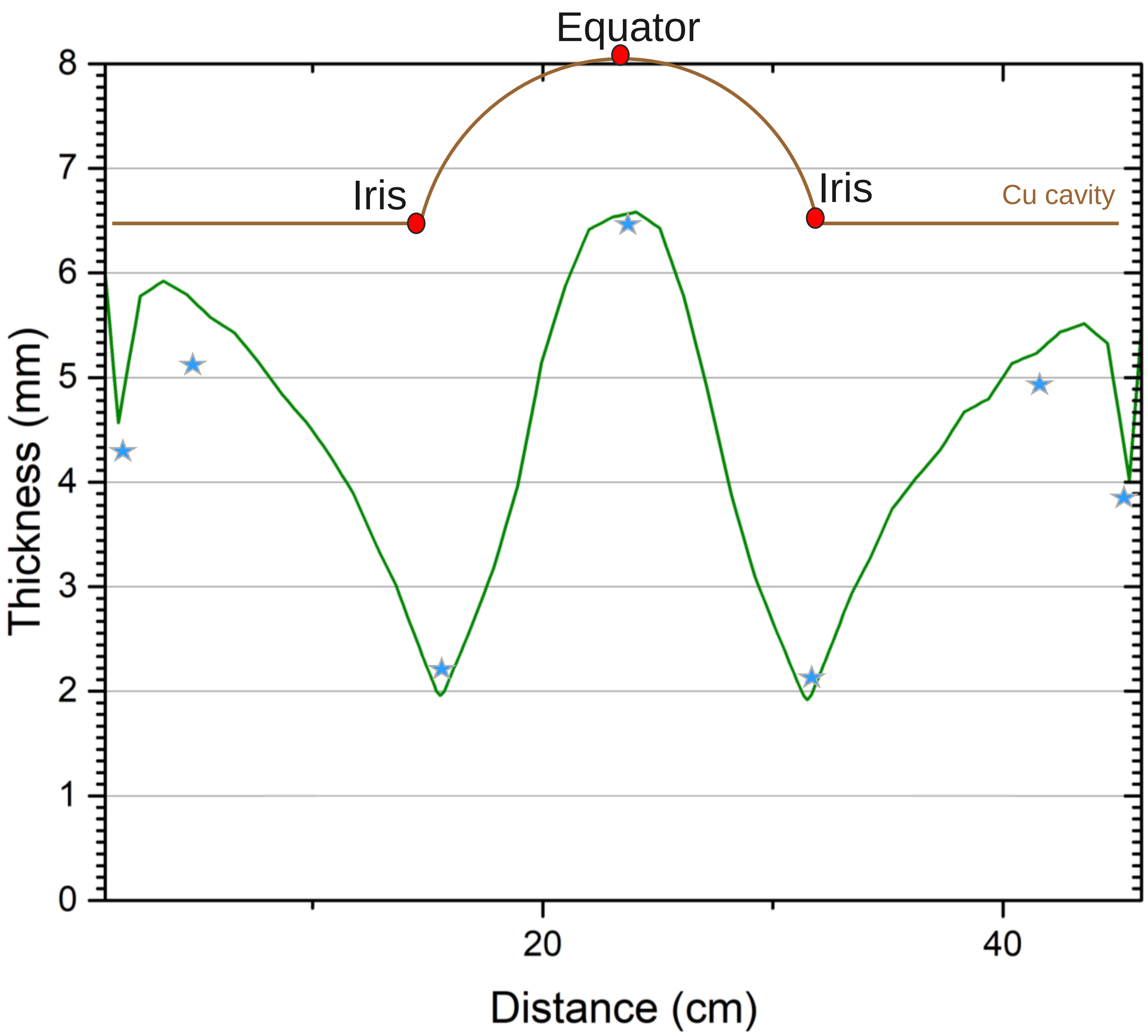
2 µm Cu layer



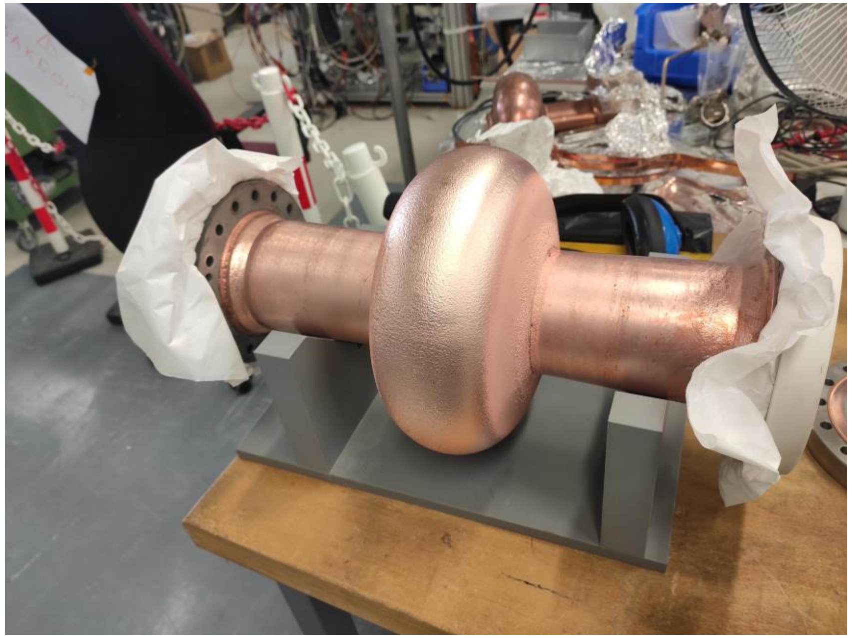
Electroforming process

330 hours of plating
(260 h pulse plating,
70 h DC plating)

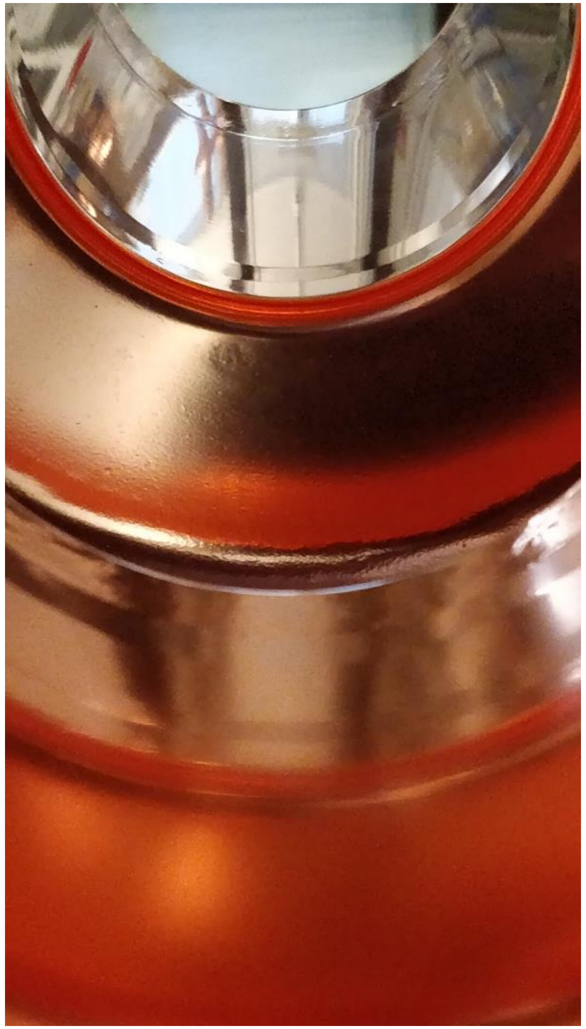
First 1.3 GHz cavity



- 2 mm plating at the iris
- 6.4 mm plating at the equator



1.3 GHz cavity production



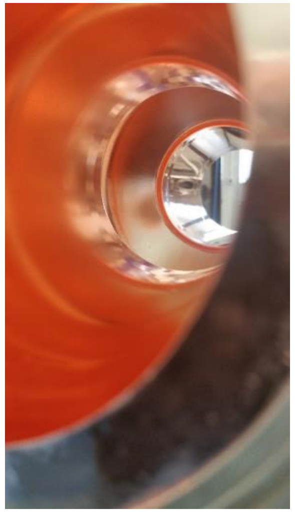
Aluminum dissolution NaOH 5M

Surface preparation: SUBU

First 1.3 GHz cavity

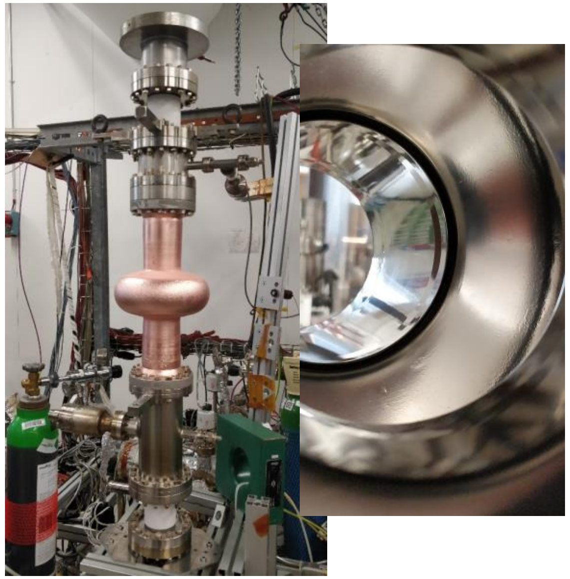
Workflow succesfully evaluated on electroformed cavity

Surface preparation



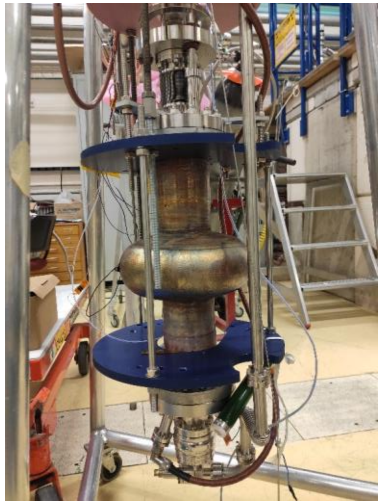
HPR

Nb Coating



HPR

RF testing

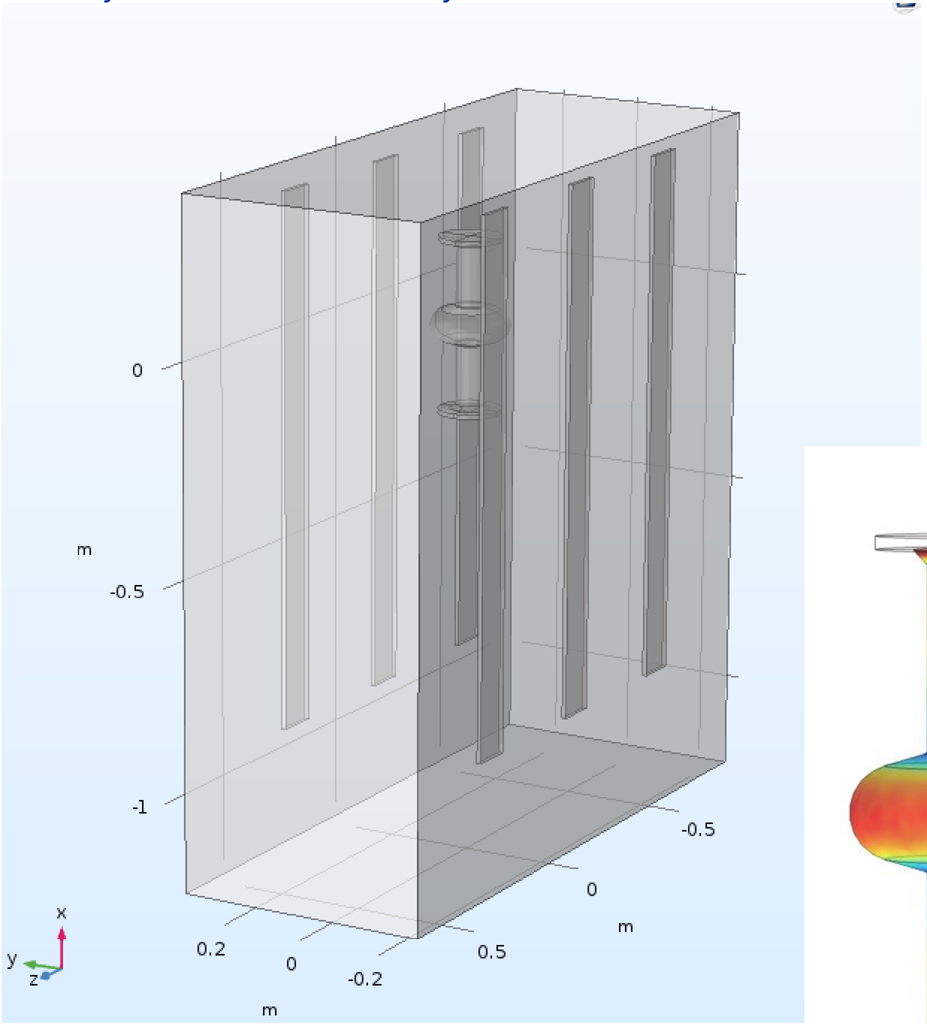


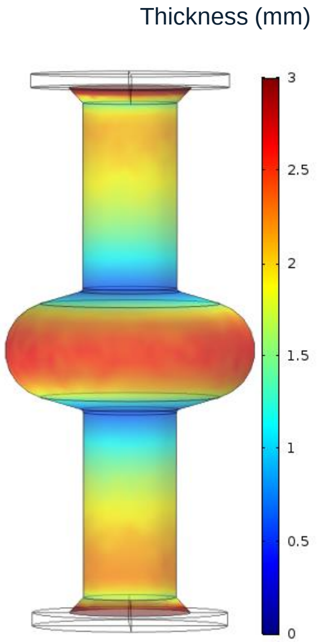
Nb stripping

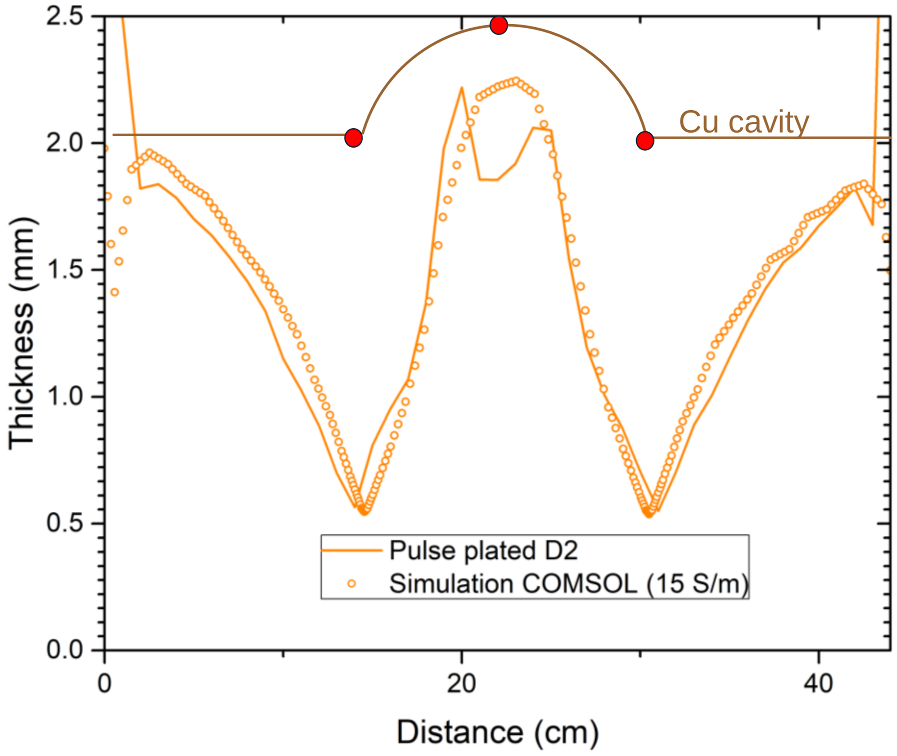
COMSOL simulations for optimization

Thickness profile simulated with COMSOL

Physics module: Secondary current distribution



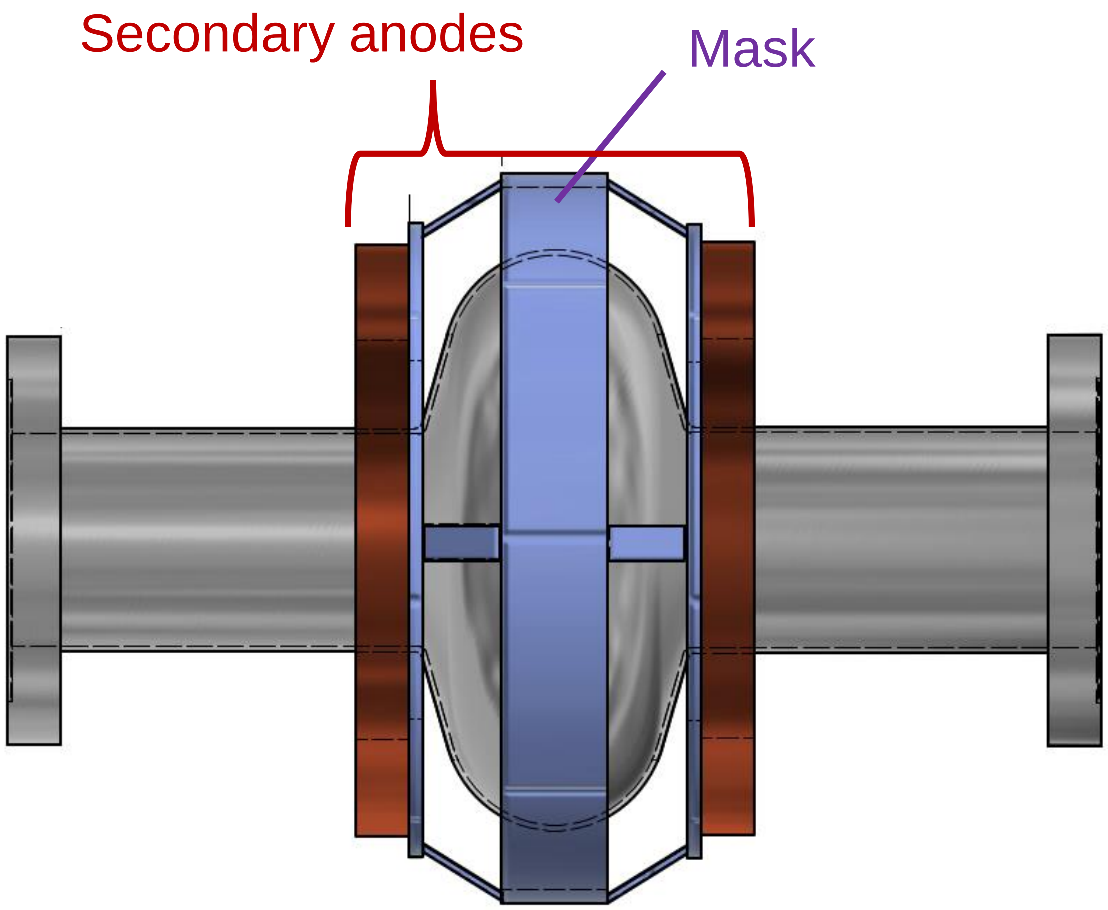




- Good agreement between simulation and experimental.
- Simulation can be used for optimization of anodes and mask.

Design of secondary anodes and masking

- Solution for uniformity: Secondary anodes positioned at the iris to promote plating, mask at the equator to reduce the deposition.

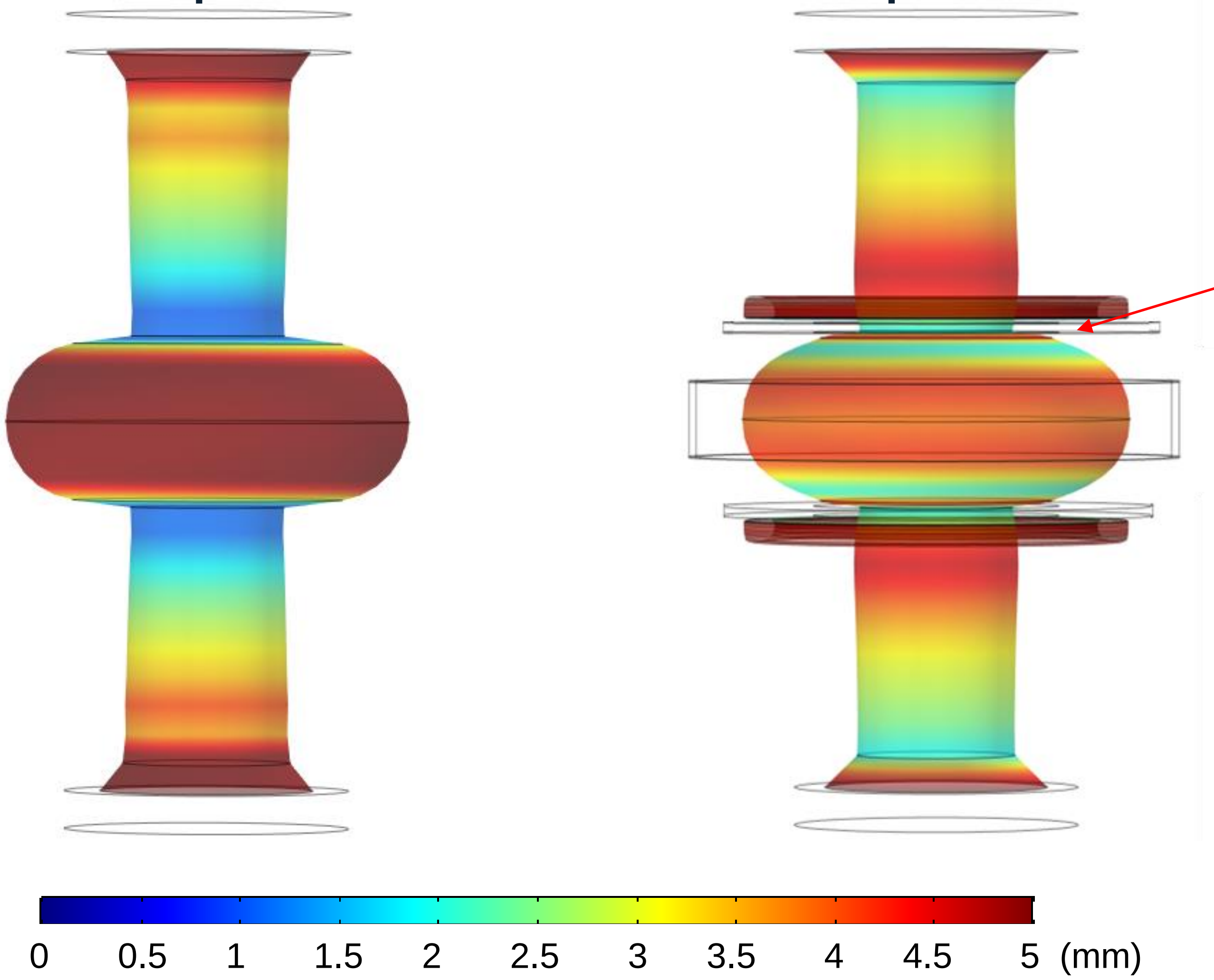


Design of secondary anodes and masking

Thickness profile simulated with COMSOL

Without optimization **After optimization**

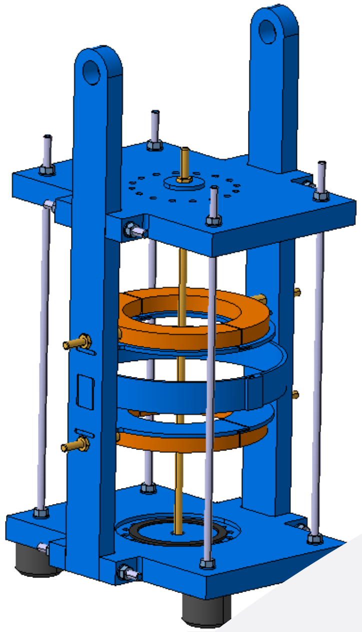
Thickening on equator

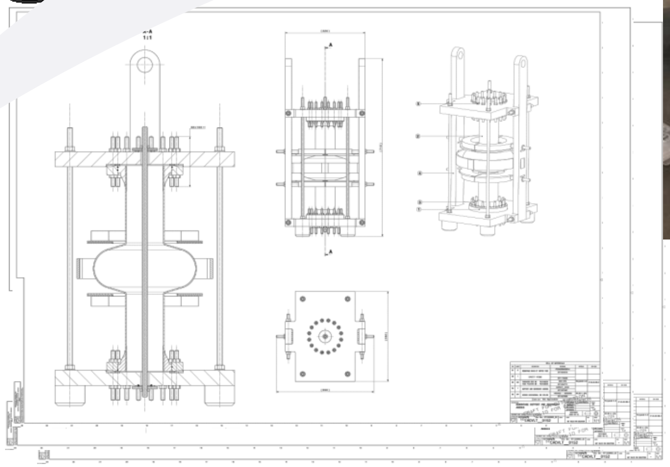


Thickening also on iris

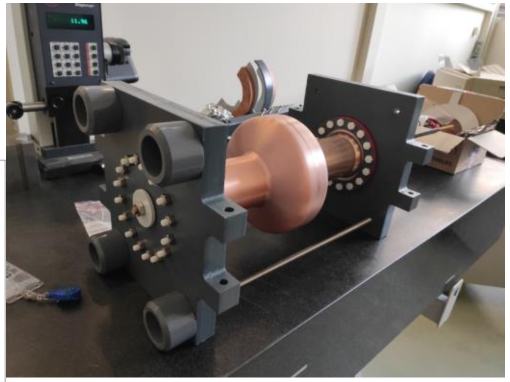
- 175 hours plating time for 2 mm at the iris
- Decrease plating time in half

Implementation of support

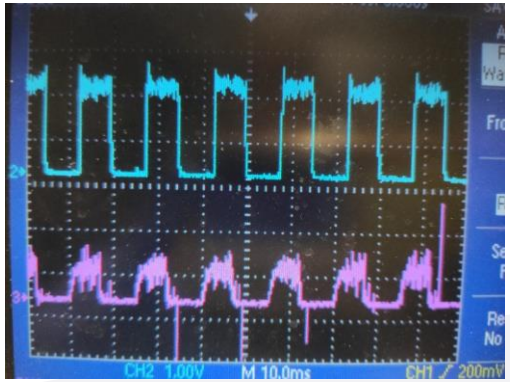




Design



Fabrication



Commissioning

Summary

- Cavity lifecycle (production-coating-rinsing-testing-stripping) feasibility has been demonstrated with the electroformed 1.3 GHz cavity.

- The main drawback of the electroforming approach is the non-uniform thickness distribution along the cavity.

  Solution: secondary anodes and masking to the cavity. The plating time will be reduced by half.

Future steps

- 1.3 GHz cavity production and validation of the secondary anodes support.
- Nb thin film coating using best recipe and RF testing.
- Different mandrels surface state: electroforming on polished mandrels.
- Implement inverse Nb coating.

3. Reverse thin film coatings for SRF cavities

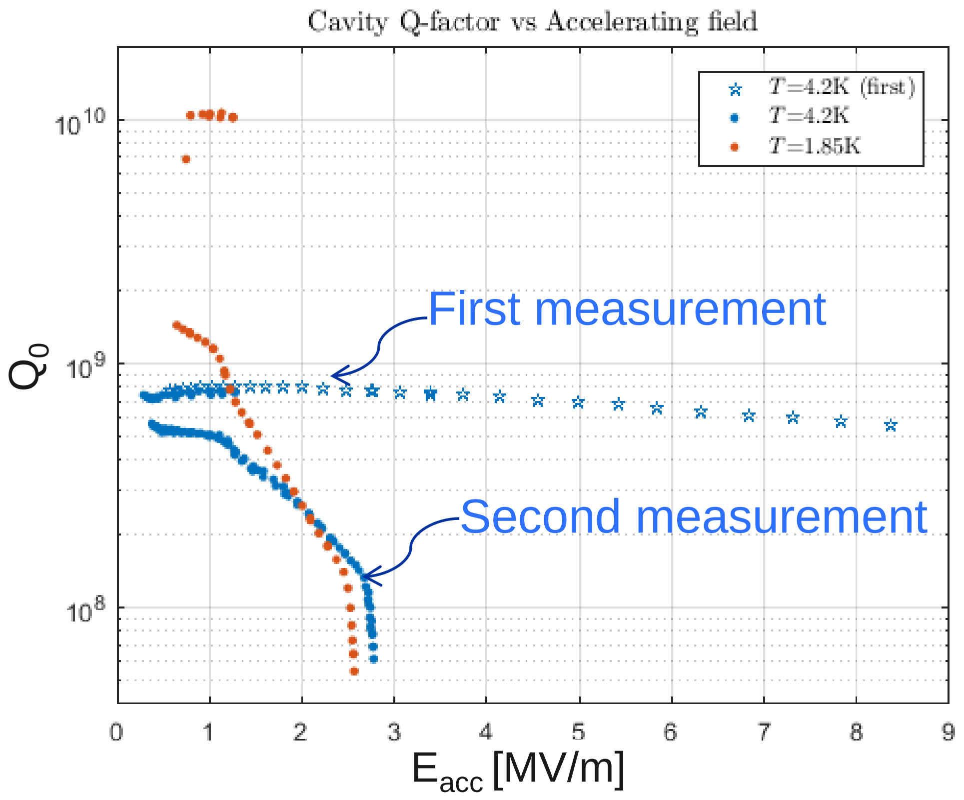
We have seen we can successfully produce SRF copper substrates.

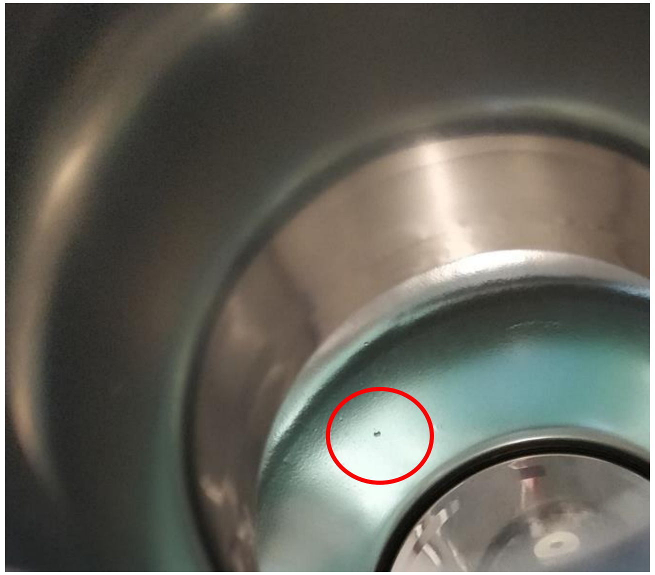
Can we integrate also a functional thin film coating in the process?

First Nb coated 1.3 GHz electroformed cavity

RF testing

L. Vega et al., presented in the International Workshop on Thin Films and New Ideas for Pushing the Limits of RF Superconductivity, 2021





Blister and Peel-off at cell

- First scan at 4.2K very good accelerating field and Q_0
- Second scan stopped at low accelerating field
  - Have we induced a peel-off?

Next trial: Nb coating with EP cavity preparation

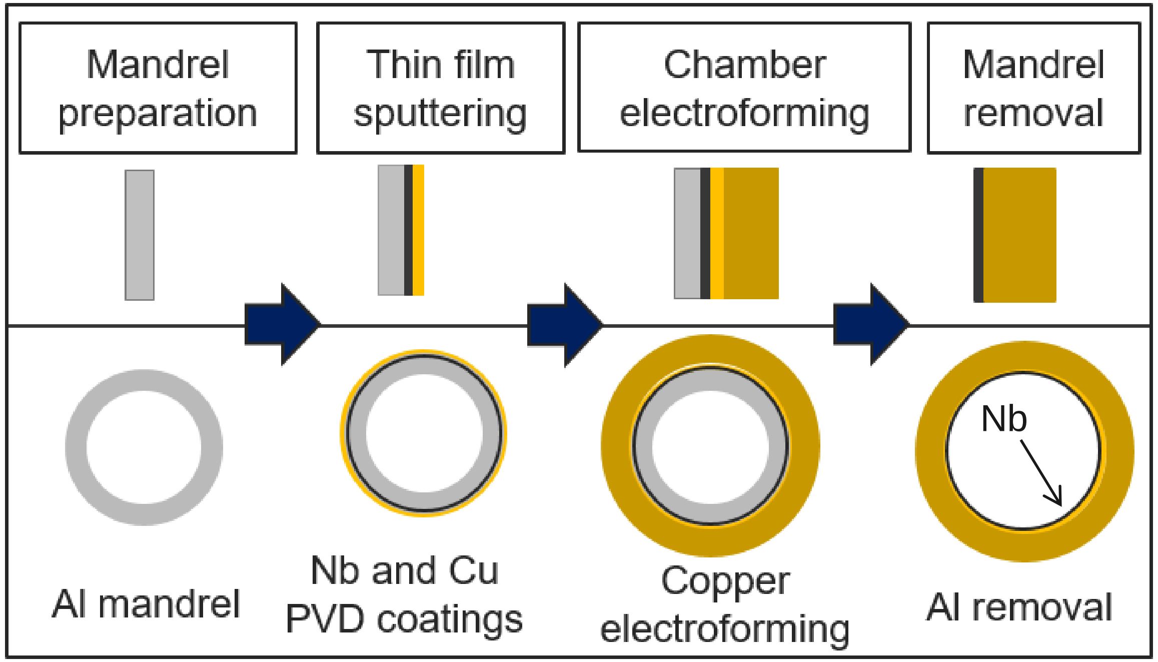
Inverse Nb coating on SRF cavities

One of the main bottlenecks of the standard Nb coating process, is the achievement of good adhesion at the Nb/Cu interface.

Solution

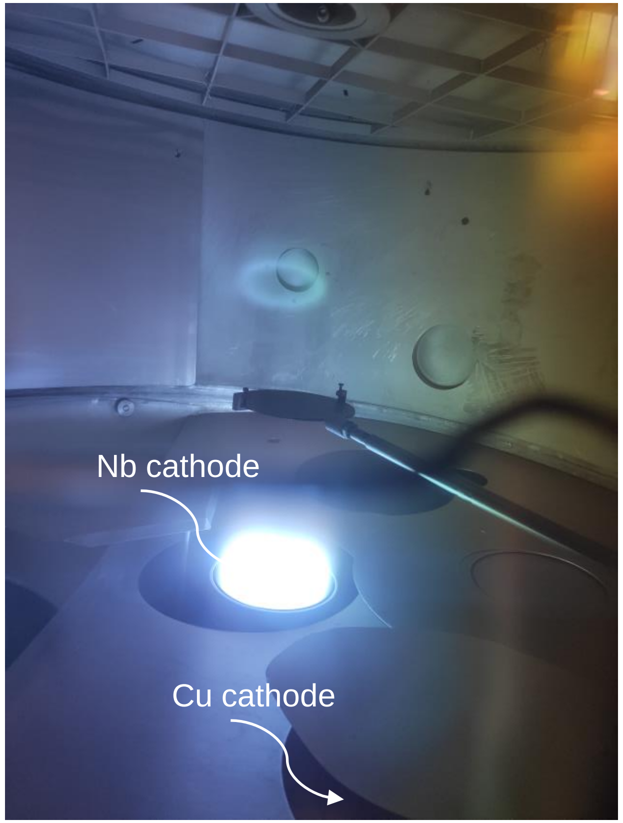
Produce the coated SRF cavity just in one process, improving the adherence between Nb and Cu layers and removing the chemistry surface preparation step.

Integrate the Nb coating on the production step

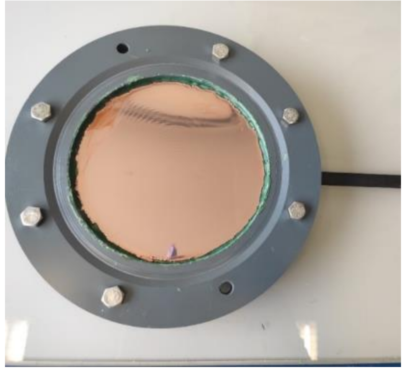


Based on idea from reverse NEG coatings: L. Lain Amador, CERN-THESIS-2019-160

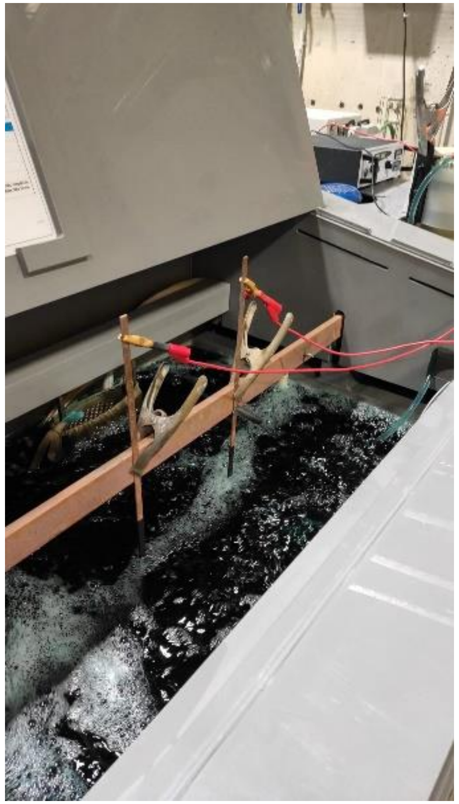
Inverse Nb coating on flat samples



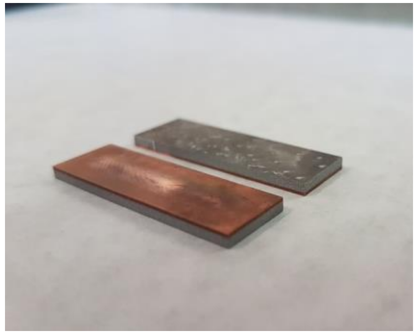
Nb and Cu coating



Preparation electroplating



0.5 mm electroplating

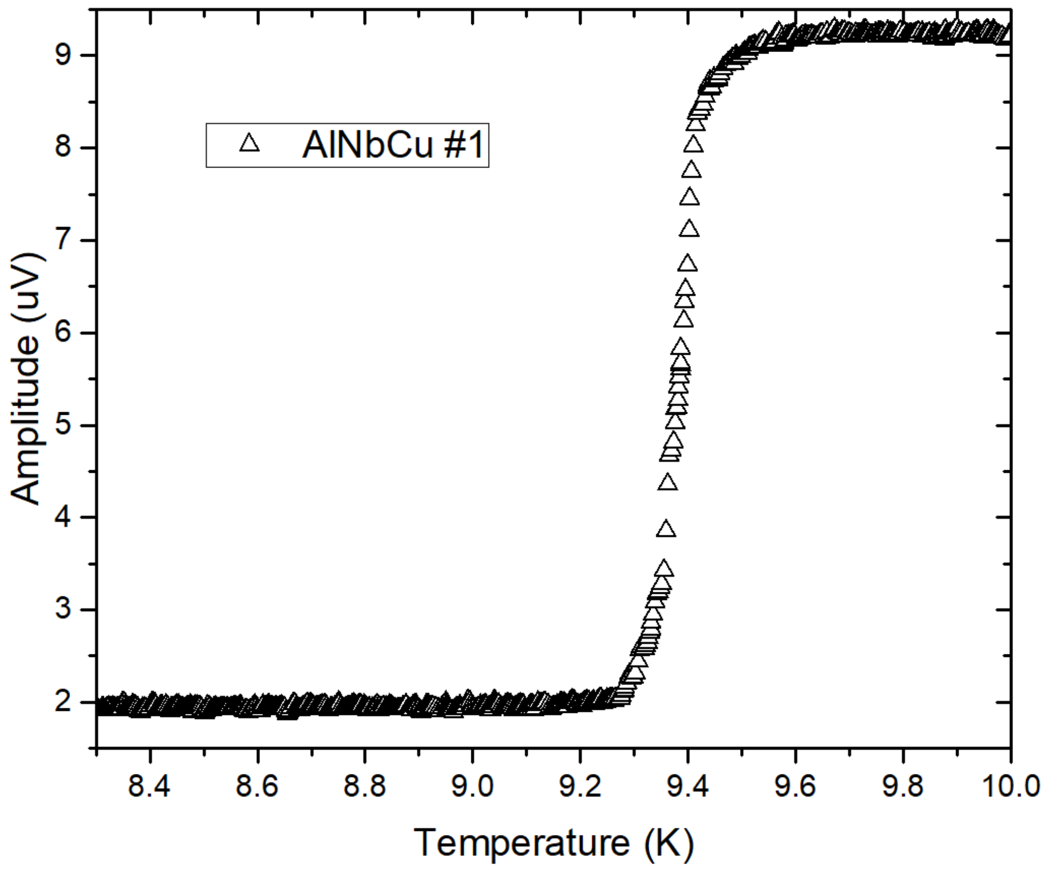


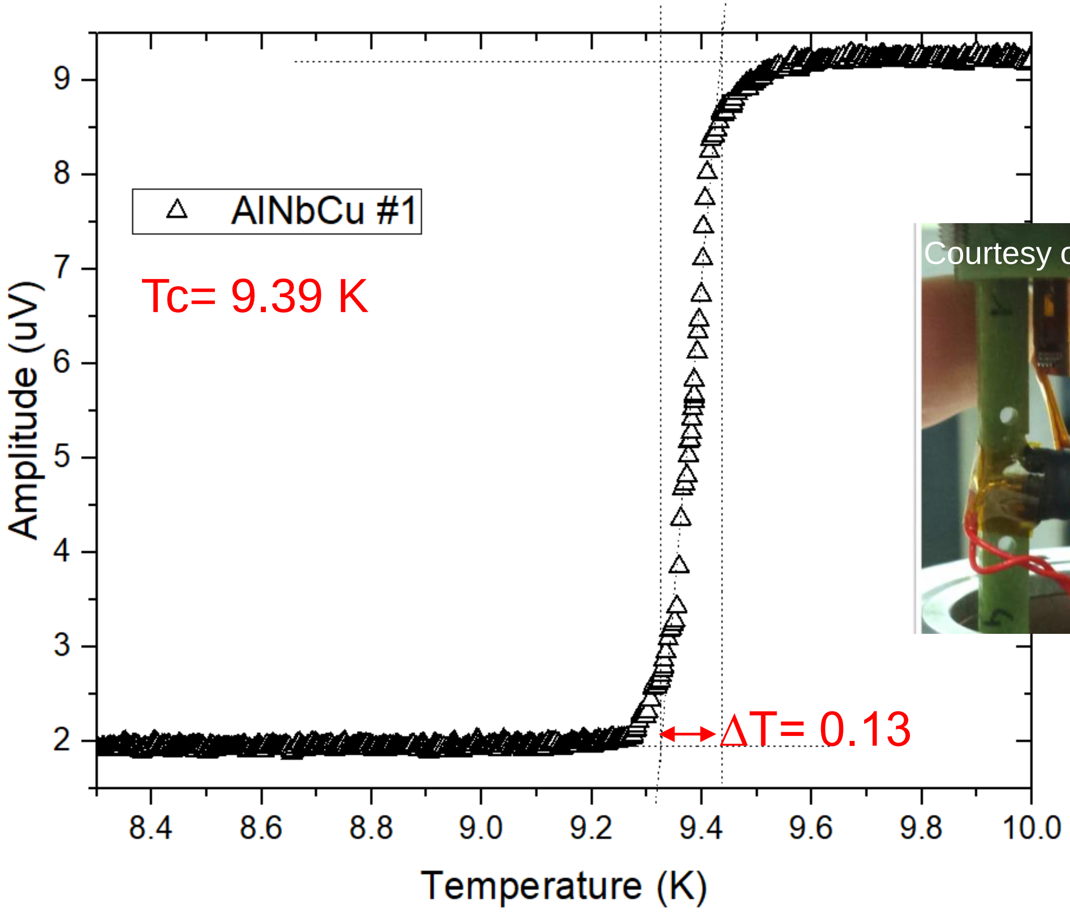
Samples cutting

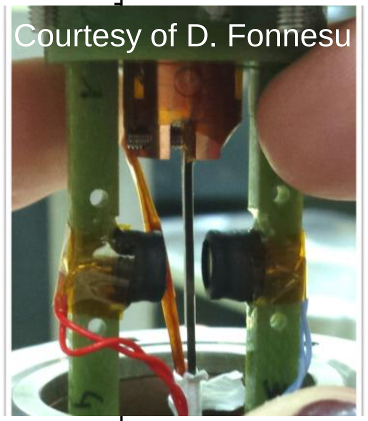
Al disk (1050 alloy), 150 mm diameter, 1.5 mm thickness

Nb Tc measurements (before etching)

1.5K/min







- Tc in agreement with Nb thin film literature values (Tc=9.25 – 9.45)[1]

[1] V. Palmieri et al. Proceedings of the Fifth Workshop on RF Superconductivity, DESY, Hamburg, Germany

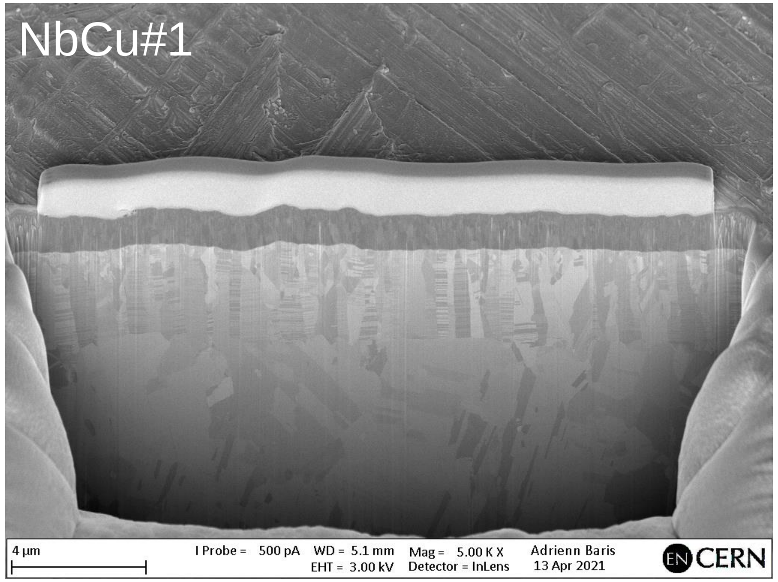
Additional challenges

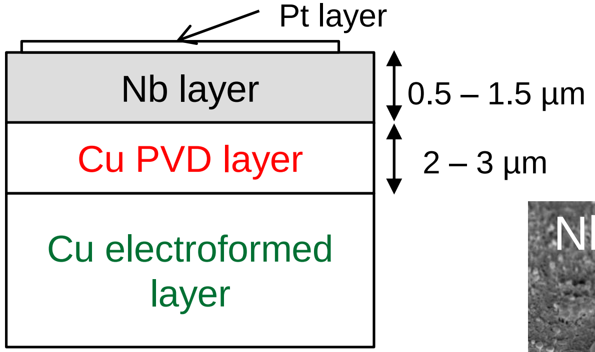
- Removal of the aluminium mandrel without damaging the Nb thin film

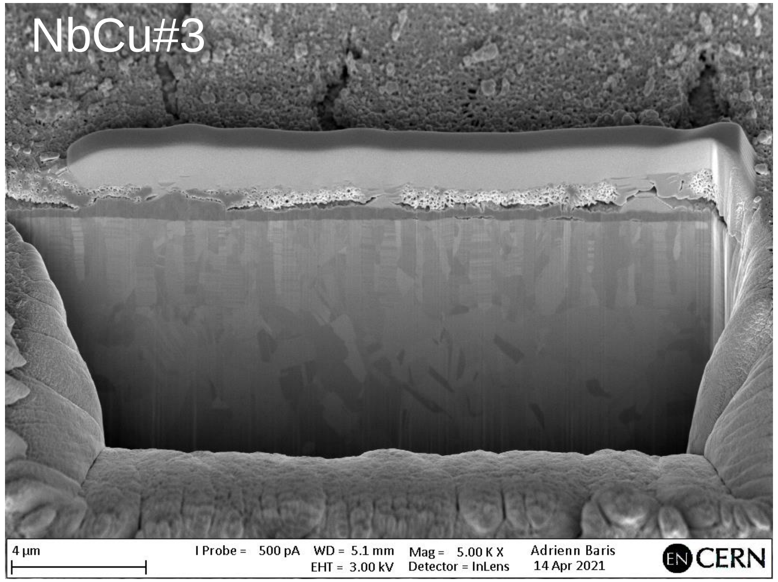
$$2\ Al(s) + 2\ NaOH(aq) + 2\ H_2O(aq) \rightarrow$$
$$2\ NaAlO_2(aq) + 3\ H_2(g)$$

Nb coating characterization

FIB cross-section and SEM analysis





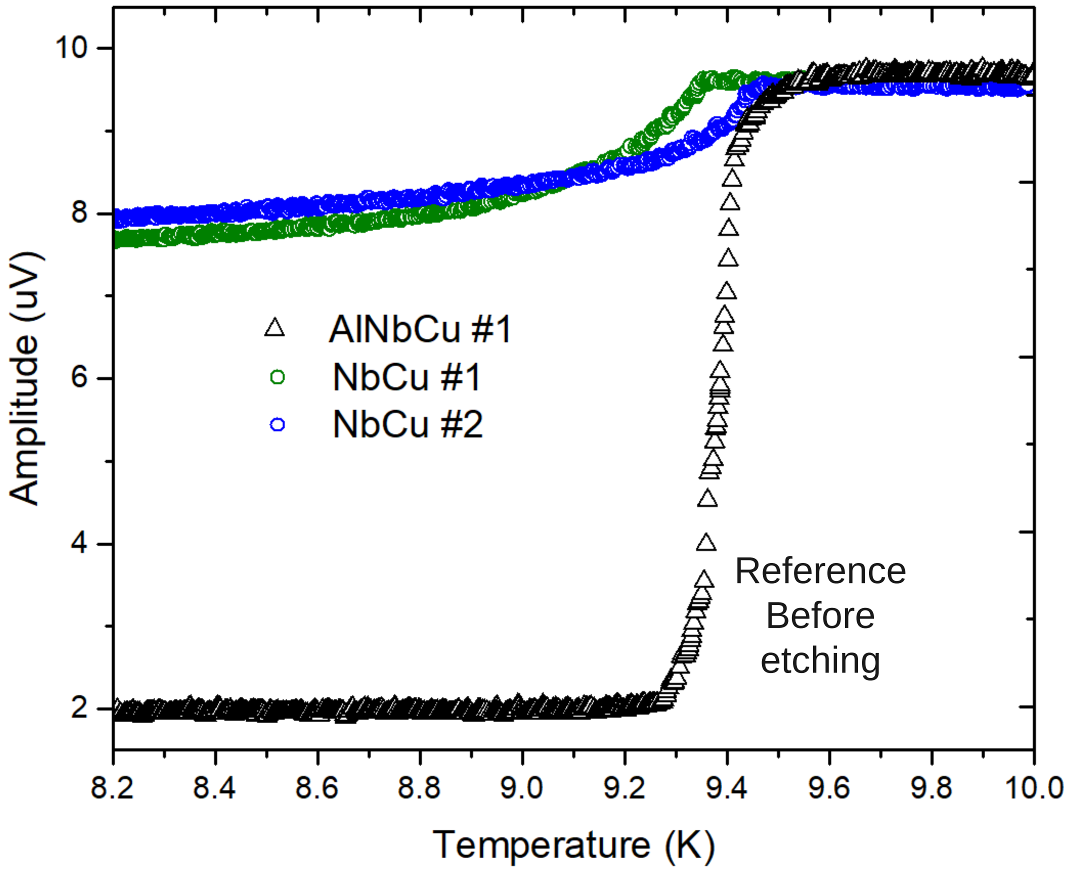


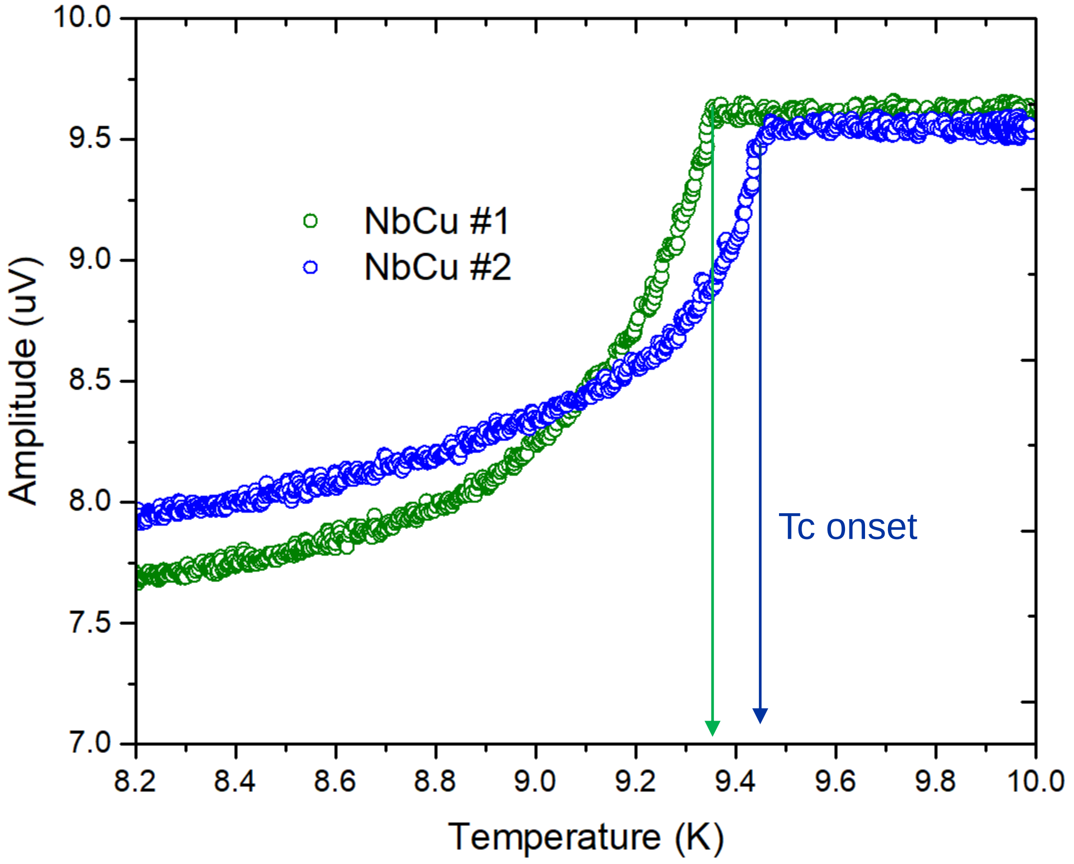
- Nb/Cu sharp interface without voids: Good adherence
- Nb coating topography follows the extrusion lines of the aluminium mandrel

- Some samples exposed for longer times to NaOH present Nb damaged layer
- Formation of porous Nb-O layer on surface.

Nb Tc measurements (after etching)

1.5K/min

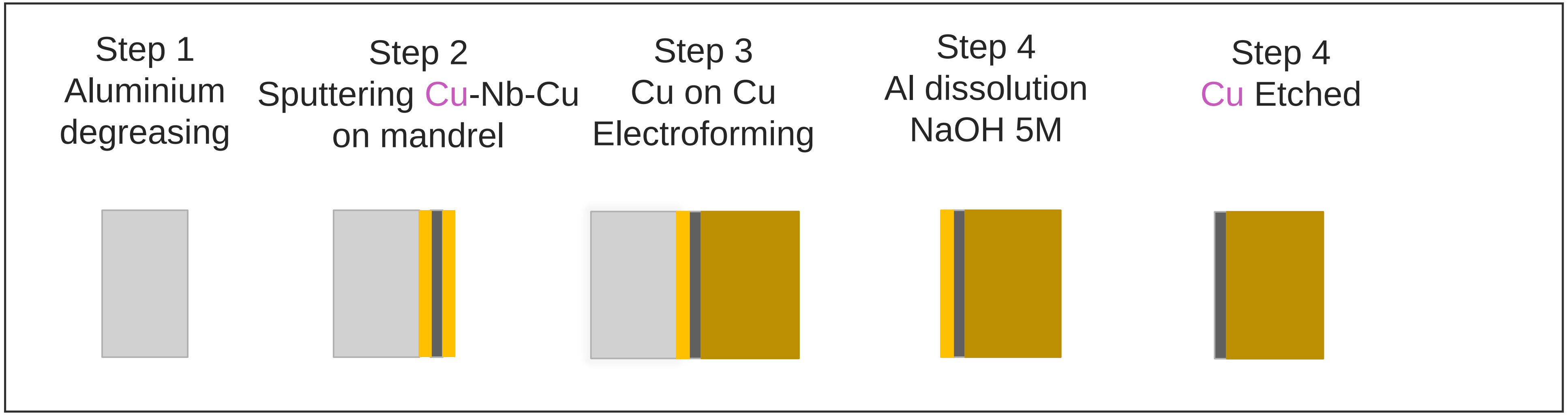




- Degradation of the superconducting performance
- Curves present a transition-like behaviour

Use of protective layer

- Good Tc until aluminium mandrel removal.
- A protective layer between the Nb and the aluminium will prevent the attack of the NaOH 5 M solution.



Conclusions

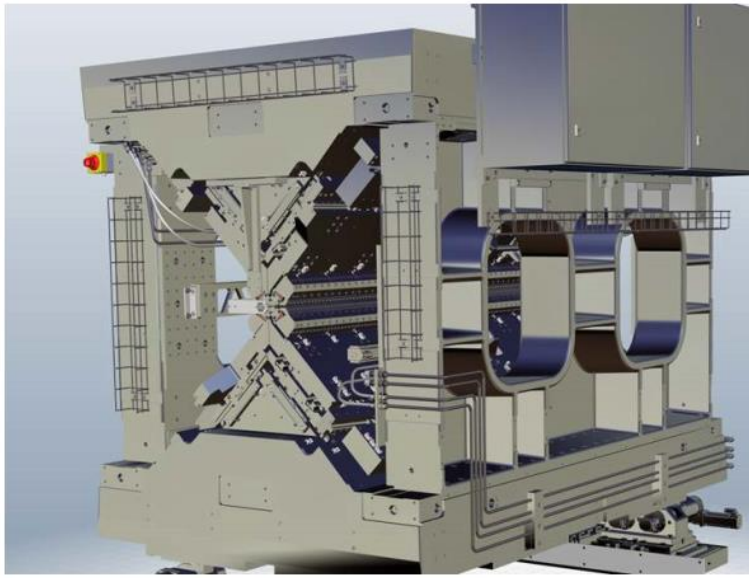
- Incorporation of the Nb layer to the electroforming process was successfully achieved.
- The NaOH attack the Nb layer when exposed for long times.
- Degradation of the superconducting performance.

Perspectives

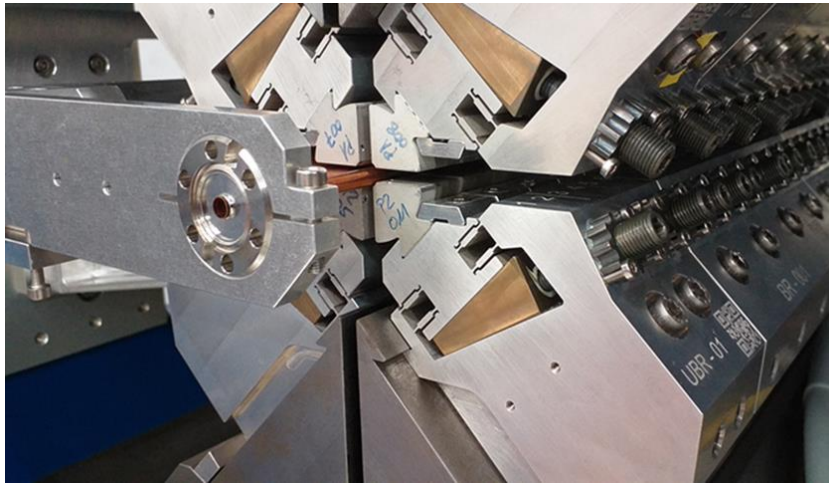
- Barrier layer between the Aluminium and the Nb (Cu layer good candidate)
- Annealing of coatings for possible H contamination
- If Tc is good, asses RF performance.

4. Development of thin-walled copper electroformed vacuum chambers for undulators

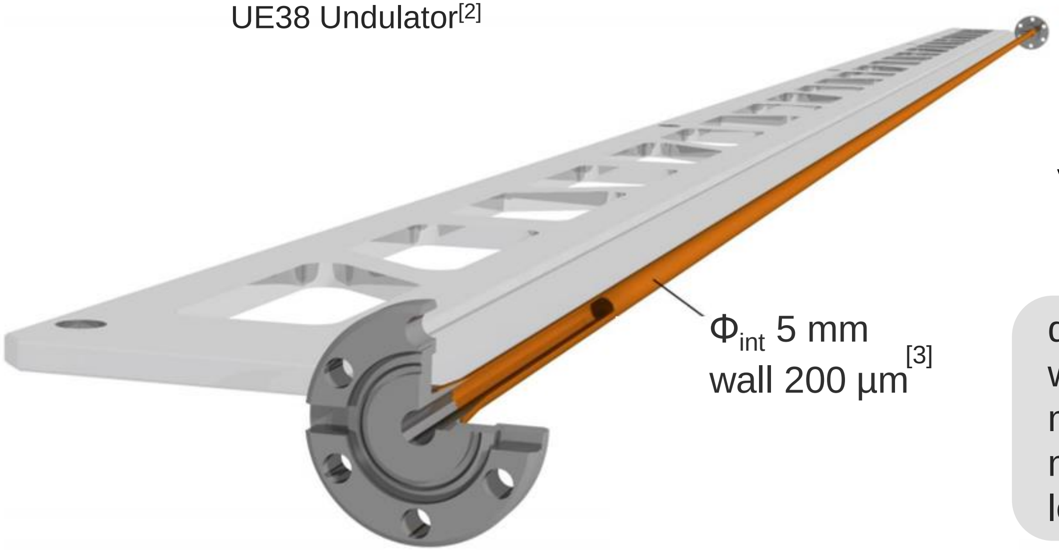
Electroformed undulator vacuum chamber (SwissFEL)



UE38 Undulator[2]



UE38 Undulator[2]



Vacuum chamber dimensions

diameter	5.0 mm
wall thickness	0.2 mm
magnet aperture	6.5 mm
minimum gap	3 mm
length	2040 mm

Other requirements

Cu Stiffener	2 mm
Ra (internal)	0.3 µm

[2] H. Joehri at al., MEDSI 2018

[3] Thomas Schmidt, Apple-X undulator for SwissFEL Athos and EU-XFEL (SASE 3)

Electroformed undulator vacuum chamber (SwissFEL)

Chamber manufacturing process by conventional methods

1. Extruded Cu tube of 200 µm wall thickness

2. Welding of the copper tube to the stainless steel flanges

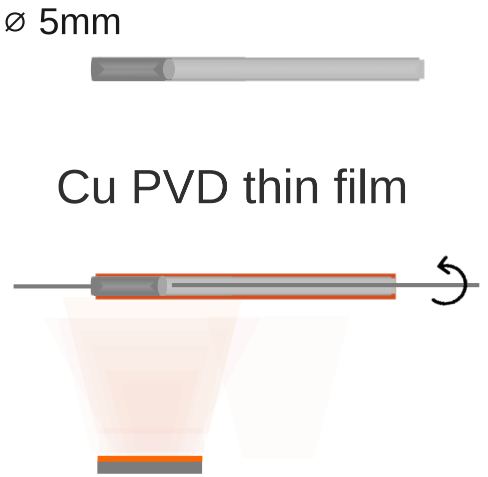
Stiffener can not be welded! (penetrated groove will damage the smooth inner surface)

3. Stiffener is glued
   Poor mechanical performance
   Glue cannot be heated up at high temperature
   Unknown glue behaviour under radiation

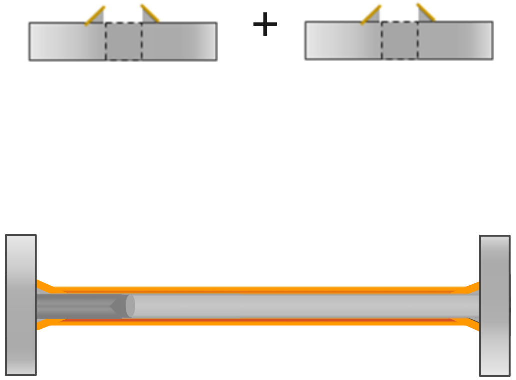
Can the thin-walled chamber be produced by electroforming?

Chamber electroforming approach

1. Preparation of Al mandrel

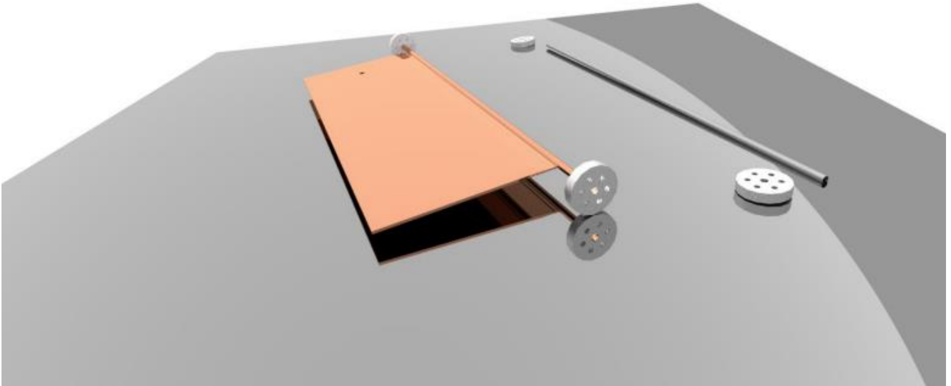


2. Cu electrodeposition 200 µm (step1)



3. Cu electrodeposition Stiffener addition (step2)

4. Mandrel etching



Starting point: 400 mm long chamber

Goal: 2 m length

Chamber electroforming approach

Preparation of Al mandrel

Cu coating (3 microm)

Cu coating process is performed by planar magnetron sputtering.

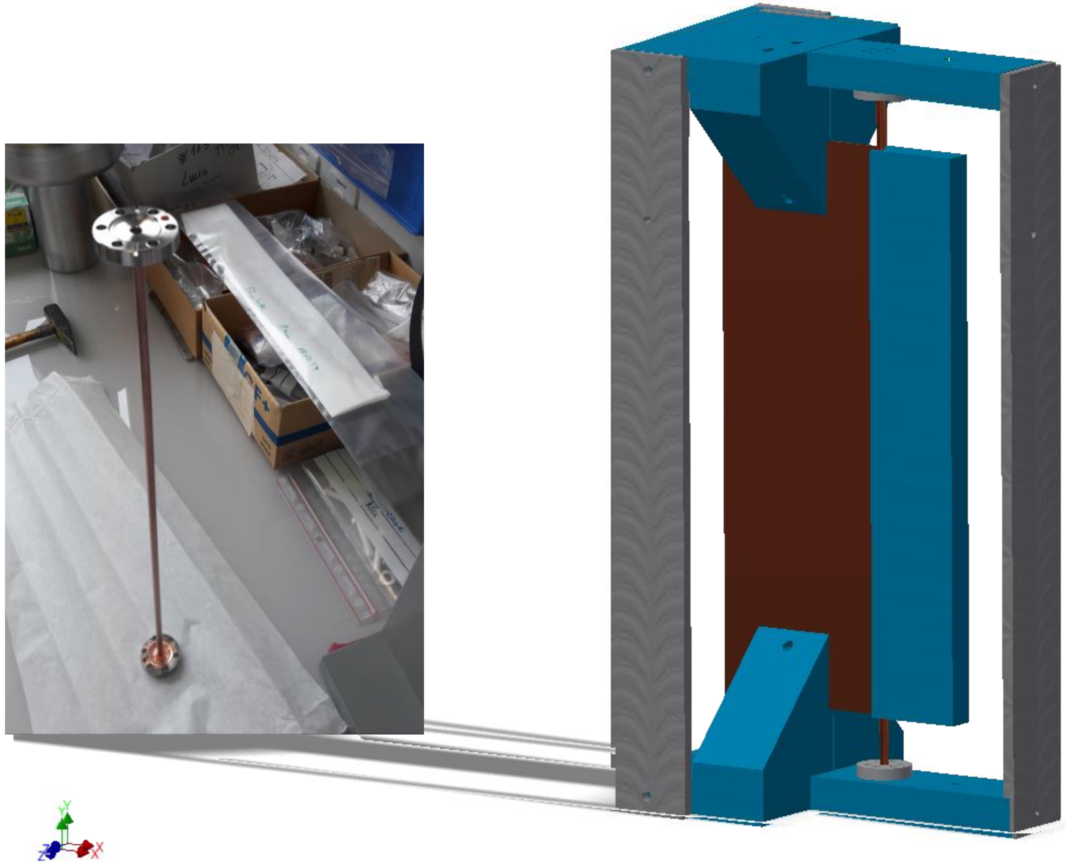
- Kr sputtering gas
- 2 coating steps with rotation of the mandrel

Preparation of the flanges

Modified DN16 flanges

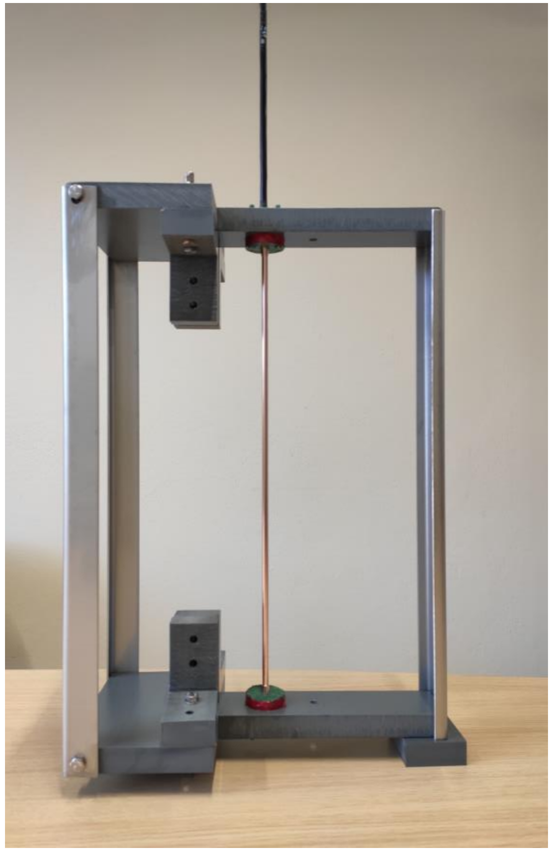
Cu plating is not adherent on SS. We need a Ni flash plated layer

Ni and Cu plating on stainless steel



Chamber electroforming approach

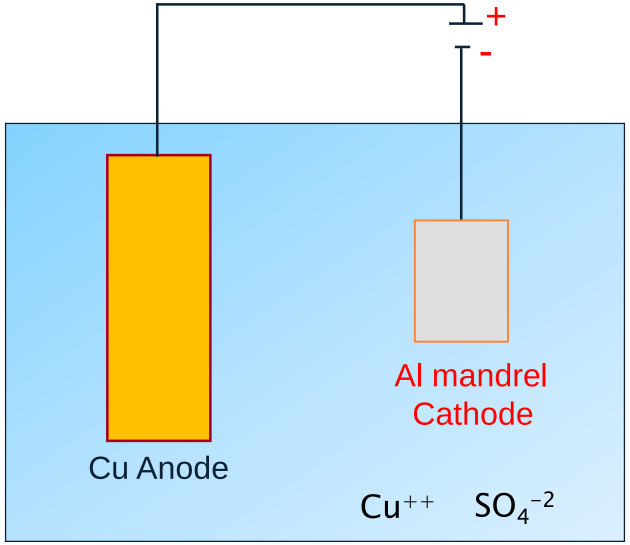
First plating: 200 µm thickness on the tube



Acidic copper sulphate with brigthener bath

6 hours plating

Direct Plating



Cathode (reduction):

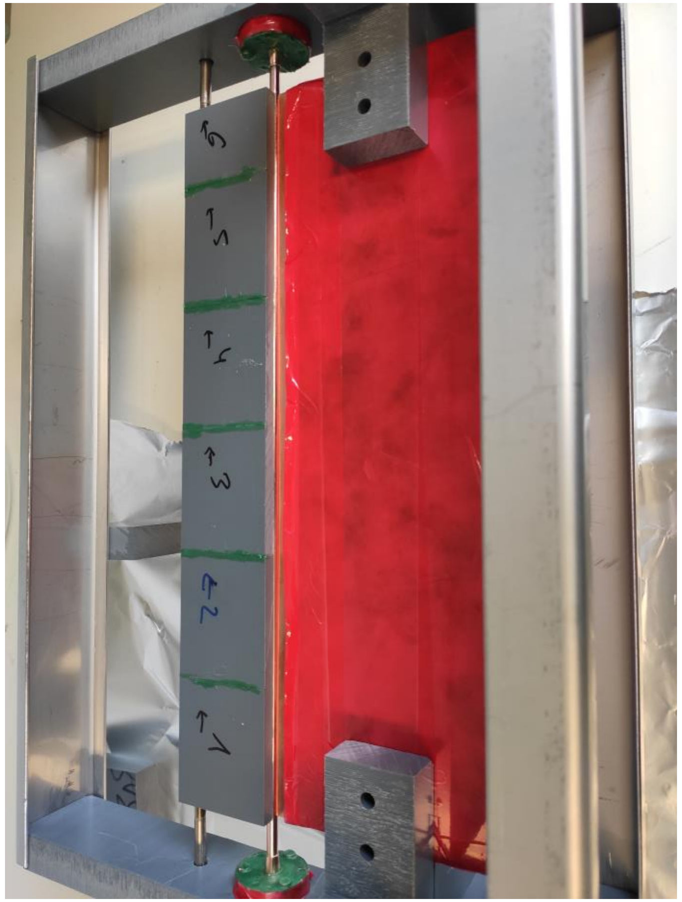
$Cu^{2+} + 2e^{-} \rightarrow Cu$

Anode (oxidation):

$Cu \rightarrow Cu^{2+} + 2e^{-}$

Chamber electroforming approach

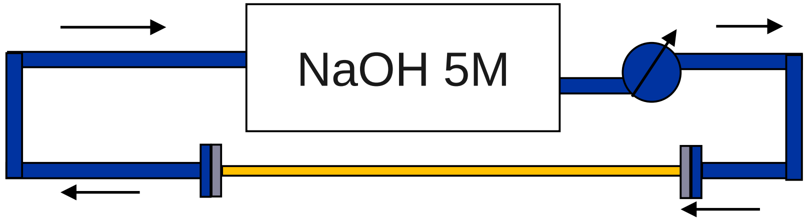
Second plating: Addition of the stiffener



Mask-tube-stiffener

24 hours plating

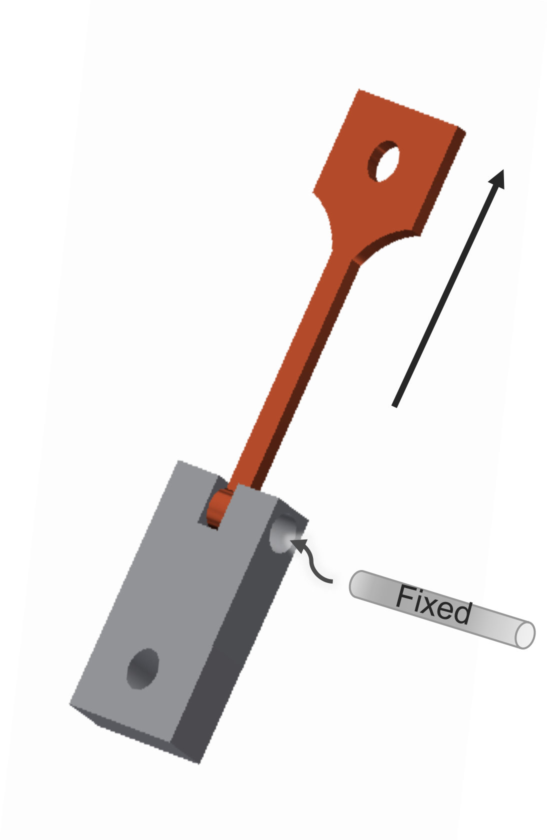
Mandrel etching: Aluminium dissolution NaOH 5M



Main challenges

The stiffener-tube junction has to be mechanically strong.

Tensile tests of the junction



Tensile specimens

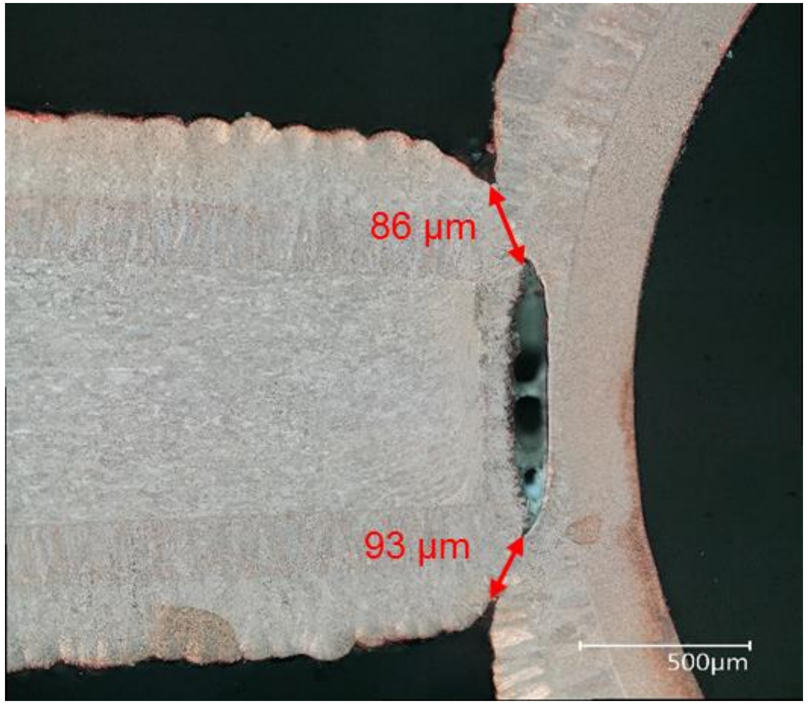
- No standard specimens
- No values of strain but values of stress

Metallographic cuts

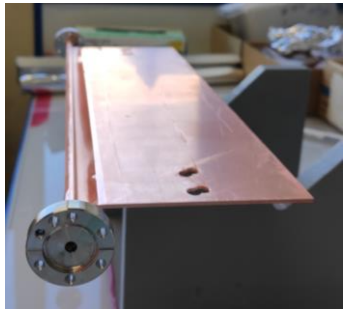
- Microstructure observation
- Junction properties

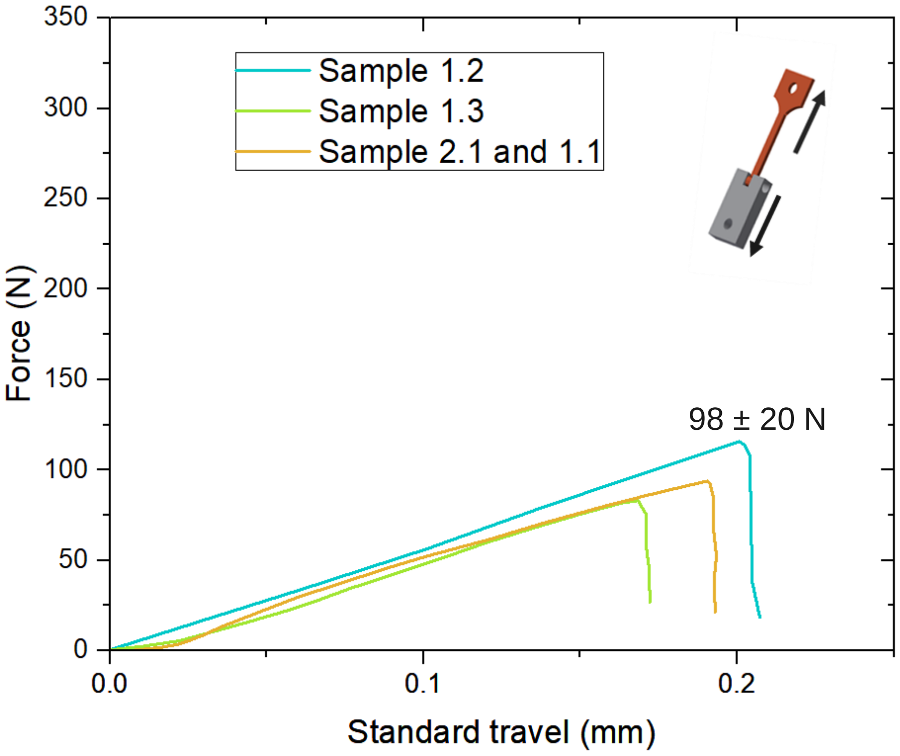
Tensile tests of the junction

Prototype 1- Starting point (10 hours)



Connection of
2 x 90 µm



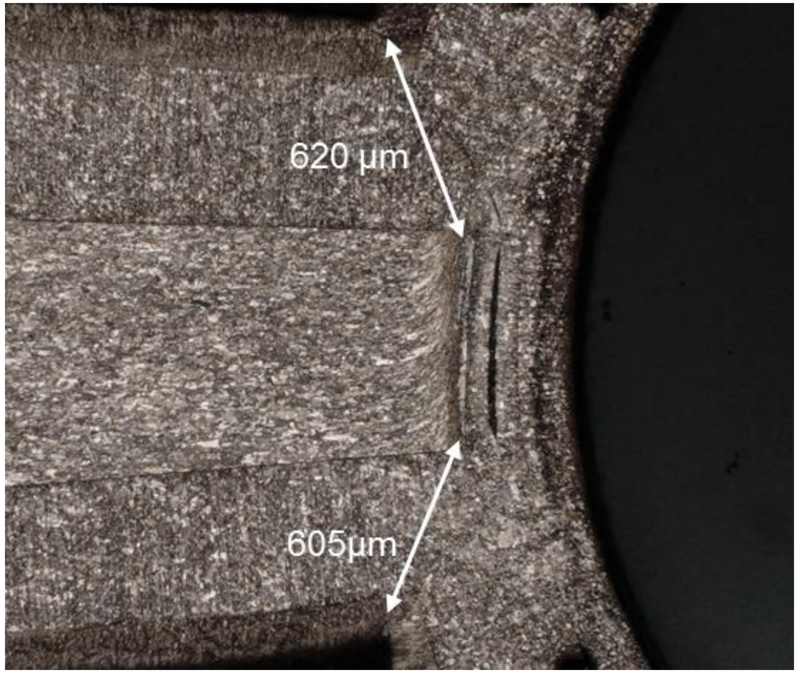


- Samples broke on the junction
- For a 34 cm stiffener, this translates on a max. load of 8000N.

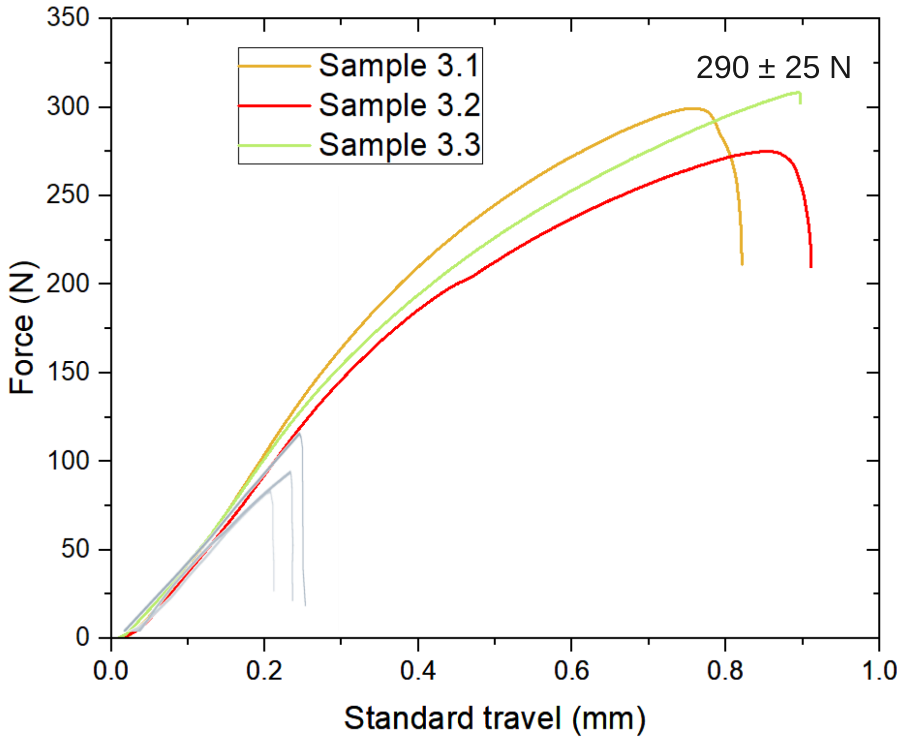
It holds the chamber

Tensile tests of the junction

Prototype 2 - Towards optimization (40 hours)



Connection of
2 x 612 µm



- Samples broke on the tube

Always for a thickness greater than 200µm (tube wall).

- **Triplicated max. load**: 24000N.

Strong connection

Main challenges

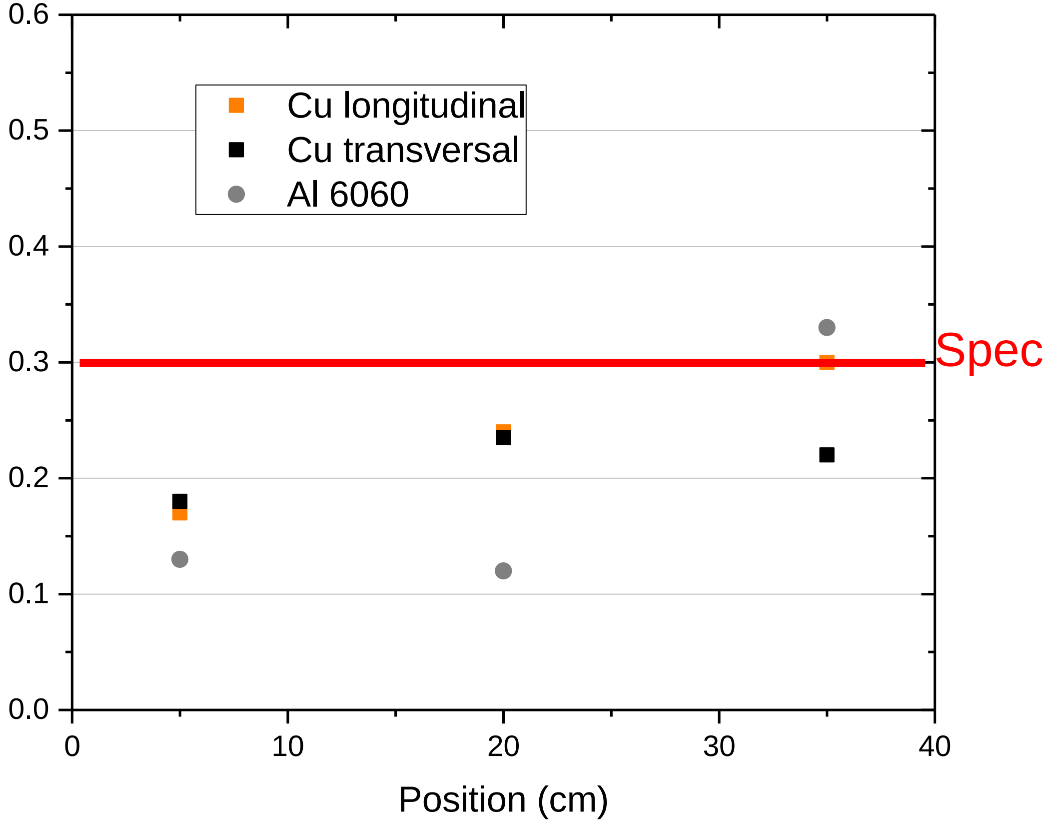
The stiffener-tube junction has to be mechanically strong.

The inner surface must guarantee a roughness of less than 0.3 µm over the length of the tube.

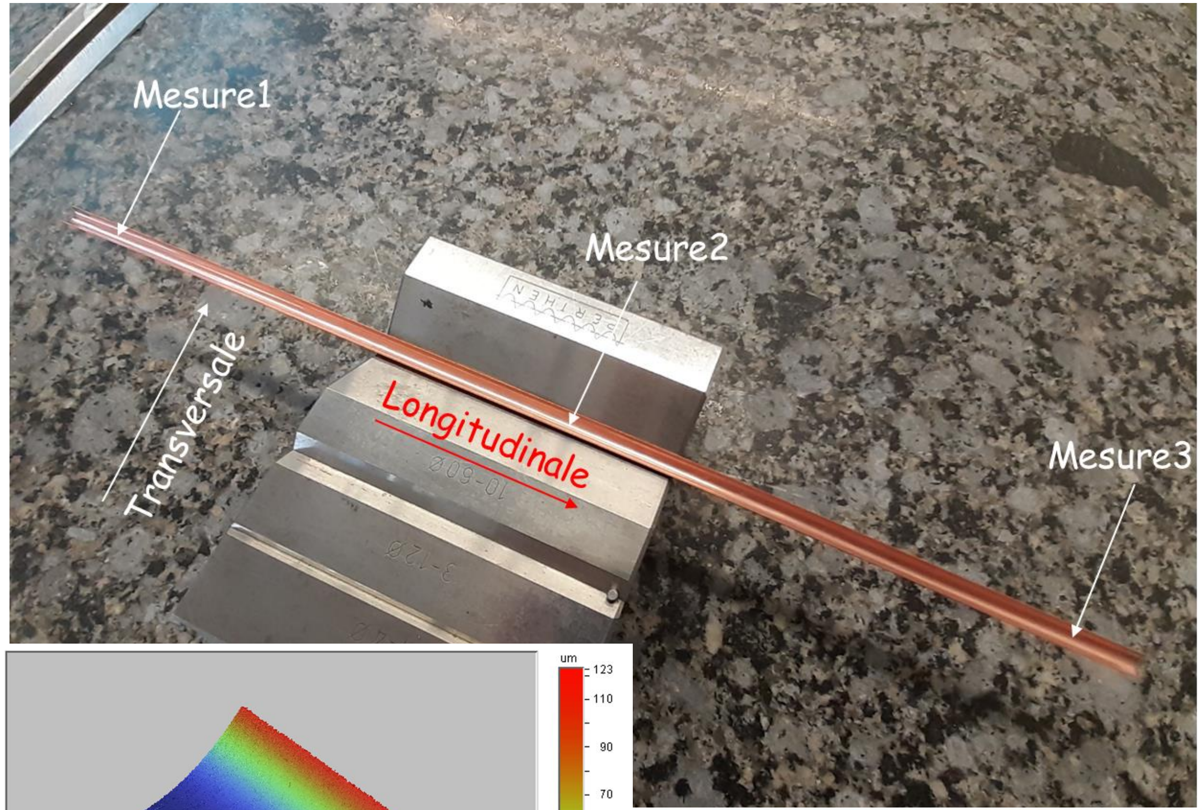
Roughness of inner copper tube surface

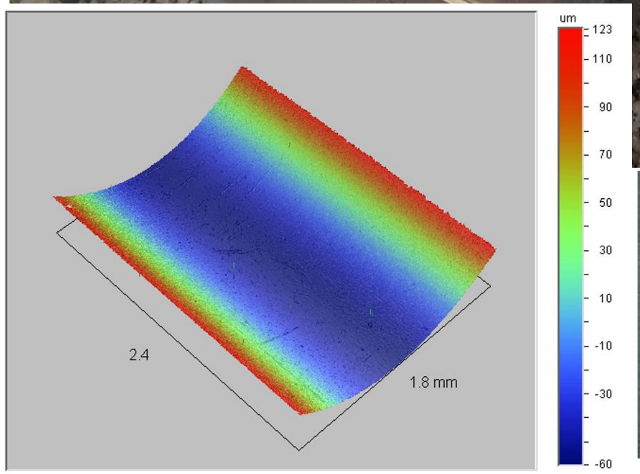
Measure on surface optical profiler (non-contact)

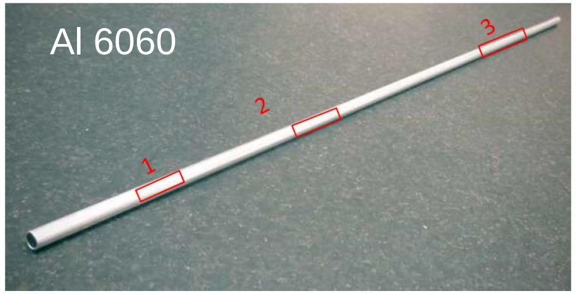
Roughness (Ra in µm)



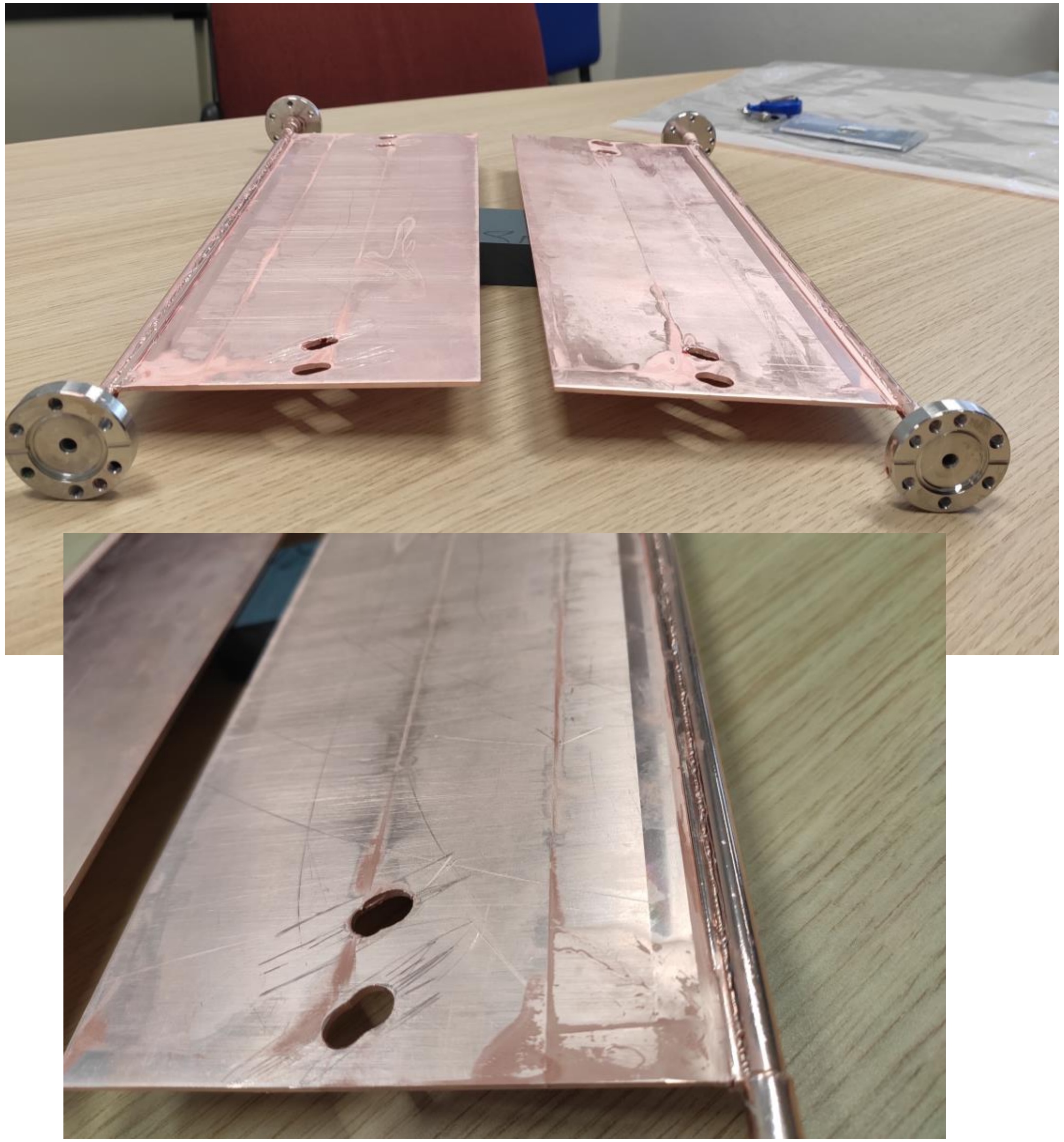
It replicates the roughness of the aluminium







Successful prototypes



Reproducibility

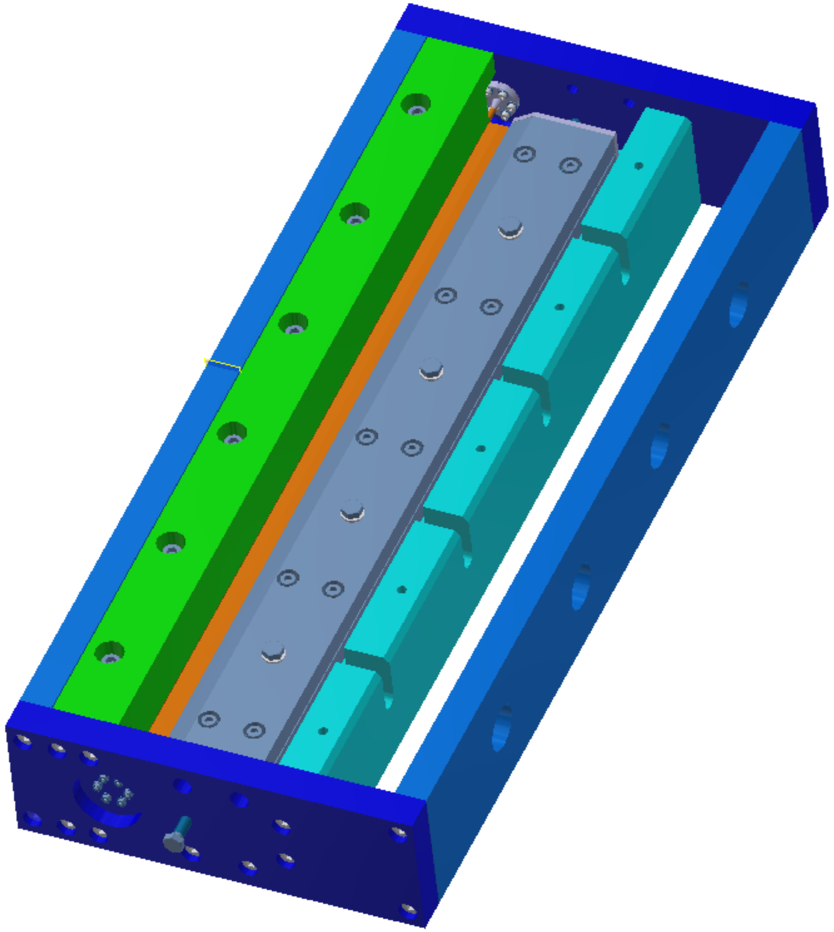
Several prototypes meet the specifications

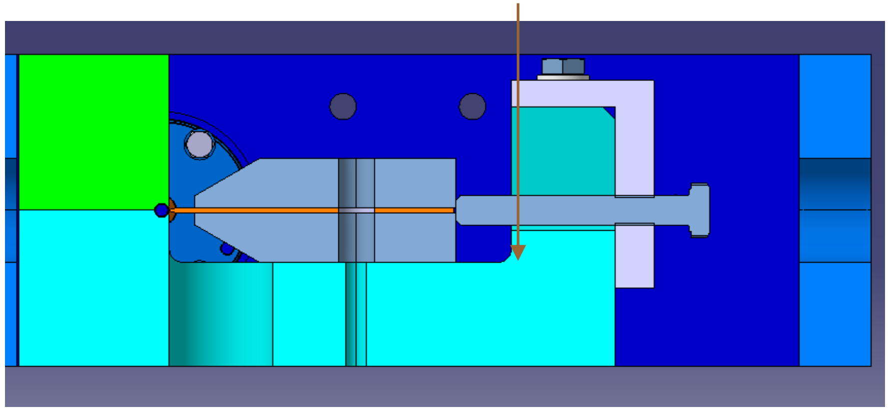
Strong connection

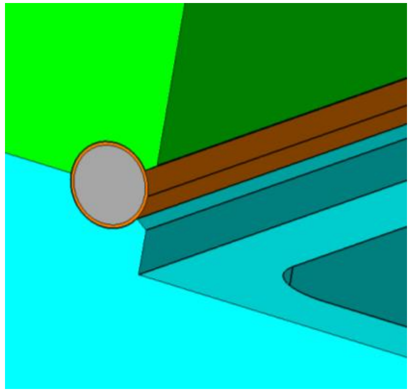
Wall thickness tube 200 µm

Smooth inner surface

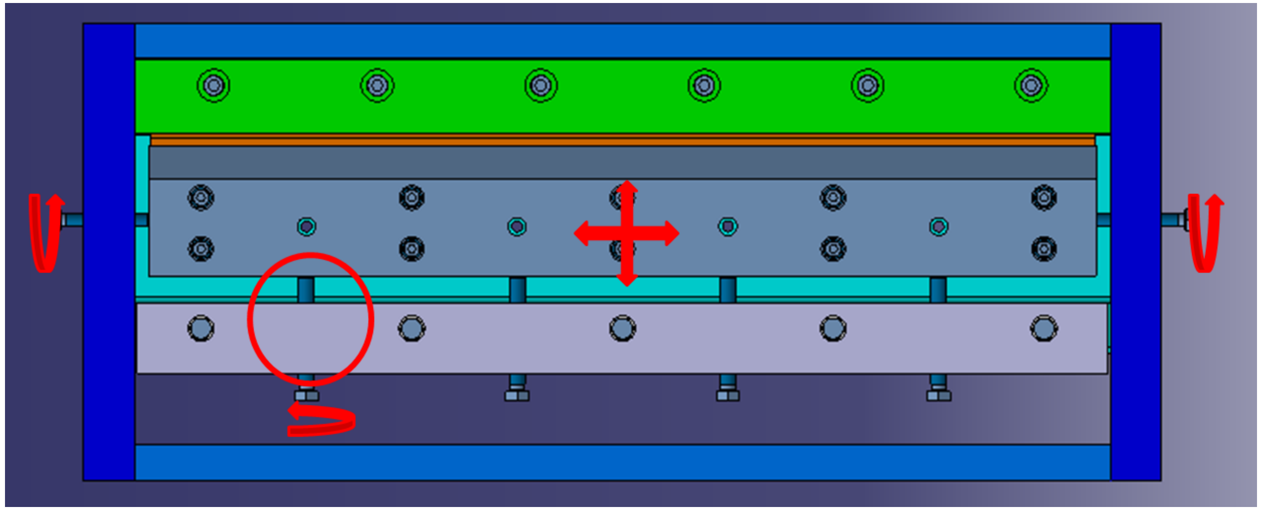
Towards meter-length chamber



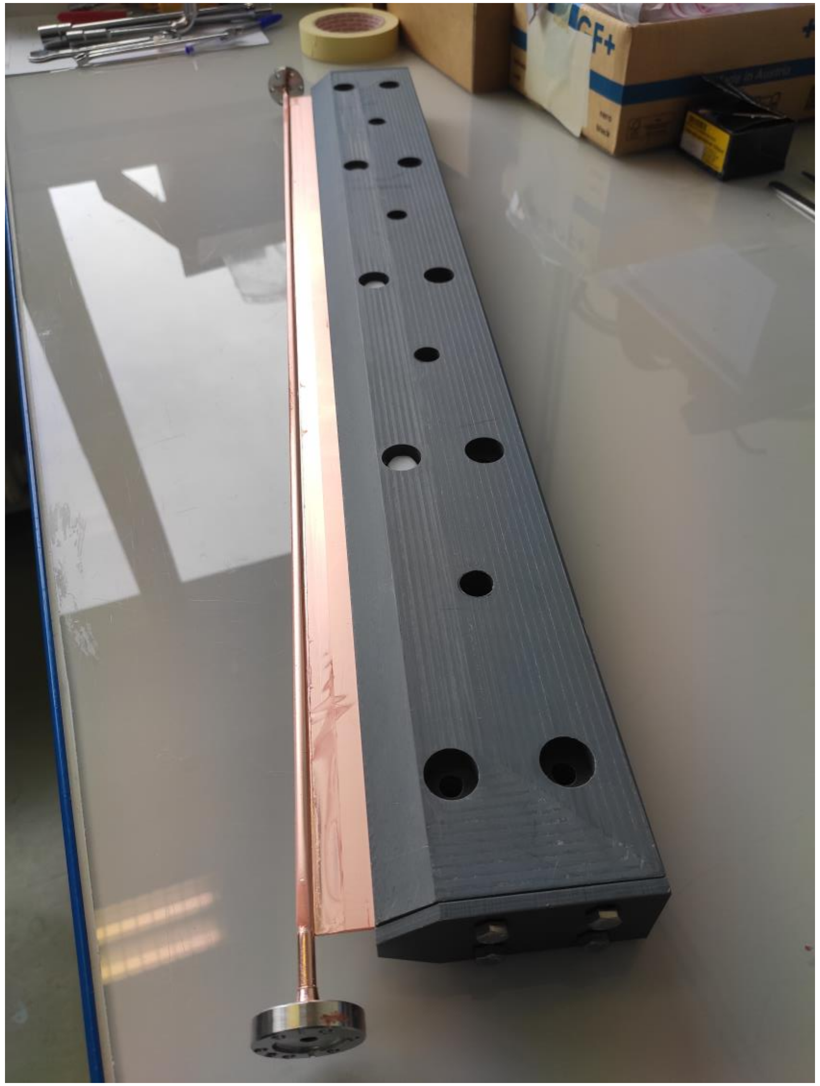


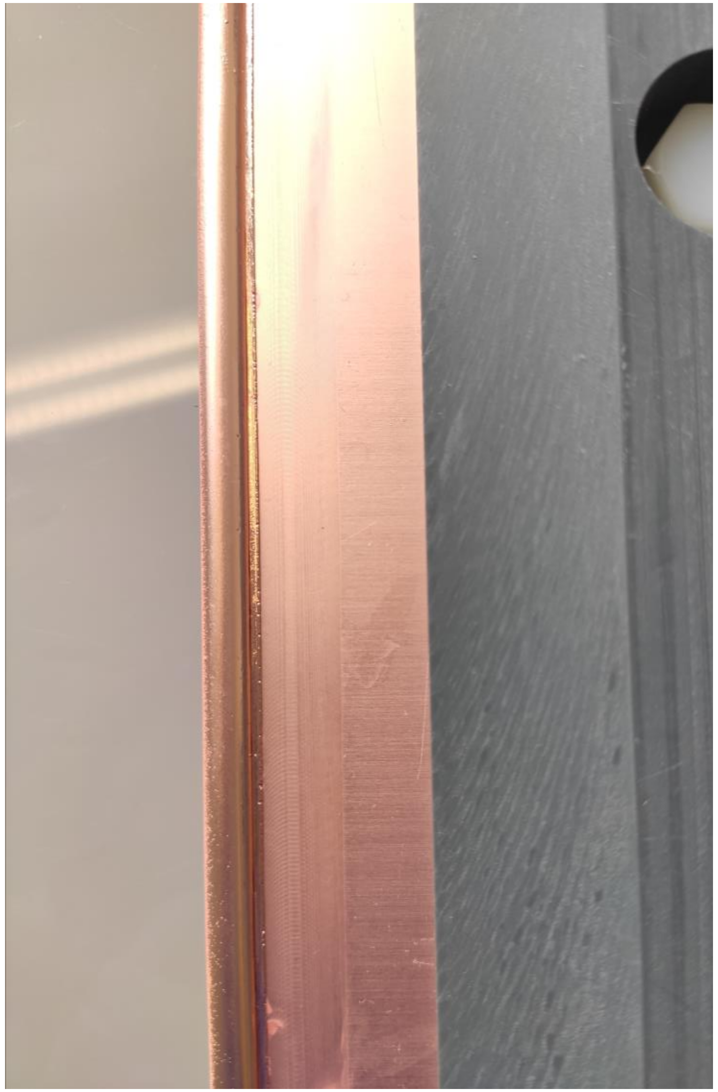


- Improved alignement stiffener-tube
- Improved masking



Thin-walled meter-length chamber





- Meter-length prototype succesfully produced
- List of measurements
  - Straigthness
  - Pump down
  - Bake-out
- Reproducibility?

Conclusions

- The feasibility of producing the thin-wall chambers, up to a meter, was demonstrated.
- The strength of the junction to the stiffener is large enough to hold and handle the chamber.
- The roughness of the inner surface is within specifications.

Perspectives

- Continue prototyping-campaign of 1 meter length chambers.
- Extend to 2 m length.
- Delivery of vacuum chambers.

Publications

- L. Lain Amador, P. Chiggiato, L. M.A Ferreira, V. Nistor, A. T. Perez Fontenla, M. Taborelli, W. Vollenberg, M-L Doche, J-Y Hihn, J. Vac. Sci. Technol. A, 36, 021601 (2018).

- L. Lain Amador, J Rolet, M-L Doche, P. Massuti-Ballester, M-P Gigandet, V. Moutarlier, M. Taborelli, L. M. A. Ferreira, P. Chiggiato, J-Y Hihn, ECS Transactions 85(13): 815-822 (2018).

- L. Lain Amador, J. Rolet, M-L Doche, P. Massuti-Ballester, M-P Gigandet, V. Moutarlier, M. Taborelli, L. M. A. Ferreira, P. Chiggiato, J-Y Hihn. Journal of The Electrochemical Society 166:10, D366-D373 (2019).

- L. Lain Amador, CERN-THESIS-2019-160, (2019)

- L. Lain Amador, L. M.A Ferreira, M. Taborelli, Proceedings of European COMSOL conference (2020).

- L. Lain Amador, P. Chiggiato, L. M.A Ferreira, E. Garcia-Tabares, T. Koettig, M.S. Meyer, A. T. Perez Fontenla, K. Puthran, G. Rosaz and M. Taborelli, Phys. Rev. Accel. Beams (submitted)

Thank you for your attention!