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

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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

standard

diamond

Cu coating by planar magnetron sputtering

Cu cathode

3 µm Cu
Electroforming process

Cu electroforming

Two copper sulphate-sulphuric acid baths

- Bath without additives
- Bath with brightener

Setup Schematic

Chemistry

Cathode (reduction):
\[ Cu^{2+} + 2e^- \rightarrow Cu \]

Anode (oxidation):
\[ Cu \rightarrow Cu^{2+} + 2e^- \]

- Electrodeposition of Cu, 2 A/dm²
  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)

<table>
<thead>
<tr>
<th></th>
<th>DC</th>
<th>PP</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>352 ± 41 MPa</td>
<td>174 ± 6 MPa</td>
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</table>

E modulus – impact excitation

<table>
<thead>
<tr>
<th></th>
<th>DC</th>
<th>PP</th>
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<tbody>
<tr>
<td></td>
<td>124 ± 15 GPa</td>
<td>131 ± 15 GPa</td>
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</table>
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.

\[
\lambda = \frac{Q L}{A \Delta T}
\]

- **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

<table>
<thead>
<tr>
<th></th>
<th>Standard mandrel machining (Ra 0.49 µm)</th>
<th>Diamond mandrel machining (Ra 0.002 µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ra (µm)</td>
<td>DC plated 0.39</td>
<td>Pulse Plated 0.65</td>
</tr>
<tr>
<td>Cu</td>
<td></td>
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</table>

Cu layer reproduces mandrel topography
Electroformed copper properties

- **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

More suited for:

- Thin-walled vacuum chambers
- SRF copper substrates

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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

- 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.

1) Preparation of Al mandrel  
2) Cu PVD thin film  
3) Cu electroforming and flanges assembly  
4) Mandrel etching

- 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

For the moment: Standard machining finishing
1.3 GHz cavity production

Cu PVD coating

2 µm Cu layer

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 successfully evaluated on electroformed cavity
COMSOL simulations for optimization

Thickness profile simulated with COMSOL

- 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

Thickening profile simulated with COMSOL

Without optimization

After optimization

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

Thickening also on iris

Thickening on equator

0       0.5      1       1.5      2       2.5       3      3.5       4      4.5       5  (mm)
Implementation of support
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

- 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

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

Cu cathode

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)

- 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

\[
2 \text{Al}(s) + 2 \text{NaOH}(aq) + 2 \text{H}_2\text{O}(aq) \rightarrow 2 \text{NaAlO}_2(aq) + 3 \text{H}_2(g)
\]
**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, assess RF performance.
4. Development of thin-walled copper electroformed vacuum chambers for undulators
Electroformed undulator vacuum chamber (SwissFEL)

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

UE38 Undulator

[2] H. Joehri at al., MEDSI 2018
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?
The chamber is produced by copper electroforming around a sacrificial aluminium mandrel which has the internal shape of the vacuum chamber.

Starting point: 400 mm long chamber

Goal: 2 m length

1. Preparation of Al mandrel

- Ø 5mm
- Cu PVD thin film

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 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 brightener bath

6 hours plating

Direct Plating

Cathode (reduction):
\[ \text{Cu}^{2+} + 2e^- \rightarrow \text{Cu} \]

Anode (oxidation):
\[ \text{Cu} \rightarrow \text{Cu}^{2+} + 2e^- \]
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)

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

Connection of 2 x 90 µm
Tensile tests of the junction

Prototype 2 - Towards optimization (40 hours)

- Samples broke on the tube always for a thickness greater than 200µm (tube wall).
- Triplicated max. load: 24000N.

Connection of 2 x 612 µm
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)

- Cu longitudinal
- Cu transversal
- Al 6060

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 alignment stiffener-tube
- Improved masking
Thin-walled meter-length chamber

- Meter-length prototype successfully produced
- List of measurements
  - Straightness
  - 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


Thank you for your attention!