Cu coating conductivity measurements

WP2/WP5 meeting

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Outline

- Measurement technique (H011 cavity)
  - Sputtering: DCMS vs HiPIMS
  - Cleaning & Positioning → Impact of thickness
  - Measurements on the blocks
- Conclusions and further studies

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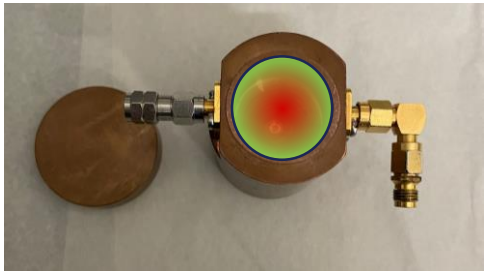
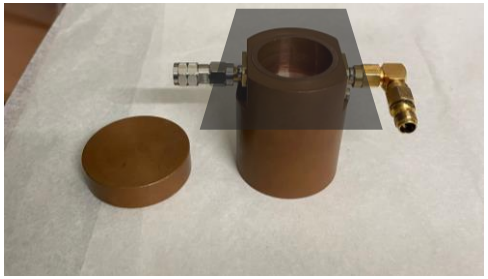
Measurement technique

- Measure the conductivity of coating only (i.e. the EM field must penetrate less than the coating thickness).





- H_{011} mode cavity.
- Resonating @ ~16.5 GHz
  - Skin depth of Cu ~0.5 μm
  - Sensitive to surface roughness
- Measuring the Q factor…
- Resistivity / Conductivity value

Calibration procedure using known conductivity samples

C. Accettura, et al. «Resistivity Characterization of Molybdenum-Coated Graphite-Based Substrates for High-Luminosity LHC Collimators». *Coatings*, vol. 10, fasc. 4, aprile 2020, p. 361. *DOI.org (Crossref)*, https://doi.org/10.3390/coatings10040361.

The effect of surface roughness

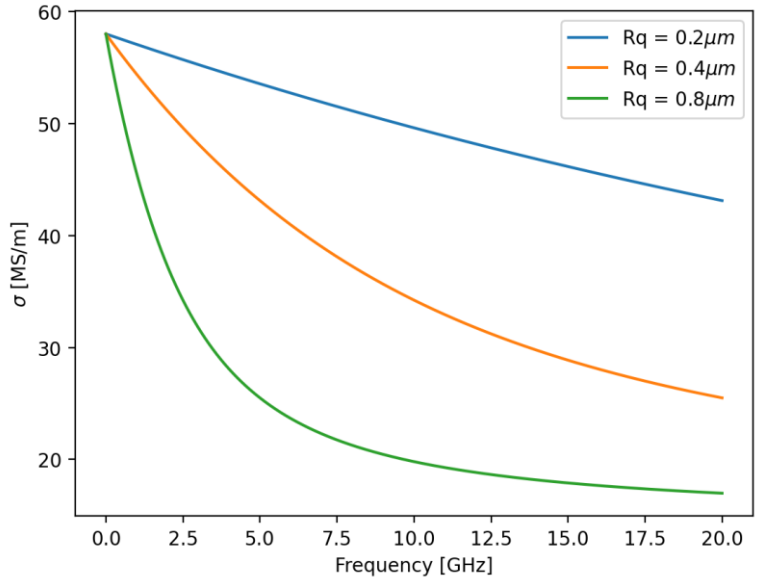
Draft

Surface roughness impacts studied with semi-empirical approach (**Hammerstad model**)

$$\sigma_{eff} = \frac{\pi f \mu}{R_s^2}$$

$$R_s = \sqrt{\frac{\mu\omega}{2\sigma}}\left(1 + \frac{2}{\pi}\mathrm{arctg}(0.7\mu\omega\sigma R_Q^2)\right)$$

Root mean square value of the surface height variation



C. Zannini, Update of TL wall with inclusion of roughness (Here)

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DCMS VS HiPIMS

- Investigation on two magnetron sputtering techniques for producing the coatings (direct current and high-power impulse (70 and 130 V)).
- Tests done on both **Cu on Glass** and **Cu on Graphite**.

Cu on Graphite

ID	Coating Procedure	$\sigma\ [MS/m]$ (Single Spot)	$\sigma\ [MS/m]$ (Avg on surface)
1	DC - centre	18.36 +/- 0.61	16.87 +/- 4.00
2	DC – side	12.80 +/- 0.27	15.13 +/- 1.30
3	HiPIMS – centre	29.38 +/- 0.31	35.02 +/- 7.23
4	HiPIMS – side	39.10 +/- 6.64	34.47 +/- 5.54

- **HiPIMS 130 V** gives consistently a higher conductivity.
  - Consistent with previous measurements of Mo on Graphite : https://indico.cern.ch/event/883715/contributions/3723861/attachments/1995848/3329786/Colusm_28022020_NB.pdf

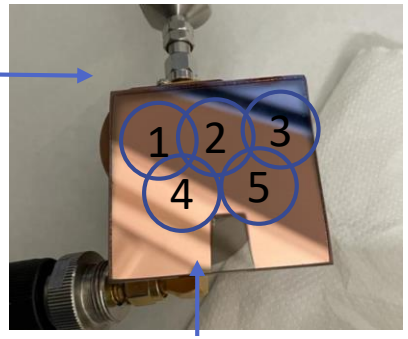
Bulk	Mo coating	DC	ECT	RF
MoGr	DCMS	120 ± 64	440 ± 80	470 ± 37
	HIPIMS	47 ± 21	68 ± 4	61 ± 2
Graphite	DCMS	410 ± 140	628 ± 5	679 ± 41
	HIPIMS	47 ± 17	145 ± 2	112 ± 1

Resistivity values in $n\Omega \cdot m$

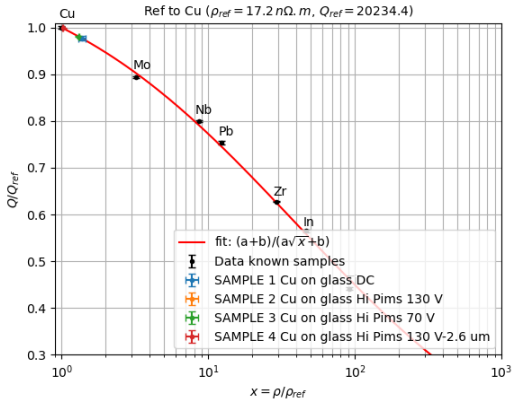
The case of Cu on Glass

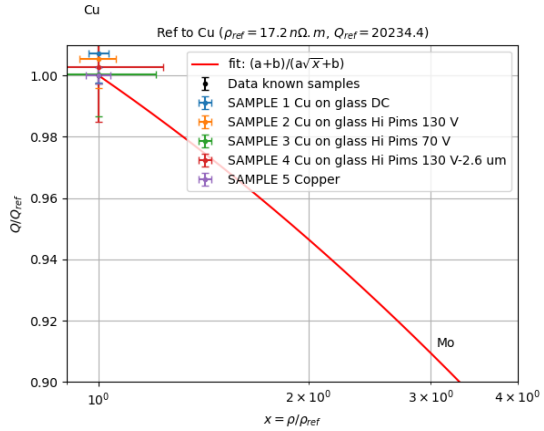
- When investigating DCMS vs HiPIMS → 4 samples of Cu on Glass provided.

ID	Coating Procedure	$\sigma\ [MS/m]$ (Single Spot)	$\sigma\ [MS/m]$ (Avg. on Surface)
S1	DCMS	42.37 +/- 2.42	58.00 +/- 1.90
S2	HiPIMS 130 V	58.00 +/- 0.00	58.00 +/- 3.49
S3	HiPIMS 70 V	44.39 +/- 0.46	58.00 +/- 12.01
S4	HiPIMS 130 V-2.6 um	57.13 +/- 0.72	58.00 +/- 13.80



Five Q values each taken with a different position of the sample on the cavity.





- Avg. conductivity above the calibration Cu sample
  Might be an indication that the roughness of the surface is playing a significant role (@16.5 GHz).

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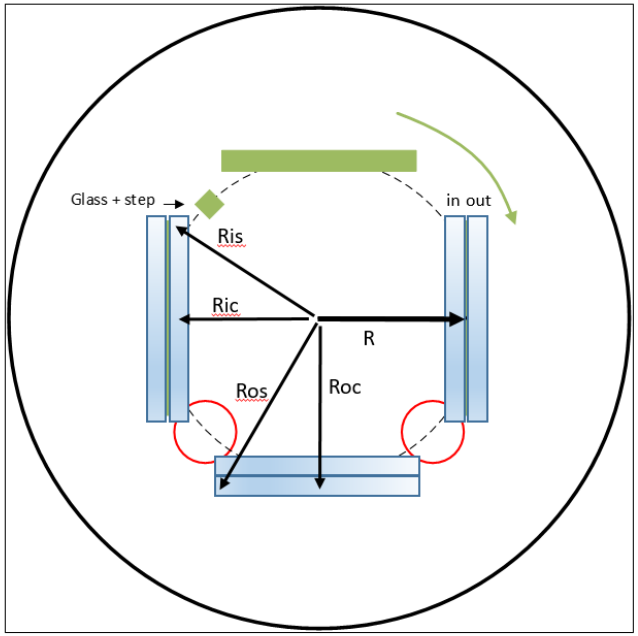
Positioning

Courtesy of Wil Vollenberg

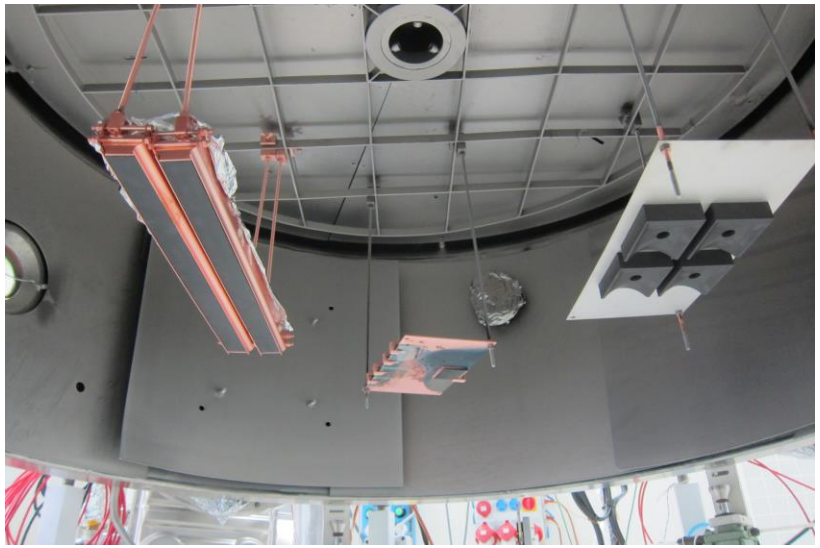
TE-VSC-SCC

For **optimizing production**: loading of 8 blocks of $50\ cm$.



Possible loading of 8 blocks

Ti / Cu cathode



In this configuration, the radius change is $27 - 42\ cm$

The **position** of the blocks in the oven **affects** the ultimate **coating thickness**.

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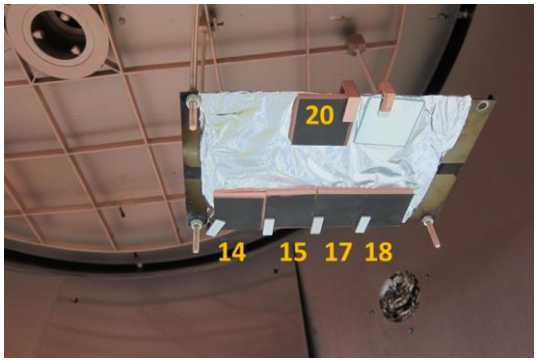
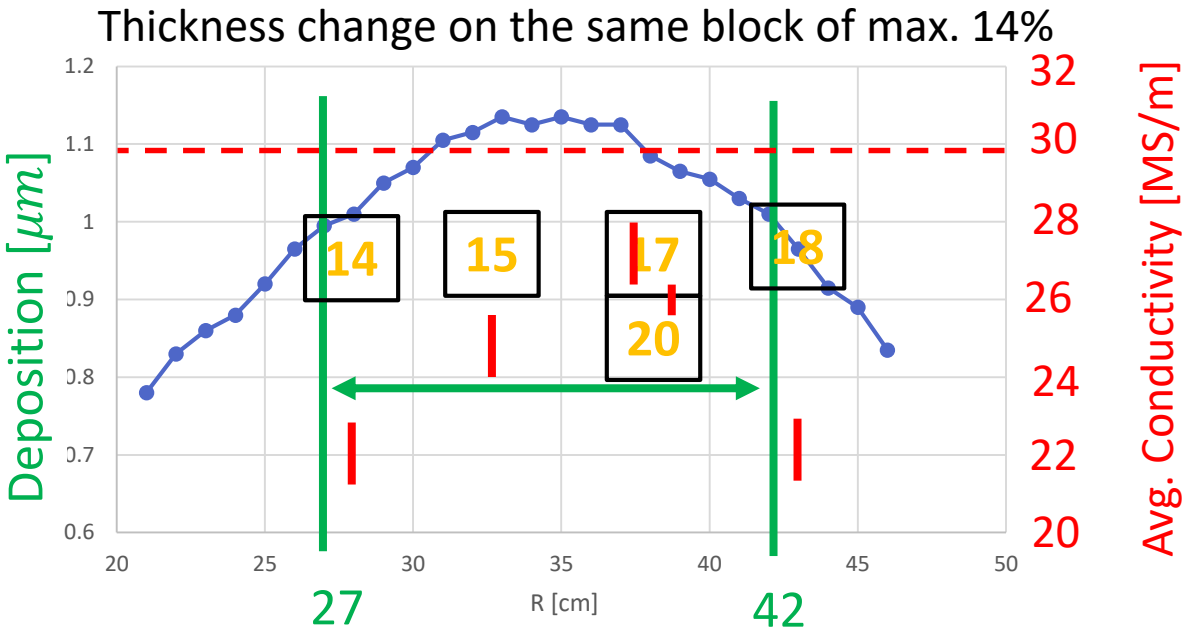
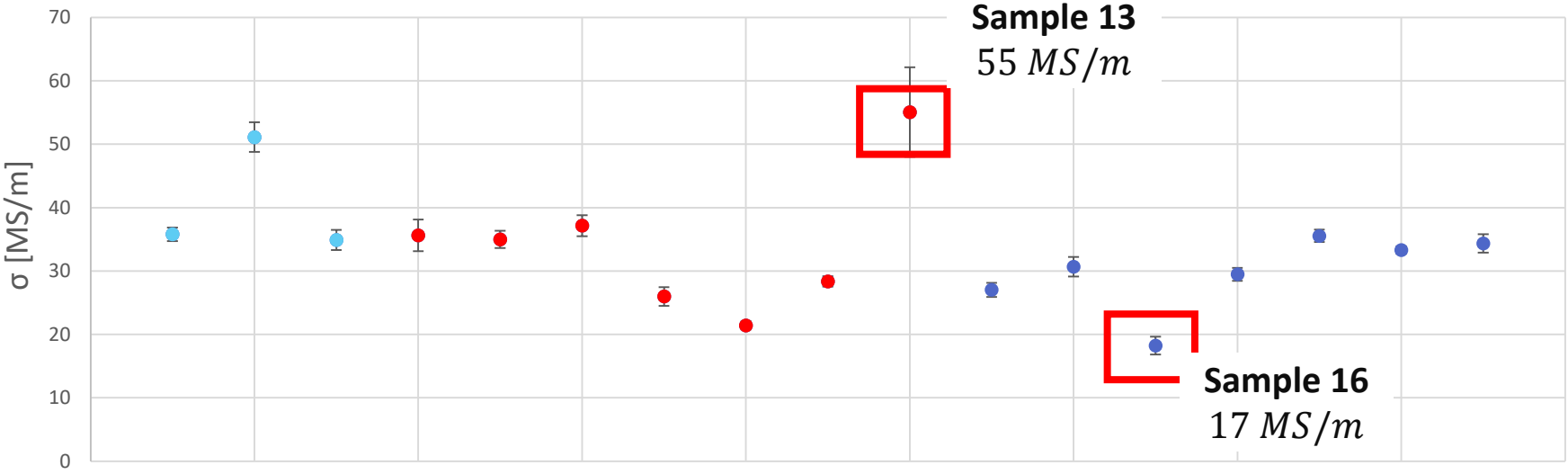




- Conductivity values are relatively **lower** → Adhesion layer is Nb and not Ti
- The conductivity seems to follow same trend as thickness
- However:
  - Coating thickness is lower ($\sim 2.5\ \mu m$) but still enough for the field to decay
  - The same trend is not shown in the measured blocks.

Cleaning

Three cleaning procedures tested on 20 samples. **Firbimatic chosen**.



U.S. with propanol

38.5 ± 7.2 $[MS/m]$

Not allowed for blocks

U.S. with detergent cleaning

38.3 ± 12 $[MS/m]$

Firbimatic + U.S. cleaning in demineralized water

30.0 ± 5.7 $[MS/m]$

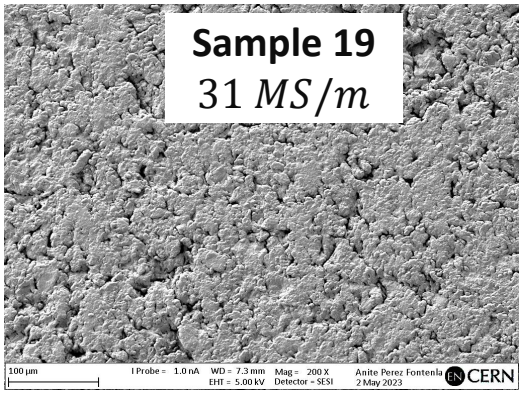
- Conductivity not significantly worse (see outliers)
- Well established process
- Samples with different positions and angles with the target

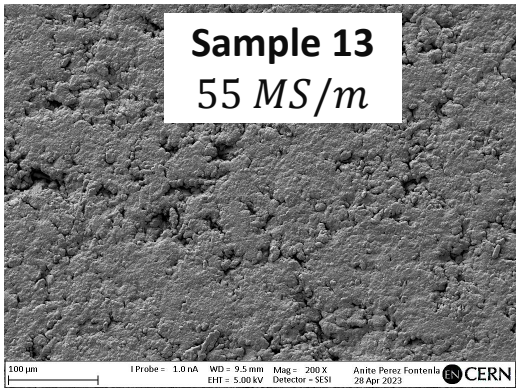
Courtesy of
Carlotta Accettura

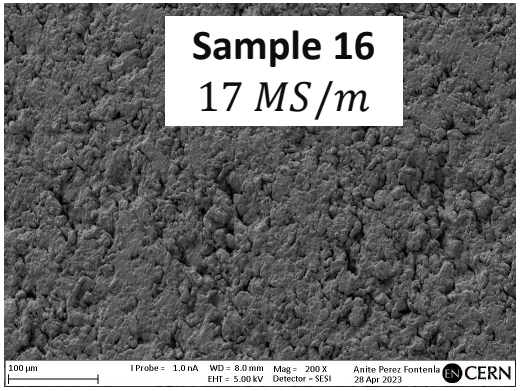
EN-MME-EDS

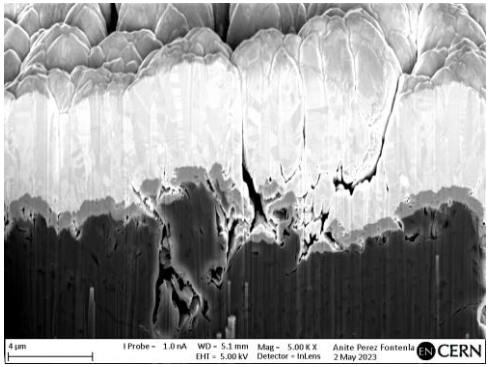
Microscopy

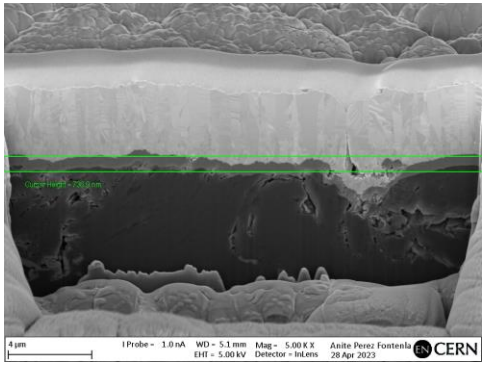
More than 20 samples measured. High variability of the conductivity of the coating.
Investigation on significant samples (close to avg, max and min).

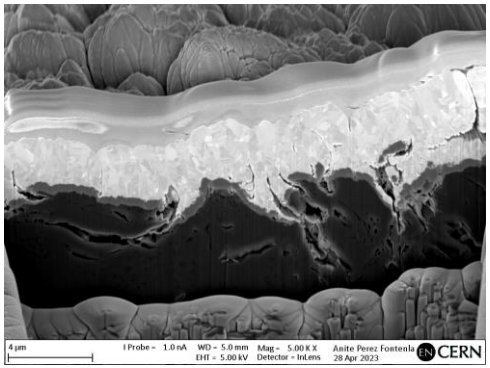












- Variation between #19 and #13 could be related to local inhomogeneities.
- Cu coating on sample #16 has lower thickness and grain size smaller, less columnar.

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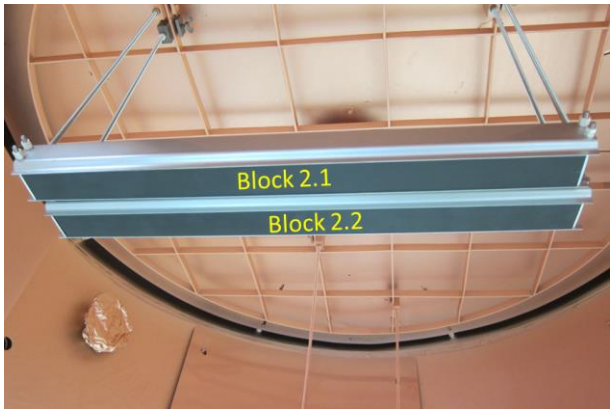
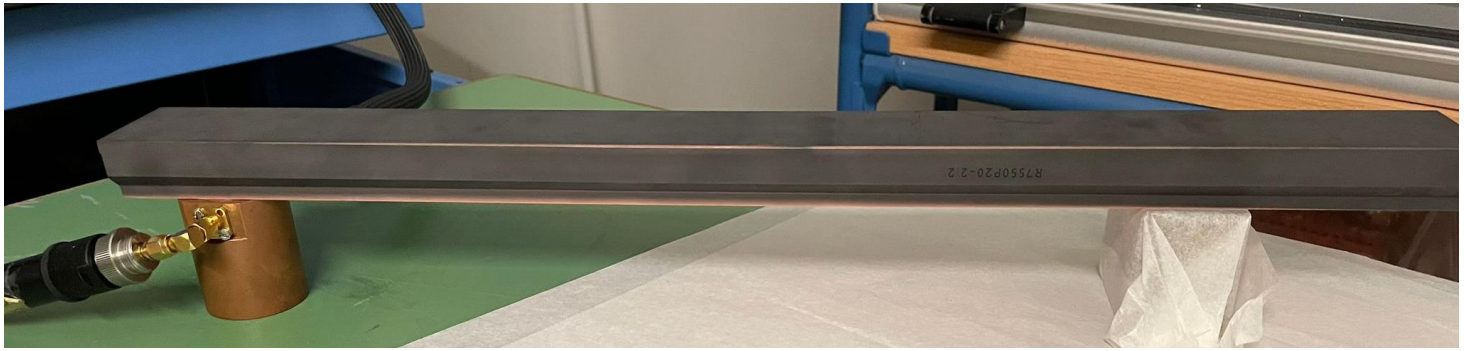
Blocks

Two Blocks measured before UHV tests**:**

- 2.1: outside
- 2.2: inside



- Blocks placed upside-down on the cavity: **minimize moving the cabling**.
- Effort to keep everything **vacuum compliant**.



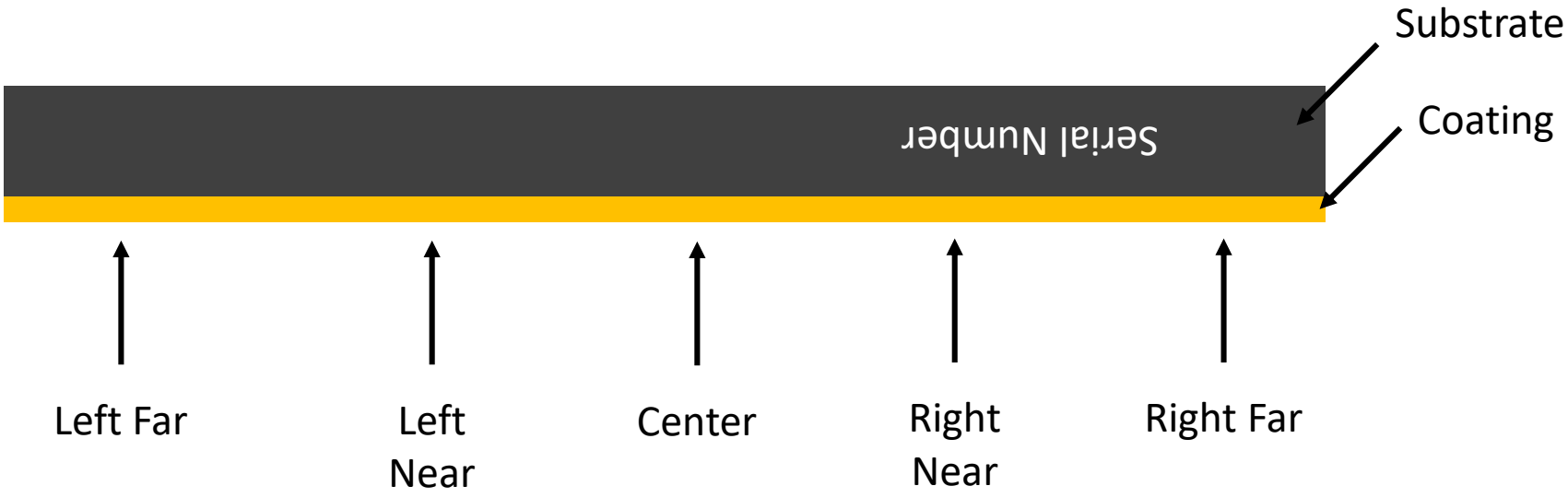
1. Difficult to measure the blocks without the **risk of damaging** them
2. **Different pressures** applied (the cavity is "closed" slightly differently)

Blocks

Each block measured in **5 points** (5 measurements taken for each point):

- **Left far**
- **Left near**
- **Center**
- **Right near**
- **Right far**

Near or far from the center of the block



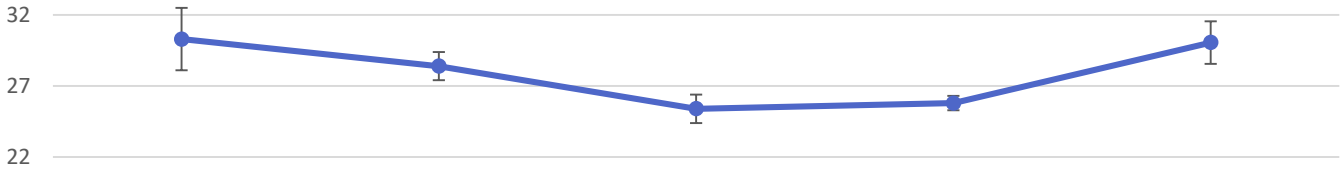
Blocks

Serial Number

2.1

ID	Left Far	Left Near	Center (34cm)	Right Near (36cm)	Right Far (42cm)
$\sigma\ [MS/m]$	30.3 +/- 2.2	28.4 +/- 1.0	25.4 +/- 1.0	25.8 +/- 0.5	30.6 +/- 1.5

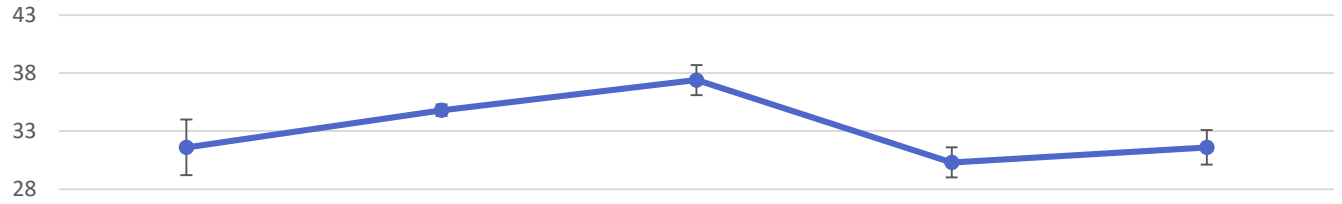
Avg. 28.10 [MS/m]



2.2

ID	Left Far	Left Near	Center (29cm)	Right Near (32cm)	Right Far (38cm)
$\sigma\ [MS/m]$	31.6 +/- 2.4	34.8 +/- 0.5	37.4 +/- 1.3	30.3 +/- 1.3	31.6 +/- 1.5

Avg. 33.15 [MS/m]



Opposite trends with the position (radius) in the oven.
This behavior might be explained by a surface roughness due to substrate inhomogeneities.

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Summary

- Investigation on:
  - Sputtering type → HiPIMS 130V
  - Position and cleaning → Optimization of the production process

- Measurements of the first Cu coated blocks

  - Measured conductibility around $\sim 30\ MS/m$ (with variability).

    - Still not fully clear the dependence with positioning.
    - Impact of surface roughness to further study, it may explain the discrepancies.

Due to measurement @ $16.5\ GHz$

Further work

- Measuring of the blocks after UHV treatment.
- Measuring of two more blocks (ready since 26/06):
  - Input from Wil: should the blocks be exposed to air?
- Measuring of glass samples → Further study on the positioning in the oven

- Measuring at different frequency points → Further study on the surface roughness
  - Maybe loading the cavity with ferrite.

Thank you for the attention

Cu coating conductivity measurements

Chiara Antuono & Leonardo Sito (BE–ABP–CEI)