Cu-coated graphite TCSPMs - overview of tests and planning

C. Accettura

With contribution of C. Antuono, G. Cattenoz, L. Gentini, L. Giacomel, D. Glaude, L. Hannemann, N. Mounet, FX Nuiry, D. Pugnat L. Sito, W. Vollenberg

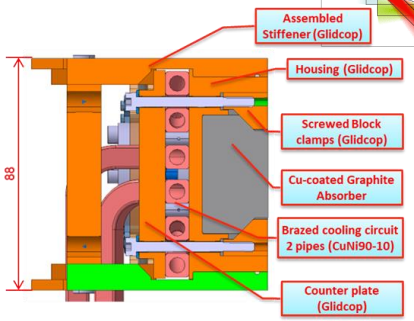
Special Joint WP2/WP5 meeting

CERN, Geneva

June, 27th 2023

Why graphite with copper?

- Absorbers for **12 TCSPM** collimators should be produced during LS3 (see LHC-TC-ER-0006 v.1)
- Technical material assessment of studied options summarized in the collimator's jaw's materials table LHC-TC-ER-0008 v.1



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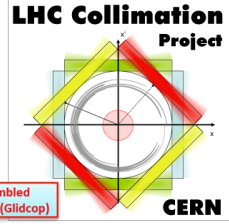

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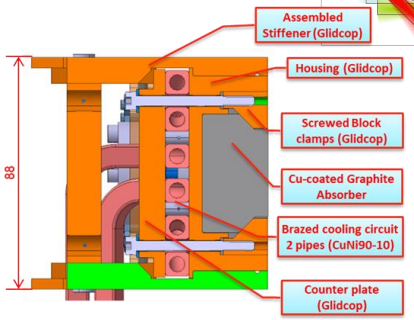
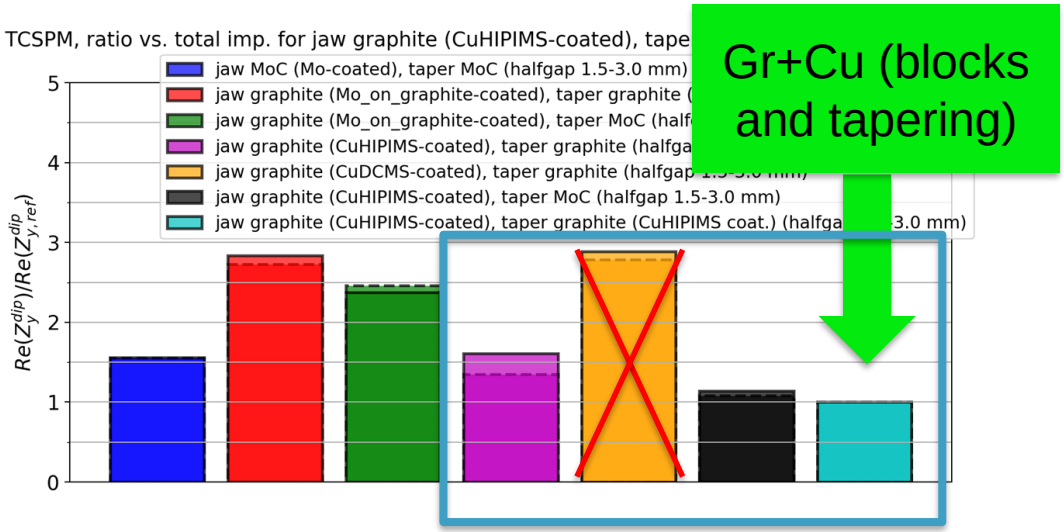


- Graphite with 3 µm Cu coating selected at the WP5 PSM on 12/07/2022 for the blocks (see F.-X. Nuiry, "Jaw materials option for LS3 collimators", 162nd HL-LHC TCC , 14th September 2022)
  - Good HiRadMat results (Multimat 2) (see J. Guardia, 'Report on MultiMAT2', ColUSM#153, 2nd September 2022)
  - Cheaper (see F.-X. Nuiry, "Jaw materials option for LS3 collimators", 162nd HL-LHC TCC , 14th September 2022)
  - Good solution for impedance (see N. Mounet, "Impedance consideration in material master table", WP5.2 Technical Meeting, 19th September 2022)
    - Good indication from electrical conductivity measurement (See A. Kurtulus, "Update on Resistivity Measurements Coated Collimators and Control Procedure", EDMS 2735237)

Why graphite with copper?

- It was recently pointed out that the **tapering** contribution to impedance is not negligible→ preferred solution graphite with copper coating



Activities overview

Optimization of tests and procedures for Cu-coated graphite in view of the series production

Preparation of the series production (offer request, planning of activities at CERN)

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Order of a small pre-series (6 blocks, 20 plates)

- Follow-up and reporting:
- WP5.2 #28
- WP5.2 #37
- WP5.2 #38

Activities overview

Optimization of tests and procedures for Cu-coated graphite in view of the series production

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OBJECTIVES

*proposal to increase the length to 500mm to easy assembly

METROLOGY

Verify achievable tolerances on long block*

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Verify achievable tolerances on long block*

**test a cleaning procedure suitable for series production

UHV

Verify UHV compatibility before and after coating**

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Select a high-el. conductivity coating procedure

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METROLOGY	Verify achievable tolerances on long block*
UHV	Verify UHV compatibility before and after coating**
IMPEDANCE	Select a high-el. conductivity coating procedure
IMPEDANCE	Prove the repeatability of the el. conductivity achieved

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IMPEDANCE

Replicate a high-el. Conductivity coating on blocks and tapering

IMPEDANCE

Select a high-el. conductivity coating procedure

IMPEDANCE

Prove the repeatability of the el. conductivity achieved

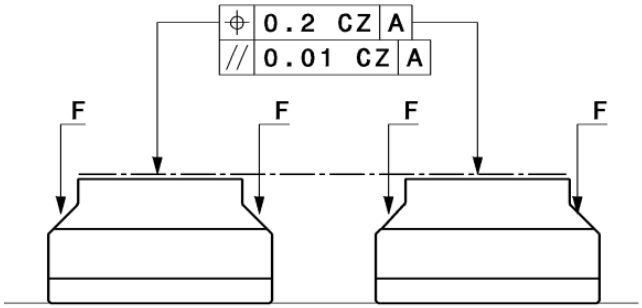
Verify achievable tolerances on long block*

Metrology

- Summary of the results:

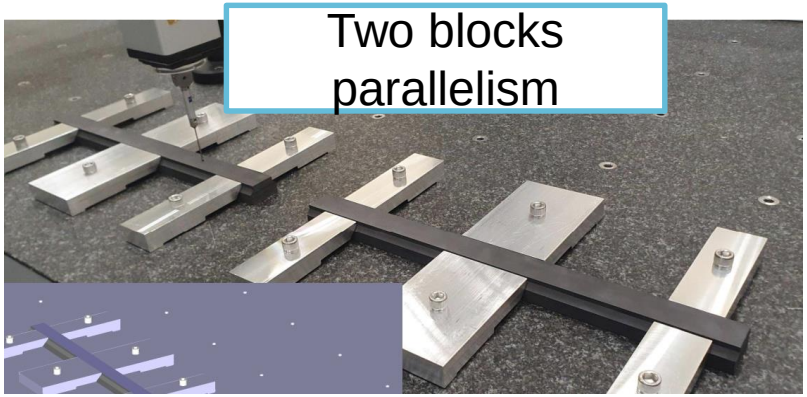
✓ Required parallelism achieved on the blocks

✓ Coherence with the measurement done at the enterprises



SGL

	measurement 2x500			
block	flatness	position	height	parallelism
1.1	0.009	0.048	24.976	0.0114
1.2				
2.1	0.01	0.0432	24.978	0.0121
2.2				
3.1	0.009	0.0523	24.944	0.0119
3.2				



CERN

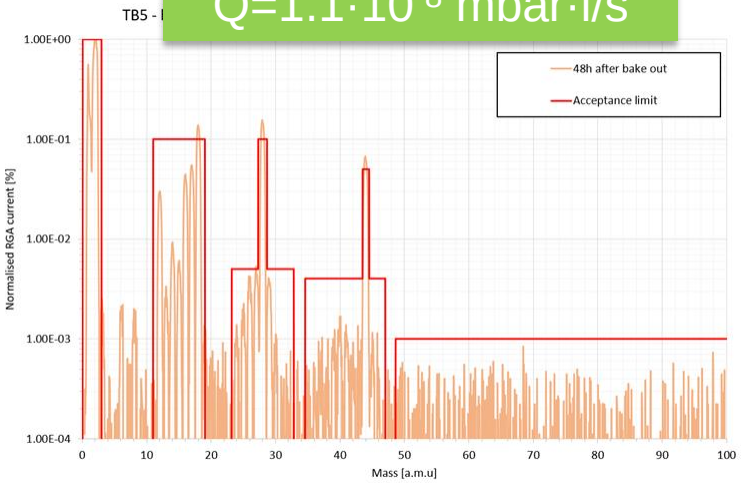
	measurement 2x500			
block	flatness	position	height	parallelism
1.1	0.004	0.042	24.979	0.009
1.2				
2.1	0.004	0.04	24.98	0.009
2.2				
3.1	0.004	0.041	24.979	0.008
3.2				

Verify UHV compatibility before and after coating**

Outgassing test

- Summary of the results:
- First test after cleaning (Firbimatic+US demineralized water)and 16h at 950°C interrupted before bake-out because a contamination was detected→ RGA shows a signal compatible with the detergent used during the cleaning
- An additional thermal treatment of 56h was performed
- ✓ UHV test of 4 cleaned blocks **ok** after a total of 72h hour of treatment
- ✓ UHV test of 2 cleaned and coated blocks **ok** (report under preparation)

$Q=1.1\cdot10^{-8}$ mbar·l/s



Courtesy of G. Cattenoz

Select a high-el. conductivity coating procedure

Which coating process?

- HIPIMS generally better that DCMS, but DCMS faster and well mastered at CERN→ test to discard this option on 4 Gr plates
- HIPIMS conductivity (**34 MS/m**) 2 times higher than DCMS→ go on with the HIPIMS

Comparison with previous measurements

- Example of Mo on Graphite from previous measurements:
  https://indico.cern.ch/event/883715/contributions/3723861/attachments/1995848/3329786/Colusm_28022020_NB.pdf

Resistivity values in $n\Omega \cdot m$

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

HiPims shows lower resistivity values compared to DCMS

		NOT MOVING		MOVING	
	ID	$\rho\ [n\Omega \cdot m]$	$\sigma\ [MS/m]$	$\rho\ [n\Omega \cdot m]$	$\sigma\ [MS/m]$
DCMS	1	54.48 +/- 1.82	18.36 +/- 0.61	59.29 +/- 14.00	16.87 +/- 4.00
	2	78.17 +/- 1.68	12.80 +/- 0.27	66.11 +/- 5.67	15.13 +/- 1.30
HiPims	3	34.03 +/- 0.36	29.38 +/- 0.31	28.56 +/- 5.89	35.02 +/- 7.23
	4	25.58 +/- 4.34	39.10 +/- 6.64	29.01 +/- 4.67	34.47 +/- 5.54

Same trend observed in the measurements of Copper on Graphite.

See next talk of L. Sito for measurement details!

Courtesy of C. Antuono, L. Sito, A.Kurtulus, N. Biancacci, N. Mounet, B. Salvant

Prove the repeatability of the el. conductivity achieved

Is it reproducible?

- HIPIMS coating test on 20 Gr plates:
  - Different cleaning
  - Different orientation to mimic the tapering surface
  - Different positions
- Average conductivity 34MS/m, but high scattering (min 17MS/m – max 55MS/m) and *poor statistical analysis* to address causes

See next talk of L. Sito for measurement details!

Prove the repeatability of the el. conductivity achieved

Is it reproducible?

- From where does it come the difference?
  - Position
  - Orientation
  - Cleaning
  - Material

Prove the repeatability of the el. conductivity achieved

Is it reproducible?

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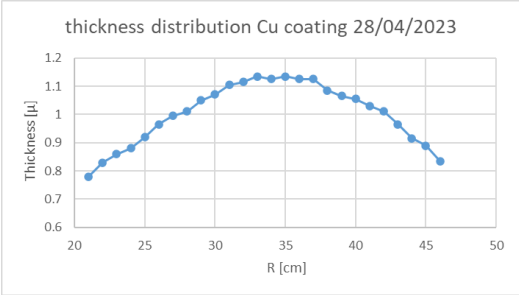
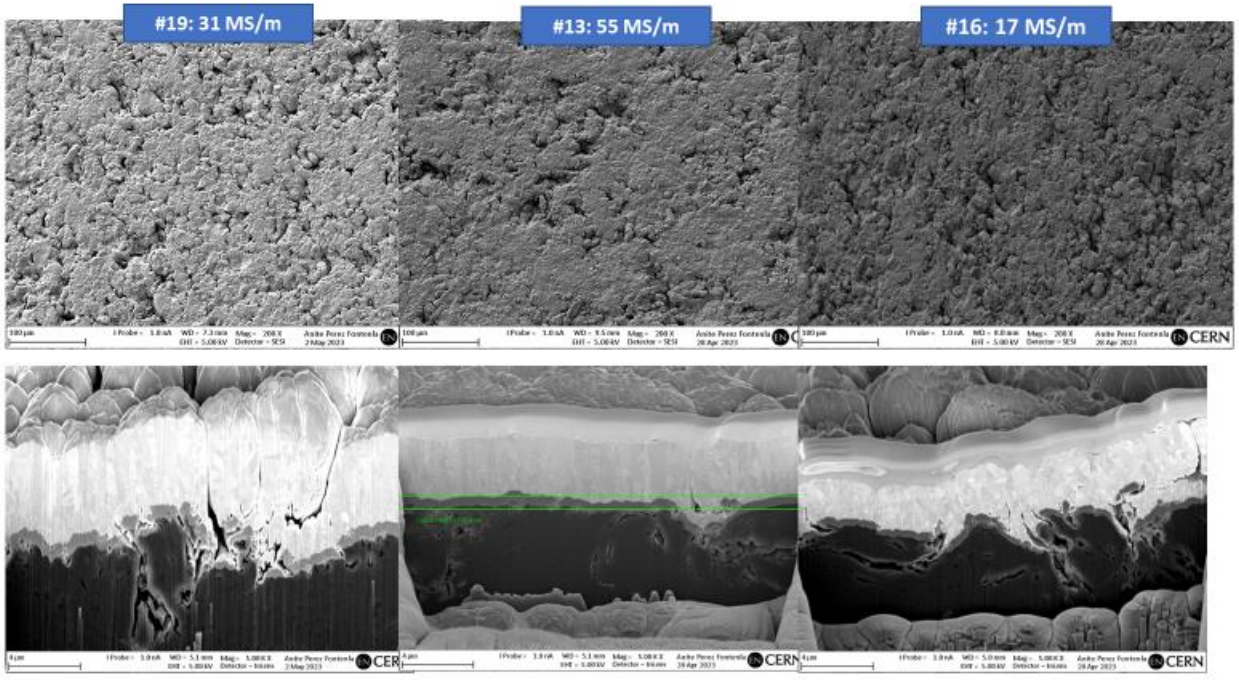
1-Test on glass:
→ thickness as a function of position



2-Microscopy:
→ Grain size/columnar structure function of the thickness
→ Lower thickness in the low-conductivity sample



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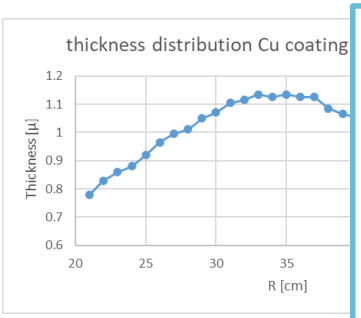

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→ Grain size/columnar structure function of the thickness
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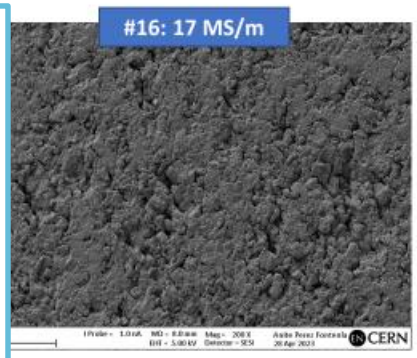
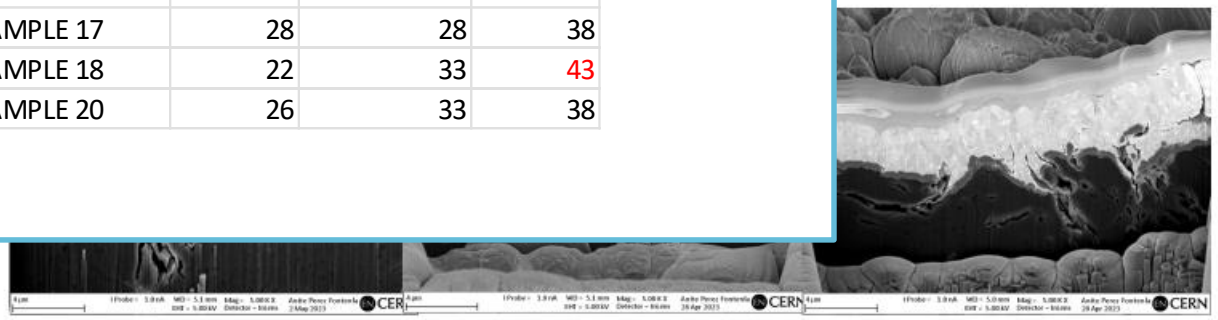
3-Repeat a test only on flat samples:
→ No influence of the cleaning (same sample -50%)
→ Influence of the position

	Conductivity [MS/m]		
Sample ID	run 3	run 2	R [cm]
SAMPLE 14	22	31	28
SAMPLE 15	26	27	33
SAMPLE 17	28	28	38
SAMPLE 18	22	33	43
SAMPLE 20	26	33	38





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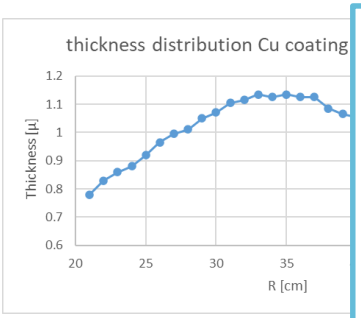

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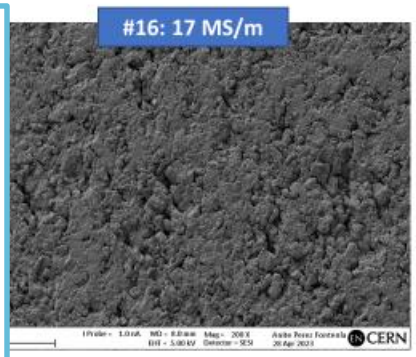
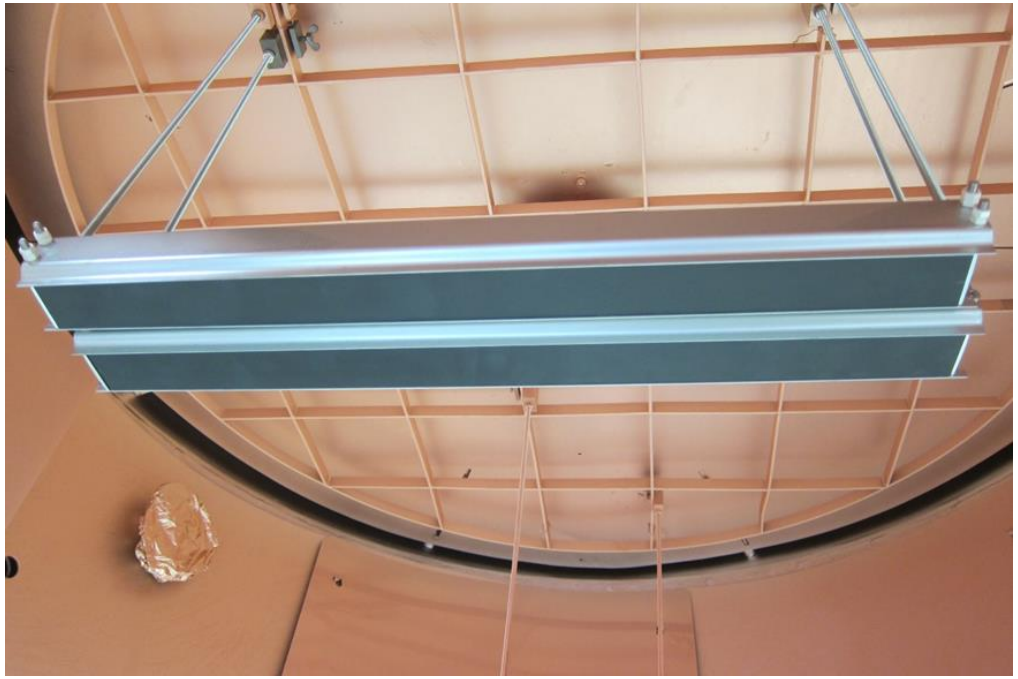
Try to increase the coating thickness!



Replicate a high-el. Conductivity coating on blocks and tapering

Coating on real blocks

- First positive test on blocks
  - Block 1: average conductivity 33±2.5 MS/m (variation along the length)
  - Block 2: average conductivity 28±2.5 MS/m (variation along the length)
  - Is the difference related to position? Is it repeatable?



See next talk of L. Sito for measurement details!

Conclusions

- Cu-coated graphite is selected as absorber material for LS3 TCSPM
- A small pre-series production has been launched to optimize the post-production treatments and coating at CERN
- With this pre-production we have positively tested different points:
  - Tight tolerance achievable and CMM report from the company coherent with CERN's one
  - Optimal parameters for the thermal treatment found. UHV compliant after cleaning and after the coating
  - Achievable value of Cu coating~ 34MS/m both on samples and blocks
  - Possibles reason for high conductivity variation analyzed and coating parameters were corrected
- **Almost 10 coating runs done and almost 40 samples measured in few months → many thanks to the UHV colleagues and to the impedance team for all the work done!!**

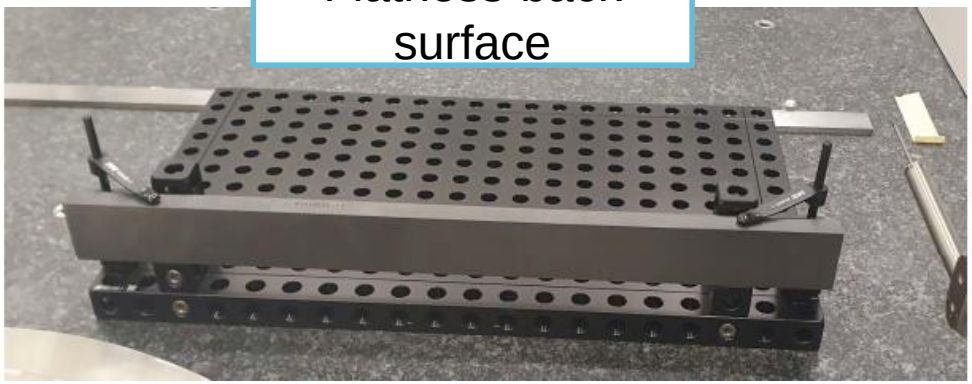
Next steps

- 2 blocks and 4 tapering coated→ impedance measurement planned
- **Definition of coating planning and measurement strategy**:
  - If the difference is confirmed, is it worst reducing the coating production rate? (15→30 weeks!!)
  - Should we measure the conductivity of all the blocks? In all the positions?
  - Can we rely on witness samples?
  - What is the minimum acceptable conductivity? Can we rely on the average one (if measured in different longitudinal location?)
  - What are the limits for the tapering?

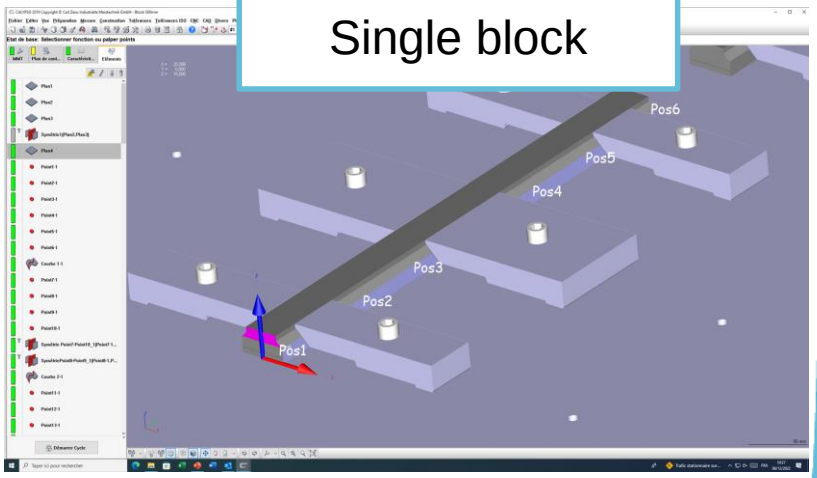
Metrology

- 3 measurement required at CERN

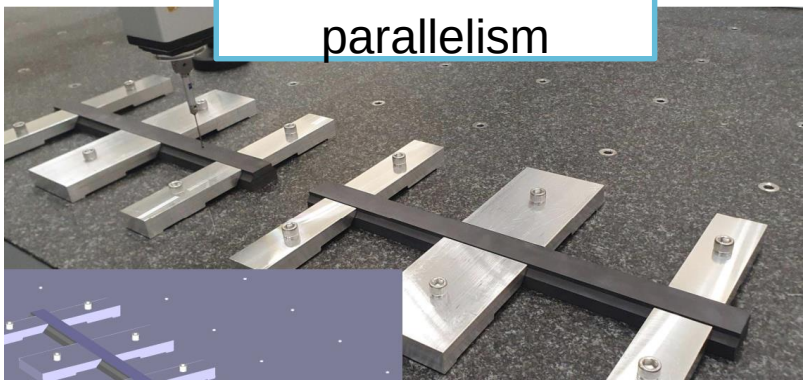
Flatness-back surface



Single block



Two blocks parallelism



Metrology

- 3 measurement required at CERN

	flatness	measurement 500			measurement 2x500				
block	flatness A	max 45mm	max 500	position	flatness	position	height	parallelism	max length
1.1	0.035	45.026	500.064	500.047	0.004	0.042	24.979	0.009	1000.099
1.2	0.011	45.026	500.035	500.03					
2.1	0.025	45.026	500.043	500.04	0.004	0.04	24.98	0.009	1000.112
2.2	0.03	45.026	500.069	500.054					
3.1	0.019	45.027	500.04	500.036	0.004	0.041	24.979	0.008	1000.109
3.2	0.022	45.025	500.069	500.059					
max	0.02	45.05	500.05	500.05		0.2		0.01	

all values ok
minor values not ok
important values not ok

Metrology

- 3 measurement required at CERN

	flatness	measurement 500			measurement 2x500				
block	flatness A	max 45mm	max 500	position	flatness	position	height	parallelism	max length
1.1	0.035	45.026	500.064	500.047	0.004	0.042	24.979	0.009	1000.099
1.2	0.011	45.026	500.035	500.03					
2.1	0.025	45.026	500.043	500.04	0.004	0.04	24.98	0.009	1000.112
2.2	0.03	45.026	500.069	500.054					
3.1	0.019	45.027	500.04	500.036	0.004	0.041	24.979	0.008	1000.109
3.2	0.022	45.025	500.069	500.059					
max	0.02	45.05	500.05	500.05		0.2		0.01	

- We are measuring with the clamps; thus the final parallelism of the beam surface would be close to the one measured with the blocks installed in the jaw. However, a very high tolerances of the flatness could compromise the contact and hence the heat evacuation. These values are considered acceptable.

Metrology

- 3 measurement required at CERN

	flatness	measurement 500			measurement 2x500				
block	flatness A	max 45mm	max 500	position	flatness	position	height	parallelism	max length
1.1	0.035	45.026	500.064	500.047	0.004	0.042	24.979	0.009	1000.099
1.2	0.011	45.026	500.035	500.03					
2.1	0.025	45.026	500.043	500.04	0.004	0.04	24.98	0.009	1000.112
2.2	0.03	45.026	500.069	500.054					
3.1	0.019	45.027	500.04	500.036	0.004	0.041	24.979	0.008	1000.109
3.2	0.022	45.025	500.069	500.059					
max	0.02	45.05	500.05	500.05		0.2		0.01	

- Even if the total length does not respect the tolerances, the sum of the 2 blocks allow a safe mounting of the jaw.

Metrology

- Comparison with SGL
  - Good agreement on all the measurement, but for the symmetry ad the profile (see next slide)
  - An error was detected in the CERN program, reference and tolerance switched to calculate the parallelism→ does SGL noticed it? To be checked
  - CERN 500mm measured with external tangent plane to represent the worst case, not done in SGL→ to be changed

SGL

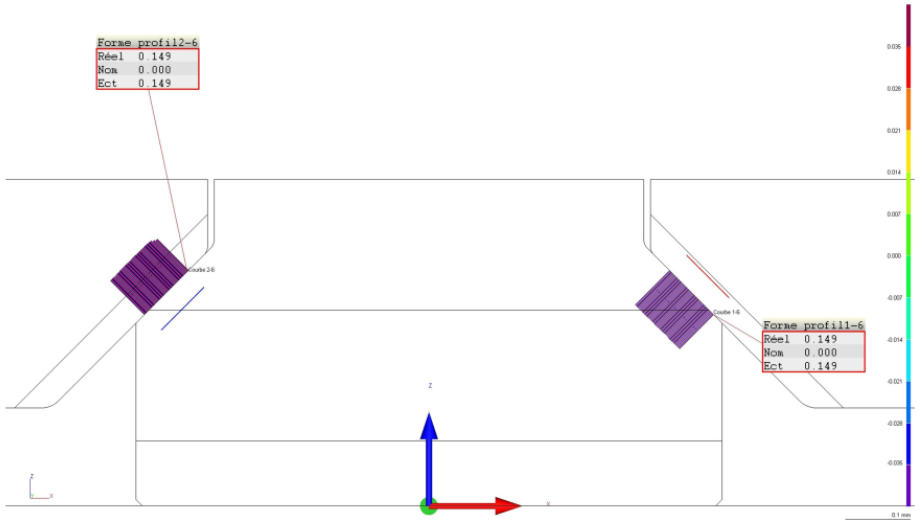
	measurement 500		measurement 2x500			
block	max 45mm	position	flatness	position	height	parallelism
1.1	45.023	500.0056	0.009	0.048	24.976	0.0114
1.2	45.024	500.007				
2.1	45.022	500.0075	0.01	0.0432	24.978	0.0121
2.2	45.024	500.0052				
3.1	45.024	500.0043	0.009	0.0523	24.944	0.0119
3.2	45.024	500.0056				

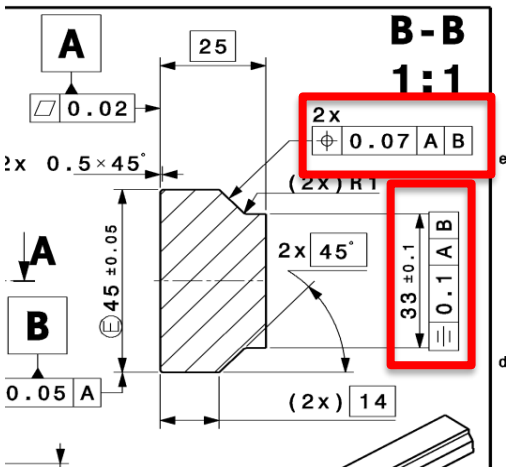
CERN

	measurement 500		measurement 2x500			
block	max 45mm	position	flatness	position	height	parallelism
1.1	45.026	500.047				
1.2	45.026	500.03	0.004	0.042	24.979	0.009
2.1	45.026	500.04				
2.2	45.026	500.054	0.004	0.04	24.98	0.009
3.1	45.027	500.036				
3.2	45.025	500.059	0.004	0.041	24.979	0.008

Metrology

- 4 blocks with non-conformities at the level of symmetry and profile (not detected in SGL)
- Worst case analyzed (block 3.2)
- Symmetry 0.226mm→ the gap between the housing and the blocks will be extended to 0.4mm→ this asymmetry can be accepted
- Profile 0.149mm→ this could push the clamp toward the beam. However, the clamp will slight deform the surface and even if the clamp is outward no problem (anticollision mechanism to be aware of this)





Metrology

- Continuous vs by point measurement→ some marks appeared on the surface (removed with gloves, to be checked on cleaned blocks)
  - 48min vs 169 min
  - ~40% of difference in the results (probably related to the continuous pressure exerted on the surface)

continous	flatness	position	height	parallelism
1.1	0.004	0.042	24.979	0.009
1.2				

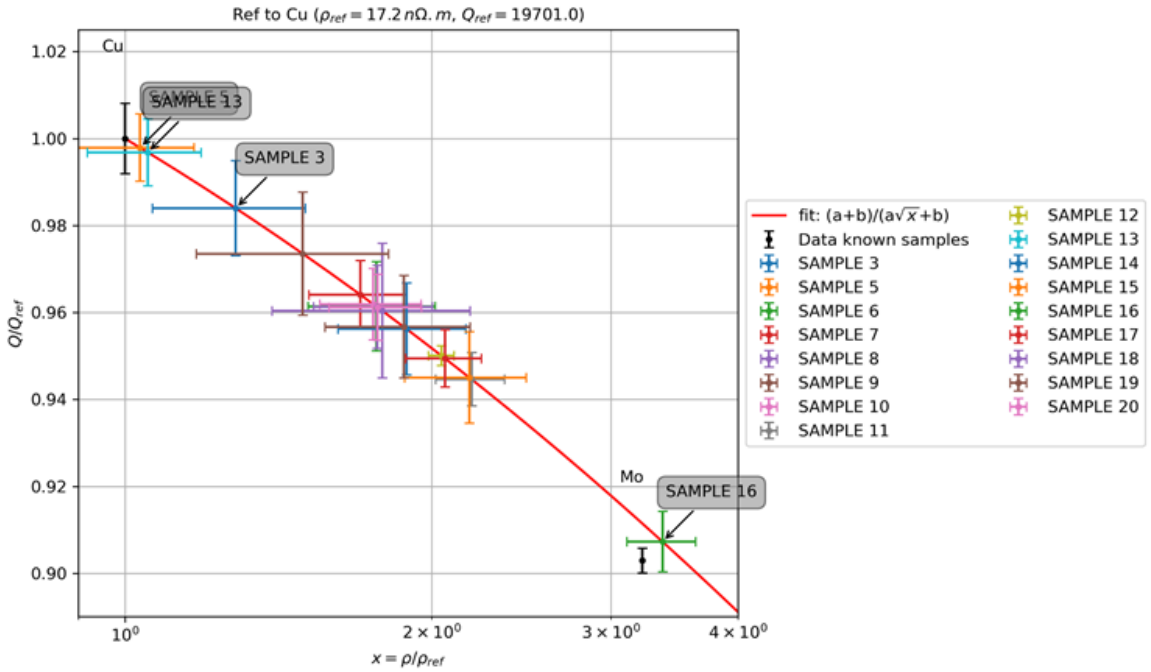
by point

1.1	0.004	0.041	24.98	0.013
1.2				

Should we change the program for the series?

Second coating run

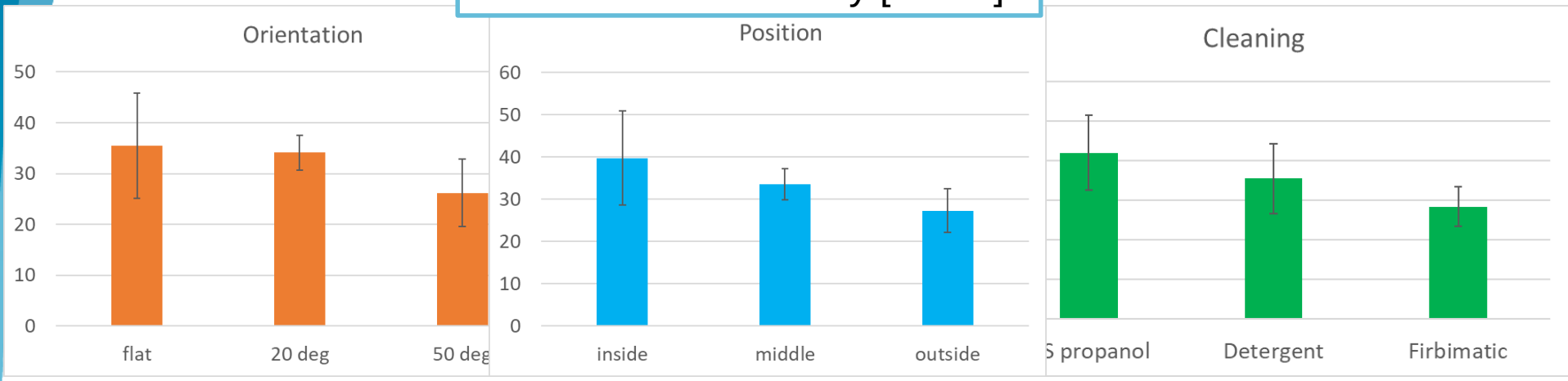
- Different cleaning, orientation and position tested
- Different orientation to mimic the tapering surface
- ‘Statistical’ analysis performed to understand the influence of different parameters
- ***Topic to be addressed at the WP2 meeting***



Courtesy of C. Antuono, L. Sito, A.Kurtulus, N. Biancacci, N. Mounet, B. Salvant

Second coating run

Electrical conductivity [MS/m]



	Conductivity [MS/m]		
Orientation	#sample	average	dev
flat	10	35.52461	10.33261
20 deg	3	34.11268	3.42512
50 deg	2	26.20744	6.654802

	Conductivity [MS/m]		
Position	#sample	average	dev
inside	6	39.75345	11.21432
middle	4	33.49665	3.719578
outside	6	27.2832	5.178647

	Conductivity [MS/m]		
Cleaning	#sample	average	dev
US propanol	2	42.0145	9.418866
Detergent	7	35.46399	8.81331
Firbimatic	7	28.43653	5.048292

- An average conductivity of 34MS/m is reached.

Block 2.1 VS Block 2.2

Serial Number					
Sample ID	Left far	Left near	Center	Right near	Right far
Resisitvity [nOhm.m]	33.01455	35.238622	39.276428	38.741712	32.727704
Error on Resisitivity	2.413862	1.279696	1.583135	0.686026	1.630721
Conductivity [MS/m]	30.289676	28.377954	25.460564	25.811973	30.555153
Error on Conductivity [MS/m]	2.214633	1.030549	1.026252	0.45707	1.522469

Average Resistivity: 35.80 [nOhm.m]
Average Conductivity: 28.10 [MS/m]

Serial Number					
Sample ID	Left far	Left near	Center	Right near	Right far
Resisitvity [nOhm.m]	31.606848	28.743126	26.772739	32.972479	31.625235
Error on Resisitivity	2.361193	0.440461	1.310587	1.359024	1.470169
Conductivity [MS/m]	31.638713	34.790928	37.351426	30.328323	31.620319
Error on Conductivity [MS/m]	2.363574	0.533138	1.828437	1.25004	1.469941

Average Resistivity: 30.34 [nOhm.m]
Average Conductivity: 33.15 [MS/m]

Next step:

1. Repeat 2 blocks coating
2. Confirm the difference, evaluate the impact on the production if 4 blocks per batch not 8
3. Decide how to measure the blocks during the series (all? Samples?)
4. Minimum allowable conductivity? Average ?
5. Tapering