

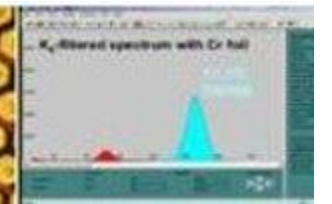
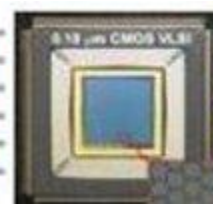
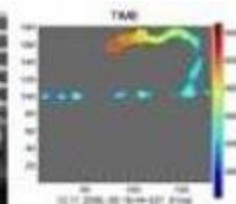
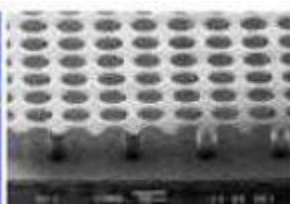
Status of the DLC machine

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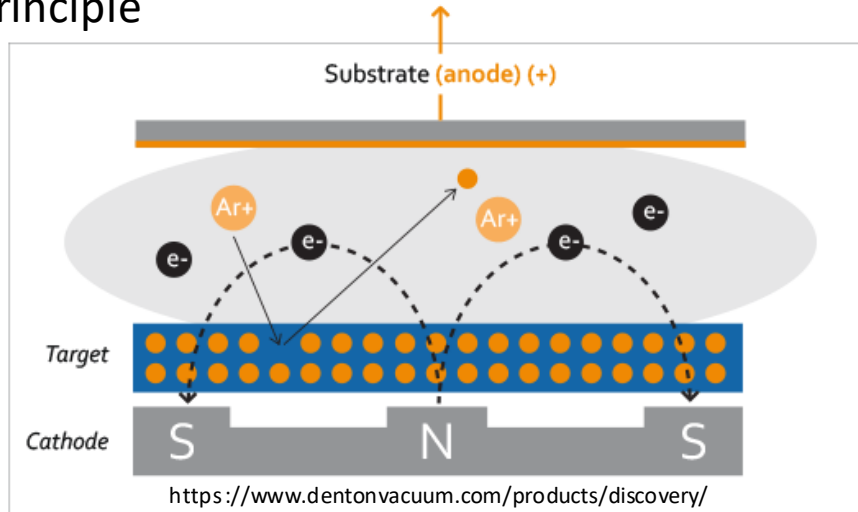
CONTRIBUTION

INFN-CSN1



The magnetron sputtering machine

Working principle



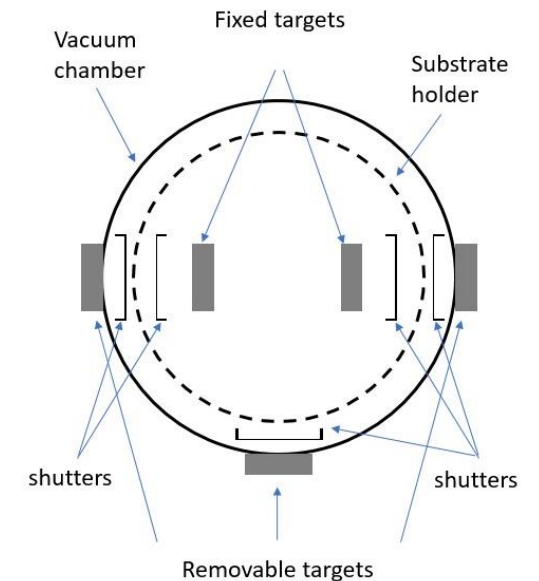
- The field creates ion-electron pairs in the plasma
- The electrons are accelerated and further ionize the plasma generating new ions
- A magnetic field concentrates the ions in a peculiar region of the target (speed track)
- The ions drift towards the target and by collision the material is extracted flying all over the vacuum chamber



The graphite target



The three external cathodes



DLC tests

The machine phase-space is quite huge; thanks to Serge's experience the plan for the tests have been focused to few parameters.

QUICK GLOSSARY

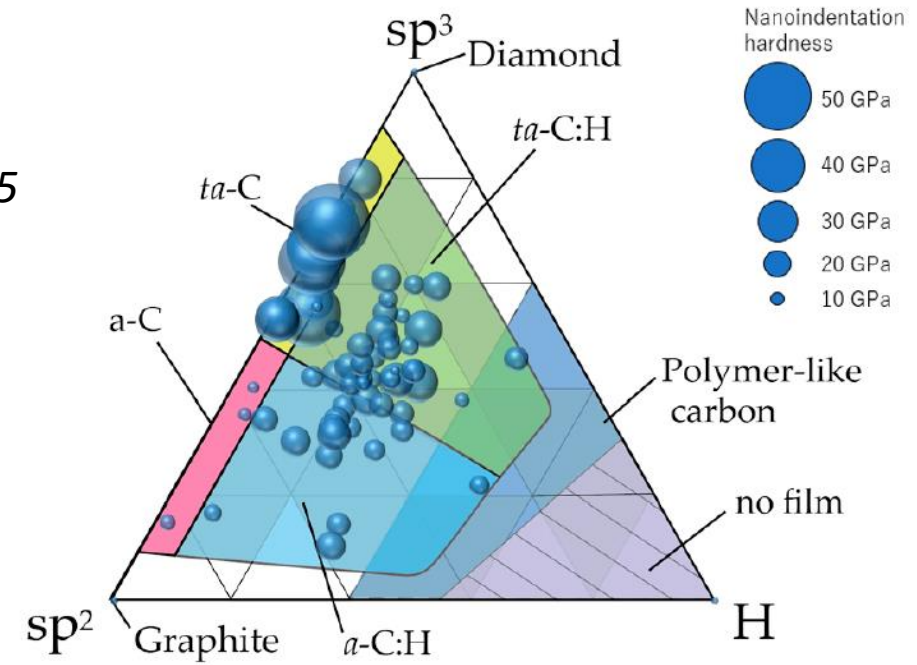
Pressure limit: *the pressure of the vacuum at which the run starts. Typically 2.E-5 mbar*

Pressure process: *the pressure of the plasma during the sputtering phase*

Power: *the maximum power limit output from the DC PS on the cathode*

Time deposition: *the duration of the sputtering process*

- Ar-N₂ plasma tests
 - Scan in nitrogen percentage at a given pressure process
 - Scan in pressure process at a given nitrogen percentage
 - Tests with different time deposition
- Ar-C₂H₂ plasma tests
 - Scan in acetylene percentage at a given pressure process and power
 - Scan in time deposition at a given pressure process, acetylene percentage and power
 - Uniformity tests



DLC tests: modus operandi

SAMPLES

- APICAL foil dried in the oven (at least 16h at 100°C)
- Three rectangular samples, 15 x 10 cm² (machine operating in oscillation mode), to check the uniformity of the deposition along z
- A small 1.5 x 1.5 cm² glass next to each sample for thickness measurement

RUN

- Pure Ar-based plasma surface treatment (plasma etching)
- Pure Ar-based pre-sputtering process
- Sputtering process

POST RUN

- Resistivity measurements
- Baking of one sample from each run (2h at 220°C) to simulate the thermal shock during detector manufacturing
- Monitoring of the resistivity during the following days (stability check)



Repetitivity and stability with N2

First tests with N2 answered to two urgent questions:

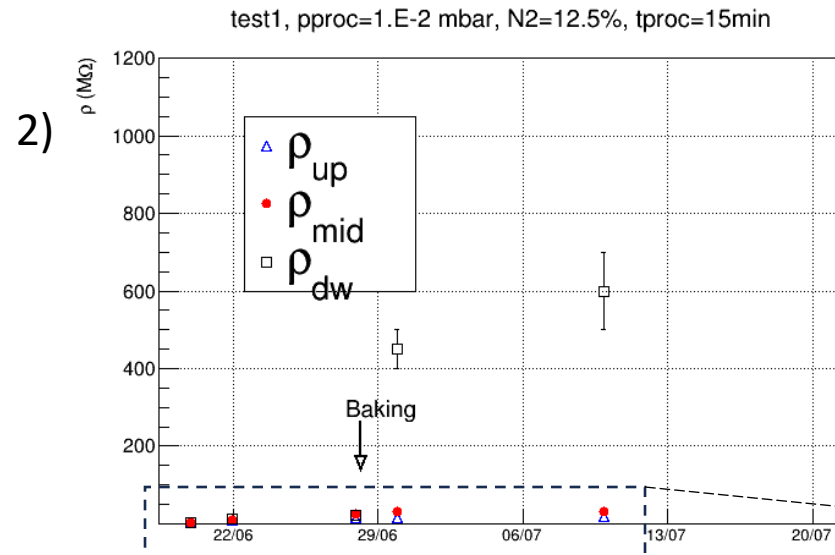
- 1) Standing the same deposition parameters, will the resistivity be the same or will it change?
- 2) How does the resistivity change along the time?

| 1) Test | | | 20/06 | 22/06 |
|---------|---|--|-----------------|-------------|
| 1 | $P_{\text{proc}}=1.E-2$ $N_2 = 12.5\%$ $T_{\text{proc}} = 15\text{min}$ | ρ_{bot} ρ_{mid} ρ_{up} | 2.7 2.5 2 | |
| 2 | $P_{\text{proc}}=1.E-2$ $N_2 = 12.5\%$ $T_{\text{proc}} = 15\text{min}$ | ρ_{bot} ρ_{mid} ρ_{up} | 2.5 3 2 | |
| 3 | $P_{\text{proc}}=1.E-2$ $N_2 = 12.5\%$ $T_{\text{proc}} = 15\text{min}$ | ρ_{bot} ρ_{mid} ρ_{up} | | 3 3 4 |

Measurements in Mohm/sq.

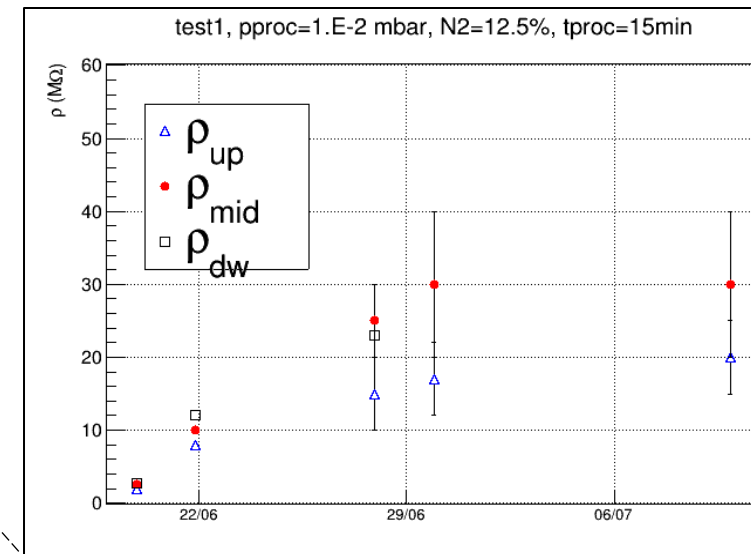
Quite satisfactory repetition capability

Further confirmations for other dep. conditions



We baked one of the three sample and the resistivity jumped up of a factor 200

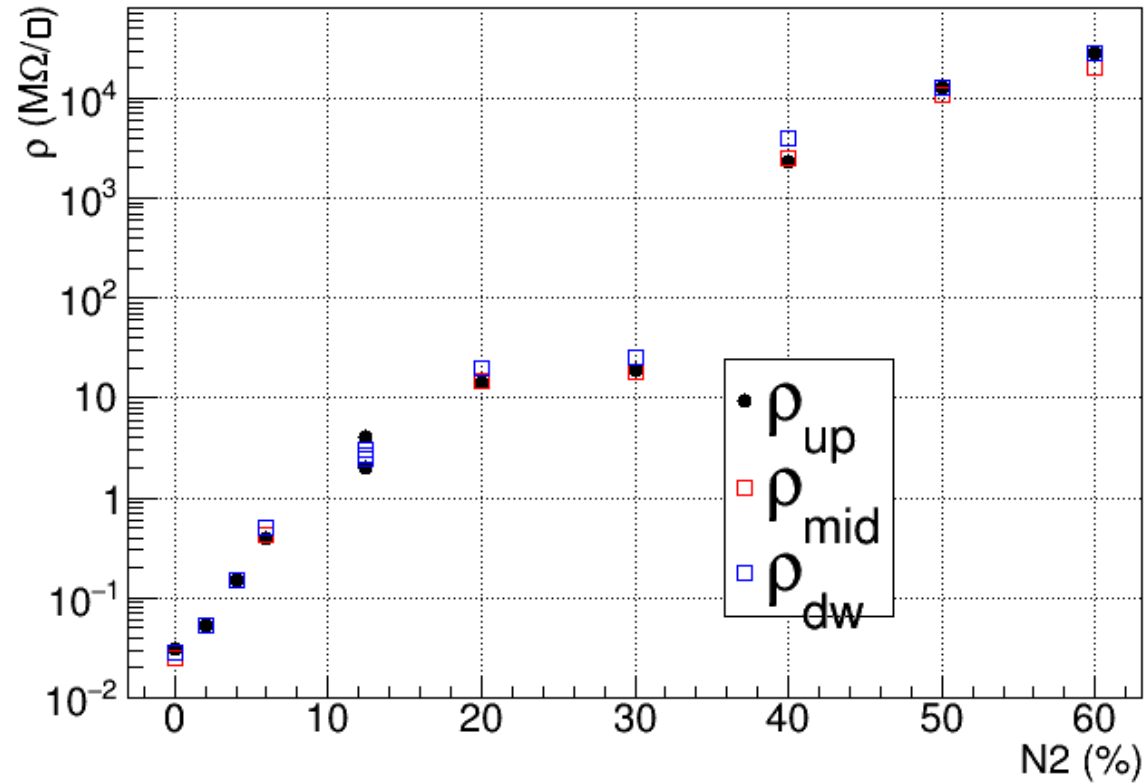
Anyway a drift towards upper values of resistivities is observed after few days
WE NEED STABLE VALUES



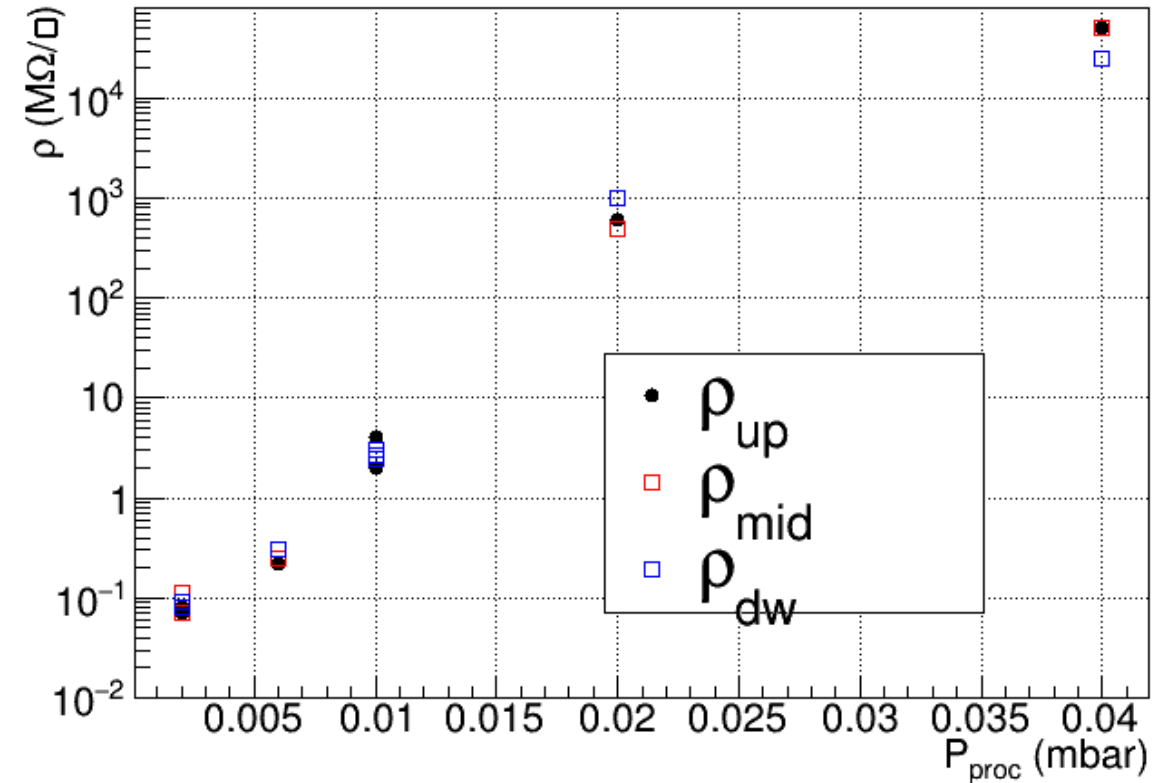
Summary of the test with N₂ (June 2023)

Nevertheless these tests have been helpful to understand the dependance of the resistivity on the quantity of the second component of the plasma and on the pressure process

N₂ scan at $p_{\text{proc}} = 1.E-2$ mbar and $\Delta t_{\text{proc}} = 15$ min



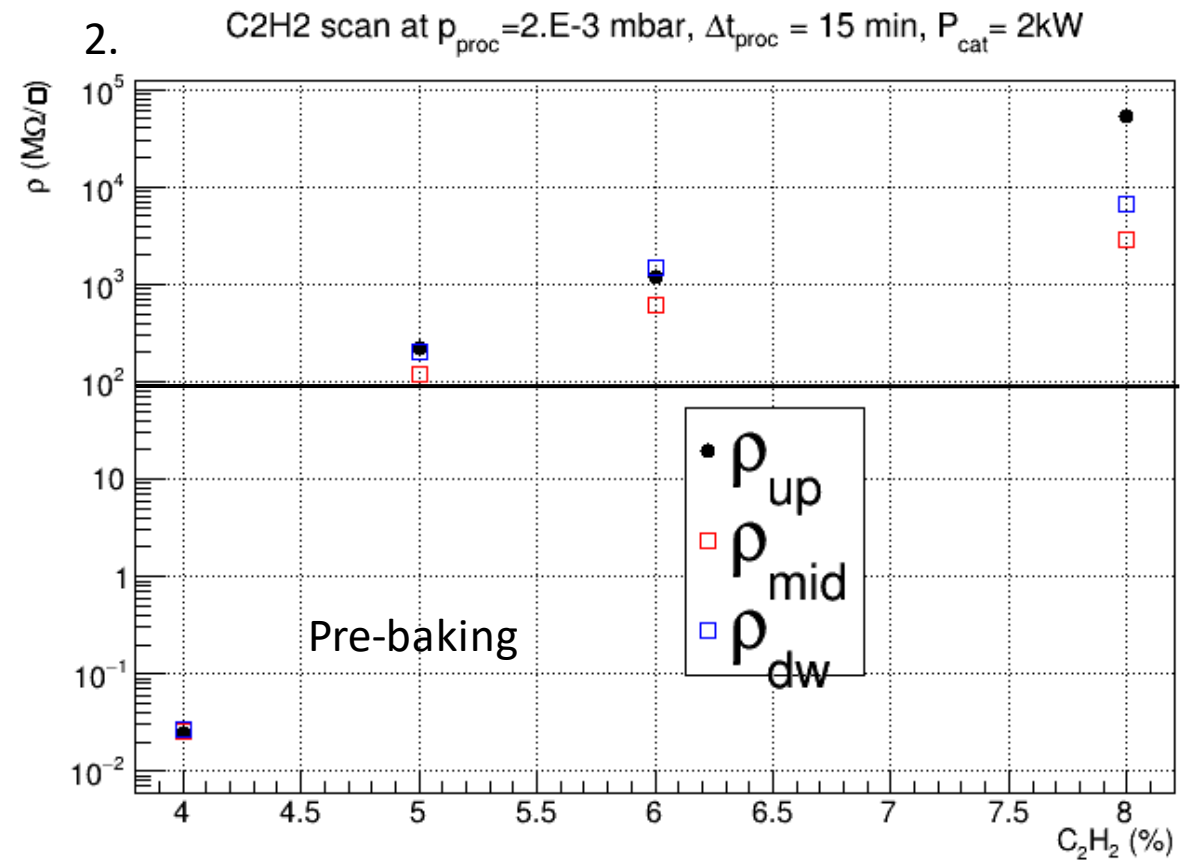
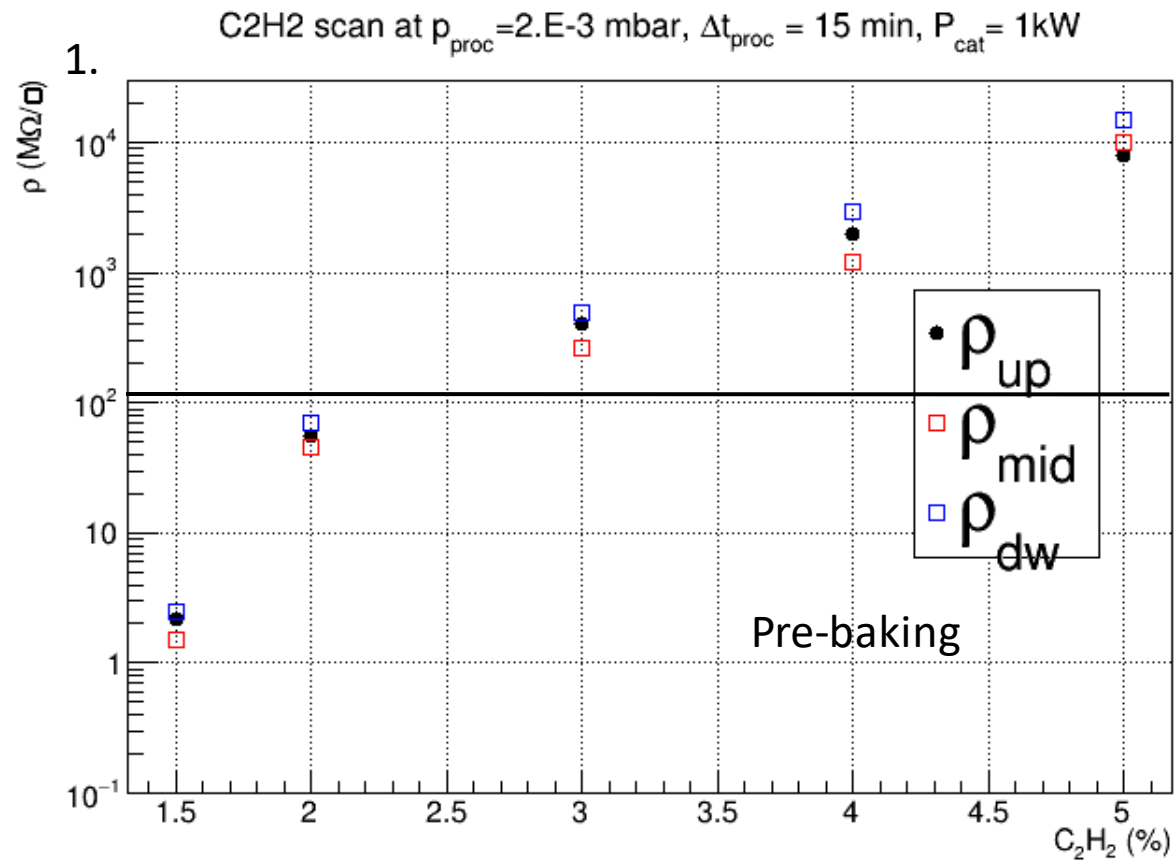
P_{proc} scan at 12.5% N₂ and $\Delta t_{\text{proc}} = 15$ min



Very large amount of nitrogen to reach the target resistivity (50 – 200 Mohm/sq.)

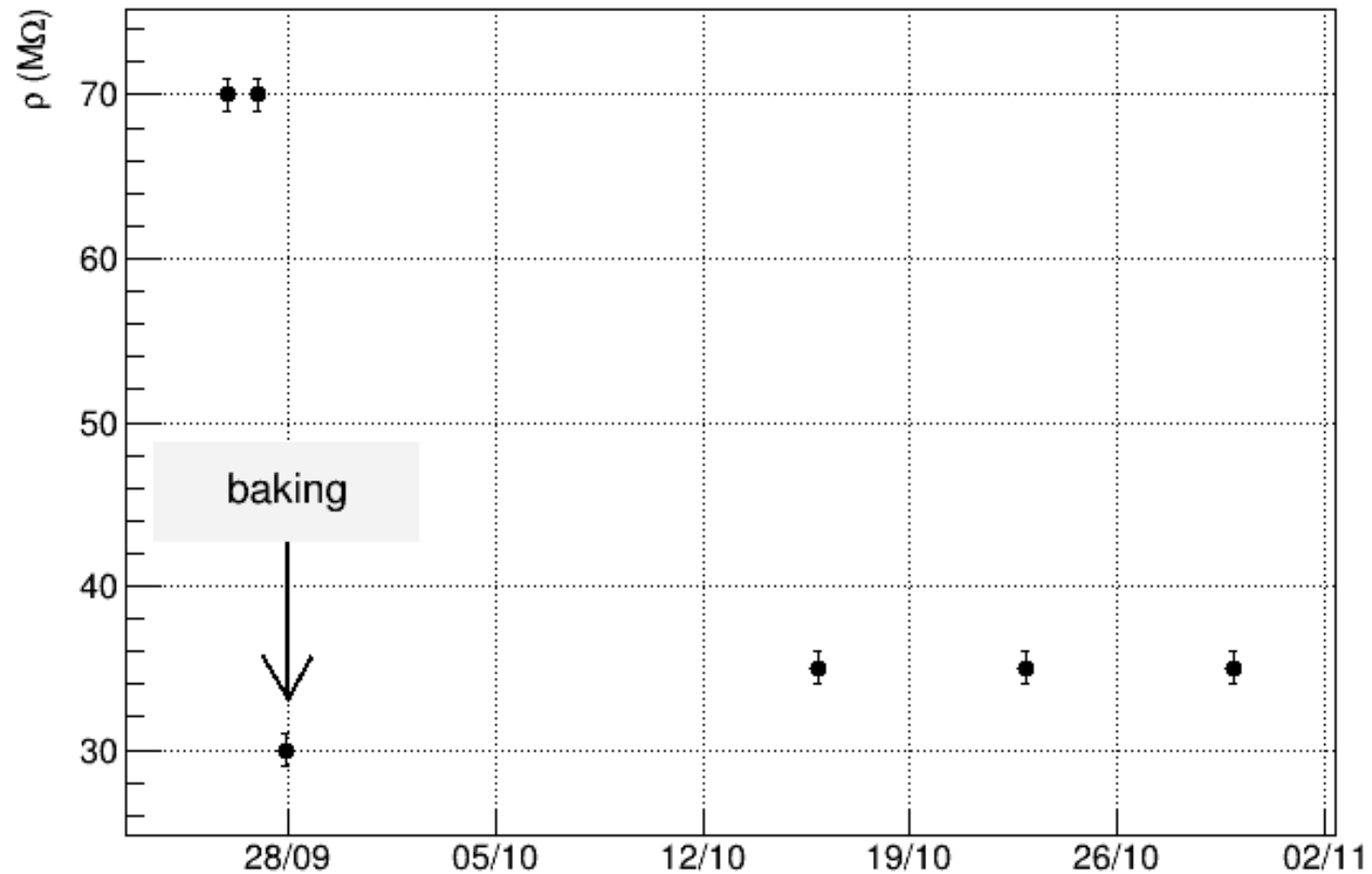
Summary of the test with C₂H₂ (Sept. 2023)

1. Scans in acetylene percentage (1.5% - 5%) at $p_{\text{proc}} = 2.E-3$ mbar, $t_{\text{dep}} = 15$ m and $P_{\text{cat}} = 1$ kW
2. Scans in acetylene percentage (4% - 8%) at $p_{\text{proc}} = 2.E-3$ mbar, $t_{\text{dep}} = 15$ m and $P_{\text{cat}} = 2$ kW
3. Test at different deposition time (15 m – 240 m) with 4% of C₂H₂ at $p_{\text{proc}} 8.E-3$ and $P_{\text{cat}} = 1$ kW
4. Repetitvity tests (C₂H₂ 3%, $p_{\text{proc}} = 2.E-3$, $P_{\text{cat}} = 1$ kW, $t_{\text{dep}} = 22.5$ m)
5. Uniformity tests



Stability with C₂H₂ (Sept. - Oct. 2023)

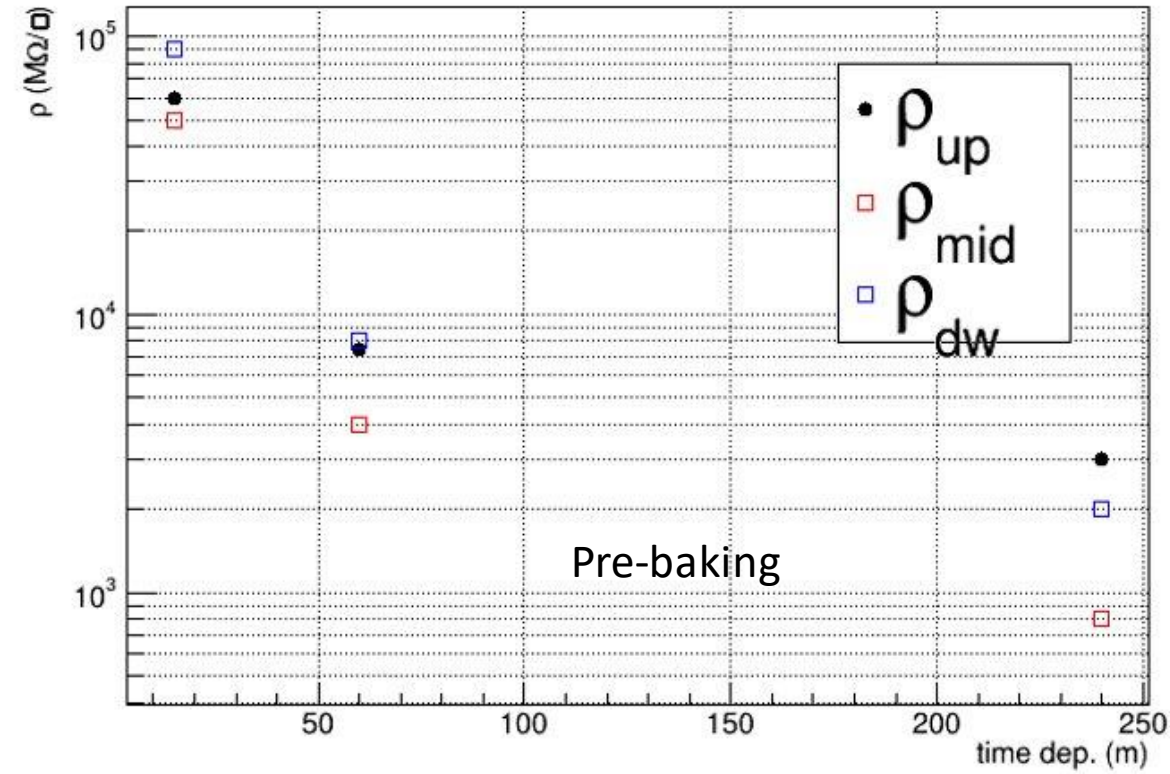
C₂H₂ 2%, p_{proc} = 2E-3 mbar, t_{dep} = 15 min, P_{cat} = 1kW



A drop after the baking and a small drift, but then very stable values

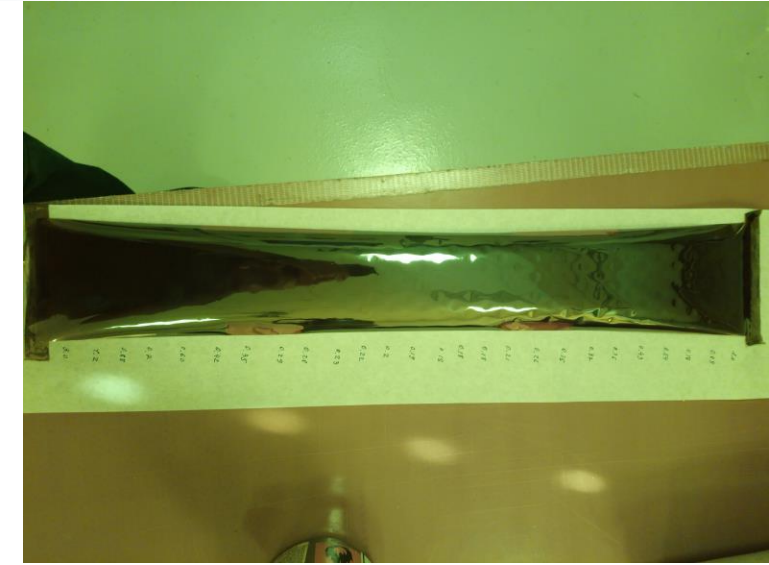
Deposition time and repetitivity test

time dep. scan at C2H2 = 4%, $p_{\text{proc}} = 8.E-3$ mbar, $P_{\text{cat}} = 1$ kW

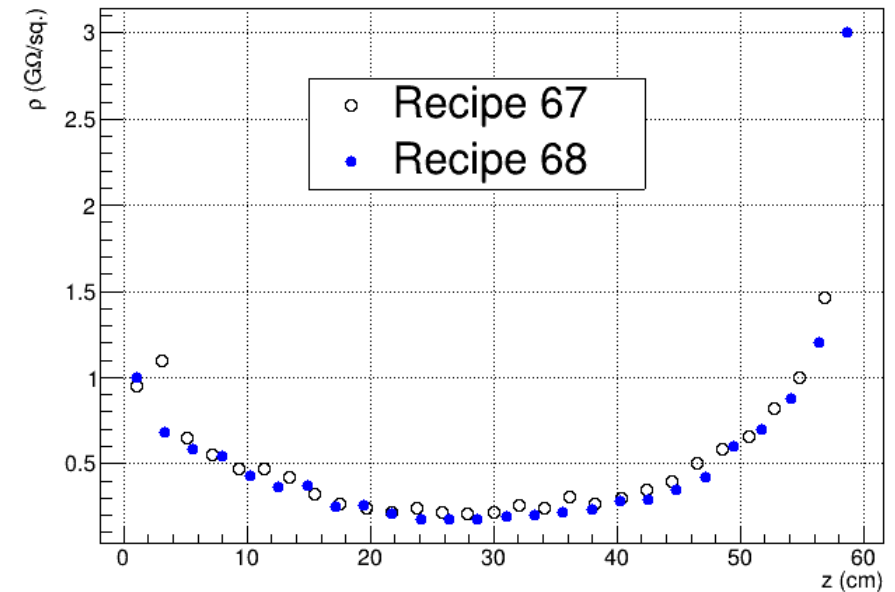


Confirmed the behaviour $1/t$
Time can be an important parameter for the resistivity adjustment
WARNING = evidence of non-uniformity along z!

From now on, we use a single APICAL sample 60 x 10 cm²
We measure the resistivity along the longitudinal axis

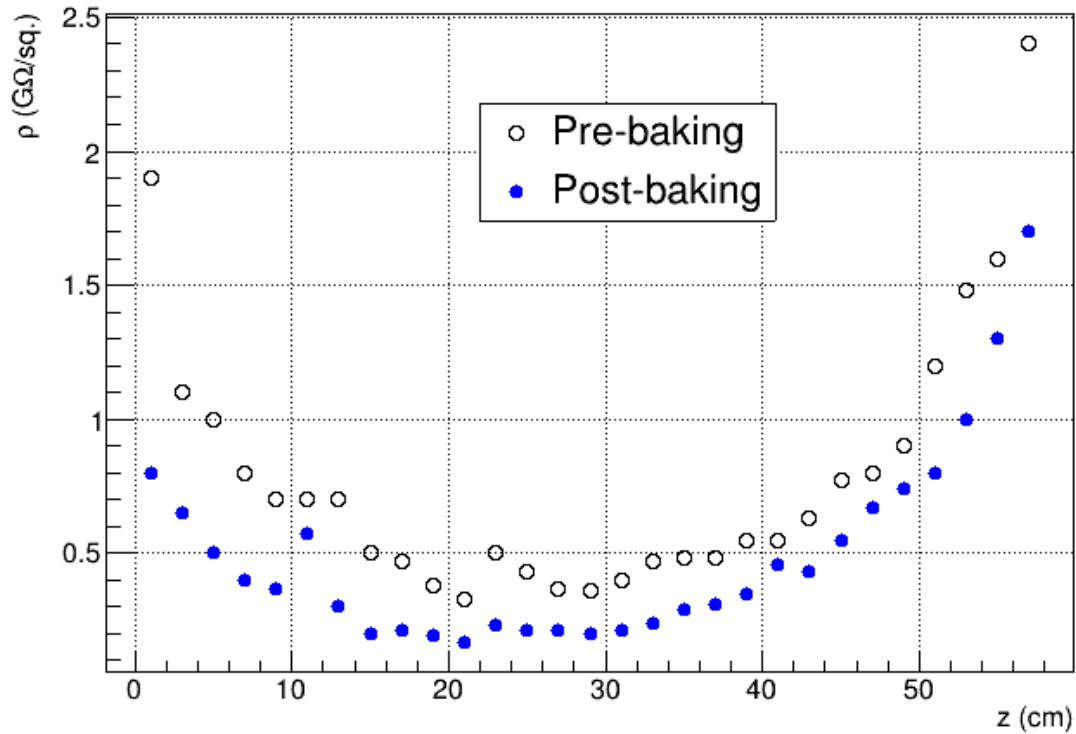


C2H2 3%, 2.E-3 mbar, time = 22.5 min, P = 1 kW

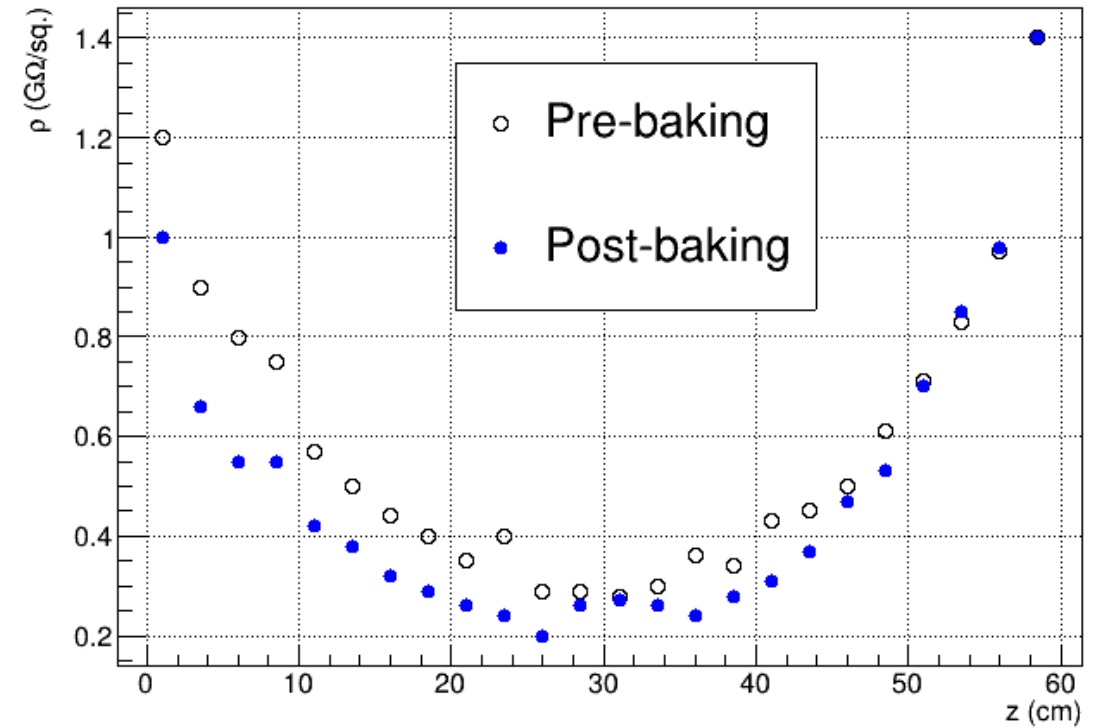


Uniformity test

C2H2 4%, 2.E-3 mbar, time = 15 min, P = 1 kW



C2H2 5%, 2.E-3 mbar, time = 10 min, P = 2 kW



The baking, as expected, doesn't change the thickness profile

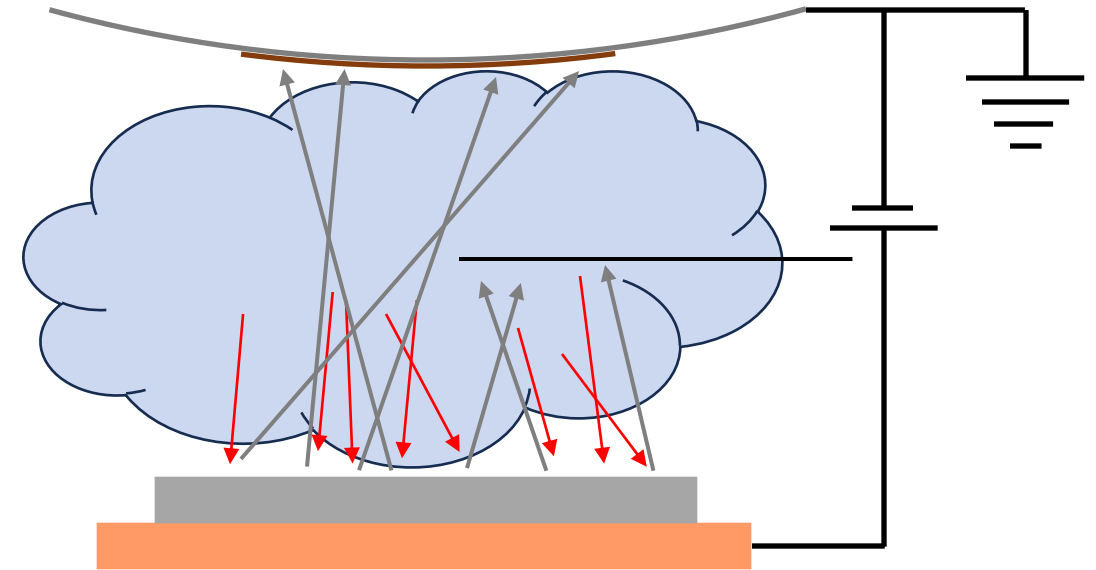
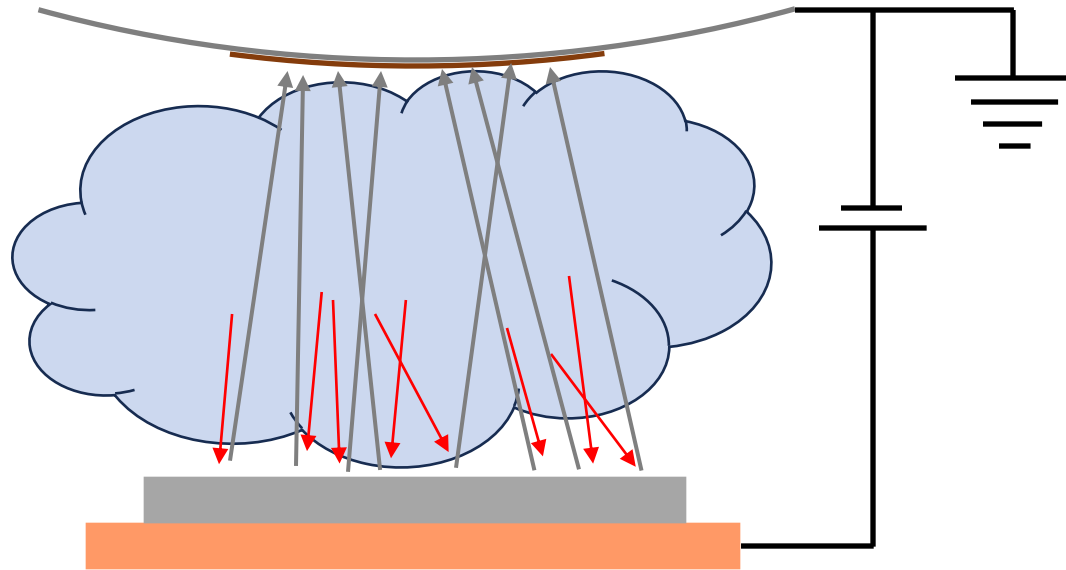
These tests pointed out that the deposition, along the axis, is uniform in a very narrow central region

We would like to have a uniformity $\leq 15\%$

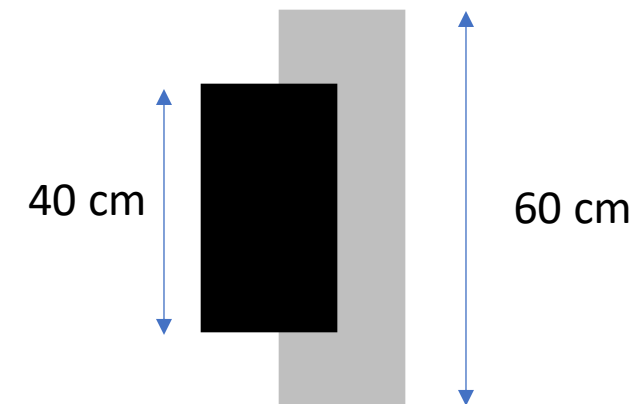
THIS REGION MUST BE EXTENDED

Summary of the test with C_2H_2 (Nov. 2023)

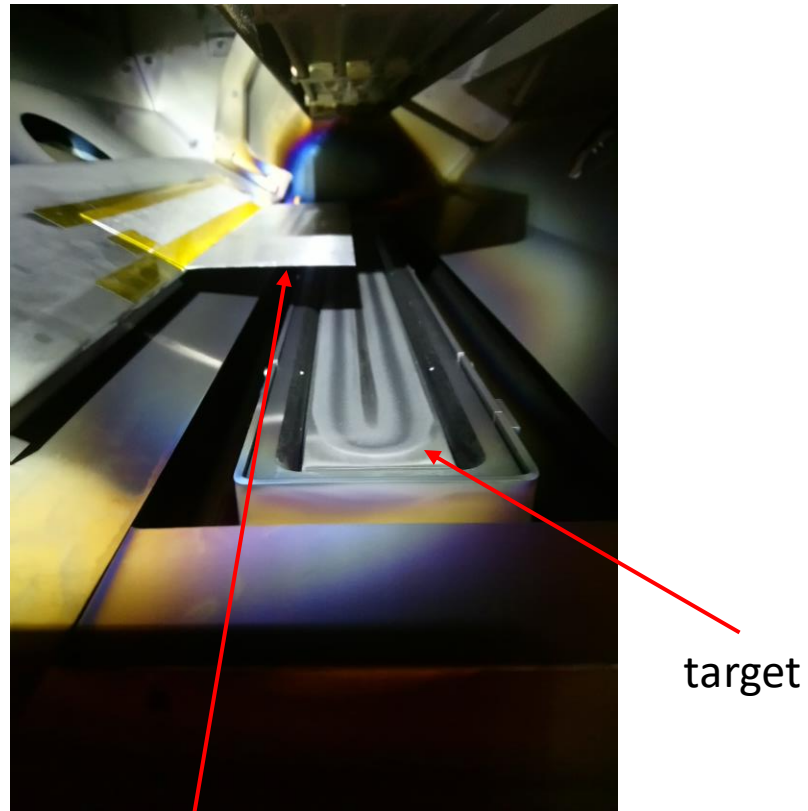
To improve the uniformity, Serge's idea is to install a mask stuck to the shutter to reduce the material extraction in the central part of the target



Lower extraction → thinner deposition → larger resistivity



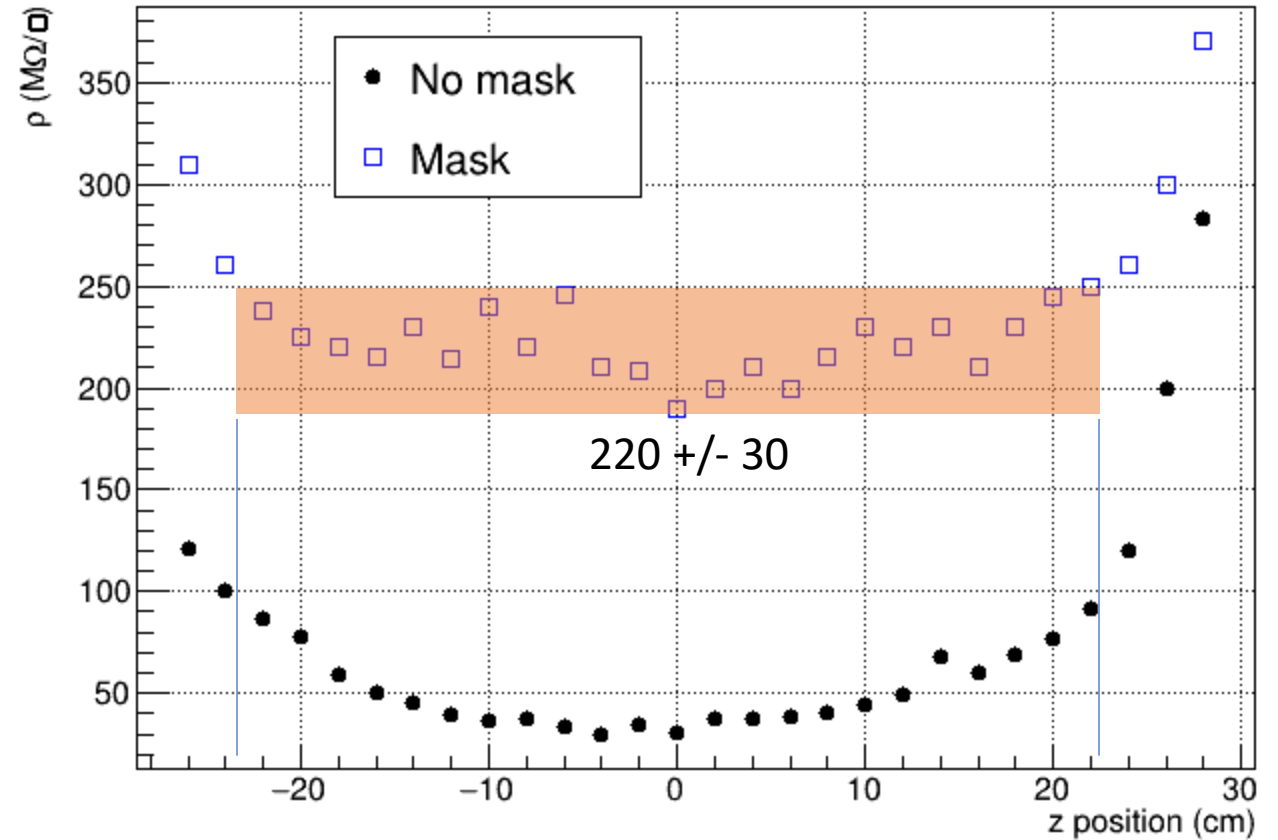
Summary of the test with C₂H₂ (Nov. 2023)



mask

target

Ar 150 sccm, C₂H₂ 3 sccm, p_{proc} 2E-3 mbar



The mask seems promising. The uniformity region has been extended to about 45 cm. Further improvements can be achieved with a different mask shape

Summary and plans

- Repetitiveness of the results have been confirmed with different plasma composition
- The first tests with nitrogen have been very helpful to understand and confirm the dependence of the resistivity on the plasma composition and pressure
- Nitrogen does not help in stability
- Acetylene seems very promising as the wanted resistivity can be obtained with different recipes
- The uniformity along the vertical axis have been remarkably improved with the insertion of an aluminum mask (to be optimized)
- Next tests (early 2024) dedicated to deposition on a large area APICAL foil (170 x 60 cm²)