



Università
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Possible Material-Saving Approach of Sputtering Techniques for Radiopharmaceutical Target Production

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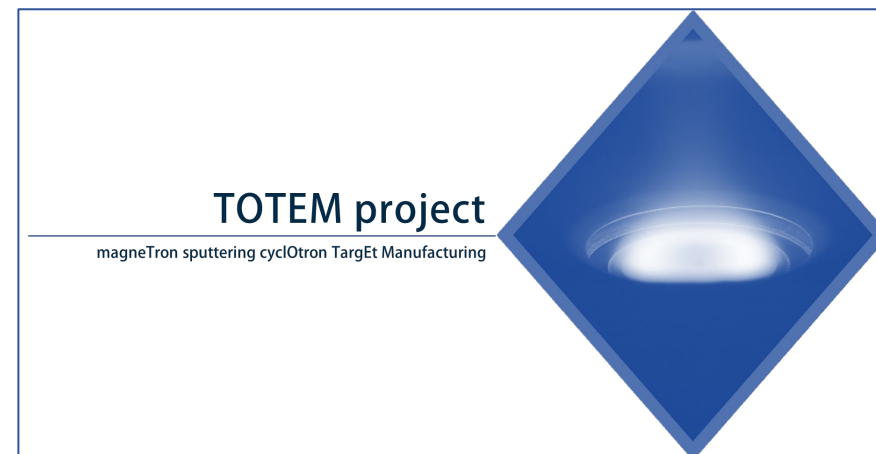


18th Workshop on Targetry and Target Chemistry
Whistler, BC August 22-26, 2022



Work Goal

To evaluate the main magnetron sputtering technique issues and to found a possible solution to use this technique for solid target production



This research was funded by CSN5 of the INFN, Italy for 2018–2022, in the framework of METRICS project. PI: J. Esposito and by INTEFF program at INFN funded by MISE, Italy – in the framework of TOTEM project PI: S.Cisternino.

Outline

1

Introduction to Sputtering techniques

2

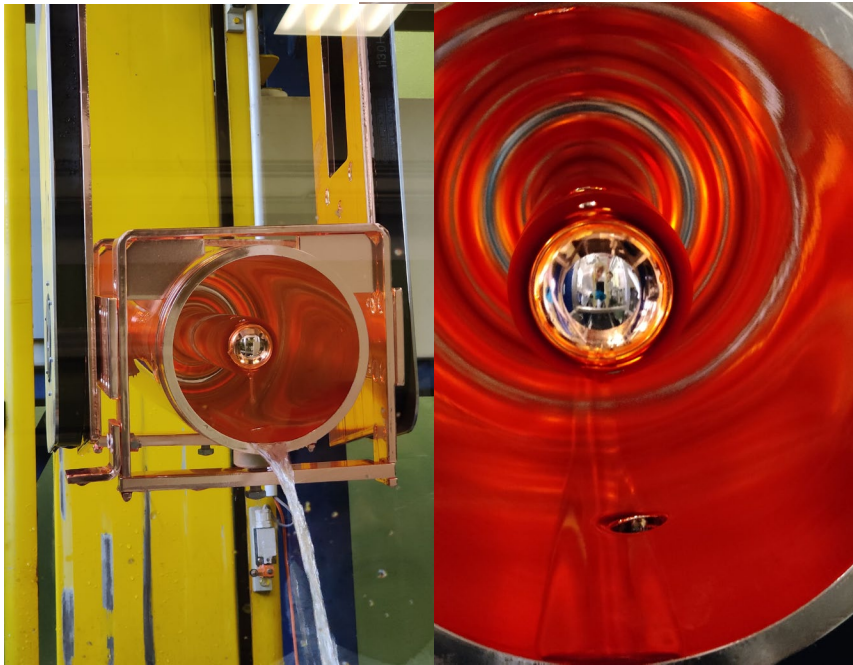
Results on magnetron sputtering optimization

3

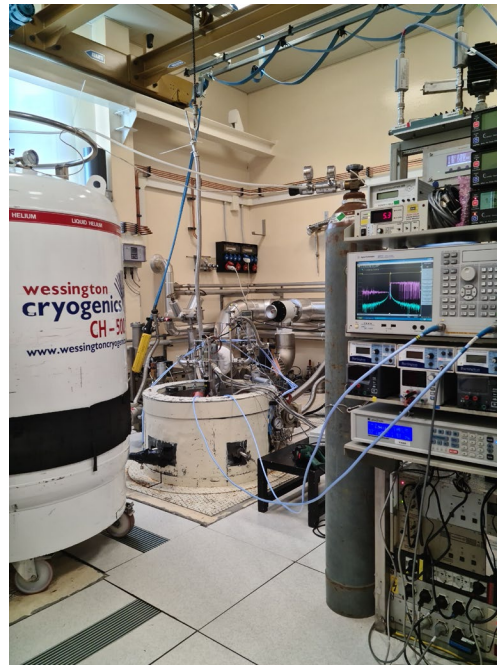
Results on nat-ZnO targets realization

Surface Technologies and Superconductivity Service

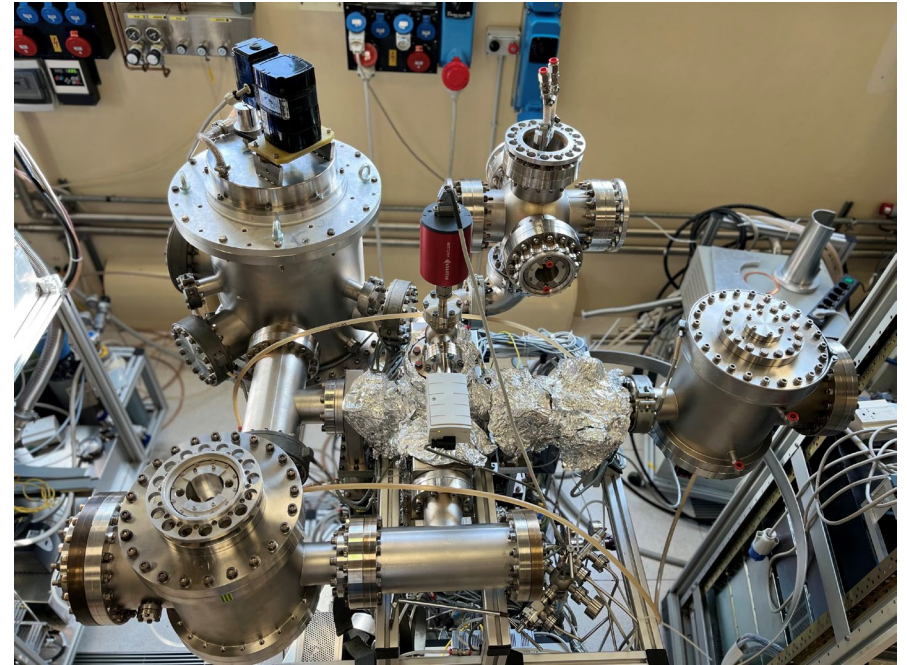
Chemistry laboratory



Cryo laboratory



Sputtering laboratory



Advanced surface treatments for Cu and Nb accelerating cavities

Superconductive Nb depositions

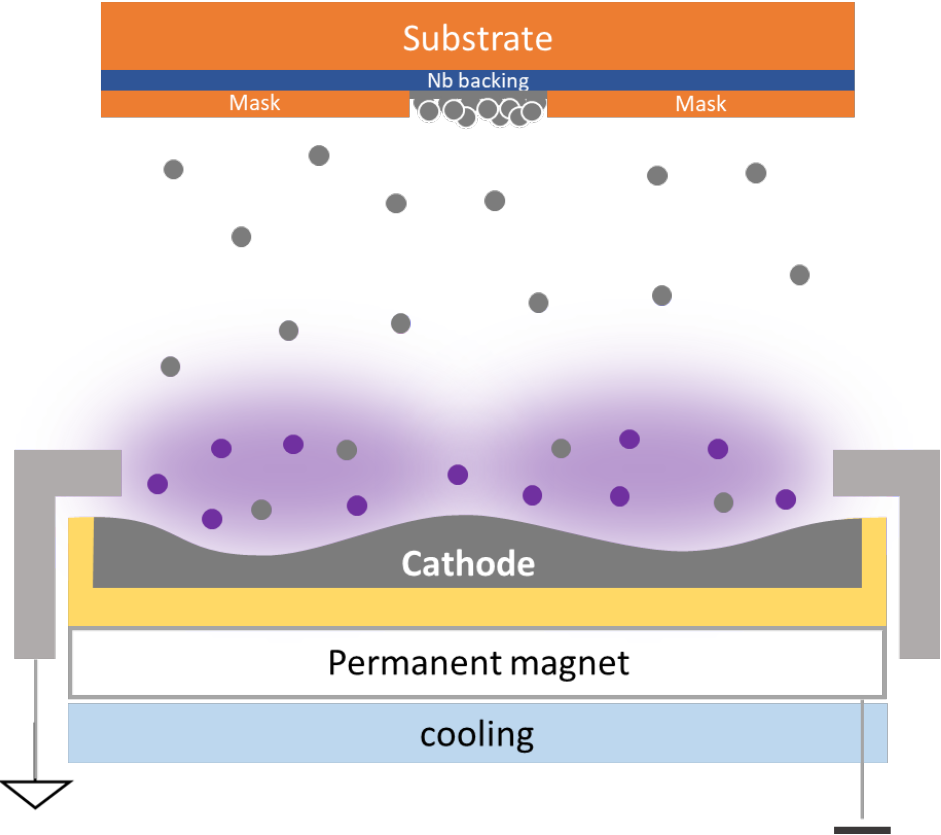
Cryogenic characterization

- Various PVD machines for industrial and scientific interests
- Deposition of metals and ceramic compounds on different substrates

1

Magnetron sputtering

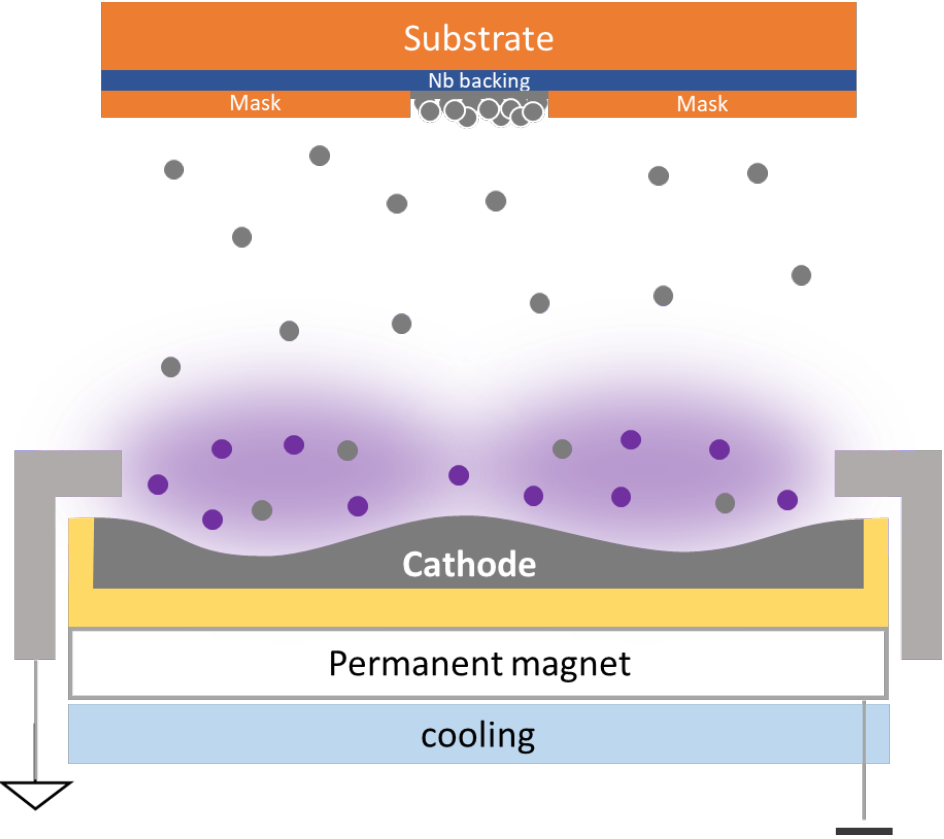
Planar magnetron sputtering



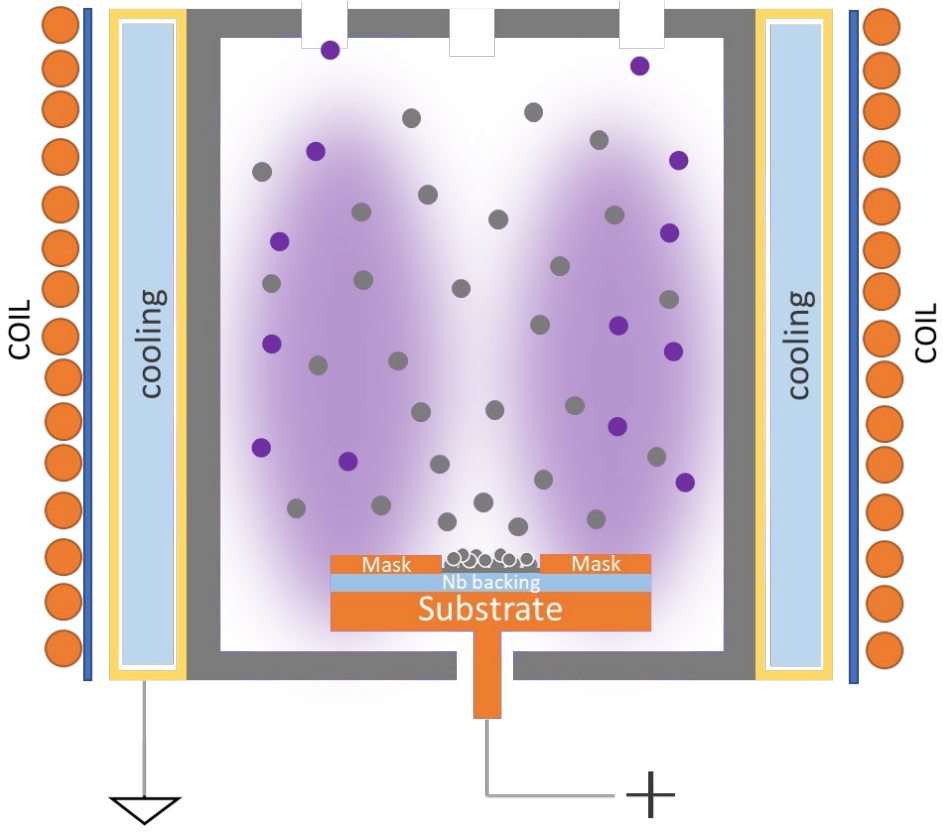
1

Magnetron sputtering

Planar magnetron sputtering



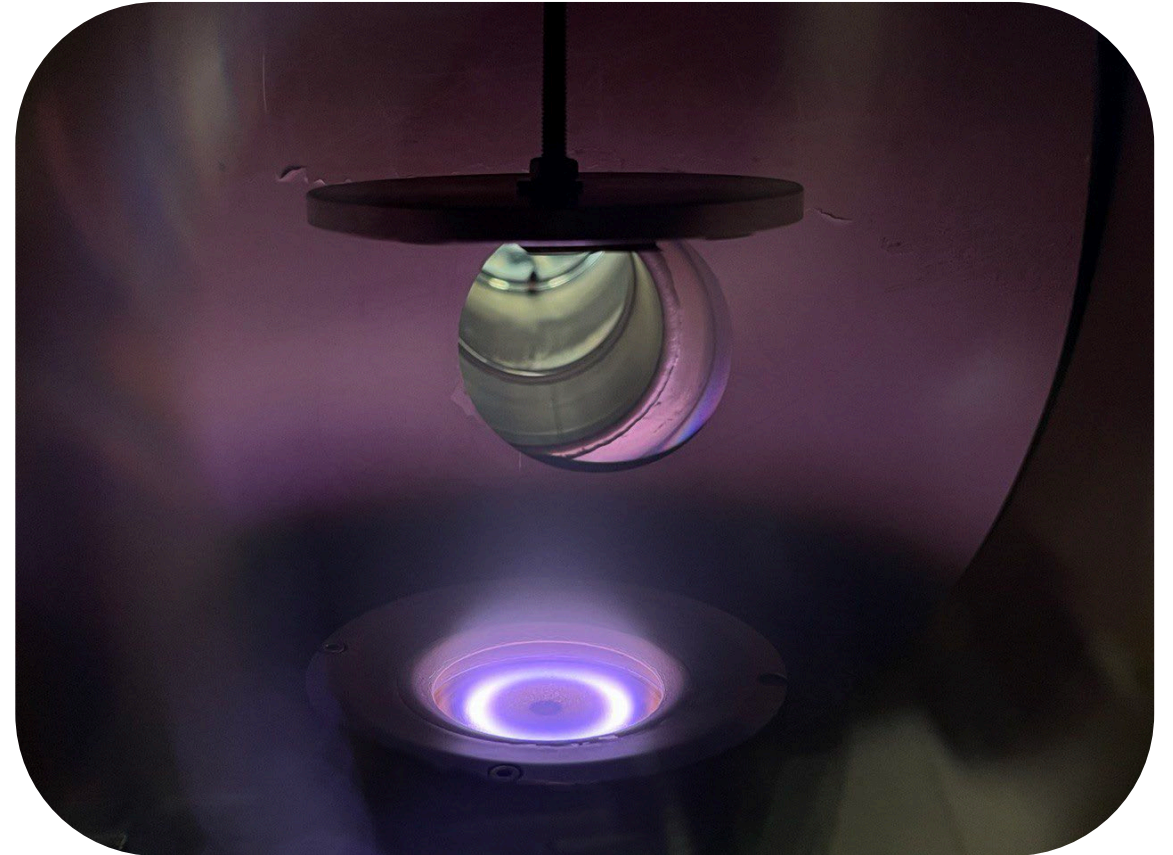
Idea of inverted magnetron sputtering



Magnetron sputtering - characteristics

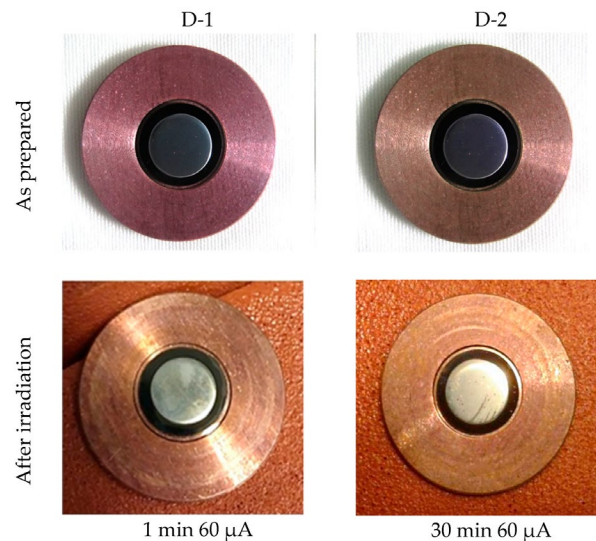
- ✓ Precise thickness control
- ✓ High adherence
- ✓ Densification of the deposition

- Material sputtered everywhere in the vacuum chamber
- Material losses – 80%



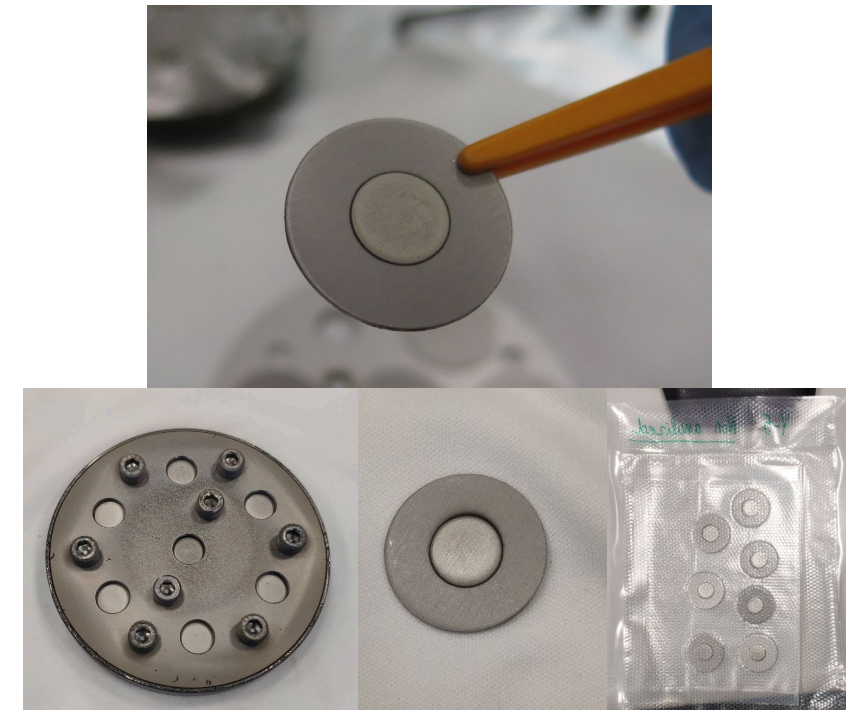
Magnetron sputtering for target production

nat-Mo targets preparation



[Patent n. WO 2019/053570]
[Skliarova et al., Molecules 2021]

nat-Y target preparation

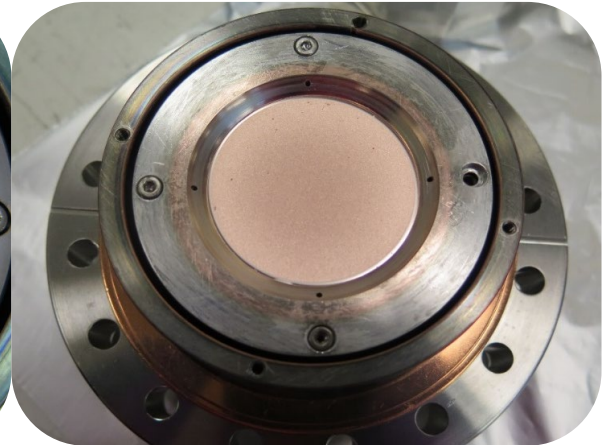
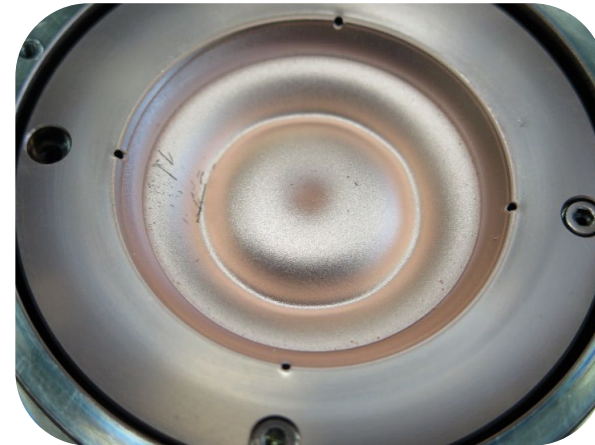
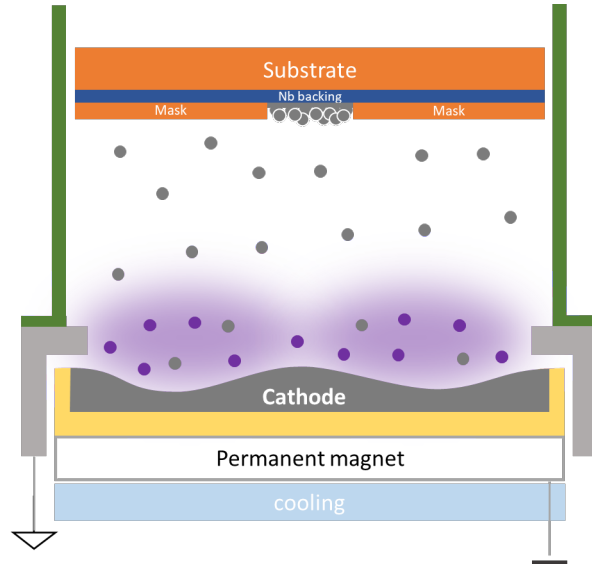
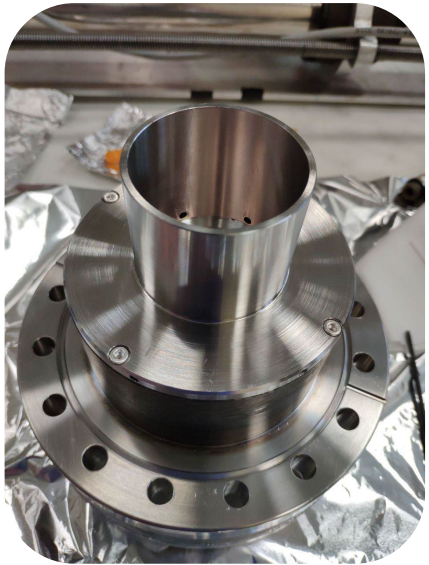


- 7 targets simultaneous depositions
- Thickness range – 150-200 μ m

2

Possible material-saving approach

2 inch planar magnetron evaluation

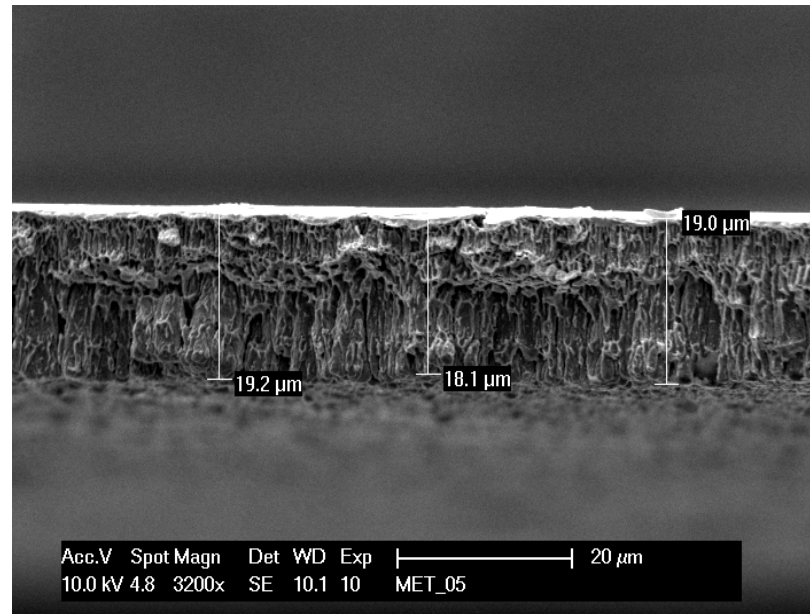
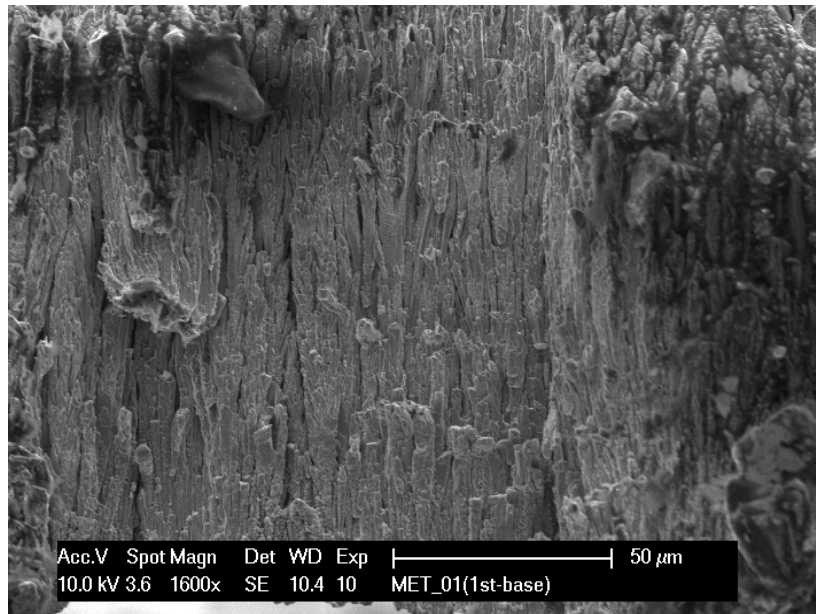


Recovering shield

Magnetron and diode sputtering techniques

Possible material-saving approach

Material results	Magnetron,%	Diode, %
On the substrate	38,9±0,003	21,6±0,035
On the shield	56,3±0,005	71,6±0,113
Losses	4,8±0,008	6,8±0,148

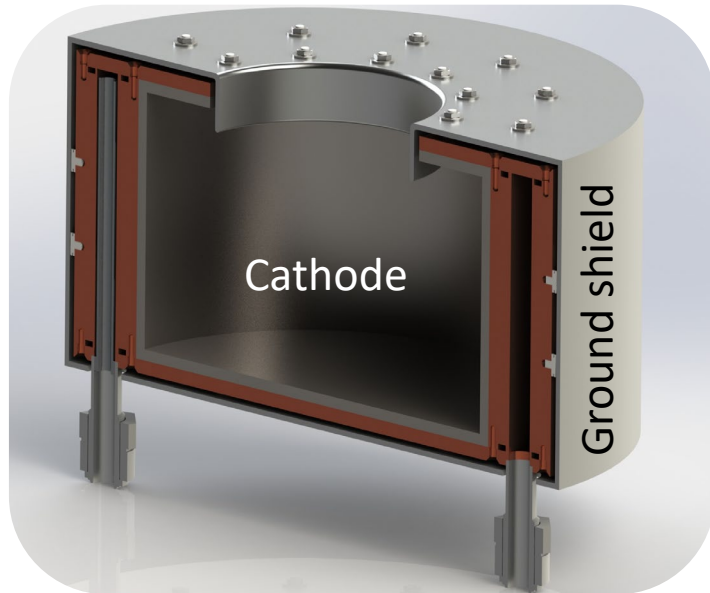


[Kotliarenko et al., Applied Science 2021]

Growing behaviour of MS and Diode depositions

2 Inverted magnetron idea

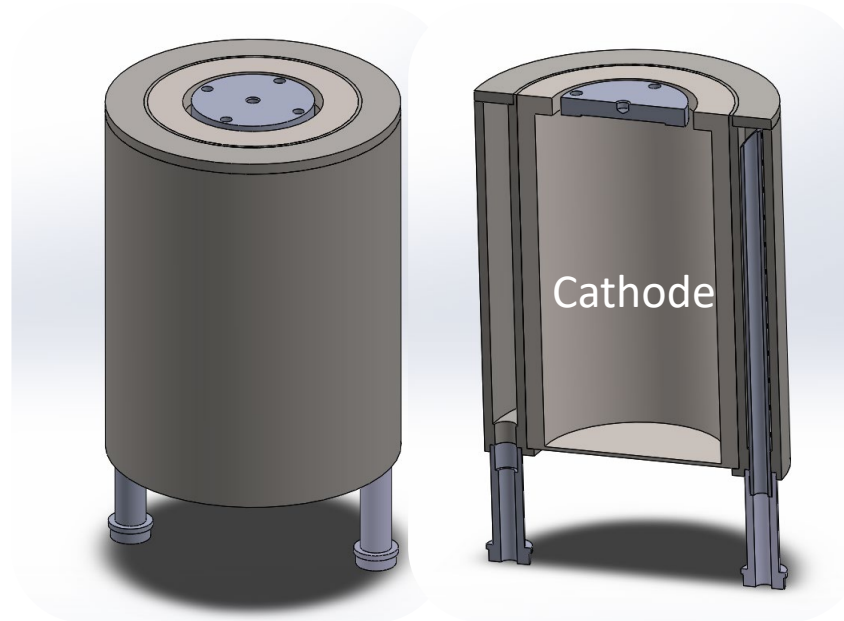
Initial design



Dimensions:

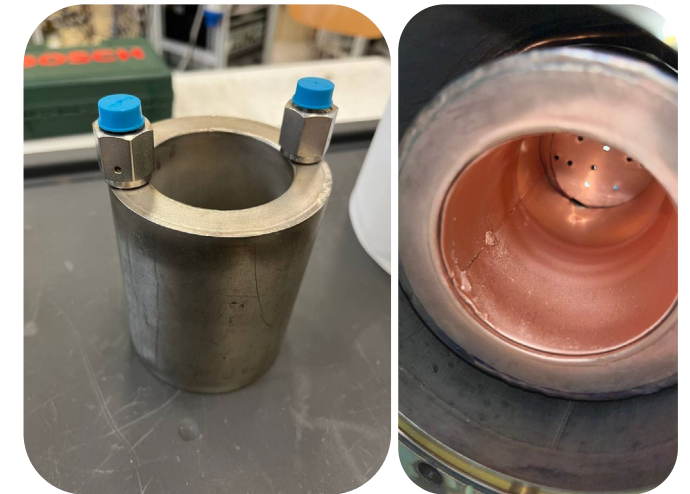
External **h** 80 mm **Ø** 160 mm
Cathode **h** 60 mm **Ø** 100 mm

Prototype design



- Simpler system
- Does not require shielding
- Easy maintenance

Produced prototype



Dimensions:

External **h** 106 mm **Ø** 89 mm
Cathode **h** 106 mm **Ø** 59 mm

2

Inverted magnetron - proof of concept

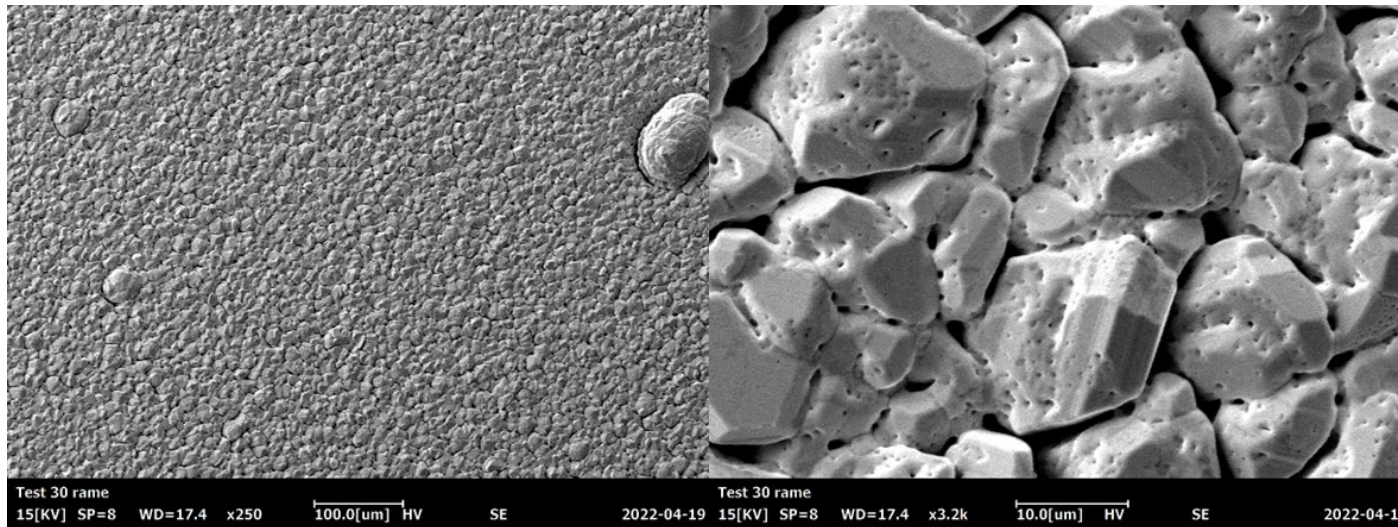
Deposition parameters

Gas flows: Ar – 3,5 sccm

Power: I_{const} 1 A;
U ~500 V

Time: 150 min

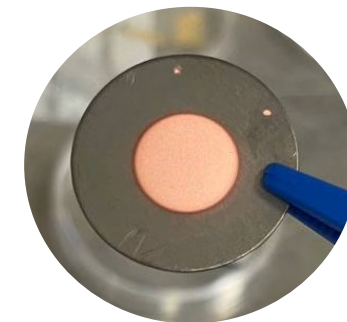
Coil power: 5,5 A



Morphology of Cu deposition

DONE

TEST 30



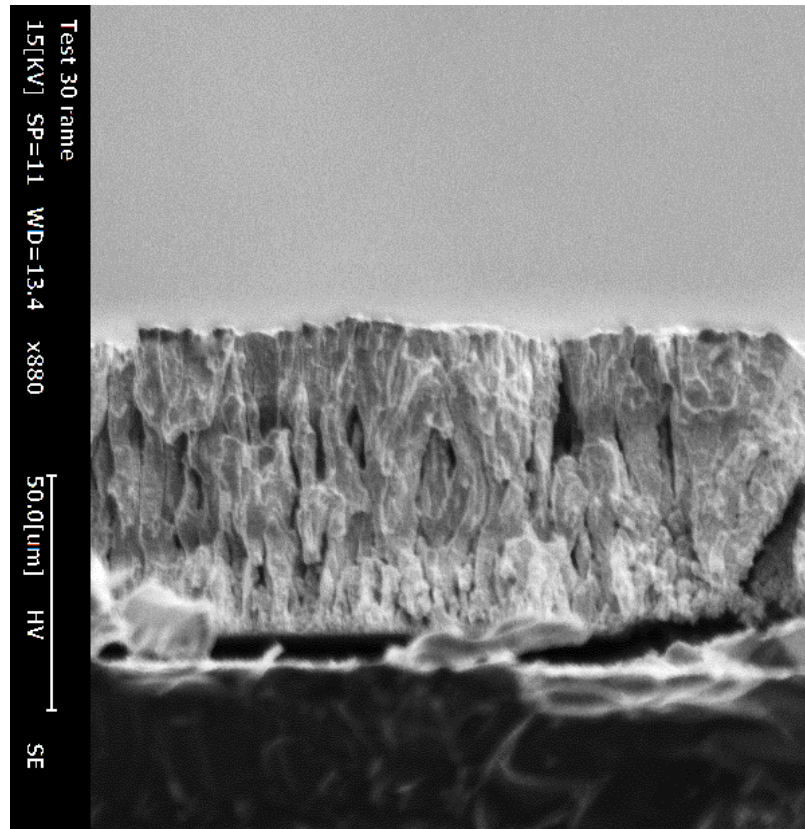
Cu deposition:

Ø10 mm x 40 µm

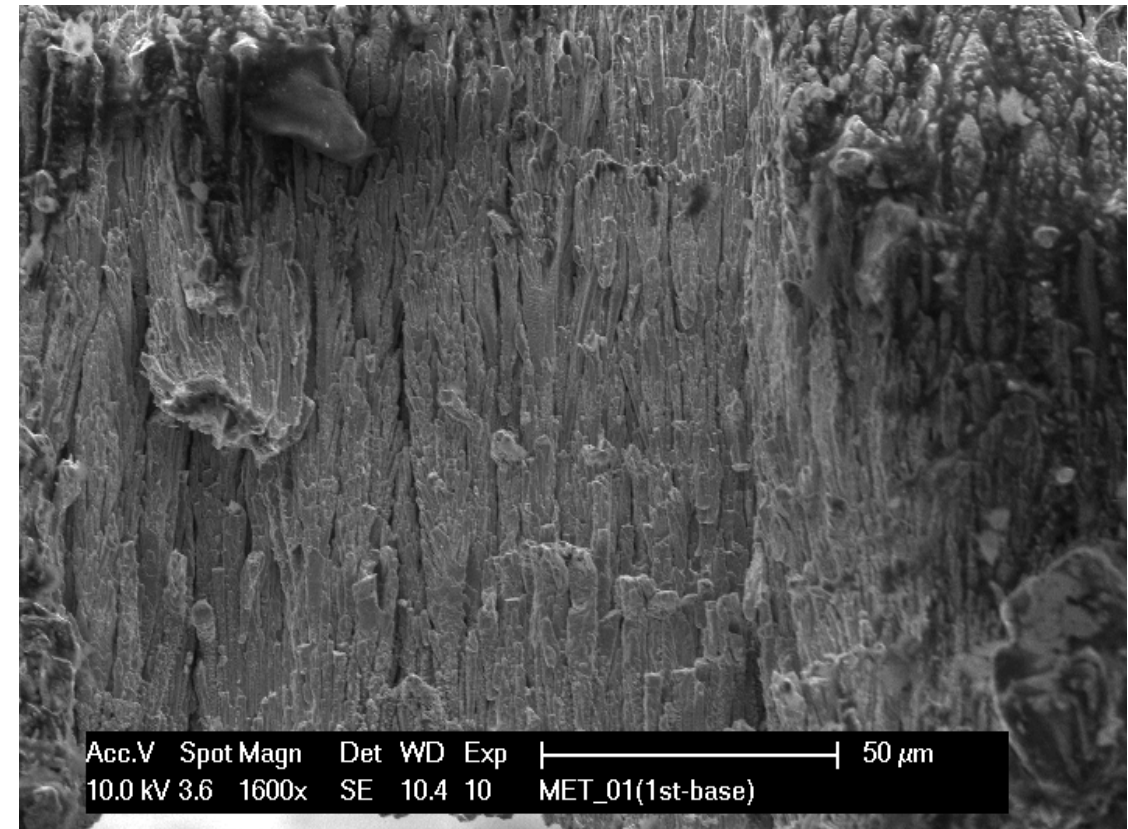
Nb: Ø24 x 0,5 mm

Inverted magnetron – standart magnetron

Inverted MS



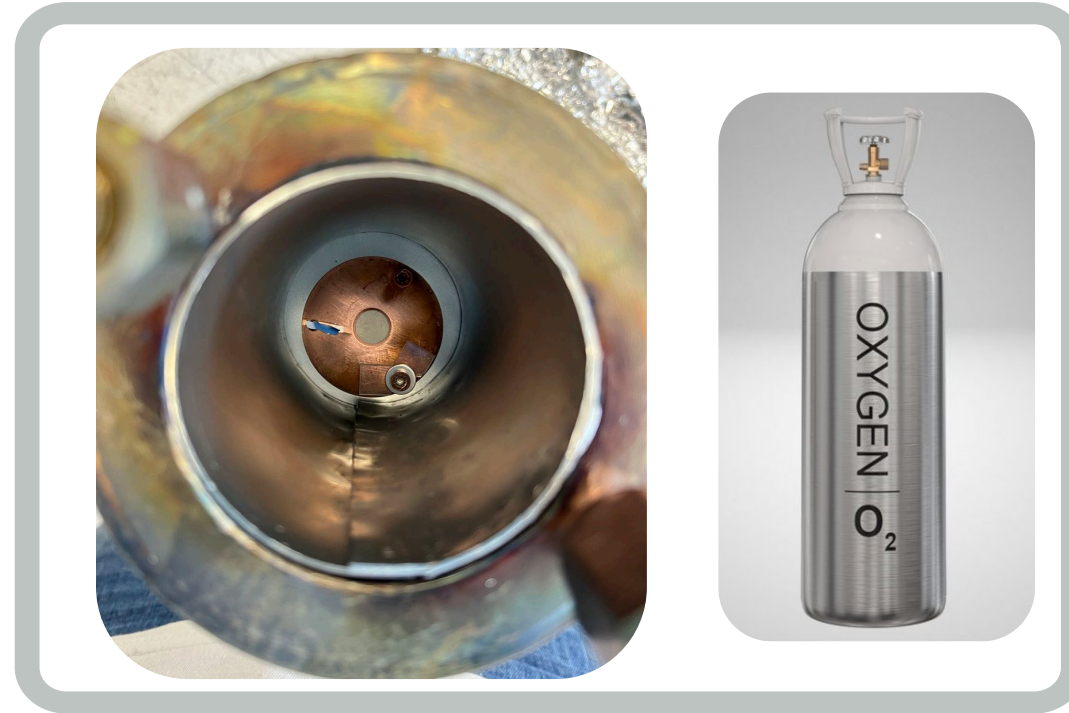
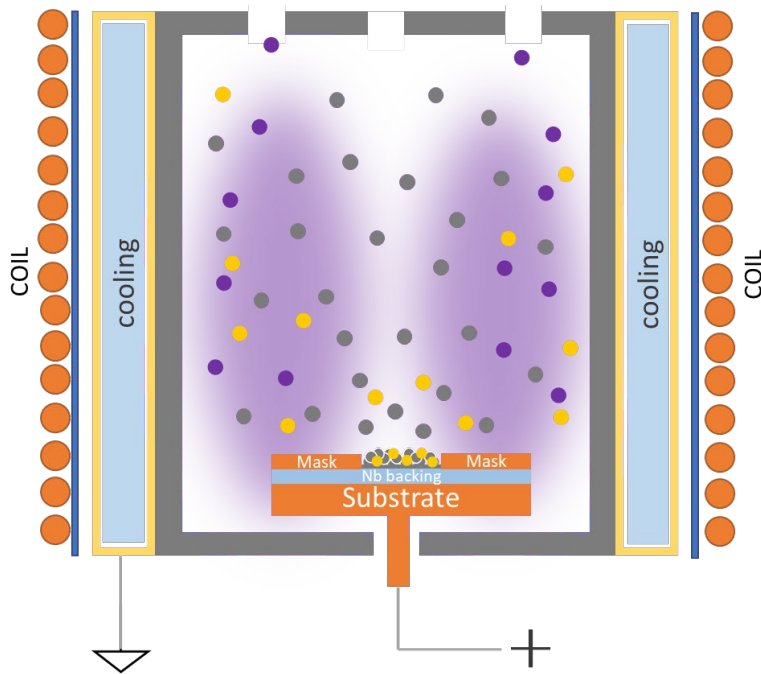
Standard MS



Growing behaviour of Cu deposition

3 nat-ZnO deposition test

Reactive sputtering process



Zn cathode

Reactive gas – O₂

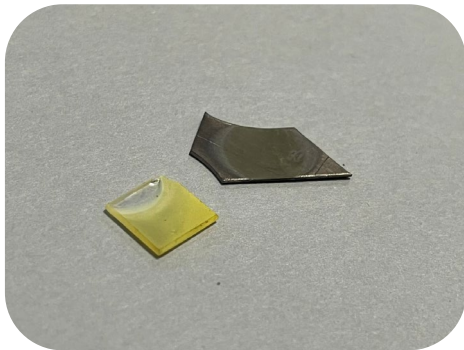


Reactive process of ZnO

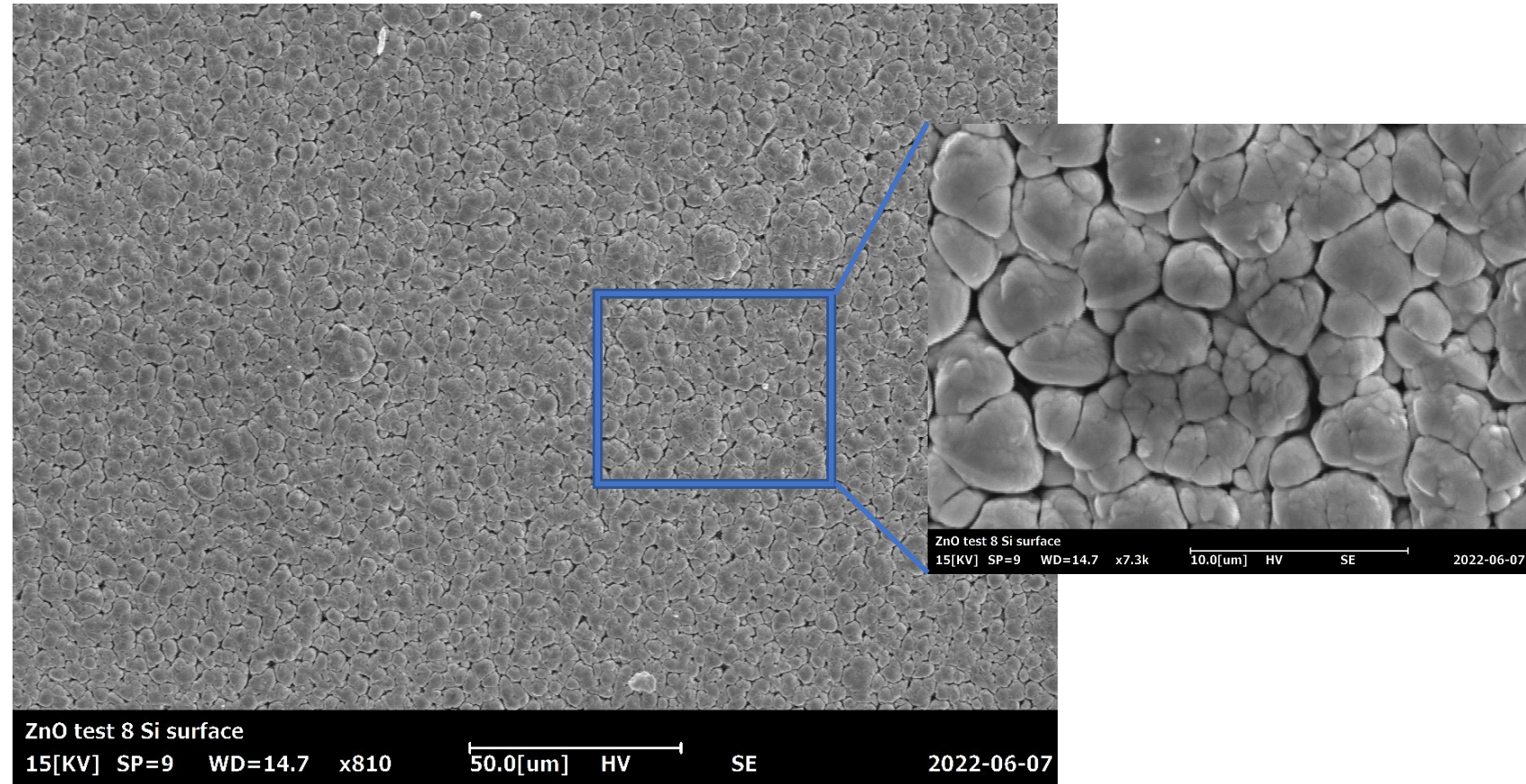
nat-ZnO deposition test - results

TEST ZnO - 8

Gas flows: Ar - 4 sccm
 O₂ - 2 sccm
Power: I_{const} 0,7 A;
 U ~450 V
Time: 115 min
Coil power: 6 A



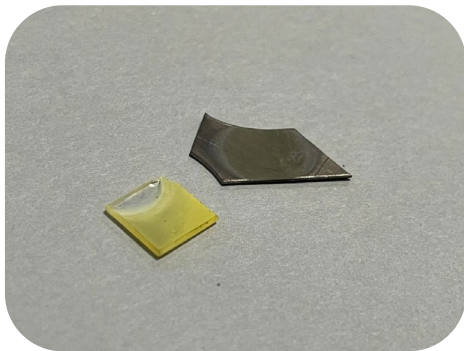
Morphology of ZnO deposition



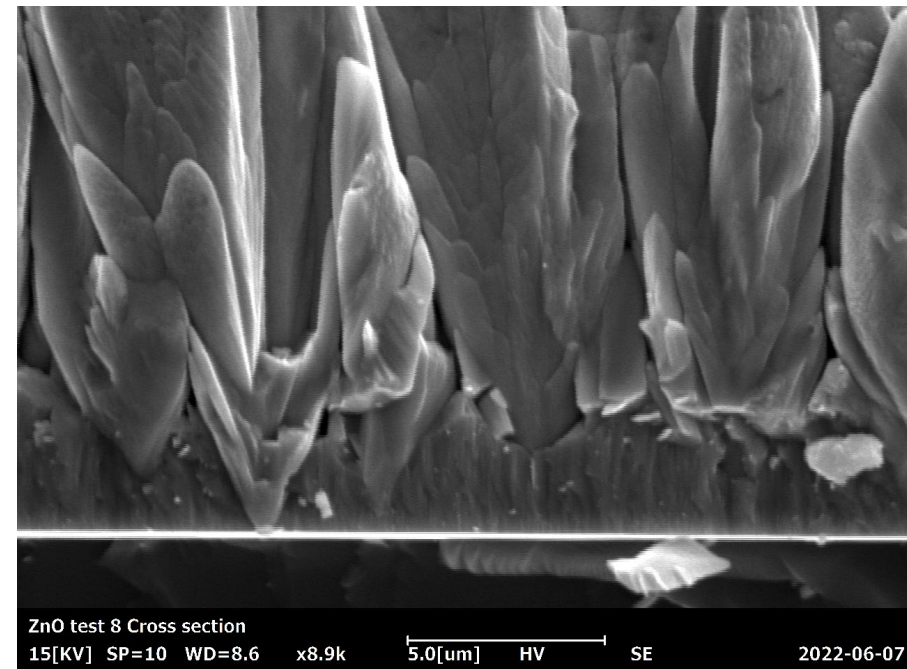
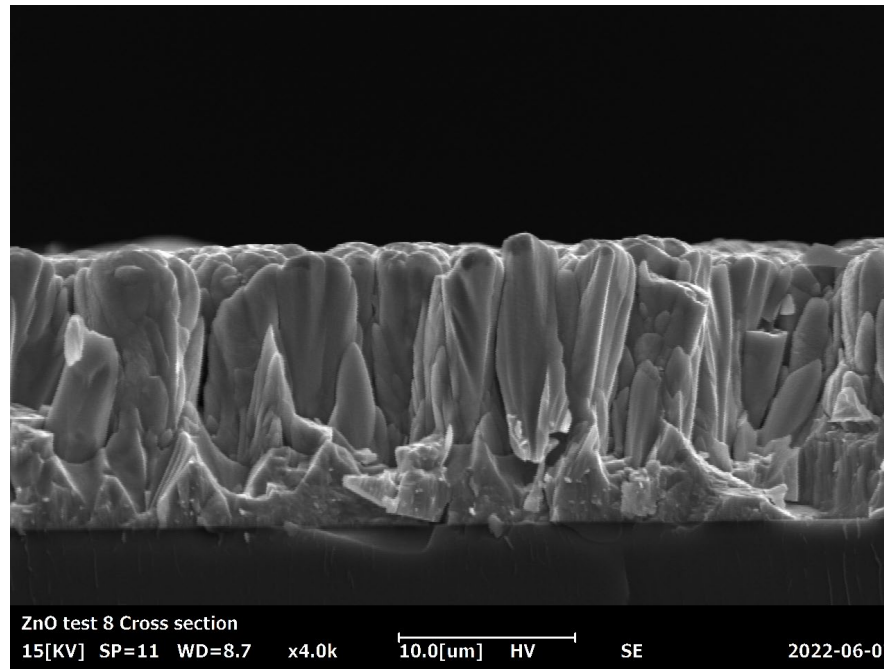
nat-ZnO deposition test - results

TEST ZnO - 8

Gas flows: Ar - 4 sccm
 O₂ - 2 sccm
Power: I_{const} 0,7 A;
 U ~450 V
Time: 115 min
Coil power: 6 A



Polycrystalline growing behaviour of ZnO deposition



Deposition rate: 130 nm/min

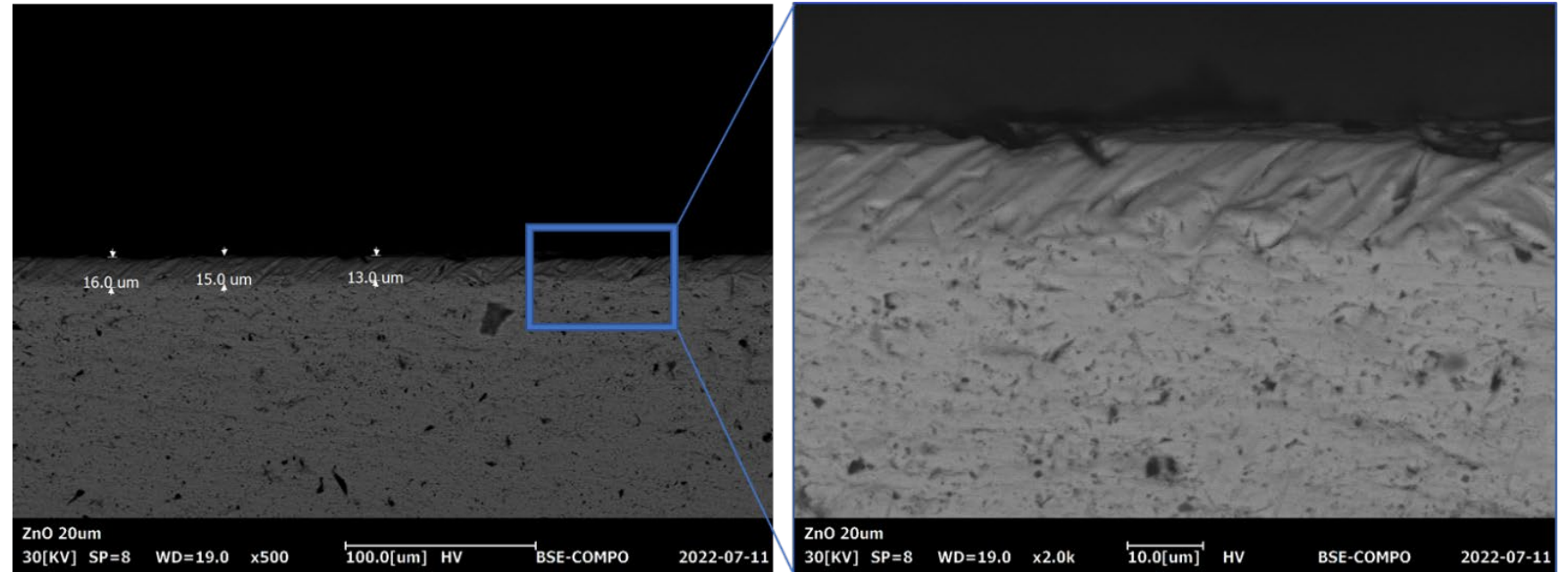
nat-ZnO deposition test - results

Interface analysis



Sample preparation

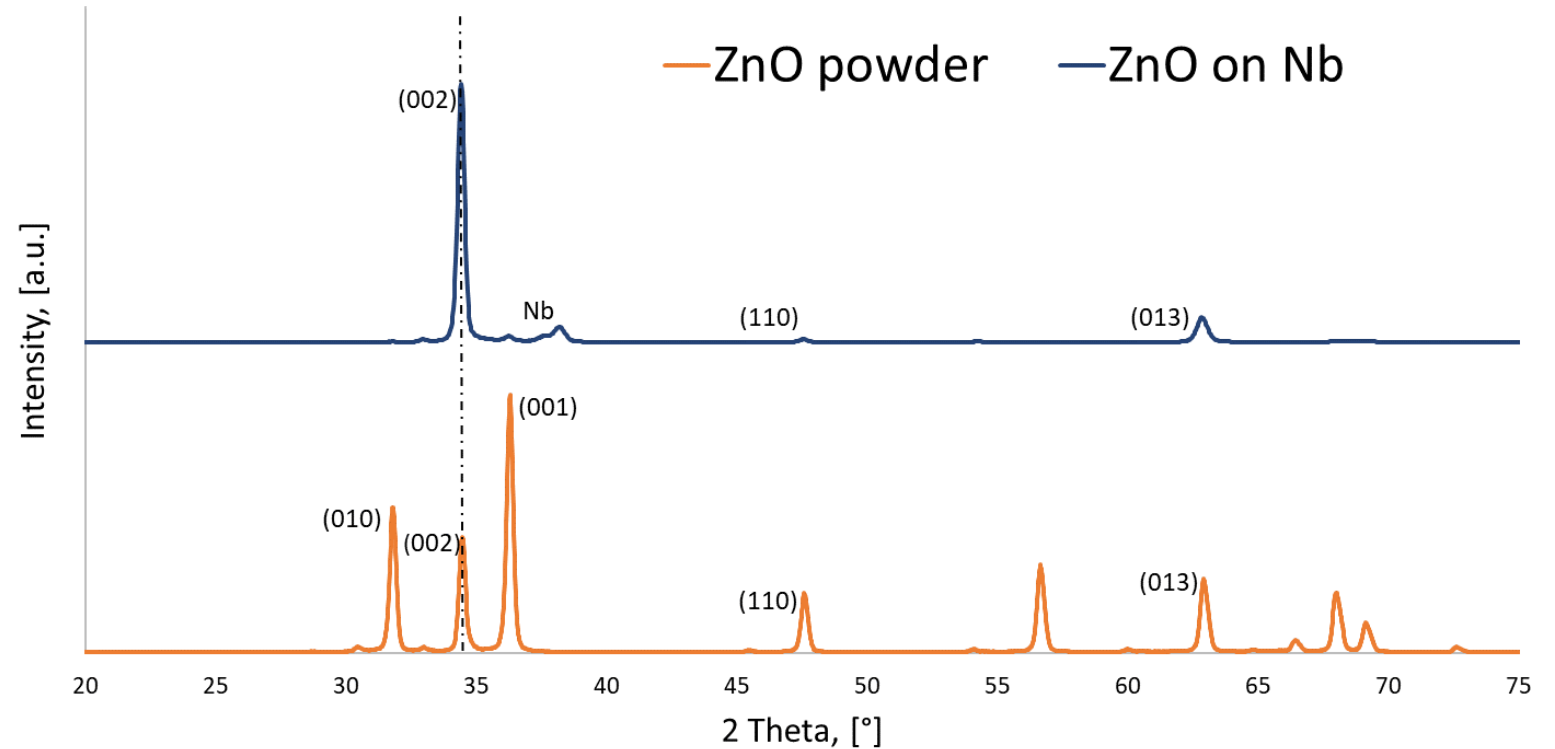
Cutting with electro erosion and step-lapping with different abrasive papers



High adhesion of ZnO to Nb

nat-ZnO deposition test - results

- Crystalline structure
- Similar to nat-ZnO powder
- Preferential (002) crystal growing



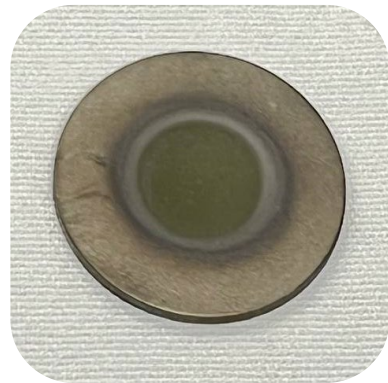
XRD diffractogram

3

nat-ZnO deposition test - results

Sputtering parameters

Gas flows: Ar - 4 sccm
 O₂ - 2 sccm
Power: I_{const} 0,7 A;
 U ~450 V
Time: 130 min
Coil power: 6 A



20 μm

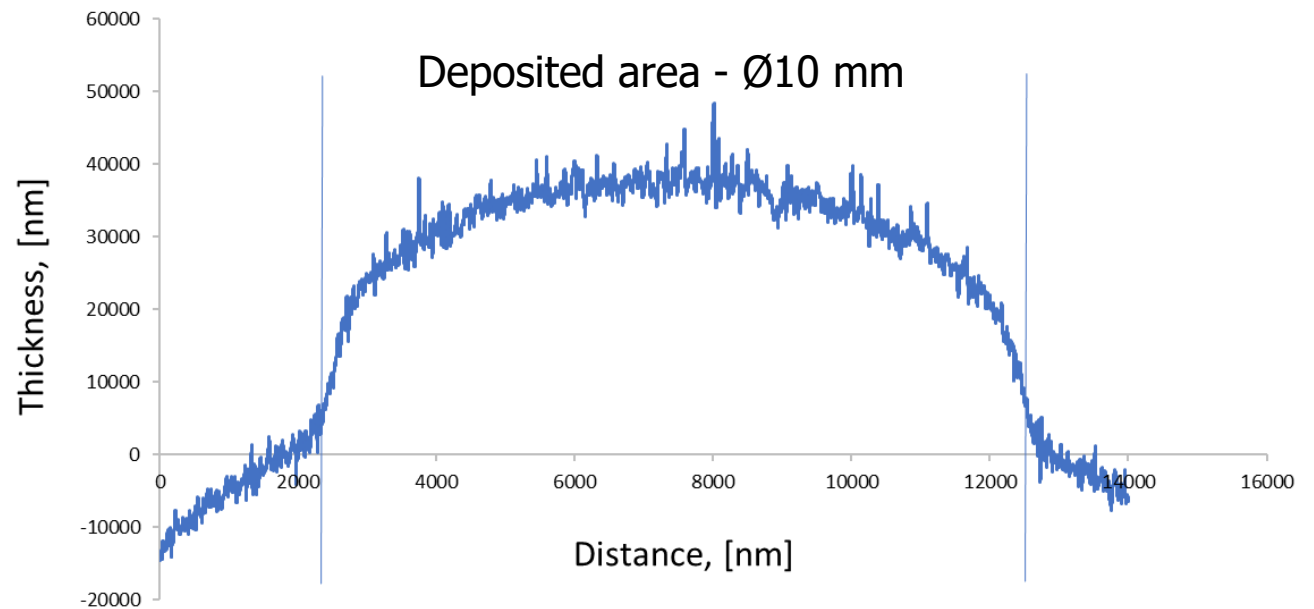
Gas flows: Ar - 4 sccm
 O₂ - 2 sccm
Power: I_{const} 0,7 A;
 U ~500 V
Time: 150 min
Coil power: 6 A



30 μm



Thickness uniformity measurements by profilometer



3

nat-ZnO deposition test - results

Targets parameter

ZnO deposition:

Ø10 mm x 20 µm;

$\rho_{\text{ZnO}} \sim 73 \%$ bulk

Nb: Ø23.7 x 1,2 mm

Areal $\rho \sim 8,4 \text{ mg/cm}^2$



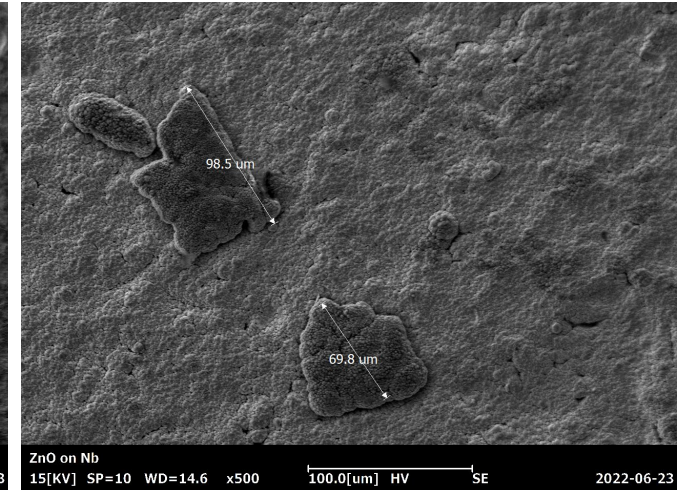
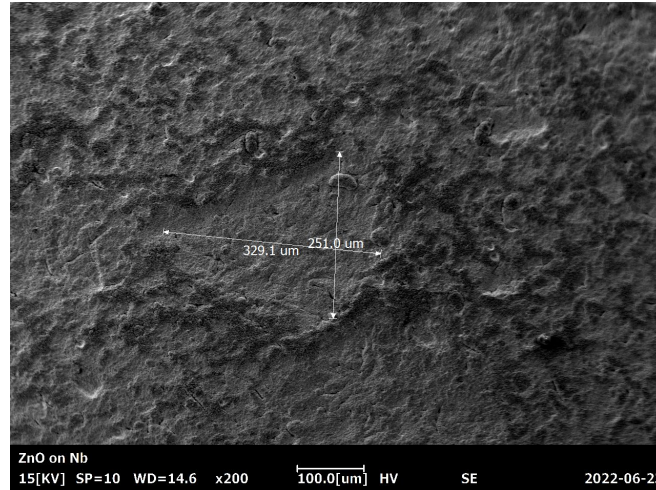
ZnO deposition:

Ø10 mm x 30 µm;

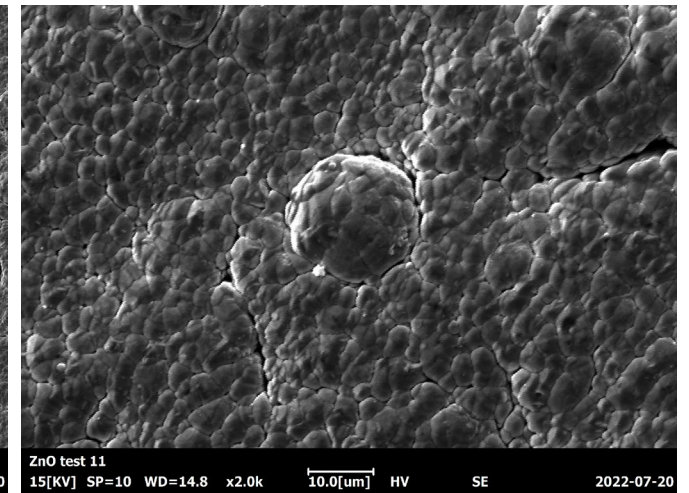
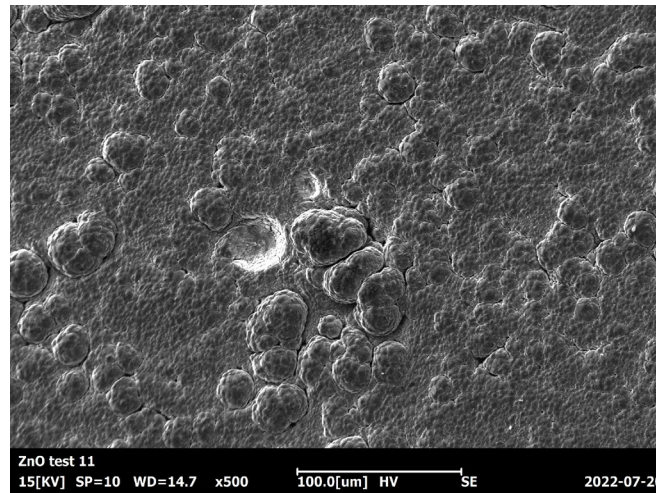
$\rho_{\text{ZnO}} \sim 71 \%$ bulk

Nb: Ø23.7 x 1,2 mm

Areal $\rho \sim 12,1 \text{ mg/cm}^2$



Morphology of ZnO targets



Targets parameter

ZnO deposition:
 $\text{Ø}10 \text{ mm} \times 20 \text{ }\mu\text{m}$;
 $\rho_{\text{ZnO}} \sim 73 \%$ bulk
Nb: $\text{Ø}23.7 \times 1,2 \text{ mm}$
 Areal $\rho \sim 8,4 \text{ mg/cm}^2$



ZnO deposition:
 $\text{Ø}10 \text{ mm} \times 30 \text{ }\mu\text{m}$;
 $\rho_{\text{ZnO}} \sim 71 \%$ bulk
Nb: $\text{Ø}23.7 \times 1,2 \text{ mm}$
 Areal $\rho \sim 12,1 \text{ mg/cm}^2$



Irradiation parameter

Energy 19 MeV,
 p+ current 10 μA
 5 min

Thermo-mechanical irradiations tests



Energy 19 MeV,
 p+ current 20 μA
 5 min



Conclusions

- Evaluation of sputtering techniques from the material point of view
- Realization of recovering shield for standard magnetron deposition

- An alternative sputtering configuration proposed and realized
- First nat-ZnO targets realization by inverted magnetron technique



Is the magnetron sputtering technique suitable for ZnO target production?

Thank you for your attention!

Conclusions

- Evaluation of sputtering techniques from the material point of view
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Is the magnetron sputtering technique suitable for ZnO target production?

WORK IN PROGRESS

Thank you for your attention!



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