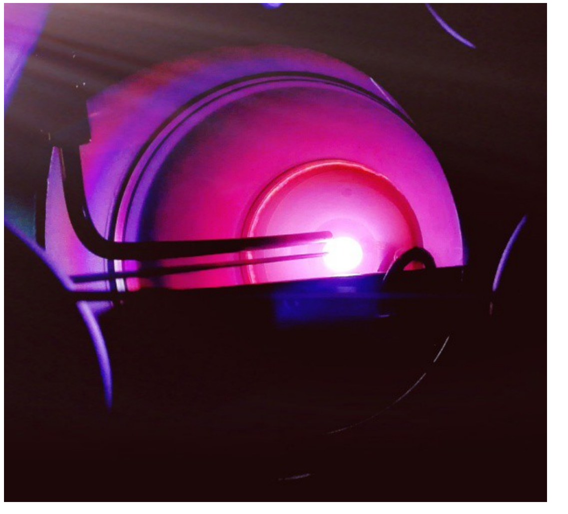
Optimization of magnetron sputter-deposition process of thin film coatings on a moving cylindrical substrate



PhD. Student Seminar 07/04/2016

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UGITECH
Providing special steel solutions

Outline

Context

→ Why do we want to coat wire ?
→ Scientific issues

Employed technology

→ Main specifications of reactive sputtering in an inverted cylindrical magnetron (ICM)

Commercial magnetron

→ Characterization of the first used commercial magnetron

Ugitech made magnetron

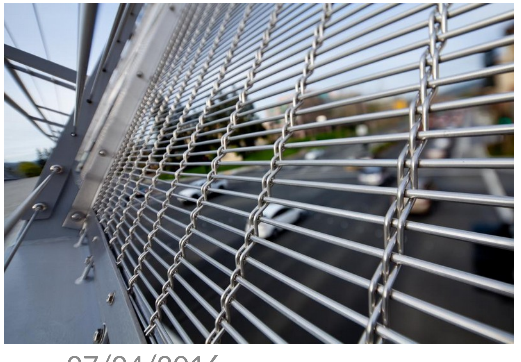
→ Characterization of the magnetron designed by the company

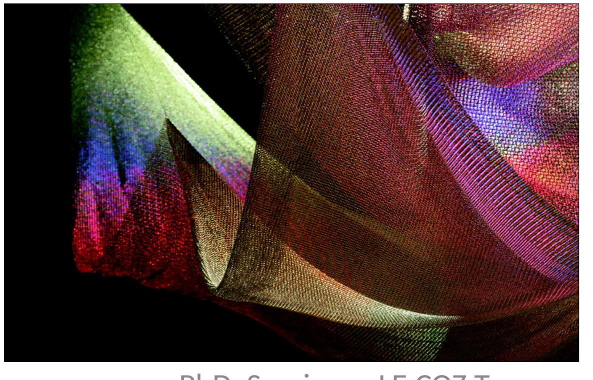
Conclusions and Forthcoming work

Context

Wide amount of stainless steel wire used in many fields :

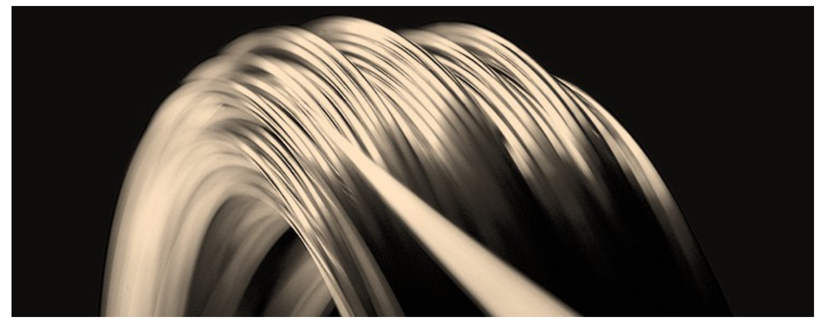
→ Architecture
→ Urbanization
→ Art
→ Cold heading
→ Automotive
…





Context

Scientific issues

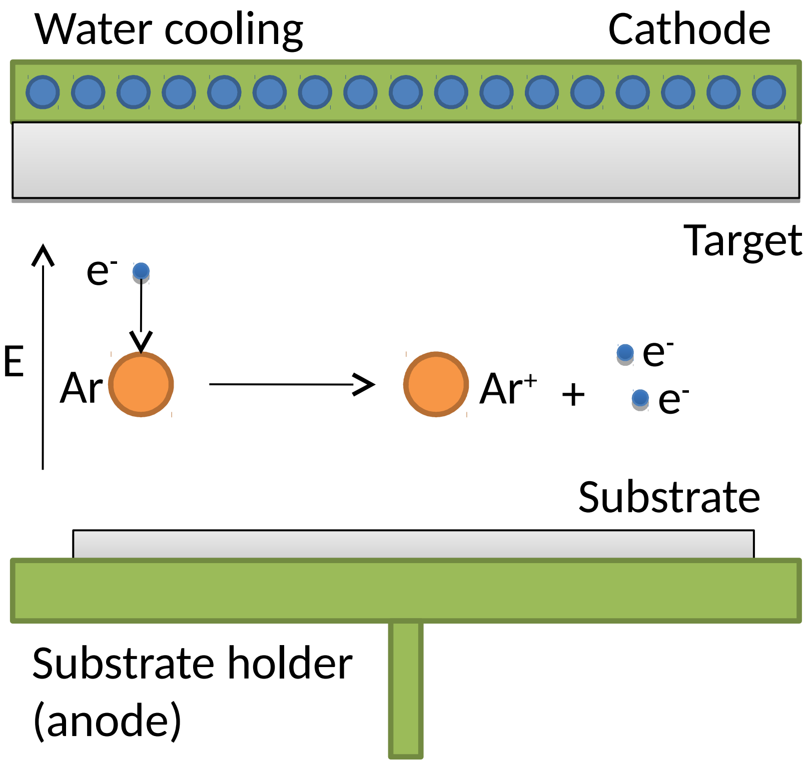


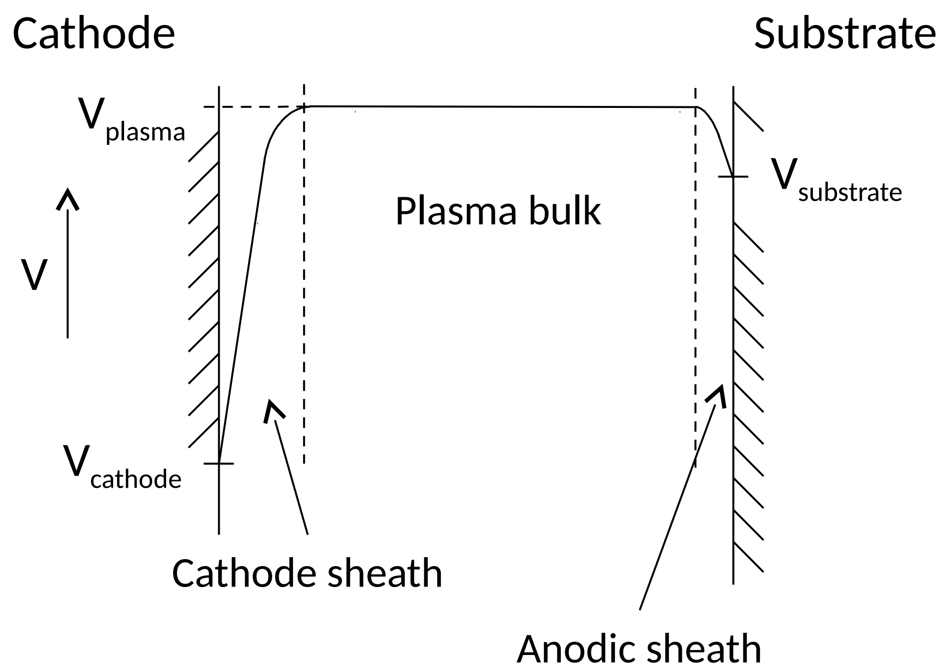
Sizing the system for :
→ Fast roll-to-roll process
→ Uniform coatings
→ High quality coatings

Considerations :
→ Geometry
→ Electric
→ Magnetic
→ Thermal

Employed technology

Physical Vapor Deposition (PVD)





→ Ionization of argon atoms by surrounding electrons

→ Potential drop in the sheath accelerates the ions used to sputter the target

Employed technology

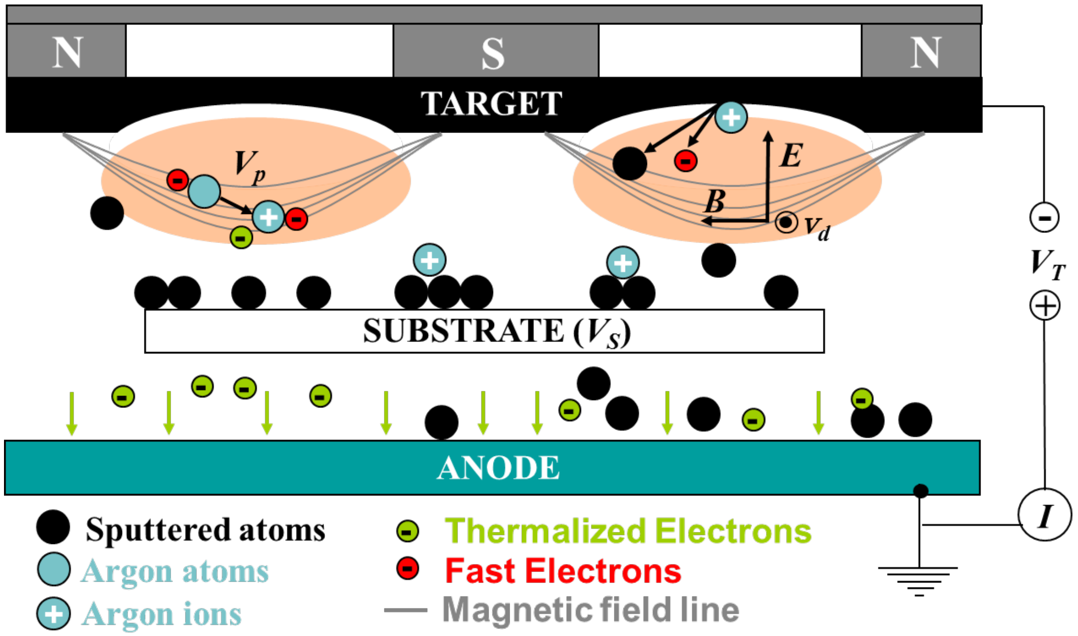
Magnetron sputtering

→ Plasma close to the target

→ High density (10^{10} – 10^{11} cm^{-3})

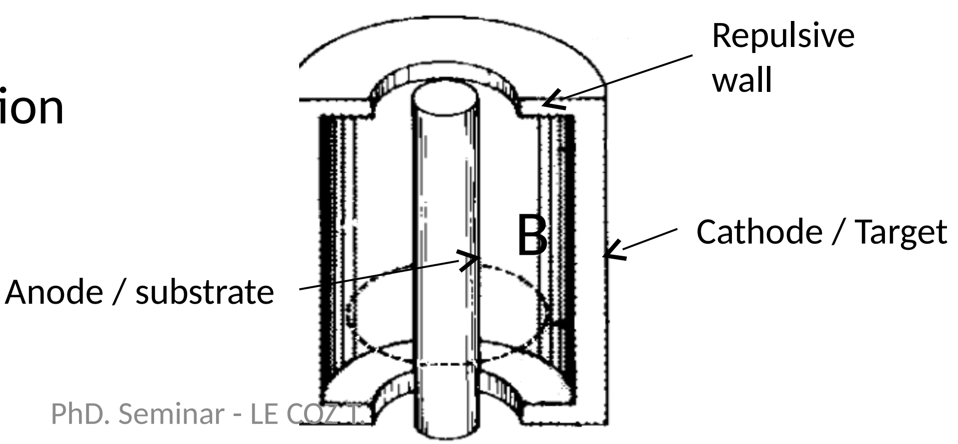
→ Low pressure (0.1 Pa)

→ Race tracks leading to non-homogeneous coatings



Inverted cylindrical configuration

→ Conformal geometry
→ No loss of matter on the walls



Employed technology

Reactive sputtering

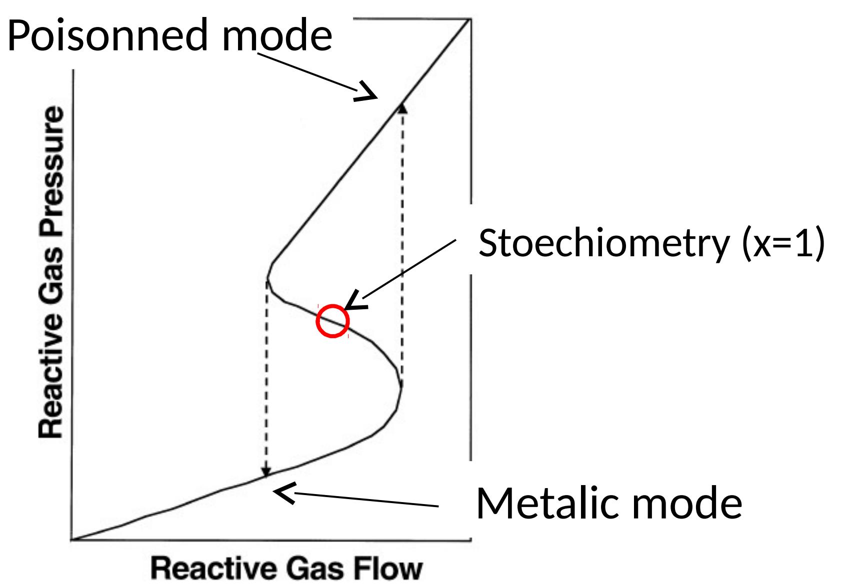


Fig. 1. Typical hysteresis behavior found in reactive sputtering. When only the reactive gas flow is controlled, the partial pressure jumps as shown by dotted lines and hysteresis is seen. With feedback the entire curve can be measured.

→ Argon plasma
→ Nitrogen as reactive gas
→ Titanium target

→ Formation of TiN_x on target and substrate

→ Instability of the stoechiometric point

Lindberg et al., « Reactive deposition of nitrides and oxides using a twin-cathode inverted cylindrical magnetron », Surface and Coatings Technology 133-134 (2000) 484-488.

Employed technology

Reactive sputtering

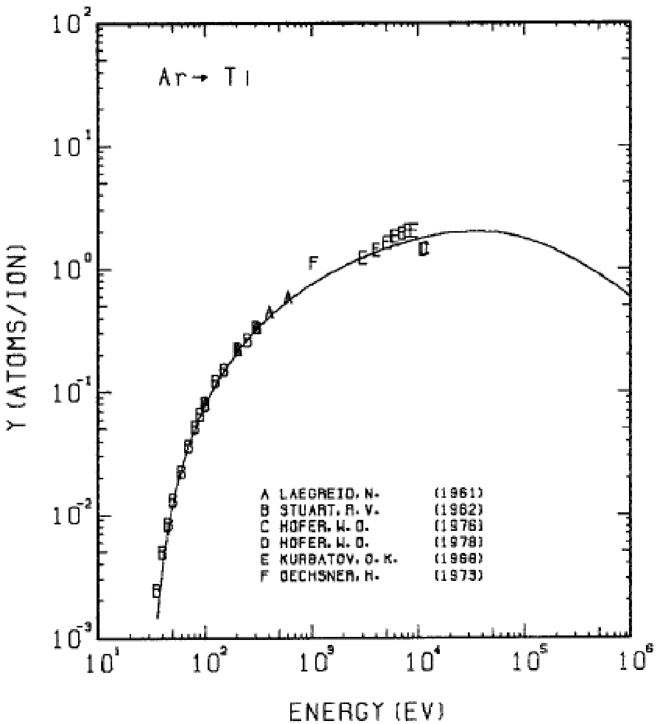


FIG. 50 ENERGY DEPENDENCE OF THE SPUTTERING YIELD OF TI WITH AR⁺.

Atomic Data and Nuclear Data Tables, Vol. 62, No. 2, March 1996

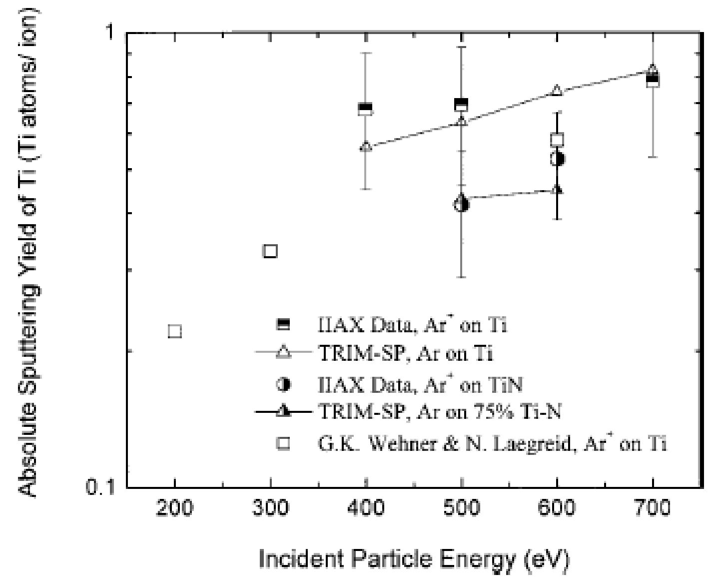


FIG. 2. Absolute sputtering yield of Ti data from Ar^+ bombardment on Ti and TiN targets in the range of 200–700 eV at normal incidence and TRIM.SP simul

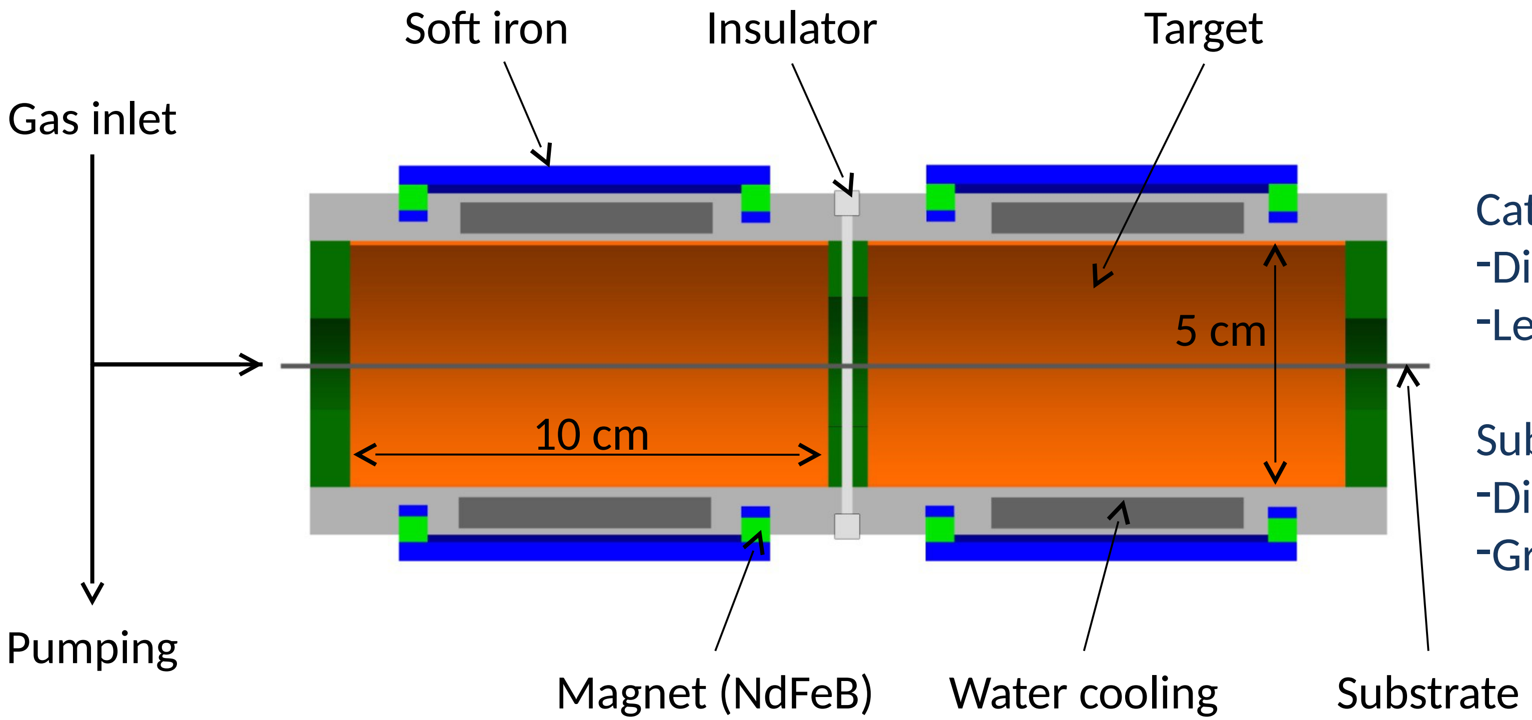
	Energy (eV)	IIAX (atoms/ion)	TRIM.SP (±3%)
Ar^+ on Ti	400	0.676±0.227	0.560
	500	0.694±0.234	0.634
	600		0.741
	700	0.782±0.248	0.827
Ar^+ on TiN	500	0.417±0.129	0.429
	600	0.527±0.142	0.450

J. Vac. Sci. Technol. A, Vol. 19, No. 3, May/Jun 2001

→ Sputtering yield depends on the material

Commercial magnetron

Geometry



Cathode :
- Diameter : 5 cm
- Length : 2*10 cm

Substrate :
- Diameter : 2 mm
- Grounded

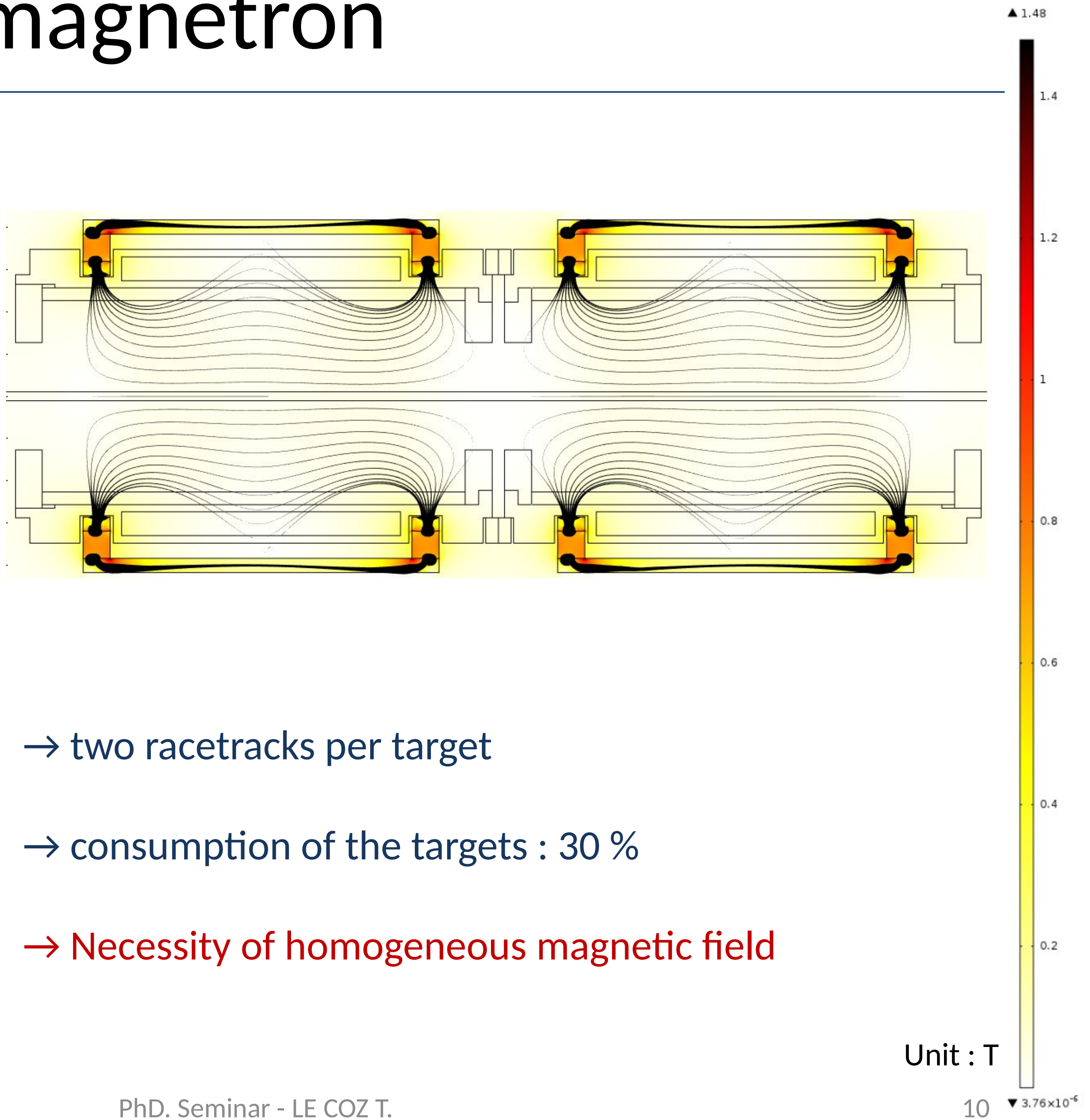
→ Two isolated targets for RF sputtering – we use DC sputtering

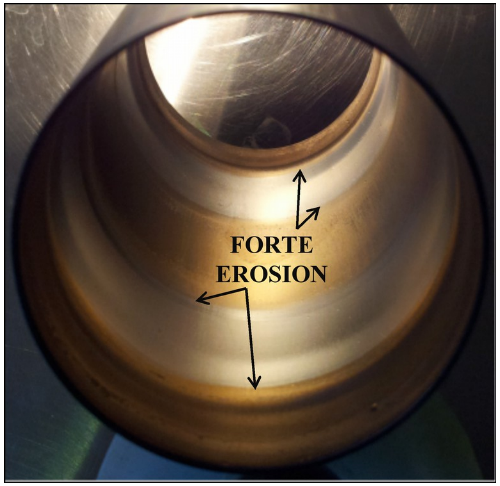
Commercial magnetron

Magnetic configuration

→ 4 rings of 30 magnets

→ heterogeneity of the magnetic field





→ two racetracks per target

→ consumption of the targets : 30 %

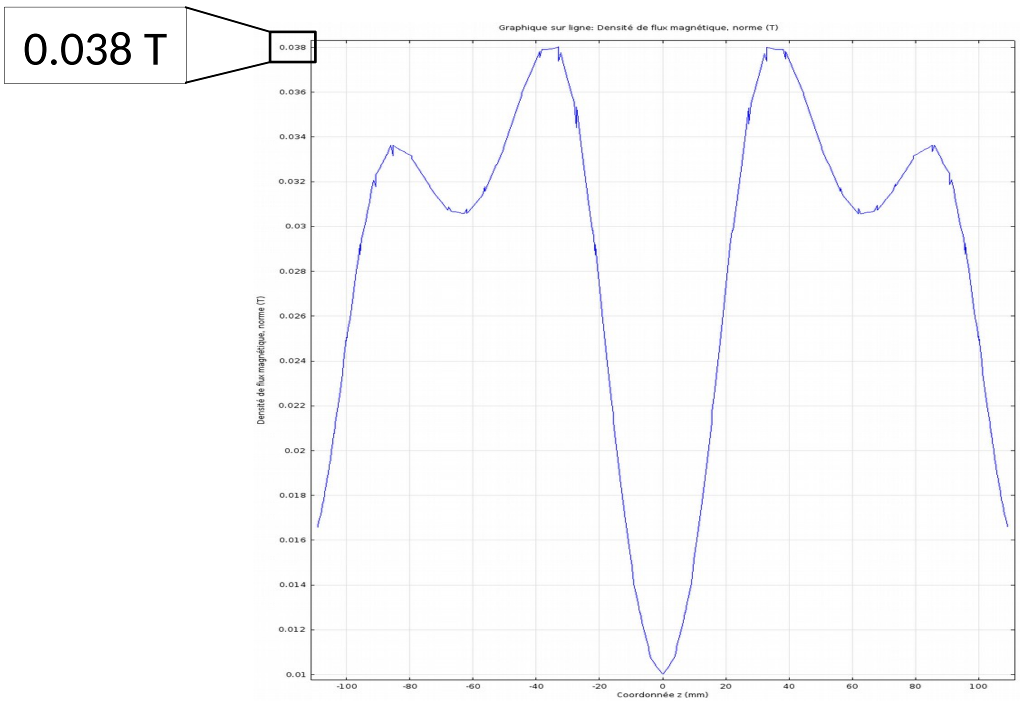
→ Necessity of homogeneous magnetic field

Unit : T

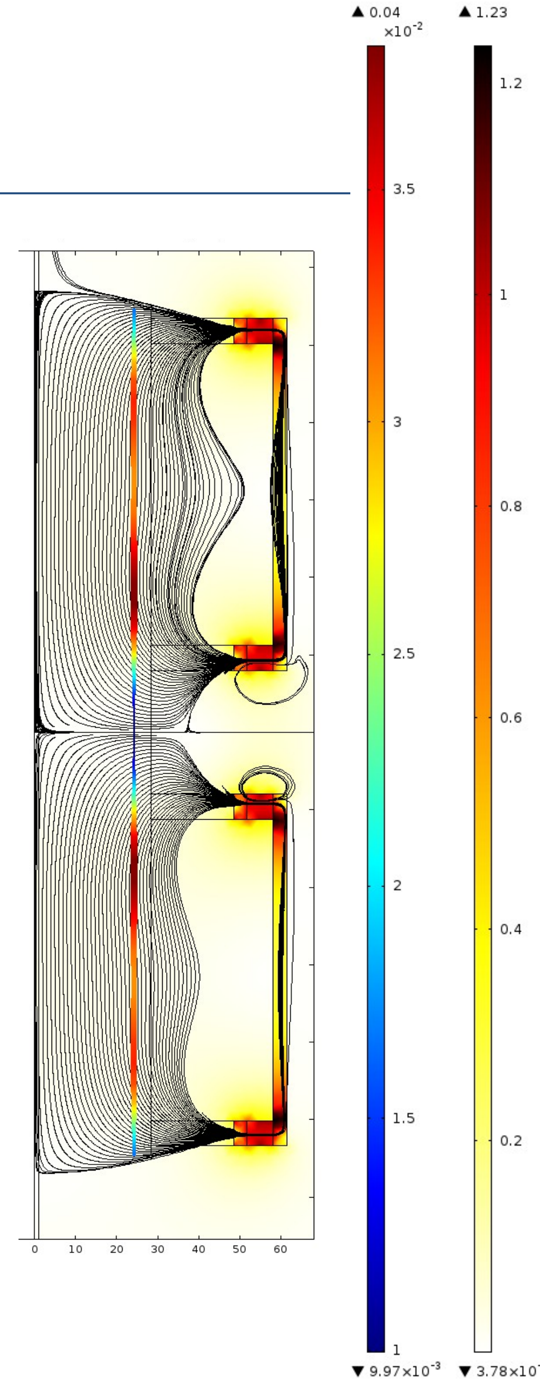
Commercial magnetron

Magnetic configuration

→ Two ways to get a homogeneous magnetic field :
- increase the diameter of the magnetic rings
- use a coil



Magnetic field intensity on the target surface



Unit : T

Commercial magnetron

Magnetic configuration

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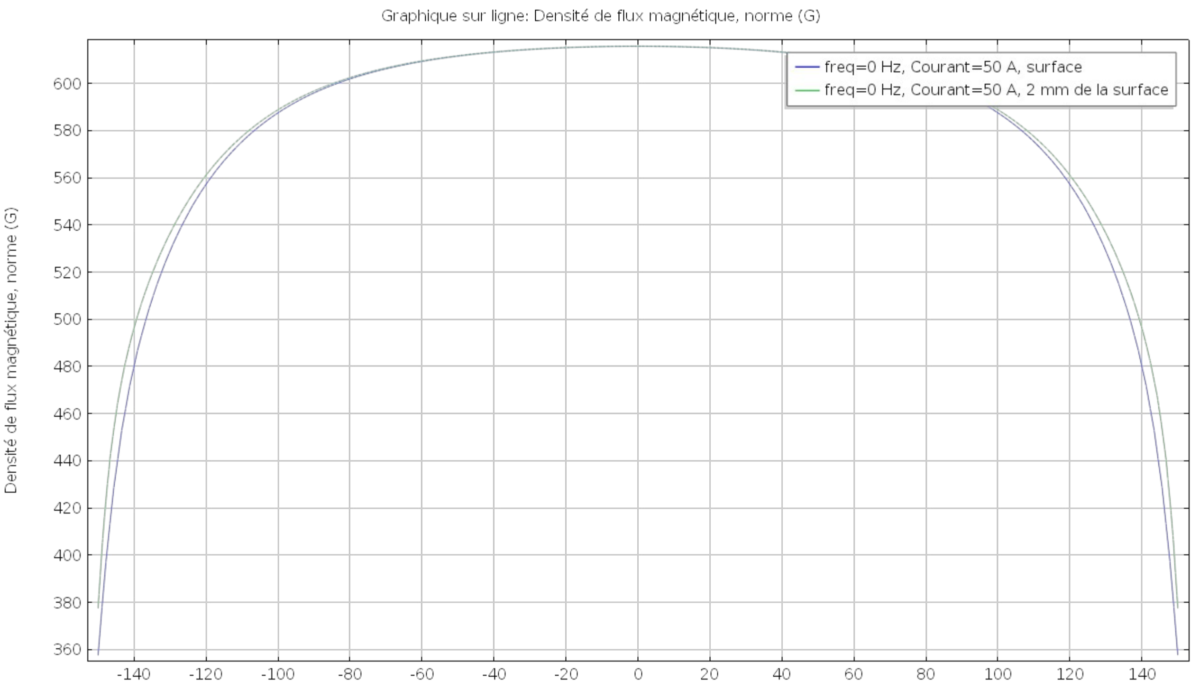
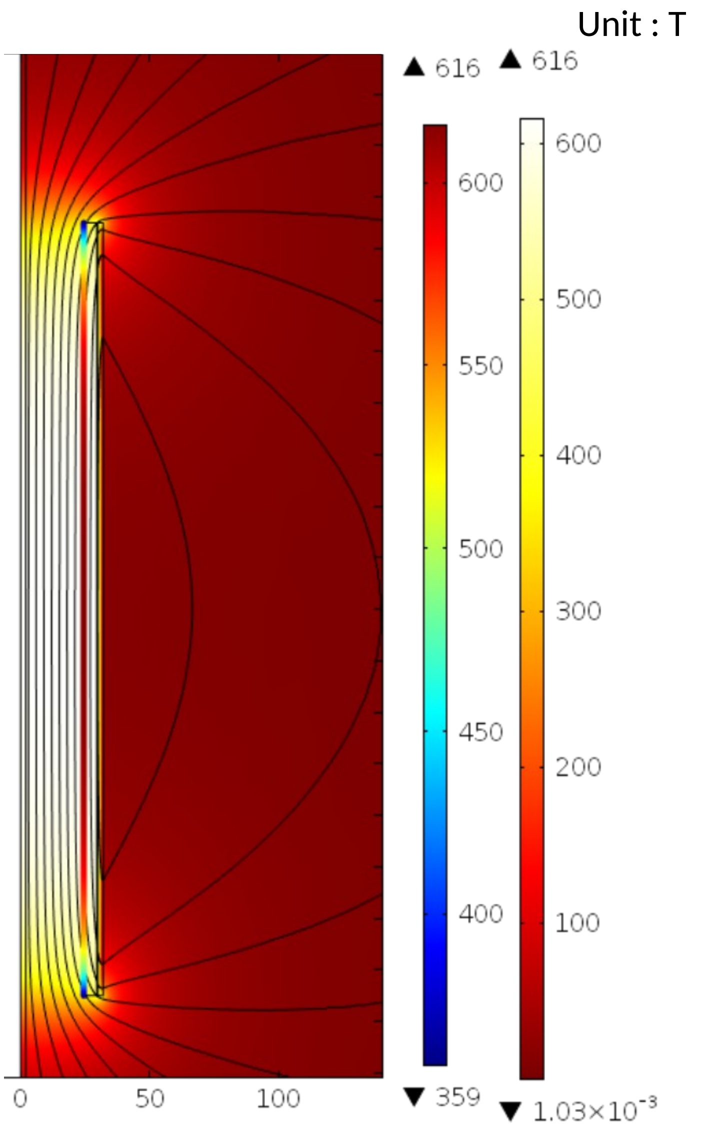
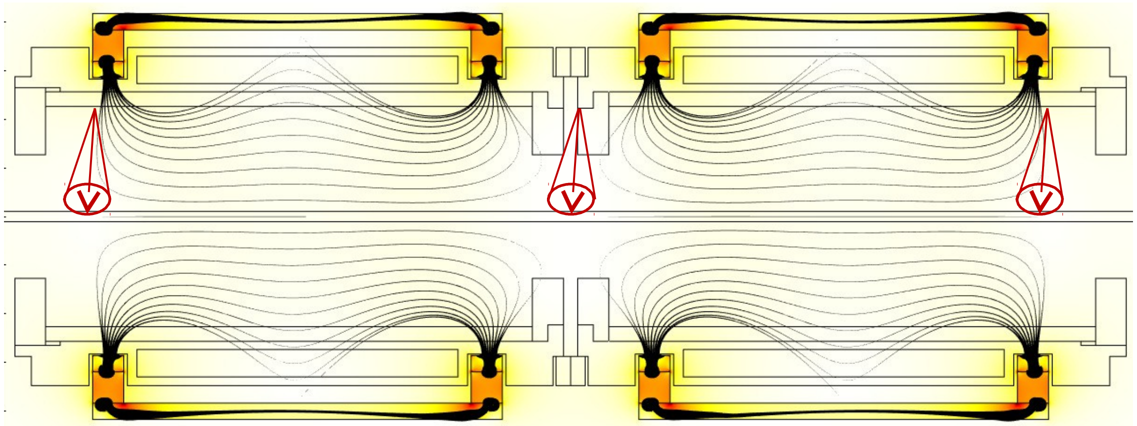


Magnetic field intensity on the target surface

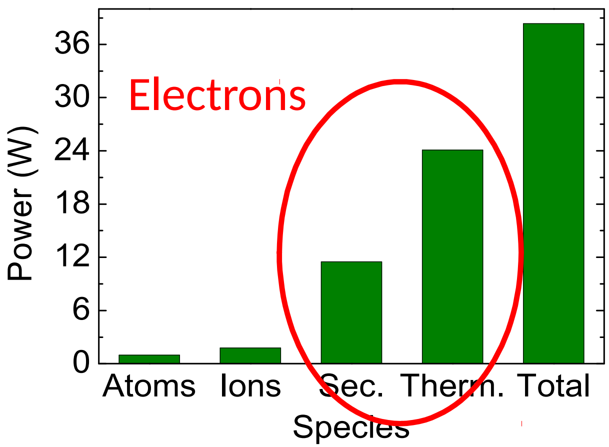


Commercial magnetron

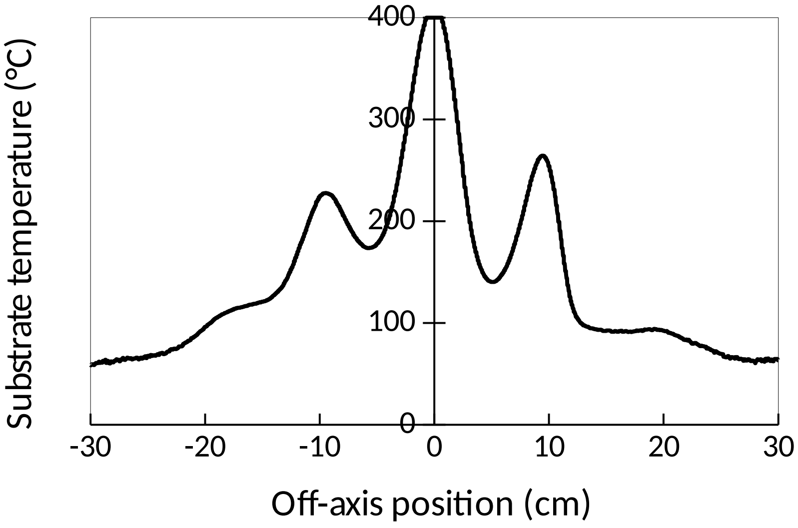
Magnetic configuration



→ Electron leakage leading to the creeping and breaking of the wire



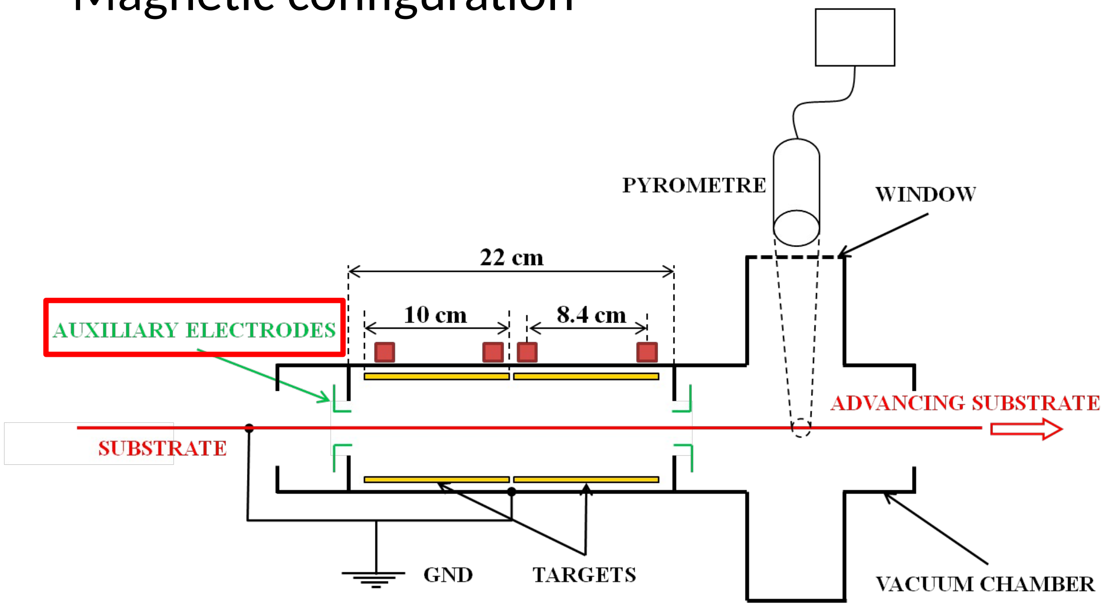
Species contributions to the heat load of the substrate



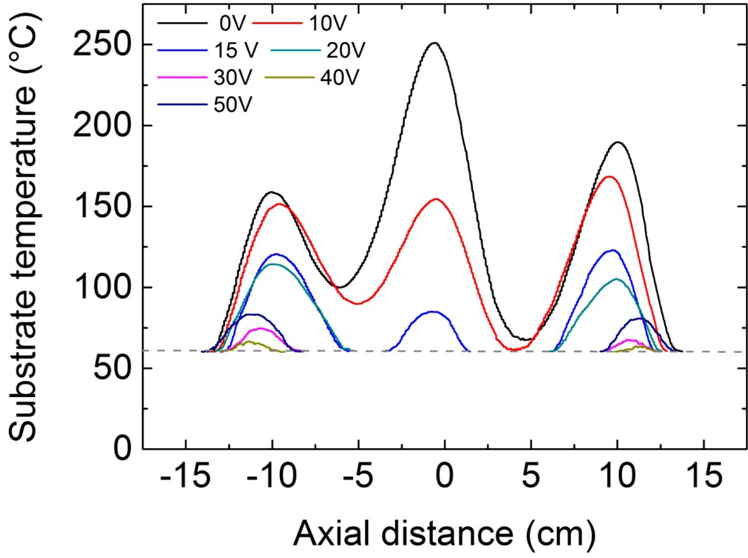
→ Substrate to repulsive potential

Commercial magnetron

Magnetic configuration



Schematic of the deposition chamber and pyrometric setup



→ Decrease of the substrate temperature

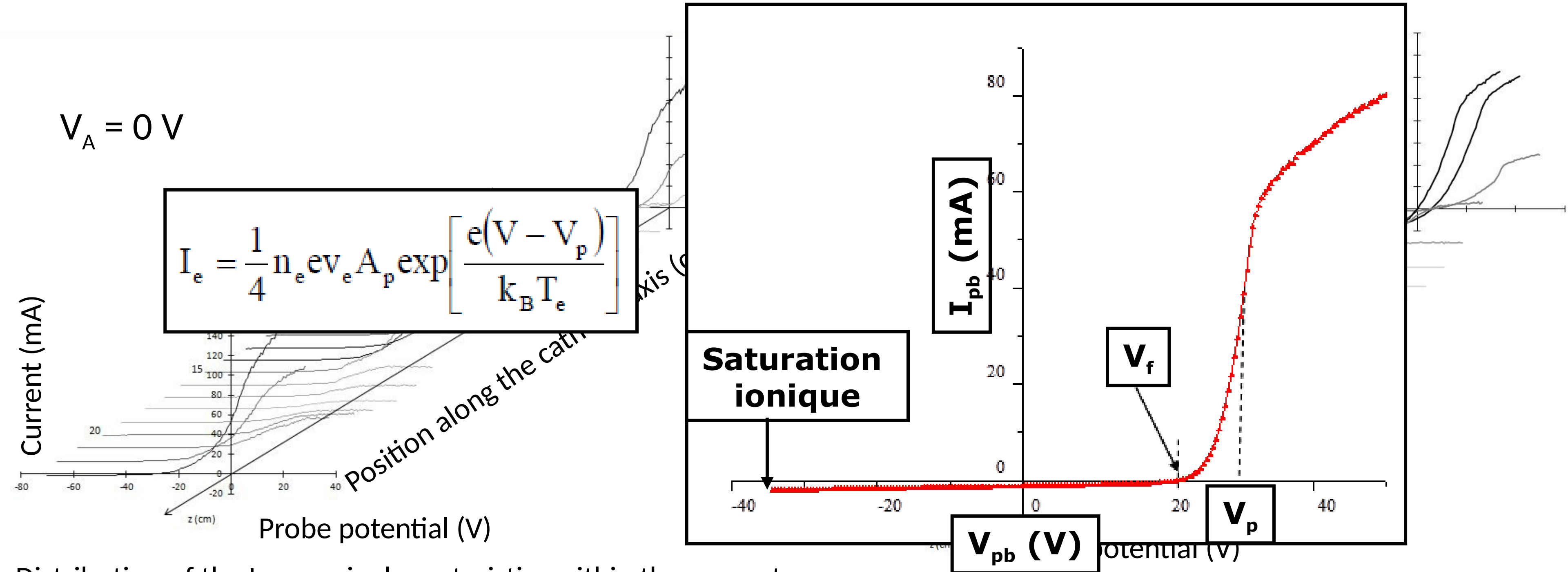
→ Modification of the spatial distribution of the plasma

Commercial magnetron

Magnetic configuration

→ Plasma characterization through Langmuir probe measurements.

$V_A = 0$ V



$$I_e = \frac{1}{4} n_e e v_e A_p \exp\left[\frac{e(V - V_p)}{k_B T_e}\right]$$

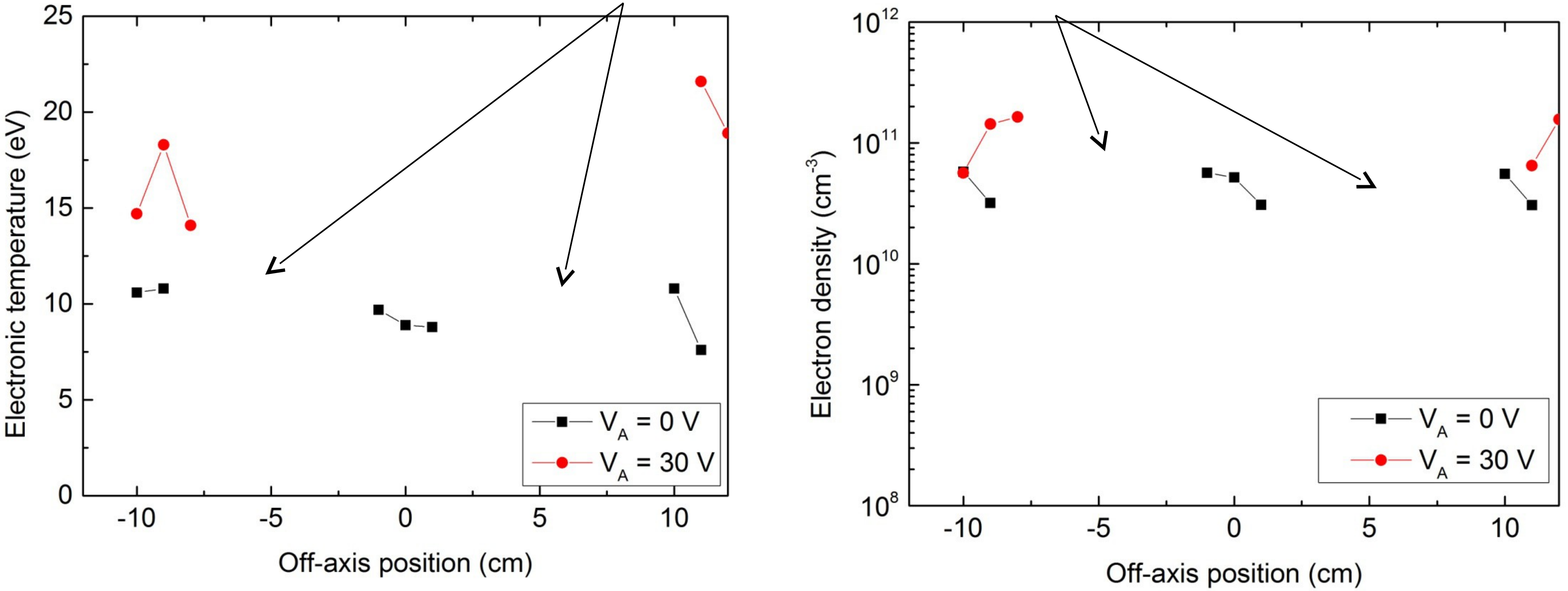
Distribution of the Langmuir characteristics within the magnetron

→ Spatial redistribution of the plasma with the variations of the anode potential

Commercial magnetron

Magnetic configuration

No data because of a lack of collected current

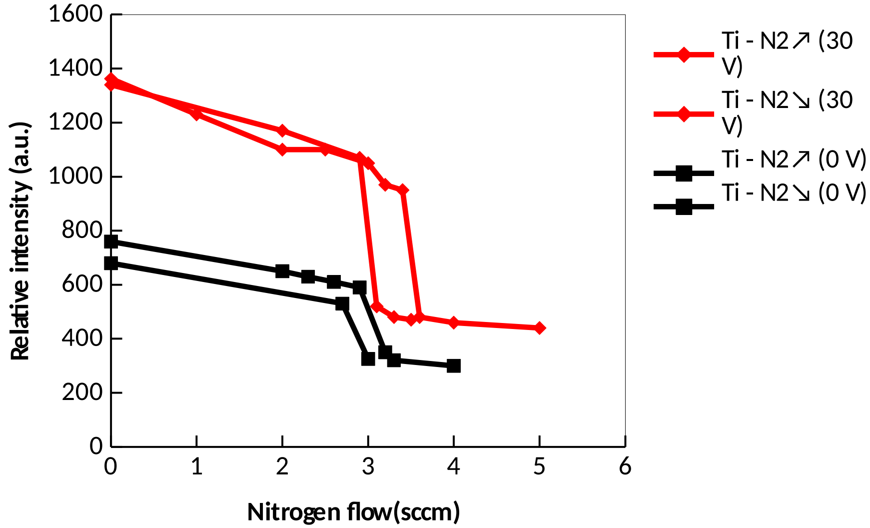


→ More energetic electrons when polarizing and electron density slightly superior

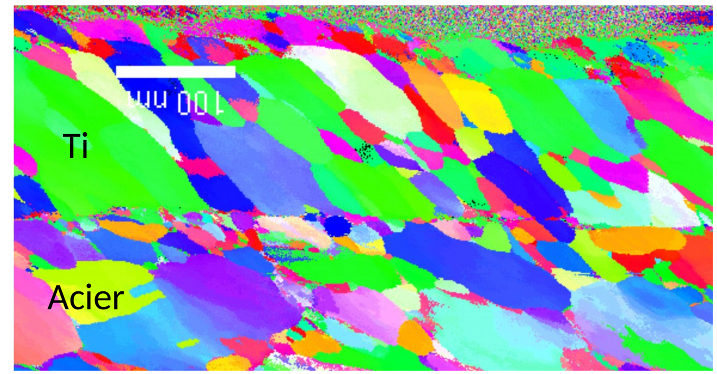
→ Better ionization but spatial redistribution of the plasma

Commercial magnetron

Influence of the anode potential



V_A = 30 V



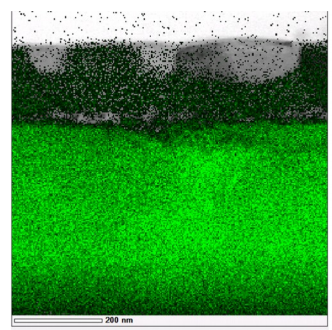
ASTAR pictures made by S. Grosso, SIMaP

→ Hysteresis shifted to higher nitrogen flows

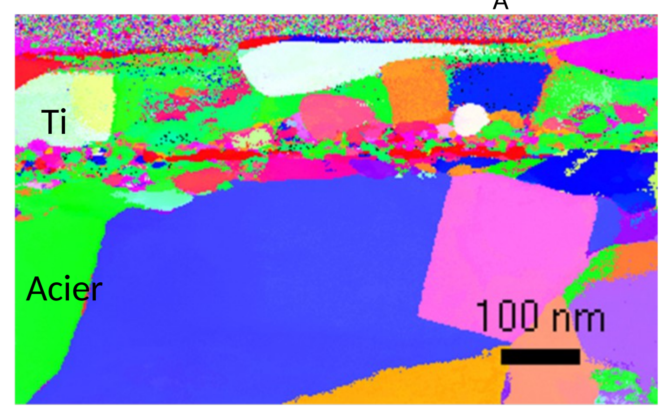
→ Grain size variation due to temperature

→ Diffusion of iron in the titanium coating

V_A = 0 V



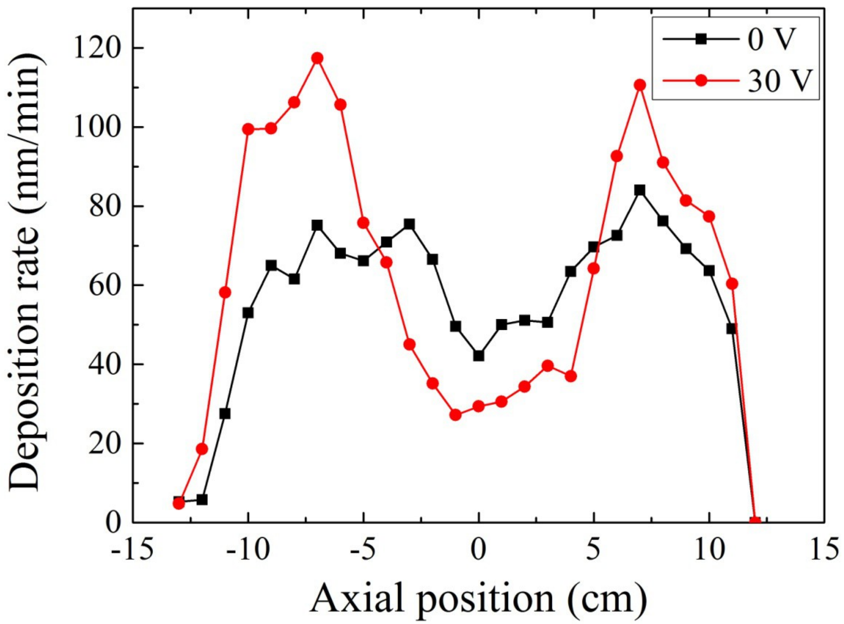
V_A = 0 V

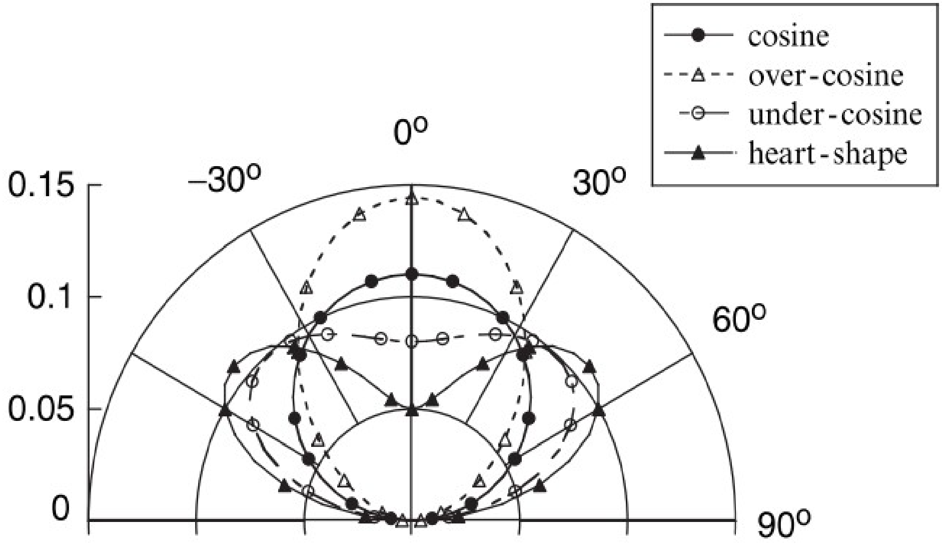


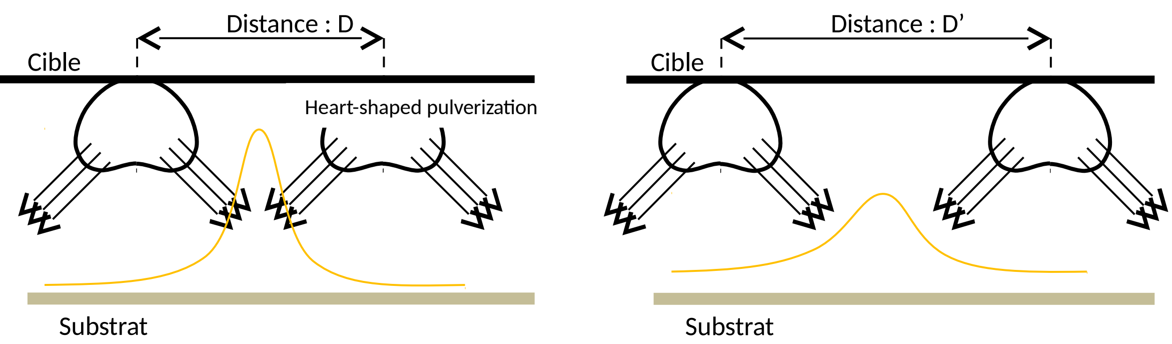
EDX characterization made by S. Grosso, SIMaP

Commercial magnetron

Influence of the anode potential





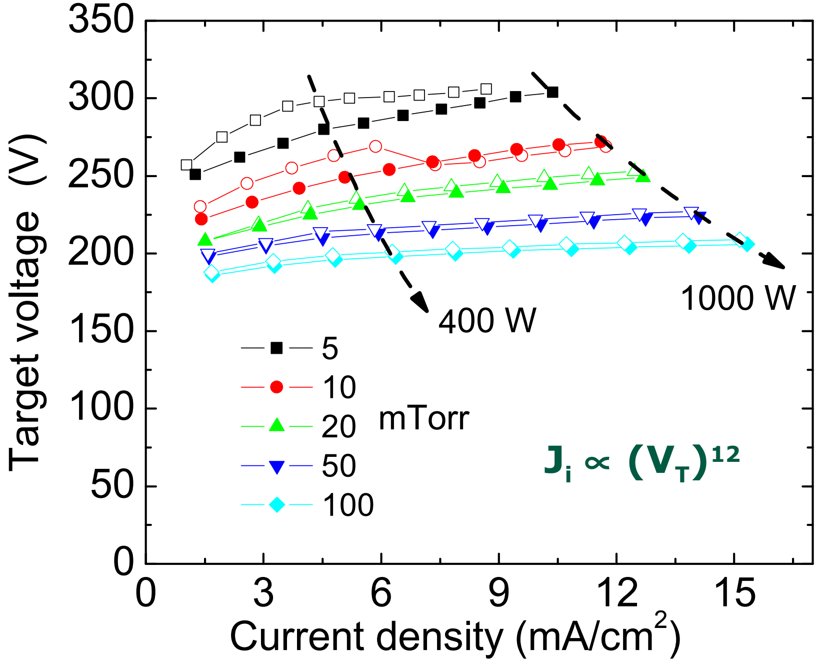


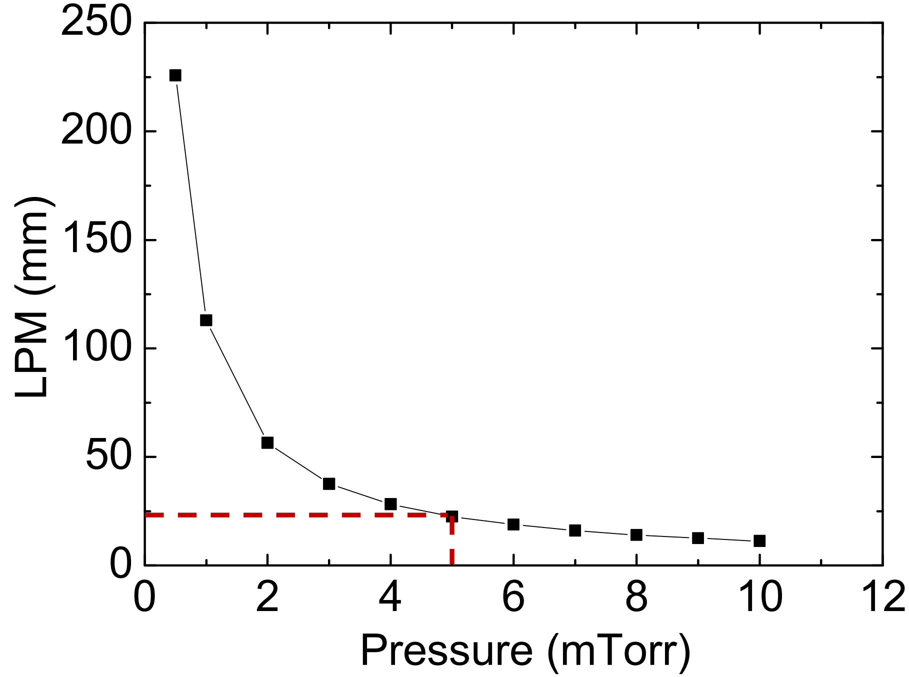
→ Deposition rate ≈ 60 nm/min

→ Deposition profile is changed due to plasma redistribution

Commercial magnetron

Operating parameters





Alexandru Todoran, « Magnetron sputtering in inverted cylindrical configuration : 3D depositoin on moving substrate », thèse 2014

→ J_T α $(V_T)^{12}$

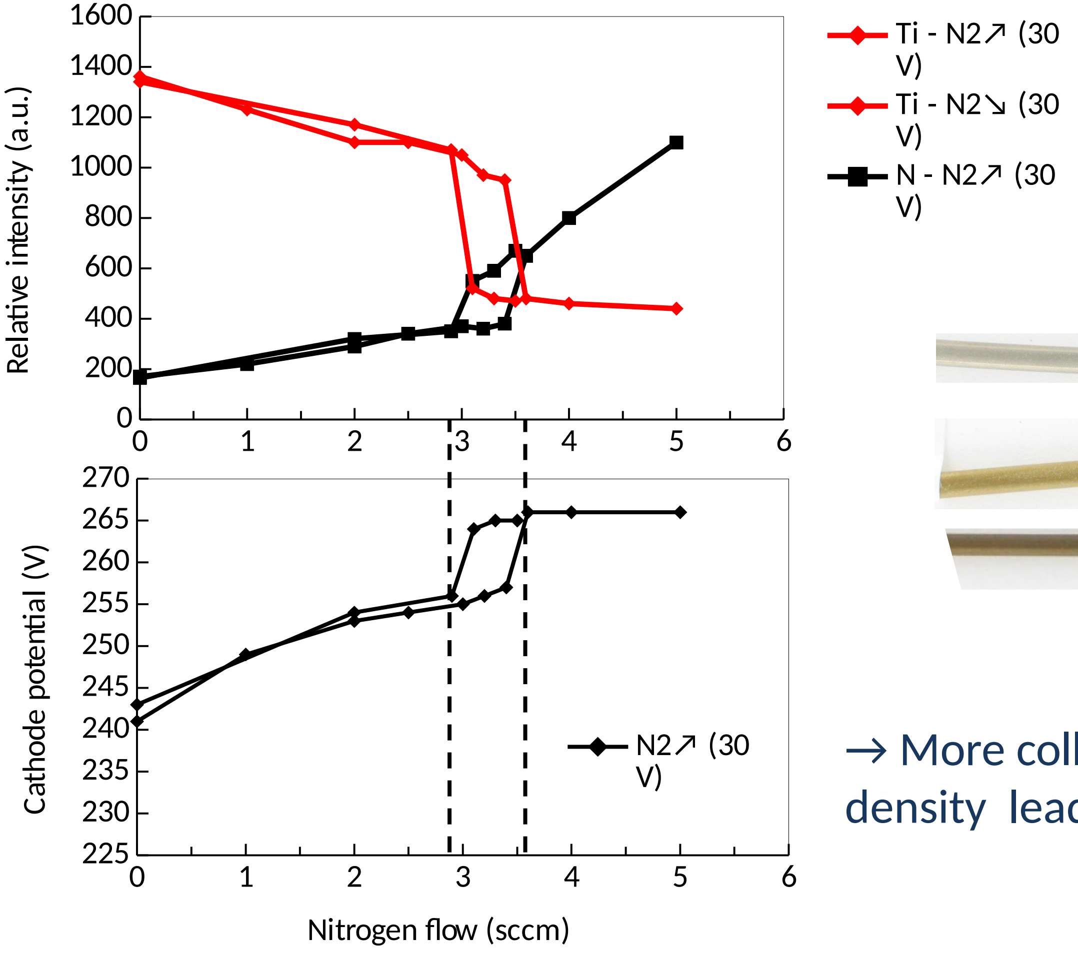
→ Minimal turn-on pressure : 5 mTorr

→ Mean free path at 5 mTorr ≈ 25 mm = Distance cathode-target

→ Maximal operating power : 1000 W (by construction)

Commercial magnetron

Operating parameters



→ 1 : under-stoechiometric
→ 2 : stoechiometric
→ 3 : over-stoechiometric

→ More collisions due to the increase of species density leading to faster thermalization

Commercial magnetron

Intermediate conclusion

→ *Necessity of homogeneous magnetic field*

→ *Control of the substrate temperature by polarizing auxiliary electrodes …*

→ *… which influences the plasma distribution within the reaction chamber*

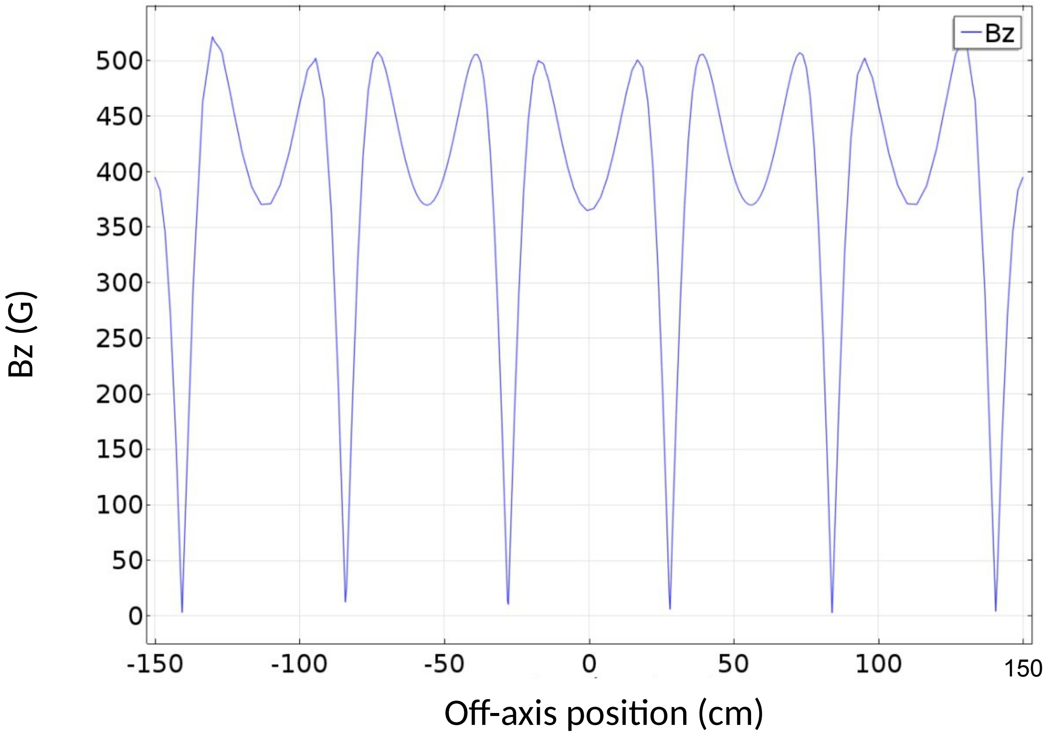
→ *Low deposition rate*

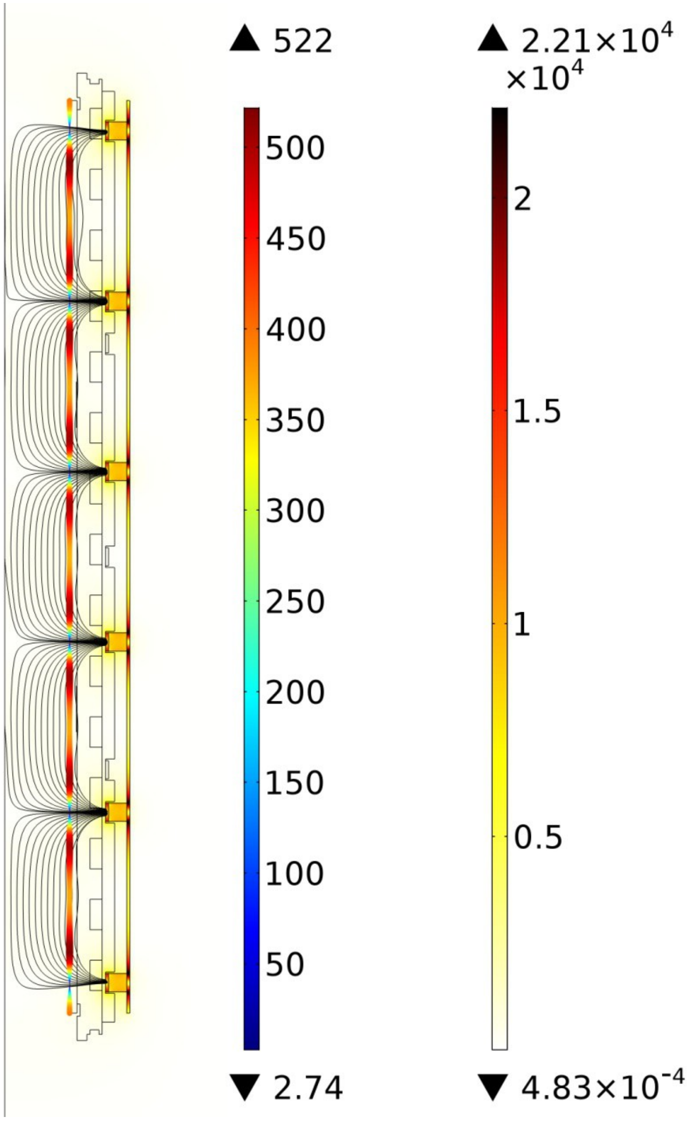
→ *Power input limited*

→ *Unstable conditions for stoechiometric coatings*

Ugitech's magnetron

Magnetic configuration



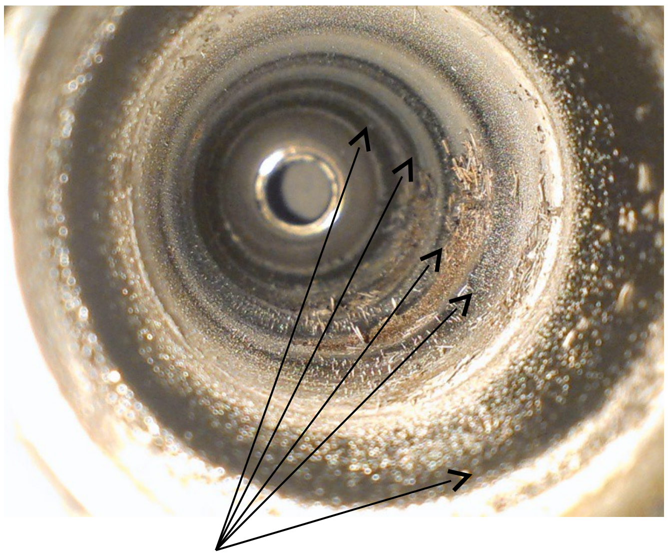


→ Non homogeneity of the magnetic field close to the target.

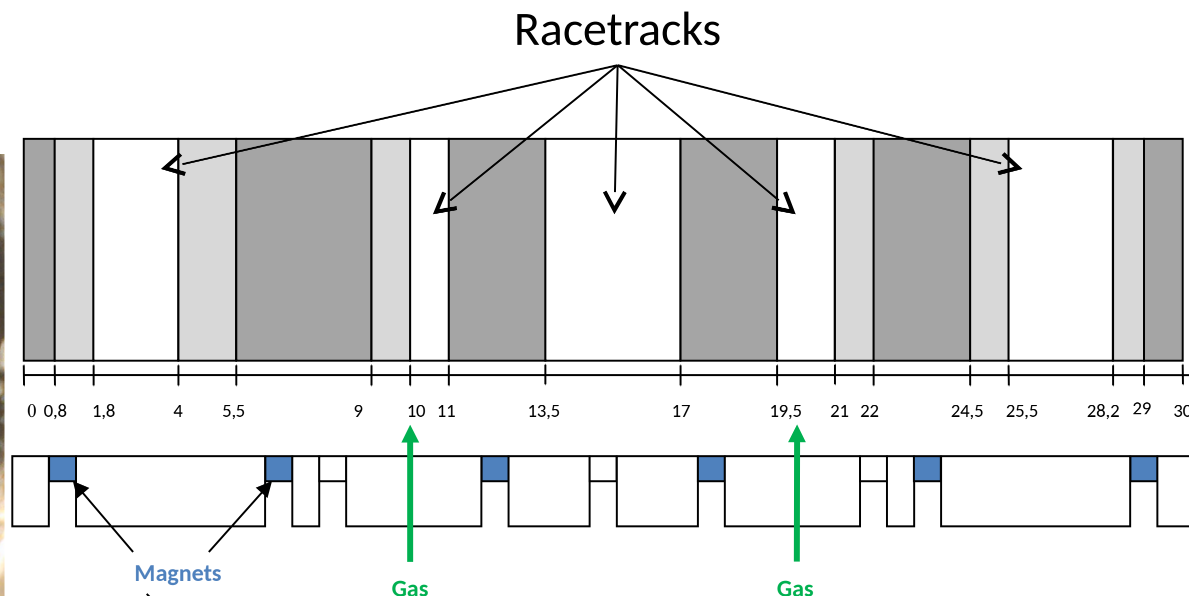
Unit : G

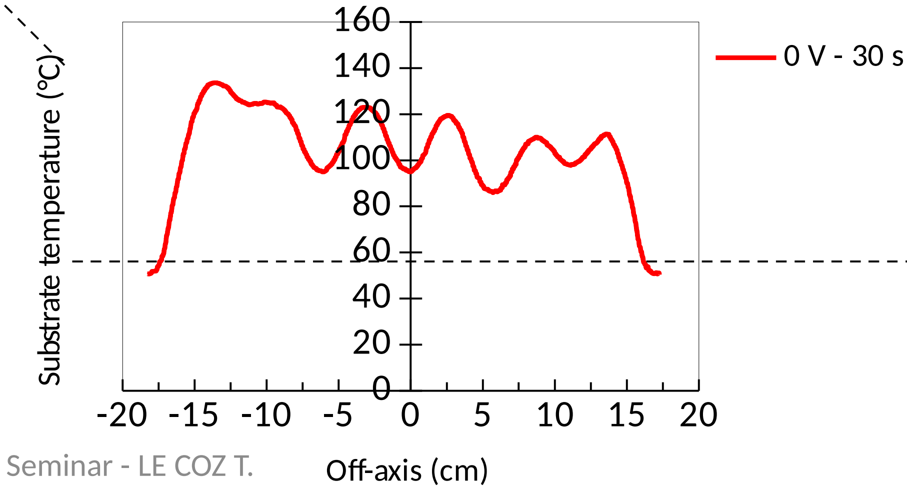
Ugitech's magnetron

Magnetic configuration



Racetracks

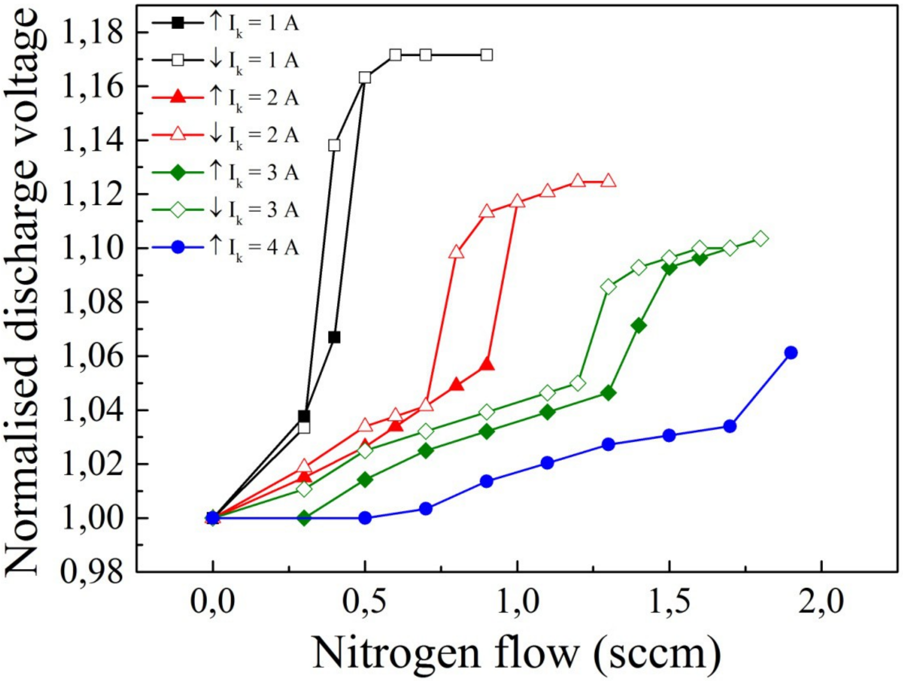




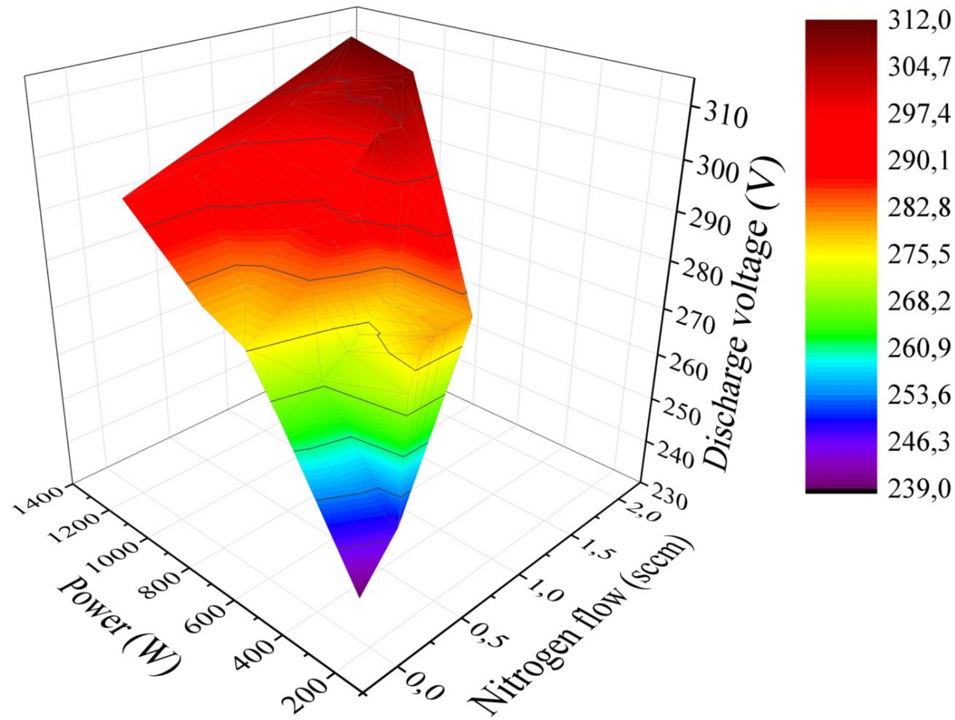
→ 5 racetracks
→ 6 electrons leakage zones
→ Target consumption: 36 %

Ugitech’s magnetron

Operating parameters



Hysteresis curves for different target currents

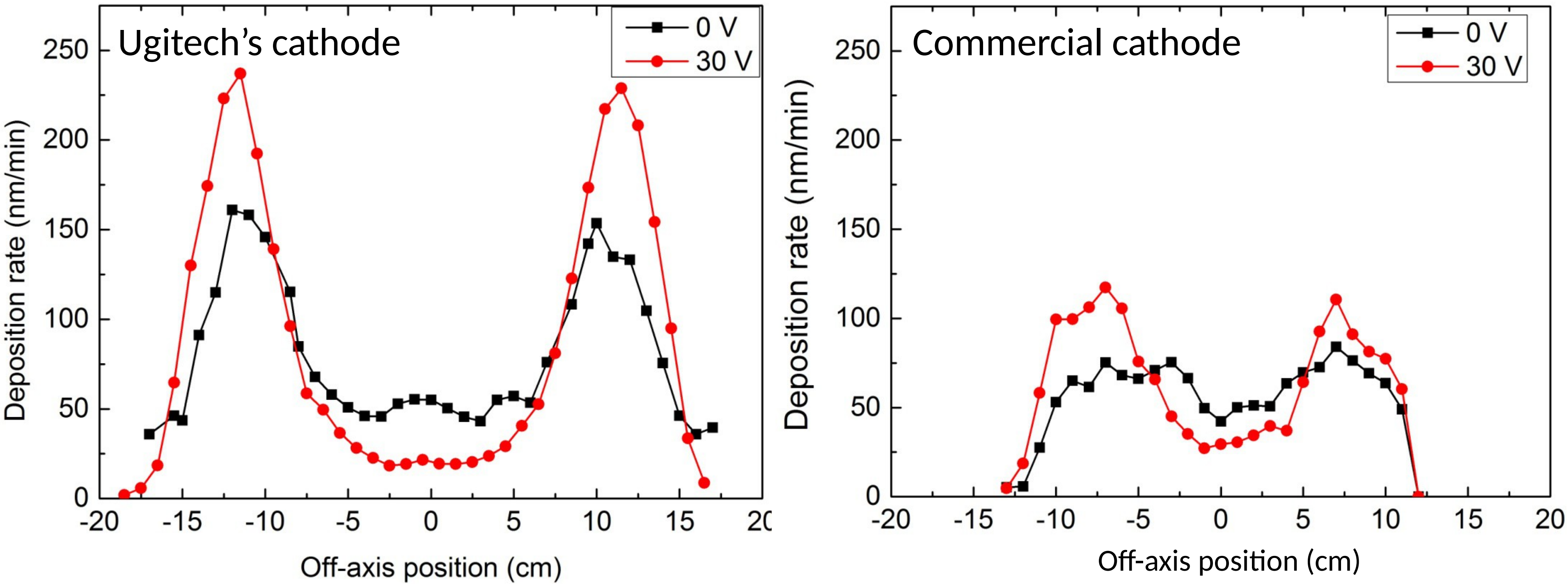


→Higher sputtering for higher current
→Increase of the ionization rate

→ Hysteresis for low nitrogen flows

Ugitech’s magnetron

Operating parameters



Deposition rate as a function of the position in the cathode

→ Mean deposition rate : 100 nm/min against 60 nm/min in commercial cathode
→ Heterogeneous thickness
→ Polarization influences spatial plasma distribution

Ugitech’s magnetron

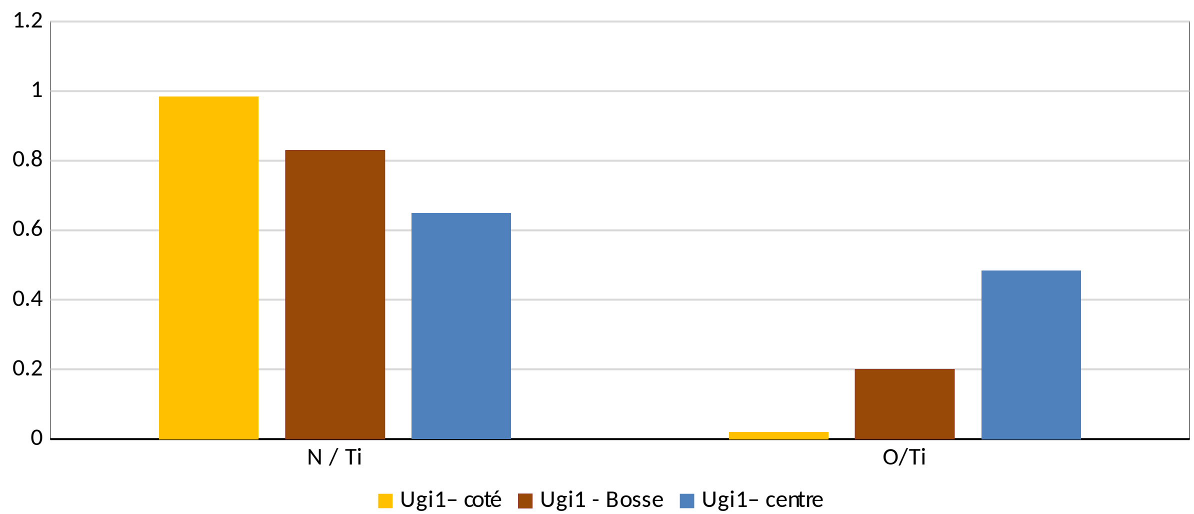
Titanium nitride coatings

Coated wire made in reactive mode under 30 V of polarization, 5 min static deposition

→ Oxygen contamination

→ Gold yellow is TiN

→ Blue is TiN_xO_y



Ratio of nitrogen and oxygen concentrations with respect to the colors on the wire

Ugitech’s magnetron

Intermediate conclusions

→ *Necessity of homogeneous magnetic field*

→ *Control of the substrate temperature by polarizing auxiliary electrodes …*

→ *… which influences the plasma distribution within the reaction chamber*

→ *Deposition rate greater than for the commercial cathode*

→ *Unknown limit of power input – grater than that for the commercial cathode*

→ *Unstable conditions for stoechiometric coatings*

→ *Oxygen contamination – Unknown origin*

Forthcoming work

→ *Sizing of a coil*

→ *Study the influence of the diameter of the targets*

→ *Modeling of the sputtering process*

→ *Modeling of the gas dynamics*

→ *Find the origin of the oxygen contamination*

→ *Ultimately design a new optimized magnetron*