

# A simple approach to transpose the deposition conditions in two different magnetron sputtering chambers: a case study of oxidation of sputter-deposited VN films

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## Introduction

- Magnetron sputtering is a vacuum coating technique characterized by high purity films, high deposition rates, outstanding uniformity on large-area substrates, and remarkably high adhesion of films. These peculiarities render this technique suitable for several applications, such as hard and wear-resistant coatings, decorative coatings, electronic thin films [1].
- Large-scale sputtering chambers are employed to make possible these industrial applications. They differ from standard laboratory sputtering reactors in numerous attributes like volume, cathode dimension, pumping speed, among others.
- This work presents a simple approach to compare the deposition parameters between two different sputtering reactors: a semi-industrial and a laboratory-scale one.
- A comparison of VN deposition conditions and afterward oxidation of the as-deposited films in a furnace under atmospheric pressure at 550 °C for up to 3.5 minutes is carried out to test the criterion.
- The oxidation of the VN films obtained in the semi-industrial machine revealed that the best thermochromic behavior occurred when the samples were completely oxidized (550 °C for 3.5 minutes) [2].
- Raman spectrometry, SEM and infrared camera analysis are employed to compare the results obtained from the oxidized films of the lab-scale machine with the ones from the semi-industrial.

## Theoretical criterion

- The criterion for comparing films obtained in different sputtering chambers is based on strong approximations. It will not be considered:
  - the difference in volume between the deposition chambers,
  - the different pumping speeds in each machine,
  - the size of the magnetron,
  - the difference in the flow of sputtered atoms to reach the substrate in each deposition chamber.
- It is assumed that the fundamental parameter that governs the growth of thin films is the energy at which the sputtered atoms arrive on the substrate. Hence, the criterion to form analogous films from both machines is that **the sputtered atoms coming from the target arrive with the same energy at the substrate.**

$$E_F = (E - k_B T_g) e^{n \ln \left( \frac{E_f}{E_i} \right)} + k_B T_g \quad \rightarrow \quad \text{energy of the atoms arriving at the film surface [3]}$$

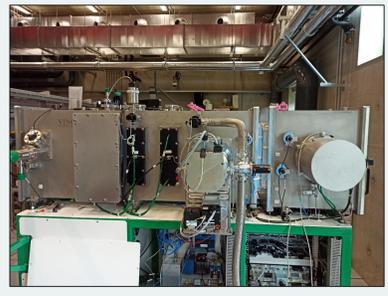
$$\left\langle \frac{E_f}{E_i} \right\rangle = 1 - 2 \frac{m_g m_s}{(m_g + m_s)^2} \quad \rightarrow \quad \text{average ratio of energy of the sputtered particle after a collision [4]}$$

$$n = \frac{d}{\lambda} \quad \text{average number of collisions before reaching the substrate} \quad \lambda = \frac{k_B T_g}{\sqrt{2} \pi d_s^2 P} \quad \text{mean free path of the sputtered particle}$$

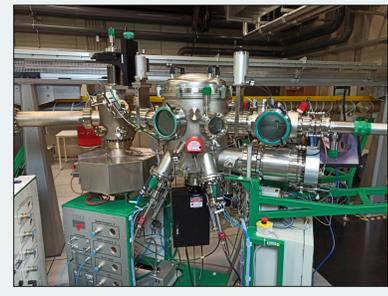
Parameters controlled during deposition: pressure (P) and target-substrate distance (d)

## Experimental conditions

### Semi-industrial machine



### Lab-scale machine



### Main differences between the two machines

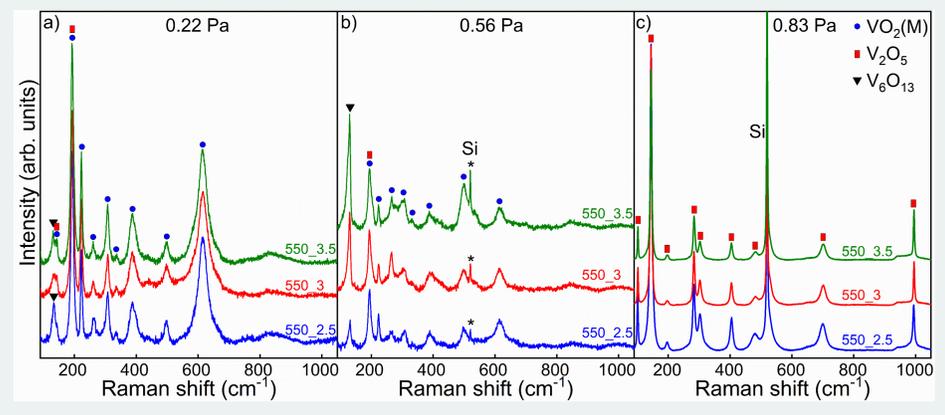
	Semi-industrial	Lab-scale
<b>Cathode size</b>	330 x 50 x 6 mm <sup>3</sup>	2" diameter
<b>Deposition chamber volume</b>	≈ 350 L	≈ 70 L
<b>Target-substrate distance (d)</b>	fixed to 5 cm	9 – 19 cm

### VN deposition conditions

	Semi-industrial	Lab-scale
<b>Substrate</b>	Si	Si
<b>Working pressure (Pa)</b>	1	0.22, 0.56, 0.83
<b>Target-substrate distance (cm)</b>	5	9
<b>Deposition temperature (°C)</b>	No intentional heating	
<b>V target current (A)</b>	1.5	0.5
<b>Ar flow rate (sccm)</b>	30	30
<b>N<sub>2</sub> flow rate (sccm)</b>	5	5
<b>P × d (Pa cm)</b>	5	1.98, 5.04, 7.47

## Structural characterization

### VN oxidized films from the lab-scale machine



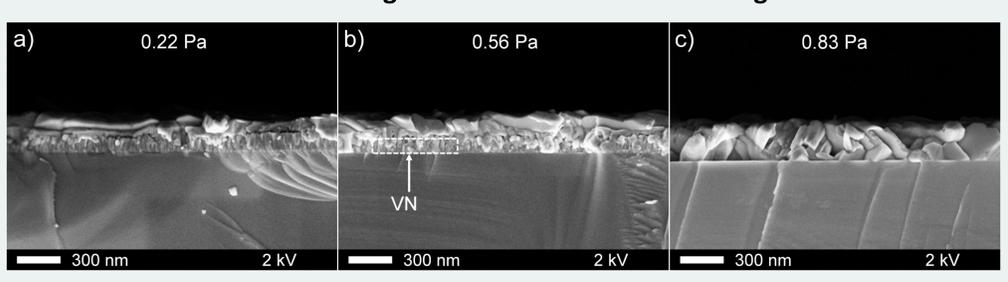
Mixture of phases, VN and VO<sub>2</sub> are the predominant phases. None of the oxidized films present the Si vibration band (VN residual layer). VO<sub>2</sub> is the predominant oxide phase.

Mixture of phases, VN and VO<sub>2</sub> are the predominant phases. The intensity of the Si band (520.7cm<sup>-1</sup>) increases with oxidation time. VO<sub>2</sub> is the predominant oxide phase.

The Si band is the more intense band in all the films (no VN residual layer). V<sub>2</sub>O<sub>5</sub> is the only oxide phase detected.

## Morphology and optical properties of the oxidized VN films (lab scale machine)

### Cross-section images for the oxidized films during 3.5 min



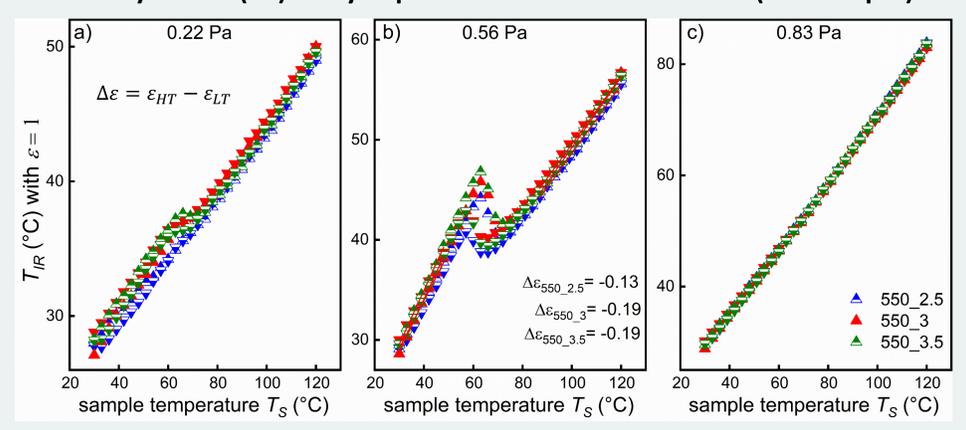
**a) 0.22 Pa:** Continuous VN layer on top of the Si substrate. VO<sub>2</sub> layer on top of the remaining VN layer.

**b) 0.56 Pa:** Discontinuous VN layer on top of the Si substrate.

**c) 0.83 Pa:** Film completely oxidized, no sign of VN. Large grains of V<sub>2</sub>O<sub>5</sub>.

The cross-section images validate the results of Raman spectrometry

### Emissivity switch (Δε) analysis performed with an IR camera (7.5 – 13 μm)



**a) 0.22 Pa:** No thermochromic behavior, important VN residual layer.

**b) 0.56 Pa:** Thermochromic behavior (characteristic hysteresis loops).

**c) 0.83 Pa:** No thermochromic behavior, V<sub>2</sub>O<sub>5</sub> is the only phase detected.

## Conclusions

- The results evidence the influence of the VN deposition parameters on its oxidation behavior to form a thermochromic VO<sub>2</sub>(M) phase.
- As predicted, the only samples that display thermochromic behavior are the ones deposited with equivalent conditions of the semi-industrial chamber (0.56 Pa), even with the presence of a residual VN layer.
- Samples deposited with lower pressure (0.22 Pa) are denser and more difficult to oxidize. On the contrary, films deposited with higher pressure (0.83 Pa) are less dense and easy to oxidize.
- These findings reveal the relevance of our criterion to produce analogous films through different sputtering machines. The conditions obtained could be employed as initialization parameters to transpose deposition from one sputtering chamber to another (even to industrial scale).

## References

[1] Kelly, P. J. *et al.*, *Vacuum*, 56, (2000), 159–172.  
 [2] García-Wong, A.C. *et al.*, *Sol. Energy Mater. Sol. Cells*, 210, (2020), 110474.  
 [3] Meyer, K. *et al.*, *J. Appl. Phys.*, 52, (1981), 5803-5805.  
 [4] Gras-Marti, A. *et al.*, *J. Appl. Phys.*, 54, (1983), 1071-1075.

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