

Influence of the inclined columnar microstructure of ZrO₂ thin films on the surface reactivity

Aurélien BESNARD^{(a)*}, Asma GZAIEL^(b,c,d), Khalil AOUADI^(d,e), Faker BOUCHOUCHA^(d), Corinne NOUVEAU^(b), Yoann PINOT^(b), Amina GUESSABI^(f), Boudjemaa BOUAOUINA^(g)

(a) *Université Marie et Louis Pasteur, SUPMICROTECH, CNRS, Institut FEMTO-ST, Besançon, France.*

(b) *Arts et Métiers Institute of Technology, LaBoMaP, UMLP University, Cluny, France*

(c) *University of Carthage, Tunisia Polytechnic School, La Marsa, Tunisia*

(d) *Research Laboratory Physics, Mathematics, Quantum Modeling and Mechanical Design, University of Carthage, IPEIN, Nabeul, Tunisia*

(e) *Engineering and durability of materials center, Moroccan Foundation for Advanced Science, Innovation and Research, University Mohammed VI Polytechnic, Ben Guerir, Morocco*

(f) *Plant and Microbial Biotechnology, Moroccan Foundation for Advanced Science, Innovation and Research, Mohammed VI Polytechnic University (UM6P), Ben Guerir, Morocco*

(g) *Département de Physique, Unité de Recherche Matériaux, Procédés et Environnement, Université de Boumerdès, Boumerdès, Algeria*

* Corresponding Author: aurelien.besnard@femto-st.fr

Keywords : ZrO₂, Oblique angle deposition (OAD), Microstructure, Antibacterial activity

Abstract : Zirconia has attracted attention for its potential antibacterial activity and good mechanical performance. It is widely used as a bulk material to produce femoral heads of hip prosthesis. The reactivity of the devices is directly linked to the properties of the surface in contact with the biological environment. The use of thin films can enhance these performances, thanks to the fine control of the roughness and porosity at a very local range [1]. Among all PVD deposition techniques, magnetron sputtering using oblique angle deposition (OAD), allows the growth of films dominated by the “shadowing effect”. This

specific configuration induces an inclined columnar microstructure and a higher porosity and roughness.

To investigate various incidence configurations, a custom-designed substrate holder was employed [2]. It consists of a base plate onto which individual holders are mounted, each with a specific out-of-plane inclination (substrate tilt angle θ) and two possible in-plane orientations (substrate orientation angle Φ). The target-to-substrate distance was fixed at 85 mm, and the center of each substrate was positioned 30 mm from the system's central axis. A schematic representation of the system geometry is shown in Figure 1. Seven substrate tilt angles θ were available on the substrate-holder: 15, 30, 45, 60, 75, and 85°, each with two possible in-plane orientation angles Φ : 0° (facing the target) and 180° (facing the chamber wall). Using SIMTRA simulations, six $[\theta; \Phi]$ configurations were selected to cover a wide range of incidence angles α , from 7° to 74° [3]. Structural analyses were conducted via X-ray diffraction, while microstructure and surface morphology were examined using scanning electron microscopy and atomic force microscopy. Nanoindentation tests were performed to assess hardness and elastic modulus. Wettability was assessed on a contact angle meter system using two liquids (physiological water and ethylene glycol). The antibacterial activity of ZrO₂ films was examined according to the standard plate count method against Gram-positive pathogens (*Staphylococcus aureus* ATCC 29213).

Results revealed that increasing the incidence angle α from 7 to 74° significantly affected surface morphology, microstructure, film thickness, and columnar tilt. The hardness and Young's modulus of the films remained constant for incidence angle α between 7 and 50°, but decreased with the increasing incidence angle α . The films exhibit neither distinctly hydrophobic nor hydrophilic behavior at incidence angles α below 45° while at incidence angles α above 45°, corresponding to more inclined columns and increased surface roughness, the films become hydrophobic for both liquids. Furthermore, the films exhibited strong antibacterial activity against Gram-positive pathogens (*Staphylococcus aureus*), particularly at the highest incidence angle α , with inhibition rates exceeding 90%.

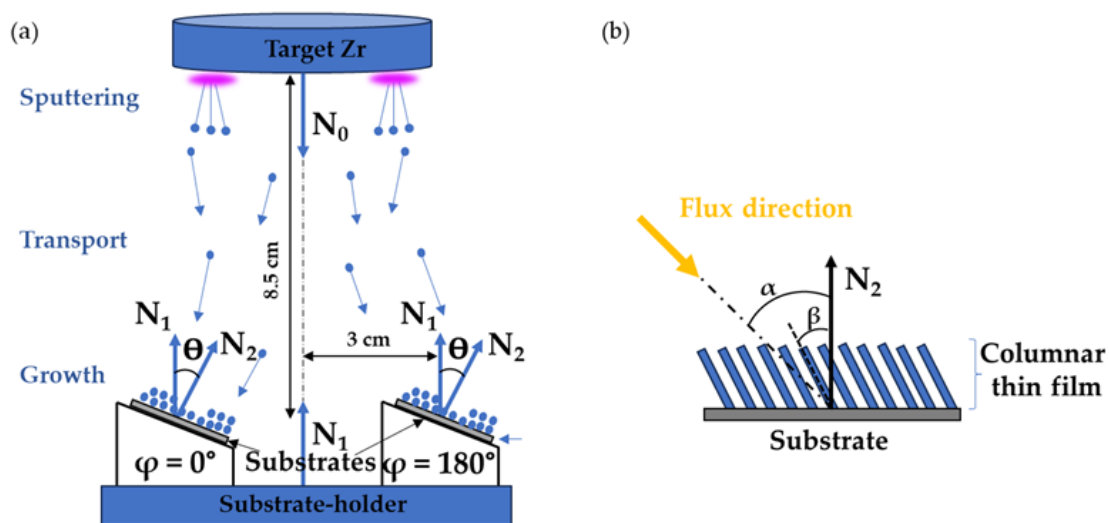


Figure 1: Principle of the OAD technique: (a) Schematic representation of the system geometry showing the out-of-plane substrate tilt angle θ and the in-plane substrate orientation

Φ ; (b) Illustration of the inclined columnar microstructure (column tilt angle β) formed within the deposited film due to the incidence of the flux (angle α).

REFERENCES :

- [1] A. GZAIEL, K. AOUADI, A. BESNARD, Y. PINOT, C. NOUVEAU, F. BOUCHOUCHA, Y. AGZENAI BEN SALEM, A. GUESSABI, B. BOUAOUINA, *Micro*, **6**, 4, (2026). <https://doi.org/10.3390/micro6010004>
- [2] K. RAHMOUNI, A. BESNARD, K. OULMI, C. NOUVEAU, A. HIDOUSSE, L. AISSANI, M. ZAABAT, *Surface and Coatings Technology*, **436**, 128310, (2022). <https://doi.org/10.1016/j.surfcoat.2022.128310>
- [3] A. BESNARD, H. GERAMI, M. RASCHETTI, N. MARTIN, *Nanomaterials*, **15**, 620, (2025). <https://doi.org/10.3390/nano15080620>