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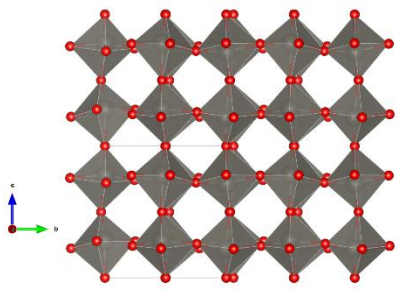
Annealing Temperature and Pressure Effects on the Structural and Photoelectrochemical Properties of Sputtered WO_3 Thin Films

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Introduction

Tungsten Trioxide (WO_3)



Type: n-type semiconductor

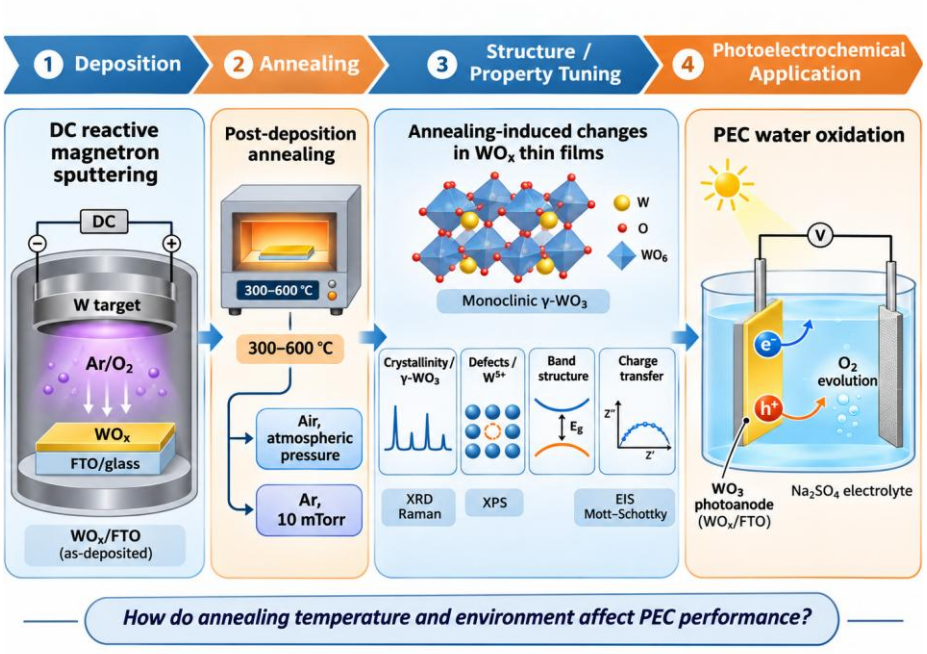
Bandgap: 2.6–3.2 eV

Applications: Photocatalysis, gas sensing, smart windows.

- Tungsten oxide (WO_3) is a promising n-type semiconductor for photoelectrochemical (PEC) water oxidation [1].
- However, its performance is strongly influenced by crystallinity, defect chemistry, and charge-transfer processes.
- Post-deposition annealing can modify these properties, but the combined effects of annealing temperature and environment on PEC performance remain insufficiently understood [2].

In this work, we investigate how **annealing at different temperatures** (300-600 °C), under **atmospheric pressure (air)** and **reduced pressure (10 mTorr, Ar)**, affects the structural, optical, electronic, and PEC properties of WO_x thin films deposited by DC reactive magnetron sputtering.

Methodology

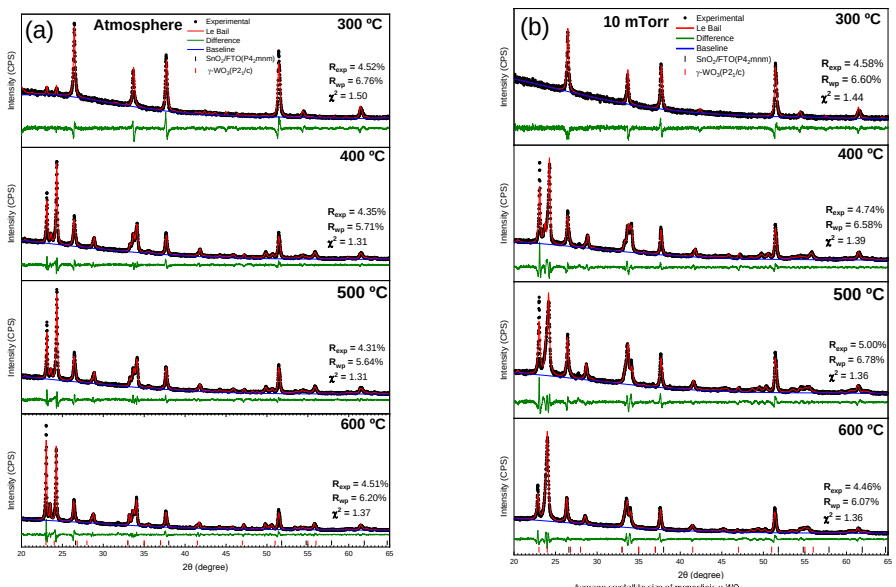


Results

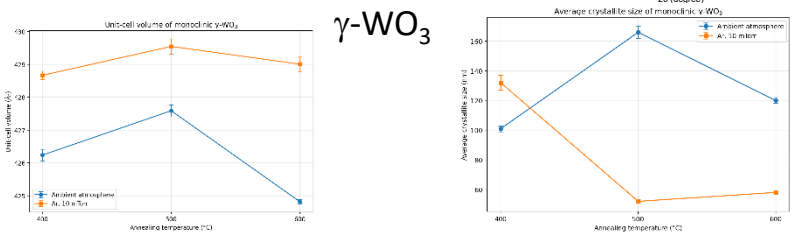
XRD

Ambient

10 mtorr



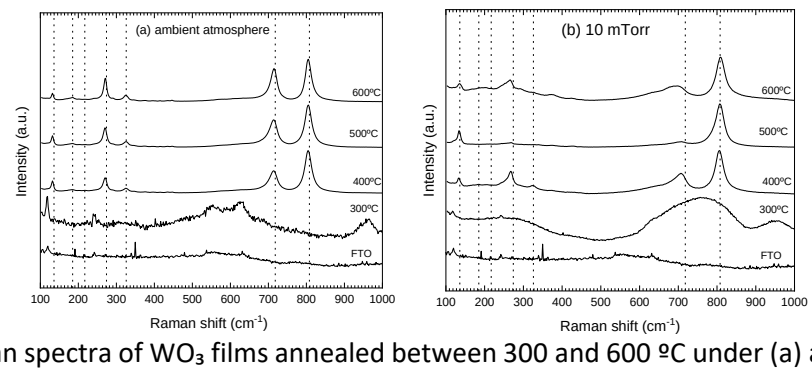
γ-WO_3



Unit Cell Volume

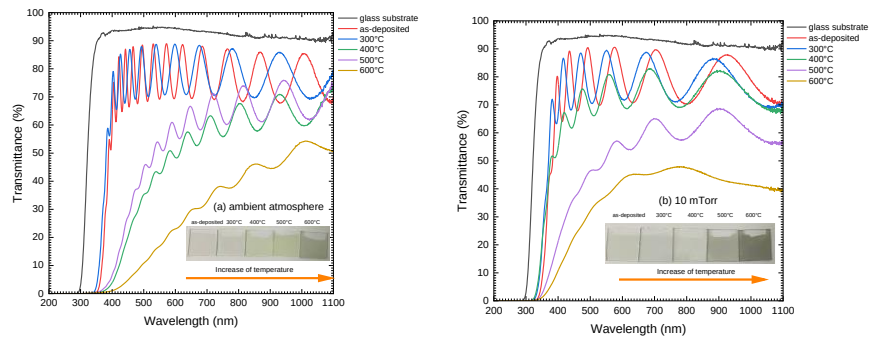
Mean Crystallite Size

Raman Scattering

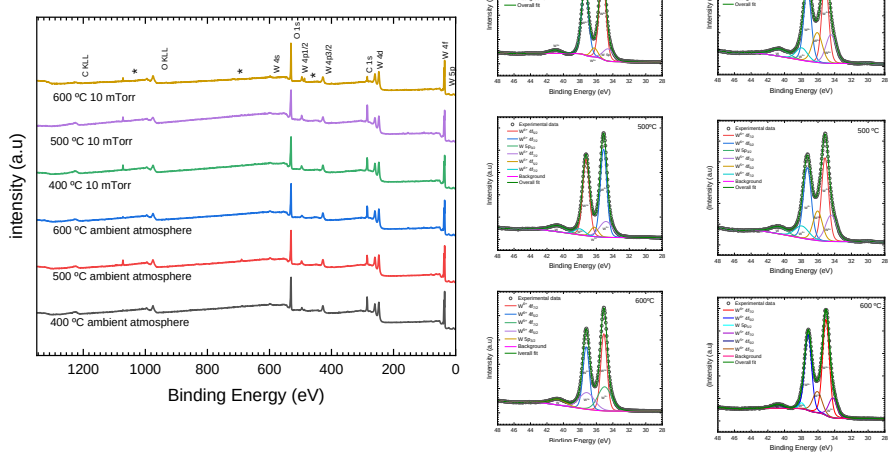


Raman spectra of WO_3 films annealed between 300 and 600 ºC under (a) ambient atmosphere and (b) 10 mTorr.

Transmittance

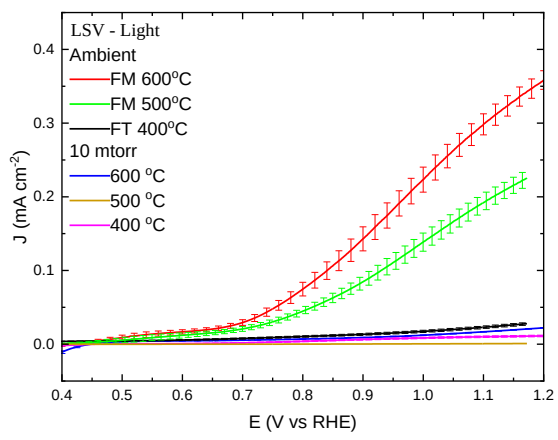


XPS

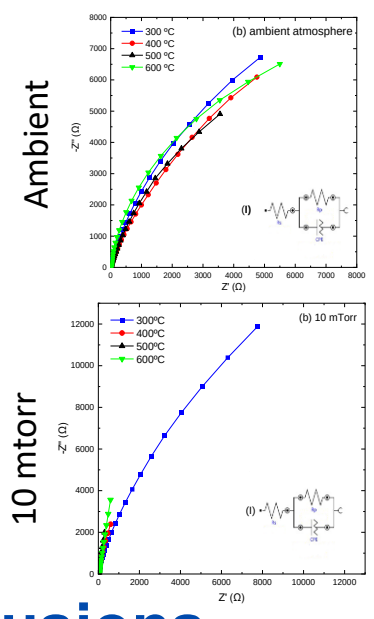


PEC

Ciclic Voltammetry



EIS



Conclusions

- Post-deposition annealing at **400 ºC and above** promoted the crystallization of monoclinic γ-WO_3, as confirmed by XRD and Raman spectroscopy.
- Annealing under **Ar at 10 mTorr** resulted in larger unit-cell volumes and smaller crystallite size at 500-600 °C compared with atmospheric annealing.
- XPS revealed predominantly W^{6+}, with W^{5+} contributions and changes in surface oxygen environments depending on annealing conditions.
- The best PEC performance was achieved at **600 °C under atmospheric pressure** (~0.35 mA cm^{-2}), while **400 °C was optimal at 10 mTorr** (~0.20 mA cm^{-2}), both at 1.2 V vs. RHE.
- PEC performance depends on the interplay between crystallinity, defect-related properties, and interfacial charge-transfer processes.

Acknowledgments

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Eneva H_2 Turquoise Project.

References

[1] O. Samuel, et al. Ceram. Int. 48 (2022) 5845–5875.

[2] S. Farid, et al. Nanotechnology 31 (2020).