MICROESTRUCTURAL CHARACTERIZATION OF WO_3 THIN FILMS

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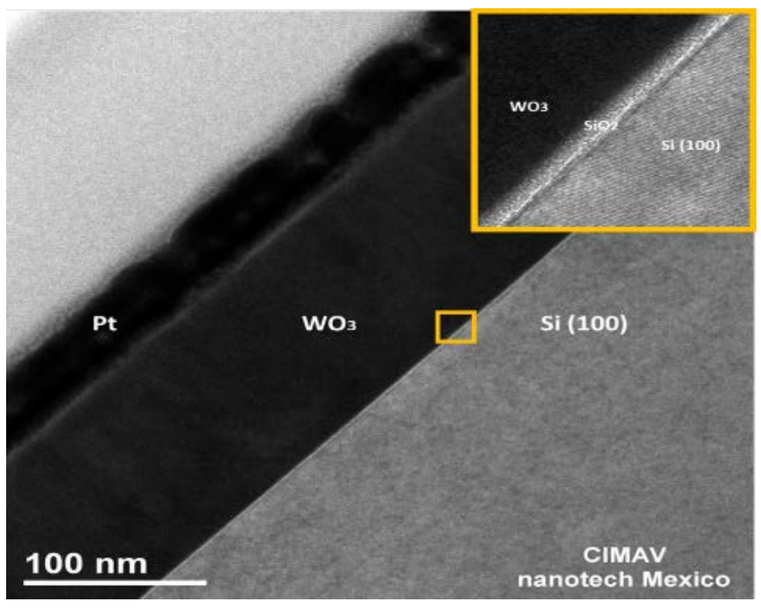
Abstract

The thin films of WO_3 were grown by reactive magnetron sputter deposition and two-step annealing, here are few studies on the microstructure and crystallography of WO_3 thin films. The study of the structure and crystalline phases of WO_3, Determine the direction of growth of the thin films, the thickness formed on the deposition and the tilt of octahedrons of WO_6 .

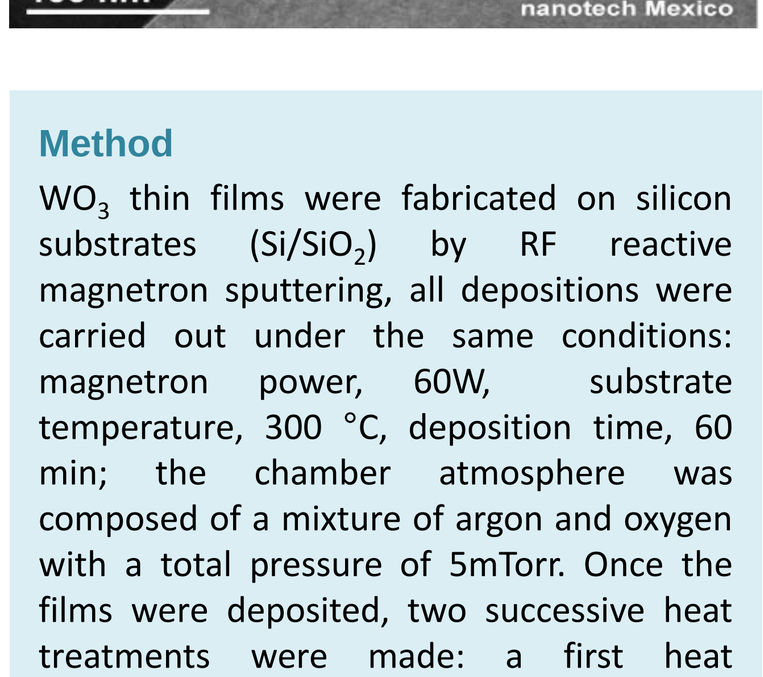
Introduction

There are few studies on the microstructure and crystallography of WO_3 thin films. The study of the structure and crystalline phases of WO_3 is really important, due to the fact that electrochromic , catalytic and gas sensing properties depends on structure and crystalline phase present.

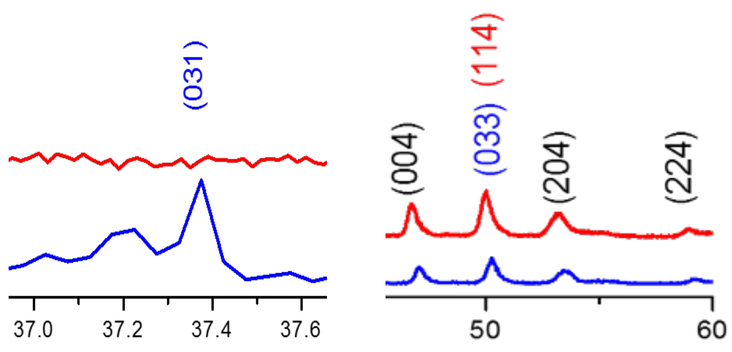


Method

WO_3 thin films were fabricated on silicon substrates (Si/SiO_2) by RF reactive magnetron sputtering, all depositions were carried out under the same conditions: magnetron power, 60W, substrate temperature, 300 °C, deposition time, 60 min; the chamber atmosphere was composed of a mixture of argon and oxygen with a total pressure of 5mTorr. Once the films were deposited, two successive heat treatments were made: a first heat treatment at 400 °C for six hours followed by a second heat treatment at 350 °C for one hour.



Grazing Incidence X-Ray Diffraction (GIXRD): The grazing angle was 0.2 with a step size of 0.02° and a integration time of 10 s per step.



Reflection conditions

$0kl$: $k+l = 2n$
hkl : $h+l$, $k = 2n$

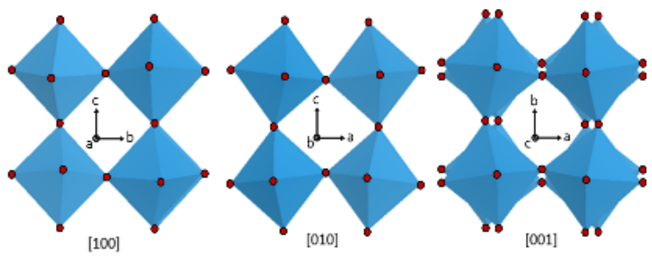
$0kl$: $k = 2n$
hkl : $h+k$, $l = 2n$

Interplanar distance d_{hkl}

$$\frac{1}{d_{hkl}^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}$$

d_{hkl}=1.82 Å

Grazing Incidence X-Ray Diffraction (GIXRD): Reflection conditions on the plane (031) and Interplanar distance on planes (033)/(114).

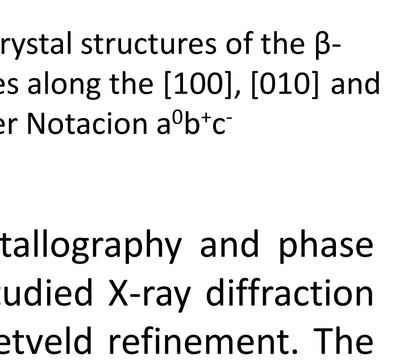


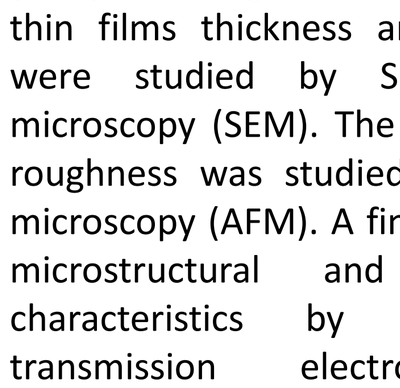
Projections of the crystal structures of the β-WO_3 viewing tilt angles along the [100], [010] and [010], Glazer Notacion $a^0b^+c^-$

Results

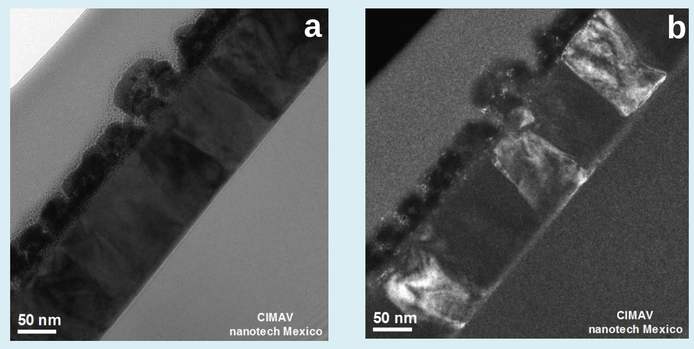
The thin films crystallography and phase composition was studied X-ray diffraction (XRD) including Rietveld refinement. The thin films thickness and microstructure were studied by Scanning electron microscopy (SEM). The thin films surface roughness was studied by atomic force microscopy (AFM). A fine study about the microstructural and crystallographic characteristics by high resolution transmission electron microscopy (HRTEM) allowed to determine the growth directions and planes.

High-resolution SEM: Surface morphologies of WO_3 films after two-step annealing

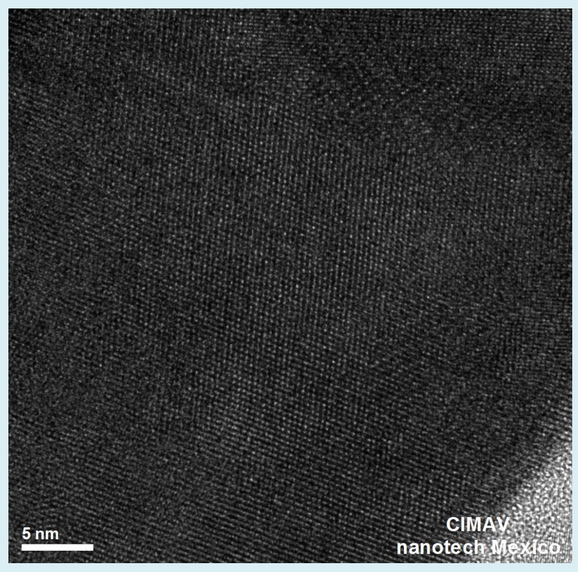




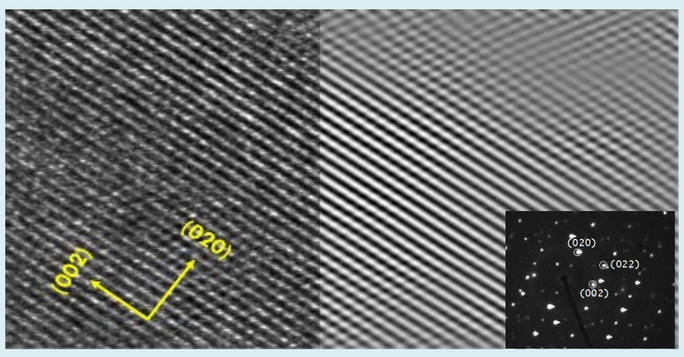
AFM image (1μm x 1 μm) : Showing the surface morphology of WO_3 after two anealinngs



TEM images of WO_3 thin film whit two-step annealing at 250 °C for 5 h: (a) bright field TEM image; (b) dark field TEM image obtained on the same area.



HR-TEM images of WO_3



HR-TEM images of WO_3: image processing using a mask with the Digital Micrograph software, in order to highlight the planes (002) and (020)

Conclusion

WO3 thin film after heat treatment present two orthorhombic phases, krasnagorite and β-WO3, where this last phase, presents inclinations in their octahedral with glazer´s notation $a^0b^+c^-$, the preferential growth direction is (002) and grows in a columnar form.

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