



An Attempt to Study MoO₃-Like TCO Nanolayered Compound in Terms of structural and Ethanol Sensitivity Application



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Abstract

MoO₃ thin films were successfully synthesized by a simple, inexpensive, and reproducible spray technique for gas sensing applications by using Ammonium molybdate tetrahydrate [(NH₄)₆Mo₇O₂₄·4H₂O]10⁻²M aqueous solution. XRD shows that MoO₃ prepared thin films crystallizes in an orthorhombic structure with space group Pnma (62) related to α-MoO₃ phase. On the other hand, AFM observations show some fibers-shape parallel to the substrate. Finally, ethanol detection test shows good sensitivity with reproducible behavior.

Keywords: Molybdenum oxide; Spray; Gas sensors

Introduction

During the last years, the semiconductors based on metal oxides have known many interests in the research as well as in the field of the application. Indeed, these oxides have been applied in several areas such as, optical windows [1], surface acoustic devices [2] and solar cells [3]. In the field of the environment, oxides have played an important role, particularly in photocatalysis and gas detection.

Several metal oxides have been tested as gas sensors such as ZnO [4], In₂O₃ [5], SnO₂ [6].

Recently, MoO₃ [7-10] is starting to take a place in the field of the environment.

Thin binary belongs to metallic transition element has emerged as a promising active oxide for various applications. Also, it is found that MoO₃ is a n-type semiconductor this property is related to oxygen vacancies in this binary compound. This binary exists in three main crystal structures which are orthorhombic α-MoO₃, monoclinic β-MoO₃ and hexagonal h-MoO₃ [11-14]. Particularly, α-MoO₃ has been considered as a potential photocatalyst material [15,16].

Numerous methods have been carried out to prepare MoO₃ thin films, such as MOCVD [17], chemical vapor transport (CVT) [18], sputtering [19] laser ablation [20] and spray pyrolysis [21,22].

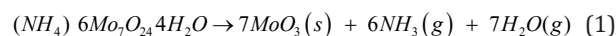
The latter is best suited for the preparation of thin films due to its performance in terms of the thin films homogeneity, the possibility of giving crystallized films without resorting to annealing

and the possibility of controlling the grains size by controlling the deposition conditions. The present work is devoted essentially to the study of ethanol detection by MoO₃ sprayed thin film.

MoO₃ Films Preparation and Characterization Techniques

Molybdenum oxide thin films were deposited on glass substrates at 460 °C using 10-2M Ammonium molybdate tetrahydrate [(NH₄)₆Mo₇O₂₄·4H₂O] aqueous solution. Spraying was assisted by a stream of nitrogen ensuring good fragmentation of the drops of the solution. During the deposition process, the precursor mixture flow rate was taken constantly at 4mL/min and the nozzle-to-substrate plane distance was fixed at 27cm.

The formation reaction of molybdenum trioxide from the solution of this precursor can be described by the following reaction:



After thin film preparation, a systematic study by XRD was carried out for the determination of films' structure. The atomic force microscope was used for morphological study. On the other hand, the Finally, The sensing properties of MoO₃ films based sensors under ethanol vapor at 300°C were Investigated.

Results and Discussion

Structural properties

XRD spectrum of prepared thin film (Figure 1) depicts the

presence of (020), (040), (131) and (261) peaks related to α - MoO_3 orthorhombic phase with (020) and (040) preferred orientations. From thin spectrum, orthorhombic lattice parameters were calculated. Their values are listed in Table 1. These values are closed with others reported elsewhere [23,24].

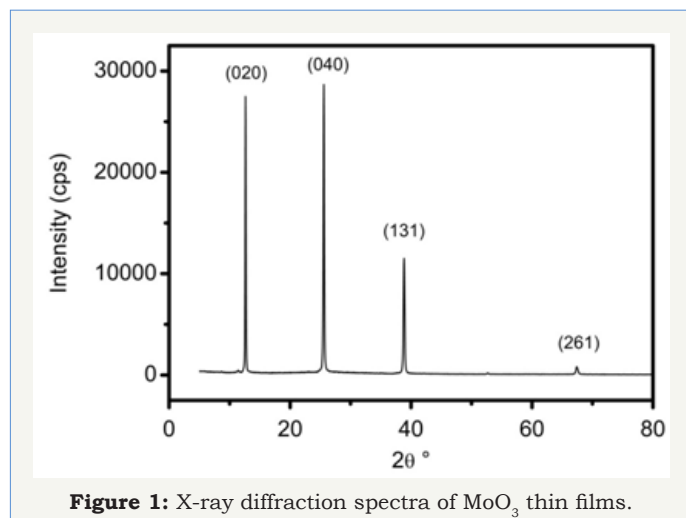


Figure 1: X-ray diffraction spectra of MoO_3 thin films.

Table 1: Lattice parameters of MoO_3 thin films.

a (Å)	b (Å)	c (Å)
3.926	14.007	3.645

Atomic force microscopy (AFM) observations

Surface topography of the MoO_3 thin film investigated by atomic force microscopy (AFM) is shown in Figure 2.

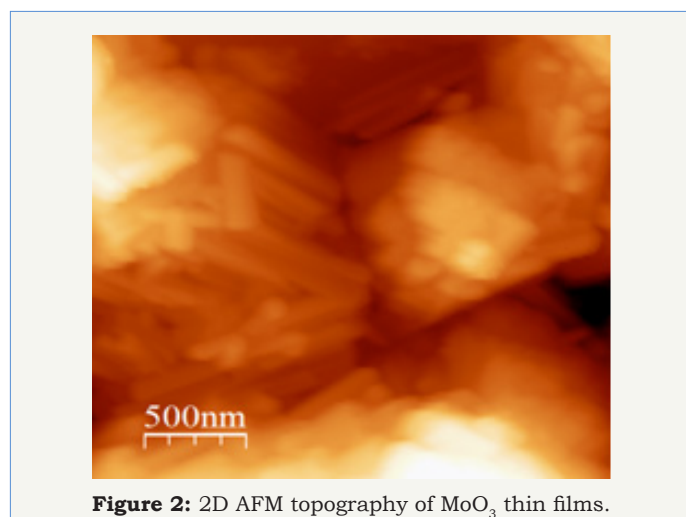


Figure 2: 2D AFM topography of MoO_3 thin films.

This AFM observation shows that the MoO_3 film is formed of parallelepiped shaped wires which can be considered as assembling orthorhombic meshes. The average wires length's is estimated at 500nm and the roughness mean square is around 121nm.

The sensing property of MoO_3 films against ethanol vapor was studied. by means of an appropriate experimental protocol. Figure 3 shows MoO_3 film responses at 500ppm ethanol measured at 300 °C.

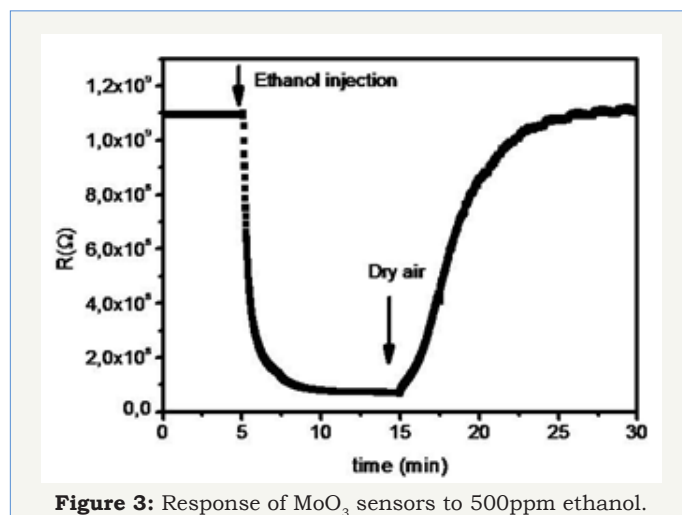


Figure 3: Response of MoO_3 sensors to 500ppm ethanol.

The exploitation of this response allows deducing the sensitivity of the detection which is given by the following relation [25]:

$$s = 100 \frac{R_{gas} - R_{air}}{R_{air}} \quad (2)$$

Where R_{gas} and R_{air} are the resistance of the device in the presence of ethanol and in air respectively. The value obtained from the sensitivity is estimated at 93%.

This value seems good. Indeed, it is related to a large variation in the resistance value, and therefore a good ethanol sensitivity using molybdenum trioxide. On the other hand, the study of several cycles shows reproducible ethanol detection (Figure 4). This shows that MoO_3 can be considered as a potential candidate as ethanol sensors.

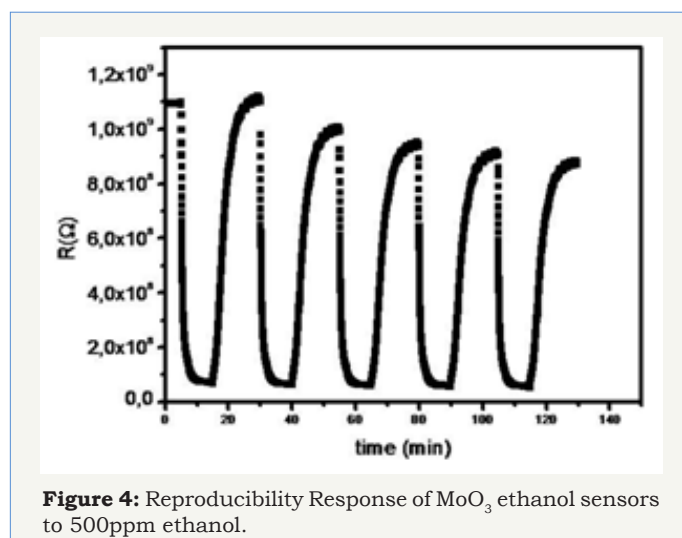


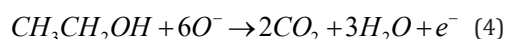
Figure 4: Reproducibility Response of MoO_3 ethanol sensors to 500ppm ethanol.

On the other hand, by examining the variation of the resistance of MoO_3 sample, it is possible to interpret the mechanism of ethanol sensing as follows.

The reaction mechanism of MoO_3 adsorbing ethanol gas is given by:



and



Where O^- denotes the oxygen ion on the surface of MoO_3 film and e^- is a conduction electron. When the zinc oxide film adsorbs ethanol gas, the mobility of electrons in the sensitive film increases because the electrons in the film increase which results in the decrease of MoO_3 thin film. The increase of the free carriers can be attributed to the n-type MoO_3 semiconductor binary compound [26-28]. This can explain the decrease of the resistance following the ethanol adsorption.

Conclusion

The molybdenum trioxide thin films have been successfully prepared on a glass substrate by a simple spray pyrolysis process at 460 °C. X-ray diffraction analysis shows that the prepared film is mainly formed by orthorhombic α - MoO_3 phase. The crystallites are preferentially oriented along (020) and (040) directions parallel to the substrate plane.

MoO_3 thin film sensors exhibited a high ethanol sensing. This result seems interesting. Indeed, with simple thin film prepared by cost-effective spray pyrolysis technique has been used to prepare such films. Further studies are in progress to improve the sensitivity of MoO_3 thin films in terms of effective reproducibility as well as its selectivity.

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