Silver Doped Niobium Pentoxide nanostructured thin film, Optical Structural and Morphological Properties

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Silver Doped Niobium Pentoxide nanostructured thin film, Optical Structural and Morphological Properties

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Abstract. Ag-Nb₂O₅ thin films were successfully prepared using a drop casting method. Structural properties ensure the presence of silver atom in the Nb₂O₅ polycrystalline thin films. The estimated energy gap from the optical absorption showed a reduction in the Eg value from (2.37 to 2.05) eV with the presence of Ag atom. SEM and EDs result also shows the presence of the silver atom at the structure of the film.

Keywords: Nb₂O₅ thin film, Ag dopant, XRD, energy gap, SEM, EDX

1. Introduction

Due to its versatile industrial applications, Nb₂O₅ has been widely used in electro-optic technology. It is a promising counter electrode material in optoelectronic devices [1-3]. These thin films also important electrochromic cathodic materials because of the large optical transmittance difference between the oxidized and reduced states. Niobium-based oxide thin films show a wide energy band gap ranged between (2.2- 3.4) eV so that, it would be expected to be capable of catalyzing contaminant degradation under UV irradiation. It has been found that Niobium oxide showed better photo-catalytic activity than other material such as zinc oxide [4, 5].

This material has a remarkable stability in the chemical reaction in addition to its resistance to corrosion in both base and acid media. Therefore it is used in a different electrochromic application. It’s also worth to know that, the properties of Nb₂O₅ thin films dependent directly on preparation method and the presence of dopants which show a considerable effect on the material characteristics [1, 6, and 7]. In the last 20 years, the large variety and the wide available possibilities make it possible to prepare doped and pure Nb₂O₅ thin film with specific properties. Such prepared thin films have been used to fabricate different important devices such as nanobatteries, solar cells, besides its application in the field of electrochromic interaction and catalysis [8].

In the previous work, lithium was used as a dopant, it found to enhance the electrochemical properties comparing with the pure crystalline Nb₂O₅ thin films [9]. In another work, the carbon-modified Nb₂O₅ was also investigated and found to have an excellent photochemical activity. In the oxide semiconductor materials, the most important difficulties that faced the photochemical and photoconductive properties is the electron – hole pares recombination process, remarkable efforts have been made to limit this effect in order to enhance electron – hole generation and recombination and hence the overall Photochemical efficiency. It has been found that coating semiconductor material with metal nanoparticles such as Ag, Pt, Pd, and Au, can exhibit enhanced PC activity due to the sinks effect induced by Nobel metal electrons.[10, 11] besides that, due to the lower Femi level value of the silver dopant compared with Nb₂O₅ thin films,
the PC would be highly enhanced. Another important application was reported dependent on the antibacterial activity of Ag nanoparticle, where Nb$_2$O$_5$ nanobelt decorated with Ag nanoparticle was investigated and found to have a considerable biomedical applications and strong synergistically antibacterial activities [12-15] in the present work, some physical properties of pure and Ag-doped Nb$_2$O$_5$ was characterized, we look forward to opening a new horizon to use chemical methods for deposition of films that could be used in photonics and renewable energy applications.

2. Experimental work

The required niobium pentoxide colloidal solution was obtained using a method depended in our previous published [16]. (0.2 g) of 99.99% Nb$_2$O$_5$ powder was dissolved using HF acid, ammonia, DIW, and ethanol with (1:1:4:8) Molar ratio. The reaction usually takes the following chemical formula

$$\text{Nb}_2\text{O}_5 + 10HF \rightarrow 2\text{H}_2\left[\text{NbOF}_2\right] + 3\text{H}_2\text{O}$$

(1)

$$2\text{H}_2\left[\text{NbOF}_2\right] + 10\text{NH}_4\text{OH} \rightarrow \text{Nb}_2\text{O}_5 \downarrow + 10\text{NH}_4\text{F} + 7\text{H}_2\text{O}$$

(2)

A drop casting method was employed to obtain homogeneous Nb$_2$O$_5$ thin films. The colloidal solution was deposited on silicon and quartz substrates at 100°C for 5 minute. Ag as impurities introduced to the film structure using UV activation method, where the Nb$_2$O$_5$ thin film was immersed in silver nitride AgNO$_3$ solution and then UV illumination.

T60 UV-Vis spectrophotometer was used to find the optical properties of the prepared films. Cu-ka x-ray source from Shimadzu 6000 was used to determine the structural properties, and finally, scanning electron microscope of (AA-3000) type was used to find morphological properties and EDX analysis.

3. Results and discussion

Figure (1) shows the X-ray diffraction results obtained for pure and Ag doped Nb$_2$O$_5$ thin films. All the obtained results are coinciding with the standard Card (00-030-0872) x-ray diffraction peaks. The successful preparation of the multi crystalline niobium pentoxide is obvious and could notice through the presence of the diffraction peak related to the diffraction planes (111), (213), (311), (704), (002), and (422) of the Nb$_2$O$_5$ substance. The diffraction peak at 20 = 21.1° is also clear which is related to (011) diffraction plane of the NbO$_2$ substance, similar diffraction peaks could be found elsewhere [4, 16].

![Figure 1. X-ray diffraction results of pure Nb$_2$O$_5$ thin films and Ag-Nb$_2$O$_5$](image-url)
The insertion of Ag atom into Nb$_2$O$_5$ thin films could recognize through the presence of the silver diffraction peak at 2$\theta$ = 38.2° which is related to the x-ray diffraction from (111) silver diffraction plane. In addition, the presence of the diffraction peak at 2$\theta$ = 35.4° is related to silver oxide (002) Ag$_2$O diffraction plane. A deviation of about 0.7° degree from the standard card may be related to the dislocation or stress in the films structure. These results are coinciding with x-ray diffraction standard card No. 01-087-0717 and 03-065-2871. Some of the diffraction peak related to the Nb$_2$O$_5$ found to disappear after doping with silver nanoparticle. This may be attributed to the recrystallization of the base material due to the insertion of dopant atoms into the main structure. Figure (2a, b) shows the absorption spectra of the pure and Ag-doped Nb$_2$O$_5$ thin films and the estimated band gaps values for both films prepared on quartz substrate.

![Absorption spectra](image)

**Figure 2.** Absorption results and $(\alpha h\nu)^2$ Vs. incident photon energy(hv) to estimate energy gap of a-pure
and b- doped Nb$_2$O$_5$ thin films

The clear absorption peak (300-400) nm of the Uv region is one of the specific characteristics of the Nb$_2$O$_5$ thin films. The increase in the absorption value for Ag-doped Nb$_2$O$_5$ thin shown in figure (b) is related to the presence of silver nanoparticle in addition to the presence of the silver oxide nanoparticle as shown in the x-ray diffraction results. The estimated energy gaps value for both pure and doped films dependent on the approximation formula of Tauc law i.e, $(\alpha h\nu) = A (h\nu-E_g)^{1/2}$ [17, 18] where $\alpha$ is the absorption coefficient and $h\nu$ is the incident photon energy and its absorbance A is given by the following mathematical expression [19] The energy gaps were obtained by extrapolating of the straight line of the $(\alpha h\nu)^2$ Vs. photon energy, the reduction in the obtained energy band gap value ensure the successful insertion of the Ag nanoparticles atom into the electronic structure of the Nb$_2$O$_5$ thin films.

The surface morphology of the prepared pure and Ag-doped Nb$_2$O$_5$ is shown in figure (3a, b). A dense sand dune like nanostructure is clear in figure (a) where the pure Nb$_2$O$_5$ thin films are prepared using simple and very simple drop casting method. The presence of such structure is related to the nature of the deposition method which permits to the chemical colloidal suspension of the reactant raw material to dry slowly at the substrate drop by drop growth.

The particularly uniform surface in figure (b) presences due to the effect of the immersion of the base sample i.e Nb$_2$O$_5$ thin film in the AgNO$_3$ chemical solution followed by the reduction using UV light for about twenty minutes resulting in presence of the Ag nanoparticles and absence of the sand dunes like structure.

The following figure (4) represents the EDs results for the Ag-doped Nb$_2$O$_5$ thin film. The presence of Nb, O, Ag elements are clear with their highlighted colors in figure (a). In the typical Nb$_2$O$_5$ structure the ratio of Nb/O has to be about 2.3 the deviation from this value to be about 0.36 is related to the presence and the insertion of the Ag atoms into the crystal base and be part of the crystal structure. The high percentage of the silicon is related to the substrate.
4. Conclusion

Poly-crystalline niobium pentoxide thin films were successfully doped by Ag atoms. This was ensured by the x-ray diffraction results. A shift in the absorption edge of the doped films towards the visible region coincidence with the fact that Ag and AgO molecules were introduced to the electronic structure of the prepared films resulting in energy gap reduction of the doped films. Eds results also agree with the previous physical properties.
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