Electrical conductivity inversion for Nb$_2$O$_5$ nanostructure thin films at different temperatures

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Abstract

The effect of treatment temperature on structural, morphological, and electrical properties of Nb$_2$O$_5$ thin films using the precipitation method is presented in this work. Clear enhancements, such as decreased stress and dislocation density values, are recognized in structural properties after heat treatment. AFM result shows increased average grain size and RMS of the prepared thin film with treatment temperature from 87.87 °C to 110.97 °C and rising temperature from 25 °C to 600 °C. Conductivity type conversion from n- to p-type is observed when the heat treatment is increased from 600 °C to 700 °C based on Hall effect measurement results.

1. Introduction

Niobium oxides (Nb$_2$O$_3$) elicit the interest of investigators due to their wide charge states, morphologies, phases, and connected characteristics. Nb$_2$O$_3$ can be found in different structures based on the number of niobium atoms, such as NbO, NbO$_2$, and Nb$_2$O$_3$ [1–4].

The fabrication method plays a significant function in the morphological characteristics of thin films and their implementation. Nb$_2$O$_3$ enforcements are broadly spreading; for example, Nb$_2$O$_3$ nanostructure is utilized in dye-sensitized solar cells as a photoanode due to its broad band gap, outstanding electron injection adequacy, and chemical stabilization [5–9]. Moreover, the high surface area of the nanostructured layers is appropriated for catalytic [10], photodetectors [11], sensing [12], and battery [13] instruments. Regarding optical coatings, soft and monotonous Nb$_2$O$_3$ films are preferred to reduce the dissipation of light [14]. The Nb$_2$O$_3$ preparative processes are strongly effective on the morphology and the functional characteristics [15].

New reports focused on Nb$_2$O$_3$ incorporation in electrode materials in the implementations of the supercapacitor, which has produced remarkable achievements ascribed to the pseudocapacitance effect [16–18].

However, the niobium–oxygen system is especially compound due to the considerable influence of tight distortions from the accurate stoichiometry in Nb$_2$O$_3$ on the material physical characteristics. For example, a slight oxygen reduction results in the transformation from n-type insulating to semiconducting conduct [19].

Structural, dielectric, and optical properties of amorphous Nb$_2$O$_3$ films have already been reported in literature results [20–25]. These results show that film properties strongly depend on the fabrication method and post-deposition treatments, such as annealing [26–29]. A progressive redshift in the energy gap values from 3.62 eV to 3.07 eV is observed with rising annealing temperatures [20–32]. This redshift can be due to the crystal phase transformation and the rising oxygen ion vacancy, which increases the localized state concentration in the band structure. Moreover, film crystallinity considerably influences the material refractive index as it drops from 2.3 to 2.2 via annealing [33–36]. Furthermore, the amorphous Nb2O5 thin-film phase is altered via annealing to pseudo-hexagonal crystal (TT-Nb$_2$O$_3$) and orthorhombic (T-Nb2O5) phases [37–40]. The atomic force microscope results showed that extreme annealing temperature realized the smallest grain sizes and large grain
As shown in previous works, the effect of treatment temperatures on the optical, structural, and morphological properties of Nb$_2$O$_5$ films has been investigated [41–49] as a continuation.

The current work focuses on the effect of post-annealing on the electrical properties and types of electrical conductivity of Nb$_2$O$_5$ thin films synthesized using the chemical method.

2. Experimental work

The Nb$_2$O$_5$ suspension was prepared using the precipitation method by adding hydrofluoric acid and 12 M ammonium hydroxide [17, 50] to Nb$_2$O$_5$ powder. Thin films were deposited on the silicon substrate (for structural and morphological measurements) and the glass substrate (for electrical measurements) on the (2 × 2 cm) glass substrate (for electrical measurements) by employing a spin-coating method at 3000 rpm, and the thickness of the deposited film was (450 nm).

The prepared thin films were treated at different temperatures ranging from 200 °C to 700 °C using the classical method employing furnace (Nabertherm GmbH, Germany).

The structural properties of all prepared films deposited on the silicon substrate were analyzed with x-ray diffraction (XRD) methods. XRD measurement was performed and matched with the ASTM cards, and structural analyses were performed using Shimadzu 6000 x-ray diffractometer with wavelength $\lambda = 1.5406$ Å using the Cu-K$_\alpha$ target. The thin films were scanned from $(2\theta = 10^\circ–60^\circ)$ Structural parameters were extracted from structural properties using the following equations: [51–56]

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$ \hspace{1cm} (1)

$$\delta = \frac{1}{D^2}$$ \hspace{1cm} (2)

$$\varepsilon = \frac{\beta}{4 \tan \theta}$$ \hspace{1cm} (3)

where $D$ is the grain size in (nm) unit, $\beta$ is the full width of the peaks at the half maximum in (rad) unit, $\theta$ is the Bragg angle in (deg) unit, $\delta$ is the density of dislocation that indicates the number of dislocations in a unit volume, and $\varepsilon$ is the stress [57–61].

Morphological properties, such as average grain size, surface roughness, and root mean square of the prepared Nb$_2$O$_5$ thin film deposited on a silicon substrate, were investigated by atomic force microscopy (AFM) (SPM AA3000, Angstrom Advanced Inc., USA). The contact mode was selected to scan the samples for RMS surface roughness calculation.

The resistance of the Nb$_2$O$_5$ thin films deposited on the glass substrate was measured in the temperature range of 40 °C to 200 °C (with 10 °C temperature step) by using (KEITHLEY 616) meter. Aluminum metal electrodes were deposited on the films by using masks. The distance between the two contacts is 2 cm, while the contact dimensions are 0.5 cm × 1 cm. The activation energy (Ea) value was extracted by plotting log $\sigma$ versus 1000/T. The Ea value was evaluated from the slope of the straight line, as provided in equation (4) [62, 63]:

$$Ea = 0.86 \times \text{slope}$$ \hspace{1cm} (4)

The conductivity type, carrier concentration, and carrier mobility were measured using (HMS3000 Ecopia).

3. Result and discussion

3.1. Structural properties

The crystal structure and orientation of the Nb$_2$O$_5$ films before and after heat treatment at different temperatures were demonstrated by XRD patterns, as shown in figure 1.

The crystallization of the prepared films was enhanced. This enhancement demonstrated an increase in the peak intensity with heat treatment. The figure shows no change in the diffraction peak position. Moreover, new additional diffraction peaks have emerged. This emergence indicates the absence of new phases with heat treatment according to the standard cards (No. 00-037-1468).

The intensity of the main diffraction peak at $2\theta = 17.2^\circ$ corresponding to the (301) diffraction plane increased and sharpened with the temperature. In addition, the intensity of the second diffraction peak at $2\theta = 14.2^\circ$ corresponding to the (−203) diffraction plane underwent a relative reduction in the intensity with a low full width at half maximum (FWHM). The small peak at $2\theta = 18.8^\circ$ corresponding to the (−403) diffraction plane completely disappeared at 300 °C. These phenomena exhibited an improved crystalline structure and indicated the general conduciveness of this structure. This result agrees with that of other published data.
Structural parameters, such as grain size, stress, and density of dislocation, were investigated by respectively using equations (1), (2), and (3), as listed in Table 1.

The table shows that the crystallite size values increased from 9.49 nm to 34.7 nm due to the increase in the heat treatment temperature from 200 °C to 600 °C. This result may be attributed to the particle formation through the heat treatment process. During heating, these crystallite structures collided, increased, and coalesced with one another, thus forming a large particle. This result may also be imputed to the merging process induced by annealing treatment. Numerous dangling bonds concerning the defects of metal and oxygen were observed in metal oxides. Such defects were located at the grain boundaries. Consequently, these defects were preferred for the merging process to facilitate the production of large grains with increased treatment temperature [56, 65]. The stress value decreased due to the reduction in heat treatment from 0.23 to 0.06 caused by the increase in the heat treatment temperature from 200 °C to 600 °C. This phenomenon indicated the relaxation in Nb2O5 thin films. This relaxation may be attributed to the reduction in the number of imperfections during deposition. As the atoms deposited with a slight disordering, that is, as the temperature increased, the atoms had additional energy to adjust their position [57, 66]. Dislocation density also decreased with temperature, indicating reductions in lattice imperfection concentration [58]. An increase in annealing temperature up to 700 °C also demonstrated a relative reduction in the crystallite size, which results in the breaking of Nb-O bonds. This phenomenon facilitated the free movement of atoms to their identical stable sites, leading to further defects and additional stress as shown in Figure 2. These results are consistent with the published data [67, 68].

Table 1. Structural properties extracted from XRD result.

| Temperature (°C) | D (nm) | Stress | dislocation density (1/nm²) |
|-----------------|--------|--------|----------------------------|
| RT (as-deposited)| 9.49   | 0.23   | 1.11E-02                   |
| 200             | 9.96   | 0.22   | 1.01E-02                   |
| 300             | 10.90  | 0.20   | 8.41E-03                   |
| 400             | 12.78  | 0.17   | 6.12E-03                   |
| 500             | 17.08  | 0.13   | 3.43E-03                   |
| 600             | 34.70  | 0.06   | 8.30E-04                   |
| 700             | 33.90  | 0.07   | 8.70E-04                   |

Figure 1. XRD results of the Nb2O5 thin film treated at different temperatures.
3.2. Surface roughness
The effect of heat treatment on the surface roughness of the prepared Nb2O5 thin films at 12 mol l\(^{-1}\) molarity is illustrated in figure 3. The surface roughness of the film varied with the heat treatment, and all films also attained the shape of the hills and the valley structure.

Table 2 shows that the grain size increased from 87.87 nm to 110.79 nm as the temperature rose from 200 °C to 600 °C. This increase is related to the migration stimulation of grain boundaries at high temperatures, resulting in the coalescence of two or more grains through annealing. The result also indicates that treatment with high temperatures will produce additional energy for atoms. Thus, atoms can diffuse through the crystal and occupy suitable sites, producing grains with low surface energy; this finding is consistent with other research\[69]\.

An additional increase in the treatment temperature caused a reduction in grain size. These results were previously confirmed by XRD measurements and agreed with other research works\[70]\.

The grain size value calculated by AFM analysis was larger than the average crystallite size measured from the XRD data possibly due to the formation of large grains by the small crystallites\[71]\.

The surface roughness influenced the performance of the devices based on transparent conductive thin films. Thus, mapping the surface roughness of the fabricated films before the production of photoelectric devices is crucial. The roughness of the treated thin film decreased with the increase in heat treatment, but the decrease was minimal due to the original low value. The value rose as the temperature was increased up to 700 °C. The most uniform and homogenous thin films with low roughness value were produced by treatment at 600 °C. Thus, these temperatures are the most suitable heat treatment temperatures from the topography viewpoint.

3.3. Electrical properties
The negative sign of the Hall coefficient over the range of 200 °C–600 °C heat treatment indicated the production of donor (n-type) conductivity thin films, as shown in figure 4.

A dramatic change in conductivity was observed at 700 °C. This change is attributed to lattice displacement, vacancies, and interstitial atoms. This conductivity conversion depending on the number of acceptor levels or ‘holes’ relevant to the heat treatment can be explained using the following two mechanisms: chemical and mechanical. In the chemical mechanism, the effective concentration of acceptor impurities, such as the change in solubility with temperature, must be modified by heat treatment. In the mechanical mechanism, heat treatment is assumed to break the valence bonds through the displacement of atoms in the lattice to create hole-like defects that recompense the existing donors and sequentially result in p-type conversion. Beyond the conversion point, additional heat treatment introduces added defects, which also improve the conductivity. Heat treatment at low temperatures around 500 °C enables the treatment of these defects, demonstrating a return to their initial properties as shown in other articles\[73, 74]\.

The dependence of conductivity conversation
Figure 3. Two and three dimensional AFM images of the Nb₂O₅ thin films at different heat treatment temperatures A: 200 °C, B: 400 °C, C: 500 °C, D: 600 °C, E: 700 °C.
on temperature is a function of material impurity, that is, the transformation temperature increases with the impurity level. This condition extends to a determined level at which the conductivity converts as much as the temperature increase, as shown in other works [73].

The n- to p-type conductivity conversion was observed in many semiconductor materials, such as pure germanium at 800 °C, GaAs at 700 °C, and N: Mg at 220 °C, as shown in other works [75–77].

The variation in carrier concentration as a function of heat treatment temperature is presented in figure 5. This variation is due to the change in the donor–acceptor balance. In doped materials, the p-type conductivity is generally proportional to the number of impurity atoms introduced in the material. By contrast, the conductivity in pure materials may arise from acceptor levels introduced by lattice defects due to the heat treatment. These defects resulted in atom displacement from the original lattice sites.

Furthermore, if donor and acceptor carrier levels occur concurrently, then a compensating action emerges such that the mechanism and magnitude of the electrical conductivity depend on the abundance of the carrier level and its final concentration. In the n-type film, as the treatment temperature increases, the acceptor concentration slightly rises and the final donor concentration decreases, as shown in figure 6(I). An additional increase in the treatment temperature up to 700 °C also raises the acceptor level. Consequently, the acceptor net concentration exceeds the donor concentration, as shown in figure 6(II).

The resistivity value increased due to the heat treatment effect and reached a maximum value at 600 °C, where the concentration of donors and acceptors only equalize or compensate each other, as shown in figure 6(I). Moreover, an increase in heat treatment temperature beyond 700 °C acceptor concentration is regarded as an excess. Thus, the material will be p-type, and the acceptor concentration will continuously increase. Furthermore, the resistivity accordingly decreases (figure 6(II)).

![Figure 4. Hall coefficient for Nb₂O₅ thin films as a function of the heat treatment temperatures.](image)

**Table 2.** Average Grain Size and Root mean square of the prepared thin film extracted from AFM result.

| Heat treatment temperature (°C) | Average grain size (nm) | RMS (nm) |
|-------------------------------|-------------------------|----------|
| 200                           | 87.87                   | 1.51     |
| 400                           | 92.35                   | 1.42     |
| 500                           | 98.84                   | 1.21     |
| 600                           | 110.79                  | 1.16     |
| 700                           | 107.34                  | 3.27     |
The mobility of free carriers in semiconductor thin films could be interpreted via the barrier effect of the grain boundary due to the abundance of grains in any film. These grains are separated with boundaries containing a pair of atomic layers of disorganized atoms. This disorganization creates many defects and prevents the passing of carriers from one grain to another, working as potential barriers; the height of this potential barrier depends on the grain size \[41\].

Figure 7 shows the relationship between grain size and mobility of carriers as a function of temperature. The
carrier mobility increases as the temperature rises from RT to 600 °C due to the increase in the grain size caused by the heat treatment, as shown in figure 7 (blue curve). This result agrees with published data for different materials [78]. Further increase in the temperature above 600 °C resulted in an evident decrease in mobility despite the relative reduction in grain size. This behavior is attributed to the lower mobility of the hole (majority carrier) in p-type thin films (transformation at a treatment temperature between 600 °C–700 °C) than that in n-type thin films.

As shown in figure 8, the electrical resistivity of Nb$_2$O$_5$ thin films was estimated across a broad range of temperatures (40 °C–200 °C) to test their semiconducting characteristics as a function of heat treatment temperature (200 °C, 300 °C, 400 °C, 500 °C, 600 °C, and 700 °C).
The figure reveals that the resistivity of all thin films decreases with the annealing temperature, thus exhibiting semiconductor behaviors [79]. The resistivity also increased with the heat treatment temperature, reaching its maximum value at more than 600 °C and then decreasing again. This result coincides with that extracted from Hall effect measurements.

A plot of the natural logarithm of the conductivity versus 1000/T was used to evaluate the Ea. Taking the slope of the fitted straight portion of the curve shown in figure 9, the relationship between activation energy and heat treatment temperature is displayed in figure 10.

Figure 10 shows the relationship between the activation energy of the thin films and the treatment temperature. The values of the activation energy increased from 0.72 to 0.77 with rising treatment temperature.
from RT to 600 °C. This result may be attributed to the decrease in carrier concentration with heat treatment. Further increase in the temperature caused a slight reduction in activation energy value because the hole density in the p-type thin film treated at 700 °C slightly exceeded that of the electron in n-type treated at 600 °C.

4. Conclusion

High-quality Nb₂O₅ thin films could be obtained using the precipitation method. The properties of these films could be improved via treatment at different temperatures. The crystallization of the prepared films was enhanced with heat treatment. The most important achievement was the conversion of the electrical conductivity of the prepared films from n- to p-type at high heat treatment of approximately 700 °C. Optimum physical properties of the films could be obtained via treatment at 600 °C.

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