Investigation on structural and morphological properties of molybdenum trioxide (MoO$_3$) thin films

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Research Article

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Abstract

The present study reports on the effect of substrate temperature on structural, morphological and optical properties of MoO$_3$ thin films. MoO$_3$ thin films were deposited on pre-heated glass substrate using spray pyrolysis technique. The substrate temperature was varied from 300 °C to 400°C with a step interval of 50 °C. Structural studies were studied using X-ray diffraction technique. It is observed that all the diffraction peaks exactly match with JCPDS card No. 05-0508. The as –deposited MoO$_3$ thin films exhibits orthorhombic crystal structure. It is found that the crystalline nature increases with the increases of substrate temperature. FESEM micrographs show that the grains are distributed uniformly over the surface without any void. An optical property reveals that the transmission of MoO$_3$ thin film in the visible region increases with increases of deposition temperature.

1. Introduction

Scientists are drawn much attention towards transition metal oxides due to their interesting physics and engineering applications. As a result of their different oxidation states, they exhibit promising structural, electrical, magnetic, and optical properties, as well as high melting points\[1–2\]. Molybdenum trioxide (MoO$_3$) is one of the transition metal oxides, which attracted scientific community owing to its attracting properties such as tunable band gap, various stable oxidation states \[3\]. In additions, molybdenum oxide (MoO$_3$) exhibits interesting structural, chemical, electrical, and optical properties that make it an ideal metal oxide for use in chemical, optical, and electronic applications \[4\].

Due to the various exceptional properties MoO$_3$ finds potential applications in diverse fields such as batteries, smart window, optical switches, catalysis, solar cells, gas sensors, and energy storage \[5–8\]. MoO$_3$ thin films exist in three phases, namely: (i) orthorhombic $\alpha$-phase, (ii) monoclinic $\beta$-phase, and (iii) hexagonal $h$-phase \[9\]. The $\alpha$-MoO$_3$ phase is formed by two bi-layers parallel to the (010) plane. The edge forms the $\alpha$-phase of MoO$_3$ and corner-sharing MoO$_6$ octahedral linked to create bilayer sheets that are stacked in the (010) direction. Additionally, MoO$_3$ has versatile morphological structures, including nanobelts, nanorods, nanowires, etc., offering a high surface-to-volume ratio \[10–12\]. Gases such as nitrous oxide, nitrogen dioxide, carbon monoxide, and ammonia are extremely sensitive to MoO3 thin films at temperatures between 573 and 873 K \[13\]. Because of their morphology, MoO3 nanobelts have a high rate capacity, so they are used in hybrid electric vehicles as cathode materials for rechargeable lithium batteries. Molybdenum oxide has the highest oxygen concentration and acts as a doping center that controls the material's electrical and optical properties \[14\].

The MoO$_3$ thin films have been deposited using different deposition techniques such as sputtering \[15\], thermal evaporation \[16\], atomic layer deposition \[17\], spray pyrolysis \[18\], hydrothermal method \[19\], sol-gel spin coating \[20\] etc. Nirupamaetal et al \[21\] reported the formation of nanostructured -MoO$_3$ thin films by magnetron sputtering was reported in \[22\]. Bouzidietal. \[23\] analysed the effects of substrate temperature on the structural and optical properties of MoO$_3$ thin films.
In the present research work spray pyrolysis technique were used for the deposition of MoO$_3$ thin films on glass substrate for different substrate temperature. The effect of deposition temperature on structural and morphological properties of MoO$_3$ thin films was investigated.

2. Experimental details

Molybdenum trioxide (MoO$_3$) thin films were deposited on ultra-sonicated clean and pre-heated glass substrate using spray pyrolysis technique. The substrate temperature was varied from 300 to 400 °C with a step interval of 50 °C. Initially, 0.01M of ammonium hexamolybdate tetrahydrate was dissolved in de-ionized (D.I) water and stirrer for 30 minutes to get homogenous solution. The spray nozzle and substrate distance was fixed at 45 cm.

The chemical reaction between solvent and solute is given as follows.

$$(NH_4)_6Mo_7O_{24} \cdot 4H_2O + H_2O \rightarrow 7MoO_3(s) + 6NH_3(g) + 7H_2O(g)$$

The MoO$_3$ thin films which are deposited at 300 °C, 350 °C and 400 °C were coded as MoO$_3$ (300), MoO$_3$ (350) and MoO$_3$ (400) respectively. Structural studies were carried out using X-ray diffractometer (XRD, Bruker, Germany) with CuK$_{a1}$ radiation of wavelength 1.5406Å. Surface morphology of the films was studied from Field Emission Scanning Electron Microscope (FE-SEM, JEOL-6701F, Japan).

Description of experimental set-up

Fig. 1 shows the schematic illustration of spray pyrolysis technique. A suitable mixture of chemical compounds is dissolved in the solvent (usually deionized water, D.I) to form the precursor solution. The prepared final solution was sprayed upon a pre-heated substrate using a carrier gas. The fine droplets of the final prepared solution undergo pyrolytic decomposition upon reaching the hot substrate, and eventually, uniform thin films of semiconductors were deposited on the substrate. The important components of the home made spray pyrolysis set-up include (i) a spray nozzle, a hot plate, a carrier gas regulator, and an airtight chamber.

Upon applying the pressure, a vacuum is formed at the nozzle’s tip as result the prepared solution were sprayed upon pre-heated glass substrate using spray gun (Spray nozzle). The hot plate is usually made of an iron disc with a thermocouple at the center to measure the temperature. The carrier gas regulator controls the pressure of the carrier gas flowing through the spray gun. When the precursor solution was sprayed, the toxic gases could be released. Hence, the entire spray set-up was mounted with an airtight chamber.

3. Results and discussion

3.1 Structural studies
X-ray diffraction pattern of MoO$_3$ (300), MoO$_3$ (350) and MoO$_3$ (400) thin films is shown in fig. 2. It is observed that all the diffraction peaks are well matched with JCPDS card number 05–0508 and also confirm the α-orthorhombic structure of as-deposited MoO$_3$ thin films [24]. Furthermore, no structural rearrangement and impurity phase were observed which shows the purity of prepared MoO$_3$ thin films. More importantly, XRD pattern of MoO$_3$ reveals that on increasing substrate temperature the crystallinity of MoO$_3$ thin film also increases. The crystallite size of thin film was calculated using Sherrer formula [25] and it is found the average value of crystallite size varied from 36 nm to 42 nm. The relation used for the crystallize size of MoO$_3$ thin films is given as follows:

\[
\text{Crystallite size (D)} = \frac{0.9\lambda}{\beta \cos (\theta)}
\]  

(1)

Where \(\lambda\) is wavelength of x-ray (\(\lambda=1.5406\ \text{Å}\)), \(D\) denotes crystallite size.

The value of the internal strain and dislocation density of MoO3 thin films was calculated using the following relation [25,26]. Table 1 shows the crystallite size, position of (021) peak, strain (\(\varepsilon\)), and dislocation density (\(\delta\)) values of MoO3(20), MoO3(30), MoO3(40) and MoO3(50) thin films.

\[
\text{Stain (}\varepsilon\text{)} = \frac{\beta \cos \theta}{4}
\]  

(1)

\[
\text{Dislocation density (}\delta\text{)} = \frac{1}{D^2}
\]  

(2)

where \(\beta\) denotes the full width at half maximum (FWHM), \(\theta\) denotes the diffraction angle and \(D\) is crystallite size of the deposited MoO3 thin films. Table 1 shows the crystallite size and average thickness, strain and dislocation density of MoO3 (300) MoO3 (350) MoO3 (400) thin films.

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### 3.1 Morphological studies

Fig. 3 exhibits the FESEM micrographs of MoO$_3$ thin films. The FESEM micrograph of as-deposited thin films reveals that the grains are distributed uniformly over the surface without any voids. From the analysis it is found that the crystalline nature of MoO3 thin films enhanced with the increase of substrate temperature.

### 4. Conclusion

In summary, MoO$_3$ thin films for different substrate temperature were deposited on glass substrate using spray pyrolysis technique. The effect of deposition temperature on structural and morphological properties of MoO$_3$ thin films was studied. Structural analysis reveals that as the deposition temperature
increases the crystallinity nature of as-deposited MoO3 thin films also increases. Morphological studies reveal that the grain is distributed uniform over the surface. Hence it has been concluded that the substrate temperature is an important factor which determines the quality of thin films.

References


**Table**

Table 1: the crystallite size and average thickness, strain and dislocation density of MoO$_3$ (300) MoO$_3$ (350) MoO$_3$ (400) thin films.
<table>
<thead>
<tr>
<th>Sample</th>
<th>Crystallite size (nm)</th>
<th>Thickness (nm)</th>
<th>Strain ($\varepsilon$) $\times 10^2$</th>
<th>Dislocation density ($\delta$) $\times 10^{14}$ (lines/m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MoO$_3$ (300)</td>
<td>39</td>
<td>410</td>
<td>2.5</td>
<td>9.6</td>
</tr>
<tr>
<td>MoO$_3$ (350)</td>
<td>41</td>
<td>385</td>
<td>2.4</td>
<td>8.7</td>
</tr>
<tr>
<td>MoO$_3$ (400)</td>
<td>44</td>
<td>325</td>
<td>2.2</td>
<td>7.2</td>
</tr>
</tbody>
</table>

**Figures**

**Figure 1**

The schematic illustration of spray pyrolysis technique
Figure 2

X-ray diffraction pattern of MoO$_3$ (300), MoO$_3$ (350) and MoO$_3$ (400) thin films.

Figure 3
FESEM micrographs of (a) MoO$_3$ (300) and (b) MoO$_3$ (400) thin films.