

Research Article

Growth of MoO₃ Films by RF Magnetron Sputtering: Studies on the Structural, Optical, and Electrochromic Properties

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Molybdenum oxide (MoO₃) films were deposited on glass and silicon substrates held at temperature 473 K by RF magnetron sputtering of molybdenum target at various oxygen partial pressures in the range 8×10^{-5} – 8×10^{-4} mbar. The deposited MoO₃ films were characterized for their chemical composition, crystallographic structure, surface morphology, chemical binding configuration, and optical properties. The films formed at oxygen partial pressure of 4×10^{-4} mbar were nearly stoichiometric and nanocrystalline MoO₃ with crystallite size of 27 nm. The Fourier transform infrared spectrum of the films formed at 4×10^{-4} mbar exhibited the characteristics vibrational bands of MoO₃. The optical band gap of the films increased from 3.11 to 3.28 eV, and the refractive index increased from 2.04 to 2.16 with the increase of oxygen partial pressure from 8×10^{-5} to 8×10^{-4} mbar, respectively. The electrochromic performance of MoO₃ films formed on ITO coated glass substrates was studied and achieved the optical modulation of about 13% with color efficiency of about 20 cm²/C.

1. Introduction

Transition metal oxides constitute an interesting group of semiconducting materials because of their technological applications in various fields such as display devices, optical smart windows, electrochromic devices, and gas sensors [1, 2]. Among the transition metal oxides, molybdenum oxide (MoO₃) exhibits interesting structural, chemical, and optical properties. MoO₃ finds application as a cathode material in the development of high energy density solid state microbatteries [3, 4]. It is considered as a chromogenic material since it exhibits electro-, photo-, and gasochromic (coloration) effects by virtue of which material is of potential for the development of electronic display devices [5]. MoO₃ films in nanocrystalline form also find applications in sensors and lubricants [6]. It is also a promising candidate as a back contact layer for cadmium telluride solar cells in superstrate configuration because of its high work function, which possibly reduces the back contact barrier [7]. Various physical thin film deposition techniques such as thermal evaporation [8, 9], electron beam evaporation [10, 11], pulsed laser deposition [12, 13], and sputtering [14–18] and chemical methods such as electrodeposition [19], chemical vapour deposition [20],

spray pyrolysis [21, 22], and sol-gel process [23–25] were employed for the growth of MoO₃ films. Among these films deposition techniques, magnetron sputter deposition is an industrially practiced technique for the growth of oxide films. The physical properties of the sputter deposited MoO₃ films depend critically on the sputter parameters such as oxygen partial pressure, substrate temperature, substrate bias voltage, sputter power, and sputter pressure. The influence of annealing temperature on the structural and optical properties of RF magnetron sputtered MoO₃ films was earlier reported [18]. In the present investigation, MoO₃ films were formed by RF magnetron sputtering of metallic molybdenum target at different oxygen partial pressures. The effect of oxygen partial pressure on the chemical composition, crystallographic structure, surface morphology and optical properties was studied, and the results were reported.

2. Experimental Details

2.1. Preparation of MoO₃ Thin Films. MoO₃ thin films were deposited onto glass and silicon substrates held at temperature of 473 K by sputtering of pure metallic molybdenum

target in oxygen and argon gas mixture using reactive RF magnetron sputtering technique. Metallic molybdenum target (99.99% pure) with 50 mm diameter and 3 mm thickness was used as sputter target. The sputter chamber was evacuated using a diffusion pump and rotary pump combination to achieve ultimate pressure of 4×10^{-6} mbar. Pressure in the sputter chamber was measured with digital Pirani-Penning gauge combination. Oxygen and argon (99.99% purity) were used as reactive and sputter gases, respectively, for deposition of MoO₃ films. After achieving ultimate pressure, required quantities of oxygen and argon gases were admitted into the sputter chamber through fine controlled needle valves followed by Aalborg mass flow controllers (Model GFC 17). RF power of 150 W was supplied to the sputter target using power supply (Advanced Energy Model ATX-600W) for deposition of the experimental films. The sputter parameters fixed during the growth of the MoO₃ films are given in Table 1.

2.2. Characterization Methods of MoO₃ Thin Films. The MoO₃ films formed at various oxygen partial pressure were characterized by studying their chemical composition, crystallographic structure surface morphology, chemical binding configuration, and optical properties. The thickness of the deposited films was measured with a mechanical Veeco Dektak (Model 150) depth profilometer. The chemical composition of the films was analysed with energy dispersive X-ray analysis (Oxford Instruments Inca Penta FETx3) attached to a scanning electron microscope (Carl Zeiss, model EVO MA15). The crystallographic structure of the films was determined by X-ray diffractometer (Bruker D8 advance diffractometer) using copper K_{α} radiation with wavelength of $\lambda = 0.15406$ nm. The X-ray diffraction profiles were recorded in the 2θ range $10-60^{\circ}$ in steps of 0.05° . The surface morphology of the films was analysed with a scanning electron microscope (Hitachi SEM Model S-400). The chemical binding configuration of the films formed on silicon substrates was analyzed with Fourier transform infrared spectrophotometer (Nicolet Magana IR 750), recorded in the wavenumber range $300-1500$ cm⁻¹. The optical transmittance of the films formed on glass substrates was recorded using UV-Vis-NIR double beam spectrophotometer (Perkin Elmer Spectrophotometer Lambda 950) in the wavelength range $300-1500$ nm. The electrochromic properties of the MoO₃ films formed at oxygen partial pressure 4×10^{-4} mbar were investigated by three-electrode cell with platinum as a counter electrode, Ag/AgCl as a reference electrode, and the ITO coated MoO₃ films as a working electrode using an EC (Model-CHI 608). The colored and virgin states of the films were measured by UV-Vis-NIR double beam spectrophotometer.

3. Result and Discussion

3.1. Deposition Rate. The thickness of the deposited MoO₃ films determined by using Veeco depth profilometer was $1.3 \mu\text{m}$ for the films deposited at 8×10^{-5} mbar. As the oxygen partial pressure increased from 2×10^{-4} to 4×10^{-4} mbar, the thickness was increased from 1.8 to $2.2 \mu\text{m}$. Further increase

TABLE 1: Sputter parameters fixed during the growth of the MoO₃ films.

| Sputter target | Molybdenum (50 mm diameter and 3 mm thick) |
|--|--|
| Target to substrate distance | 65 mm |
| Ultimate pressure | 4×10^{-6} mbar |
| Oxygen partial pressure (pO ₂) | 8×10^{-5} – 8×10^{-4} mbar |
| Substrate temperature (T_s) | 473 K |
| Sputter pressure | 4×10^{-2} mbar |
| Sputter power | 150 W |
| Deposition time | 120 min |

of oxygen partial pressure to 8×10^{-4} mbar decreased the thickness to $1.9 \mu\text{m}$. The deposition rate of the films was calculated from the measured film thickness and duration of deposition. Figure 1 shows the dependence of deposition rate of MoO₃ films on the oxygen partial pressure. At low oxygen partial pressure of 8×10^{-5} mbar, the deposition rate of MoO₃ films was 11.2 nm/min. The deposition rate gradually increased to 15.5 nm/min with the increase of oxygen partial pressure to 2×10^{-4} mbar and reached 18.3 nm/min for oxygen partial pressure 4×10^{-4} mbar. On further increase of oxygen partial pressure to 8×10^{-4} , the deposition rate decreased to 16.2 nm/min. At low oxygen partial pressures, the increase of deposition rate with the increase of oxygen partial pressure was due to the effective reaction between the metallic molybdenum and oxygen and hence, the increase in the deposition rate. At higher oxygen partial pressures, the chemical reaction between the target surface and the reactive oxygen gas forms molybdenum oxide layer on the target which reduced the deposition rate as observed by Mohamed et al. [26]. Scarminio et al. [5] also noticed such an increase of deposition rate with the increase of oxygen partial pressures and then a decrease at higher oxygen partial pressures in RF sputtered MoO₃ films. The observed decrease in the deposition rate with increase of oxygen partial pressure was due to target poisoning by oxygen atoms, the negative ion impingement on the target surface reduce the film growth [27].

3.2. Composition Analysis. The chemical composition of the MoO₃ films was determined by using energy dispersive X-ray analysis (EDAX). Figure 2 shows the representative EDAX spectra of the MoO₃ films deposited at oxygen partial pressures of 8×10^{-5} and 4×10^{-4} mbar. The chemical constituents present in the MoO₃ films formed at different oxygen partial pressures are given in Table 2. It is seen that the films formed at low oxygen partial pressure of 8×10^{-5} mbar contained less quantity of oxygen required to form a compound of molybdenum oxide. It revealed that the deposited films contained MoO₃ along with molybdenum. As the oxygen partial pressure increased to 4×10^{-4} mbar, the formed films showed that the atomic ratio of oxygen to molybdenum was $2.98:1$ which indicated that the stoichiometric MoO₃ films were deposited at 4×10^{-4} mbar. Siciliano et al. [28] also conclude that the atomic ratio of O and Mo was $3:1$ in

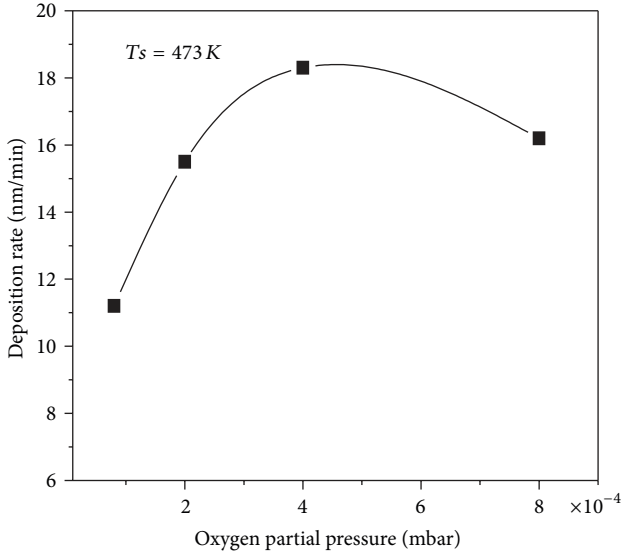


FIGURE 1: Variation of deposition rate of MoO₃ films with oxygen partial pressure.

the sputter deposited MoO₃ films. It revealed that oxygen partial pressure of 4×10^{-4} mbar is an optimum to achieve stoichiometric MoO₃ films. Further with the increase of oxygen partial pressure to 8×10^{-4} mbar, the atomic ratio of oxygen to molybdenum was found to be 3.03. This indicates that the films were overstoichiometric.

3.3. Structural Studies. Crystallographic structure of the films was analyzed by the X-ray diffraction. Figure 3 shows the X-ray diffraction (XRD) profiles of the MoO₃ films deposited at different oxygen partial pressures. The films formed at low oxygen partial pressure of 8×10^{-5} mbar showed that the X-ray diffraction peak at $2\theta = 26.13^\circ$ was related to the (040) reflection of MoO₃. Another peak observed at 38.1° (JCPDS card no. 50-0739) indicated the growth of the mixed phase of MoO₃ and MoO₂. The presence of this (100) peak revealed that the films formed at low oxygen partial pressure of 8×10^{-5} mbar contained the mixed phase of MoO₂ and MoO₃ because of insufficient oxygen present in the sputter chamber to achieve stoichiometric films. When oxygen partial pressure increased to 4×10^{-4} mbar, the films showed a diffraction peak $2\theta = 12.82^\circ$ related to the (020) along with (040) reflections of the orthorhombic α -phase of MoO₃ (JCPDS card no. 76-1003) in the amorphous background. Thus, the films formed at low oxygen partial pressure were the mixed phase of MoO₂ and α -MoO₃, and with the increase of oxygen partial pressure the films were transformed into orthorhombic α -phase MoO₃ nanocrystals within the amorphous background. The films formed at higher oxygen partial pressure of 8×10^{-4} mbar showed sharp (020) peak and reduction in the intensity of (040) reflection. Nirupama et al. [29] observed the coexistence of the mixed phase of α - and β -MoO₃ along with elemental molybdenum at oxygen partial pressures $< 2 \times 10^{-4}$ mbar and single α -MoO₃ achieved at oxygen partial pressure $\geq 2 \times$

TABLE 2: Chemical composition analysis of MoO₃ films analyzed by EDAX.

| Oxygen partial pressure (mbar) | O (at %) | Mo (at %) | O/Mo |
|--------------------------------|----------|-----------|------|
| 8×10^{-5} | 72.4 | 27.6 | 2.62 |
| 2×10^{-4} | 73.2 | 26.8 | 2.73 |
| 4×10^{-4} | 74.8 | 25.2 | 2.97 |
| 8×10^{-4} | 75.2 | 24.8 | 3.03 |

10^{-4} mbar. Subbarayudu et al. [18] have grown polycrystalline MoO₃ films with the mixed α - and β -phases at oxygen partial pressures 1.9×10^{-1} and 4 mbar and at substrate temperature of 573 K. On increasing the substrate temperature to 923 K, the films were of α -phase MoO₃. Gretener et al. [7] deposited MoO₃ films with 20% of oxygen content at substrate temperature 473 K which consisted of MoO₂ and with 50% of O₂ and at higher substrate temperature of 673 K consisted of MoO₃ associated with Mo₉O₂₆ phases. Thus, the grown phases of sputtered MoO_x films strongly depend on oxygen partial pressure prevailed in the sputter chamber during the deposition of the films.

The crystallite size (L) of the films was calculated from the X-ray diffraction reflections by using the Debye-Scherrer relation [30]

$$L = \frac{0.89\lambda}{\beta \cos \theta}, \quad (1)$$

where λ is the wavelength of the X-ray, β the full width at half maximum of diffraction intensity of the diffraction peak measured in radians, and θ the diffraction angle. The crystallite size of the films increased from 24 to 27 nm with the increase of oxygen partial pressure from 2×10^{-4} to 4×10^{-4} mbar, respectively. On further increase of oxygen partial pressure to 8×10^{-4} mbar, the crystallite size of the films decreased to 15 nm.

3.4. Surface Morphology. Figure 4 shows the scanning electron microscope images (SEM) of MoO₃ films formed at different oxygen partial pressures. MoO₃ films deposited at low oxygen partial pressure of 8×10^{-5} mbar consisted of grains on cracking background uniformly distributed on the surface. This grain growth was due to MoO₂ along with MoO₃ at low oxygen partial pressure. When oxygen partial pressure increased to 2×10^{-4} mbar, tiny spherical grain growth started. After that, the films exhibited needle shaped grains uniformly distributed on the surface of substrate at an oxygen partial pressure of 4×10^{-4} mbar. The size of needle shaped grains was about 800 nm long and 120 nm diameter. It is evident that the oxygen partial pressure strongly influenced the surface morphology of the deposited MoO₃ films. Further due to the increase of oxygen partial pressure to 4×10^{-4} mbar, the films showed uniform large size grains which was attributed to the growth of α -phase MoO₃. This uniformity of the films was due to the formation of stoichiometric α -MoO₃. The films grown at higher oxygen partial pressure of 8×10^{-4} mbar showed the fine grain structure. Ramana and Julien [12] found

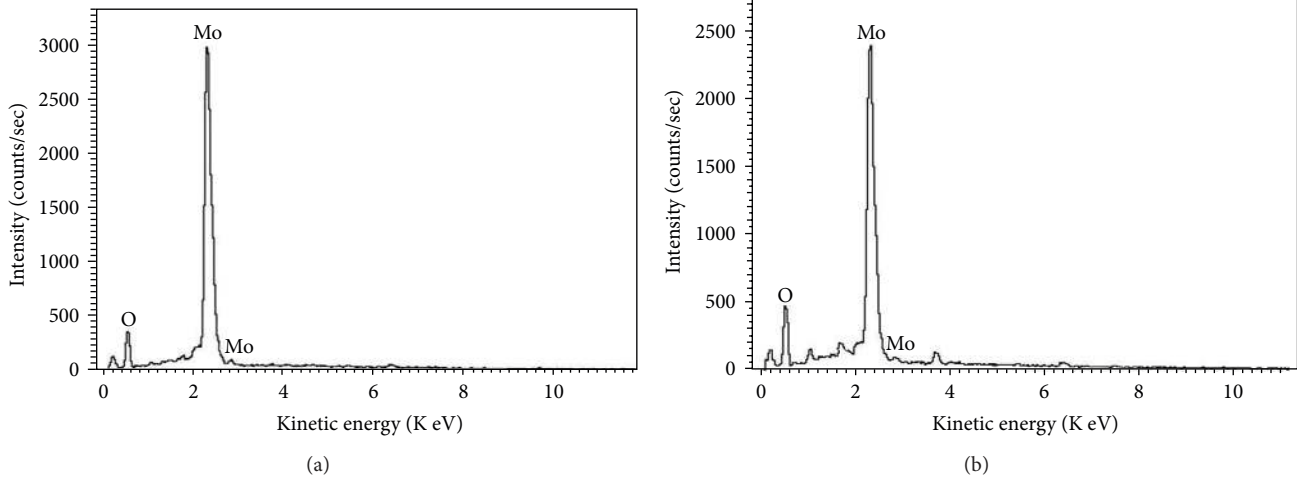


FIGURE 2: EDAX spectra of MoO_3 films deposited on glass substrates at oxygen partial pressures: (a) 8×10^{-5} mbar and (b) 4×10^{-4} mbar.

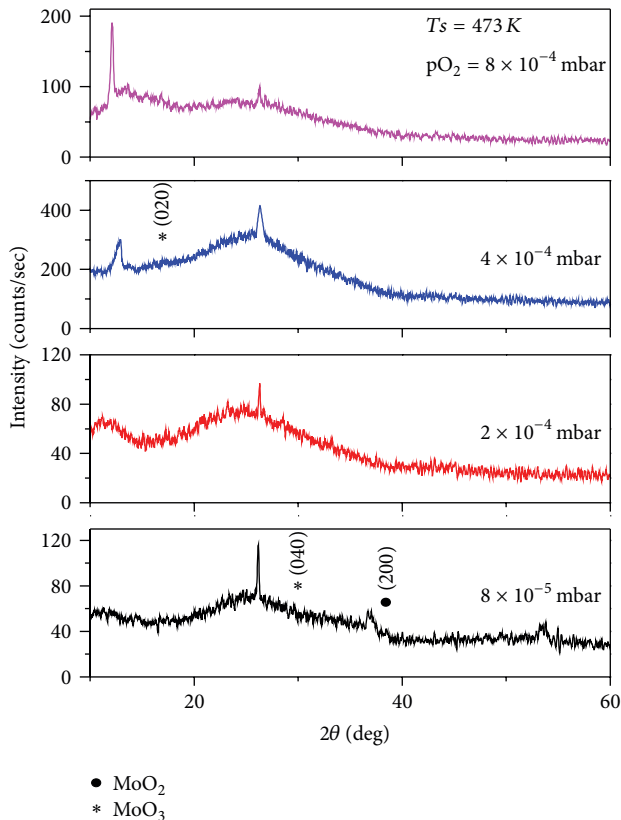


FIGURE 3: XRD profiles of MoO_3 films deposited on glass substrates at different oxygen partial pressures.

that the MoO_3 films grown at 61% content of oxygen pressure exhibited small grains along with the thin long bars or needle shape grains due to incomplete oxidation.

3.5. Fourier Transform Infrared Spectroscopy. Fourier transform infrared transmittance spectra of MoO_3 films formed

on silicon substrates at various oxygen partial pressures were recorded in the wavenumber range $300\text{--}1500\text{ cm}^{-1}$ in order to see the chemical binding configuration in the films. Figure 5 shows the Fourier transform infrared transmittance spectra of MoO_3 films formed at different oxygen partial pressures. The FTIR spectra of the films formed at low oxygen partial pressure of 8×10^{-5} mbar contained broadband in the wavenumbers between 600 and 1000 cm^{-1} . The absorption band located at 566 cm^{-1} was due to transverse optical vibrations of Mo-O-Mo, and broadband centered around 794 cm^{-1} was the characteristic bridging vibration of Mo-O. Ivanova et al. [31] observed the transverse optical vibrations of Mo-O-Mo at 558 cm^{-1} . When the oxygen partial pressure increased from 8×10^{-5} to 2×10^{-4} mbar, the FTIR spectra show the absorption bands at $990, 810, 689,$ and 573 cm^{-1} . The FTIR spectra of the films formed at oxygen partial pressure of 4×10^{-4} mbar showed the absorption bands at about $809, 689,$ and 572 cm^{-1} . The absorption band observed at 811 cm^{-1} was attributed to the bridging vibrations of Mo = O and indicated that the existence of Mo^{6+} oxidation state was related to α -phase MoO_3 . The films deposited at oxygen partial pressure of 8×10^{-4} mbar showed a shift in the absorption bands to $820, 687,$ and 558 cm^{-1} . The intensity of the absorption band seen at 990 cm^{-1} was associated with the Mo = O stretching vibration. Nirupama et al. [29] noticed that the absorption bands at 894 and 1002 cm^{-1} were corresponding to the growth of α -phase MoO_3 in DC magnetron sputtered MoO_3 films formed at oxygen partial pressure of 4×10^{-4} mbar. These studies confirmed that the oxygen partial pressure of 4×10^{-4} mbar is an optimum to produce the films with stoichiometric α -phase MoO_3 .

3.6. Optical Properties. Figure 6 shows the wavelength dependence optical transmittance of the MoO_3 films formed at different oxygen partial pressures. The optical transmittance of the films formed at low oxygen partial pressure of 8×10^{-5} mbar exhibited the average optical transmittance $<50\%$ about the fundamental optical

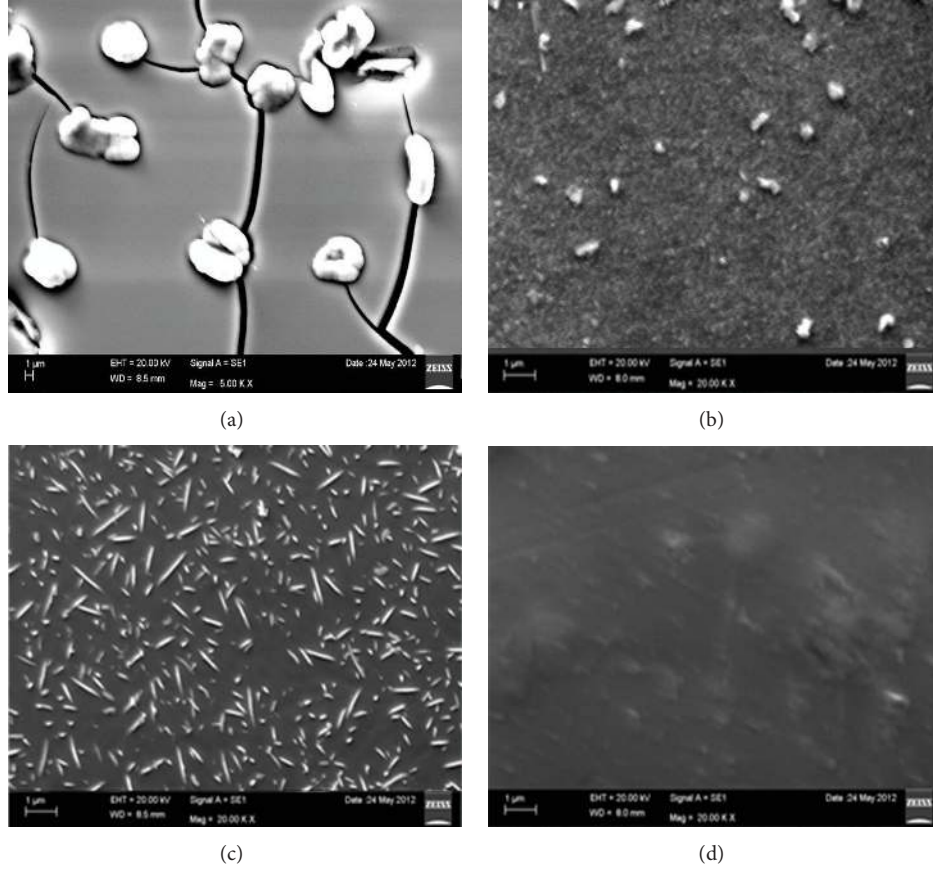


FIGURE 4: SEM images of MoO_3 films formed on glass substrates at oxygen partial pressures: (a) 8×10^{-5} mbar, (b) 2×10^{-4} mbar, (c) 4×10^{-4} mbar, and (d) 8×10^{-4} mbar.

absorption edge. This low optical transmittance at low oxygen partial pressure of 8×10^{-5} mbar was due to the formation of substoichiometric MoO_3 films which characterize the blue color due to the oxygen ion vacancies [29, 32]. The broadband absorption above wavelength 500 nm was mainly due to the presence of MoO_2 atoms which act as scattering centers of light and hence the decrease in the optical transmittance. As the oxygen partial pressure increased to 4×10^{-4} mbar, the optical transmittance increased to about 85% due to oxygen ion vacancies decrease, and the films transformed into nearly stoichiometric α - MoO_3 films. As the oxygen partial pressure increased to 4×10^{-4} and 8×10^{-4} mbar, there was not much variation in the optical transmittance due to the formation of MoO_3 films as conformed by the EDAX data. The fundamental optical absorption edge in the films was observed in the wavelength range 300–400 nm. The optical absorption edge of the films shifted towards lower wavelength side with the increase of oxygen partial pressure.

The optical absorption coefficient (α) of the films was evaluated from the optical transmittance (T) data using the relation:

$$\alpha = -\left(\frac{1}{t}\right) \ln(T), \quad (2)$$

where t is the film thickness. The optical band gap (E_g) of the films was evaluated from the optical absorption coefficient using the Tauc relation [33] assuming that the direct transition takes place in these films

$$(\alpha h\nu) = A(h\nu - E_g)^{1/2}. \quad (3)$$

The plots of $(\alpha h\nu)^2$ versus photon energy ($h\nu$) of the films formed at different oxygen partial pressures are shown in Figure 7. The optical band gap of the films was determined from the plot of $(\alpha h\nu)^2$ versus photon energy ($h\nu$). The extrapolation of the linear portion of plots of $(\alpha h\nu)^2$ versus photon energy to $\alpha = 0$ yields the optical band gap of the films. The optical band gap of the MoO_3 films formed at low oxygen partial pressure of 8×10^{-5} was 3.11 eV, and it increased from 3.23 to 3.28 eV with the increase of oxygen partial pressure from 2×10^{-4} to 4×10^{-4} mbar, respectively. On further increase of oxygen partial pressure to 8×10^{-4} mbar, the optical band gap of the films reached 3.35 eV. The low value of the optical band gap of the films formed at low oxygen partial pressures was due to the formation of substoichiometric films, which is the mixed phase of MoO_2 and MoO_3 . The films formed at oxygen partial pressure of 4×10^{-4} mbar were of nearly stoichiometric MoO_3 films. These MoO_3 films exhibited the optical band gap of

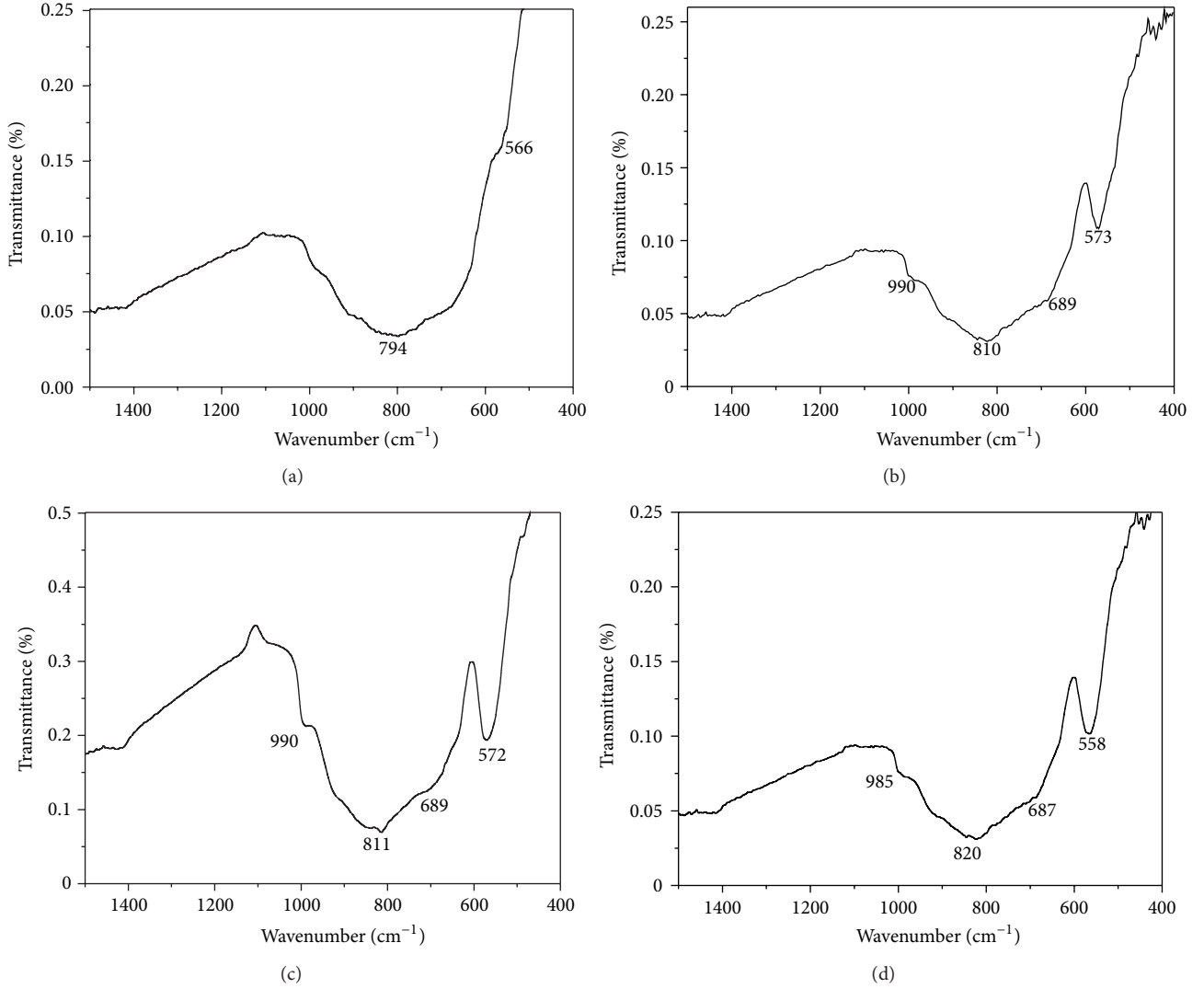


FIGURE 5: FTIR spectra of MoO₃ films formed on silicon substrates at oxygen partial pressures: (a) 8×10^{-5} mbar, (b) 2×10^{-4} mbar (c) 4×10^{-4} mbar, and (d) 8×10^{-4} mbar.

3.28 eV. In the literature, Okumu et al. [34] observed that the optical band gap of the MoO₃ films increased from 3.0 to 3.2 eV with the increase of oxygen partial pressure from 8×10^{-2} to 1.2×10^{-1} mbar in DC magnetron sputtering. Mohamed and Venkataraj [17] noticed that the value of the optical band gap of the films initially increased from 2.64 to 2.69 eV with the increase of oxygen partial pressure from 0.17×10^{-5} to 2.6×10^{-1} mbar and then decreased to 2.67 eV with further increase to 6.4×10^{-1} mbar which was due to the reduction of defect centers and hence improved in stoichiometry. Boudaoud et al. [22] achieved a high optical band gap of 3.35 eV in spray pyrolysis deposited MoO₃ films. It is to be noted that low optical band gap values between 2.60 and 2.70 eV in DC magnetron sputtered [17] and RF magnetron sputtered [35] films were due to the growth of β -MoO₃.

The refractive index (n) of the films was determined from the optical transmittance interference data employing

Swanepoel's envelope method used in the following relation [36],

$$n(\lambda) = \left[N + \left(N^2 - n_0^2 n_1^2 \right)^{1/2} \right]^{1/2}, \quad (4)$$

$$N = 2n_0 n_1 \left[\frac{(T_M - T_m)}{T_M T_m} \right] + \left[\frac{(n_0^2 + n_1^2)}{2} \right],$$

where T_M and T_m are the optical transmittance maxima and minima and n_0 and n_1 are the refractive indices of air and substrate, respectively. Figure 8 shows the wavelength dependence of refractive index of MoO₃ films formed at different oxygen partial pressures. In general, the refractive index of the MoO₃ films decreased with the increase of wavelength. The refractive index of MoO₃ films (at the wavelength of 500 nm) increased from 2.04 to 2.16 with the increase of oxygen partial pressure from 8×10^{-5} to $8 \times$

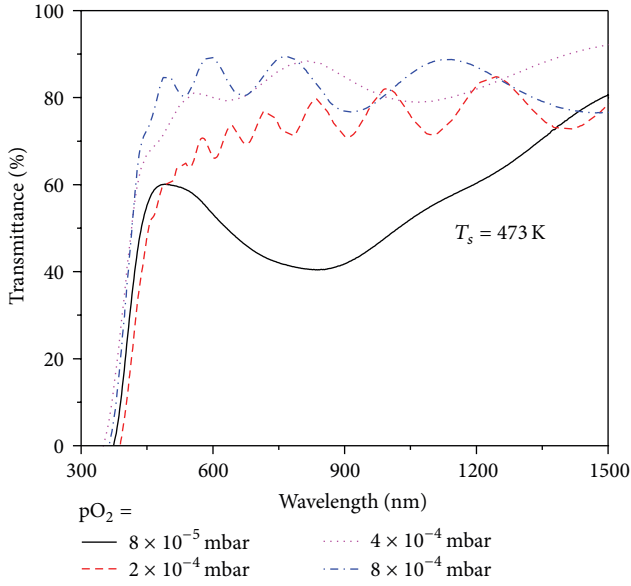


FIGURE 6: Variation of optical transmittance of MoO₃ films deposited on glass substrates at different oxygen partial pressures.

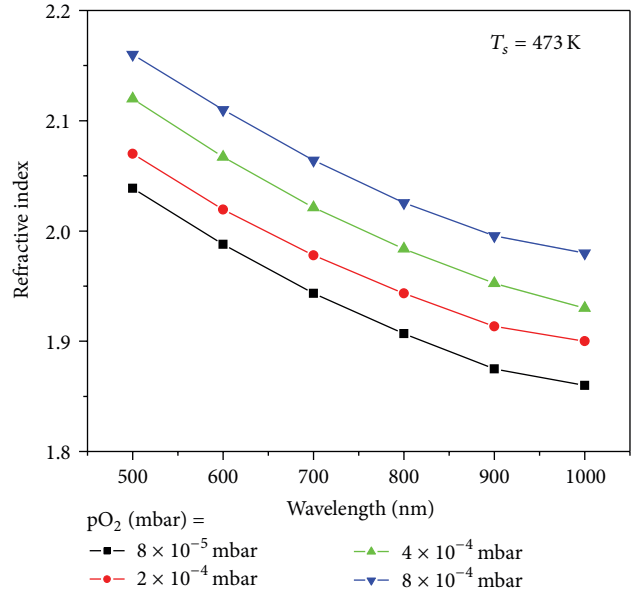


FIGURE 8: Wavelength dependence of refractive index of MoO₃ films deposited on glass substrates at different oxygen partial pressures.

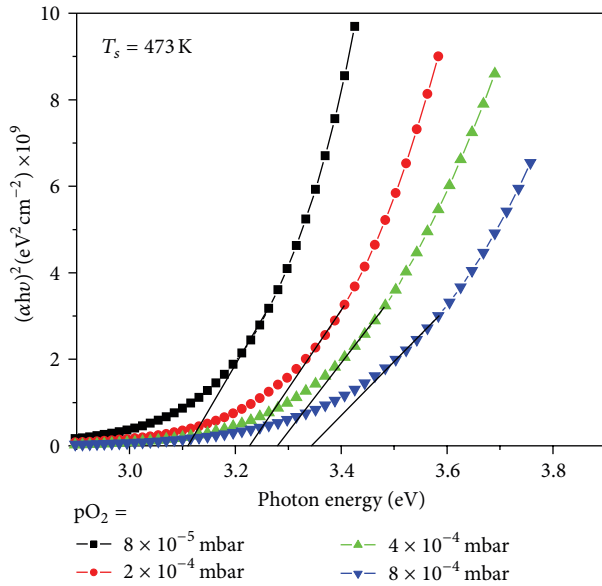


FIGURE 7: Plot of $(\alpha h\nu)^2$ versus photon energy ($h\nu$) of MoO₃ films deposited on glass substrates at different oxygen partial pressures.

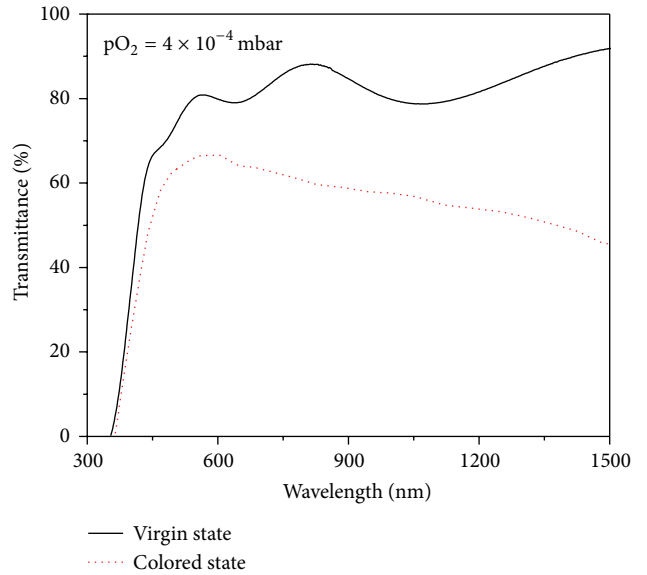


FIGURE 9: Optical transmittance spectra of virgin and colored states of MoO₃ film deposited on glass substrates at oxygen partial pressure of 4×10^{-4} mbar.

10^{-4} mbar, respectively. The low value of refractive index at low oxygen partial pressure of 8×10^{-5} mbar was due to the presence of MoO₂ along with the MoO₃. The increase in the refractive index at higher oxygen partial pressures was due to the formation of single phase α -MoO₃ and increase in the packing density of the films. It is to be noted from the literature that the refractive index value of 1.8 was achieved by Reyes-Betanzo et al. [37] in thermal evaporation films, while Cárdenas et al. [38] reported 1.9 in pulsed laser deposited films. The refractive index of the DC magnetron sputtered

MoO₃ films increased from 2.03 to 2.10 with the increase of substrate temperature from 303 to 573 K [14].

3.7. Electrochromic Properties. In order to study the electrochromic properties, the stoichiometric MoO₃ films were formed on ITO coated glass substrates at oxygen partial pressure of 4×10^{-4} mbar. The electrochromic properties of the films were investigated by three-electrode cell, with platinum as a counter electrode, Ag/AgCl as a reference electrode and the indium tin oxide coated MoO₃ films as a

working electrode using an electrochromic cell model (HI 608). The colored and virgin states of the films were measured by UV-Vis-NIR spectrophotometer. In the electrochromism, the coloration is due to the reduction of Mo^{6+} to Mo^{5+} state by insertion of Li^+ ions into the MoO_3 films. In the reverse scan, the virgin state can be achieved by the intercalation charge removed from the films, resulting in the virgin state due to the oxidation of Mo^{5+} to Mo^{6+} state. Figure 9 shows the colored and virgin states of the MoO_3 film formed at oxygen partial pressure of 4×10^{-4} mbar. The optical modulation (ΔT) of the films at 550 nm is about 13%. This optical modulation is mainly dependent on the quantity of Li^+ insertion into the MoO_3 films. The color efficiency (η) at a particular wavelength correlated to the optical contrast; that is, the change in optical density with charges intercalated per unit electrode area and can be expressed with relation [39]:

$$\eta = \frac{\log(T_b/T_c)}{(Q/A)} = \frac{(\Delta OD)}{(Q/A)}, \quad (5)$$

where T_b is the bleaching transmittance, T_c the colored transmittance, Q the charge inserted into the films, and A the area of the films. The color efficiency of the MoO_3 film formed at oxygen partial pressure of 4×10^{-4} mbar was $20 \text{ cm}^2/\text{C}$. Lin et al. [40] reported that the coloration efficiency achieved a value of $25.1 \text{ cm}^2/\text{C}$ in the MoO_3 films formed at room temperature subsequently annealed in air at 573 K.

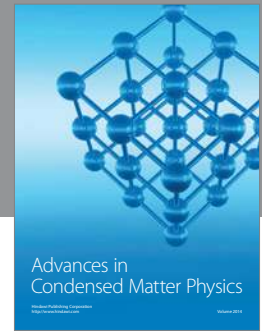
4. Conclusions

Thin films of molybdenum oxide were deposited on glass and silicon substrates held at temperature of 473 K by RF magnetron sputtering method. The films were formed by sputtering of metallic molybdenum target at various oxygen partial pressures in the range 8×10^{-5} – 8×10^{-4} mbar. The energy dispersive X-ray analysis revealed that the films formed at oxygen partial pressure of 4×10^{-4} mbar were nearly stoichiometric. X-ray diffraction studies indicated that the films formed at oxygen partial pressure $< 4 \times 10^{-4}$ mbar were the mixed phase of MoO_2 and MoO_3 , while those deposited at 4×10^{-4} mbar were single phase α - MoO_3 with crystallite size of 28 nm. Scanning electron microscopic studies revealed that the films grown at 4×10^{-4} mbar exhibited that the grown grains are of needle shape grains with size of about 800 nm. The Fourier transform infrared transmittance spectra indicated the presence of characteristic vibrations of MoO_3 in the films formed at oxygen partial pressure $\geq 4 \times 10^{-4}$ mbar. The optical band gap of the films increased from 3.11 to 3.28 eV, and the refractive index of the films increased from 2.04 to 2.16 with increase of oxygen partial pressure from 8×10^{-5} to 8×10^{-4} mbar, respectively. The electrochromic performance of the stoichiometric MoO_3 films formed on ITO coated glass substrates was studied and achieved the optical modulation of about 13% with color efficiency of about $20 \text{ cm}^2/\text{C}$.

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