

RF Sputtered MoO₃ Thin Film on Si (100) for Gas Sensing Applications

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ABSTRACT

Molybdenum Trioxide (MoO₃) films are grown on Si(100) substrates by reactive RF magnetron sputtering in plasma containing a mixture of Argon and Oxygen, using a pure Molybdenum target. In this paper, we report the deposition of (MoO₃) films on Si(100) substrates under varying gas flow (O₂ + Ar gas) (20 sccm to 30 sccm with the duration of deposition ~ 1hr) by RF reactive magnetron sputtering at room temperature. To get crystalline MoO₃ films annealing in O₂ environment at 500 °C for 4 h is done. Phase formation and orientation of the film is characterised by Glancing incidence X-ray diffraction (GIXRD). The identification of the orthorhombic MoO₃ phase is investigated by XRD and Raman spectroscopy. Raman lines at 819 cm⁻¹ and 995 cm⁻¹ are due to the (A_{1g}, B_{1g}) symmetric stretching (Mo–O–Mo) bond and asymmetric stretching band (Mo=O) respectively. Surface morphology and cross-sectional image of the deposited thin films were investigated by FE-SEM image. UV-Visible reflectance and cross-sectional FE-SEM image confirm the thickness of the MoO₃ films with oxygen-rich and oxygen deficient phase formation occur. Reverse leakage current density of 20 sccm 1hr sample is low (1×10⁻⁶ mA/cm²) as compared to 30 sccm 1hr sample (1×10⁻³ mA/cm²). The higher leakage is due to crack formation during the ex-situ annealing of MoO₃ films. This MoO₃ films can be used in Gas sensing and switching devices.

Keywords: MoO₃; Sputtering; XRD; Reflectance; FESEM; Leakage current; Gas sensing

1. INTRODUCTION

Among the transition metal oxides, Molybdenum oxide (MoO₃) is one of the most potential materials for the variety of technological applications such as large-scale electrochromic devices, coatings for optical switching and memory devices¹. MoO₃ is intensively used for sensing various gases like NO₂, NH₃, Ethanol, Methanol, H₂ and H₂S gases² and various volatile organic compounds (VOC)³. The thin films of MoO₃ can also be used in solar cells, micro-batteries, supercapacitors etc⁴⁻⁵.

MoO₃ is a wide bandgap semiconductor of bandgap approximately 3.3 eV. Usually, MoO₃ gets crystallised in two different phases⁶. Orthorhombic, α -MoO₃, which is the most stable phase and the other one, is a metastable monoclinic β -MoO₃ crystal phase. MoO₃ has good chemical stability and as-deposited films normally have amorphous structure which on annealing at 300 °C crystallizes to the monoclinic phase and above 400 °C to 600 °C, it crystallizes orthorhombic α -MoO₃ phase.

MoO₃ thin films can be prepared by different growth techniques like sputtering⁷⁻⁸, molecular beam epitaxy⁹, pulsed laser deposition¹⁰⁻¹¹, atomic layer deposition¹², chemical vapor deposition¹³, spray pyrolysis¹⁴ and sol-gel¹⁵. Among them, sputtering is more preferred for the deposition of high quality

of MoO₃ films due to ease in operation and effective in cost. The commonly used substrate for MoO₃ thin films are Glass, Quartz, Si and GaAs.

Sarkar⁶, *et al.* have studied the interface study of MoO₃ thin films on GaAs substrate for Solar cell applications. Arfaoui¹⁶, *et al.* have studied the structural and morphological properties MoO₃ and WO₃ thin films prepared by thermal evaporation for gas sensing and photocatalytic applications. Touihri¹⁷, *et al.* studied the annealing effect of MoO₃ films and found the multiphase of MoO₃ films.

Researchers have identified the MoO₃ multiphase usually by XRD and Raman analysis¹⁸⁻¹⁹ but proper analysis of ex-situ annealed RF sputtered MoO₃ films by structural, optical and electrical analysis to identify the multiphase with oxygen deficiency or oxygen-rich in MoO₃ films are less studied. Therefore in this study it is planned to grow the MoO₃ thin films by RF sputtering by varying the oxygen pressure. After growing the film its annealing effect under oxygen environment has studied using structural, optical and electrical characterisations.

This paper discusses the deposition of MoO₃ thin films on Si (100) substrate by RF sputtering technique (with Mo target) at different oxygen partial pressure and thereafter ex-situ annealed at 500 °C. The phase and microstructure of the annealed samples are reported. The optical and electrical properties of the MoO₃ films are also reported.

2. EXPERIMENTAL TECHNIQUES

The two MoO₃ films were deposited using RF reactive magnetron sputtering on Si (100) substrate by varying the oxygen + Ar flow (10 sccm+10 sccm) to (15 sccm+ 15sccm)), together with same deposition time (~1 h) and are named as sample S1 and S2 respectively. After deposition ex-situ annealing was done in O₂ environment at 500 °C for 240 min. The structural evolution together with microstructural and optical properties of MoO₃ thin films was studied using X-ray Diffraction (XRD), Raman spectroscopy, UV-visible reflectance and Field Emission Scanning Electron Microscopy (FE-SEM) measurements. X-ray diffractogram of the MoO₃ films were measured in thin-film geometry using glancing incidence x-ray diffraction (GIXRD) technique. In this geometry x-ray incidence angle (α) is kept constant around $\sim 2^\circ$ from the sample surface. In the diffracted arm. A diffracted signal was detected by the detector by scanning with 2theta angle from 10° to 75° to record the diffractogram. In XRD using Cu K α radiation PANalytical X-Pert Pro MRD HRXRD system is used for phase and orientation parameter analysis. The surface morphology of the films and their cross-section were examined by FE-SEM (Carl Zeiss Model: SUPRA-50). Raman spectroscopy experiments were performed in the backscattering geometry at room temperature by the confocal Micro-Raman spectrometer system (Model Horiba Jobin Yuvan LABRAM HR Evolution) using frequency-doubled NdYAG laser at 532.15 nm excitation with cooled UV enhanced CCD detector. UV-visible reflectance measurements were done by Agilent make UV-visible spectrophotometer from 200 nm to 850 nm. For electrical I-V characteristics have been obtained from -2 V to +10 V using Keithley 2611B source meter.

3. RESULTS AND DISCUSSION

The XRD patterns of synthesised films in grazing incidence geometry (GIXRD) are shown in Fig. 1. The MoO₃ films is found to be polycrystalline in nature having orientations along (020), (110), (040), (021), (130), (111), (041), (150), (200), (002), (112), (109), (202) planes corresponding to the 2 θ angles of 12.68°, 23.33°, 25.65°, 27.32°, 29.45°, 33.81°, 35.65°, 38.8°, 45.78°, 49.11°, 55.25°, 64.91° and 69.73° in XRD diffractogram. In the XRD pattern of samples S1 and

S2 relatively intense (110) and (021) peak is observed as compared to other XRD peaks respectively. The peak position of XRD pattern is very well matched with the α -orthorhombic phase of MoO₃. Some unidentified extra XRD peaks were also observed, marked star (*) in the Fig. 1. These peaks may be oxygen-deficient molybdenum oxide XRD peaks. As shown in figure sample S1 is more oxygen-deficient as compared to sample S2. The average crystallite size determined by Scherer relation is 16 nm and 20 nm for sample S1 and S2 respectively. The degree of crystallinity for sample S1 (~65%) is relatively low as compared to sample S2 (~70%). The film's orientation changes along (021) to (110) directions as oxygen and Ar flow increases (20 sccm to 30 sccm). Texture Coefficient (TC) of the films along (021) for sample S1 and (110) for sample S2 are determined as equation^{20, 21} written below

$$TC(\gamma) = \frac{I_{hkl}}{I_{ohkl}} \quad (1)$$

$$= \frac{1}{N} \sum_{i=1}^N \frac{I_{hkl}}{I_{ohkl}}$$

In the above equation the measured intensity from XRD pattern is I_{hkl} and I_o is intensity taken from JCPDS data (file No-05-508). N belongs to the number of diffraction peaks. If the texture coefficient value is less than unity for any crystallographic plane, the film orientation is not preferred along that direction. The calculated texture coefficient of sample S1 along (021) plane is 1.7; while sample S2 along (110) plane is 1.2. Texture coefficients of the MoO₃ sample confirm the crystallites are partially oriented along (021) and (110) for sample S1 and S2 respectively.

XRD peak intensity of sample S1 is low as compared to sample S2 and the degree of crystallinity of S2 is higher as compared to S1. It seems after annealing sample 1(20 sccm, 1h) is still oxygen-deficient as compared to S2.

Figure 2 shows the image of the surface and cross-sectional morphology of sample S1 and sample S2 using FESEM. Figures 2(a) and 2(b) shows the FESEM image of annealed MoO₃ films image of 1 hour grown (Ar +O₂ flow ~ 20 sccm) sample S1. The average grain size of the sample S1 shows the variation from 200 nm to 300 nm. Some large and small grains are distributed in MoO₃ films, apart from grain distribution some small cracks were also observed in the films. Large grains may be MoO₃ flakes that are formed during the annealing in oxygen environment. Figure 2(c) shows the surface morphology of sample 2. The grain size is significantly increased in sample 2 (average grain size is around 300-400nm) which is quite large as compared to sample S1. In the sample S2, small cracks become large and the size of the crack ~20 nm. Figures 2(c) and 2(d) show the cross-sectional image of sample S1 and S2 respectively. The thickness of MoO₃ films measured from the cross-sectional FESEM images is 1.54 μ m and 1.6 μ m for sample S1 and S2 respectively. Interestingly in Fig. 2(b) two interfaces are observed. One from the top surface of the film up to ~0.45 μ m with large grain sized (~200-300 nm), another from this interface to substrate interface (~1.1 μ m) with grain size below 150 nm. The cross-sectional image of sample-2 shows the uniform grain size ~300 nm. The continuous layer

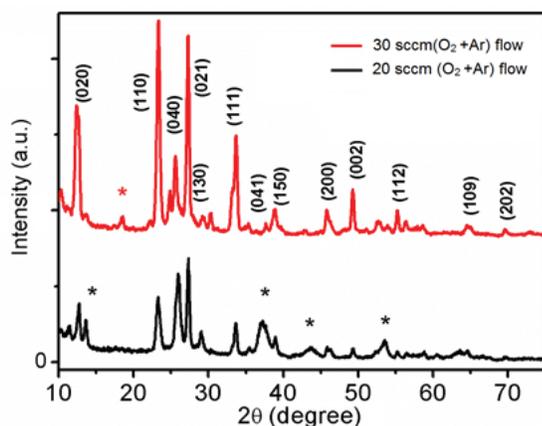


Figure 1. XRD pattern of Annealed MoO₃ films with different O₂ flow.

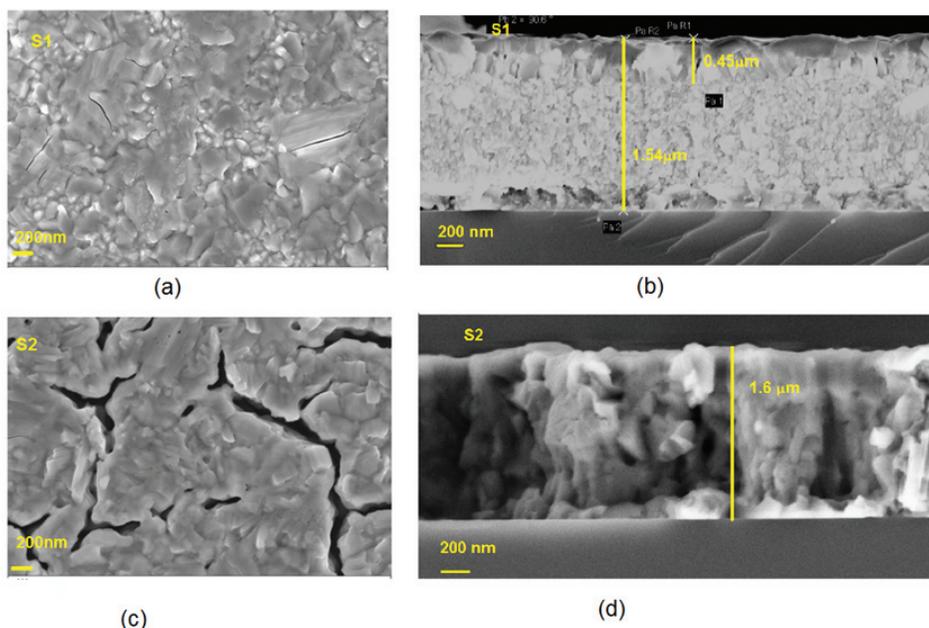


Figure 2. FESEM images of MoO₃ films (a) and (b) planer and cross-sectional for sample S1(20sccm-1h), (c) and (d) planer and cross-sectional for sample S2(30sccm-1h).

from the surface of the film to the films-substrate interface is observed with the thickness ($\sim 1.6 \mu\text{m}$).

In the crystalline orthorhombic $\alpha\text{-MoO}_3$ structures, layers formed by distorted MoO₆ octahedra which are weakly bound by Vander Wall (vdW) forces and all three lattice constants (a, b and c) are different^{22,23}. In the unit cell of $\alpha\text{-MoO}_3$ there exist 16 atoms, four represent molybdenum and others twelve are oxygen. There exist a total 45 phonon modes. The irreducible representation of orthorhombic MoO₃ crystal with the space group (Pbmn) is given by

$$\Gamma = 8A_g + 8B_{1g} + 4B_{2g} + 4B_{3g} + 4A_u + 3B_{1u} + 7B_{2u} + 7B_{3g}$$

where Raman active modes are Ag, B_{1g}, B_{2g}, and B_{3g}. Therefore total 24 phonon modes are Raman active and others (17 Modes = 3B_{1u} + 7B_{2u} + 7B_{3g}) are IR active. Apart from these normal phonon modes 4A_u are silent. Figure 3 shows the Raman spectra of MoO₃ thin films excited from the green laser. Figure 3 shows the prominent peaks at 118, 152, 199,

218, 247, 280, 338, 367, 382, 472, 660, 819, and 995 cm⁻¹, which confirms $\alpha\text{-MoO}_3$ orthorhombic phase³. These Raman active peaks can be indexed with modes: 118 (B_{2g}), 129 (B_{3g}), 160 (Ag, B_{1g}), 199 (B_{2g}), 218 (Ag), 247 (B_{3g}), 285 (B_{2g}, B_{3g}), 338 (B_{1g}, Ag), 367 (Ag), 382 (B_{1g}), 472 (Ag, B_{1g}), 667 (B_{2g}, B_{3g}), 820 (Ag₁, B_{1g}) and 995 (Ag₁, B_{1g}) cm⁻¹. The prominent Raman peaks at 819 cm⁻¹ and 995 cm⁻¹ are due to the symmetric stretching (Mo=O) band and asymmetric stretching band (Mo=O) respectively. The other Raman lines 667 cm⁻¹ is the asymmetric stretching band of O-Mo-O, 338 cm⁻¹ is a bending mode of O-Mo-O and 285 cm⁻¹ is wagging mode of O=Mo=O. The relative Raman intensity and peak position are almost matching with previously reported MoO₃ Raman modes²². Raman peaks are intense and prominent for sample S2 (30 sccm 1hr) as compared to S1. Due to the high degree of crystallinity determined by XRD pattern, Raman intensity of sample S2 (30 sccm 1hr) is more intense as compared to sample S1 (20 sccm 1h).

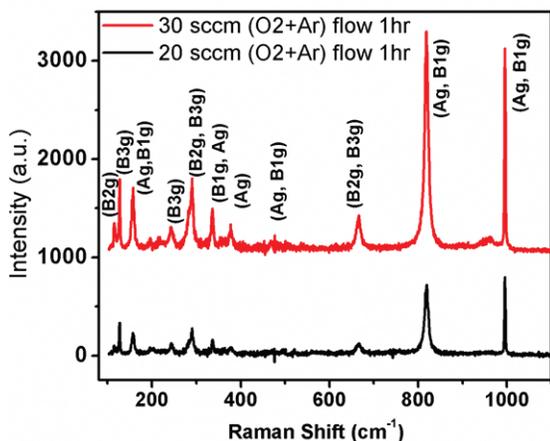


Figure 3. Raman spectra of annealed MoO₃ films with different O₂ flow excited by green 532nm LASER.

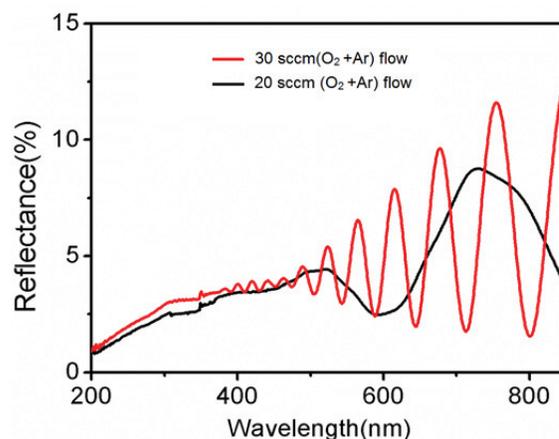


Figure 4. UV-Visible reflectance plot of MoO₃ films.

The UV-visible Reflectance measurement of MoO₃ films grown on Si (100) substrates is done at room temperature. Due to the low bandgap of Si substrates as compare to MoO₃, the transmittance study for samples 1 and 2 is not possible in the UV-visible range, Therefore reflectance measurements from 200-850 nm are performed. Fig. 1 shows the Reflectance of the two samples S1 (20 sccm 1 hr) and S2 (30 sccm 1 hr). The percentage of reflectance of sample 1 and 2 is around 5 % to 12 %. Less reflectance per cent may be due to scattering from large number of grains and grain boundaries present in the MoO₃ samples.

Sample 1 reflectance is slightly less as compare to sample S2. It might be due to high grain boundary density as analysed by XRD and SEM sections. In the reflectance, interference fringes are observed and the thickness of MoO₃ films is determined from fringe separation in the wavenumber scale. The determined thickness of MoO₃ films was 0.5 μm and 1.6 μm for S1 and S2 respectively. The optical absorption of the films is in the range of ~3.25 eV. The optical absorption of MoO₃ films seems to be very close but slightly less than actual bandgap (~3.3eV)²² of the materials. The less value is due to a slight stoichiometric variation of Mo and O in MoO₃ formation. The thickness of the film in sample S2 determined by the FESEM image is very well matched with thickness determined by UV-visible reflectance. Whereas in sample S1 the thickness determined by reflectance fringes match only from the surface to the first interface of the sample.

The two-layer in sample 1 observed by FESEM images is also seen as the two different phases from XRD. By SEM image upper layer is the orthorhombic α-MoO₃ and other layers may be the oxygen-deficient molybdenum oxide phase. While in sample-2 two-phase of α-MoO₃ and oxygen-deficient MoO₃ phase is present in the whole film therefore large cracks are observed. Since the binding energy of Mo and O in these two phases is different therefore observed cracks from SEM images are large. It is evident from XRD, Raman and SEM images after annealing under Oxygen environment low oxygen and argon flow (20 sccm) films is still oxygen-deficient (sample S1) while high oxygen and argon flow ((30 sccm) film (sample S2) shows the relatively less oxygen deficient.

Further, electrical characterizations of the MoO₃ films are studied by utilizing Au/MoO₃/Si/Au structure. Figure 5(a) shows the electrical setup for I-V measurement for the Au/MoO₃/Si/Au structure. For electrical contacts, gold (Au) was deposited on the top of the MoO₃ film 60 nm thick Au electrodes (diameter~ 0.6 mm) by shadow masking and the backside at room temperature by thermal evaporation.

Figure 5(b) shows the J-E characteristics for sample 1 and sample 2. The films are showing Schottky diode characteristics. The reverse leakage current density of sample S2 is found to be ~1x10⁻⁴ mA/cm² (high) as compare to sample S1 (~1x10⁻⁶ mA/cm²). The high leakage current density of sample 2 is due to large cracks observed in the film and also the excess of oxygen ion vacancies in the MoO₃ films. While low leakage current

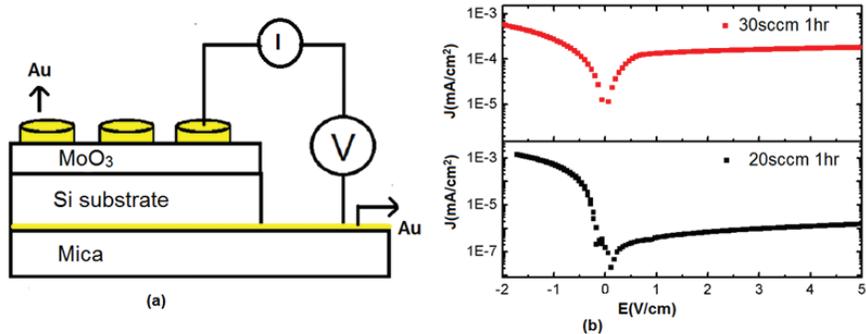


Figure 5. (a) Electrical characterisation setup and (b) J-E characteristics of MoO₃ thin films.

density in sample S1 is due to large and small grains of the films. These MoO₃ thin films can be utilised for gas sensing applications²⁴.

4. CONCLUSIONS

The MoO₃ thin films have been successfully deposited on Si substrate by reactive RF magnetron sputtering by varying Ar and O₂ flow (20 sccm and 30 sccm) with 1 h deposition duration with ex-situ annealing in oxygen environment at 500 °C for 4 h. The deposited thin films were found to be a polycrystalline orthorhombic α-MoO₃ phase with preferred orientations along <021> and <110> direction with deficient oxygen MoO₃ films. MoO₃ films grain size varies from 200-400 nm for determined by FESEM. Layer thickness determined by reflectance is very well matched with the cross-sectional FESEM images. The reverse leakage current density of 30 sccm 1-hr sample is large (~1x10⁻⁴ mA/cm²) as compare to 20 sccm 1-hr sample (~1x10⁻⁶ mA/cm²). These thin MoO₃ films may show a potential candidate for resistive random access memory (ReRAM) and gas sensing applications.

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CONTRIBUTORS

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In the current study, He contributed in the measurement and analysis of planner and cross-sectional FESEM image of MoO₃ thin films on Si(100) substrates.

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In the current study, he has reviewed the work, validated the results, continuously provided the guidance and given many valuable inputs.