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Research Article

Characterization of Silver Oxide Films Formed by Reactive RF Sputtering at Different Substrate Temperatures

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Silver oxide (A_2O) films were deposited on glass and silicon substrates held at temperatures in the range 303–473 K by reactive RF magnetron sputtering of silver target. The films formed at room temperature were single phase Ag_2O with polycrystalline in nature, while those deposited at 373 K were improved in the crystallinity. The films deposited at 423 K were mixed phase of Ag_2O and Ag. Atomic force micrographs of the films formed at room temperature were of spherical shape grains with size of 85 nm, whereas those deposited at 473 K were with enhanced grain size of 215 nm with pyramidal shape. Electrical resistivity of the single phase films formed at room temperature was 5.2×10^{-3} Ωcm and that of mixed phase was 4.2×10^{-4} Ωcm. Optical band gap of single phase films increased from 2.05 to 2.13 eV with the increase of substrate temperature from 303 to 373 K, while in mixed phase films it was 1.92 eV.

1. Introduction

Silver-oxygen system (Ag-O) was extensively attracted by researchers due to its novel applications in high density optical storage devices, gas sensors, photovoltaic cells, photo diodes, and antibacterial coatings [1–6]. This system exists in different defined compounds, namely, Ag_2O, AgO, Ag_3O_4, Ag_4O_3, Ag_2O_3, and Ag_4O_4. Among these oxides, Ag_2O is the most thermodynamically stable. The compound Ag_2O possesses a simple cubic structure at room temperature [7]. Ag_2O in thin film form is a p-type semiconductor with a band gap ranging from 1.2 to 3.4 eV due to the deviation in the stoichiometry, structure and crystallinity, phases, and physical properties arising from the employed deposition technique [8]. Thermal decomposition of silver oxide into oxygen and silver is the unique characteristics which led to the promising technological applications. Kim et al. [9] reported that silver oxide films also act as a mask layer in magneto-optical disk to enhance the magneto-optical signal. However, the high threshold of thermal decomposition temperature >673 K for silver oxide films has been a bottleneck of application in optical and magneto-optical storage [10, 11]. The Ag_2O films grown with (111) orientation by rapid thermal annealing process at temperature of 473 K find application as readout layer in a magneto-optical disk. Peyser et al. [12] achieved strong photoactivated emission of nanoscale Ag_2O for excitation with a wavelength <520 nm find application in blue optical lasers. Nanoparticles of Ag_2O embedded in ZnO inhibit the degradation in the performance of photodetector when annealed in oxygen ambient at temperature of 473 K [3]. Büchel et al. [13] effectively employed silver oxide as a substrate for the surface enhanced Raman spectroscopy for molecular level detection. Her et al. [14] incorporated silver oxide films into super resolution near field structures in optical memories.

Thin films of silver oxide can be prepared by various techniques such as thermal oxidation of silver films [5], thermal evaporation [15, 16], electron beam evaporation [17], pulsed laser deposition [18], chemical vapour deposition [19], electrodeposition [20], DC sputtering [8, 10, 21–25], and RF sputtering [7, 11, 26, 27]. Among these deposition techniques, RF magnetron sputtering is one of the promising techniques for preparation of Ag_2O films because of the advantages of high deposition rates, uniformity on large area

Table 1: Sputter deposition parameters fixed for the growth of silver oxide films.

Deposition method	RF magnetron sputtering
Sputter target	Silver (50 mm dia. and 3 mm thick)
Target to substrate distance	65 mm
Base pressure	5×10^{-4} Pa
Oxygen partial pressure	2×10^{-2} Pa
Sputter pressure	4 Pa
Substrate temperature (T_s)	303–473 K
Sputter power	65 W

substrates, precise control on the chemical composition and physical properties. In RF magnetron sputtering, the physical properties of the deposited thin films critically depend on the sputter parameters such as oxygen partial pressure, substrate temperature, and substrate bias voltage, sputtering pressure and sputter power. The influence of oxygen partial pressure on the structural, electrical, and optical properties of silver oxide films formed by RF magnetron sputtering was earlier reported [28]. In the present investigation, nanocrystalline Ag_2O films were deposited on glass and silicon substrate by RF magnetron sputtering at different substrate temperatures. The effect of substrate temperature on the crystallographic structure and surface morphology, core level binding energies, and electrical and optical properties was systematically studied and the results were reported.

2. Experimentation

Thin films of silver oxide were deposited on glass and silicon substrates using RF magnetron sputtering method. Metallicsilver (99.9% pure) of 50 mm diameter and 3 mm thickness was used as sputter target. The base pressure of 5×10^{-4} Pa was achieved in the sputter chamber using diffusion pump and rotary pump combination. Argon was used as the sputter gas and oxygen as reactive gas. The required quantities of reactive gas of oxygen and sputter gas of argon were admitted into the sputter chamber through fine controlled needle valves. The distance between the target and substrate maintained was 65 mm. The sputter target was powered with Advanced Energy RF power generator. The power fed to the sputter target was 65 W. Films were deposited at oxygen partial pressure of 2×10^{-2} Pa and sputter pressure of 4 Pa and at different substrate temperatures in the range 303–473 K. The sputter deposition parameters maintained for preparation of silver oxide films are given in Table 1.

The deposited silver oxide films were characterized by studying their structural, morphological, electrical, and optical properties. The thickness of the deposited films determined with Veeco Dektak (model 150) depth profilometer was in the range 95–125 nm. The crystallographic structure of the films was determined with X-ray diffraction (XRD) taken on a Bruker D8 Advanced diffractometer using monochromatic Cu $K_{\alpha 1}$ radiation with wavelength of 0.15406 nm. The core level binding energies of the films was analyzed with Philips X-ray photoelectron spectrometer (Model PHI 300).

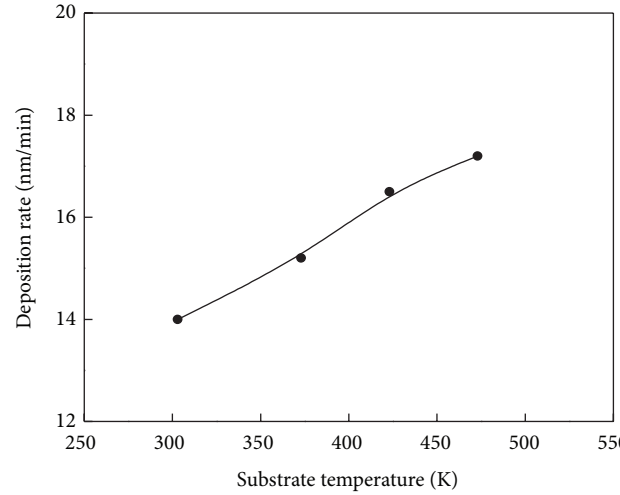

Figure 1: Variation in the deposition rate of Ag_2O films with substrate temperature.

The surface morphology of the films was analyzed with atomic force microscope (Model SPA 400). The electrical resistivity of the films was measured at room temperature using four point probe (Jandel multiposition probe) technique. The optical transmittance of the films was recorded with Perkin-Elmer double beam spectrophotometer in the wavelength range 500–2500 nm.

3. Results and Discussion

The deposition rate of the films was calculated from the thickness and the duration of deposition. The deposition rate of the films was highly influenced by the substrate temperature. The variation of deposition rate with the substrate temperature of the films was shown in Figure 1. The deposition rate of the films increased from 14 to 17.5 nm/min with the increase of substrate temperature from 303 to 473 K, respectively. It is to be noted that the formation of oxide phase during reactive sputtering occurs very nearto the substrate surface and the rate of reaction is increased with substrate temperature hence of higher deposition rate [26].

Figure 2 shows the X-ray diffraction profiles of the silver oxide films deposited on glass substrates held at temperatures in the range 303–473 K. The films formed at room temperature (303 K) showed a strong X-ray diffraction peak at $2\theta = 32.7^\circ$ and two weak peaks at 54.7° and 68.7° (shown in inset of Figure 2). These diffraction (111), (220), and (222) reflections are related to the cubic structure of Ag_2O (ICCD Card number: 00-41-1104). It indicates that the grown films were of polycrystalline in nature. The films formed at substrate temperature of 373 K showed that the enhancement in the intensity of (111) reflection indicated the increase in the crystallinity of the Ag_2O films. As the substrate temperature increased to 423 K the intensity of the (111) reflection of Ag_2O decreased with the presence of additional three diffraction peaks at 38.0°, 44.1°, and 54.8°. The diffraction peak situated at 38.0° is related to the (200) reflection of Ag_2O/(111) of Ag, and the peaks at 44.1° and 54.8° correspond to the (200) and

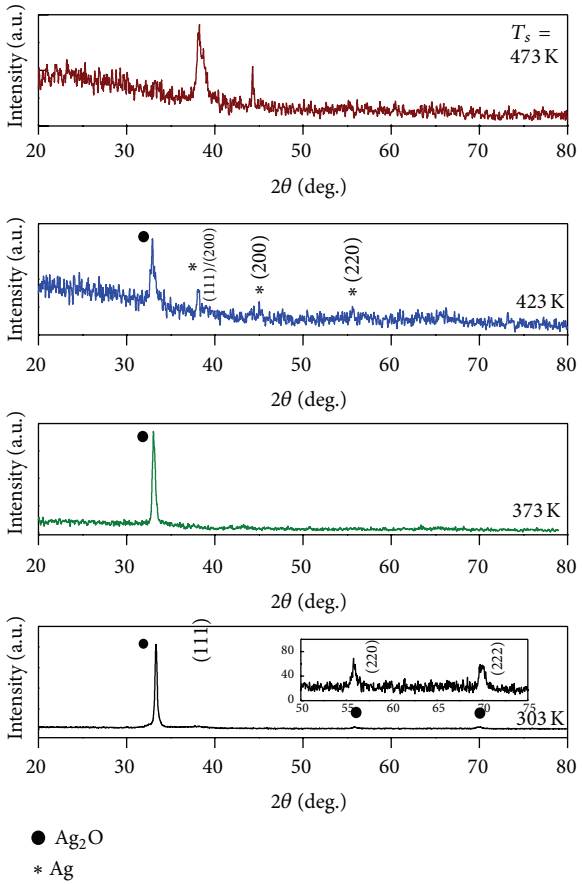

Figure 2: XRD profiles of Ag_2O films formed at different substrate temperatures.

(220) reflections of metallic silver (ICCD Card number: 00-004-0783). It revealed that the films formed at 423 K were of mixed phase of Ag_2O and Ag. The films are deposited at higher substrate temperature of 473 K, the (111) reflection of Ag_2O disappeared, and increase in the intensity of the peak at 38.0° increased. Based on the intensities of the diffraction peak, at higher substrate temperature of 473 K, the films were transformed from Ag_2O phase to metallic silver. The decomposition of Ag_2O to silver was also noticed by Gao et al. [21] in DC magnetron sputtered Ag_2O films. It was also reported that the Ag_2O decomposed into Ag after the heat treatment in the temperature range 473–673 K [29]. Pierson and Rousselot [7] noticed that the single phase Ag_2O films formed by RF sputtering with oxygen flow rate of 9 sccm were decomposed into silver by heat treatment at 473 K, while the mixed phase (Ag_2O + Ag) films formed with 6 sccm of oxygen flow rate were transformed into silver at temperature of 673 K.

The crystallite size (L) of the films was evaluated from the full width at half maximum intensity of X-ray diffraction peaks using Debye- Scherrer's relation

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

where k is a constant with a value of 0.89 for copper X-ray radiation and β the full width at half maximum intensity

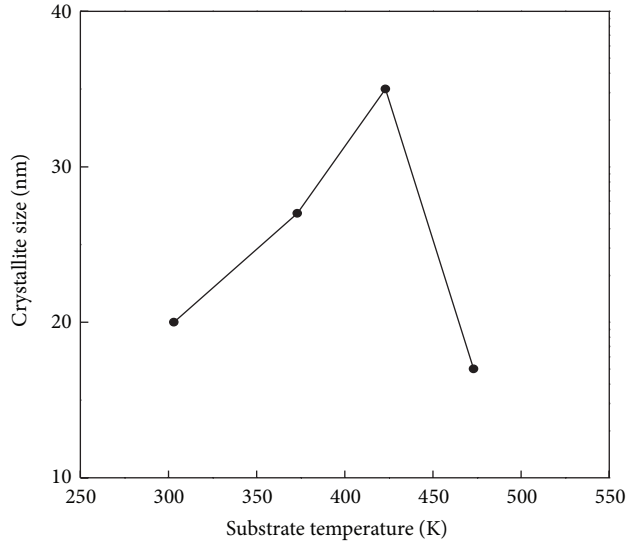

Figure 3: Dependence of crystallite size of Ag_2O films on the substrate temperature.

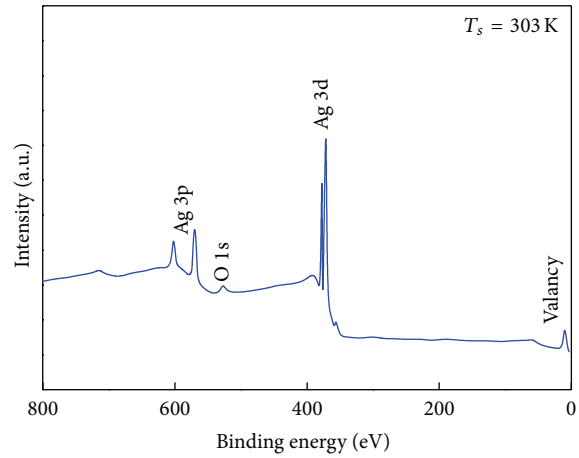

Figure 4: XPS survey scan of silver oxide films formed at substrate temperature of 303 K and 473 K.

of diffraction peak measured in radians. The crystallite size increased from 20 to 35 nm (Figure 3) with increase of substrate temperature from 303 to 373 K due to improvement in crystallinity of the films. The mixed phase films formed at 423 K showed the crystalline size of 14 nm and at higher substrate temperature of 473 it decreased to 10 nm as shown in Figure 3. The sharp decrease in the crystallite size at substrate temperature of 473 K was due to decomposition of Ag_2O into Ag.

The X-ray photoelectron spectroscopic studies were performed on the films formed on silicon substrates held at different temperatures in order to determine the core level binding energies present in the films. Figure 4 shows a representative survey X-ray photoelectron spectrum of silver oxide films formed at 303 K. The spectrum showed the characteristic core level binding energies at about 368 and 374 eV related to the Ag $3d_{5/2}$ and Ag $3d_{3/2}$, respectively, due

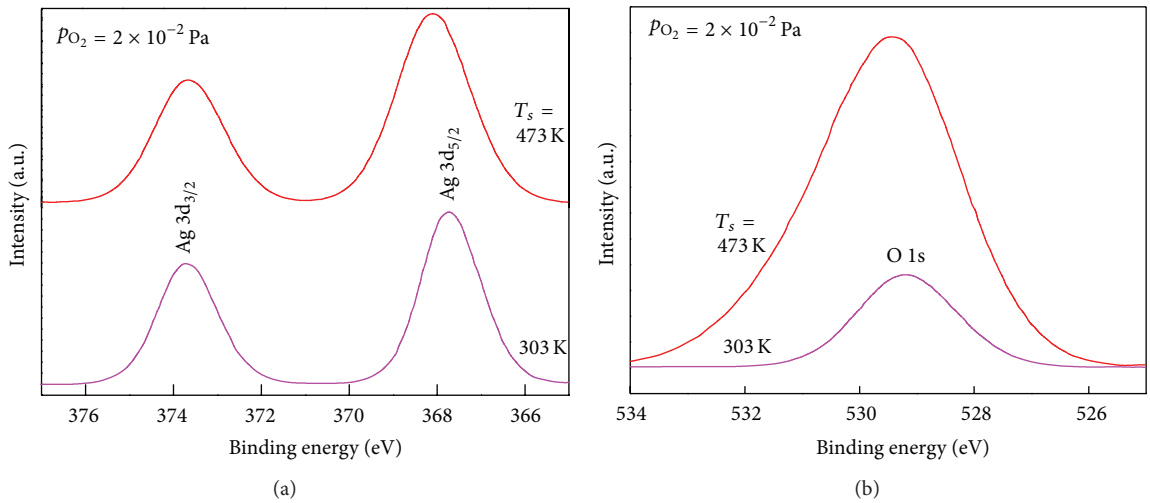

FIGURE 5: XPS narrow scan spectra of silver oxide films formed at substrate temperature of 303 K and 473 K: (a) Ag 3d and (b) O 1s.

the spin-orbit splitting of energy levels, and peaks situated at 573 eV and 604 eV correspond to the core levels of Ag $3p_{3/2}$ and Ag $3p_{1/2,}$ respectively, related to the Ag_2O films [30]. Figures 5(a) and 5(b) show the narrow scan spectra of Ag 3d and O 1s of silver oxide films formed at 303 K and 473 K. The films formed at 303 K showed core level binding energies of Ag $3d_{5/2}$ at 367.7 eV and O 1s at 529.2 eV. In the case of the films formed at substrate temperature of 473 K, the core level binding energy of Ag $3d_{5/2}$was shifted to 367.3 eV and O 1s shifted to 530.2 eV. The achieved core level binding energies in the Ag_2O films formed at 303 K were in agreement with the reports of Abe et al. [31] in reactive RF sputtered films. It is to be noted that Ag_2O core level binding energy was in the range 367.6–367.8 eV and O 1s was in the range 529.2–529.5 eV, while in pure metallic silver the Ag $3d_{5/2}$ was in the range 368.0–368.3 eV [10, 31, 32]. From these studies it is revealed that the films formed at 303 K were of single phase Ag_2O, while those deposited at 473 K were of mixed phase of Ag_2O and metallic silver. It was also confirmed by X-ray diffraction studies. It indicated that the films formed at substrate temperature of 303 K and 373 K were of Ag_2O, mixed phase of Ag_2O and Ag, at 423 K and at higher temperature of 473 K the grown films were of metallic silver.

Figure 6 shows three-dimensional and two-dimensional atomic force micrographs of films formed at different substrate temperatures. The micrographs showed different morphology of the grain growth depending on the substrate temperature. Atomic force micrographs of the films formed at 303 K showed spherical shape grains with size of 85 nm. When substrate temperature increased to 473 K the size of the grains increased to 215 nm and also transformed from the spherical shape grains into pyramidal-like shape. The films formed at substrate temperatures 303 K were uniform with root mean square roughness of 4.5 nm. The root mean square roughness of the films increased from 8.0 to 10.9 nm with increase of substrate temperature from 423 K to 473 K, respectively. The increase of surface roughness of the films with the substrate temperature may be due to the decomposition of Ag_2O phase into silver and oxygen.

The electrical resistivity of the deposited thin films is very sensitive to the grown phase and its microstructure. The substrate temperature has high influence on the electrical properties of the deposited films. The dependence of electrical resistivity of the films on the substrate temperature is shown in Figure 7. The single phase Ag_2O films formed at room temperature exhibited the electrical resistivity of 5.2 $\times 10^{-3}\,\Omega$ cm. The electrical resistivity of the films formed at substrate temperature of 373 was $3.0 \times 10^{-3}\,\Omega$ cm. The decrease of electrical resistivity with increase of substrate temperature up to 373 K was due to the improvement in the crystallinity of the films. The films formed at 423 K exhibited the electrical resistivity of $1.8 \times 10^{-3}\,\Omega$ cm. The low electrical resistivity of the films formed at 423 K was may be due to the presence of mixed phase of Ag_2O and Ag. Further increase of substrate temperature to 473 K; resistivity of the films decreased to $4.2 \times 10^{-4}\,\Omega$ cm because of transformation into metallic silver. The phase transformation from Ag_2O to Ag was also confirmed by the X-ray diffraction. Varkey and Fort [19] reported that the Ag_2O and AgO films formed on glass substrates by chemical bath deposition showed the electrical resistivity of 0.5 Ω cm and 0.12 Ω cm, respectively. Ravi Chandra Raju et al. [18] reported that the electrical resistivity increased from 1×10^{-2} to $4 \times 10^{2}\,\Omega$ cm with the increase of oxygen partial pressure from 9 to 50 Pa in pulsed laser deposited silver oxide films. The reported electrical resistivity in the Ag_2O films varied depending on the deposition method employed and the process conditions maintained during the growth of the films.

Figure 8 shows the wavelength dependent optical transmittance of the films formed at different substrate temperatures. The optical transmittance of the films formed at 303 K was about 24% (at wavelength of 1000 nm). The optical transmittance of the films increased to 58% with increase of substrate temperature up to 373 K. With further increase

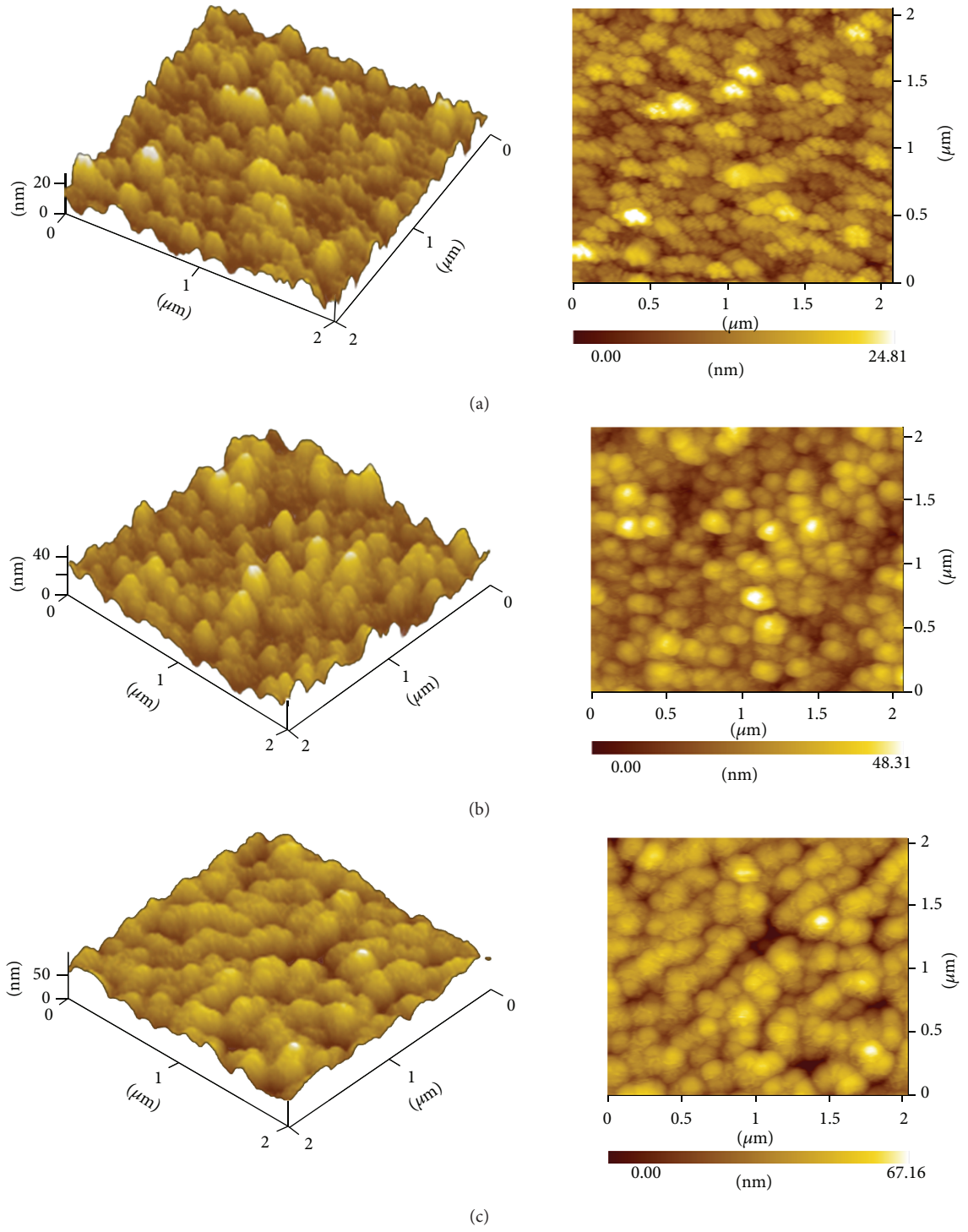

FIGURE 6: AFM 3d- and 2d-micrographs of Ag_2O films formed at substrate temperatures: (a) 303 K, (b) 423 K, and (c) 473 K.

of substrate temperature to 423 K the transmittance of the films decreased to 46%. The decrease in the transmittance in the films formed at 423 K was due to the mixed phase of Ag_2O and Ag where the metallic silver scatters the photons hence decreased in the transmittance. The films formed at higher substrate temperature of 473; there was decrease in the transmittance to 28%. The optical absorption edge of the films was shifted towards lower wavelength side with increase of substrate temperature from 303 to 373 K. With further increase of substrate temperature the absorption edge shifted towards higher wavelength side as shown in Figure 8. The absorption coefficient (α) of the films was calculated from the optical transmittance (T) data using the relation

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

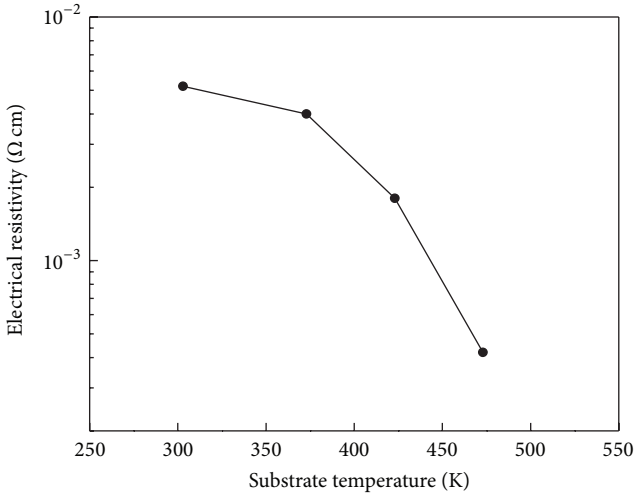

FIGURE 7: Dependence of electrical resistivity of Ag_2O films on the substrate temperature.

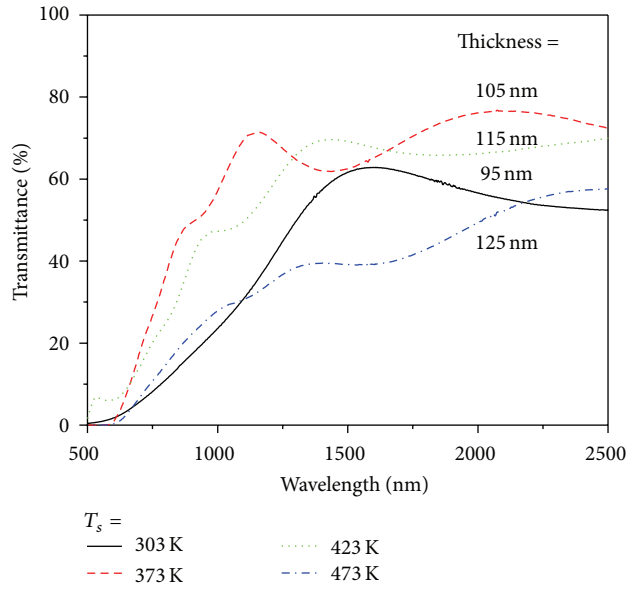

FIGURE 8: Optical transmittance spectra of Ag_2O films formed at different substrate temperatures.

where t is the film thickness. The optical band gap (E_g) of the films was estimated from the plots of $(\alpha h\nu)^2$ versus photon energy ($h\nu$) using Tauc's relation [33]:

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

where A is the absorption edge width parameter. Extrapolation of the linear portion of the plots of $(\alpha h\nu)^2$ versus photon energy to $\alpha = 0$ resulted in the optical band gap of the films. The optical band gap of the films increased from 2.05 to 2.13 eV with the increase of substrate temperature from 303 to 373 K. Further it decreased to 1.92 eV at substrate temperature of 423 K. Varkey and Fort [19] reported an optical band gap of 2.25 eV in Ag_2O films produced by chemical bath deposition. Pierson et al. [26] achieved an optical band gap of 2.23 eV in Ag_2O films formed by DC reactive magnetron sputtering. Rivers et al. [34] achieved a high optical band gap of 3.3 eV in Ag_2O films formed by evaporation of silver in the presence of ECR oxygen plasma. Ma et al. [27] reported an optical band gap decrease from 3.25 to 2.77 eV with increase of substrate temperature from 373 to 498 K in RF reactive magnetron sputtered Ag_2O films. The variations on the optical band gap for Ag_2O films depend on the deposition method employed and the process parameters maintained during the growth of the films.

4. Conclusions

Silver oxide films were deposited on glass substrates by RF magnetron sputtering of pure silver target under various substrate temperatures in the range 303–473 K. The effect of substrate temperature on the core level binding energies, structure and surface morphology, and electrical and optical properties was investigated. The films deposited at 303 K were polycrystalline with cubic structure of Ag_2O. As the substrate temperature increased to 373 K, the crystallinity of the films increased. The films formed at substrate temperature of 423 K were of mixed phase of Ag_2O and Ag, while those deposited at 473 K were of single phase Ag. The phase transformation from Ag_2O to Agwas also confirmed from the core level binding energies determined by X-ray photoelectron spectroscope. Atomic force microscopic studies on the films indicated that the grain growth transformed from spherical to pyramidal-like shape with increase of substrate temperature from 303 to 473 K, respectively. Single phase Ag_2O films formed at 303 K exhibited the electrical resistivity of $5.2 \times 10^{-3}\ \Omega$ cm, while those deposited at 473 K decreased to $4.29 \times 10^{-4}\ \Omega$ cm due to the formation of metallic silver. The optical band gap of the Ag_2O films increased from 2.05 to 2.13 eV with increase of substrate temperature from 303 to 373 K due to improvement in the crystallinity, while in the case of mixed phase of Ag_2O and Ag films deposited at substrate temperature of 423 K the optical band gap of 1.92 eV was shown.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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