Research Article

Effect of Oxygen Partial Pressure on the Electrical and Optical Properties of DC Magnetron Sputtered Amorphous TiO_2 Films

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Titanium dioxide (TiO_2) thin films were deposited on p-Si (100) and Corning glass substrates held at room temperature by DC magnetron sputtering at different oxygen partial pressures in the range 9×10^{-3} – 9×10^{-2} Pa. The influence of oxygen partial pressure on the structural, electrical, and optical properties of the deposited films was systematically studied. XPS studies confirmed that the film formed at an oxygen partial pressure of 6×10^{-2} Pa was nearly stoichiometric. TiO_2 films formed at all oxygen partial pressures were X-ray amorphous. The optical transmittance gradually increased and the absorption edge shifted towards shorter wavelengths with the increase of oxygen partial pressure. Thin film capacitors with configuration of Al/ TiO_2 /p-Si have been fabricated. The results showed that the leakage current density of films formed decreased with the increase of oxygen partial pressure to 6×10^{-2} Pa owing to the decrease in the oxygen defects in the films thereafter it was increased. The current transport mechanism in the TiO_2 thin films is shown to be Schottky effect and Fowler-Nordheim tunnelling currents.

1. Introduction

Titanium dioxide (TiO_2) thin films are widely used because of their remarkable electrical and optical properties. TiO_2 can be used as an alternative dielectric to silicon dioxide films in ultra-large-scale integration due to its much higher dielectric constant [1], lower leakage current, and higher breakdown strength than that of SiO_2 [2]. TiO_2 thin films have attracted considerable attention for applications in photocatalysts and solar cells [3]. These films also exhibit a large potential in the fields such as protective antireflection coatings [4], gas sensors [5], and air purification [6]. TiO_2 films were prepared by numerous deposition techniques such as metal organic chemical vapour deposition, spray pyrolysis, sol-gel process, thermal oxidation, pulsed laser deposition, and reactive magnetron sputtering [7–10]. However, the electrical properties of the oxide layer are affected by the growth technique due to reactions at the oxide/Si substrate interface [11]. Among these techniques, magnetron sputtering method provides more advantage in controlling the microstructure and composition of the films. The structural and electrical properties are known to be easily affected by the deposition

conditions such as the substrate temperature, substrate bias voltage, and oxygen partial pressure as well as the postdeposition annealing. The effect of substrate temperature [12] and substrate bias voltage [13] on the structural, electrical, and dielectric properties of magnetron sputtered TiO_2 films was reported. In this investigation, the influence of oxygen partial pressure on the structural and electrical behavior of DC magnetron sputtered TiO_2 thin films was studied.

2. Experimental

2.1. Thin Film Preparation. Thin films of TiO_2 were deposited on RCA-cleaned and HF-treated, low-resistivity (0.01 – $0.02 \Omega\text{cm}$) p-type silicon (100) and Corning glass substrates by DC reactive magnetron sputtering technique. Pure titanium target of 100 mm diameter and 3 mm thick was used as sputter target. The vacuum pumping system employed for sputtering was a combination of diffusion and rotary pumps. Pure oxygen and argon were used as reactive and sputtering gases, respectively. After achieving ultimate pressure of 2×10^{-4} Pa, the fixed quantities of

FIGURE 1: Sample configuration for electrical measurement.

FIGURE 2: Variation of deposition rate of TiO_2 films with oxygen partial pressure.

oxygen and argon gases were admitted into the sputter chamber through the flow controlled needle valves and their flow rates were monitored individually employing Aalborg mass flow controllers. The TiO_2 films were deposited at different oxygen partial pressures by fixing the other process parameters kept constant. The deposition conditions maintained during the growth of the TiO_2 films are given in Table 1. In order to fabricate metal-oxide-semiconductor structure, TiO_2 layer was formed on the p-Si (100) wafers. Aluminium top contacts were formed by sputtering using shadow masks over the oxide layer with fixed area of $9 \times 10^{-4} \text{ cm}^2$. The back contacts of thick Al layer, which was deposited by sputtering. High doping in Si ensured that the back contacts were ohmic. The sample configurations for metal insulator-semiconductor for electrical measurements are shown in Figure 1.

TABLE 1: Deposition conditions maintained during the preparation of TiO_2 films.

Sputter target	Titanium target (99.9% pure)
Target to substrate distance	80 mm
Ultimate pressure	$2 \times 10^{-4} \text{ Pa}$
Oxygen partial pressure	$9 \times 10^{-3} - 9 \times 10^{-2} \text{ Pa}$
Sputter pressure	0.5 Pa
Substrate temperature	303 K
Sputter power	200 W

2.2. Thin Film Characterization. The X-ray photoelectron spectroscopic (XPS) studies were performed to analyze the core level binding energies by using SPECS GmbH spectrometer (Phoibos 100 MCD Energy Analyzer) with $\text{MgK}_{\alpha 1}$ radiation (1253.6 eV). The chemical binding configuration, structural properties of TiO_2 thin films were determined using Raman spectra recorded at room temperature using Jobin Yvon (Model HR 800 UV) Raman spectrometer in the wavenumber range $100-1000 \text{ cm}^{-1}$. X-ray diffractometer (Seifert model 3003 TT) with $\text{CuK}_{\alpha 1}$ radiation ($\lambda = 0.15405 \text{ nm}$) was used to analyze the crystallographic structure of the films formed on silicon substrates. The physical thickness of the films was measured by using α -step profilometer. The optical transmittance of the films formed on Corning glass substrates was recorded by using UV-Vis-NIR double beam spectrophotometer (Perkin Elmer model Lambda 950) in the wavelength range 300–1500 nm. The electrical properties of the TiO_2 thin films were evaluated by current-voltage (I-V) measurements. The test samples with metal-insulator-semiconductor (MIS) capacitor structures were prepared on p-type Si (100) substrates. Aluminum electrodes were prepared by using DC reactive magnetron sputtering. The current-voltage characteristics of the Al/ TiO_2 /p-Si capacitors were measured by using Hewlett Packard (model hp 4140 B) pA meter.

3. Results and Discussion

3.1. Structural Properties. Figure 2 shows the dependence of deposition rate of TiO_2 films on the oxygen partial pressure. The deposition rate of the films formed at low oxygen partial pressure of $9 \times 10^{-3} \text{ Pa}$ was 4.6 nm/min. The deposition rate of the films is decreased to 1.9 nm/min at an oxygen partial pressure of $9 \times 10^{-1} \text{ Pa}$. The high deposition rate at low oxygen partial pressure was due to the high sputtering yield of metallic titanium and insufficient oxygen present in the sputter chamber. The decrease of deposition rate at higher oxygen partial pressure was related to the chemical reaction between the target surface and the reactive gas of oxygen. This led to the formation of oxide layer on the target surface and in turn reduces in the deposition rate [14]. Jagadeesh Chandra et al. [15] also observed the decrease of films thickness with the increase of oxygen partial pressure in RF magnetron sputtered Ta_2O_5 films.

Figure 3 shows the XPS survey spectra of TiO_2 thin films formed at different oxygen partial pressures. The survey

FIGURE 3: XPS survey scan spectra of TiO_2 films formed at different oxygen partial pressures.

spectra show the characteristic of titanium, oxygen, and carbon peaks. A carbon 1s peak was observed at a binding energy of 284.4 eV on all samples before ion bombardment. The presence of this peak is related to organic surface contamination, which corresponds to the fact that the samples are exposed to air before measurements. The carbon peak disappeared from the surface of TiO_2 films after presputtering with argon ion bombardment.

Figure 4(a) shows the narrow scan spectra of Ti 2p in TiO_2 films formed at different oxygen partial pressures in the range 9×10^{-3} – 6×10^{-2} Pa. The Ti 2p signal is associated with two peaks which are representative for Ti $2p_{3/2}$ to Ti $2p_{1/2}$ spin-orbital splitting. The films formed at 9×10^{-3} Pa showed the core level binding energies of Ti $2p_{3/2}$ and Ti $2p_{1/2}$ is 457.2 and 462.7 eV (energy separation of 5.5 eV). The core level binding energies of the films formed at 6×10^{-2} Pa shifted to 458.2 and 463.9 eV (energy separation of 5.7 eV) which indicated the presence of Ti^{4+} oxidation state in TiO_2 . Liu et al. [16] reported the core level binding energies of Ti 2p with two peaks located at 458.1 and 463.7 eV for Ti $2p_{3/2}$ and Ti $2p_{1/2}$ with energy separation of 5.6 eV. Figure 4(b) showed the narrow scan spectra of O 1s at different oxygen partial pressures. The core level binding energy of O 1s peak shifted from 529.7 to 530.5 eV with increase of oxygen partial pressure from 9×10^{-3} to 6×10^{-2} Pa, respectively. The chemical composition of the deposited films was determined from the area under the peak using the sensitivity factors ($\text{C } 1s = 0.25$, $\text{Ti } 2p = 1.8$, and $\text{O } 1s = 0.66$). The films deposited at 6×10^{-2} Pa showed the ratio of oxygen to titanium, that is, $\text{O}/\text{Ti} = 1.97$, which indicated the growth of nearly stoichiometric films [17].

Figure 5 shows the X-ray diffraction profiles of the TiO_2 thin films deposited at different oxygen partial pressures. It can be seen that all the films have not shown any diffraction peaks indicating the films were amorphous in nature. The

(a)

(b)

FIGURE 4: XPS narrow scan spectra of TiO_2 films: (a) Ti 2p and (b) O 1s.

amorphous structure of TiO_2 thin films can be attributed to low surface mobility of deposition particles [18].

Anatase has a space group D_{4h} ($I4_1/amd$) containing two formula units per primitive unit cell. From the group analysis, there are six Raman active modes, $A_{1g} + 2B_{1g} + 3E_g$ and three infrared active modes, $A_{2u} + 2E_u$. The Raman spectra for anatase single crystal were investigated by Ohsaka [19] who noticed six allowed bands in the first order Raman spectra were identified at 144 cm^{-1} (E_g), 197 cm^{-1} (E_g), 399 cm^{-1} (B_{1g}), 513 cm^{-1} (A_{1g}), 519 cm^{-1} (B_{1g}), and 639 cm^{-1} (E_g). The Raman spectra for the as-prepared TiO_2 thin films at

FIGURE 5: X-ray diffraction profiles of TiO_2 films formed at different oxygen partial pressures.

FIGURE 6: Raman spectra of TiO_2 films formed at different oxygen partial pressures.

different oxygen partial pressures were shown in Figure 6. Raman peaks seen at 153, 516, 659, and 810 cm^{-1} are assigned as $B_{1g}(\nu_6)$, $A_{1g} + B_{1g}(\nu_2 + \nu_3)$, $E_g(\nu_1)$, and B_{1g} Ti-O modes related to the anatase phase [20]. It is noted that the Raman peaks at 302 cm^{-1} and 520 cm^{-1} appeared from Si-Si bonding mode of the Si substrate. The Raman peaks at 516 cm^{-1} were not present because the intensity of Si dominates the intensity of B_{1g} doublet peaks. The Raman peaks at 233, 430, and 620 cm^{-1} related to the rutile phase [21]. It is seen that the peak at 153 cm^{-1} (E_g mode) strengthens, with increase in oxygen partial pressure from 9×10^{-3} to 6×10^{-2} Pa, thereafter it weakens with the oxygen partial pressure up to 9×10^{-2} Pa due to the reduction of film thickness. The rutile

peak intensity was also decreased with increasing oxygen flow ratio as well as film thickness.

3.2. Optical Properties. The optical transmittance spectra of the TiO_2 films formed at different oxygen partial pressures are shown in Figure 7. Obviously, the optical transmittance is high in the visible region and it increased with the increase of oxygen partial pressure from 9×10^{-3} to 9×10^{-2} Pa, respectively. The optical transmittance of the films formed at low oxygen partial pressure of 9×10^{-3} Pa was low maybe due to the formation of nonstoichiometric TiO_2 films. As the oxygen partial pressure increased to 6×10^{-2} Pa, the adequate oxygen can repair the oxygen vacancies and hence increase the transmittance. The relation between the absorption coefficient and concentration of free carriers can be written as [22]

$$\alpha = \frac{Ne^2\lambda_0^2}{8\epsilon_0\pi\Gamma m^*nc^3}, \quad (1)$$

where α is the absorption coefficient, N the concentration of free carriers, n the refractive index, m^* the effective mass of free carrier, and λ_0 the absorption wavelength. Equation (1) shows that the absorption coefficient of TiO_2 films is directly proportional to the concentration of free carrier, namely, twofold concentration of oxygen vacancies. Hence, the substoichiometry of the TiO_2 films induced by insufficient oxygen content is the main cause of transmittance differences. The transmittance of TiO_2 thin films deposited at 9×10^{-3} Pa is low, which indicated that the films have more oxygen vacancies. As the oxygen partial pressure increased to 6×10^{-2} Pa, the transmittance of the films increased since the oxygen ion vacancies tend to decrease and hence the composition of the films approached to the stoichiometric TiO_2 . Heo et al. [23] also observed the increase in optical transmittance with the increase of oxygen flow by RF magnetron sputtering method. The optical absorption edge of the films shifted markedly towards lower wavelengths side with the increase of oxygen partial pressure. The optical absorption coefficient (α) of the films was evaluated from the optical transmittance (T) and reflectance (R) data using the relation

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

where t is the thickness of the film. The optical band gap (E_g) of the films was estimated from the intercept of the plots of $(\alpha hv)^2$ versus photon energy (hv) which assumes the direct transition between the top of the valance band and the bottom of the conduction band using the relation

$$(\alpha hv) = A(hv - E_g)^{1/2}, \quad (3)$$

where A is the optical absorption edge width parameter. The optical band gap of the films increased from 3.45 to 3.60 eV with the increase of oxygen partial pressure from 9×10^{-3} to 9×10^{-2} Pa. The low value of optical band gap of the films formed at low oxygen partial pressure was due

FIGURE 7: Optical transmittance spectra of TiO_2 films formed at different oxygen partial pressures.

FIGURE 8: Current-voltage characteristics of $\text{Al}/\text{TiO}_2/\text{p-Si}$ capacitors.

to the formation of nonstoichiometric films. In the present investigation, the stoichiometric films deposited at an oxygen partial pressure of 6×10^{-2} Pa showed an optical band gap of 3.50 eV.

3.3. Electrical Properties. Figure 8 shows the leakage current density versus applied voltage characteristics of the $\text{Al}/\text{TiO}_2/\text{p-Si}$ capacitors. The leakage current density (J)

measured at applied voltage $V = 1.5$ V was decreased from 6.1×10^{-5} to 1.3×10^{-6} A/cm^2 with the increase of oxygen partial pressure from 9×10^{-3} to 6×10^{-2} Pa. At higher oxygen partial pressure of 9×10^{-2} Pa, the leakage current density was increased to 5.5×10^{-6} A/cm^2 [15, 24]. The increase in leakage currents at higher oxygen partial pressures was due to the decrease in film thickness. To validate the current transport mechanism through the $\text{Al}/\text{TiO}_2/\text{p-Si}$ capacitors, two main conduction mechanisms were examined. At low electric fields, the current density was proportional to the square root of the applied electric field (Schottky emission), while at higher electric fields it may be the possibility of Fowler-Nordheim conduction mechanism that was observed. The Schottky emission is given by the relation [17]

$$J_{\text{SE}} = A^* T^2 \exp \left\{ \frac{-1}{kT (q\Phi - \beta_{\text{SE}} \sqrt{E})} \right\}, \quad (4)$$

where A^* is the effective Richardson constant, Φ the Schottky barrier height q the electronic charge, k the Boltzmann constant, E the electric field, and h the Planck's constant. The constant β_{SE} is given by the relation

$$\beta_{\text{SE}} = \left(\frac{q^3}{4\pi\epsilon_0\epsilon_r} \right)^{1/2}, \quad (5)$$

where ϵ_0 is the permittivity of free space and ϵ_r the dielectric constant of the insulator. At higher bias voltages, the Fowler-Nordheim tunnelling of electrons through the triangular barrier to the conduction band of oxide will occur. The Fowler-Nordheim tunnelling current J_{FN} can be expressed as a function of the electric field in the gate oxide by the relation

$$J_{\text{FN}} = A E^2 \exp \left[-\frac{B}{E} \right], \quad (6)$$

where $A = q^2/8\pi h \phi_B$ and $B = (8\pi \sqrt{2qm^*}/3h) \phi_B^{3/2}$ are constants depending on m^* and barrier height. m^* , the electron effective mass in dielectric layer which is $m^* = 0.3 m_0$ where m_0 is the free-electron mass [25].

Figure 9 shows $\ln(J)$ versus $E^{1/2}$ plots of the $\text{Al}/\text{TiO}_2/\text{p-Si}$ capacitors formed at different oxygen partial pressures in the range 9×10^{-3} – 9×10^{-2} Pa with the applied positive bias voltage from 0 V to +10 V. Schottky emission follows at the interface between the TiO_2 films and the aluminum electrode where a Schottky barrier is formed. This effect was attributed to the absorption current due to the dielectric relaxation phenomenon of the TiO_2 capacitors. The plot $\ln(J)$ versus $E^{1/2}$ showed a linear relationship with the applied electric field. This result indicates the I-V characteristics of TiO_2 capacitors can be explained by Schottky emission in lower electric field region.

Figure 10 shows $\ln(J/E^2)$ versus $E^{1/2}$ (Fowler-Nordheim) plots of $\text{Al}/\text{TiO}_2/\text{p-Si}$ capacitors at positive substrate bias. It is noted that there are two significant regimes shown in the Fowler-Nordheim plot. At low electric fields, Schottky emission is the dominant current transport mechanism, as shown by the leakage current density versus applied gate voltage. At higher electric fields, Fowler-Nordheim tunnelling was observed.

FIGURE 9: Schottky plots of $\ln(J)$ versus $E^{1/2}$ of Al/TiO₂/p-Si capacitors.

FIGURE 10: Fowler-Nordheim plots of $\ln(J/E^2)$ versus $E^{1/2}$ of Al/TiO₂/p-Si capacitors.

4. Conclusions

Titanium oxide thin films were formed on glass and p-silicon (100) substrates deposited by DC magnetron sputtering method under various oxygen partial pressures in the range 9×10^{-3} – 9×10^{-2} Pa. The deposition rate of the films was

correlated with the oxygen partial pressure. From XPS studies it was confirmed that the films formed at an oxygen partial pressure of 6×10^{-2} Pa were nearly stoichiometric. X-ray diffraction studies indicated the films deposited at all oxygen partial pressure were amorphous. The optical transmittance of the films was increased with the increase of oxygen partial pressure; the transmittance edge shifted towards the lower wavelength side. This may be ascribed to the decrease of oxygen vacancies as the increase of oxygen partial pressure. The optical band gap of the films increased from 3.45 to 3.60 eV with the increase of oxygen partial pressure from 9×10^{-3} to 9×10^{-2} Pa, respectively. Thin film capacitors with configuration of Al/TiO₂/p-Si have been fabricated. The leakage current density of the films formed at low oxygen partial pressure of 9×10^{-3} Pa was 6.1×10^{-5} A/cm² and it was decreased to 1.3×10^{-6} A/cm² with the increase of oxygen partial pressure to 6×10^{-2} Pa and thereafter it was increased to 5.5×10^{-6} A/cm². The leakage current is found to be dominated by the Schottky emission at low electric field, whereas Fowler-Nordheim tunnelling currents appear at higher electric fields.

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