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Electron transport mechanism through ultrathin Al₂O₃ films grown at low temperatures using atomic–layer deposition

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Abstract

Alumina (Al₂O₃) films of different thicknesses have been grown at different low temperatures (100–250 °C) by atomic–layer deposition on n–type Si substrate. The robustness of the Al₂O₃ film as a barrier has been investigated based on Al/Al₂O₃/Si metal–insulator–semiconductor structures. The electron transport through the Al₂O₃ layer was fitted well by the Fowler–Nordheim tunneling mechanism, from which the barrier heights (conduction band offset between Si and Al₂O₃) were deduced. It was discovered that the growth temperature and film thickness both influenced the carrier transport and barrier height. The Al/Al₂O₃/Si structure with an ultrathin 3 nm Al₂O₃ fabricated at 150 °C showed the largest barrier height, the lowest tunneling current density (4.9×10^{-8} A/cm² at 5 MV/cm), and the highest breakdown field strength of 18.3 MV/cm. Using Au to replace Al as the electrode could suppress the tunneling current significantly. The Al₂O₃ films were also examined by X–ray photoelectron spectroscopy to determine their chemical constituents.

Keywords:

ultrathin Al₂O₃, tunneling current, low temperature, atomic–layer deposition

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1. Introduction

Alumina (Al_2O_3), a high- κ dielectric material ($\kappa \sim 9$), is widely used in microelectronic devices [1, 2]. Being an outstanding barrier material, Al_2O_3 is often used to encapsulate electronic devices to enhance the reliability through preventing H_2O and O_2 penetration [3-5]. Due to its large bandgap ($E_g \sim 8$ eV), few electronic defects, high breakdown electrical field, and superior thermal and chemical stability, Al_2O_3 is also employed to substitute SiO_2 as the gate dielectric in thin-film transistors (TFTs) to realize low-voltage operation and suppress the leakage current [6-9]. However, when an ultra-thin Al_2O_3 film was used as the gate dielectric in TFTs, carriers in channel could tunnel through the Al_2O_3 layer into the gate electrode, and positive charges can be generated inside the gate insulator as reported in reference [10]. In addition, thin Al_2O_3 films are also an excellent blocking and/or tunneling barrier in flash memory devices. For example, researchers have explored the effects of Al_2O_3 on lowering the operation voltages in charge trapping memory (CTM) devices based on $\text{TaN}(\text{TiN})/\text{Al}_2\text{O}_3/\text{Si}_3\text{N}_4/\text{SiO}_2/\text{Si}$ (TANOS) [11, 12], $\text{Metal}/\text{Al}_2\text{O}_3/\text{Si}_3\text{N}_4/\text{SiO}_2/\text{Si}$ (MANOS) [13] and $\text{Metal}/\text{Al}_2\text{O}_3/\text{HfO}_2/\text{Al}_2\text{O}_3/\text{Si}$ (MAHAS) [14] memory structures. In these devices, the transport of electrons and holes through the tunneling layer is critical. The barrier height between Si and Al_2O_3 (as tunneling layer) or between the electrode and Al_2O_3 (as blocking layer) plays an important role in device performance, especially for programming and erasing efficiencies. Thus, it is worth to deeply understand the electron transport mechanism and dielectric behavior of ultrathin Al_2O_3 films.

In recent years, much interest has also been generated to develop flexible transistors and memories for wearable electronics, item-level radio-frequency identification tags, etc. Owing to the temperature sensitivity of flexible substrates, developing a high quality dielectric that can be fabricated at low temperatures for flexible devices and circuits is crucial [15, 16]. Ultrathin Al_2O_3

films can be grown at low temperatures on flexible substrate such as polyimide by atomic-layer deposition (ALD), which can precisely control the film thickness with angstrom precision [17]. For Al_2O_3 film growth, there are two similar reactions

Al_2O_3 films grown by ALD are highly uniform and conformal, and hence has a great promise for future gate-all-around devices [18, 19] and 3D NAND applications [20, 21].

This work focused on the investigation of the electron transport mechanism and dielectric behavior of ultrathin amorphous Al_2O_3 films grown at low temperatures (100–250 °C) using the thermal ALD technique. Current–voltage (I – V) measurements of metal oxide semiconductor (MOS) capacitor structures were conducted to determine the electron transport mechanism and barrier height by fitting current density–electric field (J – E) curves through Fowler–Nordheim model, which is confirmed by X–ray photoelectron spectroscopy measurements. We exact electric field by simply using the equation of $E = V/t$, where E is the electric field, V is the bias voltage, and t is the thickness of the insulation layer. Capacitance–voltage (C – V) measurements were used to investigate the dielectric characteristics including flat band voltage, border trapped oxide charges (N_{bt}), and fixed oxide charges (N_{fc}). Both the growth temperature and film thickness have been varied to identify the optimum conditions to achieve the best electrical properties.

2. Experimental Methods

The diagram of the fabricated Al/ Al_2O_3 /n–Si structure is shown in Fig. 1(a). A 5–nm–thick Al_2O_3 layer was first grown by ALD on n^+ –Si wafers (resistivity ~ 0.008 – $0.02 \ \Omega\cdot\text{cm}$) for I – V measurements. The deposition temperatures were varied from 100 °C to 250 °C. Trimethylaluminum (TMA) and deionized water were selected as reaction precursors with N_2 as

the carrier gas. For one ALD cycle deposition, the sequence was TMA dose (0.03 s)–N₂ purge (30 s)–H₂O dose (0.015 s)–N₂ purge (30 s) at a chamber pressure of 0.15 Torr. The deposition rate was ~1 Å per cycle. Then, 100 nm Al top electrodes were deposited using thermal evaporation deposition and patterned into circular pads with a radius of 150 µm by a shadow mask. Al₂O₃ films of different thicknesses (10, 7, 5, and 3 nm) were grown at 150 °C to explore the thickness effects on device performance. Besides the Al electrode, a high work function metal, Au, was also applied to 5 nm Al₂O₃ devices. *I–V* and *C–V* measurements were conducted using Agilent B2900 and Agilent B1500A, respectively (Agilent Technologies, USA). The elemental constituents of Al₂O₃ films were examined by X-ray photoelectron spectroscopy (XPS) with ESCALAB 250 (ThermoFisher Scientific, Canada).

3. Results and discussion

To explore the carrier transport mechanism and test the robustness of the Al₂O₃ films, *I–V* measurements were carried on Al/Al₂O₃/n⁺–Si capacitor structures. During the measurements, the voltage was applied on the Al electrode of devices with grounding the Si substrate. Figure 1(b) shows the *J–E* curves of devices with 5 nm Al₂O₃ layers grown at different temperatures. The statistics of the breakdown electric fields are shown in Fig. 1(c). As seen in Fig. 1(c), the breakdown electric field increases monotonically until the temperature reaches 220 °C, at which the highest breakdown electric fields is 15.6 MV/cm. While the tunneling currents under the same electric field became large after 150 °C. For deeper analysis of the tunneling mechanism, we matched the tunneling current curves using many models, including Poole–Frenkel (P–F) emission, Fowler–Nordheim (F–N) tunneling, direct tunneling, and Schottky emission. By comparing the fittings of *J–E* curves for various models, the F–N tunneling provided good agreement with the tunneling current. The F–N tunneling current can be given by the following formula:

$$\frac{J}{E^2} = C_1 e^{-C_2/E}, \quad (3)$$

where E is the electric field across the oxide and J is the current density. C_1 and C_2 are given by

$$C_1 = q^3 m / 16 \pi^2 \hbar m_{\text{ox}} \phi_0 \quad \text{and} \quad (4)$$

$$C_2 = \frac{4}{3} \frac{(2m_{\text{ox}})^{1/2}}{q \hbar} \phi_0^{\frac{3}{2}}, \quad (5)$$

where m_{ox} and m are the electron mass in the oxide and in free space respectively, $m_{\text{ox}} = 0.23m$ [22], $2\pi\hbar$ is Planck's constant, q is the electron charge, and ϕ_0 is the barrier height [23].

FIG. 1. (a) Schematic diagram (b) J - E curves and (c) statistics of breakdown electric fields of Al/Al₂O₃/n-Si devices with 5 nm Al₂O₃ films grown at different temperatures.

The tunneling currents for 5 nm Al₂O₃ films grown at different temperatures were fitted by Eq. 3, and the linear fittings are shown in Fig. 2(a). The dotted points are the experimental results and the solid lines are the fittings. F-N tunneling is determined by a triangular barrier, and carriers only tunnel through the part of the insulator layer.[23] Figure 2(c) shows that a narrower barrier occurs with a higher field. Barrier heights (conduction band offset (ΔE_C) between Si and Al₂O₃) can be deduced from the fittings. Statistics of barrier heights and tunneling currents at 3 V bias voltage are

shown in Fig. 2(b). It is obvious that large barrier heights can suppress the tunneling current well. As shown in Fig. 2(b), 5 nm Al_2O_3 grown at 150 °C possesses a large barrier with 2.36 eV and the tunneling current was suppressed at 0.2 μA under 3 V bias voltage. All the key parameters of Al_2O_3 films grown at different temperatures are summarized in Table I. The differences in barriers and tunneling currents among these films may be caused by the varied extents of reaction between two precursors (TMA and H_2O) when the growth temperature differs. It was reported that the growth of Al_2O_3 film was dependent on surface chemistry and surface species, which were influenced by the temperature during every cycle of ALD growth [24].

FIG. 2 (a) Linear fittings for tunneling currents of the 5 nm Al_2O_3 layers grown at different temperatures by the F-N tunneling model. (b) Statistics of barrier heights and tunneling currents at 6 MV/cm. (c) Energy-band diagrams of

biased Al/ Al_2O_3 /n-Si capacitors with the F-N tunneling mechanism.

TABLE I. Summary of properties of 5 nm Al_2O_3 films fabricated at different growth temperatures.

Temperature (°C)	100	150	180	200	220	250
Breakdown electric fields (MV/cm)	10.4	12.2	12.2	12.8	15.6	14.6
Current at 6 MV/cm (μA)	0.6	0.2	9.5	4.5	5.9	3.8
Barrier height (eV)	2.19	2.36	1.80	1.86	1.82	1.90

Figure 3(a) shows C - V test results of Al/ Al_2O_3 /n-Si devices with 10 nm dielectric layers at

different growth temperatures. Herein, the 10 nm Al_2O_3 thick films were used to accurately test the dielectric properties, and the substrate is a slightly n-doped Si wafer with a resistivity of 2.5–4.0 $\Omega\cdot\text{cm}$. The dielectric constant of the Al_2O_3 film deduced from the C – V measurement was slightly dependent on the fabrication temperature. The density of border trapped oxide charge (N_{bt}) and fixed oxide charge (N_{fc}) were obtained from the C – V results, as shown in Fig. 3(b). N_{bt} and N_{fc} can be calculated using the following equations:

$$N_{\text{bt}} = (C_{\text{max}} \Delta V_{\text{fb}})/qA \text{ and} \quad (6)$$

$$N_{\text{fc}} = -C_{\text{max}}(V_{\text{fb}} - \phi_{\text{ms}})/qA, \quad (7)$$

where C_{max} is the maximal value of capacitance, V_{fb} is the value of flat band voltage, ϕ_{ms} is the difference work function of Al electrode and n-Si substrate, and A is the electrode area [25]. Values of flat band voltage, found to be –0.73, 0.08, 0.44, and 0.52 V, were obtained from the C – V curves for films grown at different temperatures. It was reported that H_2O did not react with the hydrogen-terminated silicon (H-Si) surface even increasing H_2O dose amount, while some reaction of TMA with the H-Si surface could occur.[24] During the low temperature ALD process, some positive fixed charges could be formed due to the existence of dangling bonds of the Si substrate, which leading to a negative shift of V_{fb} [25]. With increasing the growth temperature, the dangling bonds may be saturated with TMA precursors and the V_{fb} shifted positively. As shown in Fig. 3(a), almost no hysteresis was observed for Al_2O_3 films grown at 100 °C and 150 °C, while ΔV_{fb} was found to be 0.16 V and 0.38 V for films grown at 200 °C and 250 °C, respectively. The increase of border traps may be caused by the desorption of H_2O at high temperature, which leads to the incomplete reaction.[24] The values of N_{fc} and N_{bt} and the dielectric constant of 10 nm Al_2O_3 films fabricated at different growth temperatures are summarized in Table II. To ensure good dielectric quality at low temperature, we chose 150 °C as the optimum growth temperature.

FIG. 3. (a) C - V tests of Al/Al₂O₃/n-Si devices with different growth temperatures. (b) Statistics of the density of border trapped oxide charge (N_{bt}) and fixed oxide charge (N_{fc}).

TABLE II. Summary of properties of 10 nm Al₂O₃ films fabricated at different growth temperatures.

Temperature (°C)	100	150	200	250
Dielectric constant	6.3	6.3	6.2	6.3
V_{fb} (V)	-0.73	0.08	0.44	0.52
N_{bt} (x10 ¹¹ cm ⁻²)	0.69	0.35	5.54	13.16
N_{fc} (x10 ¹² cm ⁻²)	3.91	1.14	-0.14	-0.42

To explore the influence of film thickness on electrical conduction, 10, 7, 5, and 3 nm Al₂O₃ films were prepared at 150 °C, and Al/Al₂O₃/n⁺-Si devices were fabricated for J - E tests. Figure 4(a) shows the J - E curves of devices with different Al₂O₃ thicknesses. The breakdown voltage increased with the rise in Al₂O₃ thickness, while the breakdown electric field strength was inversely proportional to the film thickness. The Al/Al₂O₃/n⁺-Si structure with an ultrathin 3 nm Al₂O₃ showed the largest barrier height, low tunneling current density (4.9×10^{-8} A/cm² at 5 MV/cm), and a high breakdown electric field of 18.3 MV/cm. It is known that the growth of the Al₂O₃ film occurs during alternating exposures of TMA and H₂O. However, the layer-by-layer growth during the ALD process does not always provide the expected yield because the growth is influenced by surface species and chemistry [24]. With the increasing in the number of cycles, a few incomplete

reactions may exist, which could deteriorate film quality and reduce the film's breakdown field strength. The tunneling currents of devices with different Al_2O_3 thicknesses were also fitted using Eq. 3. Barrier heights were deduced from fittings and summarized in Fig. 4(b) with tunneling current density at 5 MV/cm. Barrier heights decrease with the increase in film thickness, leading to an increase in current density under the same electric field. The properties of Al_2O_3 films of different thicknesses grown at 150 °C are summarized in Table III.

FIG. 4. (a) J - E curves of devices with different Al_2O_3 thicknesses grown at 150 °C. (b) Statistics of barrier heights and tunneling current densities at 5 MV/cm.

TABLE III. Summary of the properties of Al_2O_3 films of different thicknesses grown at 150 °C.

Thickness (nm)	3	5	7	10
Breakdown electric field (MV/cm)	18.3	12.2	9.7	8.7
Current density at 5 MV (A/cm^2)	4.9×10^{-8}	3.9×10^{-6}	9.5×10^{-5}	1×10^{-3}
Barrier height (eV)	2.84	2.36	2.17	1.95

Different electrode materials were also applied to explore the influence on carrier transport. A high work function metal, Au, was also deposited on the 5 nm Al_2O_3 film grown at 150 °C to form Au/ Al_2O_3 /n⁺-Si devices. Figure 5(a) shows the breakdown tests of devices with two different electrodes. When a metal electrode with high work function was used, the tunneling current could be suppressed significantly. The barrier heights obtained from the F-N fitting results are 2.36 eV

and 2.67 eV for Al and Au devices, respectively, as shown in Fig. 5(b). Metal work functions on high- κ materials have been observed to differ from the corresponding values in vacuum or on SiO₂ [26]. The work functions are 3.9 eV and 5.1 eV for Al and Au contacting with Al₂O₃, respectively [26]. The little difference of barrier height may be caused by the influence of different metal work functions on band warping.

FIG. 5. (a) Breakdown tests of 5 nm Al₂O₃ devices with different electrodes. (b) Linear fittings for currents by the F-N tunneling model.

To confirm the validity of our J - E fitting results, the 10 nm Al₂O₃ sample grown at 150 °C was subjected to XPS measurement using ESCALAB 250 equipped with an Al K α source. Al 2p, O 1s, and O 1s energy loss spectra and the valence band spectrum were measured and discussed. All the XPS spectra were calibrated with C 1s peak of 284.8 eV. ΔE_C and valence band offset (ΔE_V) between the Al₂O₃ film and n-Si substrate were obtained based on XPS measurements. The chemical constituents of Al₂O₃ film grown by ALD were also determined by the XPS results.

Figures 6(a) and 6(b) show the XPS spectra of Al 2p and O 1s core level data of the Al₂O₃ film grown at 150 °C. The peaks could be fitted as symmetric single peaks at 74.7 eV for Al 2p and 531.6 eV for O 1s, respectively. An excellent Al/O composition ratio of the sample is 2/3, which is

identical to the theoretical value. The E_g value of 6.48 eV for the Al_2O_3 film was obtained from the energy loss spectra shown in Fig. 6(c). The valence band of Al_2O_3 and Si substrate was determined by measuring the valence band spectra of the Al_2O_3 film and the n-Si substrate, as demonstrated in Fig. 6(d). The ΔE_V value of 3.30 eV was obtained between Al_2O_3 and n-Si. The band gap value of Si is known to be 1.12 eV. Thus, the ΔE_C value can be easily derived by the equation

$$\Delta E_C = E_g - \Delta E_V - 1.12 \text{ (eV)}. \quad (8)$$

Thus, ΔE_C between Al_2O_3 and n-Si was calculated to be 2.06 eV. The barrier height (ΔE_C) obtained from the J - E fitting curves was 1.95 eV for 10 nm Al_2O_3 , which is well in agreement with the value obtained from the XPS result. The effective barrier fitted by F-N tunneling mechanism is a little lower than that from the XPS analysis, which is due to the combined effects of field and the image force [23, 27].

FIG. 6. XPS spectra for (a) Al 2p core level and (b) O 1s core level from the Al_2O_3 film. (c) O 1s energy loss

spectra. (d) XPS valence band spectra taken from the Al_2O_3 film and n-Si substrate.

4. Conclusion

In summary, the robustness and the carrier transport mechanism of different Al₂O₃ films grown by atomic-layer deposition at different temperature were systematically studied through an Al/Al₂O₃/n⁺-Si capacitor structure. The carriers transport through the Al₂O₃ layer derived from fitting *J-E* curves was found to be governed by the Fowler–Nordheim tunneling mechanism, which was confirmed by X-ray photoelectron spectroscopy measurements. The results indicate that both the growth temperature and film thickness influenced the carrier transport and barrier height. The ultrathin 3 nm Al₂O₃ film fabricated at 150 °C shows excellent dielectric properties, a low tunneling current, and a high breakdown voltage, which is promising for the tunneling layer in the fabrication of flash memory and/or 3D-NAND devices.

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