Using Anodization to Oxidize Ultrathin Aluminum Film for High-*k* Gate Dielectric Application

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Very low cost room-temperature process to fabricate ultrathin Al₂O₃ high-*k* gate dielectrics with equivalent oxide thickness of 10 to 35 Å was studied in this work. Anodic oxidation (anodization) in deionized water followed with rapid thermal annealing was used to oxidize ultrathin aluminum film. Unlike thermal oxidation, anodic Al₂O₃ possesses a speciality of self-limited oxide thickness. This work also demonstrated that the anodic Al₂O₃ exhibits gate dielectric quality characteristics, including low gate leakage current, low interface states, and low bulk trap density. The dielectric constant of anodic Al₂O₃ is estimated to be 9.7. In addition, the gate current density, *J*_g, is almost independent of the measurement temperature, revealing a low trap density and high thermal stability. These good insulator characteristics are believed to be related to the leakage path self-readjustment nature under electric field influence in liquid during anodization.

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For the silicon complementary metal oxide semiconductor (CMOS) scaling beyond 100 nm technology node, the exponential increase in tunneling current with decreasing film thickness becomes an important factor for limiting the reduction of oxide thickness. This thickness-scaling limit is considered to be 12 Å due to high gate leakage and degraded transistor performance.¹ Therefore, to keep device area small and prevent high leakage current while maintaining the same gate capacitance, a thicker gate dielectric with *k* > 3.9 is necessary for the advanced technology node. Several high-*k* materials including Al₂O₃, Si₃N₄, ZrO₂ and HfO₂ had been proposed to provide a substantial thicker dielectric film for reduced leakage current and improved gate capacitance.²⁻⁴

However, for most high-*k* materials, higher dielectric constant comes at the expense of narrower bandgap, which can itself result in leakage current. It is noted that due to directly abutting the transistor channel, high-*k* gate dielectrics must be thermally stable during subsequent high temperature process if they are incorporated into a conventional CMOS process flow. Among the existing high-*k* materials, Al₂O₃ is a strong candidate, since it has a bandgap of ~9 eV similar to SiO₂,⁵ and is an inert high temperature material which is expected to withstand silicon processing conditions. Many methods had been proposed to prepare the Al₂O₃ films, such as thermal oxidation under O₂ ambient, atomic layer deposition (ALD) and sputter deposition-based techniques.^{2,5-7} Generally, a substantial interfacial silicon oxide layer was observed during high temperature procedure due to the rapid oxygen diffusion through thin Al₂O₃.⁸ Although it is well known that SiO₂ has an excellent interface quality with Si, an uncontrolled amount of SiO₂ formation will decrease the capacitance of the dielectric, which can be detrimental to the higher *k* value. We believe that if an ultrathin SiO₂ layer could be formed between Al₂O₃ and Si during process, the interface quality could be comparable to that of SiO₂/Si or the interface between SiO₂-based gate dielectric and Si. This would avoid the need of additional interface engineering, so the higher dielectric constant value could be fully utilized.

In this work, we present a liquid phase oxidation method to oxidize the Al film. Room temperature anodization in deionized (DI) water is used to oxidize Al then rapid thermal annealing (RTA) follows for the preparation of the high-*k* gate dielectric. According to the existing literature,^{9,10} anodic SiO₂ exhibits excellent electrical characteristics such as lower leakage current and higher breakdown field endurance than conventional thermal SiO₂. With the additional merits of low thermal budget and low cost, anodic oxidation is attractive in high-density integrated circuit application. Also, an ap-

propriate annealing is efficient in controlling the electrical properties of anodic oxide.⁹ The results, show that the anodic Al₂O₃ shows gate dielectric quality characteristics of low leakage current, low interface states, and low bulk trap density. This is of interest for the high-*k* gate dielectrics preparation by using the technique in this work.

Experimental

Prior to anodization, 3 in. p-type Si wafers were treated with standard RCA cleaning. A final HF dip procedure was adopted to remove the native oxide. 1%-Si-doped Al was then evaporated on the Si wafer with different thicknesses from 7 to 43 Å.

In the following anodization procedure, wafer with Al film act as an anode electrode. Constant electrolyzing field of 10 V/cm was applied from 5 to 30 min separately at room temperature by using DI water as electrolyte. After anodization, postanodization annealing (PAA) with RTA system was treated in N₂ ambient from 500 to 900°C. All the samples were examined by high resolution Auger electron spectrometry (AES; Physical Electronics, AES PHI670xi), X-ray photoelectron spectroscopy (XPS; Physical Electronics, ESCA PHI 1600), and ellipsometry to characterize the depth profile, near-surface region bonding condition, and the optical (physical) thickness, respectively.

After the aluminum oxide dielectric was formed, standard lithography and etching processes were utilized to fabricate MOS capacitor structure devices with Al metal gate area of 150×150 μm. In addition, conventional SiO₂ dielectrics prepared by rapid thermal oxidation (RTO) system were also fabricated at the same time for comparison. After capacitor devices were implemented, capacitance-voltage (C-V) and current density-voltage (J-V) measurements were used to analyze the electrical characteristics. Also, the equivalent oxide thickness (EOT) was obtained from two high-frequency (100 KHz and 1 MHz) C-V measurements in strong accumulation region using a dielectric constant of 3.9, considering the series resistance and quantum mechanical effect.^{11,12}

Results and Discussion

Depth profile and near-surface region bonding analysis.—After 43 Å aluminum was anodized under a constant field of 10 V/cm for 20 min, the depth profile of anodic Al₂O₃ before and after 900°C PAA was inspected by AES with a very low sputter rate as shown in Fig. 1. It is observed that the anodization could oxidize the Al into a very uniform Al₂O₃ at room temperature. After PAA, the Al distribution was not significantly different from that before annealing, which reveals that the anodic Al₂O₃ is quite thermal stable under 900°C annealing ambient. Also, a very thin SiO₂ layer, which is estimated as thinner than 5 Å, was observed in the Al₂O₃/Si inter-

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Figure 1. AES depth profile of O, Al, and Si relative atomic concentration for one anodic Al_2O_3 sample before and after PAA.

face after N_2 annealing. We believe that hydrated alumina ($\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$) and $\text{Al}(\text{OH})_3$ could coexist in the anodic Al_2O_3 after anodization. While PAA was treated to evolve the hydrogen from the anodic Al_2O_3 , the excess oxygen interacted with the bulk silicon at the same time. Thus, an ultrathin SiO_2 interfacial layer could be self-formed between Al_2O_3 and Si during high temperature annealing. This interfacial SiO_2 is helpful to obtain a satisfactory interface for device application.

The near-surface region bonding condition was characterized by *ex situ* XPS using monochromatic $\text{Mg K}\alpha$ radiation. Figure 2a and b show the Al 2p XPS spectra of original evaporated Al and anodic Al_2O_3 , respectively. In Fig. 2a, it is seen that the Al 2p bondings of metallic-state Al (Al^0) and Al_2O_3 (Al^{3+}) coexist on the evaporated Al surface, which indicates the easily formed aluminum native oxide. After 10 min anodization, no signal from metallic Al is observed in Fig. 2b. The binding energy is 75.9 eV, which implies that the metallic Al was almost converted into stoichiometric Al_2O_3 after anodization.¹³

In Fig. 3, the evolution of Si 2p spectra varied with the anodization time was shown to observe the interfacial transformation. For the silicon oxide, four different bonding configurations were admit-

Figure 2. XPS Al 2p spectra for Al films (a) before and (b) after 10 min anodization. Al was almost converted into Al_2O_3 after anodization.

Figure 3. Evolution of XPS Si 2p spectra for the evaporated Al films anodized after 10, 20, and 30 min. The underneath Si signals demonstrate the relative SiO_2 intensity increases with the anodization time. The inset depicts the structure after anodization.

ted, *i.e.*, Si^{1+} , Si^{2+} , Si^{3+} , and Si^{4+} , which have distinguishable energy shifts to the bulk silicon, *i.e.*, 1.0, 1.8, 2.7, and 3.5 eV, respectively.¹⁴ After 10 min anodization, the Si^{2+} spectrum with a peak around 101.6 eV can be attributed to the mixture of Si and Al_2O_3 at interface. We may suppose this mixture layer is composed of silicon oxide and silicon suboxide. While the anodization time increased, we can see the Si^{2+} signal disappeared and transferred to Si^{4+} (SiO_2). From the AES and XPS analyses, we can see that this interfacial SiO_2 is grown during anodization and annealing procedures. Therefore, controlling the anodization and annealing processes, would eliminate the need of additional interface engineering. The schematic structure of anodic Al_2O_3 is shown in the inset of Fig. 3.

Behavior of Al_2O_3 anodization.—From the recent reports,^{2,8,13} the bottom silicon oxide beneath thin Al_2O_3 could be easily formed during high temperature oxidation process. Any annealing treatment with excess oxygen present will lead to rapid oxygen diffusion through the oxides, and therefore resulting in an SiO_2 or SiO_2 -containing interface. The SiO_2 /Si interface is well known to possess high quality characteristics, *i.e.*, low interface state density $\sim 10^{10} \text{ cm}^{-2} \text{ eV}^{-1}$, and low trap density. Nevertheless, a thick interfacial SiO_2 can seriously limit the EOT scaling under 12 Å. Therefore, it is worth studying how to preserve the critical high quality nature of the SiO_2 interface without sacrificing the higher- k merit.

In Fig. 4, EOT of anodic Al_2O_3 vs. anodization time is shown. It is observed that the Al film can be rapidly oxidized into Al_2O_3 and subsequently self-limited to a saturated oxide thickness, especially when the evaporated Al thickness is thicker than 30 Å. We can attribute this result to the electrical field limitation. After Al had been converted into Al_2O_3 , most voltage between electrodes would drop over Al_2O_3 due to its higher resistance, which could lead rapidly to a reduced oxidation rate. This is verified by extracting the resistances of DI water and Al_2O_3 layer from our anodization system and C-V measurement. The resistance of DI water is calculated to be $\sim 10^4 \Omega$ under 10 V/cm constant field anodization, and the Al_2O_3 (40 Å) layer extracted from two frequency C-V measurements is about $\sim 10^6 \Omega$.¹¹ Based on this characteristic, it may be possible, if an appropriate anodization field is selected, to precisely control the ultimate interfacial oxide thickness without sacrificing the higher- k value.

In order to find out the dielectric constant of anodic Al_2O_3 , the equations shown below are utilized to determine the k value

Figure 4. EOT of anodic Al_2O_3 vs. anodization time. This chart shows a self-limited oxide thickness characteristic during anodization.

$$\varepsilon_{\text{ox}}/\text{EOT} = \varepsilon_{\text{Al}_2\text{O}_3}/T_{\text{Al}_2\text{O}_3} \quad [1]$$

and

$$\Delta\text{EOT}/\Delta T_{\text{Al}_2\text{O}_3} = \varepsilon_{\text{ox}}/\varepsilon_{\text{Al}_2\text{O}_3} \quad [2]$$

where EOT is the equivalent oxide thickness with the consideration of quantum mechanical effect, $T_{\text{Al}_2\text{O}_3}$ is the thickness of Al_2O_3 that is obtained from ellipsometer with $n = 1.66$, $\varepsilon_{\text{ox}} = 3.9\varepsilon_0$ is the dielectric constant of SiO_2 , and $\varepsilon_{\text{Al}_2\text{O}_3}$ is the dielectric constant of Al_2O_3 .

In Fig. 5, EOT vs. Al_2O_3 optical thickness is plotted to determine the dielectric constant of anodic Al_2O_3 . From the slope, one obtains a dielectric constant of anodic Al_2O_3 of $\varepsilon_{\text{Al}_2\text{O}_3} \sim 9.7\varepsilon_0$.

Electrical characteristics of anodic Al_2O_3 .—Figure 6a and b shows the C-V curves of anodic Al_2O_3 before and after N_2 RTA annealing, i.e., PAA, respectively. In Fig. 6a, the C-V hysteresis loop is apparent ($\Delta V_{\text{fb}} \sim 0.5$ V). From the inserted figure, one can also observe the C-V curve appears a stretched out phenomenon, which

Figure 5. EOT vs. optical thickness of Al_2O_3 . The slope yield a dielectric constant of $k \sim 9.7$.

Figure 6. 1 MHz C-V curves for anodic Al_2O_3 (a) before and (b) after 900°C N_2 RTA. The simulated C-V curves are shown in the inserted figures for comparison. In (a), the C-V curve shows apparent hysteresis and stretch out phenomenon.

reveals noticeable trapped charges in the dielectric before annealing.¹⁵ After the anodic Al_2O_3 films were treated with 900°C N_2 RTA annealing, the hysteresis loop almost disappears, as shown in Fig. 6b. Also, the C-V curve fits the theoretical 25 Å oxide C-V curve very well,¹² indicating an excellent gate dielectric quality film. In addition, the capacitance is obviously increased after annealing, which implies a denser dielectric formed after PAA.

It is known that the trapping of positive charges under a constant current stress will result in a reduction in gate voltage, while that of negative charges will result in an increase in gate voltage. Figure 7 shows the variation of V_g under a -1 A/cm² constant current stress. It shows a very different trapping mechanism. The anodic Al_2O_3 film without annealing contains a great amount of hole and electron traps with respect to the annealed one, the latter exhibits fewer net electron traps. High frequency C-V curves with 100 KHz and 1 MHz measurements are shown in the inset of Fig. 7. The capacitance variation in the accumulation region denotes the series resistance effect, which exists in the device structure and the measurement system.¹¹ Furthermore, we can observe the depletion and inversion region curves are almost independent of the frequency. This implies that the interface state density is very low, because a higher interface state density makes the C-V curve response as frequency going down. Many works have shown that the OH^- related chemical species involved in the SiO_2 could act as trapping centers and degrade the device performance due to high trap-assisted current and poor interface quality.^{16,17} This phenomenon is also found

Figure 7. Variations of $\Delta V_g/V_g$ vs. stress time under a constant current density of -1 mA/cm^2 for one sample before and after PAA. Two frequency C-V curves with 100 KHz and 1 MHz are shown in the inset.

in as-anodized Al_2O_3 . High temperature PAA is necessary after anodization because it can effectively evolve the hydrogen from the anodic Al_2O_3 , suppress the trap center generation, and make it a denser dielectric.

From the previous discussion, anodic Al_2O_3 without PAA showed a poor interface quality, *i.e.*, too many interface states and trap densities. PAA was carried out to improve the interface quality. J-V plots of original native oxide and anodic Al_2O_3 with and without PAA treatment are shown in Fig. 8. The native Al_2O_3 film shows a very poor insulator characteristic. After anodization, the J-V curve presents a two orders reduction in current level than native oxide at -2 V , but it is still very leaky. However after the subsequent PAA treatment, oxide resistance shows a drastic improvement, the current level was reduced for four orders of magnitude further. It is suggested that the oxidant, *i.e.*, OH^- , could drift through the anodic Al_2O_3 and then react with underlying metallic Al by breaking the Al-Al or Al-Si bonds during anodization.¹³ The drift paths could be consequently formed after anodization. After PAA treatment, the drift paths could be repaired in a high temperature ambient. From previous discussions, we can notice that PAA procedure plays an

Figure 8. Evolution of J-V plots of samples before anodization (native Al_2O_3), after anodization (ANO), and then after annealing (ANO+PAA).

indispensable role to strengthen the anodic Al_2O_3 bonding network and make it a dense insulator film.

Except the gate leakage current, interface quality is an important issue of high- k gate dielectric to substitute SiO_2 . In Fig. 9, high frequency C-V curves with superimposed ac signals of 1 MHz, 100 KHz, 10 KHz, and 1 KHz are employed to analyze the response of interface states. The raised capacitance with decreasing frequency in depletion region can attribute to the response of interface states. As can be seen, the stretch-out phenomenon is obviously reduced with the increased PAA temperature. (Fig. 9a-d). When annealing temperature was raised to 700°C , the C-V stretch-out phenomenon is much improved as shown in Fig. 9c. We believe that the OH^- related trap can be removed after PAA and the interfacial SiO_2 grow at the same time when temperature was raised to 700°C . However, the interface states induced C-V distortion in depletion region is still apparent. When the PAA temperature was raised to 900°C , the interface states response shows a very slight dependence on frequency, revealing a low interface states density. From the discussion, we can see a higher temperature annealing is critical to the interface quality of anodic Al_2O_3 .

Gate leakage current density of anodic Al_2O_3 and RTO SiO_2 as a function of EOT ranging from 10 to $35 \text{ \AA}$ (gate bias at $V_g - V_{fb} = -1 \text{ V}$) is shown in Fig. 10. The anodic Al_2O_3 shows about 1.5 orders reduced leakage current with respect to SiO_2 under the same EOT. Besides, J-V plots of anodic Al_2O_3 and SiO_2 with EOTs of 20 and $25 \text{ \AA}$ are shown in the inset of Fig. 10. The anodic Al_2O_3 with an EOT of about $10 \text{ \AA}$ is also shown for comparison. The anodic Al_2O_3 exhibits a lower leakage current and higher breakdown voltage than conventional SiO_2 that was mainly attributed to the thicker physical thickness of Al_2O_3 under the same EOT. This result reveals the room temperature anodization process followed by PAA could be utilized to obtain a desirable gate quality Al_2O_3 dielectric.

Figure 11 shows the comparison of time-zero-dielectric-breakdown (TZDB) characteristics of SiO_2 and anodic Al_2O_3 samples with an EOT of $25 \text{ \AA}$. Anodic Al_2O_3 samples can suffer $\sim 3 \text{ MV/cm}$ larger field to breakdown than the SiO_2 samples. In addition, it presents a more uniform distribution than thermally grown SiO_2 . It is known that fewer pinholes can induce a better insulator characteristic and higher breakdown field. The interfacial oxide grown during anodization and PAA exhibits a more uniform electrical characteristic due to the self-adjustment nature of the current leakage path and high temperature repairing of unsatisfied bonding. A more uniform oxide exhibits less local thinning and therefore a higher breakdown field.

Figure 12 shows the characteristics of temperature-dependent reliability of J-V curves measured from 25 to 100°C . When the device was positively biased at inversion region, *i.e.*, substrate injection region, the saturated leakage current level varied with the measurement temperature. From recent research,¹⁸ the main composition of saturated current was generated from the penetration of minority carriers via the interface states. While the minority carriers were not sufficient for supporting the tunneling current, the current could be saturated in a specific level. The specific saturated level is strongly temperature dependent since the intrinsic carrier concentration is proportional to the substrate temperature.¹⁹ Therefore, the current level increased with the measurement temperature.

In the other aspect, when the device was negatively biased at accumulation region, *i.e.*, gate injection region, the conduction characteristics had a very weak temperature dependency. Unlike Fowler-Nordheim (F-N) tunneling in SiO_2 , the dominant conduction mechanism in thermally oxidized Al_2O_3 was reported to be Frankel-Poole (F-P) emission,² in which the conduction mechanism is based on the capture and emission of carriers from traps in the bulk. In F-P conduction, the trap-assisted tunneling current is strongly dependent on the measurement temperature.^{2,20} However, in this work, the anodic Al_2O_3 shows that the current is almost independent of the

Figure 9. 1 M, 100 k, 10 k, 1 k Hz high frequency C-V curves of anodic Al_2O_3 samples after (a) 500°C, (b) 600°C, (c) 700°C, (d) 900°C PAA. Bulk traps and interface states densities decrease with annealing temperature.

measurement temperature as shown in Fig. 13. In addition, by observing the inset in Fig. 13, the J-V curve of anodic Al_2O_3 is well fitted by the F-N plot in high field region. It is believed that the lack of traps in the anodic Al_2O_3 could be responsible for the tendency of F-N like tunneling of the Al_2O_3 dielectric in this work.

Figure 10. Gate leakage current densities vs. EOT of anodic Al_2O_3 and conventional SiO_2 samples with a gate bias of $V_g - V_{fb} = -1$ V. Also, J-V plots of samples with EOTs of 10, 20, and 25 Å are shown in the inset.

Conclusion

Ultrathin Al_2O_3 high- k gate dielectric preparation by DI water anodization was demonstrated in this work. Unlike thermal oxidation, it was found that anodic Al_2O_3 has the merits of a Si-SiO₂ interface in Fig. 3, low leakage current, and low trap density char-

Figure 11. Weibull plot of TZDB characteristic for anodic Al_2O_3 and SiO_2 samples with an EOT of 25 Å.

Figure 12. J-V plots of anodic Al_2O_3 samples measured at various temperatures from 25 to 100°C.

Figure 13. Gate current density J_g vs. inverse of temperature for an Al_2O_3 sample biased at -2 and -4 V. The F-N plot of this sample is also shown in the inserted figure.

acteristics. It was supposed that the self-readjustment nature of leakage path under electric field accounts for the good insulator characteristics of anodic Al_2O_3 . The proposed technique is simple and cost effective. Also, the observed high- k characteristics of anodic Al_2O_3 are apparent for gate dielectric application. We think it is possible to use this technique to prepare other high- k materials in the near future.

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