Effect of Composition, Interface, and Deposition Sequence on Electrical Properties of Nanolayered Ta$_2$O$_5$-Al$_2$O$_3$ Films Grown on Silicon by Atomic Layer Deposition

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Abstracts
Nanolayered Ta$_2$O$_5$-Al$_2$O$_3$ composite films were grown on n-type silicon by atomic layer deposition (ALD) within the overlapped ALD window of 220–270 °C. Moreover, post-annealing treatment was carried out to eliminate defects and improve film quality. Nanolayered Ta$_2$O$_5$-Al$_2$O$_3$ composite films remain amorphous after 700 °C annealing. The effects of composition, interface, and deposition sequence on electrical properties of Ta$_2$O$_5$-Al$_2$O$_3$ composite films were investigated in detail utilizing MIS devices. The results demonstrate that the formation of Ta$_2$O$_5$-Al$_2$O$_3$ composite films by mixing Al$_2$O$_3$ into Ta$_2$O$_5$ can decrease the leakage current effectively, but it leads to the decrease of the dielectric constant and the enhancement of the hysteresis effect. The interfaces in composite films are not conducive to prevent the leakage current. The deposition sequence of Si/(Al$_2$O$_3$/Ta$_2$O$_5$)$_n$, Al$_2$O$_3$ as the first covering layer, reduces the leakage current and the hysteresis effect effectively. Therefore, the electrical properties of Ta$_2$O$_5$-Al$_2$O$_3$ composite films could be regulated by adjusting components and structures via ALD to acquire relatively great dielectric constants and acceptable leakage currents.

Keywords: Ta$_2$O$_5$, Nanolayered films, Electrical property, Atomic layer deposition, Post-annealing

Background
With the shrinking of the sizes, the limitations of silicon oxide (SiO$_2$) gate dielectric for ultra large-scale integration (ULSI) devices have been reached, hence developing new gate dielectrics for next generation of microelectronic devices has become an urgent task in semiconductor industry [1]. It is required that the leakage current of new gate dielectrics has to be lower than that of the conventional SiO$_2$ under the same equivalent oxide thickness. Therefore, various high-κ dielectric materials have been recommended to replace SiO$_2$ [2, 3].

Recently, alternative metal oxide films have been extensively investigated such as Ta$_2$O$_5$, Al$_2$O$_3$, ZrO$_2$, HfO$_2$, Nb$_2$O$_5$, and TiO$_2$. Among them, tantalum pentoxide (Ta$_2$O$_5$) has been considered as one of the most promising candidates to replace SiO$_2$ due to its relatively high dielectric constant of about 20~60 [4–8]. However, Ta$_2$O$_5$ has noticeable high-field conductivity and cannot prevent carriers leakage due to its small band gap of 4.4 eV, which means this metal oxide cannot be independently used as a dielectric film. Hence, it is necessary to introduce an excellent insulating material to block leakage current [9]. Al$_2$O$_3$ is one of the most investigated materials with large band gap (8.7 eV) and high breakdown electric field [10–13]. To optimize the electrical property of Ta$_2$O$_5$ as gate dielectric, ultrathin Al$_2$O$_3$ can be mixed into Ta$_2$O$_5$ thin films for its current-blocking capability [14–16]. This composite structure is believed to provide a high dielectric constant and an acceptable leakage current by controlling the composition and structure [17–23].
As for film deposition methods, atomic layer deposition (ALD) based on saturated self-limiting surface reactions has become an important film deposition technique in the semiconductor industry. It exhibits many advantages over other deposition routes, such as precise thickness control at atomic layer level, high uniformity over a large area, excellent conformity in many complex nanostructures, and controllable film structure and composition [24–28]. Min-Kyu et al. [29] reported the film deposition of Ta$_2$O$_5$ via thermal and ozone (O$_3$) ALD using pentaethoxytantalum as Ta precursor. Hyunchol et al. [30] reported the growth of the ZrO$_2$/Ta$_2$O$_5$ multi-laminate films by ALD and the relation between their dielectric and chemical properties. Partida-Manzanera et al. [4] reported (Ta$_2$O$_5$)$_x$(Al$_2$O$_3$)$_{1-x}$ thin films deposited by ALD using pentakis(dimethylamino)tantalum as Ta precursor and DI water as oxidizer, and the effects of tantalum doping and annealing on dielectric performance. Nevertheless, the effect of composition, interface, and the deposition sequence in composite thin films on electrical properties of Ta$_2$O$_5$-Al$_2$O$_3$ film deposited by ALD still need to be further illustrated.

In this work, we deposited nanolayered Ta$_2$O$_5$-Al$_2$O$_3$ composite thin films on n-type silicon wafers by ALD technology using pentakis(dimethylamino)tantalum (PDMATa) and trimethylaluminium (TAM) as metal precursors, as well as O$_3$ as an oxidizer. Moreover, post-annealing treatments were carried out to eliminate defects and improve film quality [31]. The electrical properties of films were studied utilizing the MIS device with Ta$_2$O$_5$ as dielectric layer [32]. The effects of film composition, interface, and the deposition sequence on electrical properties of film were investigated in detail by capacitance-voltage and current-voltage measurement.

**Methods**

Nanolayered Ta$_2$O$_5$-Al$_2$O$_3$ composite films were grown onto oriented n-type silicon wafers using an ALD reactor (MNT Ltd.). Trimethylaluminium was held at room temperature and pentakis(dimethylamino)tantalum was heated to 80°C. Ozone as an oxidant was generated from oxygen (99.999% purity) by an ozone generator (Newland Ltd.). High purity nitrogen gas (99.999%) was used as the carrying and purging gas. Moreover, the temperature of the reactor chamber and the delivery lines was remained at 230°C and 120°C, respectively. All the samples were annealed at 700°C for 2 h under nitrogen ambient. The Al electrodes on both sides of the samples were deposited by physical vapor deposition. The samples were annealed at 250°C for 0.5 h to assure reliable ohmic contacts. The samples with varying ratios and varying interface number were prepared by controlling the ALD cycles or sub-layer thickness of Ta$_2$O$_5$ and Al$_2$O$_3$.

The thicknesses and refractive indexes of all samples were measured by an ellipsometer. The crystal structure of the Ta$_2$O$_5$-Al$_2$O$_3$ films was characterized by glancing angle X-ray diffraction (GAXRD) with Cu Kα radiation. Current-voltage (I-V) measurements were carried out by a Keithley 2410 1100 V source measurement unit (Keithley Instruments Inc.) and capacitance-voltage (C-V) measurements were carried out by TH2828 S LCR meter (Tonghui Electronics). All the measurements were completed at room temperature.

**Results and Discussion**

Figure 1a shows the change of deposition rate as a function of deposition temperature. There is an overlap for ALD temperature windows of Ta$_2$O$_5$ and Al$_2$O$_3$. Therefore, Ta$_2$O$_5$-Al$_2$O$_3$ composite films can be deposited within the temperature range of 220~270°C, in which it is controllable to grow uniform and high-quality dielectric films by ALD manner. Moreover, the deposition rates of Ta$_2$O$_5$ and Al$_2$O$_3$ are constant 0.52 Å/cycle and 1.01 Å/cycle in ALD temperature windows, respectively. The deposition rates can be used to design the thickness and component contents of the composite film. Annealing treatment is regarded as a necessary process to eliminate defects and improve film quality [31]. The electrical properties of films were studied utilizing the MIS device with Ta$_2$O$_5$-Al$_2$O$_3$ as dielectric layer [32]. The effects of film composition, interface, and the deposition sequence on electrical properties of film were investigated in detail by capacitance-voltage and current-voltage measurement.

![Fig. 1](image-url) **Fig. 1** a The deposition rate of Ta$_2$O$_5$ and Al$_2$O$_3$ as a function of deposition temperatures. b The XRD patterns of Ta$_2$O$_5$, Al$_2$O$_3$, and Ta$_2$O$_5$-Al$_2$O$_3$ composite films annealed at 700°C.
defects and improve film quality [33]. Figure 1b shows the GAXRD patterns of Ta$_2$O$_5$, Al$_2$O$_3$, and Ta$_2$O$_5$-Al$_2$O$_3$ films annealed at 700 °C. Pure Al$_2$O$_3$ film remained amorphous state after 700 °C annealing. In the pattern of Ta$_2$O$_5$, the strong peaks at 22.8° and 56.8° are indexed to the orthorhombic Ta$_2$O$_5$ (PDF Card 25-0922), and the peaks at 28.5°, 36.9°, and 46.8° are indexed to the hexagonal Ta$_2$O$_5$ (PDF Card 18-1304). However, no diffraction peak was detected in the pattern of Ta$_2$O$_5$-Al$_2$O$_3$ composite films with various composition and interfaces. One possible explanation is that crystallization is inhibited in the ultrathin Ta$_2$O$_5$ sub-layers. The other is that amorphous Al$_2$O$_3$ mixed in the composite film increases the crystallization temperature of Ta$_2$O$_5$ film.

Three series of experiments, as shown in Table 1, were carried out to investigate the effects of component ratio, the number of interface, and deposition sequence on electrical properties. The nanolayered Ta$_2$O$_5$-Al$_2$O$_3$ composite films have a periodic structure consisted of several sub-layered Ta$_2$O$_5$-Al$_2$O$_3$. The electrical properties of composite films were studied utilizing the metal-insulator-semiconductor (MIS) devices, as shown in Fig. 2.

To study the effect of the component ratio in composite films on the electrical properties, in experiment I, the thickness ratios of Ta$_2$O$_5$ to Al$_2$O$_3$ in films varied from 1:0 to 0:1. Figure 3a shows that the curves of current density versus electric field intensity. For pure Al$_2$O$_3$ film, it is difficult to inject current due to its strong insulativity. For pure Ta$_2$O$_5$, it shows obvious leakage current and low breakdown field strength. In Fig. 3b, the current density of pure Ta$_2$O$_5$ (Ta$_2$O$_5$:Al$_2$O$_3$ = 1:0) film at 2 MV/cm is 0.329 A/cm$^2$ due to high-field conductivity and abundant grain boundary as the leakage paths [34]. Then, the current density decreases correspondingly with decreasing the thickness ratios of Ta$_2$O$_5$ to Al$_2$O$_3$ from 1:0 to 0:1, and it finally declines down to 2.62 × 10$^{-5}$ A/cm$^2$.

The results demonstrate that the mixing Al$_2$O$_3$ into Ta$_2$O$_5$ thin film can decrease the leakage current effectively. One reason is Al$_2$O$_3$ with wide band gap has strong insulativity and can act as a barrier layer to prevent leakage current. The other is that the amorphous phase of composite film blocks leakage current path. To calculate the dielectric constants of Ta$_2$O$_5$-Al$_2$O$_3$ composite films, the C-V measurement was carried out at 100 kHz at a ramp rate of 100 mV/s, as shown in Fig. 3c. A low capacitance state is a depletion region in the negative voltage range and a high capacitance state is an accumulation region in the positive voltage range for MIS capacitors. The capacitances decrease with reducing the thickness ratio of Ta$_2$O$_5$ to Al$_2$O$_3$. Moreover, the C-V data of Ta$_2$O$_5$-Al$_2$O$_3$ composite films display significant flat band shifts to more positive voltages and additionally significant hysteresis with

![Fig. 2 Schematic diagram for Ta$_2$O$_5$-Al$_2$O$_3$ composite film and MIS device](image-url)
increasing Al$_2$O$_3$ content ratios. The positive shifts of flat band voltage can be attributed to the negative charges from trapping of electrons as well as fixed charges at the interface or in the film. Hysteresis effect in C-V measurements is normally attributed to charge trapping in the oxide or at the interface, mobile charge, and remnant polarization [35]. In Fig. 3d, the dielectric constant of pure Ta$_2$O$_5$ (Ta$_2$O$_5$:Al$_2$O$_3$ = 0:1) and pure Al$_2$O$_3$ film was calculated at 24.6 and 6.28, respectively. For Ta$_2$O$_5$:Al$_2$O$_3$ composite films, as is expected, the dielectric constants decrease continuously with the increase of Al$_2$O$_3$ content correspondingly.

To explore the effect of interface in composite films on the electrical properties, in experiment II, the number of the interfaces varied from 14 to 100. Figure 4a shows the leakage current behaviors of Ta$_2$O$_5$-Al$_2$O$_3$ composite films with various number of interfaces. It can be found that the interface has smaller effects on leakage current compared to the film component. In Fig. 4b, the current density of Ta$_2$O$_5$:Al$_2$O$_3$ composite films is $7.81 \times 10^{-7}$ A/cm$^2$ when the number of interfaces is 14, and then it increases continuously with increasing the number of interfaces from 14 to 100 at the electric field of 2 MV/cm. These results demonstrate
that interfaces in Ta$_2$O$_5$-Al$_2$O$_3$ films are not conducive to prevent the leakage current. These defects trend to generate at interfaces due to the different ionic radius and valence states for Ta$^{5+}$ and Al$^{3+}$. Moreover, more interfaces mean thinner Ta$_2$O$_5$-Al$_2$O$_3$ sub-layers in fixed-thickness film. The interface defect density will increase with the reduction of film thickness [36], which may cause an increase of leakage current. In addition, the effect of SiO$_2$ interface on the electrical properties of the nanolayered film is relatively minor after 700 °C annealing under N$_2$ ambient. Before ALD processes, the native oxide has been removed by an HF last cleaning step immediately before the deposition. The HF step gives rise to a hydrogen-passivated surface, which becomes the initial state for the ALD process. After the film deposition, the samples were annealed at 700 °C under N$_2$ ambient. The inert gas can prevent the oxidation of Si and the further growth of SiO$_2$ interface. Moreover, the Al$_2$O$_3$ films are not permeable for oxygen diffusion [37]. Al$_2$O$_3$ as a barrier layer in nanolayered Ta$_2$O$_5$-Al$_2$O$_3$ film can suppress oxygen diffusion toward the interface between Si and nanolayered film. Therefore, the effect of the SiO$_2$ interface on the electrical properties of the nanolayered film is limited below 900 °C annealing. However, the SiO$_2$ interface has an effect on the electrical properties of nanolayered Ta$_2$O$_5$-Al$_2$O$_3$ film when the annealing temperature is above 1000 °C. As shown in Fig. 5, the reduction of leakage current and dielectric constant can be attributed to the growth of the SiO$_2$ interface during the annealing processes.

The effect of the deposition sequence on the electrical properties was compared in experiment III. The deposition sequence of composite films on silicon was first Ta$_2$O$_5$ and then Al$_2$O$_3$, which was defined as Si/(Ta$_2$O$_5$/Al$_2$O$_3$)$_n$. Otherwise, it was defined as Si/(Al$_2$O$_3$/Ta$_2$O$_5$)$_n$. Figure 6a, b depicts the leakage current behaviors and the curves of C-V. The current density of Si/(Ta$_2$O$_5$/Al$_2$O$_3$) film is higher than that of Si/(Al$_2$O$_3$/Ta$_2$O$_5$) film at the electric field of 4 MV/cm, and the breakdown field of Si/(Ta$_2$O$_5$/Al$_2$O$_3$) film is obviously weaker than that of Si/(Al$_2$O$_3$/Ta$_2$O$_5$) film. In addition, the hysteresis of the C-V curve for Si/(Ta$_2$O$_5$/Al$_2$O$_3$) film is obviously greater. It is reported that Al$_2$O$_3$ thin film has a low interface trap density [38, 39] and can improve interfacial properties [22]. It can be seen that there are lesser defects at the Si/Al$_2$O$_3$ interface compared to the Si/Ta$_2$O$_5$ interface. Moreover, the Al$_2$O$_3$ films are not permeable for oxygen diffusion. It can act as a barrier layer to cover Si in order to prevent the diffusion of oxygen in film toward Si/Al$_2$O$_3$ interface.
The above results illustrate that film composition, structure, and interface state density act as the key factors to affect the electrical properties. A compromise property was obtained by mixing Al₂O₃ into Ta₂O₅ film. The increase of film crystallinity can not only increase the dielectric constant, but also increase the leakage current due to abundant grain boundary as a leakage path. Moreover, high interface state density should be avoided for the laminated or doped film on account of the negative influence on leakage current. Therefore, the amorphous dielectric film with high dielectric constant, relatively large band gap energy, and low interface state density may be a promising gate dielectric to replace SiO₂. In addition, deposition technology also as a key factor has an important effect on electrical properties of gate dielectric.

Conclusions
Nanolayered Ta₂O₅-Al₂O₃ composite films were grown on n-type silicon by ALD. The overlapped temperature window for Ta₂O₅ and Al₂O₃ is 220~270 °C using pentakis(dimethylamino)tantalum as the Ta precursor and O₃ as the oxidant. Nanolayered Ta₂O₅-Al₂O₃ composite films remain amorphous after annealing treatment at 700 °C. The formation of Ta₂O₅-Al₂O₃ composite films by introducing Al₂O₃ into Ta₂O₅ can decrease the leakage current effectively due to the excellent insulator for amorphous Al₂O₃, but lead to the decrease of the dielectric constant. Moreover, the interfaces in composite films are not conducive to prevent the leakage current. In addition, the deposition sequence of Si/(Al₂O₃/Ta₂O₅)n, Al₂O₃ as the first covering layer, reduces effectively the leakage current and the hysteresis effect due to its thermostability and barrier effect. Therefore, the electrical properties of Ta₂O₅-Al₂O₃ composite films could be regulated by adjusting components and structures via ALD to acquire relatively great dielectric constants and acceptable leakage currents.

Abbreviations
ALD: Atomic layer deposition; C-V: Capacitance-voltage; GAXRD: Glancing angle X-ray diffraction; I-V: Current-voltage; O₃: Ozone; PDMATa: Pentakis(dimethylamino)tantalum; TAM: Trimethylaluminum; ULSI: Ultra large-scale integration

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Availability of Data and Materials
All data are fully available without restriction.

Authors’ Contributions
JL carried out the experiments and measurements. JW was involved in the measurements. JL designed the study and drafted the manuscript. JS supervised the overall study. All authors read and approved the final manuscript.

Competing Interests
The authors declare that they have no competing interests.

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