Resistive Switching Behavior of

Solution-processed AlOx and GO based RRAM at Low Temperature

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**Abstract—**This paperreports on resistive switching behavior observed in resistive random access memory (RRAM) devices fabricated with aluminum oxide (AlOx) and graphene oxide (GO) dielectric films, which were solution-processed under low annealing temperatures of 250°C and 50°C for AlOx and GO dielectric films, respectively. As representative of metal oxide and two-dimensional material, a detailed study and comprehensive comparison in view of resistive switching performance has been conducted for AlOx and GO based RRAM, including operation voltage, resistance distribution, resistance ratio, conduction mechanism and retention/endurance property. A smaller operation voltage and better stability were demonstrated in AlOx based RRAM devices while higher resistance magnitude of high resistance state (HRS) and resistance ratio were observed in GO based RRAM devices. The current study opens up promising applications of environmental-friendly solution-processed AlOx and GO films with lower energy consumption for non-volatile memory (NVM).

**Keywords—** Resistive switching, Aluminum oxide, Graphene oxide, Solution-processed.

# Introduction

There are theoretical limitations observed in conventional memories like relatively slow operation speed [1], lack of flexibility [2] and low scalability [3]. As a potential replacement for next-generation non-volatile memories (NVM), resistive random access memory (RRAM) devices based on resistive switching (RS) behavior have received considerable attention in recent years. In order to reduce power consumption, accelerate data processing and increase storage density, a large variety of solid-state materials have been investigated as storage medium for RRAM device applications, including metal oxides such as NiOx, TiOx and AlOx [4-9], organics [10-12], complex perovskite oxides [13-16] and carbon-based materials [17-19]. In particular, metal oxides and carbon-based materials are currently extensively discussed due to the material simplicity [20-22].

Among various metal oxide based materials, AlOx has attracted considerable attention as dielectric layer with advantages of high dielectric constant (~8), superior elasticity, high toughness and high thermal stability with Si and Pt [23-28], which shows a great potential as RS layer. Apart from the research on metal oxides, carbon-based materials, as an important class of materials anticipated to overcome the technological barriers of conventional semiconductor electronics, have also been explored extensively in recent years [29-31]. Related research on carbon-based materials applied in the field effect transistor (FET) devices [32, 33] indicate that it is highly important to investigate their memory behavior as well. Among carbon-based materials, graphene oxide (GO) has been actively investigated having advantages such as, easy synthesis process, nanometer-level scaling down and compatibility for flexible device applications [34-36]. He *et* al. has been the first to report reliable and reproducible RS performance of GO thin films deposited by vacuum filtration method [37]. Following on from this study, a large amount of publications have reported on high ON/OFF ratio in the GO based RRAM devices [38, 39], revealing the potential of using GO in RRAM devices. Several fabrication methods such as, atomic-layer-deposition (ALD) [22, 40-42], magnetron sputtering [43, 44], vacuum filtration [45, 46] and solution-processed (SP) methods [47, 48] have been reported to achieve good performance of RRAM devices. Compared to traditional methods, the solution-processing such as drop casting [49], dip coating [50] and spin coating [47] have considerable appeal due to advantages of low fabrication cost, easy operation and high fabrication efficiency [47-50].

In this work, the solution-processed dielectrics were fabricated at low annealing temperatures of 250°C for AlOx film and 50°C for GO film, and formed the Al/TiN/AlOx/Pt and Al/GO/Si/Al RRAM devices, respectively. A detailed study of RS performance for Al/TiN/AlOx/Pt and Al/GO/Si/Al RRAM devices have been conducted, including operation voltage/resistance distribution, retention and endurance properties. A smaller operation voltage and better stability were demonstrated in AlOx-based RRAM devices while higher resistance of high resistance state (HRS) and higher resistance ratio were observed in GO-based RRAM devices. In addition, the conduction mechanisms of RRAM devices were studied and found to be trap-assisted space-charge-limited conduction (SCLC) in Al/TiN/AlOx/Pt devices and Frenkel-Poole emission in Al/GO/Si/Al devices.

# Experimental

*Device Fabrication*

**Al/TiN/AlOx/Pt Device.** The Pt (200 nm)/Ti/SiO2/Si substrates as the bottom electrode (BE) were cleaned with an air plasma system (PDC-002 HARRICK PLASMA expanded plasma cleaner). ~18.76 g of aluminum nitrate nonahydrate (Al (NO3)3·9H2O) was dissolved in 20 mL of deionized water to produce an AlOx precursor solution. Before spin-coating, the mixture was stirred vigorously for 30 min under air ambient conditions and filtered by 0.45 μm polyethersulfone (PES) syringe filters. Then the prepared solution was spin-coated at 4500 rpm for 40 s on substrates. After deposition, films were annealed at 250 °C for 60 min under ambient conditions, resulting in the thickness of the AlOx films of around 35 nm, which was measured by Atomic Force Microscope AFM). Finally, a 70 nm Al protection layer coated on TiN thin film (100 nm) with a diameter of 0.1 mm served as the top electrode (TE), which were deposited by E-beam evaporation. The cross-section of finalized device structure is shown in Fig. 1(a).

**Al/GO/Si/Al Device.** The GO was prepared with the modified Hummers method [51, 52]. 10 g graphite powder and 30 g KMnO4 were added and stirred in 120 mL H2SO4 (98 %) for 4 h at 35 °C. Then 250 mL di-ionized (DI) water was added to make the solution diluted and stirred for 4 h. After filtering the solution, the suspension liquid was washed with HCl (4 %) on the filter paper to remove inorganic impurities and further washed with 500 mL DI water to clean the residual acid. Finally, the suspension liquid was dried to produce graphite oxide. 4 mg graphite oxide powder was added to 40 mL ethyl alcohol with 6 h ultrasonic process, which was stirred at 30 min intervals. After the ultrasonic process, the solution was centrifuged at 5000 rpm for 15 minutes with the supernatant collected. The supernatant was spin-coated on the 50 nm Si substrate at a spin rate of 1500 rpm for 40 s, 4 times, and annealed at 50 °C. The thickness of the GO layers was ~40 nm as assessed by AFM. Following GO deposition, 40 nm (nominal) Al thin films with a diameter of 0.1 mm were deposited on the GO layer and also on the reverse side of the Si substrate by e-beam evaporation. The cross-section of the completed device is depicted in Fig. 1(b).

*Electrical Measurements*

The structure of Al/TiN/AlOx/Pt and Al/GO/Si/Al RRAM devices for the current-voltage (I-V) measurements is presented in Figs. 1(a) and (b), respectively. All the resistive switching characteristics including SET/RESET voltage and resistance distribution, retention and endurance properties were tested using a two-probe station with Agilent B1500A high-precision semiconductor analyzer. The DC voltage bias was applied at TE, while the BE was grounded. The wafer prober test was carried out in the dark Faraday Cage and at room temperature.



Figure 1. A schematic of device cross-section and I-V measurement setup of: (a) Al/TiN/AlOx/Pt and (b) Al/GO/Si/Al RRAM devices.

# Results and Discussion

The typical DC sweep response of the Al/TiN/AlOx/Pt and Al/GO/Si/Al RRAM devices with 1 mA and 5 mA complicance currents (CC) are plotted in Fig. 2. Both AlOx and GO samples exhibit a reproducible and reversible resistive switching behavior after a electroforming operation with higher voltage. Bistable switching operation between high resistance state (HRS) and low resistance state (LRS) can be observed from the I-V DC sweep characteristics. The arrows in Fig. 2 indicate the directions of voltage sweep for the SET and the RESET. The SET was observed to have an abrupt rise in current before reaching the CC for both samples. It is noticeable that the current drops gradually in the RESET for AlOx samples while the corresponding RESET was observed to be abrupt for the GO samples. The values for SET voltage (VSET) for AlOx samples are around 1 V while the ones for GO samples are up to 1.8 V. In the RESET process, nearly all RESET voltages (VRESET) are -1 V approximately. For oxide RRAM, it is known that a conducting path composed of oxygen vacancies can be generated inside the dielectric after the forming or SET event [53]. It has also been reported that the concentration of oxygen ions strored in the electrode has an effect on the RESET behavior [54]. The huge amount of oxygen stored in the electrode results in gradual RESET behavior, whereas the instantaneous RESET has been achieved with the electrode having a small number of the oxygen ions [53]. Therefore, compared with GO examples, AlOx samples seem to have a higher number of vacancies in the dielectrics after the SET process, which results in a gradual RESET behaviour. As shown in Figure 2, a gradual reset behavior can be observed from the I-V curves of AlOx example. When the voltage sweeps from 0 to -2 V, the current changes gradually, which is good for controlling the device conductance. It can be inferred that a higher number of oxygen ions were stored in electrode of AlOx example during set phase and hence the filament comprised a larger amount of vacancies which were dissolved gradually [51, 53]. The current controllability of the gradual switching operation in RRAM devices enhances the possibility of using it as an artificial synapse for neuromorphic applications [53]. Furthermore, it can be seen that larger CC leads to lower LRS resistance in both samples, which demonstrate their potential for multi-bit storage in solution processed RRAM devices.



Figure 2. Typical bipolar I-V characteristics for Al/TiN/AlOx/Pt and Al/GO/Si/Al RRAM devices with 1 mA and 5 mA complicance currents.



Figure 3. (a) The variation of the SET/RESET voltage; (b) the distribution of HRS/LRS resistance measured at 0.1 V; (c) the variation of the resistance ratio, for the Al/TiN/AlOx/Pt and Al/GO/Si/Al RRAM devices.

The statistic analysis of DC voltage operation and HRS/LRS resistance based on RS parameters, such as VSET/VRESET, HRS/LRS resistance (RHRS/RLRS, measured at 0.1 V) and resistance ratio were studied as shown in Fig. 3. The data were extracted from 10 continuous I-V cycles of 5 random devices for each sample. A smaller average VSET (1.49 V) and a smaller average VRESET (-1.14 V) for AlOx samples can be seen in Fig. 3(a). The corresponding VSET and VRESET values for GO samples are 1.71 V and -1.65 V, respectively. Furthermore, the distribution of RESET voltage is found to be sharper than that for SET voltage for both samples. As reported by Pan *et* al. [55], the formation or dissolution of a conductive filament (CF) in the oxide layer leads to RS behavior. The conduction path is formed at the location of the first connected filament while the dissolution only occurs in the exsiting conductive filament. In other words, the randomness of the filament formation is much stronger that that of the dissolution. As a result, conductive filament mechanism can be verified by observing voltage distribution for both samples. The cumulative probabilities of ON and OFF state resistance for the AlOx and GO samples are illustrated in Fig. 3(b). A very small range of LRS resistance level distribution (one order) can be observed while RHRS variations are about two orders of magnitude for both samples. Moreover, the value range of RHRS for GO examples is nearly one order larger on scale than that for AlOx samples. The uniformity of RS parameters plays a significant role in high-density memory applications. It can be seen that the AlOx samples have a more centralized distribution of RHRS. The RHRS for AlOx samples distributes in a range of 2 kΩ-80 kΩ, whereas RHRS for GO examples shows more scattering within a wider range of 8 kΩ-1 MΩ. The resistance values at LRS show the preferable uniformity and both samples have a similar distribution of RLRS. Finally, the variation of the resistance ratio is shown in Fig. 3(c). It can be seen that the magnitude of resistance ratio tends to be larger for GO samples (30~4600) in comparison to that for AlOx samples (4~210). Table 1 summarizes the average values of resistive switching parameters for solution processed AlOx and GO based devices in this work and in the literature [56, 57]. It is notable that the devices in this work have larger resisitance ratios and lower absolute voltage values except for the SET voltage of AlOx based RRAM device.

Table 1. A summary of swithing parameters for solution processed AlOx and GO based RRAM devices in this work compared to literature values

| **Parameters (Average)** | **AlOx based RRAM** | **GO based RRAM** |
| --- | --- | --- |
| ***This work*** | ***[56]*** | ***This work*** | ***[57]*** |
| Vforming (V) | 3.3 | 5.3 | 2.5 | - |
| VSET (V) | 1.4 | 0.83 | 1.64 | -2.6 |
| VRESET (V) | -1.1 | -1.39 | -1.71 | 2.2 |
| HRS (Ω) | 10k | 3k | 82k | 500M |
| RLRS (Ω) | 275 | 152 | 123 | 1M |
| Ratio | 35 | 20 | 713 | 500 |



Figure 4. The curve fitting for I-V characteristics of (a) Al/TiN/AlOx/Pt and (b) Al/GO/Si/Al RRAM devices on a double logarithmic scale. The number represents the straight-line slope in each region. The inset in (b) shows the curve fitting for the typical OFF state I-V characteristic of Al/GO/Si/Al device.

Fig. 4 shows the curve fitting results for both positive and negative bias regions of I-V characteristics on a log-log scale to investigate the current conduction mechanism of the Al/TiN/AlOx/Pt and Al/GO/Si/Al devices. For both devices, there is a linear dependence with a slope closed to 1 in low electric field of OFF state and ON state [58] which satisfies ohmic conduction (I∝V). In the high-voltage range, the current increases non-linearly which implies different conduction mechanism. The OFF state for Al/TiN/AlOx/Pt device follows a trap-controlled space-charge-limited conduction (SCLC) mechanism since the current line slopes are ~1.88 (I∝V2) and 4.86 (I∝V5) at voltages higher than ~0.3 V, respectively. The curve fitting presents clearly three regions on the I-V curves referring to SCLC mechanism, which are Ohmic-like behavior, trap-filled-limited (TFL) behavior and Child’s law with increasing voltage [58]. For Al/GO/Si/Al devices, the current slope in the high-voltage region is only 1.18 which does not follow the SCLC mechanism. The linear curve fitting for Al/GO/Si/Al devices, as shown in the inset, confirmed that the conduction mechanism in OFF state in both positive and negative bias regions under high electric field is Frenkel-Poole emission. Furthermore, the possible switching mechanism for both solution-processed devices is proposed to be based on the conductive filament model. Oxygen vacancies (Vo) and some other initial defects can exist in the fresh devices based on the solution-processed films. Under the applied voltages, the conduction paths are generated in the dielectric layer during the SET process. The CFs start to be generated around the initial Vo and can be further generated with the increased temperature and electric field. A large number of oxygen ions (O2-) within the dielectric layer is absorbed by TE. More and more oxygen vacancies are left when O2- ions drift from the dielectric film towards TE as driven by the electrical force (O 🡪 Vo + O2-). At the same time, the whole defect distribution is modified as well. Then the electrons can be transported through the CFs composed of oxygen vacancies and other defects between the two electrodes and low resistance state is achieved.

 

Figure 5. (a) Retention and (b) endurance performance for TiN/AlOx/Pt and Al/GO/Si/Al RRAM devices measured at room temperature.

To investigate the reliability of the solution processed RRAM devices, Fig. 5 presents the retention and endurance properties of the Al/TiN/AlOx/Pt and Al/GO/Si/Al devices at HRS and LRS, respectively. Although there is a negligible resistance fluctuation of both ON and OFF states after 103 s, each level state can still be sustained over 104 s without significant degradation of the memory window at room temperature for both devices (see Fig. 5(a)). According to Fig. 5(b), the endurance property is evident from the stable resistance ratios for more than 100 cycles. Compared with the Al/TiN/AlOx/Pt devices, the Al/GO/Si/Al devices show much higher HRS resistance and larger resistance ratio (>100). On the other side, the Al/TiN/AlOx/Pt devices show a more uniform resistance distribution.

# Conclusion

In conclusion, an analysis in light of the switching behavior has been conducted on the solution-processed AlOx and GO dielectrics in RRAM devices prepared under low temperatures. The Al/TiN/AlOx/Pt samples exhibited smaller SET/RESET voltages, HRS resistance and resistance ratio in comparison to Al/GO/Si/Al samples. The conduction mechanisms in the ON and OFF states of the Al/TiN/AlOx/Pt and Al/GO/Si/Al RRAM devices are found to be the SCLC mechanism and Frenkel-Poole emission, respectively. The high ON/OFF ratio, low operation voltage, long retention time (104 s) and above 100 endurance cycles present the enhancement of electrical properties and stable, reliable operation of the devices. Moreover, the low temperature utilised for the fabrication of oxide films means low energy consumption. The results show promise and point to the environmentally-friendly low-temperature solution-based fabrication method as the contender for applications in the flexible memory.

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