Highly-stable write-once-read-many-times switching behaviors of 1D–1R memristive devices based on graphene quantum dot nanocomposites

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One diode and one resistor (1D–1R) memristive devices based on inorganic Schottky diodes and poly(methylsilsesquioxane) (PMSSQ):graphene quantum dot (GQD) hybrid nanocomposites were fabricated to achieve stable memory characteristics. Current-voltage (I-V) curves for the Al/PMSSQ:GQDs/Al/p-Si/Al devices at room temperature exhibited write-once, read-many-times memory (WORM) characteristics with an ON/OFF ratio of as large as 10^4 resulting from the formation of a 1D–1R structure. I-V characteristics of the WORM 1D–1R device demonstrated that the memory and the diode behaviors of the 1D–1R device functioned simultaneously. The retention time of the WORM 1D–1R devices could be maintained at a value larger than 10^4 s under ambient conditions. The operating mechanisms of the devices were analyzed on the basis of the I–V results and with the aid of the energy band diagram.

The enhancement of the electrical characteristics for memristive devices fabricated utilizing hybrid nanocomposites has been intensively investigated owing to their having excellent advantages of low cost, high flexibility, simple fabrication, and low power consumption1–3. When memristive devices are fabricated utilizing inorganic/organic nanocomposites, charge-storage and matrix materials are typically combined by using a spin-coating deposition4. Moreover, metallic Au, Al, Ag, and Cu nanoparticles are extensively used as charge-trapping materials4–6. However, because almost all metallic nanoparticles are not only expensive but also unstable at high temperatures, nanocomposites containing metallic nanoparticles have inherent problems for practical applications in memory devices. Graphene quantum dots (GQDs), which are included in the category of the ultrafine graphene family, have emerged as excellent charge-trapping materials for potential applications in memristive memory devices because of their unique chemical inertness, low toxicity, and large work function7. Furthermore, GQDs with a nanoscale size contain significant edge effects and strong quantum confinement, especially in nanoscale devices8. The crystallographic orientation of the graphene edges significantly influences the electronic properties of the GQDs, including a Coulomb blockade and a mobility gap9. In addition, poly(methylsilsesquioxane) (PMSSQ) materials have attracted much attention due to their superior physical properties of low-dielectric constant, low moisture absorption, excellent thermal stability, excellent mechanical hardness, and simple synthesis10–12. Therefore, hybrid nanocomposites based on GQDs with a remarkable charge-storage capability embedded in a polymer layer with a low-dielectric constant are very effective in serving as the active layer in memristive devices13.

Cross-talk interference between memristive cells can originate from leakage current paths through neighboring cells in cross-bar array or an excess of current, may cause electrical misreading of the device14–17. This phenomenon in memristive devices disturbs the reading process of the selected cell and must be eliminated before such devices can be used for practical applications. An effective method for eliminating the cross-talk is to connect a rectifying diode or selector to each cell18,19. Recently, a write-once, read-many-times (WORM) memory with a diode, in which undesired cross-talk was prevented, was demonstrated20. The architecture of one diode and...
one resistor (1D–1R) can improve reading accessibility in an integrated memory array structure\textsuperscript{21–23}. The 1D–1R architecture is preferred in terms of integration because it occupies less area. Furthermore, the design and the fabrication of 1D–1R devices are relatively very simple.

**Methods**

The WORM 1D–1R devices contain an active layer of GQDs embedded in a PMSSQ layer on a p-Si/Al Schottky diode. Firstly, The PMSSQ was prepared first by mixing de-ionized (DI) water, n-butanol (Aldrich), and trimethoxymethylsilane [CH$_3$Si(OCH$_3$)$_3$] (Aldrich) in a weight ratio of 1:10:4. The mixture underwent ultrasonic processing for 24 h at 60°C. Then, the GQD solution (ACS MATERIAL) was added to the PMSSQ solution in a weight ratio of 0, 10, or 20%, followed by an ultrasonic process for 2 h at room temperature\textsuperscript{25}. The p-type (100) Si substrates (containing native oxide layers) with a resistivity of 1–10 $\Omega$ cm were cleaned ultrasonically in acetone, methanol, and de-ionized (DI) water for 30 min each. After the chemically-cleaned p-Si substrates had been dried by using N$_2$ gas with a purity of 99.99%, middle Al electrodes, each with a thickness of 70 nm, were thermally evaporated at a pressure of $1 \times 10^{-6}$ Torr. Then, the memristive devices with GQDs embedded in an insulating PMSSQ layer were fabricated on Al/p-Si substrate. The PMSSQ:GQDs thin layers were formed on Al/p-Si substrates by using a spin-coating method with spin-coating speeds of 500 rpm for 3 s, 1000 rpm for 5 s, 3000 rpm for 30 s, 1000 rpm for 5 s, and 500 rpm for 3 s in series at room temperature. Then, the devices were annealed at 140°C for 1 h. The top Al electrodes, each with a thickness of 180 nm and a diameter of 1 mm, were deposited on the PMSSQ:GQDs layer by using thermal evaporation through a metal mask at a system pressure of $1 \times 10^{-6}$ Torr. Finally, to fabricate the p-Si/Al Schottky diodes, we deposited bottom Al electrodes, each with a thickness of 200 nm, on the back side of the Si substrate by using thermal evaporation at a system pressure of $1 \times 10^{-6}$ Torr.

The structural properties of the Al/PMSSQ:GQDs/Al/p-Si/Al devices were characterized by using scanning electron microscopy (SEM, NOVA NANO SEM 450). All electrical measurements on the devices were performed by using a semiconductor characterization system (Keithley 2400) at 300 K. The bottom Al electrode was grounded during the memory-effect measurements. When the performances of the resistive memory were measured, the voltage was applied to the top Al electrode and the middle Al electrode, when those of the diode were measured, the voltage was applied to the middle Al electrode and the bottom Al electrode, and when those of the 1D–1R device were measured, the voltage was applied to the top Al electrode and the bottom Al electrode.

**Results and Discussion**

Figure 1 shows a (a) schematic diagram and a (b) cross-sectional SEM image of the 1D–1R device with an Al/PMSSQ:GQDs/Al/p-Si/Al structure. The structure of the device consisted of a PMSSQ active layer containing GQDs and a Al/p-Si layer, with those two layers separating the top and the bottom Al electrodes. The thicknesses of the top Al electrode, the PMSSQ:GQDs nanocomposite film, and the Al layer on the p-Si were approximately 176, 315, and 72 nm, respectively. Figure 1(c) demonstrates that the PMSSQ:GQDs thin film exhibits a bright blue emission under 254-nm UV illumination. Figure 1(d) presents the photoluminescence spectra from the PMSSQ and the PMSSQ:GQDs thin films under excitation by 300-nm light; the results are indicative of the existence of GQDs in the deposited film.

Figure 2 shows I-V curves for the PMSSQ resistive memory devices with GQD (Al/PMSSQ:GQDs/Al) concentrations of (a) 0, (b) 10, and (c) 20%. The bottom Al electrode was grounded, and the voltage was swept from $-5$ to $15$ V; thus, both charge trapping and de-trapping currents occurred. Figure 2(a) shows the I-V curves for the devices with only a PMSSQ active layer. Even though the devices with GQD concentrations of 0% showed slight memory characteristics, their electrical characteristics were unstable. The I-V curves for the Al/PMSSQ:GQDs/Al devices with a GQD concentration of 10% demonstrated bipolar characteristics, as shown in Fig. 2(b). When the voltages applied to the device were varied from 0 $\rightarrow$ 2.1 (OFF) $\rightarrow$ 5 (ON) $\rightarrow$ 2.8 (ON) $\rightarrow$ 5 (OFF), the corresponding currents of the ON and the OFF states at 1 V were $2.29 \times 10^{-2}$ and $3.48 \times 10^{-6}$ A, respectively. Figure 2(c) presents the I-V curves for the devices containing an active layer with GQDs embedded in a PMSSQ layer at a GQD concentration or 20%. The hysteresis behaviors observed in the I-V curves for those devices, on a log scale, were similar to those of the devices fabricated with a GQD concentration of 10%. The ON and the OFF currents at 1 V were $2.4 \times 10^{-2}$ and $2.36 \times 10^{-5}$ A, respectively. The ON/OFF ratio of the devices with a GQD concentration of 20% was smaller than that of the devices with a GQD concentration of 10%.

Even though the structure of the device is symmetrical, its electrical behaviors are asymmetric. Because of the fabrication process and post-treatment, the top-Al/PMSSQ interface is actually different from the bottom-Al/PMSSQ interface. For the bottom-Al/PMSSQ interface, the PMSSQ solution is spin-coated onto the Al electrode, followed by annealing at 140°C for 1 h. However, for the top-Al/PMSSQ interface, the Al electrodes are directly deposited on the surface of the PMSSQ. Thus, the two interfaces are different. According to the I-V curves in Fig. 2 and the working mechanism based on charging/discharging of traps, which will be discussed later, electrons seem to be more easily injected into the active layer from the middle Al electrode. For the bottom-Al/PMSSQ interface, the PMSSQ layer was fabricated on Al/p-Si substrate. The PMSSQ:GQDs thin films were formed on Al/p-Si substrates by using a spin-coating method with spin-coating speeds of 500 rpm for 3 s, 1000 rpm for 5 s, 3000 rpm for 30 s, 1000 rpm for 5 s, and 500 rpm for 3 s in series at room temperature. Then, the devices were annealed at 140°C for 1 h. The top Al electrodes, each with a thickness of 180 nm and a diameter of 1 mm, were deposited on the PMSSQ:GQDs layer by using thermal evaporation through a metal mask at a system pressure of $1 \times 10^{-6}$ Torr. Finally, to fabricate the p-Si/Al Schottky diodes, we deposited bottom Al electrodes, each with a thickness of 200 nm, on the back side of the Si substrate by using thermal evaporation at a system pressure of $1 \times 10^{-6}$ Torr.

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annealed at 140 °C for 1 h, which is the reason for an Ohmic junction being formed on the front side of the p-Si substrate.

The I-V curves for the resistive memory devices fabricated utilizing PMSSQ:GQDs nanocomposites exhibited bipolar characteristics, as shown in Fig. 3(b). The electrical characteristics of the devices with a GQD concentration of 10% demonstrated the largest memory margin, as shown in Fig. 2(b). The I-V characteristics of a combined 1D–1R device with a structure of Al/PMSSQ:GQDs/Al/p-Si/Al show that the memory and the diode characteristics of the 1D–1R device functioned simultaneously, as shown in Fig. 3(c). The reversible switching characteristics of unipolar memory devices are attributed to the rectifying properties of the diodes. While the current in the 1D–1R devices dramatically increased at a threshold voltage of 2.2 V in the range of positive sweep voltages, indicative of a transition from the OFF to the ON states, no current flow of any significant size was measured under negative applied voltages. However, the current in the 1D–1R devices under an applied positive voltage was smaller than that of the resistive memory devices with a structure of Al/PMSSQ:GQDs/Al. The ON/OFF current ratios of the resistive memory and the 1D–1R devices at 1 V were 88 and 1.16 × 10², respectively. Because the p-Si/Al diode layer with a large resistivity affects the electrical characteristics of the 1D–1R devices, the decrease in the OFF current of the 1D–1R devices originates from the existence of the p-Si/Al Schottky diode, resulting in the prevention of the crosstalk effect due to the role played by the diode.

Figure 4 shows the I-V curves for the WORM 1D–1R devices during three voltage sweeps. The first and the second sweeps were done in the voltage range between −5 and 5 V. During the first voltage sweep, the devices initially maintained an OFF state at low voltages. When the applied voltage reached 2.2 V, the current rapidly increased from 9.17 × 10⁻⁸ to 1.26 × 10⁻³ A, indicative of a state transition in the memristive devices from an OFF state to an ON state. After the stage transition had occurred, the current in the devices gradually increased from 1.3 × 10⁻³ to 5.5 × 10⁻³ A with increasing applied bias voltage between 2.2 and 5 V. The current ratio between the ON and the OFF states for the 1D–1R devices at a read bias voltage of 1 V was as large as approximately 1.09 × 10⁴, which was large enough to decrease significantly the misreading probability from the memory identification of the devices. After the transition from the OFF to the ON states, the device stayed in the ON state, as shown in the curves for the subsequent voltage sweeps from 5 to 0 V. The decrease in the current of the devices when the voltage was swept from 0 to −5 V could be attributed to the rectifying properties of the p-Si/Al diode. During the second voltage sweep, the devices showed WORM characteristics. The ON state of the devices only appeared in the voltage range between 0 and 5 V. The electrical characteristics of the devices were similar in the negative applied voltage range due to the existence of the p-Si/Al diode, which is the same behavior as was observed in the I-V...
Figure 2. Current-voltage curves for the Al/PMSSQ:GQDs/Al/p-Si/Al devices with GQD concentrations of (a) 0, (b) 10, and (c) 20%.

Figure 3. Current-voltage curves and schematic diagram of electrical contact of the Al/PMSSQ:GQDs/Al/p-Si/Al devices (left insets) of the (a) p-Si/Al Schottky diode, (b) Al/PMSSQ:GQDs/Al device, and (c) Al/PMSSQ:GQDs/Al/p-Si/Al 1D–1R device.

Figure 4. Current-voltage curves of the Al/PMSSQ:GQDs/Al/p-Si/Al device during three voltage sweeps.

curves for the devices during the first voltage sweep. The third sweep was performed in the voltage range between -10 and 10V to observe the electrical characteristics at large applied voltages. The behaviors of the electrical characteristics during the third sweep stage were very similar to those during the first and the second sweeps. No resistive switch phenomena occur. The reader should note that the introduction of a diode to the memory device leads to the write-once limitation. Thus, the proposed 1D–1R memory devices can only be used in an electronic system that requires a WORM memory.
When a reverse voltage bias is applied, the reversed resistance of the diode ($R_{D_R}$) is much larger than the memory in the ON state ($R_{M_ON}$), as shown in the inset of Fig. 6(d). As a result, the actual potential applied to the memristive devices is much smaller than the resetting threshold voltage bias to the memory. For the ON state ($R_{M_OFF}$), as shown in the inset of Fig. 6(b). Thus, the electrons emitted from the p-Si/Al Schottky diode are injected into the LUMO of the PMSSQ via the F-N tunneling process, as shown in Fig. 6(b). Because the GQDs in the PMSSQ act as electron-trapping sites, they capture the electrons emitted from the p-Si/Al. The electrons captured in the GQDs generate a local internal field in the PMSSQ layer, which results in a transition from an OFF to an ON state. When a reverse voltage bias is applied, the reversed resistance of the diode ($R_{D_R}$) is much larger than that of the memory in the ON state ($R_{M_ON}$), as shown in the inset of Fig. 6(d). As a result, the actual potential applied to the memristive devices is much smaller than the resetting threshold voltage bias to the memory.
Thus, the device cannot be permanently switched to an OFF state, as shown in Fig. 6(d). As a result, the 1D–1R device is always in the ON state under the reading voltage.

Conclusion

The I-V curves for the Al/PMSSQ:GQDs/Al devices at room temperature showed that by adjusting the GQD concentration, we were able to maximize the memory margin of the devices at a GQD concentration of 10%, resulting in improved device performances. Using the measured electrical properties of the Al/PMSSQ:GQDs/Al devices, we fabricated WORM 1D–1R devices with an optimized structure of Al/PMSSQ:GQDs/Al/p-Si/Al, i.e., an optimal GQD concentration of 10% and a p-Si/Al Schottky diode, to prevent cross-talk in the Al/PMSSQ:GQDs/Al/p-Si/Al devices. The I-V curves for the WORM 1D–1R devices at room temperature demonstrated memory and rectification characteristics. The results of the retention measurements for the WORM 1D–1R devices demonstrated that they exhibited stable memory performance with retention times larger than 10^4 s without any significant electrical degradation.

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Author Contributions
S.S. and T.W.K. conceived the project, and S.S. designed and performed the experiments and collected the data. S.S., C.W., H.S.J., and T.W.K. analyzed and discussed the data. All authors discussed the results and contributed to the writing of the manuscript.

Additional Information
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