Nonvolatile floating gate memory characteristics of Sb$_2$Te–SiO$_2$ nanocomposite thin films

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Abstract

Nonvolatile floating gate memory (NFGM) device composed of Sb$_2$Te nanocrystals (NCs) as the charge storage traps embedded in SiO$_2$ matrix was prepared by the target-attachment sputtering method at various nitrogen (N$_2$) incorporation conditions. Via post annealing at 450 °C in ambient air, the sample prepared at the condition N$_2$/Ar=0.1 exhibited a maximum memory window ($\Delta V_{FB}$) shift=4.4 V and charge density=4.2×10$^{12}$ cm$^{-2}$ under ±7 V gate voltage sweep. N$_2$ incorporation not only reduced the Sb$_2$Te NC size to about 5 nm, but also suppressed the oxygen defects and antimony oxides in the sample. Feasibility of the Sb$_2$Te chalcogenide NCs to NFGM fabrication with the simplified process and relatively low annealing temperature is demonstrated.

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1. Introduction

Nonvolatile floating gate memory (NFGM) is a key component to the portable electronic products in the present day. However, the conventional NFGM suffers high leakage current and poor reliability due to the scale-down limitation [1,2]. The device composed of discrete nanocrystals (NCs) as the charge storage traps embedded in dielectric matrix is hence proposed to replace the memory device with continuous layer structure. It offers the advantages including low power consumption, fast write/erase speed, small voltage operation and high endurance [1–3]. The NFGM containing Si NCs embedded in a SiO$_2$ matrix was first proposed by Tiwari et al. [1]. Since then, various transition metal NCs, e.g., cobalt (Co) [4], ruthenium (Ru) [5], platinum (Pt) [6], and iridium (Ir) [7] have been implanted in the NFGMs due to their advantages of high thermal stability, wide range of work function, and large density of states around Fermi level. Presently, a memory window ($\Delta V_{FB}$) shift=7.4 V with charge density=$1.17 \times 10^{13}$ cm$^{-2}$ under −15 V to +13 V gate voltage sweep was achieved in NFGM containing Ir NCs [7]. However, several difficulties remain to be overcome for the metallic NC-based NFGMs, for instance, the metal interdiffusion and oxidation during high-temperature annealing (usually above 600 °C) might degrade the memory performance [4–7]. Moreover, complicated fabrication methods are frequently required in order to gain control on the thickness and quality of oxide layer presenting in between the dielectric layer and the Si substrate [8]. In order to reduce leakage current and improve thermal stability, nitrogen (N$_2$) incorporation has been adopted to eliminate the undesired features brought by the oxide interfacial layer due to its strong coupling with oxygen vacancies [9].

Chalcogenides are well-known phase-change recording media for optical data storage [10]. They possess ultra fast recrystallization rates and comparatively low recrystallization temperatures and, hence, it is the purpose of this work to explore the feasibility of chalcogenide NCs to NFGM so as to achieve a simple fabrication method in conjunction with low-temperature annealing process. Nucleation-dominated chalcogenide, Ge$_2$Sb$_2$Te$_5$ [11] and doped growth-dominated chalcogenide, AgInSbTe$_2$ [12], in NC form have been demonstrated to possess a significant NFGM property. Sb$_2$Te possesses a high work function (4.61 eV) [13,14] that may generate a deep trap for charge storage. Nevertheless, the NFGM study regarding of the dopant-free growth-dominated alloy, Sb$_2$Te, is still missing. This study prepares the Sb$_2$Te–SiO$_2$ nanocomposite thin films by the target-attachment sputtering method [12] at various N$_2$ incorporation conditions. The memory performance of NFGM containing such nanocomposite layers is evaluated and its correlations to the microstructure and chemical status of elements are discussed in the following.

2. Experimental details

A Metal-insulator-semiconductor (MIS) capacitor containing Sb$_2$Te–SiO$_2$ nanocomposite layer was deposited on p-type, Si (100) substrates (resistivity=1–10 $\Omega$·cm). A schematic illustration of the device structure is shown in Fig. 1. Prior to the deposition, all Si substrates were cleaned by standard RCA process and soaked in a diluted hydrofluoric acid solution to remove the native oxides. Afterward, 25-nm thick Sb$_2$Te–SiO$_2$ nanocomposite layers were deposited on the Si substrate by target-attachment method in a sputtering system with a background pressure better than $2.7 \times 10^{-4}$ Pa. The Sb$_2$Te pellets in disc form with 12 mm in diameter and 1.5 mm in thickness were first prepared by pressing the Sb$_2$Te powder in the mold at a pressure of 100 MPa. Appropriate amount of Sb$_2$Te pellets was then attached on

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Since the effect of N2 incorporation on the chalcogenide NC-based crystallization of Sb2Te phase in nanocomposite layers. Note that the annealing condition has been optimized to achieve the best memory property was observed in N10 sample which exhibits the asymmetric charge injection from NCs to the gate electrode. Lee et al. pointed out that the asymmetric J−E behavior of memory device is resulted from the difference in energy barrier height for charge transport [22]. In the case of gate injection (i.e., positive bias on the top Al electrode), N10 sample apparently exhibits the lowest leakage current density of 1.28×10⁻¹⁵ A/cm² at a bias field of 8 MV/cm in comparison with the other samples. This implies a good Coulomb’s blockage effect on such sample that the suppression of charge de-trapping from NCs to the substrate would benefit the data retention property [21]. In the case of gate injection (i.e., negative bias on the top Al electrode), relatively high leakage currents were observed for all samples. This indicates a preference of charge injection from NCs to the gate electrode. Lee et al. pointed out that the asymmetric J−E behavior of memory device is resulted from the difference in energy barrier height for charge transport [22].

Fig. 2(a) presents the C−V characteristics of various NFGM samples subjected to ±7 V gate voltage sweeps. It is worthy to note that the counterclockwise hysteresis loops indicate electron trapping/detrapping in NCs during the substrate injection [3]. The N00 sample exhibits a negligible ΔVFB shift of 0.4 V, indicating the poor charge storage in NCs during the memory operation. As to N10 and N20 samples, the ΔVFB shifts of 4.41 V and 3.38 V with the corresponding charge densities of 4.21×10¹² cm⁻² and 3.61×10¹¹ cm⁻² were achieved. Moreover, the significant negative shifts of C−V profiles imply the presence of positive fixed charges, e.g., oxygen vacancies, in nanocomposite layer [19]. Though the C−V results shown in Fig. 2(a) clearly indicate that N2 incorporation enhances the charge storage capability of NCs, excessive N2 addition in fact degrades the electrical performance as revealed by the C−V profile of N20 sample. This is ascribed to the formation of nitrides near the substrate which would trap the charges and suppress the Coulomb's blockage effect in the nanocomposite layer [20]. The presence of nitride phase in the samples was discussed in the following XPS analysis.

Fig. 2(b) presents the current density–electric field (J−E) characteristics of the samples deduced by the J−V measurement. At the substrate injection case (i.e., positive bias on the top Al electrode), N10 sample apparently exhibits the lowest leakage current density of 1.28×10⁻¹⁵ A/cm² at a bias field of 8 MV/cm in comparison with the other samples. This implies a good Coulomb’s blockage effect on such sample that the suppression of charge de-trapping from NCs to the substrate would benefit the data retention property [21]. In the case of gate injection (i.e., negative bias on the top Al electrode), relatively high leakage currents were observed for all samples. This indicates a preference of charge injection from NCs to the gate electrode.

The microstructures of NCs were characterized by transmission electron microscopy (TEM, FEI TECNAI G2 F20 S-TWIN) with an operating voltage of 200 kV. The cross-sectional TEM (XTEM) samples were prepared by the focused ion beam (FIB, FEI Helios 400S) technique using the 8-keV Ga⁺ ion source for sample milling. An energy dispersive spectroscopy (EDX, Link ISIS 300) attached to the TEM was adopted to analyze the stoichiometric ratio of chalcogenide NCs. The chemical status of the elements in nanocomposite samples was analyzed by an X-ray photoelectron spectroscopy (XPS, PHI Quantera SXAM) using monochromatic Al Kα(1486.6 eV) radiation as the X-ray source. The curve fitting of XPS profiles was achieved by using the XPS Peak 4.1 software. The spectra were de-convoluted with a Shirley background and optimized by allowing the 100% Gaussian with fixed full width at half maximum.

Capacitance–voltage (C−V) and charge retention properties of NFGM devices were evaluated by a HP 4284A precision LCR meter at a frequency of 1 MHz. Current–voltage (I−V) measurement was carried out by using an HP 4156B semiconductor parameter analyzer in conjunction with a probe tester (SANWA, WM-365A-1). All electrical measurements were performed in ambient air at room temperature.

3. Results and discussion

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with satisfactory electrical performance can be achieved. Moreover, it effectively saves the thermal budget of the NFGM process since the utilization of chalcogenide NCs as the charge traps dramatically reduces the temperature and time span of post annealing process in comparison with previous studies [4–7].

XTEM images of Sb$_2$Te–SiO$_2$ nanocomposite layers in the N$_{00}$, N$_{10}$ and N$_{20}$ samples are separately presented in Fig. 3(a)–(c). It can be readily seen that the amount of N$_2$ incorporation affects the sizes and distribution of Sb$_2$Te NCs in the nanocomposite layer. As shown in Fig. 3(a), the column-like Sb$_2$Te phase emerges in the N$_{00}$ sample. Severe overlapping of Sb$_2$Te NCs occurred in such a sample that it not only impoverishes the charge trap sites, but also forms the leakage channel of charges. This explains the poor C–V performance of N$_{00}$ sample as depicted in Fig. 2(a). Uniformly dispersed Sb$_2$Te NCs with size of about 5 nm were observed in N$_{10}$ sample as shown in Fig. 3(b) and (d). This illustrates that appropriate N$_2$ incorporation may result in the discrete NCs and hence suppresses the lateral charge loss [21]. Moreover, it increases the number of NCs and, hence, the number of charge trapping sites in the nanocomposite layer. A satisfactory memory performance was thus achieved in the N$_{10}$ sample. As to N$_{20}$ sample, it contains ultra small Sb$_2$Te NCs with size of about 3–4 nm as shown in Fig. 3(c). Though the number of NCs seemed to increase with increasing N$_2$ incorporation, TEM characterization revealed rather weak and vague image contrast for NCs in N$_{20}$ sample. This implies a suppression of crystallinity of chalcogenide phase due to excessive N$_2$ addition. The studies relating phase-change media for optical discs reported that the N$_2$ doping results in the formation of nitride phases which may alter the recrystallization behaviors of chalcogenides [18].

We note that EDX analysis performed in the TEM indicates the stoichiometric ratio of chalcogenide NCs is Sb:Te = 2:3 for all samples. The study on Sb$_2$Te found that its structure can be viewed as a sequential stacking of Sb$_2$Te$_3$ and Sb$_2$ layers [24]. It is inferred that the Sb portion of Sb$_2$Te might react with oxide matrix to form the antimony oxide on the NC surfaces while the Sb$_2$Te$_3$ portion becomes the dominant constitution of NCs. Evolution of such a metal oxide will be discussed in the subsequent XPS analysis. Moreover, the TEM micrographs shown in Fig. 3 revealed, regardless of N$_2$ incorporation, a SiO$_2$ layer with thickness of about 3–5 nm is present at the nanocomposite/Si interface in all samples.

### Table 1

<table>
<thead>
<tr>
<th>Sample designation</th>
<th>ΔV$_{FB}$ shift (after 10$^4$ s retention)</th>
<th>Charge loss (at ± 5 V)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N00$^a$</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>N10</td>
<td>2.05 V</td>
<td>27.8%</td>
</tr>
<tr>
<td>N20</td>
<td>2.03 V</td>
<td>28.5%</td>
</tr>
</tbody>
</table>

$^a$ Memory property diminished in less than 10$^3$ s.

![Fig. 2. (a) C–V and (b) J–E characteristics of NFGM samples. The gate voltage sweep range is ±7 V for C–V measurement. (c) Schematic energy band diagrams of NFGM samples under substrate and gate injection processes.](image-url)
Such a thin SiO$_2$ interfacial layer can be clearly seen in the enlarged micrograph shown in Fig. 3(d). It not only serves as the tunneling layer for charge transport, but also provides an effective barrier for charge storage in the nanocomposite layer.

Fig. 4(a)–(d) depict the XPS spectra and the corresponding deconvoluted profiles obtained by the Gaussian fitting method [25]. Prior to the XPS analysis, removal of about 20-nm thick nanocomposite layer was performed by 1-keV/20-mA Ar$^+$ ion sputtering in in-situ manner to expose the region enriched with chalcogenide NCs. Fig. 4(a) presents the N 1s XPS spectra of N$_{10}$ and N$_{20}$ samples in which the curve fitting of the spectra reveals the presence of Si–N (398.2 eV) and Sb–N (401.8 eV) bonds in the N$_2$-incorporated samples. This indicates that N$_2$ incorporation during sputtering may result in the nitride phases, e.g., Si$_3$N$_4$ and Sb$_4$N$_6$, in the nanocomposite layers. It is noted that the corresponding XPS peak intensities are rather weak, indicating the small nitride contents in the samples due to the low N$_2$ inlet gas flow adopted for sample preparation. As shown by Si 2p XPS spectra in Fig. 4(b), a relatively high intensity of suboxide phase, i.e., SiO$_2$ (102.4 eV), in the N$_{80}$ sample implies the abundance of oxygen defects in such sample. As to the N$_{10}$ and N$_{20}$ samples, the dominance of the SiO$_2$ (103.3 eV) component indicates that N$_2$ incorporation suppresses the oxygen defects in the nanocomposite layers. The increase of SiO$_2$ peak also indicates that a dense oxide component is built in the nanocomposite layer, implying the lower leakage and better charge retention properties of N$_{10}$ and N$_{20}$ samples as depicted in Fig. 2(a) and (b). In addition, a small amount of silicon nitride (Si$_3$N$_4$; 101.8 eV) emerging in Fig. 4(b) also manifested the presence of Si–N bonds in the matrix of nanocomposite layer. It is plausible that a portion of SiO$_2$ may transform into the oxide–nitride mixtures, e.g., SiO$_x$N$_y$, which are known to suppress the charge hopping process and improve the leakage property of the samples [26].

The Sb 3d XPS spectra shown in Fig. 4(c) reveal the Sb$_2$O$_3$ phase (530 eV in 3d$_{5/2}$ region; 539.6 eV in 3d$_{3/2}$ region) in the N$_{80}$ sample. The antimony oxide likely resulted from the oxidization of NCs during the annealing at 450 °C in ambient air by interacting with oxide matrix via the reactions 3Sb$_2$Te + 3SiO$_2$ → Sb$_2$(Si,Te)$_3$ + 2Sb$_2$O$_3$. This also explains the detection of a Sb$_2$Te$_3$ phase in the samples by EDX analysis. As to the N$_{10}$ and N$_{20}$ samples, Fig. 4(c) indicates that N$_2$ incorporation effectively eliminates the Sb$_2$O$_3$ phase and results in the small amount of Sb$_2$N$_4$ phase (530.2 eV in 3d$_{5/2}$ region; 539.6 eV in 3d$_{3/2}$ regions) in the nanocomposite layers. The removal of Sb$_2$O$_3$ component in the N$_{10}$ and N$_{20}$ samples suggests that the suppression of antimony oxide by appropriate N$_2$ incorporation benefits the memory performance.

Te 3d$_{5/2}$ XPS spectra for all samples are presented in Fig. 4(d). It can be readily seen that the N$_2$ incorporation and post annealing conditions do not affect the chemical status of Te in the interior of the nanocomposite layer which remains in a form of metallic Te (572.85 eV). Nevertheless, the Te 3d$_{5/2}$ XPS depth profile of N$_{80}$ sample shown in Fig. 5 reveals a 3-nm thick TeO$_2$ layer residing on the nanocomposite surface. The thin TeO$_2$ layer was similarly observed in other samples and it is noted that, once the nanocomposite samples were removed from the vacuum chamber, such TeO$_2$ surface layer inevitably formed on the samples as revealed by the composition analysis. This indicates that the reaction of chalcogenide with oxygen in ambient air is most likely the cause of TeO$_2$ formation. Once it formed on the sample surface, the TeO$_2$ seemed to passivate the nanocomposite layer since no Te oxidization further occurred in the interior of the nanocomposite layer as revealed in Figs. 4(d) and 5. Similar to the SiO$_2$ layer at the nanocomposite/Si substrate interface, the TeO$_2$ layer at the nanocomposite/Al electrode interface might serve as a tunneling layer for charge transport and the charge confinement layer in NFGM.

Fig. 3. XTEM micrographs of (a) N$_{00}$, (b) N$_{10}$ and (c) N$_{20}$ samples. Enlarged micrograph shown in (d) is taken from N$_{10}$ sample at the nanocomposite layer/Si substrate interface.
device containing chalcogenide NCs. However, TeO₂ is not effective as a barrier compared to the SiO₂ layer due to its lower barrier height as mentioned previously.

4. Conclusions

Chalcogenide NCs serving as the charge traps were implanted in the NFGM devices by using the target-attachment sputtering method. With a supply of Ar/N₂ inlet gas flow at the ratio of 10:1 during the sputtering deposition, a significant ΔV_{FB} shift = 4.41 V and charge density = 4.21×10^{12} cm⁻² at ±7 V gate voltage sweep were achieved in the NFGM sample. Moreover, the ΔV_{FB} shift about 2.05 V and the charge loss about 27.8% were observed after 10⁴s retention test at ±5 V gate voltage stress. Analytic results indicated that appropriate N₂ incorporation may not only suppress the number of oxygen defects, but also eliminate the formation of antimony oxides in the nanocomposite layer. This promoted the charge storage capacity and yielded a significant memory performance in NFGM device. This study demonstrated that a single chalcogenide NCs–SiO₂ nanocomposite is feasible as the programming layer of NFGM device. In addition to the simplification of device fabrication, utilization of chalcogenide NCs may also save the thermal budget in an effective manner and is a promising alternative for advanced NFGM fabrication in the future.

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Fig. 4. (a) N 1s, (b) Si 2p, (c) Sb 3d and (d) Te 3d XPS spectra for nanocomposite samples prepared at various N₂ incorporation conditions. The solid black curves with open diamond symbols correspond to raw XPS data while the black curves are the profiles obtained by Gaussian fitting.

Fig. 5. Te 3d_{5/2} XPS depth profiles for N_{10} sample.
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References