

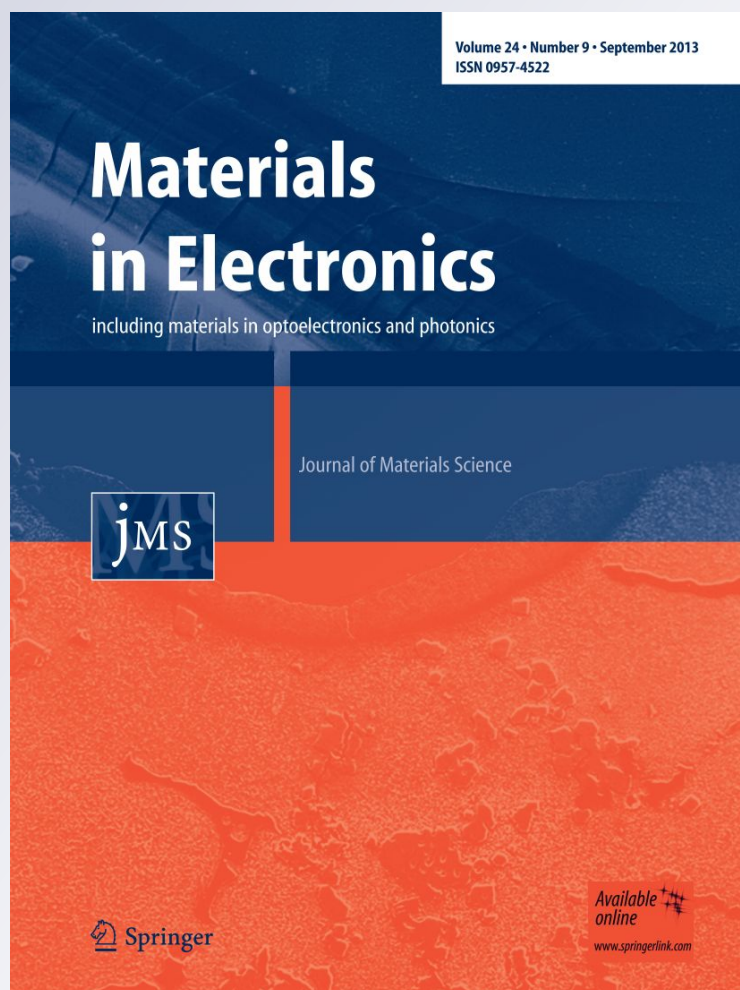
*Insulator–metal transition and ultrafast crystallization of Ga<sub>40</sub>Sb<sub>60</sub>/Sn<sub>15</sub>Sb<sub>85</sub> multiple interfacial nanocomposite films*

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# Insulator–metal transition and ultrafast crystallization of Ga<sub>40</sub>Sb<sub>60</sub>/Sn<sub>15</sub>Sb<sub>85</sub> multiple interfacial nanocomposite films

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## Abstract

The phase change behavior of Ga<sub>40</sub>Sb<sub>60</sub>/Sn<sub>15</sub>Sb<sub>85</sub> multiple interfacial film was studied systematically for its application in phase change memory. The thermal stability of amorphous state was evaluated using the Arrhenius formulas. The evolution of phase structure indicates that the Sb phase exists in the Sb-rich Ga<sub>40</sub>Sb<sub>60</sub>/Sn<sub>15</sub>Sb<sub>85</sub>, Ga<sub>40</sub>Sb<sub>60</sub> and Sn<sub>15</sub>Sb<sub>85</sub> films. The change in density and thickness during crystallization was obtained by X-ray reflectance. The nanoscale change in roughness of the Ga<sub>40</sub>Sb<sub>60</sub>/Sn<sub>15</sub>Sb<sub>85</sub> film was confirmed by atomic force microscopy. The rate for crystallization and amorphization transformation was measured by a picosecond laser pump-probe system. The phase change memory device based on [Ga<sub>40</sub>Sb<sub>60</sub> (7 nm)/Sn<sub>15</sub>Sb<sub>85</sub> (3 nm)]<sub>5</sub> thin film was fabricated. An ultra-fast switching and low power were achieved for Ga<sub>40</sub>Sb<sub>60</sub>/Sn<sub>15</sub>Sb<sub>85</sub> multiple interfacial film.

## 1 Introduction

As the promising next-generation mainstream storage technology, phase change random access memory (PCRAM) has great advantages in speed as well as storage density compared to flash memory [1]. The storage of the data is based on the reversible phase transition of the chalcogenide compound material induced by an electrical pulse or heating manner. The RESET process can be achieved with a high and short electrical pulse, while the SET one can be obtained in a low but long electrical pulse. The significant difference between the resistivity of two states is used to

distinguish the binary logic levels of “0” and “1” [2]. As the core of PCRAM, the performance of phase change materials directly affects the characteristics of devices. Therefore, the phase change material needs to meet the requirements of fast crystallization speed, good thermal stability and low operating power consumption.

With the improvement of phase change materials in recent years, the traditional material Ge<sub>2</sub>Sb<sub>2</sub>Te<sub>5</sub> (GST) has exposed more and more problems, such as the relatively low 10-year data retention temperature (85 °C), resulting in poor thermal stability. This makes it impossible to be applied in the automotive electronics sector (~125 °C) and industrial control aviation (~150 °C) [3]. In addition, GST belongs to a nucleation dominated crystallization mechanism. Its crystallization time is more than 100 ns, which is not comparable with DRAM (10 ns). To this end, we hope to develop a new material that is far superior to GST in terms of speed and power consumption. Zifang He et al. [4] have confirmed that the SnSb<sub>4</sub>/Ge multiple interfacial film has a rapid crystallization rate. XRD analysis shows that the Sb phase still exists before and after recombination, which can shorten the crystallization time of the material. The GaSb/Sb<sub>2</sub>Te<sub>3</sub> film with superlattice structure prepared by Yegang Lu [5] et al. have both good thermal stability and low operating power consumption. When these materials are applied in PCRAM, the crystallization time (SET) is less than 10 ns, and the erasing speed is improved. Based on previous researches, we combined the Ga<sub>40</sub>Sb<sub>60</sub> (GS) and Sn<sub>15</sub>Sb<sub>85</sub> (SS) materials to obtain a multiple interfacial phase change film

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in this work. The reversible phase change and crystallization behavior were investigated in details.

## 2 Experimental

GS, SS monolayer films and GS/SS multiple interfacial films were prepared by RF magnetron sputtering (single target sputtering and alternating sputtering, respectively). The total thickness of the film was fixed at 50 nm, the deposited substrate was SiO<sub>2</sub>/Si (100), and the purity of GS and SS alloy targets were over 99.99 at.%. The substrate temperature was room temperature, and the instrument was cooled by circulating water during sputtering. The background pressure of the vacuum chamber was lower than  $1 \times 10^{-4}$  Pa with a sputtering gas pressure of 0.25 Pa. A low sputtering power of 30 W was adopted to ensure the accurate control of film thickness. The substrate was ultrasonically cleaned prior to use to remove dust, organic and inorganic impurities from the surface. The sample disk kept rotating at a speed of 20 rpm to guarantee the uniformity of films with an argon flow rate of 30 sccm (sccm was standard ML/min).

The  $R$ – $T$  curves of the phase change films were measured using an in situ resistance–temperature test system to study the phase transition characteristics of the films from amorphous to crystalline state. At the same time, the crystallization activation energy ( $E_a$ ) of the film was calculated with isothermal crystallization to evaluate its thermal stability. The reflectance of the film was measured by a near-infrared spectrophotometer (NIR, 7100 CRT, XINMAO, China), and the band gap was obtained by Kubelka–Munk's law. The phase structure was analyzed using an X-ray diffractometer (XRD, PANalytical, Netherlands) with Cu target, K ray, scanning angle range of 20°–60° in the scanning speed of 1° min<sup>-1</sup>. The change of film thickness during crystallization was determined by an X-ray reflectometer (XRR). The surface morphology of the film was observed by atomic force microscopy (AFM, FM Nanoview 1000). The picosecond laser pumping probe system was employed to study the phase transition velocity by irradiating the film with a mode-locked ytterbium-doped yttrium aluminum garnet laser at a wavelength of 532 nm for about 30 ps. The  $I$ – $V$  and  $R$ – $V$  curves of the PCRAM device cells based on the GS/SS multiple interfacial film were tested using a Tektronix AWG5012B arbitrary waveform generator and a Keithley 2602 A parameter analyzer.

## 3 Results and discussion

As shown in Fig. 1, all the films are in a high-resistance state before crystallization. The resistivity increases with the thickness of the GS layer in the GS/SS multilayer phase change films. Higher resistivity means less free electrons participating

in conduction. As the temperature rises gradually, the film reaches its crystallization temperature ( $T_c$ ) [6]. As can be seen from the Fig. 1, the corresponding  $T_c$  for SS, [GS (5 nm)/SS (5 nm)]<sub>5</sub>, [GS (6 nm)/SS (4 nm)]<sub>5</sub>, [GS (7 nm)/SS (3 nm)]<sub>5</sub>, GS thin films were 189, 223, 238, 266 and 330 °C, respectively. In general, the higher crystallization temperature reveals that the phase change film has better amorphous thermal stability but higher programming power consumption. Compared with SS, the GS/SS multiple interfacial films have more stable amorphous resistance state. In addition, the value of amorphous resistance for the GS/SS multiple interfacial films significantly increases with more GS, which can reduce the pulse current of the Joule heating and is conducive to reducing the power consumption in the RESET process [7]. The resistivity of the amorphous GS/SS films differs by at least two orders of magnitude from that of the crystalline films, which is suitable for PCRAM application.

In order to further analyze the thermal stability of phase-change films, the isothermal annealing is carried out to evaluate the difficulty of crystallization. The Arrhenius formula is the common model used to quantitatively assess the data retention and can be expressed as [8]:

$$t = t_0 \exp(E_a/k_b T) \quad (1)$$

where  $t$ ,  $t_0$ ,  $k_b$  and  $T$  are the failure times, the pre-factor depends on the nature of the material, the Boltzmann constant and the absolute temperature, respectively. Figure 2 is a plot of failure time versus  $1/(k_b T)$ . The extrapolated temperatures for the 10-year lifetime ( $T_{10}$ ) of [GS (5 nm)/SS (5 nm)]<sub>5</sub>, [GS (6 nm)/SS (4 nm)]<sub>5</sub> and [GS (7 nm)/SS (3 nm)]<sub>5</sub> films are 154, 168 and 179 °C, respectively, which exceed the requirements of automotive electronics industry (120 °C) [9, 10]. From the slope of Fig. 2, the activation energy ( $E_a$ ) of the GS/SS multiple interfacial film can be

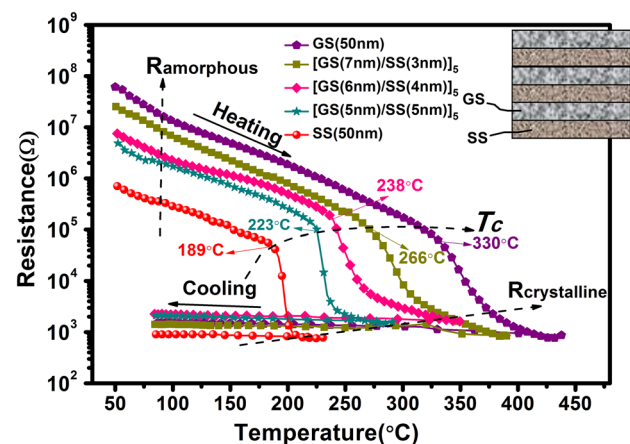


Fig. 1 Resistance as a function of temperature for GS and SS monolayer thin films, GS/SS multiple interfacial films with different thickness ratio at a heating rate of 30 °C/min

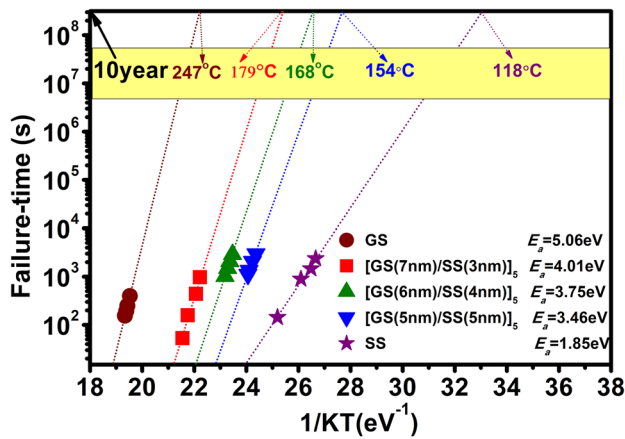


Fig. 2 Plots of failure time versus reciprocal temperature, showing the data retention

obtained. With the increase of the proportion of GS layer in the GS/SS film, the  $E_a$  increased from 3.46 eV of [GS (5 nm)/SS (5 nm)]<sub>5</sub> to 4.01 eV of [GS (6 nm)/SS (4 nm)]<sub>5</sub>. The values are just between the GS (5.06 eV) and SS (1.85 eV) monolayer films. It can be seen that changing the composition thickness in multiple interfacial films can effectively regulate the structural switching barrier in phase transition. The excellent amorphous thermal stability makes the GS/SS films can be used in higher temperature environments [11].

The reflectance spectra of GS/SS films were measured by UV–Vis–NIR spectrophotometry in the wavelength range of 400 to 2500 nm. As shown in Fig. 3, the band gap energy can be determined by extending the absorption edge onto the energy axis, where the conversion of reflectance to absorbance is obtained by the Kubelka–Munk function (K–M) [12]:

$$K/S = (1 - R)^2 / (2R) \quad (2)$$

where  $R$ ,  $K$  and  $S$  are reflectance, absorption coefficient and scattering coefficient, respectively.

Figure 3 shows that the energy band gaps of amorphous SS, [GS (5 nm)/SS (5 nm)]<sub>5</sub>, [GS (6 nm)/SS (4 nm)]<sub>5</sub>, [GS (7 nm)/SS (3 nm)]<sub>5</sub> and GS thin films are 0.648, 0.729, 0.750, 0.763 and 0.837 eV, respectively. At 380 °C, the band gaps of crystalline SS, [GS (5 nm)/SS (5 nm)]<sub>5</sub>, [GS (6 nm)/SS (4 nm)]<sub>5</sub>, [GS (7 nm)/SS (3 nm)]<sub>5</sub> and GS thin films are 0.436, 0.525, 0.606, 0.656 and 0.734 eV, respectively. The value of band gap for the overall crystalline states is smaller than that of the amorphous ones, indicating that the carrier concentration becomes larger. Besides, the energy band gap of the films increases with the thickness of GS layer, which is in accordance with the trend of

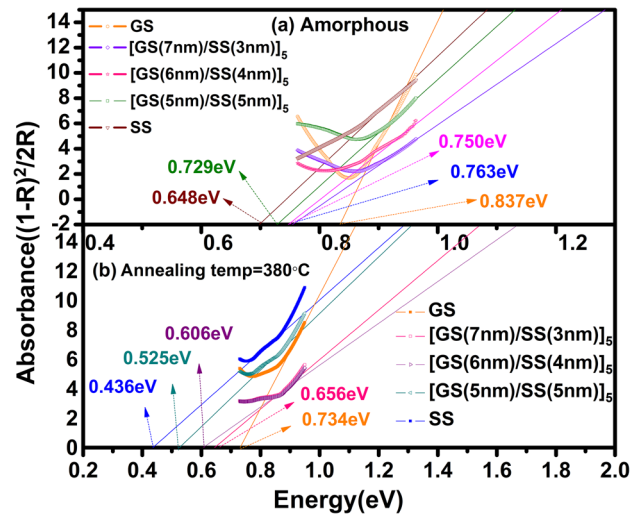


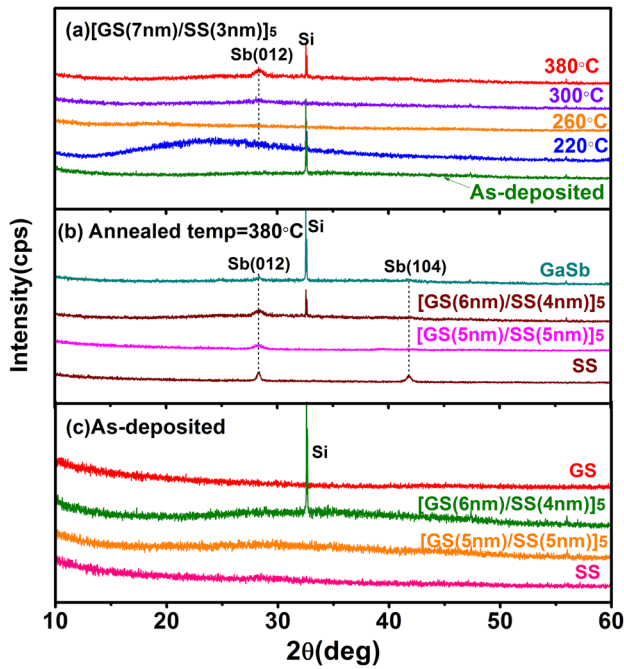
Fig. 3 The Kubelka–Munk function of GS, SS monolayer thin films and GS/SS multiple interfacial films for a amorphous and b crystalline states

the  $R$ – $T$  curves in Fig. 1 [13]. With a broader band gap, the film needs more energy to accumulate for phase change.

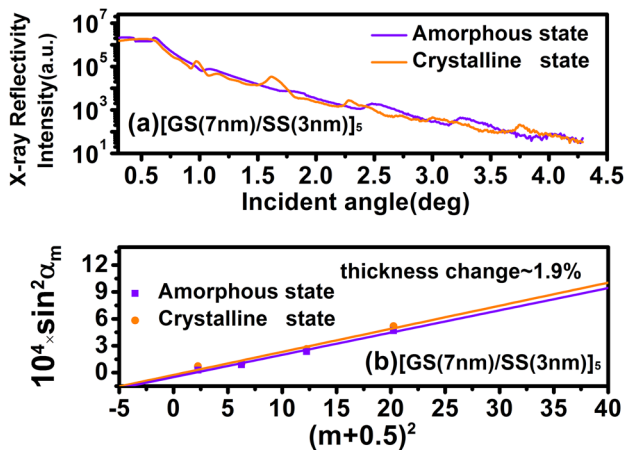
Figure 4 provides the XRD diffraction patterns of the deposited and annealed GS/SS, GS and SS films. In the deposited state (Fig. 4c), no diffraction peaks of all films were observed, indicating an amorphous structure. When the annealing temperature rises to 380 °C above the  $T_c$  for all films, the diffraction peaks (012) and (104) belonging to the Sb phase appear in Fig. 4b. It can be concluded that the Sb phase is present in the Sb-rich GS/SS, GS and SS films. In Fig. 4a, no Sb phases are observed for annealed [GS (7 nm)/SS (3 nm)]<sub>5</sub> at 220 and 260 °C, which is consistent with the results of Fig. 1. Besides, Sb (104) peak is absent for [GS (7 nm)/SS (3 nm)]<sub>5</sub> film, which may be the result of the suppression of multiple interface. It should also be noted that all of the three graphs have diffraction peaks belonging to the Si phase, indicating that Si is derived from the Si substrate [14].

The atomic rearrangement in the phase change process can change the volume or density of the film. The resulted voids are detrimental to the contact between phase change material and the electrode. It can cause the degradation of cycle durability and reliability of the PCRAM device. Figure 5a shows the XRR pattern of the crystalline and amorphous states of the [GS (7 nm)/SS (3 nm)]<sub>5</sub> film. It can be found that the incident angle corresponding to the maximum or minimum peak of the crystalline state shifts to a higher angle. The modified Bragg equation is used to determine the density change before and after crystallization [15]:

$$\sin \theta_m = 2\delta + (m + \Delta m)^2 \left( \frac{\lambda}{2t} \right)^2 \quad (3)$$



**Fig. 4** XRD patterns of **a** [GS (7 nm)/SS (3 nm)]<sub>5</sub> thin film annealed at various temperatures for 5 min; **b** GS, SS and GS/SS thin films annealed at 380 °C for 5 min; **c** as-deposited state of GS, SS and GS/SS thin films



**Fig. 5** **a** XRR curves and **b** modified Bragg fitting curves of amorphous and crystalline [GS (7 nm)/SS (3 nm)]<sub>5</sub> thin films

where  $\delta$  is a constant,  $m$  in  $\theta_m$  is the reflection coefficient,  $\lambda$  is the wavelength (0.154 nm),  $t$  is the film thickness,  $m$  in  $m + \Delta m$  is the correction coefficient, and the value of  $\Delta m$  is 0.5, and the value of  $\Delta m$  corresponds to the minimum or maximum value of the XRR diffraction peak for each reflection coefficient. The linear curve of  $\sin^2 \alpha_m$  and  $(m + \Delta m)^2$  is

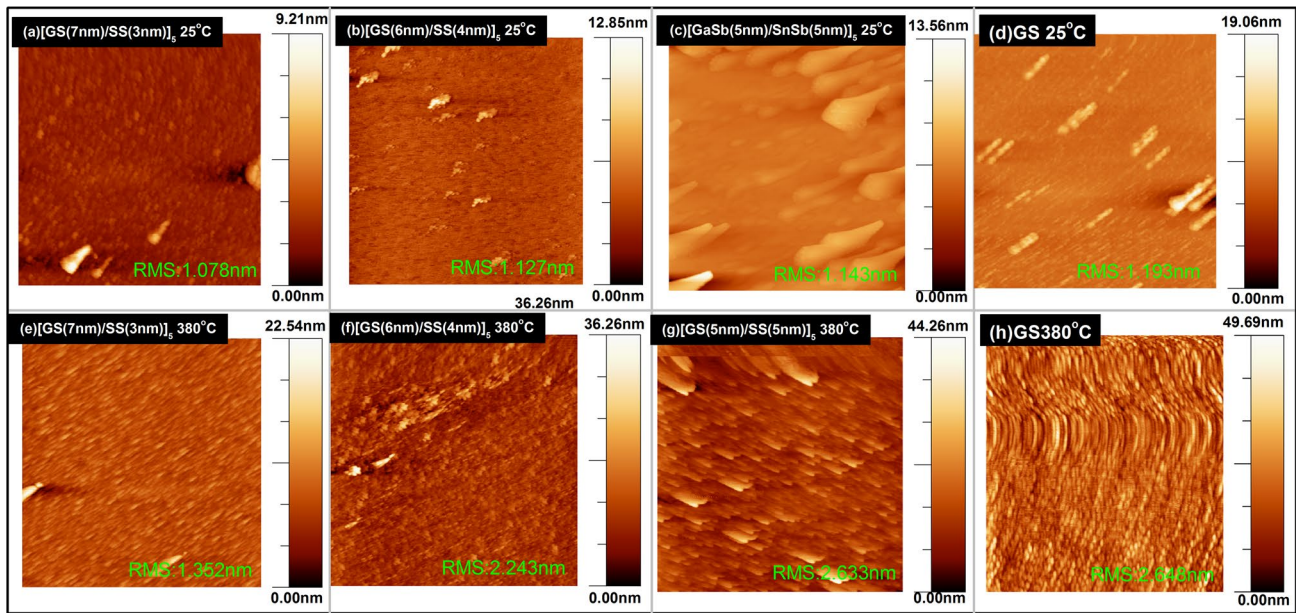
shown in Fig. 5b. The relationship between the thickness variation of the film is calculated as [15]:

$$t = \lambda / 2 \sqrt{k} \tag{4}$$

From this, it is calculated that the thickness change of the [GS (7 nm)/SS (3 nm)]<sub>5</sub> film before and after crystallization is 1.9%, which is smaller than the conventional GST (6.8%) film [16]. The existence of multiple interface restrains the phase transition, leading to a smaller volume change. The smaller thickness variation during the phase change can improve the reliability and extend the life of the PCRAM.

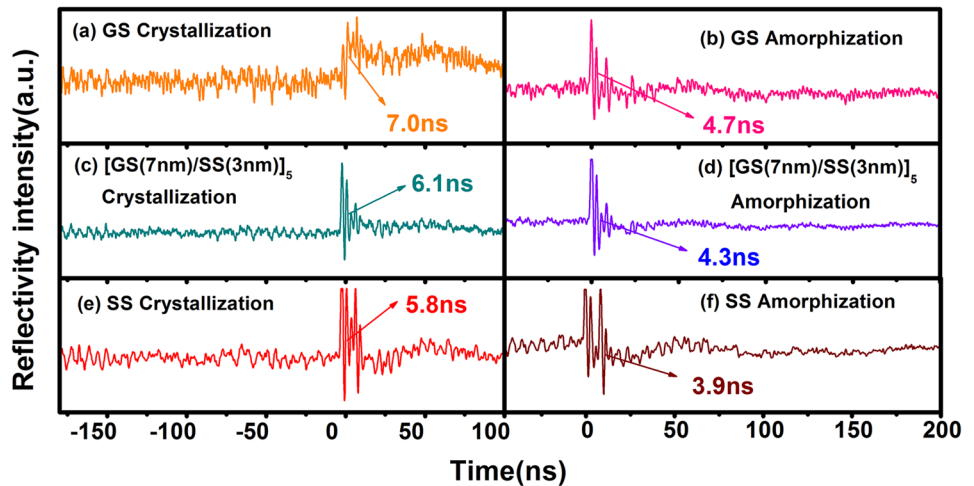
The morphology of the film surface also changes accompanied by the internal stress. Figure 6 shows the AFM images of the GS and GS/SS films before and after crystallization. As can be clearly seen in Fig. 6a–d, all film surfaces appear flat and smooth with a small Root-Mean-Square (RMS) roughness of [GS (7 nm)/SS (3 nm)]<sub>5</sub> = 1.078 nm, [GS (6 nm)/SS (4 nm)]<sub>5</sub> = 1.127 nm, [GS (5 nm)/SS (5 nm)]<sub>5</sub> = 1.143 nm and GS = 1.193 nm. After annealing at 380 °C for 10 min in Ar atmosphere, many small and dense grains can be observed, indicating the amorphous to crystalline phase transition. Accordingly, the value of RMS increases a little to 1.078 = 1.193 nm. Compared with GS, the grain quantity of GS/SS multiple interfacial films is much less and the surface roughness is lower. It should be the results of the inhibitory effect of interface clamping. This research reveals that the PCRAM device based on GS/SS multiple interfacial films can have a better contact between the phase-change layer and electrode, which can improve the fatigue durability [17].

For PCRAM, the speed of reading and writing data is crucial. Picosecond laser technology is used to study the speed of phase switching. It is well known that amorphous states exhibit high electrical resistance and low reflectivity, while the crystalline states have low electrical resistance and high reflectivity [18]. Figure 7a, c, e are the transitions from amorphous to crystalline state. The crystallization time for GS, [GS (7 nm)/SS (3 nm)]<sub>5</sub> and SS are 7.0, 6.1 and 5.8 ns, respectively. Usually the crystallization process takes more time than the amorphization one, and the reverse phase change requires higher laser pulse energy. As shown in Fig. 7b, d, f, the amorphization time for GS, [GS (7 nm)/SS (3 nm)]<sub>5</sub> and SS are 4.7, 4.3 and 3.9 ns, respectively. [GS (7 nm)/SS (3 nm)]<sub>5</sub> film has a shorter crystallization and amorphization time than GS, which is also faster than GST (17.7 and 16.5 ns) [19]. The reasons may be that the low thermal conductivity of the composite multilayer structure hinders the heat diffusion to increase heating efficiency and reduce heating time. Besides, the rich Sb atoms with a weak Sb-Sb bond can act as a nucleation center. A large number



**Fig. 6** AFM images for as-deposited and annealed films **a, e** [GS (7 nm)/SS (3 nm)]<sub>5</sub>; **b, f** [GS (6 nm)/SS (4 nm)]<sub>5</sub>; **c, g** [GS (5 nm)/SS (5 nm)]<sub>5</sub>; **d, h** GS

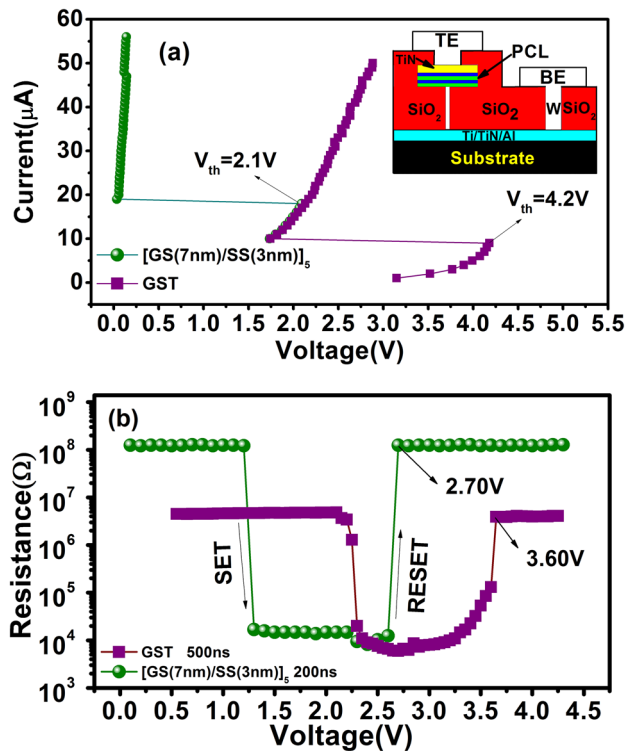
**Fig. 7** Reversible reflectivity evolution of the GS, [GS (7 nm)/SS (3 nm)]<sub>5</sub> and SS thin films induced by consecutive picosecond laser pulses with different fluencies: **a, c, e** crystallization process; **b, d, f** amorphization process



of crystal nuclei can promote the crystallization process and shorten the crystallization time [20].

PCRAM device cells based on [GS (7 nm)/SS (3 nm)]<sub>5</sub> and GST thin films were fabricated using a 0.18 μm CMOS process technology to test and verify the electrical performance. A schematic of the cross-sectional structure is shown in the inset of Fig. 8a. The role of TiN is to increase the adhesion of (top electrode) TE and (phase change layer) PCL. Figure 8a shows the current–voltage (*I*–*V*) curves for the SET operation, which corresponds to the phase transition

from the high resistance to low resistance state. The threshold voltage ( $V_{th}=2.1$  V) of [GS (7 nm)/SS (3 nm)]<sub>5</sub> film is smaller than GST ( $V_{th}=4.2$  V), indicating that the power consumption of the multiple interfacial film during the SET process is smaller [21]. From the resistance–voltage (*R*–*V*) characteristic curves in Fig. 8b, the reversible phase transition can be achieved by a pulse of 200 ns for the [GS (7 nm)/SS (3 nm)]<sub>5</sub> film, indicating that it has a faster switching speed than GST (500 ns). In addition, the RESET voltage (2.70 V) for [GS (7 nm)/SS (3 nm)]<sub>5</sub> film is smaller than



**Fig. 8** **a**  $I$ - $V$  and **b**  $R$ - $V$  curves of the PCRAM cells based on [GS (7 nm)/SS (3 nm)]<sub>5</sub> and GST thin films. The inset in **a** shows the schematic diagram of the PCRAM cell structure

GST (3.60 V), which reveals that the RESET power consumption of the multiple interfacial film is lower [22].

## 4 Conclusions

The reversible phase change and crystallization behavior of GS/SS multiple interfacial film were investigated. The comparison shows that the  $T_c$  increased from 189 °C of SS to 266 °C of [GS (7 nm)/SS (3 nm)]<sub>5</sub>. And the data retention temperature increased from 118 to 179 °C for 10 years. At the same time, GS/SS multiple interfacial films have higher  $E_a$  compared with SS. The band gap value of the crystalline composite film was smaller than that of the amorphous state, resulting in a larger carrier concentration. The Sb phase was present in the Sb-rich GS/SS films. The [GS (7 nm)/SS (3 nm)]<sub>5</sub> film had a smaller thickness change (1.9%) and roughness change before and after crystallization. The crystallization and amorphization time for [GS (7 nm)/SS (3 nm)]<sub>5</sub> are 6.1 and 4.3 ns, respectively. Based on the [GS (7 nm)/SS (3 nm)]<sub>5</sub> thin film, the  $V_{th}$  in RESET process is 2.7 V. The results showed that GS/SS multiple interfacial film was a promising phase change material with fast-speed and low-power in PCRAM application.

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