

## Comparison of the crystallization of Ge–Sb–Te and Si–Sb–Te in a constant-temperature annealing process

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The crystallization of Ge–Sb–Te and Si–Sb–Te phase-change materials has been characterized by in situ time-dependent resistance measurement and transmission electron microscopy. Although silicon has various properties that are similar to those of germanium, Si–Sb–Te and Ge–Sb–Te crystallize via different processes. Si–Sb–Te has a complex structure (mainly hexagonal) while Ge–Sb–Te has a simple face-centered cubic structure when annealed at 433 K for several hours.

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Phase-change memory (PCM) is a rapidly emerging technology for next-generation non-volatile memory [1–3]. It relies on the thermally induced reversible resistance switching of phase-change materials. When a phase-change material in the amorphous state is heated above its crystallization temperature by an electrical pulse, the material rearranges into the polycrystalline state. To switch back to the amorphous state, the material is heated by a stronger pulse above the melting temperature. Data retention is an important parameter for PCM applications. The standard procedure for data retention assessment relies on the evaluation of the time-dependent resistance change under constant-temperature annealing. In recent years, data retention has been assessed by both experimental methods and numerical simulation [4–6]. It is well known that the resistance of a phase-change material will reduce with time during time-dependent resistance ( $R-t$ ) measurements, due to crystallization of the material. In situ  $R-t$  measurements as well as transmission electron microscopy (TEM) have

been widely used to study the crystallization process [6–12]. The relationship between reflectivity and microstructure of phase-change material has also been studied for optical disk applications [13]. Study of the crystallization process of phase-change material via  $R-t$  measurement is important for PCM research and development. In this work, the electrical resistance of Ge–Sb–Te (GST) and Si–Sb–Te (SST) as a function of annealing time is measured in situ at a high-temperature. A comparison of the crystallization processes of the two phase-change materials is made using TEM.

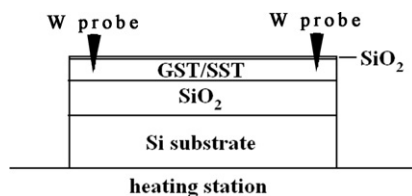
TEM is a powerful scientific technique for assessing the microstructure of materials. A transmission electron microscope equipped with a substrate heating stage and selected-area electron diffraction (SAED) has been employed to study the effects of annealing conditions on crystallization [8–14]. Crystallization of phase-change materials has been studied by in situ observation of their microstructure and morphology in a transmission electron microscope with a heating stage. However, the electrical resistance change was not monitored in those studies. Furthermore, samples for TEM experiments need to be thin. Hence, studies of phase transformations of phase-change materials are often not representative of the transitions occurring in bulk or a

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thicker film. Furthermore, an ultrathin film may result in changes in the crystallization kinetics and in the temperature that promotes crystallization [15]. Therefore, to study the intrinsic properties of phase-change materials, a thicker film is preferred. In most TEM studies reported, a phase-change thin film (<70 nm) is encapsulated between a substrate film and a dielectric protecting layer. According to the references available, the nucleation and growth parameters of these film are influenced by the dielectric capping layers if the film is ultrathin [16]. Moreover, in most recent reports, the phase-change layer in PCMs is around 100 nm [17,18]. According to Ref. [19],  $I_{\text{melt}}$  (the current pulse required to melt the phase-change material within a PCM device) will decrease with the ratio between GST thickness and electrode diameter.  $I_{\text{melt}}$  is reduced to a minimum when the GST thickness is at least equal to the electrode diameter. However, the electrode diameter will be larger than 100 nm in a traditional “T” structure even when 90 nm complementary metal-oxide semiconductor (CMOS) technology is used.

In our previous work, SST was studied for PCM applications due to its outstanding properties [20,21]. Although silicon has various properties that are similar to those of germanium, SST is different from GST in many respects. In the present work, the crystallization processes of both GST and SST are characterized using TEM.

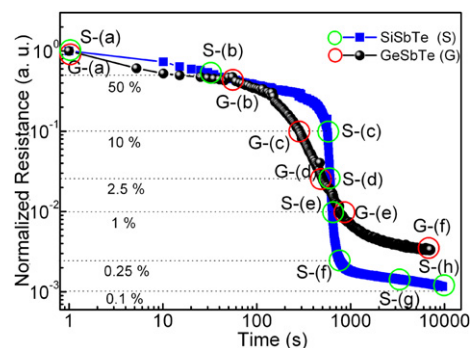
GST and SST films  $\sim 300$  nm thick were deposited on oxidized Si substrates and Al foil by co-sputtering Ge, Si, Sb and Te targets. The background pressure and Ar gas pressure are  $1 \times 10^{-4}$  and 0.21 Pa, respectively. The compositions of the GST and SST films were determined by means of energy dispersive spectroscopy (EDS) using the samples deposited on Al foil. The compositions of GST and SST were determined as Ge(24.4 at.%)–Sb(22.5 at.%)–Te(53.1 at.%) and Si(20.6 at.%)–Sb(23.6 at.%)–Te(55.8 at.%), respectively. The  $R$ – $t$  performance of the as-deposited amorphous GST and SST films was conducted under constant-temperature annealing on a temperature-controlled probe station, which has a uniform temperature distribution. It has been reported that crystal nucleation starts at the phase-change film surface and progresses towards the film–substrate interface along the film thickness [13,22]. The phenomenon might result from the oxidation of GST and SST phase-change material results in a drop in their crystallization temperature [8,21,23]. The two-point resistance measurement method was employed in this work, as shown in Figure 1. Crystallization at the film surface may affect the  $R$ – $t$  measure-



**Figure 1.** Illustration of the electrical resistance vs. time measurement method.

ment result. In order to avoid the oxidation in the  $R$ – $t$  measurement, phase-change films deposited on  $\text{SiO}_2/\text{Si}$  substrates were coated with a 5 nm thick  $\text{SiO}_2$  layer by sputtering. To determine the microstructure of the materials at the different crystallization stages, TEM is carried out to study the powder scratched from the  $\text{SiO}_2/\text{Si}$  substrates after annealing for various amounts of time for high-temperature  $R$ – $t$  measurement. A thinner film would be much more suitable for the TEM test, but it is not so easy to scratch a thin film from the substrate. It is possible that the powder scratched from the film surface might have a higher crystalline fraction compared with the powder from the film–substrate interface if surface crystallization occurs. To solve this problem, several powder samples were studied by TEM for each specimen annealed at various conditions. However, according to the TEM observation, most powder specimens are thicker than 50 nm.

For the  $R$ – $t$  measurement, the five above-mentioned as-deposited  $\text{SiO}_2/\text{GST}/\text{SiO}_2/\text{Si}$  samples ( $1 \text{ cm} \times 1 \text{ cm}$ ) were put on the heating probe station simultaneously. The probe station has a stable temperature of 433 K. Normalized resistance as a function of annealing time for GST film is shown in Figure 2. According to Figure 2, the resistance of GST drops with annealing time, with a sharp drop at around 400 s. In order to study the microstructure of GST at different crystallization stages, six areas on the  $R$ – $t$  curve were chosen for TEM measurement, as shown in Figure 2 by red circles. The six areas represent six different crystallization states of GST. During the  $R$ – $t$  measurement process, when resistance drops to 50% of the value of the initial state, one of the samples was removed from the heating station to a clean board at room temperature. This process was repeated as the resistance drops to 10%, 2.5%, 1% and 0.25% of the value of the initial state. The  $R$ – $t$  curve of SST was also determined and is illustrated in Figure 2. The resistance of SST drops steeply at around 600 s. The resistance of SST annealed for 10,000 s is about 0.1% of that of the amorphous state. Eight SST samples at different crystallization states, denoted by green circles in Figure 2, were chosen for the TEM experiments.



**Figure 2.** Normalized resistance of GST and SST as function of annealing time when the annealing temperature is 433 K. Red (GST) and green (SST) circles refer to samples, annealed for various periods of time, chosen for the TEM measurement. (For interpretation of the references in colour in this figure legend, the reader is referred to the web version of this article.)

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