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Evaluation of the nanoindentation behaviors of SiGe epitaxial layer on Si substrate

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ABSTRACT

In this paper, ultra-high vacuum chemical vapor deposition (UHV/CVD) was employed to synthesize silicon–germanium (SiGe), and sequence to endure annealing treatment. Morphological characterization, roughness, and microstructural morphology were observed by means of scanning electron microscopy (SEM), atomic force microscopy (AFM), and transmission electron microscopy (TEM). The elements distribution, crystallographic, and nanomechanical behavior were carried out using energy-dispersive X-ray spectroscopy (EDS) mapping technique, X-ray diffraction (XRD), and nanoindentation technique.

The annealing treated SiGe leads to the 2D germanium segregation on the surface. The phenomenon is interpreted in terms of dislocation-induced structural changes in annealing treatment. Thus, the dislocation propagation in the microstructure was observed. Subsequently hardness and elastic modulus were increased because of a comparatively unstable microstructure after annealing treatment.

1. Introduction

Silicon–germanium (SiGe) is one of the most attractive semiconductor materials because of its outstanding behaviours [1–3]. Compressively strained SiGe alloys can exhibit outstanding hole mobility predicted through theoretical calculations, which is due to the strain-induced heavy-hole/light-hole splitting. The holes show a light n-plane effective mass depending on alloys composition. Hence, hole mobility in SiGe alloys is predicted to be 3–10 times higher than in Si at the room temperature [3]. Therefore, SiGe alloys can be incorporated in either recessed S/D regions of PMOS to enhance drive current [4–6] or PMOS channel to enhance current gain [7–9]. Since then, SiGe have been applied to heterojunction bipolar transistor (HBT) as well as complementary metal-oxide-semiconductor (CMOS) [10–13]. However, there are several limiting factors for their applications including the long growth time, high material consumption, rough surface, and partial strain relaxation [14,15]. The high quality of the SiGe is required for devices application, that is to say, crystal defects and non-uniform composition are undesirable [16,17].

Due to large lattice mismatch between Ge and Si atoms (about 4.2%), strain relaxation induced crystal defects such as misfit dislocation and threading dislocation from interface [18,19], poor mechanical and morphological characteristic may occur in SiGe thin films. The poor thermal stability of the SiGe/Si heterostructures has probability of lowering the quality of SiGe. Meanwhile, this may degrade the performance of thin films as well as be an obstacle to the further developments. The intermixing caused by interdiffusion alters the interfacial properties and then degrades the film performance. Some groups have used different methods to change the material behaviours of SiGe films such as adding a Si buffer layer to decrease the dislocation density in the SiGe films [20], depositing the SiGe epilayers on SiGe substrate for reducing the lattice-mismatch by means of UHVCVD method [21], using annealing treatment to observe electrical conductivity [22], interdiffusion at SiGe/Si interface [23], density of crystal defects, and strengthening adhesion between SiGe and Si interface [24]. Relatively, the nanomechanical aspects for the defects induced change on the SiGe thin films and/or annealing treatment are yet to be reported.

In this study, we have employed the nanoindentation technique to investigate the defects induced phenomenon on the SiGe/Si heterostructures. As a consequence, surface morphology, roughness, and microstructure structures of SiGe/Si heterostructures were observed by using SEM, AFM and TEM. The elements distribution, and crystallographic were carried out using EDS mapping technique, and XRD.

2. Experimental procedure

The samples were prepared by a standard Radio Corporation of American (RCA) clean and a HF:H_2O (1:50) bath for 15 s, p-type

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Si(1 0 0) wafers were simultaneously introduced into load-lock chamber of ultra-high vacuum chemical vapor deposition (UHV/CVD) system. The deposition process are including three steps: (i) A 3-nm-thick Si buffer layer was deposited on the Si substrate at 500 °C for 30 min from pure SiH_4 (in 85 sccm) gas, the rate of deposition is 0.1 nm/min. (ii) A 500-nm-thick $Si_{0.8}Ge_{0.2}$ layer was deposited at 500 °C for 180 min from pure SiH_4 (in 85 sccm) and GeH_4 (in 15 sccm) mixing, the rate of deposition is 2.8 nm/min and the vacuum is achieved at 10^{-7} mbarr. (iii) In the annealing, the SiGe thin films are endured thermal treatments (400 °C and 500 °C), and *ex situ* in furnace on N_2 gas for 30 min.

Morphological characterization, roughness, and microstructure of the SiGe thin films were observed by means of scanning electron microscopy (SEM, Hitachi S-4000), atomic force microscope (AFM, Veeco D5000), and transmission electron microscopy (TEM, JEOL, JEM-2100F). From 3D patterns of AFM analysis, we mainly investigated two parameters: the height roughness parameters (R_a) and the root mean square (R_{ms}). In addition, TEM samples were prepared within mechanical polishing down to 20–30 μm, followed by Ar ion milling to electron transparency. The observations were made at 200 kV. The elements distribution and crystallographic were carried out using energy-dispersive X-ray spectroscopy (EDS) mapping technique and X-ray diffraction (XRD, PANalytical X'Pert Pro Inc. Singapore, with Cu K_α; $\lambda = 0.154$ nm).

Subsequently hardness and elastic modulus of the SiGe thin films was studied by using a Nano Indenter XP instrument (MTS Cooperation, Nano Instruments Innovation Center, TN, USA). The nanoindentation measurements used a diamond Berkovich indenter tip (tip radius ~50 nm), suggesting that plastic deformation can be generated at very small load. In addition, Hardness data obtained with Berkovich indenter can be transformed to Vickers hardness because of the same shape of a three-sided pyramid, which means a similar area-to-depth function [25,26]. The continuous contact stiffness measurement (CSM) mode executed by superimposing small oscillations on the force signal to record stiffness data along with load and displacement data dynamically, allows hardness and Young's modulus to be calculated at every data point acquired during the indentation experiment [27,28]. The instrument was calibrated by using a standard fused silica sample prior to measuring the mechanical characterizations of SiGe thin films. The drift rate preset to <0.05 nm/s before the beginning of each indentation. Frequency of 45 Hz was used to avoid the sensitivity to thermal drift and loading resolution was 50 nN [29]. Ten indents were made on samples to minimize the deviation of the results. The nanoindentations were sufficiently spaced (50 μm) to prevent from mutual interactions. In order to obtain "film-only" properties, a commonly used rule of thumb is to limit the indentation depth to less than 10% of the films thickness [27,28]. Hardness and Young's moduli were determined using the Oliver and Pharr analysis [27]. Hardness (H) means the resistance to local plastic deformation of materials, which has been conventionally obtained by measuring the projected contact area, A_c:

$$H = \frac{P}{A_c} \tag{1}$$

where P is the load. Elastic modulus E can be obtained from the contact stiff, using the following relation:

$$S = \beta \frac{2}{\sqrt{\pi}} E_r \sqrt{A_c} \tag{2}$$

$$\frac{1}{E_r} = \frac{1-\upsilon_i^2}{E_i} + \frac{1-\upsilon^2}{E} \tag{3}$$

where S is the initial unloading contact stiffness measured from the upper portion of the unloading data; β is a constant that depends on the geometry of indenter and $\beta = 1.034$ [29]; E_r stands for the reduced modulus; E_i and υ_i are Young's modulus and Poisson's ratio of indenter, respectively; E and υ are the same parameters for the specimen. The load and stiffness are directly measured during an indentation, contact area Ac and contact depth h_c has relation $A_c = 24.56h_c^2$ [30]. By inserting the calculated contacted area into Eqs. (1)and (2), the hardness and elastic modulus were evaluated. For diamond which is the usual material of a Berkovich indenter, $E_i = 1141$ GPa and $\upsilon_i = 0.07$ [27]. As the commonly done, we assume that υ is 0.3.

3. Results and discussion

In order to investigate the role of UHV/CVD grown SiGe thin films, annealing treatment was employed. The crystallographic structure of the SiGe thin films was obtained from HRXRD analysis. Fig. 1 shows the XRD spectra of the (0 0 4) reflection of the SiGe thin films, and the peak position of 67.925 (SiGe) and 69.128 (Si) are obtained. The composition of as-deposited SiGe thin films can be verified by comparing the measured rocking curves (Fig. 1a) and simulated X-ray rocking curves, which were deduced by diffraction theory [31]; therefore, Ge mole fraction of 20% was confirmed. Besides, the oscillation behaviors in this curve exhibit a high epitaxial of SiGe layer grown on Si substrate. Comparatively, the fringes fade out in Fig. 1b and c, which means that the interdiffusion-caused destroyed and broadening interfacial layer occurred after annealing treatment. The similar observation was also revealed by Ref. [17]. Furthermore, crystal quality can be estimated by observing the full width at half maximum (FWHM) of curve peak. Obviously, the FWHM of SiGe peaks increased after annealing treatment, indicating that poor crystal quality and strain relaxation appeared in the SiGe thin films [32,33]. The SiGe/Si heterostructures have highly focused in its strain-relaxed performance from annealing treatment. The intermixing alters the interfacial properties and then degrades the film performance. In addition, the SiGe thin films with 500 nm in thickness exceeds the theoretical equilibrium critical thickness and is in a metastable condition [34,35].

The height roughness parameters (R_a) and the root mean square (R_{ms}) can be used to show morphology condition as part of the quantitative analysis of AFM images [36]. We employed the AFM technique to identify the morphology of the SiGe thin films, typical measurement on a series of annealing procedure are shown in Figs. 2, in which the surface roughness and apparent feature size can be observed. The SiGe thin films characterized by a smooth manner (R_{ms} is 0.3 nm) which gradually roughens from 400 to 500 °C (R_{ms} are 4.1 nm and 7.3 nm), even if surface roughness increases rapidly at the extra thermal budget, this assumed to relieve the strain [34,37,38]. To evaluate the annealing effect, Table. 1 lists the summary of R_a and R_{ms} at different annealing temperatures. It is noted that parameters of R_a and R_{ms} increased at the annealing process and the results were agreeable with that of Zheng et al. [23]. Meanwhile, Tételin et al. [39] reported that Ge segregation and strain relaxation can be investigated to the formation of islands on the surface. Herein, the 2D state results in Fig. 2 at the annealing temperatures (400–500 °C) are in good agreement with the similar observations from Ref. [39].

In order to investigate the interdiffusion and strain alteration of the SiGe thin films, the SEM and relative EDS mapping were employed. The surface EDS mapping for the SiGe thin films and annealing in furnace environment using nitrogen as following gas are shown in Fig. 3, where (a) is the image of the sample of surface SiGe thin films grown directly on the Si(0 0 1) substrate without annealing process, and (b–c) are the images of the SiGe thin films after annealing. The surface SiGe thin films have high concentration of Si element than the Ge composition. Beside, Ge composition

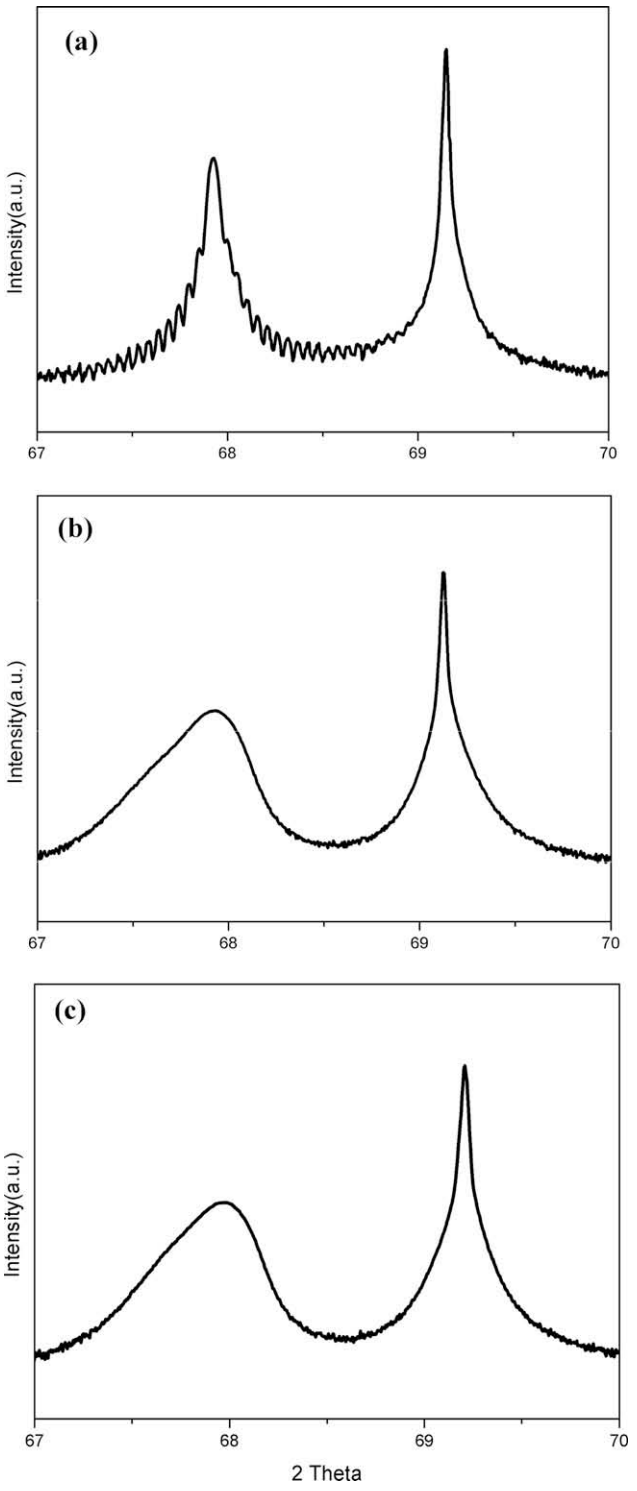

Fig. 1. The XRD rocking curve for 500 nm of the SiGe thin films deposited on Si substrates with various anneals: (a) no anneal, (b) 400 °C for 0.5 h, and (c) 500 °C for 0.5 h.

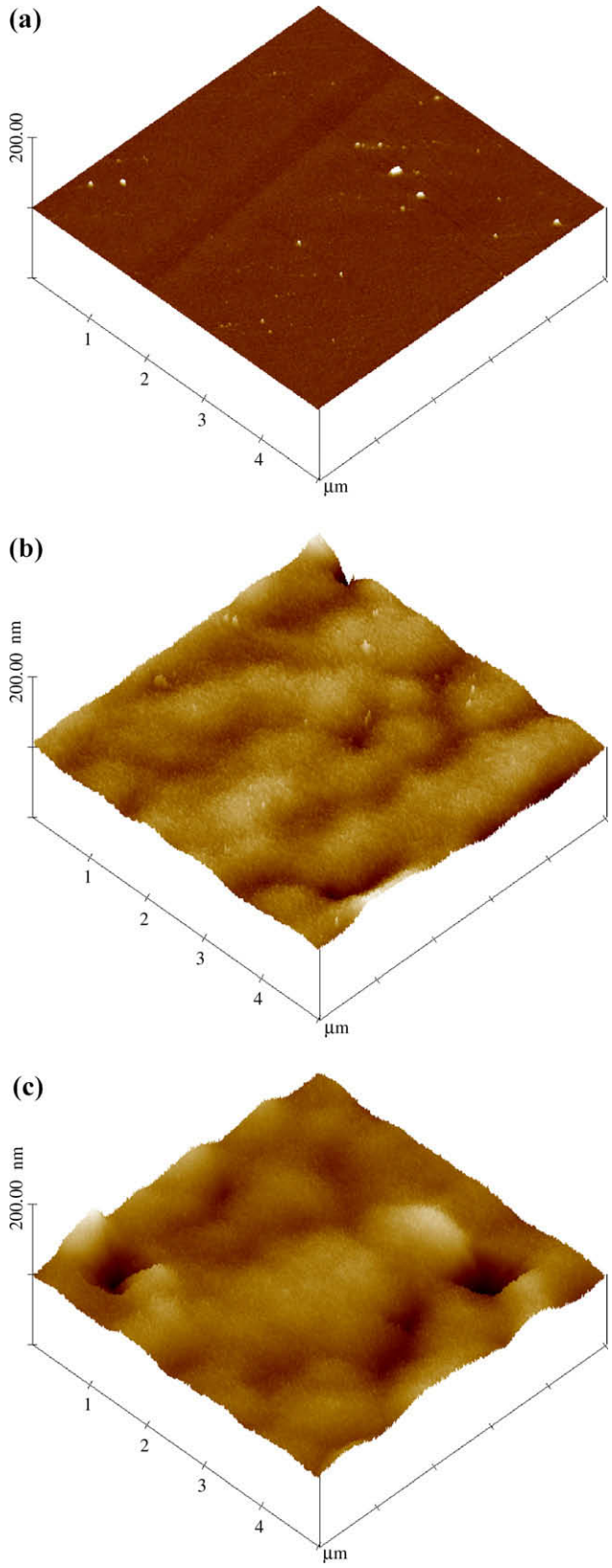

Fig. 2. AFM images of surface topography of samples; (a) before annealing and (b) after annealing at 400 °C for 0.5 h, and (c) 500 °C for 0.5 h. The averaged surface roughness values (R_{ms}) of the SiGe thin films are 0.3, 4.1, and 7.3 nm, respectively.

is suddenly increased upon to surface when annealing at $T = 500$ °C (Fig. 3c). Furthermore, the cross-section EDS mapping are shown in Fig. 4, where (a) is the Si and Ge element for the SiGe thin films without annealing process, and (b,c) is the SiGe thin films within annealing process, the heavy concentration of Ge element was transited into the surface of sample after annealing treatment. It is conjectured that annealing temperature plays an important role in promoting the interdiffusion between the SiGe thin films and Si substrate [40,41]. A serious interdiffusion in the SiGe thin films corresponds to the supporting of the energetically thermal motion

Table 1
Variation of average surface roughness (R_a) and root-mean-square surface roughness (R_{ms}) at various annealing temperatures.

Sample	Pretreatment temperature (°C)	Average roughness, R_a (nm)	Root mean square roughness, R_{ms} (nm)
(a)	–	0.2	0.3
(b)	400	3.3	4.1
(c)	500	5.1	7.3

(Fig. 4c). Thus, the different roles between Si and/or Ge element in the SiGe thin films were revealed.

Specifically, the nanoindentation technique is useful in probing the properties of thin films. The SiGe/Si heterostructures can be investigated from continuous penetration depth by means of continuous stiffness measurement (CSM) in nano-size. Figs. 5 and 6 show hardness and Young's moduli of the SiGe/Si heterostructures as functions of the indentation depth at 200 nm, following the method proposed by Oliver and Pharr [27]. For indentation depths up to about 15 nm, the hardness increased as the indentation depth increased, which is usually attributed to the transition between purely elastic to elastoplastic contact whereby the hardness is actually the contact pressure. For indentation depths greater than about 15 nm, the hardness became constant. Young's modulus followed a trend similar to that of the hardness except that its magnitude converged at an indentation depth smaller than that for hardness. Hardness and Young's moduli were therefore determined by averaging measurements at indentation depths from 100 to 200 nm, considering an adequate depth to achieve a fully developed plastic zone and meanwhile not exceeding 40% of the film thickness to avoid the more substrate effect [42]. For the measured results of the SiGe/Si heterostructures, hardness were 13.9 ± 0.7, 15.1 ± 0.3, and 15.2 ± 0.5 GPa, while Young's moduli were 190.4 ± 7.9, 205 ± 3.2, and 207 ± 5.3 GPa, respectively. Besides, It is mentionable that the oscillation and discontinuous phenomenon observed in hardness curve of as-deposited SiGe thin films may be pop-ins events caused by shear-induced dislocation slip and twinning [43,44].

After inspecting the enhancement in hardness and Young's moduli through annealing treatment of the SiGe/Si heterostructures, the microstructures are subsequently investigated. The relative defects induced mechanical properties change of the SiGe thin films are examined in microstructure observation. Fig. 7 shows that TEM profile, where (a) is the smooth interface of the SiGe thin films/Si substrate, and (b,c) is the subsequent annealing treatment that had significant interdiffusions from the misfit dislocations. This tends to form a serial nucleation seed and induces high density dislocation occurring at the interface, therefore to bring a serious slip line in our investigations. This observation is consistent with the LeGoues et al. reported [45] that the SiGe structures grown by UHV/CVD at low temperature relax by a modified Frank–Reed mechanism. The dislocations are formed by the reproduction of corner dislocations. Also, Mooney et al. reported [15] that the threading segments of dislocations annihilate and the relaxed SiGe films have low threading dislocation densities. Therefore, in the large mismatch about 4.2% between Si and Ge, the growth of nearly dislocation-free SiGe films is a major reason. In the 500 nm of SiGe layers in excess of the critical thickness for dislocation nucleation considered in these studies unexpectedly show few dislocation nucleation events. Evidently, all the samples in our experiment were endured 500 °C thermal treatment for 180 min during the growth process. This thermal treatment time was 6 times longer than that in post-treatment (30 min) and the temperature is even higher comparing with post-treatment at 400 °C.

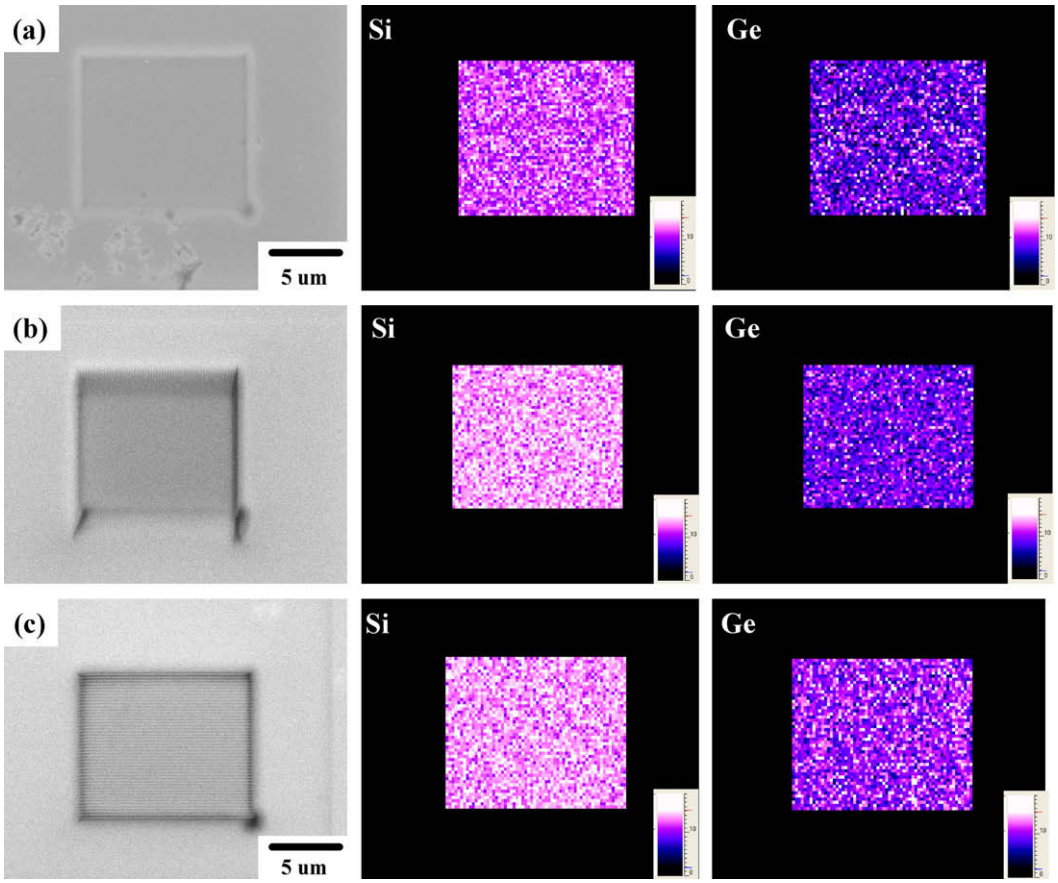

Fig. 3. The plane-view of EDS mapping analysis with the SiGe thin films samples: (a) no anneal, (b) 400 °C for 0.5 h, and (c) 500 °C for 0.5 h.

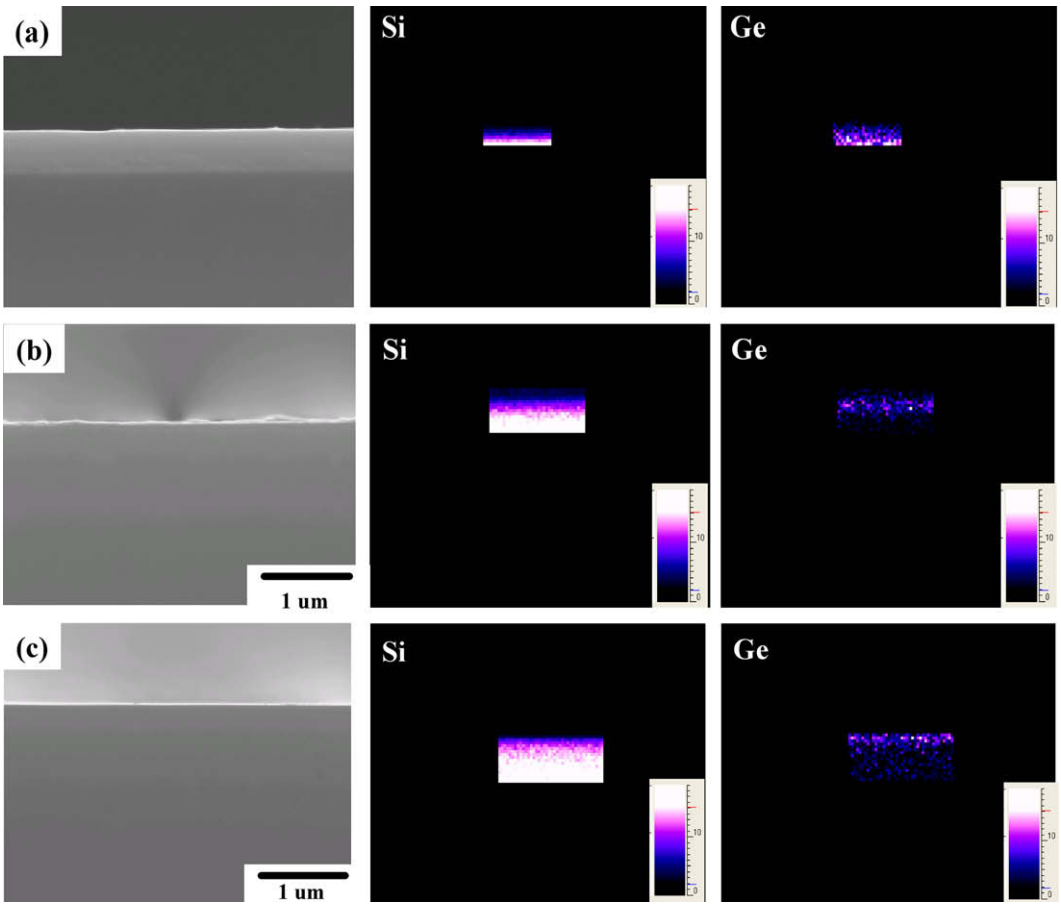

Fig. 4. The cross-sectional EDS mapping analysis with the SiGe thin films samples: (a) no anneal, (b) 400 °C for 0.5 h, and (c) 500 °C for 0.5 h.

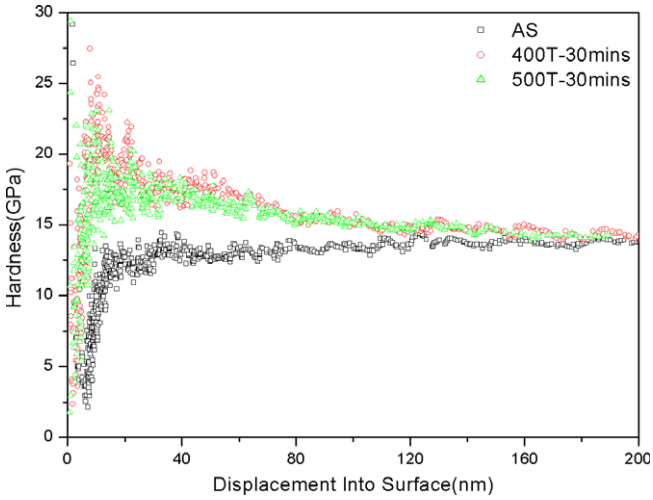

Fig. 5. The hardness of the SiGe thin films samples: (a) no anneal, (b) 400 °C for 0.5 h, and (c) 500 °C for 0.5 h.

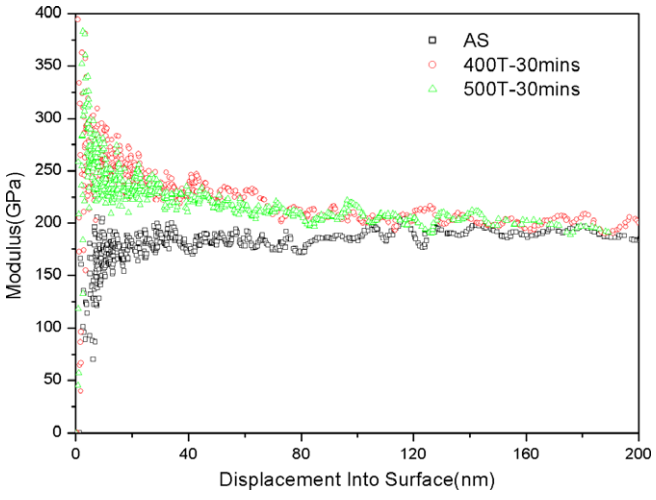

Fig. 6. The Young's moduli of the SiGe thin films samples: (a) no anneal, (b) 400 °C for 0.5 h, and (c) 500 °C for 0.5 h.

Nevertheless, Fig. 1 and particularly Fig. 7 show drastic difference between non annealed and annealed samples. It is because that in our study, to obtain a good quality of SiGe films with a thickness of 500 nm, the 500 °C thermal-budget for 180 min is necessary [46,47]. Besides, the films grown with these parameters were in a metastable condition [48], which means SiGe films can relax easily with post thermal treatment, especially while the films is under non-ultra-high vacuum environment.

As mentioned above, while the dislocation occurred on the SiGe thin films, this accompanied with the enhancement in its mechanical resistance against elastic and elastoplastic deformations. This phenomenon can be observed from Figs. 5 and 6 with the indentation depths below 50 nm, which can reflect "film-only" properties. From above analysis, temperature increased after annealing treatment not only leads nucleation seed but also enhances the hardness and Young's moduli of the SiGe thin films.

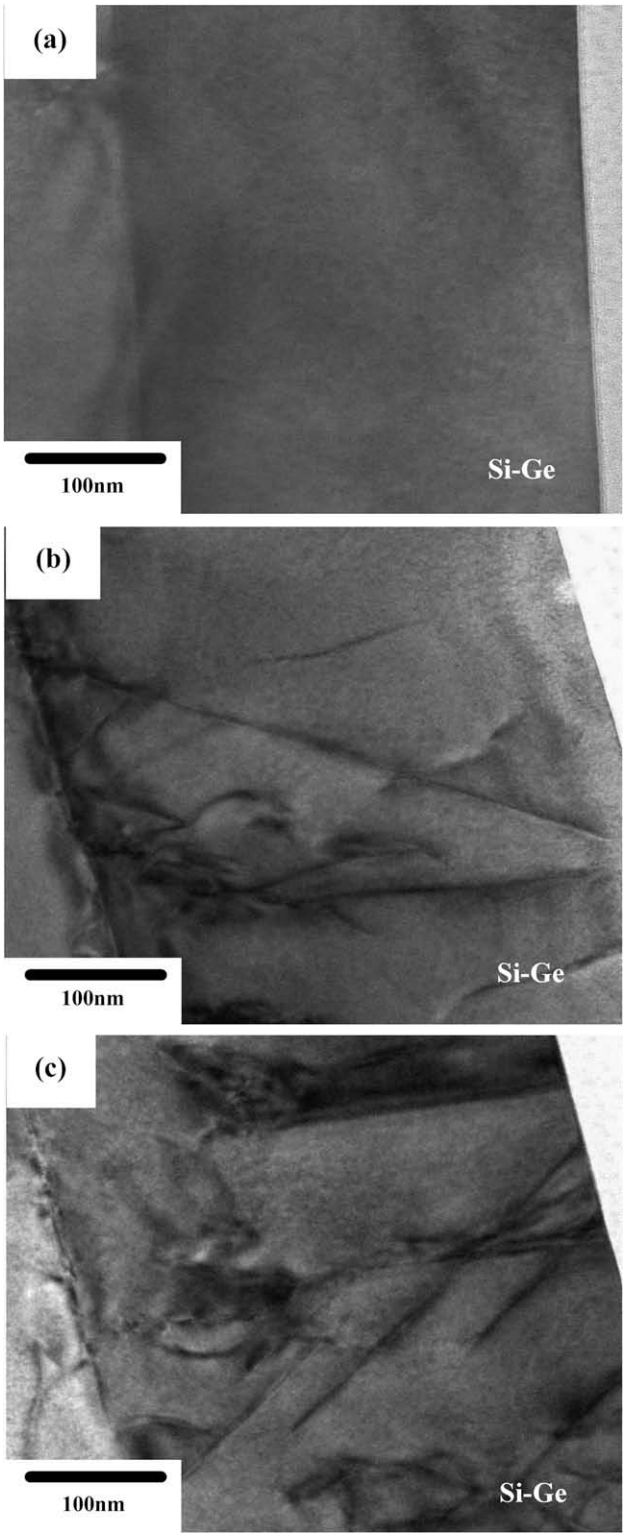

Fig. 7. The cross-sectional TEM image of the SiGe thin films samples: (a) no anneal, (b) 400 °C for 0.5 h, and (c) 500 °C for 0.5 h.

4. Conclusions

The materials analysis and nanoindentation techniques have been used to investigate surface features and nanomechanical properties of the SiGe thin films. The XRD analysis showed that the SiGe thin films featured a crystalline nature. As the annealing treatment, the SiGe thin film became predominantly oriented along the (0 0 4) peaks of position of 67.925 (SiGe) and 69.128 (Si) and the surface roughness increased. The 2D germanium segregation on the surface was observed from AFM analysis. It is also obviously observed that the smooth manner (R_{ms} is 0.3 nm) gradually roughens from 400 °C to 500 °C (R_{ms} are 4.1 nm and 7.3 nm). Results from Berkovich nanoindentation indicated that the hardness of the SiGe thin films with annealing treatment ranged from 13.9 ± 0.7 to 15.2 ± 0.5 GPa while the Young's modulus ranged from 190.4 ± 7.9 to 207 ± 5.3 GPa. The SiGe thin films were shown to slightly enhance mechanical properties due to the misfit dislocation propagation from thermal annealing.

Acknowledgments

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