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Low-Dislocation Relaxed SiGe Grown on an Effective Compliant Substrate

Y.H. LUO,[1,3] J.L. LIU,[1] G. JIN,[1] K.L. WANG,[1] C.D. MOORE,[2] M.S. GOORSKY,[2] C. CHIH,[2] and K.N. TU[2]

1.—University of California, Device Research Laboratory, Department of Electrical Engineering, Los Angeles, CA 90095-1594. 2.—University of California, Department of Materials Science and Engineering, Los Angeles, CA 90095-1595. 3.—e-mail: yuhao@ee.ucla.edu

An effective compliant substrate was successfully fabricated for growth of high quality relaxed SiGe templates. The compliant substrate was fabricated by synthesizing a 20% B_2O_3 concentration borosilicate glass in the silicon on insulator wafers through boron and oxygen implantation followed by high temperature annealing. Substrates with 5%, 10% and 20% B_2O_3 were used for 150 nm $Si_{0.75}Ge_{0.25}$ epitaxy. Double-axis x-ray diffraction measurements determined the relaxation and composition of the $Si_{1-x}Ge_x$ layers. Cross-sectional transmission electron microscopy was used to observe the lattice of the SiGe epilayer and the Si substrate, dislocation density and distribution. Raman spectroscopy was combined with step etching to measure the samples. For 20% BSG sample, the strain in the thin Si layer was calculated from the Raman shift and it matched the results from DAXRD very well. The density of threading dislocation on the surface of 500 nm $Si_{0.75}Ge_{0.25}$ layers was 2×10^4 cm^{-2} for the sample on the 20% borosilicate glass substrate. This method is promising to prepare effective compliant substrate for low-dislocation relaxed SiGe growth.

Key words: Compliant substrate, relaxed, SiGe, MBE

INTRODUCTION

Recently, high mobility strained Si, $Si_{1-x}Ge_x$, and Ge have attracted considerable attention for their potential applications in high frequency devices.[1,2] This has stimulated considerable interest in relaxed SiGe buffer layers, which are used as "virtual substrates" for the growth of high electron mobility transistors and the integration of III-V devices on Si.[3,4] However, the large lattice mismatch (~4.17%) between Si and Ge results in a high density of dislocations in SiGe buffer layers with high Ge content. Moreover, threading dislocations propagate through the SiGe buffer layer into the active layers and thus deteriorate the device performance.[5,6] To date, several strategies have been explored to prepare relaxed SiGe layers minimizing the threading dislocation density, such as a graded composition growth[7] and a low temperature buffer layer.[8] However, these layers have to be relatively thick (~µm) in order to avoid high dislocation density. In some cases, these schemes offer inadequate improvement of material quality to realize significant dividend to device performance.

The concept of compliant substrate was proposed by Lo in 1991[9] and was later realized experimentally by Powell et al. in 1994.[10] The ideal compliant substrate is a freestanding thin substrate, which would elastically accommodate the misfit strain and absorb threading dislocations. In practice, however, a thin membrane acting as a substrate is prohibitive. Currently, there are several approaches to fabricate a compliant substrate by wafer bonding.[11,12] The thickness of the silicon on insulator (SOI) layer has been reduced to the nanometer regime and successfully used as compliant substrate for GaN and SiGe epitaxy.[13–15] In the case of SiGe, the SiO_2 is expected to be rigid at the growth temperature range necessary for SiGe epitaxy (typically between 450°C and 700°C). The inability of the SOI layer to deform at these substrate temperatures limits the possibility of the Si layer to share the misfit strain during epitaxy. As a result, the relaxed SiGe layers grown on these kinds of substrates exhibited a large number of dislocations.[13] Therefore, to achieve a better compliant sub-

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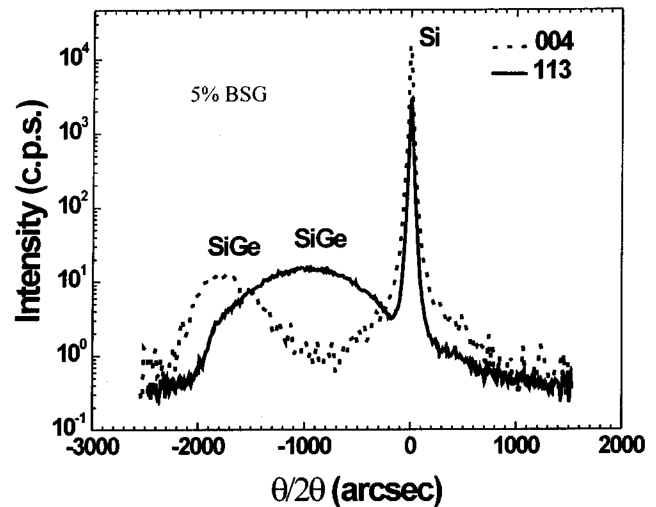
strate, it is necessary to promote the *in-situ* accommodation of misfit. This could be realized provided that the SOI could deform at lower growth temperatures. It has been well known that the addition of B_2O_3 in SiO_2 (borosilicate glass) decrease the viscosity of the SiO_2.[16] To have a 15 Poise viscosity, which is roughly the critical viscosity for the strain to transfer from the SiGe epilayer to the thin Si layer,[17] the growth temperature should be 1100°C for SiO_2, but 500°C for 20% BSG.[16] In this work, the substrates with different concentrations of B_2O_3 in the SiO_2 layer of SOI wafers were fabricated, which were called as BSG substrates. The compliant effect for SiGe growth of the BSG substrates was reported.

EXPERIMENT

An effective compliant substrate was successfully fabricated and used for high quality SiGe growth, which acted as a freestanding thin substrate during the SiGe growth and shared the misfit strain with $Si_{1-x}Ge_x$. The SOI substrates in this work were bonding and etch back SOI (BESOI) wafers produced by Sibond, Inc. The fabrication process of our borosilicate glass (BSG) substrates was as follows: SOI wafers with a 60 nm Si layer and a 400 nm buried SiO_2 were implanted with boron and oxygen with the implant dosage and energy selected according to the stoichiometrical ratio of B_2O_3 and the target concentration by weight.[18] The implantation energy was chosen to place the BSG layer in the SiO_2 layer approximately 5 nm to the Si/SiO_2 interface. After implantation, a two-step annealing process was performed to form the single crystal Si layer and the BSG layer. Solid phase epitaxy was performed at 500°C for 2 h in nitrogen ambient. The wafers were subsequently annealed at 900°C for 5 h in nitrogen ambient to form the borosilicate glass as well as minimize implantation damage. The top Si layer was thinned down to about 20 nm by thermal oxidation and then 2% HF dip. The SOI thickness was confirmed by x-ray reflectivity measurements. The surface morphology was analyzed by atomic force microscopy and found suitable for molecular beam epitaxy with the root mean squared (rms) roughness measured as 0.3 nm. The substrates were cleaned and immediately loaded into a Perkin-Elmer MBE system for SiGe epitaxy.

BSG substrates with three weight ratios, 5%, 10%, and 20% B_2O_3, were used for SiGe epitaxy. For comparison, a SOI and a planar Si substrate were grown simultaneously. The epitaxy sequence consisted of a 10 nm Si buffer layer and a 150 nm $Si_{0.75}Ge_{0.25}$ layer. The growth temperature for both layers was maintained at 500°C. The nominal deposition rate of Si and Ge were 1.2 Å/s and 0.4 Å/s, respectively. The sample grown on the 5% BSG substrate was named as 5% BSG sample in this paper, and it was the same way for other samples.

Symmetric (004) and asymmetric (113) double-axis x-ray diffraction (DAXRD) measurements were used to determine the composition and relaxation of the SiGe layers. Cross-sectional transmission electron



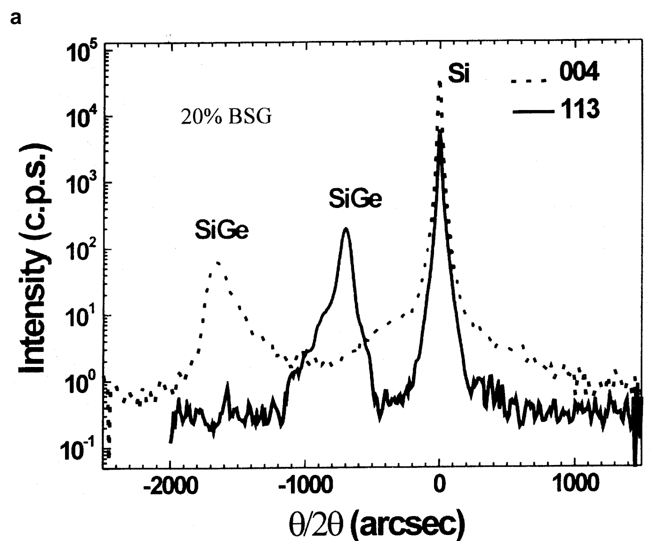


Fig. 1. Symmetric (004) and asymmetric (113) DAXRD measurements for (a) the as-grown 5% BSG sample, (b) the as-grown 20% BSG sample. The structure of the sample is 150 nm $Si_{0.75}Ge_{0.25}$ /10 nm Si/ 20 nm Si/BSG.

microscopy (XTEM) was used to observe the dislocation distribution and estimate the dislocation density. The measurement was performed using Philips CM200 FEG microscope and all the images shown here are bright field cross-sectional [110] images.

Raman spectroscopy was used to measure the strain in the thin Si layer. Raman spectroscopy has been proved to be sensitive to strain in semiconductor hetero-structures as a contactless probing techique.[19,20] Strain induced frequency shifts in the Si-Si longitudinal optical (LO) phonon modes of the thin Si layer was used to determined the strain in the Si layer. In this work, Raman spectra were taken with a microscope entrance, giving 0.7 μm laser spot on the sample. The exciting source was a 457.9 nm Ar^+ laser line.

RESULTS AND DISCUSSION

DAXRD symmetric (004) and asymmetric (113) measurement results for the as-grown 5% BSG sample and 20% BSG sample are shown in Fig. 1. From the

Table I. The Ge Compositon, Relaxation of SiGe, and FWHM of the (004) Scan SiGe Peak from DAXRD for As-Grown 5% BSG Sample and 20% BSG Sample, and 850°C Annealed Samples

Sample	Ge Composition (x)	Relaxation (%)	FWHM of SiGe Peak for (004) (arcsec)
As-grown 5% BSG	0.28 ± 0.02	79 ± 10	590
Annealed 5% BSG	0.28 ± 0.02	99 ± 3	610
As-grown 20% BSG	0.23 ± 0.01	64 ± 3	140
Annealed 20% BSG	0.23 ± 0.01	85 ± 2	233

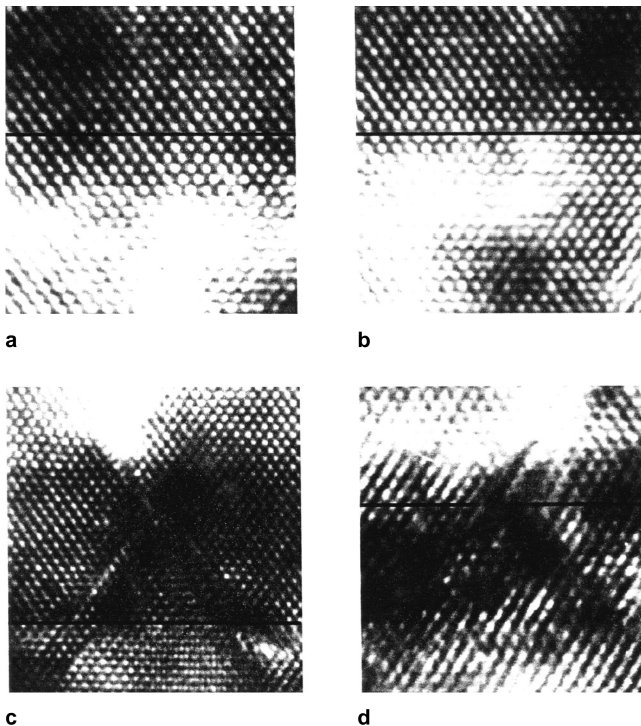


Fig. 2. Bright field [110] cross-sectional HRTEM image of (a) the as-grown 5% BSG sample, (b) the 5% BSG sample annealed at 850°C for 30 min in nitrogen ambient, (c) the as-grown 20% BSG sample, (d) the 20% BSG sample annealed at 850°C for 30 min in nitrogen ambient. The line marks the SiGe/Si interface.

Fig. 3. Bright field [110] XTEM image of (a) the as-grown 5% BSG sample, (b) the 5% BSG sample annealed at 850°C for 30 min in nitrogen ambient, (c) the as-grown 20% BSG sample, (d) the 20% BSG sample annealed at 850°C for 30 min in nitrogen ambient.

peak separation between the SiGe and Si substrate, the Ge composition and the relaxation of the SiGe layer are calculated. The scans were repeated after rotation by 180° and no tilting between the epilayer and substrate was observed. This technique of x-ray diffraction allows measurement of the relaxation independently of the dislocation density measurement, which could be estimated by XTEM.

The DAXRD results for the as-grown and annealed samples are summarized in Table I. As determined from the XTEM images, the thickness of the SiGe layer in both of the two samples is about 150 nm, which allows a direct comparison of the two samples. For the 5% BSG sample, due to the presence of misfit dislocations at the SiGe/Si interface, the SiGe peaks are broadened as a result of mosaic structures. After the annealing, the SiGe layer become fully relaxed, just as SiGe on Si substrate. For the 20% BSG sample, the much smaller full width of half maximum (FWHM) of the SiGe peaks indicates a much lower density of misfit dislocation, which will be confirmed by followed XTEM results. The reason could be that the thin Si layer became tensily strained during growth and then there would be much less misfit between the Si layer and the SiGe layer. The strain in the thin Si layer will be calculated from the Raman shift later. The strain in the thin Si layer after strain equalization of the bilayer will be calculated theoretically to confirm the strain sharing between the SiGe layer and the Si layer, too.

The quality of the SiGe layer is compared further by XTEM and the images are shown in Figs. 2 and 3. Figure 2 shows high-resolution XTEM (HRTEM) images of the as-grown 5% BSG sample (a) , the 5% BSG sample after 850°C annealing for 30 min in nitrogen ambient (b), and 20% BSG sample (c) and (d), respectively. Figure 3 shows XTEM images of the as-grown 5% BSG sample (a), the 5% BSG sample after 850°C annealing for 30 min in nitrogen ambient (b), and 20% BSG sample (c) and (d), respectively.

From careful measurement of the inter-atomic spacing, it is found that for as-grown 5% BSG sample there is lattice mismatch between the epi-layer and the Si layer (see Fig. 2a). A misfit dislocation and stacking fault can be observed in the SiGe/Si interface region too. This is expected from the DAXRD result. In Fig. 2b, it is found that after annealing the mismatch

becomes bigger than that of the as-grown sample, which matches the results from DAXRD that after annealing the SiGe layer relaxes more and SiGe peaks become wider. However, in Fig. 2c of the as-grown 20% BSG sample, every {100} double layer of the Si substrate continues into the SiGe layer, which implements that the SiGe layer makes a coherent interface with the Si layer. It normally means that the SiGe is fully strained. However, from the result of DAXRD, the SiGe layer is about 60% relaxed. Therefore, it is believed that the Si substrate expands during the growth to match the SiGe layer and there is no lattice mismatch between the Si and the SiGe layer. After annealing, as shown in Fig. 2d, no mismatch between the SiGe and the Si is observable from the inter-atomic space. However, as a stacking fault is observed in the HRTEM image, it is believed that during the annealing, the strained Si layer relaxed partially and stacking faults form in it.

In the following, all densities of misfit dislocations are estimated from several XTEM images with different magnifications. The range of observation is about 5 μm. For the as-grown 5% BSG sample, as shown in Fig. 3a, the diffuse contrast arises from the misfit dislocations and corresponding stacking faults are clearly observed in the SiGe/Si interface. The density of misfit dislocations is estimated to be 2×10^5 cm^{-1}. After annealing at 850°C for 30 min, the density of misfit dislocations at the SiGe/Si interface increases to about 4×10^5 cm^{-1}, as shown in Fig. 3b. For the 20% BSG sample, as shown in Fig. 3c, no misfit dislocations are observed at the interface of the SiGe layer and Si layer in all images, suggesting that the density is below 2×10^3 cm^{-1}. After annealing at 850°C for 30 min, as shown in Fig. 3d, the density of misfit dislocations at the SiGe/Si interface increased to about 1×10^4 cm^{-1} and a threading dislocation is observed to propagate downwards into the Si buffer layer.

As well known, the plastic strain relief δ due to the misfit dislocations with density ρ_{md} is given by $\delta = b_{eff} \times \rho_{md}$. b_{eff} is the effective Burger's vector, the component of the Burger's vector responsible for misfit strain relief. For 60° dislocation in SiGe, b_{eff} is $a_{SiGe}/2\sqrt{2}$. For fully relaxed $Si_{0.75}Ge_{0.25}$ on Si substrate, as $\delta = 4.17\% \times 0.25 = 1.04\%$, if the strain is completely released by plastic, i.e., due to the formation of misfit dislocations, then $\rho_{md0} = 5.36 \times 10^5$ cm^{-1}. For partially relaxed $Si_{0.75}Ge_{0.25}$ with R% relaxation, $\rho_{md} = \rho_{md0} \times R\%$.

For the 5% samples, the measured misfit dislocation densities are little lower than calculated values. The reason is that the threading dislocations release part of the mismatch. In the XTEM images, stacking fault contrast decorates the region of the SiGe/Si interface. Threading dislocations propagate downwards into the Si layer, as well as upwards into the SiGe layer. The measured misfit dislocation densities for 20% sample are much lower than calculated values, too. However, no threading dislocation is observed in the SiGe layer and the Si layer. The reason for fewer misfit dislocations is that the {100} lattice of the thin Si substrate expands to become tensily strained to match that of the SiGe layer. HRTEM already shows that the lattice of the SiGe and the Si are coherent for the as-grown 20% BSG sample. The strain in the Si layer will be confirmed by the followed Raman results.

In the above, we speculated that the thin Si layer became strained during the growth of 20% BSG sample. To confirm the strain in the thin Si layer buried below the SiGe layer, Raman spectroscopy was combined with step etch to measure the samples. The

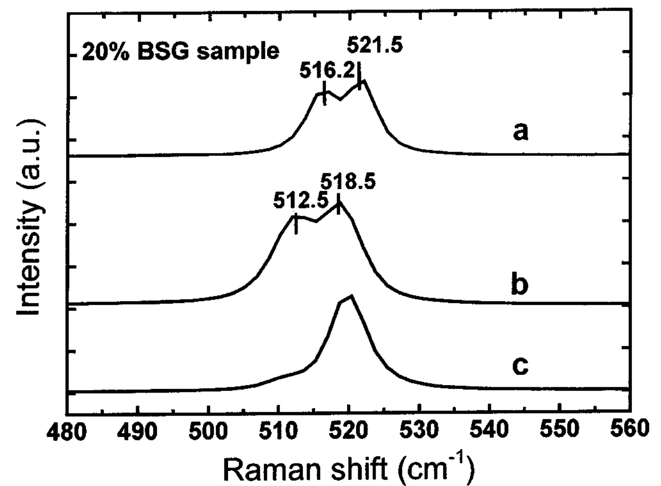


Fig. 4. Raman spectral from the as-grown 20% BSG sample without etching and after etching: (a) without etching, and (b), (c) after etching steps.

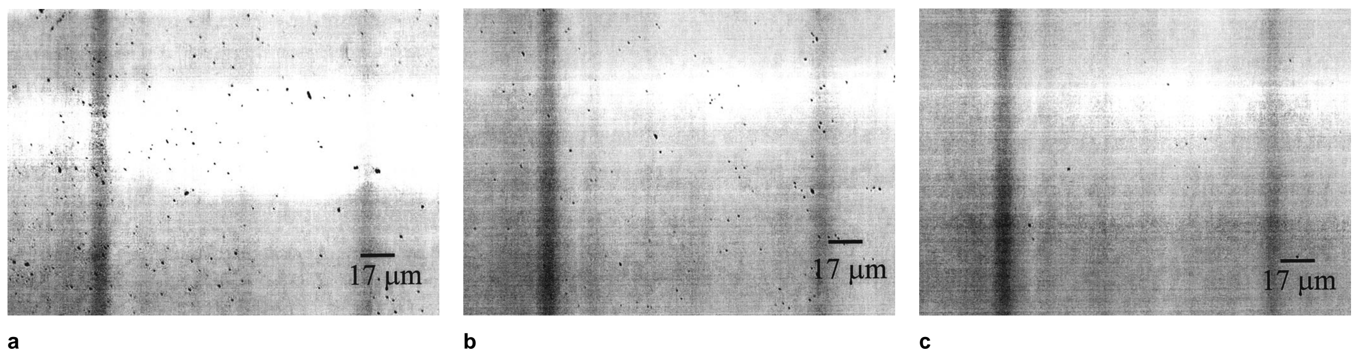


Fig. 5. Nomarski micrograph showing etch pits for the 500 nm $Si_{0.75}Ge_{0.25}$ grown on (a) 5% BSG substrate, (b) 10% BSG substrate, and (c) 20% BSG substrate.

Raman spectra of the 20% BSG sample are shown in Fig. 4. The peak positions marked in the figure are obtained from the Lorentz curve fit. For the as-grown 20% sample without etch, two peaks at 516 cm^{-1} and 521.5 cm^{-1} are observed. The 516 cm^{-1} peak could be assigned to the Si-Si LO mode of SiGe layer. However, this Raman shift 516 cm^{-1} is much larger than the reasonable value according to the Ge composition (506~512 cm^{-1} for x~0.25). Therefore, it is suspected that the 516 cm^{-1} peak is formed by the overlap of the peaks from the strained thin Si layer and the SiGe layer. So the step etching method was used to etch away the SiGe layer step by step. $HF:HNO_3:H_2O$ solvent was used. Figure 4a is the spectral from the as-grown 20% sample without etch. After some etch, as shown in Fig. 4b, two peaks appear at 512.5 cm^{-1} and 518.5 cm^{-1}. These two peaks are believed belonging to the Si-Si LO mode of the SiGe layer and the strained Si layer, respectively. After even more etch, the intensity ratio of the 512.5 cm^{-1} to 518.5 cm^{-1} dropped significantly, which confirms the sources of the peaks further. Using the formula from literature, the strain in the thin Si layer was calculated from the shift of the Si-Si peak too.[19] For the 3 cm^{-1} shift $\Delta\omega$, using $\Delta_{Si} = 34$ cm^{-1}, the normalized strain Σ is $\Delta\omega/\Delta_{Si} = 0.088$. As the full (100%) mismatch strain between pure Ge and pure Si makes $\Sigma = 1$, considering the Ge composition 0.23, the normalized strain in the thin Si layer was 0.088/0.23 = 38.3%. Assuming the strain of the Si layer accommodates all the relaxation of the SiGe layer, then the relaxation of the SiGe layer is about 1–38.3% = 61.7%. This value is quite close to the 64% obtained from the DAXRD measurement. The as-grown Si and 5% BSG samples were measured in the same way. No shift of the Si-Si LO peak at 521.5 cm^{-1} was observed. The Si-Si mode of the SiGe layer kept at the ~512 cm^{-1}. Therefore, the thin Si layer of the 20% BSG substrate works as a compliant substrate to accommodate part of the mismatch strain as the BSG is soft at the temperature and the Si layer becomes strained during the growth.

From the strain sharing model,[21] the strain for the Si ($\in_1$) and SiGe ($\in_2$) films is given by:

$$\in_1 = \frac{B_2 h_2 (1/a_1 - 1/a_2)}{a_2 (B_1 h_1 / a_1^2 + B_2 h_2 / a_2^2)}$$

and

$$\in_2 = \frac{B_1 h_1 (1/a_2 - 1/a_1)}{a_1 (B_1 h_1 / a_1^2 + B_2 h_2 / a_2^2)}$$

where a_1 and a_2 are the relaxed (cubic) lattice constants of Si and SiGe, respectively; and h_1 and h_2 are the thickness of Si and SiGe from XTEM images, 30 nm and 150 nm, respectively; B_1 and B_2 are material constants given by $2G_i(1+\nu_i)/(1-\nu_i)$ (i = 1~2), where G_i and ν_i are the shear modulus and Poisson's ratio of Si and SiGe, respectively. To minimize the total energy $E = B_1 \in_1^2 h_1 + B_2 \in_2^2 h_2$, the two films will reach a common lattice constant a_0. The lattice constants and elastic constants of Si and Ge from published data[22] are used and the corresponding constants for SiGe are interpolated. The equilibrium strain ratio of the mismatch strain in the Si layer is then calculated to be 32.6%. This value is close to the 38.3% obtained from the Raman results for the 20% BSG sample. Thus, for the 20% BSG sample, the misfit strain is believed to equalize between the SiGe layer and the Si layer during the growth.

The final purpose of our compliant substrates is to grow high quality relaxed SiGe films, especially with low density of threading dislocations. Thus, thick SiGe sample was grown on different substrates to check the performance of the compliant substrate. The sample was grown on 5%, 10% and 20% BSG substrates at the same time. The epitaxy sequence consisted of 10 nm Si buffer layer and a 500 nm $Si_{0.75}Ge_{0.25}$ layer. The growth temperature for both layers was maintained at 500°C. Material quality was evaluated by a modified Schimmel etch[23] to reveal crystalline defects and observed with Nomarski interference microscopy. The micrographs are shown in Fig. 5. The density of etch pits are estimated to be 4×10^5 cm^{-2}, 1.5×10^5 cm^{-2}, and 2×10^4 cm^{-2} for SiGe grown on 5%, 10%, and 20% BSG substrates, respectively. The much lower density of etch pits on the surface of SiGe layer grown on the 20% BSG substrate is due to the strain sharing effect of the compliant substrate during the growth. As the result of the straining sharing effect of the substrate, the lattice mismatch between the epi-layer and the substrate become much less and fewer defects form during the growth.

CONCLUSIONS

In summary, an effective compliant substrate was successfully fabricated by forming 20% concentration B_2O_3 borosilicate glass in a SOI wafer and thinning down the Si layer on top of the borosilicate glass. It was observed that the compliant substrate accommodated part of the misfit strain between the SiGe layer and the Si layer during the growth. It was also observed that the defect density decreased significantly for SiGe layer grown on 20% BSG compared with that grown on 5% BSG and 10% BSG substrates. This kind of substrate therefore has the potential to provide low dislocation density growth of relaxed SiGe layers for device applications.

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