CHIN. PHYS. LETT. Vol. 26, No. 4 (2009) 048103

Highly Strained Si Films with Ultra-low Dislocation Density Grown on Virtual Substrates of Thin Thickness *

YANG Hong-Bin(杨鸿斌), ZHANG Xiang-Jiu(张翔九)**

Surface Physics Laboratory (National Key Laboratory), Fudan University, Shanghai 200433

(Received 13 November 2008)

By using compositionally graded SiGe films as virtual substrates, tensile strained Si films with the strain of 1.5% and the threading dislocation density less than $1.0 \times 10^5\ cm^{-2}$ are successfully grown in micron size windows by molecular beam epitaxy (MBE). The thickness of the virtual substrates was only 330 nm. On the surface of the s-Si films no cross-hatched lines resulting from misfit dislocations could be observed. We attribute these results to the edge-induced strain relaxation of the epitaxial films in windows, and the patterned virtual substrates with compositionally graded SiGe films.

PACS: 81.15.Hi, 61.72.Ff, 78.30.Er

Strained Si (s-Si) films have attracted much interest in recent years for their importance in the applications of Si-based high-speed devices,[1−3] because their carrier mobility can be greatly enhanced due to the reduced effective mass and lower intervalley scattering. Usually the s-Si films require high strain, low dislocation density, thin virtual substrate and planar surface. For the growth of tensile s-Si films, one of the methods is to grow the Si films upon the virtual substrates consisting of relaxed compositionally graded SiGe layers. It has been reported that the enhancement factor of electron mobility for the s-Si films grown by this method would increase with x_t, the Ge composition of the top SiGe layer of the virtual substrate, and would reach the saturation value of 1.8 when x_t was around 0.2.[4] However, for hole mobility, the saturation value of the enhancement factor was approximately 2.4 when x_t was 0.4.[5] That is to say, if the strain of the s-Si films increases up to 1.6%, the hole mobility of the s-Si films will be over two times higher than that of the unstrained Si films. Low dislocation density is another important requirement for the s-Si films, because dislocations will cause leakage current in MOS devices. Many methods have been developed to reduce the dislocation density,[6,7] but in most of them the thickness of the virtual substrates is frequently increased up to several microns, and the thick virtual substrate will lead to a self-heating effect in MOS devices and complicate their integration. Therefore, to develop the new methods for the growth of the s-Si films to meet all these requirements is important and necessary.

In this Letter, we report experimental results about the MBE growth of highly strained Si films with ultra-low dislocations density on patterned Si (001) substrate, while the thickness of the virtual substrate of the compositionally graded SiGe films is only 330 nm.

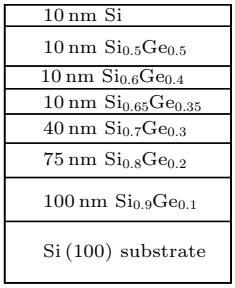

Fig. 1. Schematic illustration of the sample structure.

The p-type Si (001) wafers with a resistivity of 20 Ω-cm were used as the substrates of the samples. First a layer of Si_3N_4 of 450 nm thickness was deposited on the substrate by PECVD technology. Then square windows of $3 \times 3\,\mu m^2$ were opened by photolithography and dry etching process. On the same substrate an area of several tens of square millimetres was left unpatterned for the growth of the films for comparison. After the Si substrate was chemically cleaned by the Shiraki process,[8] the compositionally graded SiGe films and a layer of s-Si film were successively grown on the substrate at 550°C in a MBE system of Riber EVA-32. Figure 1 is a schematic illustration of the sample structure. In the sample, the total thickness of the compositionally graded SiGe layers, which were used as the virtual substrate, was 330 nm, and the thickness of the s-Si film grown over the virtual substrate was 10 nm. In order to observe the defects in the s-Si films, the samples were etched at 15°C by the modified Schimmel etching solution, which consisted of 55 vol% CrO_3(0.3 M) and 45 vol% HF (49%),[9] to reveal the etch pits of threading dislocations and the cross-hatched lines resulting from misfit dislocations. Their images were recorded by a scanning electron microscope (SEM) of Philips XL 30 FEG. The strain of the s-Si films and the SiGe virtual substrates was obtained from their Raman scatter-

*Supported by the Natural Science Foundation of China under Grant No 60376012, and the National Advanced Materials Committee of China under Grant No 2002AA313020.

**To whom correspondence should addressed. Email: xjzhang@fudan.ac.cn

© 2009 Chinese Physical Society and IOP Publishing Ltd

ing spectra, which was measured with a micro Raman spectrometer of LabRam HR800 with the excitation wavelength of 514.5 nm.

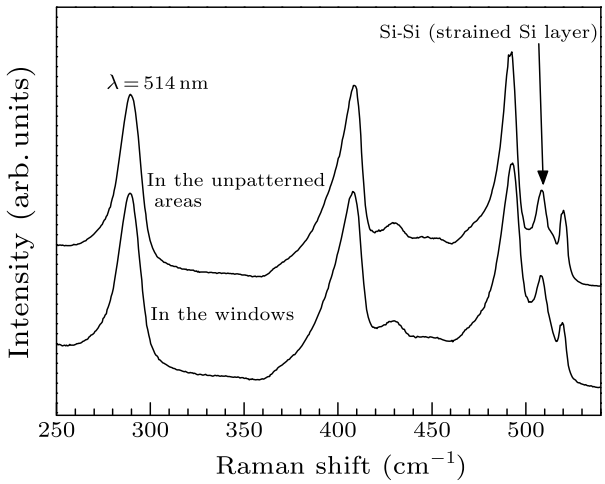

Fig. 2. Raman scattering spectra of the strained Si films and the SiGe virtual substrates in the unpatterned areas and in the windows of 3 × 3 micron.

Figure 2 shows the Raman spectra of the Si/SiGe/Si structures grown in the windows and in the unpatterned areas. The peaks near 490 cm^{-1}, 408 cm^{-1} and 289 cm^{-1} correspond to the phonon frequencies of the Si–Si, Si–Ge and Ge–Ge modes of the virtual substrates respectively. The contribution to the Raman spectrum of the virtual substrate can be regarded as only from the top layer of the $Si_{0.5}Ge_{0.5}$ film approximately, since the penetration depth into the $Si_{0.5}Ge_{0.5}$ film for the exciting laser beam of the Raman spectrometer[10] is much less than the thickness of the top $Si_{0.5}Ge_{0.5}$ layer.

The residual strain of a $Si_{1-x}Ge_x$ film can be obtained with any two of the following equations:[11]

$$\omega_{\mathrm{Si-Si}} = 520.5 - 62x - 815\varepsilon_{\mathrm{SiGe}}, \quad (1)$$

$$\omega_{\mathrm{Si-Ge}} = 400.5 + 14.2x - 575\varepsilon_{\mathrm{SiGe}}, \quad (2)$$

$$\omega_{\mathrm{Ge-Ge}} = 282.5 + 16x - 385\varepsilon_{\mathrm{SiGe}}, \quad (3)$$

where $\omega_{\mathrm{Si-Si}}$, $\omega_{\mathrm{Si-Ge}}$ and $\omega_{\mathrm{Ge-Ge}}$ are the phonon frequencies (in cm^{-1}) of the Si–Si, Si–Ge and Ge–Ge modes of the $Si_{1-x}Ge_x$ film respectively, x is the Ge composition of the $Si_{1-x}Ge_x$ film; 815, 575 and 385 are the strain-phonon coefficients (in cm^{-1}) of the corresponding modes, and $\varepsilon_{\mathrm{SiGe}}$ is the strain of the SiGe film.

The peaks at 520.5 cm^{-1} and near 509 cm^{-1} in Fig. 2 correspond to the phonon frequencies of the Si–Si mode of the Si substrate and the s-Si films. The strain of the s-Si film, $\varepsilon_{\mathrm{s-Si}}$, can be obtained from $\omega_{\mathrm{s-Si}}$ with the following equation,[12] which is deduced from Eq. (1) with $x = 0$:

$$\varepsilon_{\mathrm{s-Si}} = (520.5 - \omega_{\mathrm{s-Si}})/815. \quad (4)$$

Here $\varepsilon_{\mathrm{SiGe}}$ and $\varepsilon_{\mathrm{s-Si}}$ in the windows and in the unpatterned areas are listed in Table 1, which are obtained with Eqs. (1), (2) and (4), respectively. In order to describe the extent of the strain relaxation of the s-Si films and the top SiGe films of the virtual substrates, we define two strain relaxation factors for these two kinds of films. They are $R_{\mathrm{s-Si}} = 1 - \varepsilon_{\mathrm{s-Si}}/(0.042x_t + \varepsilon_{\mathrm{SiGe}})$ and $R_{\mathrm{SiGe}} = 1 + \varepsilon_{\mathrm{SiGe}}/0.042x_t$ respectively. Here x_t is the Ge composition of the top SiGe films of the virtual substrate. In Table 1, R_{SiGe} and $R_{\mathrm{s-Si}}$, are also listed.

In Table 1, R_{SiGe} in the windows and in the unpatterned areas reaches 92% and 89%, respectively. Based on this high strain relaxation of the $Si_{0.5}Ge_{0.5}$ films, the strain of the s-Si films in the corresponding regions reaches 1.5% and 1.4%, respectively.

Table 1. Strain and the strain relaxation factors of the s-Si films and the $Si_{0.5}Ge_{0.5}$ films of the virtual substrates grown in the windows and in the unpatterned areas. $R_{\mathrm{SiGe}} = 1 + \varepsilon_{\mathrm{SiGe}}/0.042x$ and $R_{\mathrm{s-Si}} = 1 - \varepsilon_{\mathrm{s-Si}}/(0.042x + \varepsilon_{\mathrm{SiGe}})$.

Growth region for films	$\varepsilon_{\mathrm{SiGe}}$ (%)	R_{SiGe} (%)	$\omega_{\mathrm{s-Si}}$ (cm^{-1})	$\varepsilon_{\mathrm{s-Si}}$(%)	$R_{\mathrm{s-Si}}$ (%)
In unpatterned areas	−0.22	89	508.9	1.4	25
In windows	−0.17	92	508.2	1.5	22

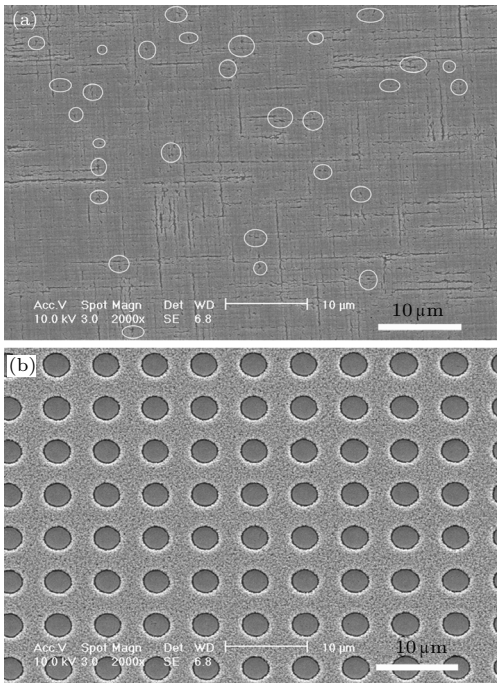

Fig. 3. SEM images of the strained Si films after etching (a) in the unpatterned area and (b) in the windows. The black dots in the white circles in (a) are the etch pits of the threading dislocations.

In addition, the dislocation density of the s-Si films in the two different regions is also studied. Figure 3 shows the SEM surface images of the samples etched by the modified Schimmel etching solution. In Fig. 3(a), on the surface of the s-Si films in the unpatterned areas, cross-hatched lines and the etch pits can be observed clearly. The cross-hatched lines result from the inhomogeneous strain fields associated with

the misfit dislocations, and the pits are due to the terminations of the threading dislocations in the strained films.[13] However, for the s-Si films in the windows, no such defects are found, as shown in Fig. 3(b). We checked several hundred windows and did not observe any etch pits of threading dislocations in the s-Si films, so we conclude that as a conservative estimate, the threading dislocation density of the s-Si films in the windows of $3 \times 3\,\mu m^2$ is lower than $1 \times 10^5\,cm^{-2}$.

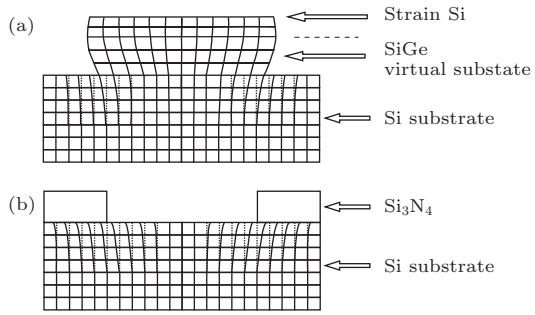

Fig. 4. Schematic illustration of the edge-induced strain relaxation in the structure of s-Si/compositionally graded SiGe layers/Si substrate within the window, (a) the strain relaxation in the s-Si film and the compositionally graded SiGe layers, (b) the effect of the edge-induced stress of the Si_3N_4 film on the surface atoms of the Si substrate.

In Table 1, it is noteworthy that the strain relaxation factors of the s-Si films grown in the windows and in the unpatterned areas reach 22% and 25% respectively. Although these two values are close to each other, the causes of inducing the strain relaxation in the two regions are quite different. For the s-Si films in the unpatterned areas, the strain relaxation is obviously induced by the dislocations. However, for the s-Si films in the windows, their dislocation density is very low, so the strain relaxation must be induced by processes other than dislocation formation. This is attributed to the edge effect of the epitaxial film in the window.[14,15] As illustrated in Fig. 4(a), the tensile strain of the s-Si film in the window is relaxed by the inward movement of the atoms in the marginal area of the s-Si film. In the movement these atoms also drag the underlying lattice planes of the virtual substrate along with them. For the heteroepitaxial films grown on the patterned virtual substrate, the edge-induced strain relaxation depends on the thickness of the virtual substrate and the effective rigidity ratio of the patterned substrate and the strained film.[16] The lower effective rigidity ratio and the thicker thickness of the virtual substrate will result in higher strain relaxation. In our experiments, the thickness of the patterned virtual substrates is thick enough to reduce the strain of the s-Si films significantly and the layered structure of the compositionally graded SiGe films of the virtual substrate makes the effective rigidity ratio lower. Thus the strain relaxation of the s-Si films in the windows becomes higher.

This edge-induced strain relaxation has significant influences on the strain and the dislocation density of the s-Si films in the windows. On the one hand, it is not expected for the s-Si films because it reduces their strain. However, similar edge-induced strain relaxation is also present in the SiGe films of the virtual substrates in the windows, which is induced due to the outward movement of the atoms in the marginal areas,[17] as shown in Fig. 4(a). This strain relaxation will be conducive to increase the strain of the s-Si films. In addition, the edge of the mask material of the window will induce another effect to relax the strain of the virtual substrates in the windows. Since the mask material of the sample is the Si_3N_4 film grown by PECVD technology, there should be intrinsic tensile stress in the film. According to Hu's model,[18] the edge-induced stress of the Si_3N_4 film will drag the surface atoms of the Si substrate near the edge of the window toward the Si_3N_4 itself, as shown in Fig. 4(b). Therefore the strain of the epitaxial SiGe film in the window will be further reduced. Due to these two edge effects, the strain relaxation of the virtual substrates in the windows will increase significantly and the strain of the s-Si films in the windows can remain at a high value. However, on the other hand, the edge-induced strain relaxation of the epitaxial film plays an important role in improving the crystallinity of the s-Si film in the window. Since this strain relaxation is of elastic relaxation, it may increase the critical thickness for the pseudomorphic growth of the s-Si films in the window and reduce the dislocation density of the s-Si film.[15,19] Additionally, the edge of the epitaxial film in the window will further reduce the dislocation density by blocking the dislocation propagation,[20] if dislocations occur. Under the influence of these edge effects, the dislocation density of the s-Si films in the windows is reduced greatly. In addition, it is important to indicate that the dislocation density of the top $Si_{0.5}Ge_{0.5}$ films of the virtual substrates in the windows was proved to be greatly reduced also due to the same reasons described above by our previous experimental results.[17] We believe that this ultra-low dislocation density of the virtual substrates is of key importance also in reducing the dislocation density of the s-Si films in the windows.

In summary, the s-Si films with the strain of 1.5% and the dislocation density less than $1.0 \times 10^5\,cm^{-2}$ have been successfully grown on the patterned Si (001) substrates. The thickness of the virtual substrates of the compositionally graded SiGe layers is only 330 nm. On the surface of the s-Si films no cross-hatched line resulting from misfit dislocations is visible. We attribute the results to the strain relaxation induced by the edge effects of the epitaxial films and the masks. The patterned virtual substrates with the layered structure make the strain relaxation of the s-Si films become higher. As the edge-induced strain relaxation is present both in the s-Si films and the virtual

substrates, the strain of the s-Si films in the windows may remain at a high value. Since this edge-induced strain relaxation is of elastic relaxation, it will increase the critical thickness for the pseudomorphic growth of the s-Si films on the SiGe virtual substrate and reduce their dislocation density. In addition, the blocking effect of the edge of the epitaxial film in the window on dislocation propagation makes a further reduction for the dislocation density of the s-Si films and the virtual substrates in the windows. This work provides a promising method for the growth of high quality strained films.

References

[1] Lee M L, Fitzgerald E A, Bulsara M T, Currie M T and Lochtefeld A 2005 *J. Appl. Phys.* **97** 011101

[2] Khare P, Schroder D and R. Sampson 2007 *Solid State Electron.* **51** 828

[3] Krishnamohan T, Krivokapic Z, Uchida K, Nishi Y and Saraswat K C 2006 *IEEE Electron. Dev.* **53** 990

[4] Takagi S I, Hoyt J L, Welser J J and Gibbons J F 1996 *J. Appl. Phys.* **80** 1567

[5] Oberhuber R, Zandler G and Vogl P 1998 *Phys. Rev.* B **58** 9941

[6] Fitzgerald E A, Xie Y H, Green M L, Brasen D, Kortan A R, Michel J, Mii Y J, and Weir B E 1991 *Appl. Phys. Lett.* **59** 811

[7] Capewell A D, Grasby T J, Whall T E and Parker E H C 2002 *Appl. Phys. Lett.* **81** 4775

[8] Ishizaka A and Shiraki Y 1986 *J. Electrochem. Soc.* **133** 666

[9] Werner J, Lyutovich K and Parry C P 2004 *Eur. Phys. J. Appl. Phys.* **27** 367

[10] Rath S, Hsieh M L, Etchegoin P and Stradling R A 2003 *Semicond. Sci. Technol.* **18** 566

[11] Tsang J C, Mooney P M, Dacol F and Chu J O 1994 *J. Appl. Phys.* **75** 8098

[12] Langdo T A, Currie M T, Lochtefeld A, Hammond R, Carlin J A, Erdtmann M, Braithwaite G, Yang V K, Vineis C J, Badawi H and Bulsara M T 2003 *Appl. Phys. Lett.* **82** 4256

[13] Hsu J W P, Fitzgerald E A, Xie Y H, Silverman P J and Cardillo M J 1992 *Appl. Phys. Lett.* **61** 1293

[14] Murakami E, Nishida A, Etoh H, Nakagawa K and Miyao M 1990 *Mater. Res. Soc. Symp. Proc.* **198** 57

[15] Xiong H, Zhang X-j, Jiang Z M, Hu J H, Shi B, Zhou X F, Jiang W R, Hu D Z and Fan Y L 2001 *J. Crys. Growth,* **233** 74

[16] Fischer A and Richter H 1994 *J. Appl. Phys.* **75** 657

[17] Lu X D, Zhang X-j, Yang H B, Fan Y L, Wang W N and Sun Y Q 2006 *Chin. Phys. Lett.* **23** 220

[18] Hu S M 1991 *J. Appl. Phys.* **70** R53

[19] Stoica T and Vescan L 1993 *J. Crys. Growth,* **131** 32

[20] Fitzgerald E A, Watson G P, Proano R E, Ast D G, Kirchner P D, Pettit G D and Woodall J M 1989 *J. Appl. Phys.* **65** 2220