Journal of the Korean Vacuum Science & Technology; JKVST
Vol.6, No.2, 2002, pp.84～87

Grazing Incidence X-ray Diffraction (GIXRD) Studies of the Structure of $Si_{1-x}Ge_x$/Si Surface Alloy

Y.Shi*, R.Zhao*, C.Z.Jiang*, X.J.Fan*

*Accelerator Lab, Department of Physics, Wuhan University, Wuhan 430072, China

(Received)

Abstract

The $Si_{1-x}Ge_x$/Si surface alloy (x = 0.3, 0.4 and 0.5), which are prepared by solid source MBE and have the SiGe epilayer thickness of 50Å, are annealed with different parameters. The surface structure analyses of the heterostructure samples are made on a triple-axis X-ray diffractometer in grazing incidence X-ray diffraction (GIXRD) geometry. It has been found that with different annealing time (1.5h, 18h, 64h) and annealing temperature (550℃, 750℃), the SiGe epilayer experienced different strain relaxation process, which was deduced from the GIXRD measurements of the in-plane (220) diffraction peak of Si(001) substrate and the relevant (220) surface diffraction of SiGe epilayer. The results show that the stress relieving and the lateral strain relaxation in the SiGe/Si heterostructure can be promoted by correct annealing, which is very helpful for the preparation of SiGe/Si strained superlattice with fine strain crystallization.

1. Introduction

Combining silicon-based technology with optoelectronics techniques, especially integrating novel optoelectronic devices onto Si chips, is one of the chief goals of microelectronics industry [1]. Because of the indirect band gap characteristics of silicon material, this goal is far from being achieved as yet. Porous Si, semiconducting silicides are once interesting candidates for Si-based light emitting devices [2], but Si-based heterostructure, especially SiGe/Si strained superlattice heterostructure, is now considered the most hopeful research direction.

Attribute to the band engineering, SiGe/Si heterostructure can improve greatly the optical and electrical properties of Si-based devices. But in practice the lattice mismatch between Si and Ge will bring about strain and distortion in the SiGe heteroepitaxy layer, lead to the mismatch dislocation in the interface. Thus the band structure and band compensation will be influenced and the properties of the Si-based optoelectronic devices will be destroyed. In fact, the major interest of this system is now more fundamental, lying in the understanding of the growth and relaxation mechanisms of SiGe strained epilayers. This is an important basis for the application of Si-based heterostructure devices. As X-ray diffraction (XRD) characterization techniques are well established for heteroepitaxial structures, we can get some valuable information from the grazing incidence XRD analyses of the surface structure of $Si_{1-x}Ge_x$/Si(001) heterostructure.

2. Experimental

SiGe is a kind of novel semiconducting alloy. SiGe alloy grown on the surface of Si wafer, i.e. $Si_{1-x}Ge_x$/Si surface alloy, were grown in a Riber 32P molecular beam epitaxy (MBE) system with a base pressure of 4 $\times 10^{-11}$ torr. Si and Ge were deposited from an electron beam evaporator and an effusion Knudsen cell, respectively. Si/Ge flux ratios were adjusted to obtain $Si_{1-x}Ge_x$ alloy with x = 0.3, 0.4 and 0.5, respectively. The deposition rate was maintained at approximately 0.3Å/s. The growth temperature was 550℃ and the thickness of the alloy layer was 50Å.

The prepared $Si_{1-x}Ge_x/Si(001)$ heterostructures were annealed with different parameters. The annealing temperature used was 550℃, 750℃ and the annealing time used was 1.5h, 18h, 64h, respectively. The analyses of $Si_{1-x}Ge_x/Si(001)$ surface structure were made on a triple-axis diffractometer using an X-ray rotating anode generator. Cu$K\alpha_1$ radiation was selected and the X-ray source was operated at a power of 50kV$\times$ 200mA. The SiGe/Si sample was mounted horizontally and the incidence angle of X-ray going down to the horizontal surface of the sample was typically 0.5°. Thus X-ray analysis beam caused diffraction only from the near surface of the sample. This is the so-called grazing incidence X-ray diffraction (GIXRD) technique. From the GIXRD measurements of the (220) surface diffraction of SiGe alloy layer and the (220) in-plane bulk diffraction peak of Si(001) substrate, the structure and strain elastic relaxation processes of the strained SiGe surface alloy can be studied.

3. Results and Discussion

The growth of SiGe heteroepitaxial layer on Si(001) substrate is in the so-called "Stranski-Krastanov" two-dimensional growth mode. Because of the 4.2% lattice mismatch between germanium and silicon, such growth results in a biaxial in-plane compression of the surface alloy. In the framework of elasticity theory, the entire lattice mismatch can be accommodated as a homogeneous strain in the alloy layers only. Some studies show that for a given Ge component x in the $Si_{1-x}Ge_x/Si(001)$ surface alloy, there is a critical thickness (Lc) of coherent epitaxy. If thinner than Lc, the deposited SiGe heteroepitaxial layer can achieve a uniform equilibrium lattice constant along the direction of interface by the mechanisms of lateral elastic deformation [3]. Thus the SiGe/Si superlattice with strained layer of low density of defects can be achieved.

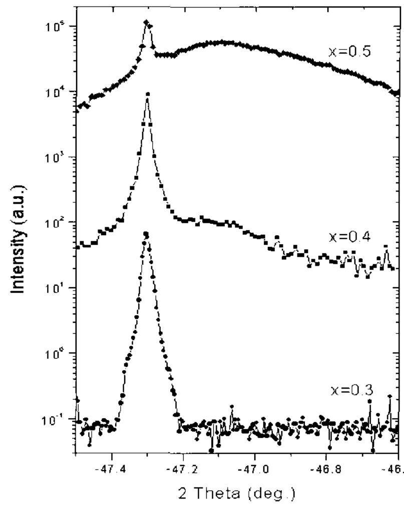

Fig. 1. The GIXRD patterns of unannealed $Si_{1-x}Ge_x/$ Si(001) surface alloy. x is 0.3, 0.4 and 0.5, respectively (bottom up).

The value of Lc is very small. For pure Ge layer (x = 1), Lc is only 15Å. Lc is 80 Å for x = 0.5 and 200 Å for x = 0.3. In this work the prepared SiGe/Si samples have all the same alloy thickness of 50 Å. Judge by the above Lc criterion, the desired SiGe/Si strained superlattice can be formed by elastic deformation after correct annealing.

To study the structure transition in the SiGe heteroepitaxy thin layer, GIXRD technique is employed, which allows precise structural characterization of thin films deposited onto a substrate surface. In the GIXRD geometry, both the signals originating from the top surface of the sample (surface diffraction) and bulk reflections located in the surface plane (in-plane diffraction) can be measured [4]. So the GIXRD analysis has been widely used to study the relaxation processes of the strain induced by the lattice misfit

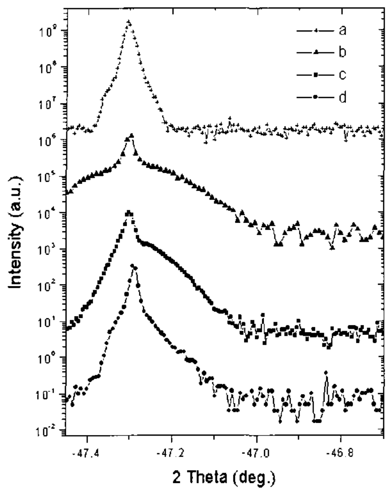

Fig. 2. The GIXRD patterns of $Si_{0.7}Ge_{0.3}$/Si(001) surface alloy annealed at 550°C. The annealing time is (a) 0, (b) 1.5h, (c) 18h and (d) 64h, respectively (top down).

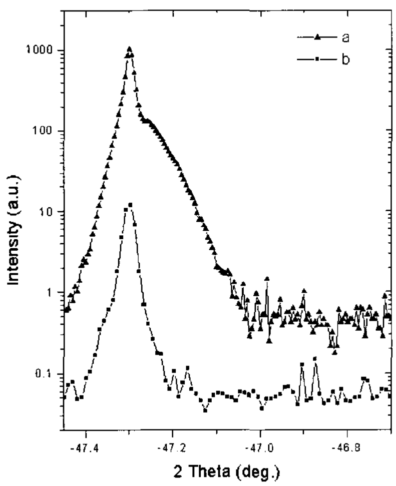

Fig. 3. The GIXRD patterns of $Si_{0.7}Ge_{0.3}$/Si(001) surface alloy after 18h's annealing. The annealing temperature is (a) 550°C and (b) 750°C, respectively.

between two crystals. For the GIXRD analyses of SiGe/Si(001) heterostructure, the strong in-plane (220) diffraction peak of Si(001) substrate can be used as a scaling reference point. By the precise measurement of the relevant (220) surface diffraction of the SiGe alloy layer, some valuable structure information of the SiGe/Si heterostructure can be got [5].

Figure 1 shows the GIXRD patterns of unannealed SiGe/Si heterostructure with different composition. The strong main peak at $2\theta = 47.302°$ (use $CuK\alpha_1$ radiation) originates from the (220) in-plane diffraction of Si(001) substrate. The shoulder at the lower anger, which reflects the relaxation expansion of lattice, comes from the SiGe strained layer. When the component of Ge is x = 0.3, there is almost no shoulder. Relaxation shoulder appears weakly when x = 0.4 and enhances obviously when x = 0.5. So for the unannealed SiGe surface alloy after heteroepitaxial growth, the lattice expansion and strain relaxation improve with the increase of Ge content.

For the SiGe surface alloy with the same Ge component, the strain relaxation is different when the samples undergo different annealing treatment. The relative GIXRD patterns are shown in Figs. 2 and 3. The $Si_{0.7}Ge_{0.3}$/Si surface alloys shown in Fig.2 are annealed with different time at 550℃. The 1.5 hours' annealing brings about a strong wide shoulder of the alloy layer. With the increase of annealing time to 18h and 64h, the width of the relaxation shoulder shrinks while the relative intensity keeps unchanged (b→c→d in Fig.2). The annealing temperature has similar effects. Keeping the annealing time unchanged (18h), the increase of annealing temperature from 550℃ to 750℃ makes the relaxation shoulder shrink obviously (shown in Fig.3).

The computer simulation of the GIXRD patterns can give us some quantitative results. The displacements between the SiGe layer's (220) shoulder peak and the reference Si(220) diffraction peak tell us the lattice mismatch between SiGe surface alloy layer and Si substrate. The crystallization quality of the SiGe alloy layer is estimated from the FWHM (full width at half-maximum) of the relaxation shoulder. Lattice coherent length (L) is calculated by the formula:

$\Delta Q = 4\pi\cos\theta \cdot \Delta\theta/\lambda = 2\pi/L$ (where Q is wave vector). As for the silicon (220) Bragg reflection using CuK(1 radiation, the formula is simplified as $L = 96.36/\Delta(2\theta)$(Å)

Table 1 The calculate results of unannealed $Si_{1-x}Ge_x/$ Si(001) surface alloy with different Ge content.

Component of Ge(x)	Coherent length in SiGe layer (A)	Lattice mismatch between SiGe and Si
0.3	8760	+0.094%
0.4	421	+0.376%
0.5	311	+0.575%

Table 2 The calculate results of $Si_{0.7}Ge_{0.3}/Si(001)$ surface alloy annealed at 550°C with different time.

Annealing time	Coherent length in SiGe layer (Å)	Lattice mismatch between SiGe and Si
1.5h	622	+0.028%
18h	1235	+0.244%
64h	1606	+0.206%

Some calculated results are shown in Tables 1 and 2, where the data indicate that the lattice cell of the SiGe alloy layer expands with the increase of Ge content, which brings about the increase of lattice mismatch between SiGe layer and Si substrate. But the strain relaxation in the unannealed SiGe alloy layer is far from complete. The stress freezing degrades the crystallization quality of the alloy layer, which is shown by the quick decrease of the lattice coherent length with the increase of Ge content. Correct annealing can promote the elastic strain relaxation in the SiGe alloy layer. Annealing at 550℃ for only 1.5h is not sufficient for the stress relieving in the SiGe alloy layer, as the lattice parameter (or d-spacing) is much less than that of equilibrium state. Increasing the annealing time to 18h brings about a 20% increase of the lattice parameter for the SiGe alloy layer. In the mean time the coherent length reaches twice as much, which indicates a great improvement of the crystallinity in the alloy epilayer. Annealing further longer to 64h has no obvious promotion of crystallinity. But annealing at higher temperature (750℃) will increase further the coherent length and bring uniformity and stabilization to the structure of the SiGe strained layer. So the lateral relaxation of lattice cell assumed by theoretical modelisations can be achieved and the SiGe/Si heterostructure with fine strain crystallization can be formed.

4. Conclusions

The $Si_{1-x}Ge_x/Si$ heterostructure samples (x = 0.3, 0.4 and 0.5), which are prepared by solid source MBE and have the heteroepitaxial thickness of 50Å, are annealed with different parameters. The surface structural analyses of the samples are made with GIXRD technique. It has been found that correct annealing (18 hours at 750℃) will promote the strain crystallization by stress relieving and lateral strain relaxation in the SiGe alloy epilayer. This is very helpful for the preparation of SiGe/Si heterostructure with fine crystallinity.

Acknowledgements

This work was founded by the National Natural Science Foundation of China (Project No. 50175082) and the Science and Technology Department of Hubei Province, China. Part of the experimental work was finished in the Centre de Recherche sur les Mecanismes de la Croissance Cristalline (CRMC2) of French Centre National de la Recherche Scientifique (CNRS).

References

[1] K. D. Hirschman, L. Tsybeskov, and S. P. Duttagupta, Nature **384**, 338 (1996).

[2] M. C. Bost and J. E. Mahan, J. Appl. Phys. **58**, 2696 (1985)

[3] I. Berbezier, B. Gallas, A. Ronda, and J. Derrien, Surf. Sci. **421-413**, 415 (1998).

[4] J. M. Gay, P. Stocker, and F. Rethore, J. Appl. Phys. **73**, 8169 (1993).

[5] F. Volpi, A. Portavoce, A. Ronda, Y. Shi, J. M. Gay, and I. Berbezier, Thin Solid Films **380**, 46 (2000).