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DCXRD Investigation of a Ge/Si(001) Island Multilayer Structure*

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Abstract: A Ge/Si(001) island multilayer structure is investigated by double crystal X-ray diffraction, transmission electron microscopy, and atomic force microscopy. We fit the satellite peaks in the rocking curve by two Lorentz lineshapes, which originate from the wetting layer region and the island region. Then from the ratio of the thicknesses of the Si and Ge (GeSi) layers as determined by TEM, the Ge compositions of the wetting layer and islands are estimated to be about 0.51 and 0.67, respectively, according to the positions of the fitted peaks. This proves to be a relatively simple way to investigate the Ge/Si (001) island multilayer structure.

Key words: Si; Ge; nano-dot; island; X-ray
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1 Introduction

Ge/Si islands formed on Si substrate by the Stranski-Krastanov growth mode have received much attention because of their novel electrical and optical properties, which are associated with quantum confinement[1]. These Ge islands on Si substrate are potential candidates for a new generation of Si-based electronic and optoelectronic devices. For many actual applications, it is important to acquire the Ge composition x of either the wetting layer or the islands. Etching technology has been used to determine the Ge composition[2,3], but the methods are very complicated. As a nondestructive examination method, double-crystal X-ray diffraction (DCXRD) has been widely used to determine the structure parameters of heterojunctions and superlattices[4,5]. However, it is difficult to use to investigate a Ge/Si island multilayer because of the special structure of this material.

In this work, we have investigated the Ge compositions of both a SiGe wetting layer and SiGe islands in a Ge/Si(001) island multilayer structure through DCXRD measurement combined with transmission electron microscopy (TEM) and atomic force microscopy (AFM) measurements. The method introduced here is much simpler than those reported previously[2,3].

2 Experiment

The investigated Ge/Si(001) island multilayer was grown by ultrahigh-vacuum chemical vapor deposition (UHV-CVD) on (001)-oriented Si substrate from pure Si_2H_6 and GeH_4[6]. The base pressure in the system was about 1×10^{-8} Pa, and that during growth was about 7×10^{-3} Pa. The substrate was cleaned in an ex-situ chemical etching process and then loaded into the growth chamber and heated up to 930℃ for 8min to deoxidize. After the growth of a 200nm Si buffer layer at 800℃, the temperature was decreased to 620℃. Then 11 layers of Ge islands separated by Si spacer layers were grown. The thicknesses of the Ge and Si spacer layers were 8 eq-ML(1eq-ML = 6.27 $\times10^{14}$ Ge atom/cm^2) and 50nm, respectively. The sample was terminated with a Ge layer.

The DCXRD rocking curve analysis was carried out on a standard double-crystal X-ray diffraction setup with the CuKα1 (λ = 0.154nm) line as the X-ray source. AFM images were recorded with a Digital Instruments Nanoscope Ⅲ AFM

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setup operating in contact mode. TEM measurement was performed on cross-sections.

3 Result and discussion

Figure 1 is a 2μm × 2μm AFM image of the morphology of the Ge islands on the top layer. Uniform dome-shaped Ge islands with an average width of 150nm and density of $2.5 \times 10^9 cm^{-2}$ have been formed clearly. The ratio between the area occupied by islands and the denuded area is about 42%.

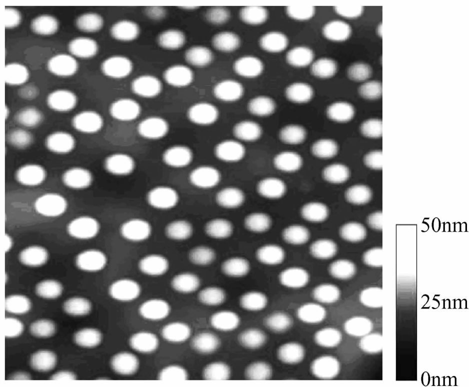

Fig.1 2μm×2μm AFM image of the top morphology of the investigated multilayer sample

In the cross-sectional TEM image of the multilayer sample shown in Fig. 2, the dark contrast regions correspond to the thin Ge wetting layers and Ge islands. There is generally a strain-driven Si-Ge inter-diffusion as growth. Therefore, the wetting layer and the island are actually composed of GeSi alloy[7]. According to the growth rate of nominally pure Ge and our reflection high energy electron diffraction (RHEED) observation during the growth, the thickness of the wetting layer with nominally pure Ge is 2.5~5.5 eq-ML. However, the thickness gotten by TEM is much larger than this value. This is because of the Si diffusion into the Ge layer driven by strain during the growth. We can get the thickness ratio of the Si spacer layer and the GeSi layer R from the TEM result. The values of R in the wetting layer region and island region are about 40 and 6, respectively.

To further investigate the average Ge composition of both the wetting layer and the islands from a macroscopic dimension viewpoint, we performed a conventional DCXRD measurement. Figure 3 shows the θ-2θ scan mode around the sub-

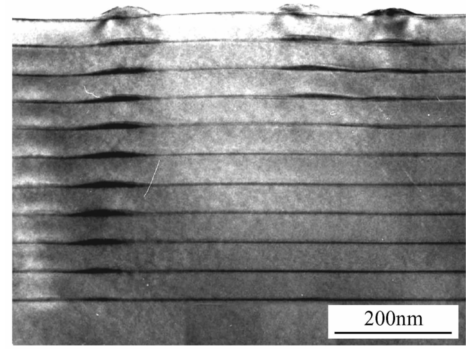

Fig.2 Cross-sectional TEM image of the investigated multilayer sample

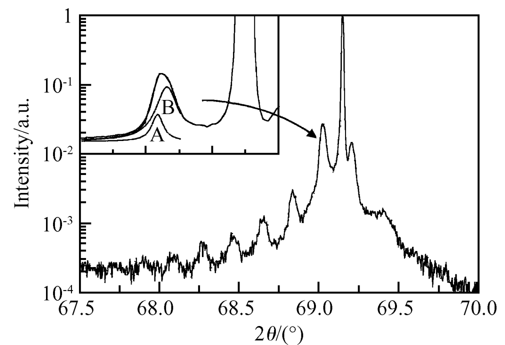

Fig.3 θ-2θ scan of the multilayer structure made around the substrate Si(004) Bragg peak with CuKα1 line as the incident light Shown in the inset is a close-up of the zero-order peak with the linear-scale ordinate, which is fitted well with two Lorentz line shapes, A and B.

strate Si(004) Bragg peak with the CuKα1 line as the incident light. The sharp peak at 69.148° is attributed to the diffraction from the (004) plane of the Si substrate and the Si buffer layer[5]. According to DCXRD kinematical theory, satellite peaks up to −6 orders originate from the Ge/Si island multilayer structure[8]. It exhibits a zero-order peak to the left of the substrate peak, which is attributed to lattice expansion introduced by Ge. The islanding and undulating in the interface of the multilayer broaden the rocking curve satellite peaks. According to Huang *et al.*'s previous investigation[9], every satellite peak can be fitted by two Lorentz lineshapes, which originate from the coupled islands column and the wetting layer, respectively. The fitting to the zero-order peak is shown in the inset of Fig.3. The positions of peak A (from islands) and peak B (from wetting layer) are 69.017° and 69.031°, respectively. The ratio of the integrated intensity of the two peaks (A/B) is

about 0.40, which is approximately equal to the ratio of 42% between the area covered by islands and the denuded area. This further supports our theory of the origin of peaks A and B[9].

Some crystal parameters in the wetting layer region and island region of the multilayer structure can be calculated from the positions of peaks A and B, respectively[8]. According to Bragg's law, the average lattice constant a_0^σ along [001] over the period is determined by the equation

$$2a_0^\sigma \sin\theta_0 = n\lambda \qquad (1)$$

where θ_0 is the position of the zero-order peak, λ is the X-ray wavelength, and n is an integer. The lattice mismatch between GeSi and Si substrate causes the GeSi epilayer to be strained, but the strain can be partly relaxed elastically or through dislocations[2]. For strained-layer superlattices with the relaxation σ and Ge content x, the lattice constants $a_{\mathrm{GeSi}}^{/\!/}$ and $a_{\mathrm{Si}}^{/\!/}$ in the growth plane (001) are expressed as $a_{\mathrm{GeSi}}^{/\!/} = a_{\mathrm{Si}}^{/\!/} = (1+\sigma)a_{\mathrm{Si}}$. The lattice constants $a_{\mathrm{GeSi}}^{\perp}$ and $a_{\mathrm{Si}}^{\perp}$ along the growth direction [001] can be determined by Vegard's law and isotropic elasticity theory:

$$a_{\mathrm{Ge}_x\mathrm{Si}_{1-x}}^{\perp} = \left(\frac{1+\nu}{1-\nu}\varepsilon x + 1 - \frac{2\nu}{1-\nu}\sigma\right)a_{\mathrm{Si}}$$

$$a_{\mathrm{Si}}^{\perp} = \left(1 - \frac{2\nu}{1-\nu}\sigma\right)a_{\mathrm{Si}}$$

Here ν is the Poisson ratio. Since the difference between ν_{Ge} and ν_{Si} is very small[10], setting $\nu=\nu_{\mathrm{Ge}}\overline{x}+\nu_{\mathrm{Si}}(1-\overline{x})$ will not cause an appreciable error. $\overline{x}$ is the average Ge content of the whole epilayer, which can be obtained from the average lattice constant a_0^σ. $\varepsilon=(a_{\mathrm{Ge}}-a_{\mathrm{Si}})/a_{\mathrm{Si}}$ is the natural lattice mismatch of bulk Ge and Si crystals, and a_{Ge} and a_{Si} are the lattice parameters of bulk Ge and Si, respectively.

Then the average lattice constant a_0^σ along [001] over the period can be written as

$$a_0^\sigma = \left(\frac{1+\nu}{1-\nu}\times\frac{1}{1+R}\varepsilon x + 1 - \frac{2\nu}{1-\nu}\sigma\right)a_{\mathrm{Si}} \qquad (2)$$

where R is the thickness ratio of the Si and the GeSi layer obtained from the TEM result.

For the wetting layer region, the strained layer is very thin and can be regarded as fully strained. From Eqs. (1) and (2), we get a Ge composition x of about 0.51, which is consistent with Alonso *et al*.'s wet chemical etching investigation value of 0.55[2]. The wetting layer of $\mathrm{Si}_{0.49}$-$\mathrm{Ge}_{0.51}$ with the ratio of $R=40$ implies that the nominally pure Ge thickness is 4.3 eq-ML, which is also in good agreement with our RHEED observation during the growth.

For the island region, the strain relaxation must be taken into account. The strain can be partly relaxed during the 3D growth both elastically and through dislocations. This reduces the vertical crystal parameter in the island region and causes the corresponding satellite peaks to shift to the substrate Si(004) peak. According to Alonso *et al*.'s wet chemical etching and Raman investigation[2], the strain of 0.8% is relaxed by elastic lattice distortion during the 3D growth. Using this value, the average Ge composition of 0.67 is obtained from Eqs. (1) and (2). Schmidt *et al*.[11] obtained the SiGe inter-diffusion in embedded Ge/Si island of about 40%～60% by PL spectra, which is consistent with our result.

Note that a small error in σ will affect the calculated result greatly, and thus it is very important to choose the value of σ carefully. However, we can still estimate the average Ge composition in island approximately from the DCXRD result. For the wetting layer region, the SiGe layer is fully strained in most cases, so the calculation of Ge composition in the wetting layer is relatively precise.

4 Conclusion

We have investigated the Ge/Si(001) island multilayer structure by DCXRD combined with cross-sectional TEM and AFM. By fitting the satellite peaks of the DCXRD curve with two Lorentz lineshapes, we got the positions of the diffraction peaks originating from the wetting layer region and island region, respectively. Then associating the thickness ratio of the Si and SiGe layers as determined by TEM, the average Ge composition of the wetting layer and islands were estimated to be 51% and 67%, respectively. This provides a relatively simple way to investigate the Ge/Si(001) island multilayer structure.

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DCXRD 分析 Ge/Si(001)多层纳米岛材料*

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摘要：结合透射电镜与原子力显微镜实验,用双晶 X 射线衍射方法分析了 Ge/Si 多层纳米岛材料.衍射的卫星峰可以被分解为两个洛仑兹峰,它们分别源于材料的浸润层区和纳米岛区.利用透射电镜得到 Si 和 SiGe 层的厚度比,估算出浸润层区与岛区的 Ge 组分分别为 0.51 和 0.67.这是一种简单估算 Ge/Si 多层纳米岛材料中 Ge 组分的方法.

关键词：Si；Ge；纳米岛；岛；X 射线

PACC：6110；7360F

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