Investigation of Strain and Thin Film Relaxation in $\text{Ge}_x\text{Si}_{1-x}/\text{Si}$ Strained-Layer Superlattice by Dark-Field Electron Holography

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Elastic strain plays an important role in modifying physical properties such as the mobility of charge carriers in semiconductors. Strain analyses reveal that the reduction of the total strain and the elastic strain could be as large as 30 and 65%, respectively in a very thin transmission electron microscopy (TEM) specimen. The strain and thin film relaxation in a cross-sectional transmission electron microscopy (XTEM) specimen of GeSi/Si strained-layer superlattice has been investigated by dark-field electron holography with a large holographic field of view (FOV) to 150 nm achieved by moving the specimen down below the front-focal plane of the objective lens in free lens control mode.

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1. Introduction

Strain engineering plays an important role in the semiconductor industry because of its enhancement in carrier mobility.¹⁾ There exist some widely available methods to investigate the strain, e.g., X-ray diffraction and Raman spectroscopy, which offer very good strain sensitivity but poor spatial resolution.^{2–4)} Transmission electron microscopy (TEM) is thus considered for measurement of strain with much higher resolution. Since the higher-order Laue zone (HOLZ) line is very sensitive to the local lattice parameters of the specimen, convergent-beam electron diffraction (CBED)⁵⁾ has been widely used to determine the local strain point-by-point with a spatial resolution of 5–10 nm.⁶⁾ Defocused CBED including large-angle convergent-beam electron diffraction (LACBED) with a shadow image of the specimen superimposed can provide more information on the local strain.^{7,8)} Various methods based on high-resolution electron microscopy (HREM) combined with image processing have been found to be highly efficient for strain mapping at the nanometer level. For instance, geometric phase analysis (GPA) is a powerful image processing technique developed by Hÿtch *et al.*⁹⁾

Recently dark-field electron holography (DFEH) has been used to map the strain distribution.¹⁰⁾ In order to obtain a wide field of view and a sufficient lateral resolution, DFEH was carried out in an expensive Cs-corrector TEM in a pseudo-Lorentz mode. In stead Sickmann *et al.*¹¹⁾ developed a new scheme of holographic set-up with a field view of 165–200 nm by decreasing the objective lens current in a conventional TEM. On this scheme, Cooper *et al.*¹²⁾ have investigated the strain distribution in InAs/InP quantum dots by DFEH.

Although TEM is powerful to characterize the strain with high spatial resolution, the elastic relaxation of strain in the very thin XTEM specimen could induce a big error in strain measurement. Gibson and Treacy¹³⁾ have shown that the relaxation is dependent on t/λ , where t is the specimen thickness, λ the superlattice wavelength on a Fourier series

model. In the case of $t/\lambda \gg 1$, the relaxation occurs only at the specimen surface, and the effect of the relaxation can be neglected apparently. In the other case of $t/\lambda \approx 0$, the specimen will be totally relaxed. Our previous studies show that the relaxation in cross-sectional specimens of $\text{Ge}_x\text{Si}_{1-x}/\text{Si}$ SLSs can be directly measured by the shift of the diffraction lines in LACBED in the SLSs from that in the Si substrate.⁸⁾ The theoretical treatments and the experiment measurements indicate that 30% of the total strain are reduced by the thin film relaxation at a misfitting interface. However, to our knowledge, the thin film elastic relaxation of strain has not been considered in the strain measurement by dark-field electron holography.

In this paper, we have used a simple sample of $\text{Ge}_{0.25}\text{Si}_{0.75}/\text{Si}$ strained-layer superlattice to demo the measurement of the strain and thin film relaxation in the XTEM specimen by LACBED and DFEH. Also we propose a new scheme of the holographic set-up modified following Sickmann's pioneering ideas in free lens control mode to obtain a wide field of view with a sufficient lateral resolution.

2. Experiments

To investigate the strain and thin film relaxation a nominal $\text{Ge}_{0.25}\text{Si}_{0.75}(5\text{ nm})/\text{Si}(21\text{ nm})$ SLS of 15 periods grown on a (001) Si wafer by molecular beam epitaxy (MBE) was used.¹⁴⁾ The XTEM specimen was prepared as follows. First, two pieces of samples were glued face to face on (001) plane, and mechanically grinded in [110] direction to a thickness of about 2 microns. Then the specimen was unglued into two pieces, and one of them was thinned to be a specimen of uniform thickness using a focused ion beam (FIB) machine for DFEH. The thickness of the specimen was about 110 nm, as measured by electron energy-loss spectroscopy (EELS) technique. LACBED and DFEH were carried out in a FEI Tecnai F20 TEM with a Gatan imaging filter system.

To assess the thin film relaxation in the XTEM specimen of $\text{Ge}_{0.25}\text{Si}_{0.75}(5\text{ nm})/\text{Si}(21\text{ nm})$ SLS, a LACBED pattern of the specimen near the [210] zone axis was recorded (Fig. 1). It is obvious that HOLZ lines become blurred as cross the interface region of about 50 nanometer wide. This implies

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Fig. 1 (a) Large angle convergent beam electron diffraction image at [210] zone axis. (b) A magnified image of area indicated by the dashed frame in (a).

that a large change of the local crystal parameters occurs in the interface region. This is caused by a combination of residual strains and elastic relaxation, and indicates that not only the superlattice but also the top of the substrate are stressed and subject to relaxation. So we can not take the interface region as a reference for DFEH and geometric phase analysis. In contrast the Kikuchi lines 480 crossing the interface perpendicularly are more curved but less blurred, which can be used to measure the thin film relaxation.

A large holographic field of view has been required not only in order to map the structure and strain of interest such as a strained-silicon channel metal–oxide–semiconductor field effect transistors, which usually exceeds more than 50 nm or even 100 nm, but also to keep the relaxed region from the reference region. Recently Sickmann *et al.*¹¹⁾ developed a new holographic set-up with fixed position of the object and the image plane of diffraction lens to meet the requirement. This set-up is based on a the double lens system consisting of objective lens defocused to form a virtual image in front of the object, and diffraction lens refocusing the image on its image plane. Here we propose an alternative holographic set-up for DFEH with objective lens current unchanged and moving the object down below the front-focal plane to form a virtual image. This set-up is also based on a double lens system consisting of diffraction lens objective and intermediate lens to transfer the virtual image to the projector lens system which is fixed.

Figure 2 shows the path of rays on the modified holographic set-up. The scheme of the ray path with no voltage applied on the biprism is shown in Fig. 2(a). Since objective lens excitation remains at eucentric current, it keeps objective lens in the best imaging condition, e.g., rotation center, stigmatism, etc., the back-focal plane at the plane of objective lens aperture exactly. Since the object falls below the front-focal plane of the objective lens, a virtual image forms above the object. In TECNAI F20 FEG TEM, the focal length of the objective lens is about 2 mm, and the magnification is about 40 times, so the distance between the

eucentric height and the front-focal plane is estimated to be 50 μm . In DFEH experiment, we moved down the specimen about 90 μm from the eucentric position, set the intermediate lens excitation to 30%, adjusted the diffraction lens current to focus the virtual image, then an interference hologram with a FOV of 150 nm and fringe spacing of 1.3 nm was acquired. Figure 2(b) shows the path of rays in DFEH on this set-up. The specimen is oriented to a two-beam centered dark-field imaging condition. The biprism was set about 50 nm away from the interface of the GeSi/Si SLS and the Si substrate to prevent reference area from relaxed area. A wide dark-field hologram can be obtained, even at small biprism voltages due to the comparably large distance between the virtual image plane of objective lens and biprism.

In this holographic set-up the FOV w_{hol} is deduced as (see Appendix)

$$w_{\text{hol}} = 2\gamma \frac{a\Delta x}{f_0} + \left(2\gamma f_0 - \frac{2rf_0}{a} \right) \quad (1)$$

where r is the radius of the biprism wire and γ the deflection angle at a specific filament voltage U_{bp} , Δx the specimen position bellow the front focal plane of the objective lens, and f_0 the focal length, a the distance between focal plane and biprism (For our F20 TEM, a equals 106 mm). Figure 3 shows the experimentally determined FOV w_{hol} and the correspondingly calculated values of the model system at the sampling limit. A good quantitative match is observed.

3. Result and Discussion

The Ge content in GeSi layers of the SLS has been derived to be about 0.24 using LACBED method described in detail in the reference.⁸⁾ It is well in agreement with the nominal value. Then the average Ge content in the SLS is 0.046, and the corresponding misfit f between SLS as an average alloy and the Si substrate is 0.19% giving by $f = (a_{\text{GeSi}} - a_{\text{Si}})/a_{\text{Si}} = \eta x$, where a_{GeSi} and a_{Si} are bulk lattice parameters of the alloy epilayer and the Si substrate respectively, x is the

Fig. 2 Path of rays. (a) Scheme of the imaging of an object by lowering the object distance. (b) Scheme of the holographic set-up in dark field.

Fig. 3 Results obtained for FOV with different specimen position Δx at three different biprism voltages U_{bp} . The hollow and solid symbols stand by theoretical results by eq. (1) and measurements on experiment, respectively.

Ge content of the $\text{Ge}_x\text{Si}_{1-x}$ epilayer, and $\eta = (a_{\text{Ge}} - a_{\text{Si}})/a_{\text{Si}} = 4.18\%$, is the natural misfit between pure Ge and Si. Then the corresponding elastic strain and total strain, under the biaxial stress, is about 3.6×10^{-3} and 3.6×10^{-3} , respectively, giving by $e_x = \frac{2\nu}{1-\nu}f$ and $\varepsilon_x = \frac{1+\nu}{1-\nu}f$, respectively, where we take Poisson's ratio $\nu = 0.3$.

Figure 1 shows that many diffraction lines are blurred depending on the L component of diffraction vector $g = \text{HKL}$ while 480 line with $L = 0$ is only shift when crossing interface. This implies that the effect of the thin film elastic relaxation mainly concentrates near the interface. Following our previous work, the relaxation $\sigma = 2.4 \times 10^{-3}$ is obtained. It is very close to $(1 + \nu)f$. It indicates that the GeSi/Si SLS

as an average alloy is totally relaxed along $[110]$, then the residual strain in $[001]$ will be $(1 + \nu)f$. The change of the strain along $[001]$ can be obtained by

$$\Delta = \frac{1 + \nu}{1 - \nu}f - (1 + \nu)f = \frac{\nu(1 + \nu)}{1 - \nu}f. \quad (2)$$

Therefore the reduction related to total strain and elastic strain is obtained as $\Delta/\frac{1+\nu}{1-\nu}f = \nu \approx 30\%$ and $\Delta/\frac{2\nu}{1-\nu}f = \frac{1+\nu}{2} = 65\%$, respectively. So the thin film relaxation could introduce a big error in the strain measurement. In contrast the diffraction lines in the superlattice are slightly zigzagged, since, for the superlattice, the ratio of thickness to modulation wavelength t/λ is about 4. Thus relaxation can be neglected.

Figure 4 shows the strain measurement obtained by DFEF at the (004) two-beam centered dark-field imaging condition. The thickness of GeSi and Si layers were 5 and 21 nm, respectively. The hologram has a FOV of 150 nm and a fringe spacing of 1.3 nm related to a resolution of 3.9 nm (Fig. 4(a)). A two-dimensional strain in the $[001]$ direction is mapped by the GPA method (Fig. 4(b)). In Fig. 4(c) the strain profile averaged across 6 nm is obtained from the region indicated by the dash line in the strain map. Since the Si substrate far from the interface is unstrained, the strain profile at the unstrained region is just on the x axis (dashed line). The average strain in the two periods at the bottom of the superlattice was measured to be about 0.26%, much smaller than that of 0.36% as expected for the specimen before thinned. The strain at the Si substrate immediately adjacent to the interface is about -0.1% , which is induced by the relaxation of SLS epilayer. The strain in the GeSi layers near the interface was measured to be $1.8 \pm 0.1\%$. In addition a small compressive strain in the Si layers was also found due to the thin film relaxation.

Fig. 4 Strain measurement by dark field holography, (a) a dark field hologram for the (004) diffracted beam, (b) a strain map for the (004) reflection, (c) profile extracted from the region indicated by the dash line and averaged across 5 nm.

4. Conclusion

We investigate the strain and thin film relaxation in the XTEM specimen of $\text{Ge}_{0.25}\text{Si}_{0.75}(5\text{ nm})/\text{Si}(21\text{ nm})$ SLS specimen by DFEH. The average strain in the two periods at the bottom of the superlattice was measured to be about 0.26%, much smaller than that of 0.36% as expected for the specimen before thinned. It is good in agreement with that by LACBED method. It indicates that the relaxation should be taken into account in the strain measurement by DFEH. The DFEH experiment was implemented by moving the specimen down below the front focal plane of the objective lens, obtaining enough FOV with the unchanged current center and astigmatism of object lens. Large holographic field of view up to 150 nm was achieved.

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Appendix

With the biprism inserted between the back-focal plane and image plane of an optical system, FOV of the hologram w_{hol} in the object plane is given by¹⁵⁾

$$w_{\text{hol}} = \frac{2\gamma b f_0}{a + b} - \frac{2r f_0}{a} \quad (\text{A1})$$

where a is the distance from back-focal plane to biprism plane, b the distance from biprism plane to image plane, λ the electron wave length, r the radius of biprism, and γ is the deflection angle, proportional to the voltage U_{bp} on biprism, given by $\gamma = \gamma_0 U_{\text{bp}}$ with $\gamma_0 = \frac{\pi}{2 \ln(r_{\text{bp}}/r)} \frac{1 + U_{\text{acc}}/511\text{ kV}}{U_{\text{acc}}(1 + U_{\text{acc}}/1022\text{ kV})}$ the r_{bp} means the distance from biprism to the ground electrode, and U_{acc} the accelerating voltage.

In our scheme, the specimen is moved down below the front focal plane of the objective lens by Δx . Then Gauss formula becomes

$$\frac{1}{f_0 - \Delta x} + \frac{1}{v} = \frac{1}{f_0}. \quad (\text{A2})$$

With the object distance $f_0 - \Delta x$, the image distance v and the focal length f_0 .

As indicates in Fig. 2 and by simple algebra we have

$$v = a + b = f_0 - \frac{f_0^2}{\Delta x}. \quad (\text{A3})$$

Since Δx is a much smaller than f_0 , we have $\frac{f_0^2}{|\Delta x|} \gg f_0$. Neglecting the term f_0 in eq. (A3) and combining eqs. (A1) and (A3), we have

$$w_{\text{hol}} = 2\gamma \frac{a \Delta x}{f_0} + \left(2\gamma f_0 - \frac{2r f_0}{a} \right). \quad (\text{A4})$$