Evaluation of threading dislocation density of strained Ge epitaxial layer by high resolution x-ray diffraction*

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(Received 24 April 2017; revised manuscript received 5 September 2017; published online 20 November 2017)

The analysis of threading dislocation density (TDD) in Ge-on-Si layer is critical for developing lasers, light emitting diodes (LEDs), photodetectors (PDs), modulators, waveguides, metal oxide semiconductor field effect transistors (MOSFETs) , and also the integration of Si-based monolithic photonics. The TDD of Ge epitaxial layer is analyzed by etching or transmission electron microscope (TEM). However, high-resolution x-ray diffraction (HR-XRD) rocking curve provides an optional method to analyze the TDD in Ge layer. The theory model of TDD measurement from rocking curves was first used in zinc-blende semiconductors. In this paper, this method is extended to the case of strained Ge-on-Si layers. The HR-XRD $2\theta/\omega$ scan is measured and Ge (004) single crystal rocking curve is utilized to calculate the TDD in strained Ge epitaxial layer. The rocking curve full width at half maximum (FWHM) broadening by incident beam divergence of the instrument, crystal size, and curvature of the crystal specimen is subtracted. The TDDs of samples A and B are calculated to be 1.41×10^8 cm^{-2} and 6.47×10^8 cm^{-2}, respectively. In addition, we believe the TDDs calculated by this method to be the averaged dislocation density in the Ge epitaxial layer.

Keywords: HR-XRD, RPCVD, threading dislocation density (TDD), etching pit density (EPD)

PACS: 73.61.At **DOI:** 10.1088/1674-1056/26/12/127309

1. Introduction

Si-based monolithic photonic integrated circuit (PIC) has become a vital research domain since it is low-cost and efficient for maintaining the performance roadmap known as Morre's Low.[1–4] However, Si material has reached its bottleneck due to the application of photodetectors or light emitting diodes (LEDs), and a material with strong absorption coefficient and large emission wavelength is desirable. High-quality thin Ge buffer layer with smooth surface, low defect density and on Si substrates has attracted more attention due to its compatibility with traditional Si complementary metal–oxide–semiconductor transistor (CMOS) technology and the application in photonic and electronic devices. The Ge films on Si substrate can be used for fabricating the high-mobility metal–oxide–semiconductor field-effect transistors (MOSFETs)[5,6] and also serve as a platform for the integration of optoelectronic devices on Si.[7,8]

However, there are some challenges of epitaxy Ge on Si for example, it is difficult to obtain the high TDD and rough surface due to the large lattice mismatch between Ge and Si (4.2%). The analysis of TDD in Ge epitaxial layer is critical for fabricating the devices. It is therefore necessary to develop a new method of determining the TDD in the strained Ge layer. Dislocation density measurements are used to identify the dislocation density of the Ge epitaxial layer by etching pit density (EPD) and transmission electron microscope (TEM). However, it is difficult to determine the dislocation density of the material by using EPD when the dislocation density is more than 10^6 cm^{-2} and TEM also has its regional limitation. In this paper, we propose an optional method to determine the TDD in strained Ge epitaxial layer via its rocking curve full width at half maximum (FWHM).

2. Theoretical model

The FWHM of XRD rocking curve measured from Ge-on-Si layer by using a double-crystal diffractometer depends on the intrinsic rocking curve FWHM of Ge single-crystalline, incident beam divergence of the instrument, angular rotation at dislocations, strain surrounding dislocations, crystal size and curvature of the crystal specimen. Therefore, the measured FWHM of strained Ge epitaxial layer is always larger than intrinsic FWHM.

The measured FWHMcan be expressed as[9]

$$\beta_m^2 = \beta_0^2 + \beta_d^2 + \beta_\alpha^2 + \beta_\varepsilon^2 + \beta_L^2 + \beta_r^2, \tag{1}$$

where $\beta_0(hkl)$ is the intrinsic FWHM of strained Ge single crystal, $\beta_d(hkl)$ is the FWHM broadening by incident beam

*Project supported by the Research Plan in Shaanxi Province, China (Grant No. 2016GY-085), the Opening Project of Key Laboratory of Microelectronic Devices & Integrated Technology, Institute of Microelectronics, Chinese Academy of Sciences (Grant No. 90109162905), the Fundamental Research Funds for the Central Universities (Grant No. 17-H863-04-ZT-001-019-01), and the National Natural Science Foundation of China (Grant Nos. 61704130 and 61474085).

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divergence of the instrument, $\beta_\alpha(hkl)$ is the FWHM broadening by angular rotation at dislocations, $\beta_\varepsilon(hkl)$ is the FWHM broadening by the strain surrounding dislocations, $\beta_L(hkl)$ is the FWHM broadening due to crystal size, and $\beta_r(hkl)$ is the FWHM broadening due to the curvature of the Ge crystal specimen.

2.1. Intrinsic FWHM of strained Ge

The intrinsic FWHM of strained Ge (004) can be described as[10]

$$\beta_0(hkl) = [r_c\lambda^2(1+|\cos 2\theta|)|F_{hkl}|] \times [\sin(\theta-\phi)/\sin(\theta+\phi)]^{1/2}/[\pi a_0^3 \sin(2\theta)], \quad (2)$$

where r_c is the radius of electron, is the wavelength of x-ray, θ is the Bragg angle, $|F_{hkl}|$ is the reflection structure factor for the (hkl), a_0 is the lattice constant of the strained Ge, and ϕ is the angle between the crystal surface and the diffracting plane.

The reflection structure factor for the Ge (hkl) can be expressed as

$$F = f\left[1+\exp\left[2\pi \mathrm{i}\left(\frac{h}{2}+\frac{k}{2}\right)\right]+\exp\left[2\pi \mathrm{i}\left(\frac{h}{2}+\frac{l}{2}\right)\right] + \exp\left[2\pi \mathrm{i}\left(\frac{k}{2}+\frac{l}{2}\right)\right]\right] \times \left[1+\exp\left[2\pi \mathrm{i}\left(\frac{h}{4}+\frac{k}{4}+\frac{l}{4}\right)\right]\right]. \quad (3)$$

(i) When h, k, and l are all odd numbers,

$$F^2 = 32f^2; \quad (4)$$

(ii) when h, k, and l are all even numbers,

$$F^2 = 64f^2. \quad (5)$$

Hence, the reflection structure factor for the Ge (004) is $64f^2$, where f is the dispersion factor of an atom.

The lattice constant of the strained Ge can be calculated using Bragg's law to extract the out-of-plane lattice parameter $(a^\perp)$ of the Ge layer from Ge (004) diffraction peak position. It is given by

$$a^\perp = \frac{2\lambda}{\sin(\theta_{\mathrm{Ge}})}, \quad (6)$$

where λ is the wavelength of the Cu Kα1 wavelength (λ = 0.15406 nm) and θ is the Bragg angle.

The in-plane lattice constant $(a^\parallel)$ of the Ge epitaxial layer can be calculated from

$$a^\parallel = \frac{1+v}{2v}\left[a_{\mathrm{Ge}} - a^\perp\left(\frac{1-v}{1+v}\right)\right], \quad (7)$$

where v is the Possion's ratio of Ge, $v = 0.271$, and a_{Ge} is the unstrained Ge lattice constant, $a_{\mathrm{Ge}} = 0.56578$ nm.

2.2. Incident beam divergence of instrument

The FWHM broadening by incident beam divergence of instrument can be expressed as[11]

$$\beta_d = \sqrt{(\mathrm{FWHM_{Si}})^2 - 2.384^2}, \quad (8)$$

where 2.384 arcsec is the theoretical intrinsic FWHM of Si single crystal, $\mathrm{FWHM_{Si}}$ is the measured FWHM of the bulk Si and the $\mathrm{FWHM_{Si}}$ is 20.6095 arcsec. In addition, Si (004) rocking curve is shown in Fig. 1.

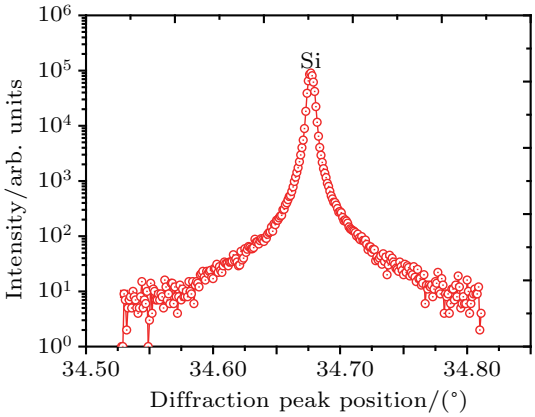


Fig. 1. (color online) (004) rocking curve scan of Si substrate obtained by HR-XRD.

2.3. Angular rotation at dislocations

The rocking curve FWHM of Ge broadening by angular rotation at dislocations can be expressed as

$$\beta_\alpha^2 = 2\pi \ln 2 b^2 D. \quad (9)$$

If the dislocations are arranged in random network, where D is the dislocation density induced by angular rotation and b is the length of Burgers vector.

2.4. Strain surrounding dislocations

The rocking curve of Ge epitaxial layer broadening by strain surrounding dislocations is expressed as[12]

$$\beta_\varepsilon^2 = \left[8\ln 2\left(\overline{\varepsilon_N^2}\right)\right]\tan^2\theta, \quad (10)$$

where $\overline{\varepsilon_N^2}$ is the mean square strain in the direction of the normal $\bar{N}$ to the diffracting planes.

Combine Eq. (9) with Eq. (10), the rocking curve of epitaxial broadening by crystal size can be expressed as[13]

$$\beta_{\mathrm{TDD}}^2 = 2\pi \ln 2 b^2 D + \left[8\ln 2\left(\overline{\varepsilon_N^2}\right)\right]\tan^2\theta. \quad (11)$$

2.5. Crystal size

The rocking curve FWHM of epitaxial layer broadening by crystal size can be expressed as[13]

$$\beta_L^2 = \left[4\ln 2/\left(\pi h^2\right)\right]\left(\lambda^2/\cos^2\theta\right), \quad (12)$$

where h is the thickness of the Ge epitaxial layer.

2.6. Curvature of crystal specimen

The rocking curve FWHM of epitaxial layer broadening by the curvature of the crystal specimen is given by[14]

$$\beta_r^2 = w^2/\left(r^2 \sin^2\theta\right), \tag{13}$$

where w is the width of the x-ray beam in the diffraction plane and r is the radius of curvature for the sample. In this sample, the thickness of Si substrate (480 μm) is larger than the Ge epitaxial layer (1 μm) and the rocking curve FWHM of Ge broadening by the curvature of the crystal specimen can be neglected.

3. Experimental details

The Ge films were deposited on a (100)-oriented Si wafer. The Si wafer was cleaned by standard RCA, and the cleaned wafer was loaded into RPCVD reactor and baked in hydrogen (H_2) at 1050 °C for 2 min to remove the thin surface oxide that is detrimental to the epitaxy process. Then, the Ge layer was grown, and the precursor for Ge is 2% germane (GeH_4) diluted in H_2 balance. A three-step Ge deposition was adopted and the thickness of the epitaxial layer was 1 μm. The three steps in the growth sequence were as follows. (I) The low temperature (LT)-Ge was grown at 350 °C to obtain a Ge seed layer and maintain a smooth surface layer; (II) temperature increase gradually from 350 °C to 650 °C at a rate of 10 °C/min; and (III) high temperature (HT) growth took place at 650 °C to achieve the required thicknessat a reasonable growth rate. The growth rates in three layers were different. High resolution x-ray diffraction (HR-XRD) patterns were recorded on a Phillips X'Pert diffractometer operating at 40 mA and 40 kV with using Cu Kα1 radiation. The 2θ–ω scan was utilized to determine the crystallinities of the Ge epilayers, and single-crystalline rocking curves are also measured.

The HR-XRD 2θ–ω scans of 1-μm thick Ge layers are shown in Figs. 2(a) and 2(b), and the figure shows a symmetric Ge (004) peak indicating good crystal quality. The FWHMs of the sample A and sample B were 208.9243 arcsec and 375.3556 arcsec, which are shown in Figs. 3(a) and 3(b). Comparing sample B with sample A, no LT-HT ramp layer was introduced in sample B. In addition, we adjusted the diffraction angle of Ge by modifying the standard diffraction angle of Si (69.132°).

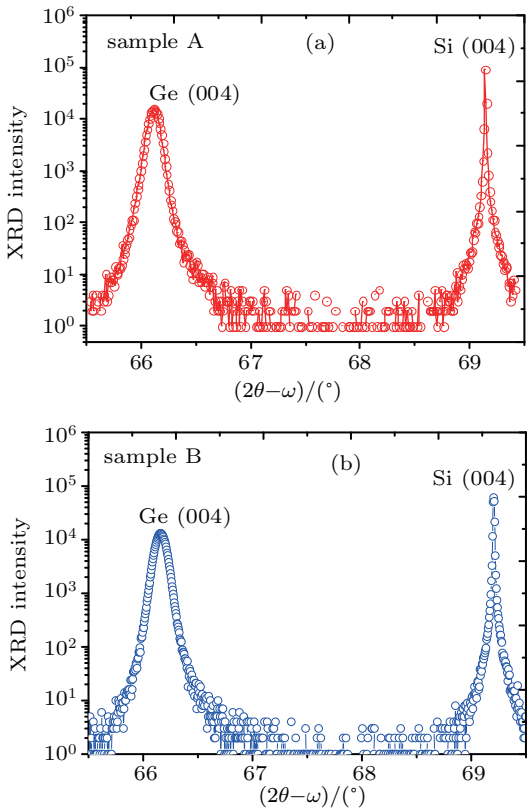


Fig. 2. (color online) 2θ–ω scans of samples A (a) A and B (b) measured by HR-XRD.

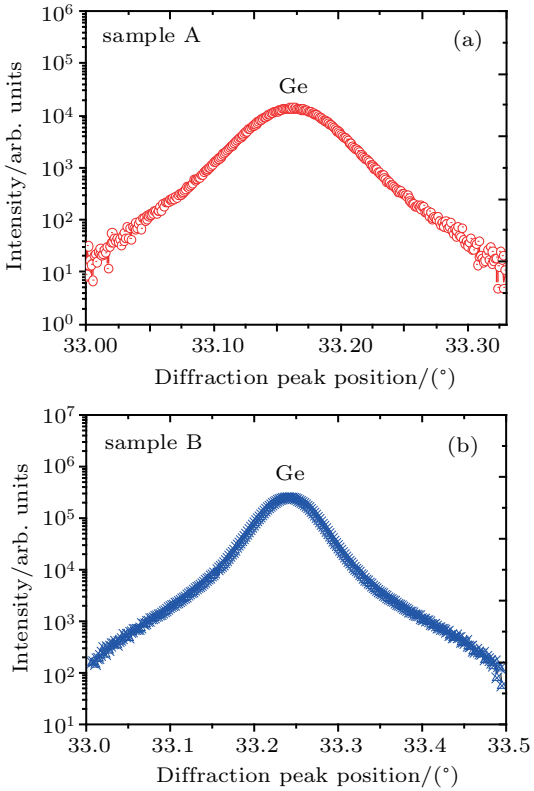


Fig. 3. (color online) (004) Rocking curve of samples A (a) and B (b) measured by HR-XRD.

4. Results and discussion

Combining Eqs. (2), (8), (11), (12), and (13) yields

$$\beta_{\mathrm{TDD}}^2 \approx \beta_m^2(hkl) - \beta_0^2(hkl) - \beta_d^2(hkl), \tag{14}$$

which is the TDD broadening in Ge epitaxial layer.

According to the Gay *et al.*'s[15] analysis, the rocking curve broadening by TDD can be written as an empirical formula:

$$D = \beta_{\mathrm{TDD}}^2/2\pi b^2 \ln 2, \tag{15}$$

where b is 0.4 nm, which is cited from Ref. [16].

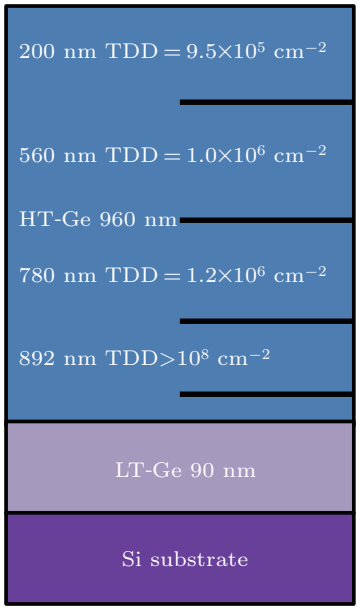


Fig. 4. (color online) Ge-on-Si depth profile of TDD in Ref. [17].

Substituting Eq. (14) into Eq. (15), TDD in Ge epitaxial layers are calculated to be 1.41×10^8 cm^{-2} for sample A and 6.47×10^8 cm^{-2} for sample B, respectively. The values of β_m^2, β_0^2, β_d^2, and β_{TDD}^2 are listed in Table 1. We find that the TDD value of sample A is smaller than that of sample B, which means that the introducing of LT-HT ramp layer can also degrade the TDD in the Ge epitaxial layer. This is due to the fact that low temperature growth rate can provide sufficient time for Ge layer to form more loops or networks. The role of LT-HT Ge layer is to allocate sufficient time for thermal exposure. If LT-HT Ge layer is replaced by a thicker LT-Ge layer, less tensile strain in the film is induced and the TDDs in LT-layer will become larger. The networks or loops can greatly reduce the dislocations from threading into the HT-Ge layer, and the averaged dislocation density in the Ge epitaxial layer also decreases.

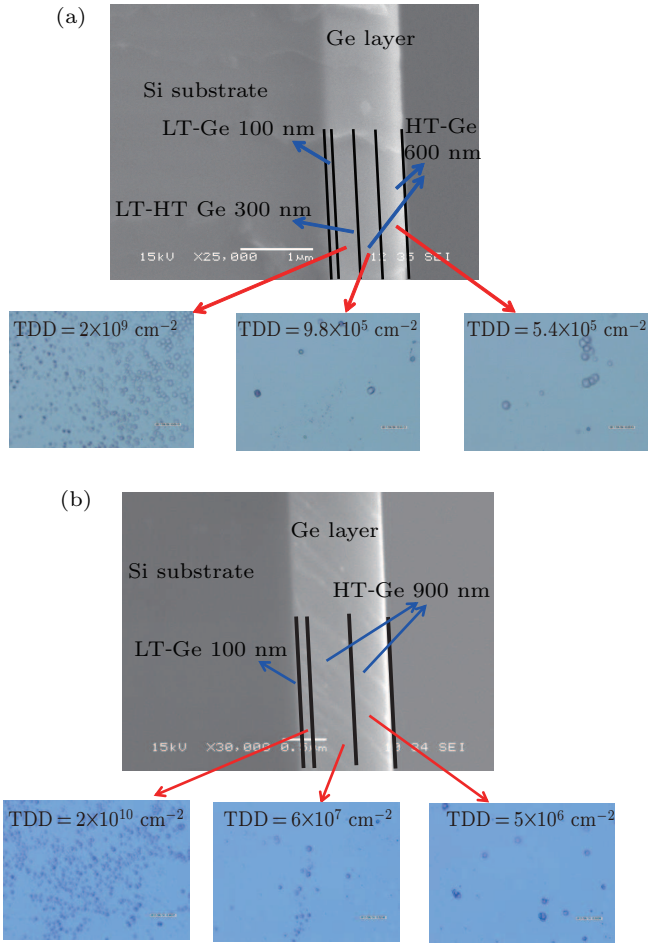


Fig. 5. (color online) Depth dependence of the TDD information of samples A (a) and B (b).

Table 1. Calculated values of β_m^2, β_0^2 , β_d^2, and β_{TDD}^2 for samples A and B.

Sample		β_m^2	β_0^2	β_d^2	β_{TDD}^2	TDD
A	66.102°	43649.363	1307.330	419.068	41922.965	1.41×10^8 cm^{-2}
B	66.106°	140891.376	1307.330	419.068	139164.978	6.47×10^8 cm^{-2}

It should be pointed out that the dislocation density in the Ge epitaxial layer varies with depth,[17] and the depth profile of TDD is shown in Fig. 4. It is demonstrated that TDD decreases with distance from Si/Ge interface and the typical TDD is about 10^6 cm^{-2} in 1-μm-thick Ge epitaxial layer grown by RPCVD. Therefore, we believe that TDD calculated by this method is the averaged dislocation density in the Ge epitaxial layer. We also use the Secco defect etching to determine the TDD in the as-grown Ge epitaxial layers, and I_2 solution ($HF:HNO_3:CH_3COOH:I_2 = 5$ ml:10 ml:11 ml:30 mg) is selected for etching counting. Firstly, we determine the etching rate of I_2 solution ($HF:HNO_3:CH_3COOH:I_2 =$ 5 ml:10 ml:11 ml:30 mg) by etching different times. In this way, the depth profile (Fig. 5) of TDD is obtained by controlling the etching time, from which we can find that the TDD in LT-Ge layer is 2×10^{10} cm^{-2}. The TDD value of LT-Ge layer in Ref. [18] is very close to 10^{11} cm^{-2}. In addition, the growth conditions of LT-Ge layer for samples A and B are the same and we believe that the TDD in LT-Ge layer keeps almost the same. At the beginning of the deposition of Ge films, a lot of dislocations exist in the LT-Ge layer and TDD in this layer is very likely to be 4 or 5 orders of magnitude larger than the TDD in HT-Ge. Since the lattice parameters of the Ge layer and Si substrate are different, LT-Ge layer is subjected to significant stress, causing stress-induced dislocation to move and form loops or networks. The networks greatly reduce the dis-

locations from threading into the HT-Ge layer. When HT-Ge grows much thicker, more and more dislocations can diminish.

In order to compare high-resolution XRD results with those obtained with EPD and TEM, we listout the recent TDD values from selected groups in Table 2. from which, we can find that the TDD values obtained by theoretical calculation are one or two orders of magnitude larger than those obtained with EPD or TEM. In addition, all the TDD values in Table 2 are obtained by TEM or EPD. What is more, it is difficult to determine the dislocation density of the material by using EPD when the dislocation density is more than 10^6 cm^{-2} and TEM also has its regional limitation. Hence, we consider the TDDs calculated by this method to be the averaged dislocation density in the Ge epitaxial layer.

Table 2. Summary of recent TDD values from selected groups.

Year	Institution	Ref.	TDD (TEM/EPD)/cm^{-2}
2008	Stanford University,	[19]	$0.8–0.1\times10^7$
2011	The University of Warwick	[20]	1×10^7
2008	CEA-LETI, Minatec	[21]	10^7
2009	CEA-LETI, Minatec	[22]	$5\times10^7–7\times10^6$
2012	Nanyang Nanofabrication Centre	[23]	10^7
2014	Lanzhou University	[24]	3×10^6
2013	SMART	[25]	10^7
2013	University of Warwick	[26]	2×10^8
2012	Nanyang Technological University	[27]	–
2012	Shanghai Institute of Microsystem and Information Technology	[28]	1×10^7
2014	University of Warwick	[29]	1×10^7
2016	Univ. Grenoble Alpes	[30]	3.8×10^7

5. Conclusions

In this study, the theory model for the calculation of TDD from rocking curve FWHM is extended to the strained Ge-on-Si layer. The Ge single crystal epitaxial layer is grown on Si (100) substrate in RPCVD system and single crystal rocking curve FWHM is measured to evaluate the TDD in Ge epitaxial layer. By using this method, the TDD of tensile strained Ge epitaxial layers are calculated to be 1.41×10^8 cm^{-2} for sample A and 6.47×10^8 cm^{-2} for sample B, respectively, and the TDD calculated by this method is the averaged dislocation density in the Ge epitaxial layer, including, LT-Ge layer, LT-HT Ge layer and HT Ge layer. We can find that the TDD values obtained by theoretical calculation are one or two orders of magnitude larger than those obtained with EPD or TEM. In addition, the TDD value of sample A is smaller than that of sample B, which means that the introducing of LT-HT ramp layer can also degrade the TDD in the Ge epitaxial layers.

Acknowledgment

The authors would like to express their gratitude to Li Xin for his help with the measurement of the HRXRD rocking curves.

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