Analysis of Strain in Semiconductor Nanoscale Thin Films

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Abstract

A thin silicon nano-overlayer (SNOL) fabricated by oxidation and etch-back on a SIMOX (separation by implantation of oxygen) wafer was investigated by grazing incident X-ray diffraction at incident angles between 0.01 and 0.1° below the critical angle of total reflection (0.18°). We measured {220} reflections by probing the sample with respect to the surface normal and found that the SNOL had finite domains under strain close to the surface. We also found that annealing the sample at 1000°C significantly reduced inhomogeneous strain and increased the size of the domains in the surface region of the SNOL.

1. Introduction

High-quality thin silicon overlayers (SOLs) on silicon-on-insulator (SOI) wafers have attracted much interest in recent years. Nanometer-thick ones, called silicon nano-overlayers (SNOLs) are expected to exhibit a clear quantum confinement effect at low temperatures [1], [2] and they have good potential as advanced Si substrates not only for high-quality epitaxial SiGe films [3], but also for nanostructure self-assembly [4]. An SNOL less than 50 nm thick is usually obtained by thermal oxidation of an SOL several hundred nanometers thick on an SOI wafer. During the thermal oxidation, the thickness of the SOL decreases as the oxide thickness increases, resulting in a kind of SiO_2/Si(<50 nm)/SiO_2 sandwich structure on the Si wafer. In this way, using cyclical thinning by thermal oxidation of a thick (>100 nm) SOL and HF etching (i.e., sacrificial oxidation) of the top silicon oxide layers (TOX), one can produce thin (<50 nm) SNOLs on top of a buried silicon oxide layer (BOX).

It is well known, however, that SOI wafers contain strain, which originates from the difference in specific volume and thermal expansion coefficients between the Si and SiO_2 at the SiO_2/Si interface. The strain usually induces defects (such as lattice distortion, finite domain size, and dislocations) at the interface and inside the SNOL. This degrades the electronic and optical properties of the final structures. Recently, Camassel et al. stated that the SOI is not a perfectly strain-relaxed system but behaves more like a balanced-strained structure [5]. Tiberj et al. also showed that tensile strain in the SNOL increases and compressive strain in the BOX decreases as the SNOL thickness decreases [6], [7]. They also showed that an SNOL with TOX layers has more strain than one without a TOX layer [6]. Accordingly, it is important to evaluate the strain and related lattice distortion in an SNOL as the SNOL thickness deceases below 50 nm in the oxidation and etch-back processes [5], [8]. In other words, to obtain a strain-controlled high-quality SNOL with a thickness of less than 50 nm, it is necessary to characterize the crystalline quality (including the strain and strain distribution) in the SNOL [9]-[11].

In this work, we characterized the lattice strain and strain distribution in a 47-nm-thick SNOL on a SIMOX wafer using grazing incidence X-ray diffraction near the critical angle of total reflection using synchrotron radiation.

2. Experiments

We used <001>-oriented SIMOX wafers with a diameter of 15 mm [12]. The oxygen dose was 4 × 10^{17} cm^{-2} and the implantation energy 180 keV. The

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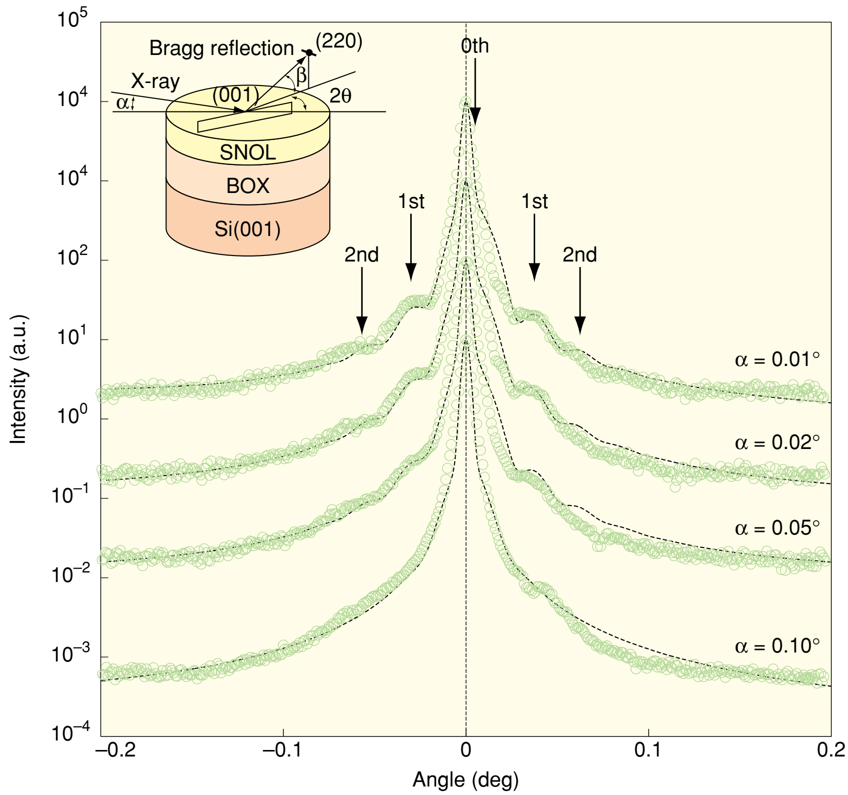

Fig. 1. Grazing incident X-ray diffraction curves of the SIMOX wafer at different grazing angles. The broken lines are the kinematical calculations based on the structural model mentioned in the text. The inset is a schematic diagram of grazing incident X-ray diffraction from the SIMOX wafer, where α, β, and 2θ represent the X-ray incident angle to the sample surface, the angle between Bragg reflection and the sample surface, and the angle between the incident X-ray and (220) plane.

wafers were annealed at 1350°C to yield a 120-nm BOX under a 62-nm-thick SOL. The density of threading dislocations was below 300 cm^{-2}. After being etched in HF dilute solution, the wafer was oxidized at 1200°C in a 0.2% O_2/Ar mixture to form a silicon oxide layer on top of the SOL and promote an internal thermal oxide layer on the bottom of the SNOL, improving the quality of the resulting interfaces [13]. Then, the thermal oxide on the SNOL was removed in an HF bath and X-ray diffraction measurements were performed from the 47-nm thick SNOL on top of the SIMOX wafer. The sample was annealed first in purified Ar gas and then in a high vacuum at 1000°C for 2 h.

Grazing incidence X-ray diffraction (GIXD) experiments were performed using the z-axis goniometer at beamline BL24XU of SPring-8 [14]. The 0.124-nm X-ray wavelength was used at incident angle α varying from 0.01 to 0.4°. This is schematically shown in the inset of **Fig. 1**. Before entering the goniometer, the X-ray beam was monochromatized and collimated to a size of 0.1 mm (V) and 1.0 mm (H) using x-y slits. An NaI detector and a solar slit with divergence of 0.2° were used to detect in-plane diffraction from the sample. A θ–2θ scan of them around the Si(220) Bragg peak was carried out to obtain a diffraction curve for different grazing incident angles.

3. Results and discussion

3.1 Strain analysis of a thin SOI wafer

The (220) Bragg diffraction curves collected from the 47-nm-thick SNOL at different grazing angles from 0.01 to 0.2° are shown in Fig. 1. The strong peak at the center (θ = 0) comes from bulk Si(220). Additional oscillating subpeaks (denoted as 0th, 1st, 2nd in Fig. 1) appear at the lower and higher sides of the Si(220) reflection. They are clearly observed for grazing angles between 0.01 and 0.1°. However, as the grazing angle increased, the peaks merged and became unclear at the shoulder of the main peak. This suggests that they originated from the surface region of the sample. In addition, it can be seen that the oscillating peaks were asymmetric with respect to the center of the Si(220) peak. The asymmetry also became clear when the grazing angle was decreased from 0.1 to 0.01°. These findings indicate that the origin of the peak oscillation is independent of the strong peak related to the Si(220) reflection, suggesting the existence of finite domains at the SNOL surface. It should also be noted that there is a small subpeak only on the right shoulder of the Si(220) peak obtained at $\alpha = 0.1°$ and that its position is different from those of the 1st subpeaks observed at $\alpha = 0.01$, 0.02, and 0.05°. This suggests that the subpeak at $\alpha = 0.1°$ has a different origin. The identification of this subpeak is under way.

For an X-ray wavelength of 0.124 nm, the critical angle of total reflection for Si is 0.18°. Below the critical angle, the penetration depths of the X-ray beam are estimated to be 2, 3, 4, and 6 nm for grazing angles of 0.01, 0.02, 0.05, and 0.1°. These estimations indicate that the bulk Si(220) peak and additional oscillating subpeaks originate mainly from the SNOL layer and surface regions of the SNOL at grazing angles from 0.01 to 0.1°.

The additional oscillating curves at the side of the main Si(220) peak can be explained in the framework of the kinematical scattering theory, which usually works well for Si with a thickness of less than 400 nm [7], [15], [16]. To reproduce the experimental curves, we constructed a structural model for the SNOL on the insulator, assuming that the SNOL is composed of two layers (surface region and underlying layers) with two different strain levels. Moreover, the surface region is composed of finite domains with less in-plane strain ε relative to the underlying SNOL. The assumption of strain domains is reasonable because Matsui et al. found evidence that spatial strain fluctuations exist [11] in thick (>5 μm) SOLs of bonded and SIMOX wafers. We also assume that the distribution of domain size is a Gaussian function with average size D and standard deviation σ. Additionally, the Bragg profile for bulk Si is of the Voigt-type. The broken lines in Fig. 1 are the results of kinematical calculations based on the two-layer model for different angles. The simulation well reproduces the experimental data involving the asymmetric features, although some deviations can be seen at the higher angle side of the Si(220) Bragg reflection. Values of parameters ε, D, and σ obtained by fitting the simulation and experimental data for different grazing angles are listed in **Table 1**. GIXD shows that the difference in in-plane strain between the surface region of the SNOL and the underlying one is on the order of 10^{-4}. The size of the strain domain D on the surface region is about 500 nm with deviation σ of 70 nm (see Table 1). The maximum strain is in good agreement with the one obtained from a 45-nm-thick SNOL in a bonded wafer ($\varepsilon \sim 10^{-4}$) by Raman spectroscopy [8]. It should be noted, however, that the strain obtained from Raman spectroscopy is an average over the whole SNOL including its surface region. The domain size is about 100 times smaller than that observed from the thick (>5 μm) SOLs on a bonded wafer [11].

Table 1. Strain ε, domain size D, and size fluctuation σ obtained by fitting the experimental results and simulations at different grazing incident angles.

α [°]	ε	D [nm]	σ [nm]
0.01	–0.00028	490	70
0.02	–0.00028	500	70
0.05	–0.00049	480	70
0.10	0.00000	400	120

3.2 Thermal stability of thin SOI wafer

To assess the thermal stability of the SNOL, we annealed the sample at 1000°C and measured GIXD at a grazing angle of 0.01°. The GIXD curves obtained during the annealing and afterwards at room temperature are shown in **Fig. 2**. At 1000°C, the additional peaks completely disappeared at the shoulder of the Si(220) (compare Figs. 1 and 2), making the peak symmetric with respect to $\theta = 0$. After the annealing, the main peak was clearly sharper and there were no additional peaks at the shoulder of the main peak. The broken lines in Fig. 2 are simulations obtained using the fitting parameters in **Table 2**. These simulations are in good agreement with the experimental curves. Comparing Tables 1 and 2, we can say that, during annealing, the strain became smaller than the detection limit of our apparatus while the strain domains at the surface increased in size from 490 to 2000 nm. These results clearly show that the post-annealing treatment is effective at improving the spatial inhomogeneous strain distribution in the SNOL.

A possible explanation of why we observe strain domains on the surface region of the SNOL is as follows. In oxidation at 1200°C in the dilute O_2/Ar mixture, the stress balance of the SIMOX wafer with a top thermal silicon oxide redistributes. The temperature is above the onset temperature of viscous flow of

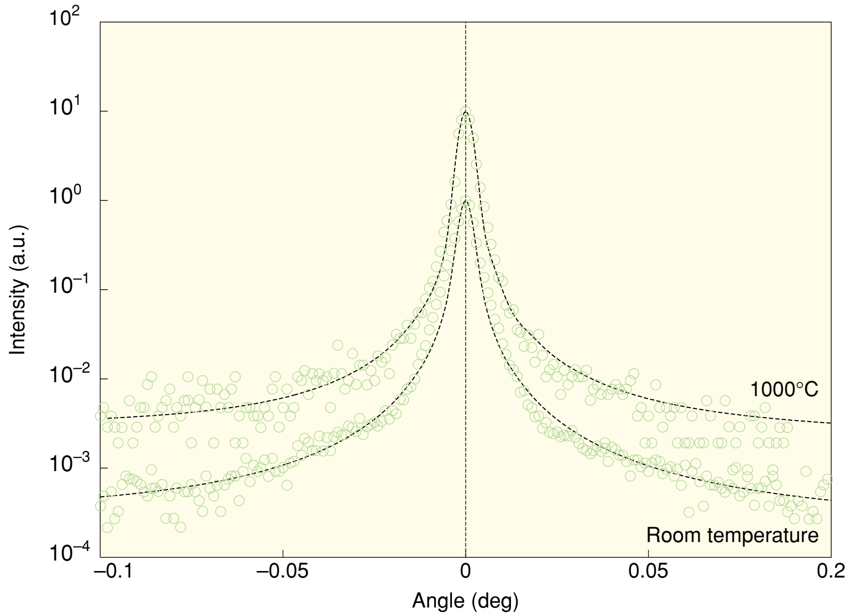

Fig. 2. Grazing incident X-ray diffraction curves of the SIMOX wafer obtained at different temperatures. The broken lines are kinematical calculations.

Table 2. Strain ε, domain size D, and size fluctuation σ obtained by fitting the experimental results and simulations at different annealing temperatures.

T [°C]	ε	D [nm]	σ [nm]
1000	–0.00028	1800	400
Room temperature	0.00000	2000	400

SiO_2 [17], indicating that the strain is fully relaxed. Moreover, the stress is expected to be nearly equal to zero at high temperature [18]. When the sample is cooled down to room temperature a new stress balance is achieved between the TOX, SNOL, BOX, and Si wafer. When the SiO_2 shrinks, the SNOL is tensily stressed by the TOX/SNOL/BOX/Si interfaces. After the etch-back of the TOX, if the SNOL is sufficiently thin (<50 nm), the tensile strain in the SNOL is partially relaxed and becomes smaller in the region close to the surface of the SNOL. This maintains the total stress balance in the SIMOX wafer, which is in agreement with the work of Camassel et al. [5]. The annealing experiment supports the idea of partial relaxation of the tensile strain in the tensilystrained SNOL. It can be considered that the defective lattice deformations on the surface region of the SNOL caused by the removal of the TOX at room temperature were sufficiently annealed out at 1000°C as a result of internal stress relaxation, yielding homogeneous strain in the SNOL of the SIMOX wafer.

4. Summary

Grazing incident X-ray diffraction was used to evaluate the lattice perfection of a thin silicon nanooverlayer (SNOL) fabricated by oxidation and etchback of a standard SIMOX wafer. We found that the surface region of a 47-nm-thick SNOL, after sacrificial oxidation at 1200°C and etch-back, was spatially inhomogeneously strained. We also found that postannealing at 1000°C improved the crystalline quality of the SNOL in the SIMOX wafer.

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