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Strain Field under the SiO_2/Si Interface Revealed by a Multiple-Wave X-ray Diffraction Phenomenon

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A multiple-wave X-ray diffraction phenomenon, i.e., interaction between Bragg reflection and crystal-truncation-rod (CTR) scattering, is applied to characterize strain field under SiO_2/Si(001) interface. Using the phenomenon it has been revealed that there is very small strain field extending over a mesoscopic-scale depth under the SiO_2/Si interface and having a static fluctuation in the lateral direction. Information on distribution of strain field has been also obtained. In the present paper depth profile of strain field induced by wet thermal oxidation at 900 ℃ is qualitatively obtained, and compared with results of dry thermal oxidation at 900 ℃ and Kr/O_2 plasma oxidation at 400 ℃.

Key words: X-ray diffraction, Silicon, Silicon oxides, Oxidation, Strain

1. INTRODUCTION

Strain near an interface affects its electronic structure, but a full understanding of such strains is still lacking even in the case of SiO_2/Si(001), which has been applied to electronic devices since 1960s. In a previous paper [1], we have proposed a new technique: the phase-sensitive x-ray diffraction (PSXD) technique. This technique is an application of a multiple X-ray diffraction phenomenon, *modulation of the crystal-truncation-rod (CTR) scattering intensity under the excitation of a Bragg reflection*, and allows us to characterize strain field in a crystal. Using the technique it has been revealed that there is very small strain field under SiO_2/Si interface, which extends over depth up to several tens of nanometer and has a static fluctuation in the lateral direction. Information on distribution of strain field has been also obtained by the technique [2]. In the present paper dependence of depth profile of strain field under SiO_2/Si(100) interface on oxidation method is presented. Depth profile of strain field induced by wet thermal oxidation at 900 ℃ is qualitatively obtained, and compared with results of other oxidation methods such as dry thermal oxidation at 900 ℃ and high-density Kr/O_2 plasma oxidation at 400 ℃ [3,4].

2. EXPERIMENT

We investigated strain fields under SiO_2/Si(100) interfaces by measuring modulation profiles of the crystal-truncation-rod (CTR) scattering on the 50 rod under the excitation of the 004 Bragg reflection (see Fig. 1) [1,2,5-11]. The PSXD technique allows us to determine the total displacement $\Delta \boldsymbol{D}_n$ (see Fig. 2) projected into the reciprocal lattice vector of the Bragg reflection $\boldsymbol{H}$. In addition the measurement becomes more sensitive to structures near the interface for a larger absolute value of Δl, which is the deviation of the momentum transfer perpendicular to the surface from a Bragg point (the 555 Bragg point in the case of Fig. 1). Thus depth profile of the strain field can be also determined; taking a lager $|\Delta l|$ allows us to measure strain field with a higher resolution in real space, which is represented by the inverse of $|\Delta l|$ [2,8]. We can change Δl by changing the wavelength of the incident X-rays.

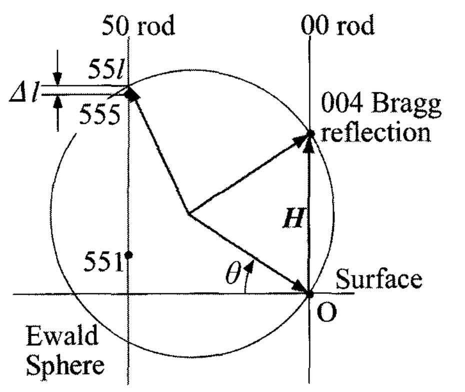

Fig. 1. Illustration of diffraction condition where intensity of CTR scattering is modulated by the excitation of a Bragg reflection. The parameter l is the momentum transfer perpendicular to the surface, and Δl is the deviation of l from the 555 Bragg point.

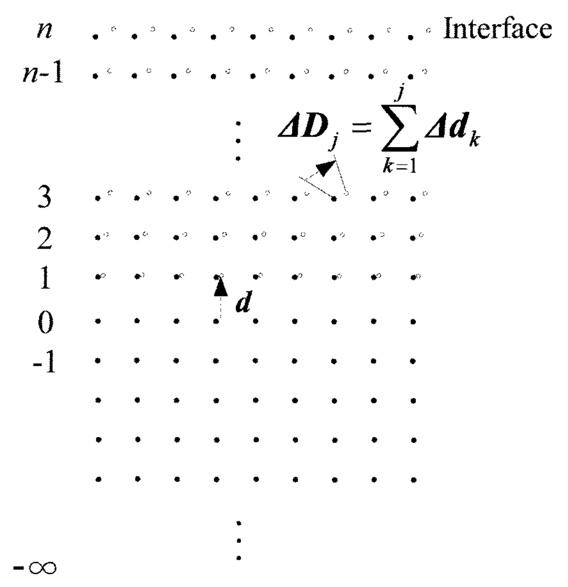

Fig. 2. Illustration of a model of mesoscopic-range strain field. A strained layer containing n atomic planes is formed on an ideal semi-infinite perfect crystal. The filled circles represent to the positions of atoms or unit cells in bulk crystal, and the open circles represents those in the strained layer under the interface. The parameter $\Delta \boldsymbol{D}_j$ represents the sum of displacements under the jth atomic plane, and $\Delta \boldsymbol{d}_j$ does the deviation of the lattice spacing between the $(j-1)$th and jth atomic planes from the lattice spacing in bulk, $\boldsymbol{d}$.

The experiment was performed at BL09XU in SPring-8, where a high-brilliance horizontally polarized X-ray beam from the undulator is available [12]. The premonochromatized SR beam was shaped by slits into a size of 1 mm (vertical) × 1 mm (horizontal), and then highly monochromatized by using two 440 Bragg reflections from two Si(220) channel-cut crystals arranged in the (+ +) geometry. The wavelengths of X-rays were fixed around 1.24 Å. The sample was first oxidized at 1000 ℃ in a dry oxygen atmosphere, so that thermal oxide layer about 500 nm was formed on it. Next the oxide layer was removed by HF solution, and oxidized again to form an oxide layer of about 1000 nm thickness. Then the oxide layer was removed again, and finally oxidized by wet oxidation process at 900 ℃. The thicknesses of the oxide layer were estimated to be 1.9 nm by an ellipsometer.

3. RESULTS AND DISCUSSION

Examples of experimentally obtained modulation profiles are shown in Fig. 3. The abscissa is the deviation of the incidence angle from the center of the 004 Bragg reflection. A modulation profile is mainly characterized by two parameters, the 'phase' and 'visibility' of the profile: the 'phase', which corresponds to the peak or dip position of the modulation profile, reflects $\Delta \boldsymbol{D}_n \cdot \boldsymbol{H}$, while the 'visibility' does to static strain fluctuation in the lateral direction [1]. In Fig. 3 a result of the wet oxidation is shown with results of Kr/O_2 plasma oxidation at 400 ℃ and dry oxidation at 900 ℃ [2]. Difference among modulation profiles is clearly seen; it reflects difference in strain field under SiO_2/Si(001) interface. On the other hand there is no difference among rocking curves of Bragg reflections for the three samples; they were explained by a perfect crystal (broken lines). This fact supports that the depths of the strain fields are sufficiently small compared with the extinction depth of the 004 Bragg reflection. The black and gray solid lines represent calculated curves for $\Delta \boldsymbol{D}_n \cdot \boldsymbol{H}$'s of −12% and −9.5%, respectively; the former is in good agreement with the experimental result of the dry oxidation, while the latter is with those of the Kr/O_2 plasma and the wet oxidations.

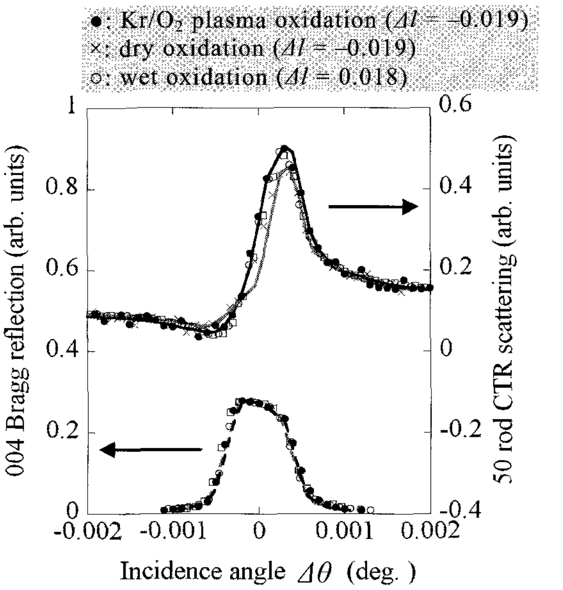

Fig. 3. Examples of experimentally obtained modulation profiles. The abscissa is the deviation of the incidence angle from the center of the 004 Bragg reflection (denoted by $\Delta\theta$). The filled circles correspond to the CTR scattering and 004 Bragg reflection in the case of the Kr/O_2 plasma oxidation, while the open circles and crosses correspond to those in the cases of wet and dry oxidations at 900 ℃. The thicknesses of the oxide layers of the three samples are 3.1, 2.6, and 1.9 nm, respectively. The broken lines correspond to the theoretically calculated curves for the 004 Bragg reflection.

Figure 4 shows depth profiles of strain fields obtained qualitatively by experimental results. In the case of the wet oxidation at 900 ℃ the distribution of strain field should be almost constant, which is supported by the fact that $\Delta \boldsymbol{D}_n \cdot \boldsymbol{H}$'s were almost constant (−9.5%) for different Δl's (different resolutions in real space) along the rod (Δl = −0.023, −0.016, −0.008, 0.015, and 0.018). In the case of the dry oxidation at 900 ℃ the tendency was same as that of the wet oxidation except that $\Delta \boldsymbol{D}_n \cdot \boldsymbol{H}$'s were somewhat lager (−12%) than that of the wet oxidation [2]. These results make contrast to the case of the Kr/O_2 plasma oxidation at 400 ℃, where strain field should change near the interface; $\Delta \boldsymbol{D}_n \cdot \boldsymbol{H}$ was −12% at Δl = 0.012, which was somewhat larger than those at $|\Delta l|$ = ±0.019 (–9.5%) [2]. This should be attributed qualitatively to drastic change in the strain field near the interface; the results of higher resolution measurements with larger $|\Delta l|$'s are considered to give more detailed information on distribution of $\Delta \boldsymbol{D}(z)$. More quantitative analysis will be presented elsewhere. Although further systematic investigation should be needed, we believe that the result should reflect how each oxide layer grows at the SiO_2/Si interface and is attributed to difference in growth mechanism of oxide layer.

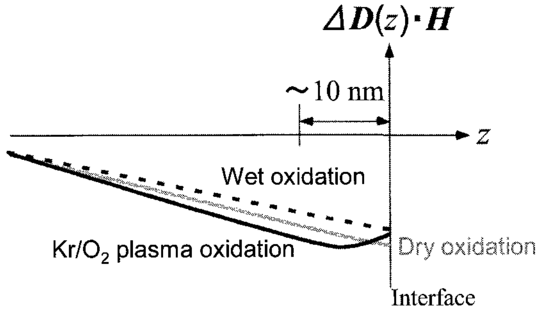

Fig. 4. Depth profiles of strain fields under the SiO_2/Si interfaces, where z represents the depth and $\Delta \boldsymbol{D}(z)$ is the sum of displacements defined in Fig. 2. The dotted and gray solid lines correspond to the wet and dry oxidations at 900 ℃, respectively, and the black solid line does to Kr/O_2 plasma oxidation at 400 ℃.

4. CONCLUSION

The phase-sensitive x-ray diffraction (PSXD) technique was applied to investigate dependence of strain field under SiO_2/Si(100) interface on oxidation method. Depth profile of strain field induced by wet oxidation at 900 ℃ was qualitatively obtained, and compared with results of dry oxidation at 900 ℃ and Kr/O_2 plasma oxidation at 400 ℃. In the case of the wet oxidation $\Delta \boldsymbol{D}_n \cdot \boldsymbol{H}$'s were almost constant (–9.5%) for different Δl's (different resolutions in real space) along the rod, which indicates that the distribution of strain field should be almost constant. This tendency was same as that of dry oxidation except that $\Delta \boldsymbol{D}_n \cdot \boldsymbol{H}$'s were somewhat lager (–12%) than that of the wet oxidation, and made contrast to the case of the Kr/O_2 plasma oxidation, where drastic change in strain field was indicated near the interface.

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