

Quantitative Analysis of Stress Relaxation in TEM specimen fabrication by Raman Spectroscopy with High-NA Oil-Immersion Lens

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1. Introduction

Transmission electron microscopy (TEM) has been used for evaluation of stress in Si (e.g., convergent beam electron diffraction and nano beam diffraction).¹ The stress induced in a micro area can be measured owing to high-spatial resolution in the TEM observation. However, stress relaxation is inevitable during the fabrication of the TEM specimen. The stress relaxation occurred remarkably in the direction perpendicular to the milling facet. It is very important to know the extent of the stress relaxation for obtaining meaningful results in stress measurements by TEM. We performed to examine the stress relaxation in the TEM specimens by using Raman spectroscopy with a high-numerical aperture (NA) oil-immersion lens. Using the high-NA oil-immersion lens, it is possible to evaluate anisotropic biaxial stress even in a (001)-oriented Si substrate, while conventional Raman spectroscopy fails to measure stress unless uniaxial or isotropic biaxial stress is assumed.²⁻⁶

2. Experimental Procedure

Strained-Si on insulator (SSOI) was used as the sample. SSOI has a 70-nm thick strained-Si layer on 145-nm thick buried oxide followed by a Si substrate (See Fig. 1). TEM specimens of SSOI were fabricated by focused ion beam (FIB). Various thicknesses of the TEM specimens were prepared, as shown in Fig. 2, which were 2820, 1020, 630, 470, and 240 nm, respectively. The coordinate system in the experiments is also shown in Fig. 2.

The extent of the stress relaxation was evaluated by Raman spectroscopy with a high-NA oil-immersion lens. The anisotropic biaxial stresses σ_{xx} and σ_{yy} can be measured by using the high-NA oil-immersion lens.³ NA and the refraction index of oil are 1.7 and 1.8, respectively. Nd YAG laser ($\lambda = 532$ nm) was used as the excitation source. The maximum angle of the collection cone inside Si is 24.5° in the immersion condition.⁶ Figure 3 shows the maximum angle as a function of NA. Figure 3 also shows the ratio of the electrical field intensity of z-polarized light (perpendicular to the Si surface) to laterally polarized light (parallel to the Si surface). The ratio is obtained by the integration over the solid angle conducted to the collection cone for the scattered light and incident light.⁷ As is seen in Fig. 3, the ratio remarkably increases with an increase in NA, which is 3.8% at NA of 1.7. TO phonon modes in Si at a Γ point can be excited with the use of the z-polarized light.

3. Results and Discussion

Excitation of TO phonon mode in Si

Figure 4 shows the Raman spectra from SSOI in the

LO- and TO- active conditions. The excitation source light penetrates through the strained-Si layer and the buried oxide layer and reaches the Si substrate. Therefore, two peaks are observed in the Raman spectra. It is noted that the peak positions on the higher wavenumber side originating in the Si substrate were kept constant, nevertheless, the peak positions on the lower wavenumber side originating in strained-Si were changed. This result indicates the Raman polarization selection rule. We therefore conclude that the TO phonon mode can be excited by using the high-NA oil-immersion lens. The wavenumber shift of the lower peak from the wavenumber of the Si substrate (defined to be 520 cm^{-1} in this study) is -4.56 cm^{-1} in the LO-active condition, while that in the TO-active condition is -3.54 cm^{-1} . The relationship between wavenumber shifts and biaxial stresses σ_{xx} and σ_{yy} are as follows,

$$\Delta\omega_{TO} = -2.31 \times \sigma_{xx} - 0.37 \times \sigma_{yy}, \quad (1a)$$

$$\Delta\omega_{LO} = -1.93 \times \sigma_{xx} - 1.93 \times \sigma_{yy}. \quad (1b)$$

The calculated biaxial stresses σ_{xx} and σ_{yy} in the strained-Si layer of SSOI were 1.35 and 1.02 GPa, respectively.

Stress relaxation in TEM specimens

Figures 5 and 6 show the Raman spectra from the TEM specimens of SSOI with the thicknesses varied from 2820 to 240 nm obtained in the LO- and TO- active conditions, respectively. The Raman spectra remarkably change as the specimen thicknesses reduce from 2820 to 240 nm.

Figure 7 shows the wavenumber shifts of the LO- and TO- phonon modes in SSOI as a function of the TEM specimen thickness. As is seen here, both the wavenumber shifts originating in the strained-Si layer decreases with a decrease in the thickness. The reason lies in that the stress in SSOI relaxed by the FIB milling. Figure 8 shows the biaxial stresses σ_{xx} and σ_{yy} in SSOI as a function of the TEM specimen thickness. The biaxial stresses in the TEM specimen with the thickness of 2820 nm are almost the same as the value of the SSOI substrate. However, as thinning the specimen, the value of the stress decreases especially in σ_{yy} perpendicular to the FIB milling facet. In addition, we note that σ_{xx} as well as σ_{yy} also decreases with a decrease in the thickness. As a result, the stress relaxation is summarized in Fig. 9, which is calculated by the equation below;

$$\text{Stress relaxation} = \frac{(\text{Stress in SSOI}) - (\text{Stress in TEM specimen})}{(\text{Stress in SSOI})}$$

The values of the relaxation of σ_{yy} and σ_{xx} reached to 75% and 30%, respectively, at the thickness of 240 nm.

3. Conclusions

The stress relaxation in the TEM specimens of SSOI was evaluated by Raman spectroscopy with high-NA oil-immersion lens. TO phonon mode can be excited by the z-polarized light owing to the use of high-NA lens. This allows us to measure the anisotropic biaxial stresses σ_{xx} and σ_{yy} . The stress relaxation in the TEM specimens increases as the specimen thickness decreases. It was confirmed that the values of the relaxation of σ_{yy} and σ_{xx} were 75% and 30%, respectively, at the thickness of 240 nm.

Acknowledgements

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References

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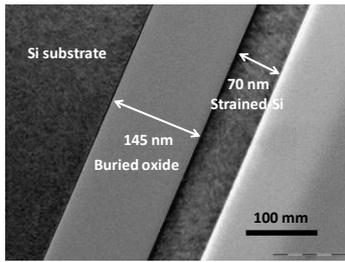


Fig. 1 TEM image of SSOI structure.

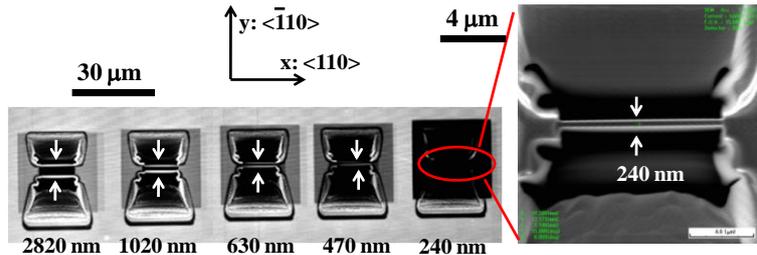


Fig. 2 TEM specimens of SSOI fabricated by FIB.

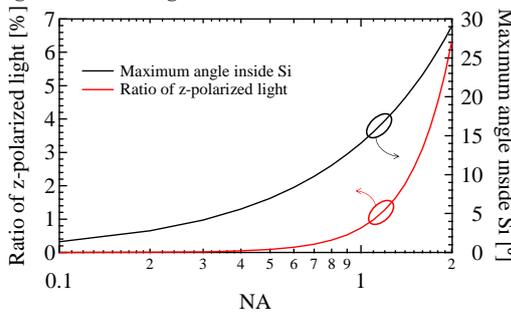


Fig. 3 Maximum angle of collection cone inside Si and ratio of electrical field intensity of z-polarized light as a function of NA.

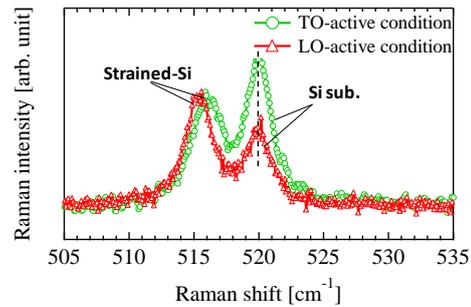


Fig. 4 Raman spectra from SSOI in LO- and TO-active conditions.

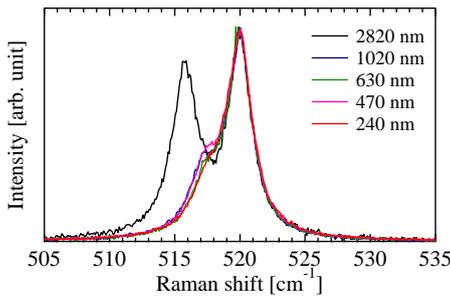


Fig. 5 Raman spectra from TEM specimens of SSOI with the thicknesses varied from 2820 to 240 nm obtained in LO-active condition.

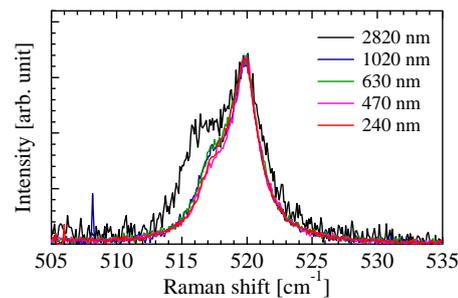


Fig. 6 Raman spectra from TEM specimens of SSOI with the thicknesses varied from 2820 to 240 nm obtained in TO-active condition.

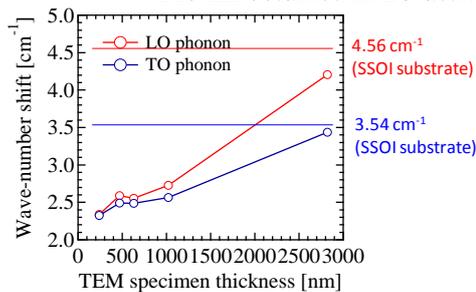


Fig. 7 Wavenumber shifts of LO- and TO-phonon modes in SSOI as a function of TEM specimen thickness.

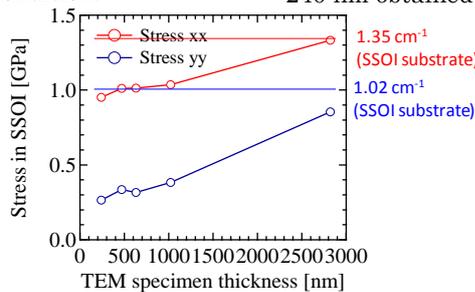


Fig. 8 Biaxial stresses σ_{xx} and σ_{yy} in SSOI as a function of TEM specimen thickness.

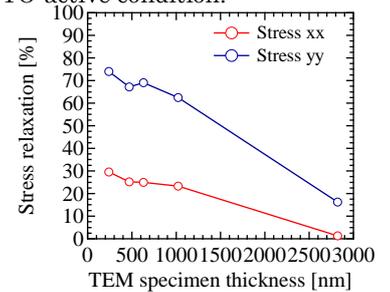


Fig. 9 Stress relaxation in TEM specimens of SSOI as a function of specimen thickness.