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Effect of stress on defect transformation in hydrogen implanted silicon and SOI structures

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ABSTRACT

Transformation of defects in hydrogen implanted silicon and silicon-on-insulator structures caused by external pressure of argon ambient (up to 1.5 GPa) at the stage of defect removal in implanted material and high temperature annealing SOI structures is reported. The results are compared to these for crystals annealed at argon atmosphere of ambient pressure. Formation of the new phase crystallites was found in SOI structures annealed at high temperature in conditions of high pressure (1.2 GPa). Small insulations were also observed in hydrogen implanted silicon, which can be patterns of the new phase. Two reasons can cause phase transformation in the top silicon layer of as-bonded SOI structures: high hydrogen concentration and high local strain.

Keywords: silicon, hydrogen implantation, silicon-on-insulator, high pressure, new phase

1. INTRODUCTION

Hydrogen implantation is known to be used for Silicon-On-Insulator (SOI) structure fabrication by Smart-Cut or similar technologies¹⁻³. This SOI technology includes the bonding of hydrogen implanted wafer with another substrate at relatively low temperatures (400 - 600°C) and demands the high temperature (1100°C) annealing of SOI to improve structural and electrical properties^{1,3}. Furthermore, silicon layers oversaturated with hydrogen are seemed to be perspective for optical applications⁴. The utilisation of high pressure (HP) at the stage of high temperature (HT) treatments can affect defect transformation⁵⁻⁷. As it was found in⁵ HP-HT treatments cause the increase in concentration of oxygen precipitates and decrease in their sizes in Cz-Si, the increase in thermal donor concentration in hydrogen implanted silicon⁶ and decrease in dislocation density in oxygen implanted crystals⁷. The aim of the present efforts was to investigate the transformation of defects and structure in Si:H and SOI structures caused by annealing at external pressure.

2. EXPERIMENTAL

The Czochralski- and Floating Zone grown silicon with (100) and (111) orientation were used as initial crystals. Hydrogen implantations (H_2^+) were carried out with the energy of 130 keV in the dose range of 4 - 6×10^{16} cm⁻². The ion projected range is equal to 0.52 μ m. SOI structures were fabricated by bonding at the temperature of 450°C and with the final annealing at 1100°C for 1 hour. The thicknesses of layers of the SOI structure were about 0.48 μ m for the top silicon layer and 0.4 μ m for the buried oxide. Hydrostatic pressure of argon ambient up to 1.5 GPa was used at the stage of defect removal in implanted material and high temperature annealing SOI structures. The results are compared to those for crystals annealed at argon atmosphere of ambient pressure (AP).

X-ray investigations were performed using a High-resolution diffractometer in a double and triple configuration. Rocking curve and reciprocal space maps were recorded after HP-HT and AP-HT treatments. A high-resolution experimental set-up was realised by employing a four-crystal Ge (220) Bartels-type monochromator in the primary beam

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and a channel-cut double-reflection Ge (220) analyser in the diffracted beam. Secondary ion-mass spectroscopy (SIMS) (Cameca Riber, sputtering by Cs^+ ions), electron diffraction, and transmission electron microscopy (TEM and HREM) were also used for investigation. Electrical measurements with using of high-frequency capacity-voltage (CV) technique were done for Si:H and SOI samples. CV measurements were done with using a mercury probe.

3. RESULTS

The hydrogen depth distributions in implanted silicon annealed at different temperatures and pressures are presented in Fig.1. Some delay in hydrogen out-diffusion is observed in the case of HP treatments. But the hydrogen sheet concentration in the last case is higher than that in AP treated sample by approximately 10-20%. The AP annealing of Si:H samples at 1100°C for 1 hour leads to decrease in hydrogen concentration lower than limit of SIMS sensitivity, whereas HP treated Si:H samples still conserve some hydrogen atoms.

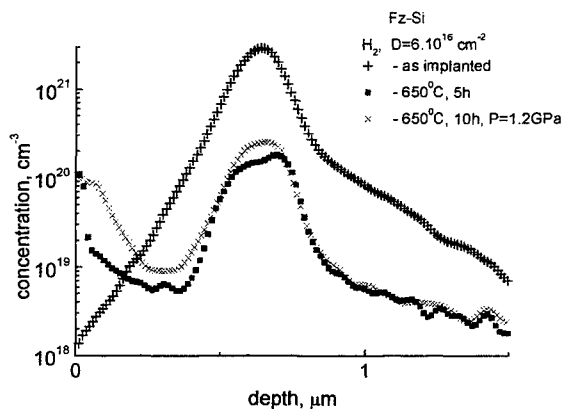


Fig.1 Hydrogen profiles obtained by SIMS for samples annealed at different temperatures and pressures.

The hydrogen profiles for as-bounded and annealed SOI structures are presented in Fig.2. As-bounded SOI contains the high hydrogen concentration in the top silicon layer (about $1.6 \times 10^{16} \text{ cm}^{-2}$). Practically complete removal of hydrogen from SOI structure was found for the sample annealed at 1100°C at AP for 0.5 hour. A slightly higher hydrogen concentration ($\sim 10\%$) in the top silicon layer and Si/SiO₂ interface was observed for the sample annealed at 1100°C in HP conditions.

Figure 3 presents the cross-section of hydrogen implanted samples (Si:H) and samples annealed at the temperature of 450°C at ambient and high pressure. The HP effect, which one can observe in Fig.3 is a suppression of formation of large cracks at the depth of maximum destruction of defects produced by ions. If for samples annealed at 450°C some small and very narrow (about 10 nm) cavities are observed than for samples annealed at $T \geq 650^\circ\text{C}$ cavities are not practically seen. The second interesting moment is that visible defects are expanded towards the surface in the case of HP-HT treatments. The size of these defects becomes smaller but they occupy the main part of the layer from surface to ion projected range (R_p). On the contrary the end-range defects have a lower density in the case of HP-HT treatments.

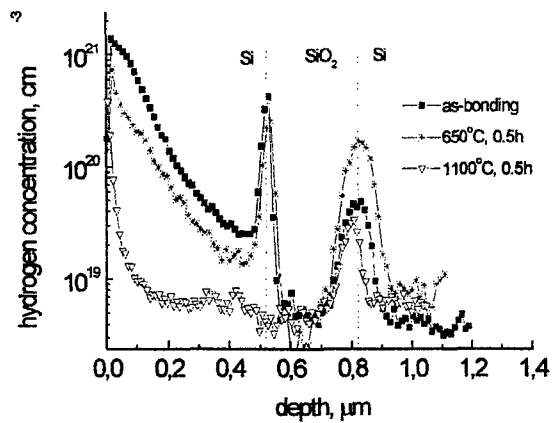


Fig.2 Depth hydrogen distribution for as-bonded SOI structure and annealed one

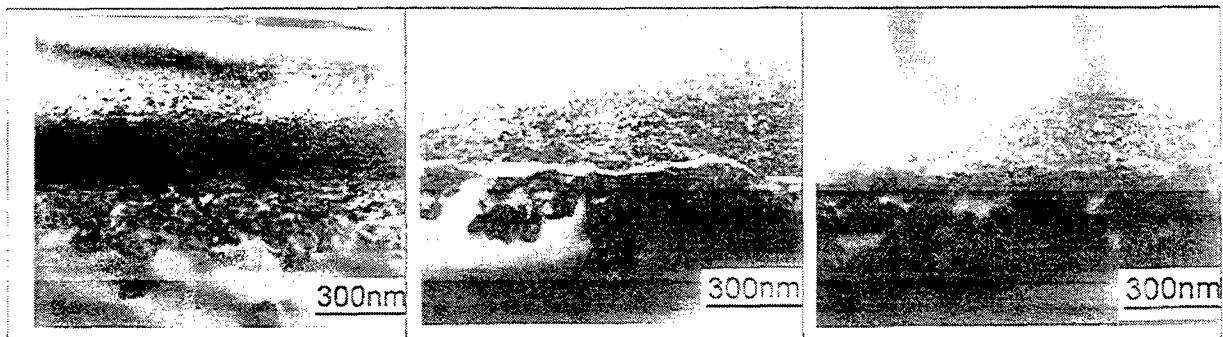


Fig.3 Cross section of hydrogen implanted sample (left), and samples annealed at 450°C for 2 hours at atmospheric pressure (middle) and 1.2 GPa (right).



Fig.4 Cross section with atomic resolution (HREM) of hydrogen implanted sample (crack) annealed at 650°C for 2 hours at 1.2 GPa.

Some incorporations can be seen near the cavities in HP treated samples (Fig.4). These incorporations have very small summary volume, and the measurement of electron diffraction for this sample gives the usual diffraction pattern as for a single crystal silicon.

The rocking curve full widths at half maximum (FWHM) for ω scans in double mode for Si:H samples are given in Table 1. Enhanced pressure of argon ambient during treatment at 1100°C results in high FWHM in comparison with AP annealed samples. The reciprocal space maps for HP-HT treated Si:H samples shows the high diffuse scattering intensity. These facts indicate development of mosaic structure (high defect concentration) after HP-HT treatments.

The rocking curves in double configuration (ω -scan) and $2\theta/\omega$ -scan of the (004) reflection for the SOI structures annealed at 1100°C for 1 hour at different pressures are presented in Figs. 5a-d. The peaks from the top silicon layer and the substrate are separated in the ω -scan (Fig.5a,b). This separation of peaks is connected to the strain in the SOI top layer and also with the tilt between the layer and the substrate. The $2\theta/\omega$ -scan for the AP annealed SOI indicates the presence of the thickness fringes. The thickness value calculated from the fringes is equal to that obtained by different methods. Lack of the thickness-related fringes in the HP treated SOI (Fig.5d) is probably caused by unhomogeneity of top silicon layer. The FWHM values for the top silicon layers of the SOI structures and substrates are given in Table 1. The FWHM value for the Si substrate did not differ from that of a typical silicon wafer. Broadening of the rocking curve for top silicon film in a double and triple configuration may be caused by the fluctuation of the lattice constant (caused by thickness effect, presence of defects, local strains, new phase, and so on). The calculated FWHM broadening for the 0.4 μm thick top Si layer, caused by the thickness effect, is equal to about 25 arcsec. As it is seen from Table 1, the FWHM value for the silicon layer of the AP annealed SOI structures is higher than that of the typical silicon single crystal even considering for thickness effect. In the case of HP treated SOI the FWHM value is very high.

Table 1 FWHM obtained in double and triple beam configurations and strain values for investigated samples.

Sample	FWHM Double conf.		FWHM Triple conf.		Strain
	film	substrate	film	substrate	
Si:H as-implanted	27		-		-
Si:H, 1100C, AP	15		-		-
Si:H, 1200C, 1.5GPa	23		-		-
SOI, as-bonded	135	15	80	9	5×10^{-4}
SOI, 1100C, AP	60	15	48	9	2×10^{-5}
SOI, 1100C, 1.2GPa	125	15	108	9	1.5×10^{-4}

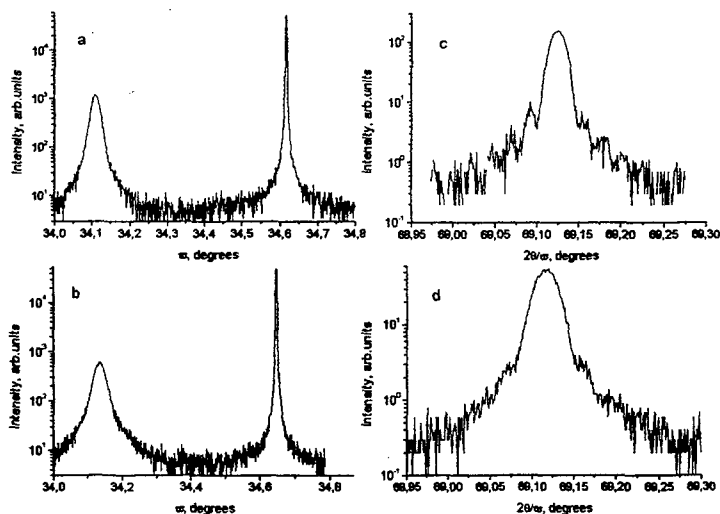


Fig.5. ω -scan of the 004 reflection in double configuration (a,b) and $2\theta/\omega$ -scan (c,d), from the SOI structures annealed at 1100°C for 1 hour at atmospheric pressure (a,c) and 1.2 GPa (b,d). Left and right peaks in ω -scans correspond to the top silicon layer and substrate, respectively.

If AP annealed SOI subjects to additional treatment at HP the perfection of the silicon top layer does not change.

The strain in the top silicon layer (defined as the relative change of the (004) plane spacing for the top Si layer with respect to that for the substrate) is determined as $\frac{\Delta a}{a} = \frac{a_L - a_S}{a_S}$, where a_L and a_S are the lattice

constants for the top Si layer and the substrate, respectively. The $\frac{\Delta a}{a}$ values measured from the rocking curves of the

same reflection for $\varphi = 0^\circ$ and 180° (φ denotes a rotation of the sample around the axis perpendicular to the sample surface) for the SOI samples are also given in Table 1. The strain value is high in the as-bonded SOI structure and in HP treated one.

Electron diffraction patterns and dark field TEM images for SOI structures annealed at different pressure are presented in Fig.6. AP annealing of SOI structure leads to a single crystalline top silicon layer. Mosaic-like structure is observed in TEM images. Additional annealing at HP of the AP treated SOI does not change defect structure. In the case of HP treated SOI the formation of new phase crystallites was found. Electron diffraction pattern reveals additional reflexes and twinning of reflexes.

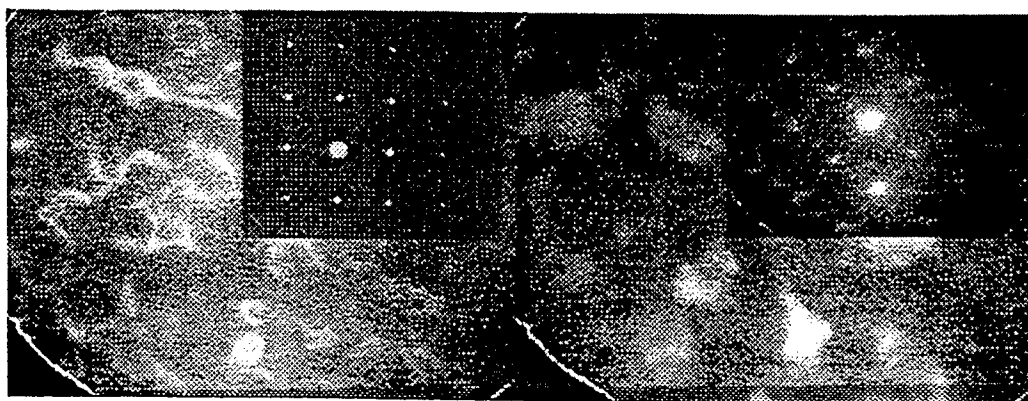


Fig.6 Dark field TEM micrograph and electron diffraction pattern from the (001) oriented top silicon layer of SOI structures annealed at atmospheric pressure (left) and at 1.2 GPa (right). The micrograph size is $1\mu\text{m} \times 0.75\mu\text{m}$.

CV measurement shows that the top silicon layer in as-bounded SOI structures has n-type of conductivity with concentration about $(2-3) \times 10^{17} \text{ cm}^{-3}$. For SOI annealed at 1100°C in AP condition the electron concentration is equal to $(3-5) \times 10^{15} \text{ cm}^{-3}$. The silicon layer of HP treated SOI is a high resistive layer, which can not be measured by CV technique.

4. DISCUSSION

HP treated Si:H samples and SOI show a very different structure as compared to AP annealed ones. The most pronounced changes are observed in SOI. According to data presented in Fig.6, the patterns of a new phase are formed in the top silicon layer. Phase transformation is obviously the reason of high FWHM value and strain for this structure. The small insulations observed for Si:H samples can also be patterns of a new phase or twins. They are too small for real identification.

Stable hexagonal-wurtzite silicon crystallites up to $20\mu\text{m}$ were found to form in a-Si:H layer directly deposited at low pressure using ultraviolet laser ablation [8], in a silicon oxide deposition process with NN2O-Silane plasma [9], or in CVD silicon [10]. In all cases there were layers which contained high hydrogen concentration. Presence of hydrogen in our structures can stimulate phase transformation. In as-bounded SOI effect can be intensified by high local strain presented in the top silicon layer due to the roughness of the Si/SiO₂ interface: the layer and the substrate were not contacting at all points of the interface with the same strength resulting in incomplete adhesion. The insulations observed in Si:H samples are also formed in the place of maximal local strain in hydrogen implanted sample – at the border of the cavity.

The AP-HT annealing of SOI leads to homogeneous adhesion on bonded interface and practically complete removal of hydrogen from the top silicon layer. As a result, the additional HP-HT treatment of AP annealed SOI does not cause phase transformation.

5. CONCLUSIONS

Formation of the new phase crystallites was found in SOI structures annealed at high temperature in conditions of high pressure (1.2 GPa). The significantly low pronounced effect is observed in Si:H samples. Two reasons can cause phase transformation in the top silicon layer of as-bonded SOI structures: high hydrogen concentration and high local strain.

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