Electrical properties of multiple-layer structures formed by implantation of nitrogen or oxygen and annealed under high pressure

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Silicon-on-insulator-like structures formed in either oxygen- or nitrogen-implanted silicon during anneals under atmospheric and enhanced hydrostatic pressure are characterized by means of electrical techniques (current-voltage and capacitance-voltage measurements). It was found that the application of high pressure (~1 GPa) stimulates the formation of a perfect top silicon layer and results in the degradation of the properties of the buried insulator. The latter effect is caused by defect accumulation in the buried insulator and leads to a decrease in the effective thickness of the insulator layer as extracted from capacitance-voltage measurements. Pressure-stimulated formation of electrically active centers (donors and acceptors) in the top silicon layer and substrate was found. The fixed charge in the oxide was found to be independent on the pressure applied during anneals, whereas the negative charge in the nitride increased with pressure. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2168233]

I. INTRODUCTION

Separation of silicon layer from substrate by oxygen or nitrogen implantation is a usual technique for the development of silicon-on-insulator materials. Implantation of oxygen to produce the separation by implantation of oxygen (SIMOX) structures is now a well-established and widely used technique for the creation of silicon-on-insulator (SOI) wafers.[1] Due to unsolved problems (e.g., crystallization of the nitride layer, large leakage current, and nonuniform Si/Si_3N_4 interface) nitrogen implantation is not widely applied in modern electronics.[2] In order to benefit both from the advantages of the produced silicon nitride (radiation hardness of Si_3N_4 insulator, diffusion inhibition of contaminations, and impurity gettering) and of the silicon dioxide (low leakage current and a planar interface between the top Si layer and the buried oxide), coimplantation of oxygen and nitrogen was employed for the fabrication of SOI material with combined insulator [separation by implantation of oxygen and nitrogen (SIMON) structures].[3] SIMON SOI materials are considered as very promising structures for the development of total-dose–radiation-hardened materials.[4] The common problem for all implantation-based technologies of SOI fabrication is the complete removal of radiation defects introduced due to the high doses used.

A perspective way to manage defects and impurities in implanted silicon crystals is the application of high pressure (HP) during high-temperature (HT) postimplantation anneals. Crystals with more homogeneous distributions of higher concentration of defects or/and impurity clusters of smaller sizes are the main effect of the HP treatment.[5,6] In the present work, the electrical and structural properties of silicon material implanted with high dose either with oxygen or nitrogen ions and subsequently heat treated under enhanced hydrostatic pressure were studied. It was found that high pressure (~1 GPa) applied during anneals is an effective tool to manage the annihilation and redistribution of the radiation defects in the implanted samples. It was also found that the application of HP during postimplantation anneals changes the defect distribution between the insulator layer and the top silicon layer. It also affects the charges at the interfaces and the donor concentration in the top silicon layer.

II. EXPERIMENT

(100)-oriented float-zone silicon (FZ-Si) and Czochralski-grown silicon (Cz-Si) crystals of *p*-type conductivity were implanted with oxygen or nitrogen ions. The energy of the implanted O^+ ions was 170 keV and doses of 6×10^{17} and 2×10^{18} cm^{-2} were used. The projected range of the O^+ ions was 0.35 μm. The energy of the implanted N_2^+ ions was 140 keV and doses of 1×10^{17} and 8.5×10^{17} cm^{-2} were used. The projected range of the N_2^+ ions was 0.18 μm. Postimplantation anneals were performed in the temperature range of 800–1300 °C under hydrostatic pressure up to 1.4 GPa, in argon ambient. This pressure value is the upper limit of the used equipment. Secondary-ion-mass spectroscopy (SIMS), cross-sectional transmission electron microscopy (XTEM), and current-voltage (*I-V*) and capacity-voltage (*C-V*) measurements were employed for sample characterization. High-frequency (1 MHz) *C-V* mea-

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surements were performed on vertical mesa structures with the use of Al contacts or a mercury probe. Vertical structures consist of a top Si layer, a buried oxide, and a substrate. *I-V* measurements were performed with the use of point probe on the top silicon layer.

Approximate values of the electrical parameters of the SOI-like structures are extracted from *C-V* characteristics, where the SOI structure is considered as two metal-dielectric-semiconductor structures connected in a series.[7] From the maximum values of the capacitance in the *C-V* curves we extracted an effective thickness of the buried insulator. The effective thickness of the buried insulator should be equal to the real thickness of the insulator layer or even higher if a depletion layer exists in silicon for all the used voltages. Other parameters extracted from the *C-V* curves are the fixed charges in the insulator near both interfaces, that is, the charge Q_f at the top Si/buried insulator interface and the charge Q_s at the buried insulator/substrate interface. The charge values were estimated with the use of the flatband voltage, V_{FB}, and the middle gap voltage, V_{mg}.[8] In the first case, the charge value is the sum of the fixed charge in the buried insulator and the charge at the interface traps. In the second case, only the fixed charge in the buried insulator is estimated. The difference between these two values of charges allows us to estimate the density of the interface traps. The range of the capacitance modulation was used for the estimation of the carrier concentration in the top silicon layer or the substrate. It was necessary to take into account that the extracted value of the carrier concentration N_f in the top silicon layer could be reduced depending on the thickness of the top silicon layer.

III. EXPERIMENTAL RESULTS

A. The structures prepared by oxygen implantation

XTEM images of samples implanted with oxygen ions and subsequently annealed at different temperatures and pressures, for the same time duration, are presented in Fig. 1 (compare Refs. 9 and 10). For lower annealing temperatures (1130 °C/1200 °C, 1 GPa) [Figs. 1(a) and 1(b)] there are no defects in the silicon layer. Comparison of the images for samples annealed at the same high temperature (1300 °C) under low and high pressures [Figs. 1(c) and 1(d)] shows that the application of high hydrostatic pressure (>0.1 GPa) [Fig. 1(d)] leads to the creation of a perfect top silicon layer and a substrate with a lot of defects formed in the buried oxide. Also, layers with dislocations are formed at the interface of the top silicon layer and the buried oxide. Nevertheless, anneals of implanted samples at 1300 °C, under low pressure (<0.1 GPa) [Fig. 1(c)] result in the formation of a relatively low concentration of defects in the buried oxide, with a lot of defects observed in the top silicon layer and the substrate. The thickness values of the top silicon layer and the buried oxide are 300 nm/140 nm and 280 nm/160 nm for anneals at 1300 °C, under 10^{-2} and 1 GPa, correspondingly. In the latter case, the thickness value of the buried oxide includes the thickness value of the layer with dislocations at the interface.

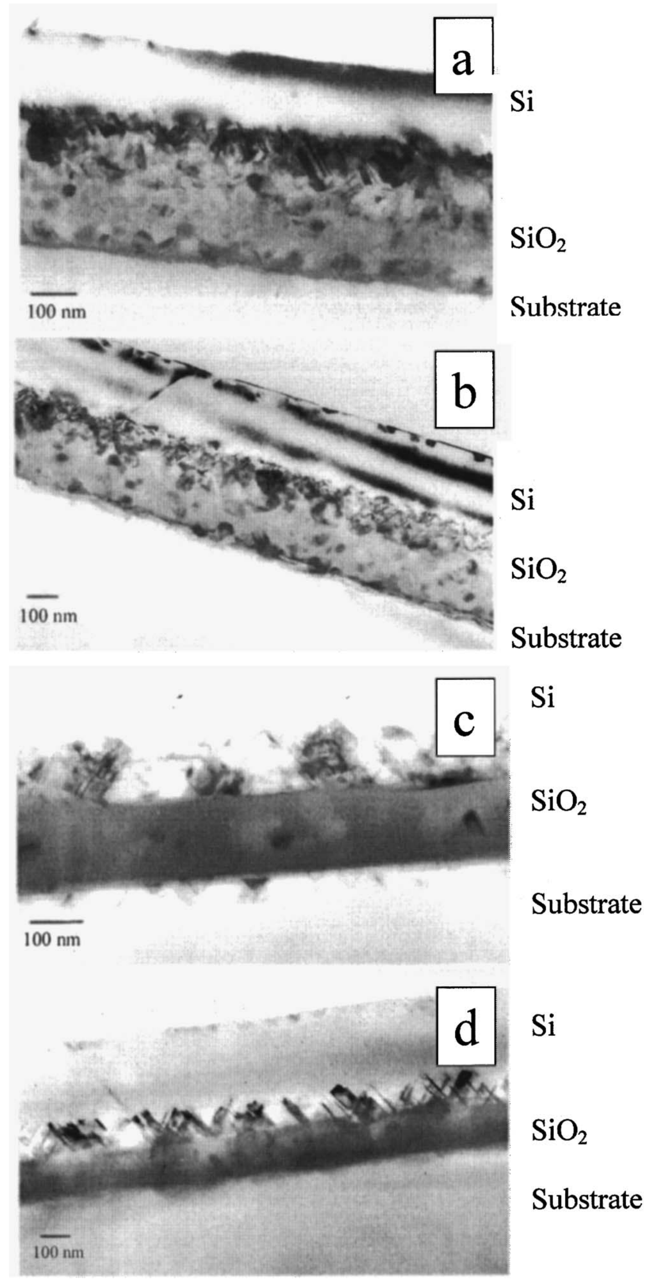

FIG. 1. XTEM images of silicon implanted with O^+ ions of 6×10^{17} cm^{-2} dose and subsequently annealed at 1130 °C, 1.0 GPa, 5 h (a); 1200 °C, 1.0 GPa, 5 h (b); 1300 °C, 10^{-2} GPa, 5 h (c); and 1300 °C, 1.2 GPa, 5 h (d).

The current through the buried oxide of the Si:O structures was lower than 1 μA for the used regimes of the HP-HT treatments. *C-V* characteristics for structures annealed at 1130 °C under different pressures are presented in Fig. 2. There are two voltage intervals where the capacitance decreases. To understand the relation between the formation of the depletion areas in the top silicon layer or in the substrate and the intervals of capacitance modulation, etching of the top silicon layer and repeated *C-V* measurements were made. The first interval, at negative voltages, corresponds to the formation of a depletion regime in the substrate while an accumulation regime in the top layer still exists. In the second interval, at positive voltages, a depletion regime occurs both in the top Si layer and the substrate. Considering that

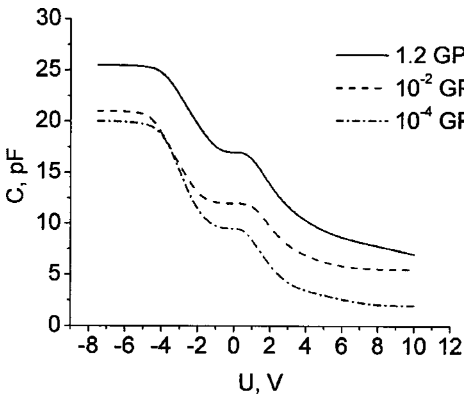

FIG. 2. *C-V* characteristics for samples implanted with O^+ ions of 6×10^{17} cm^{-2} dose and subsequently annealed at 1130 °C, under different pressures, for 5 h.

the initial silicon material was of *p*-type conductivity, it is concluded that the application of HP-HT treatments converts the conductivity of the top silicon layer from *p* type to *n* type. The estimated electron concentrations, from the *C-V* curves, in the top layer, are 3.3×10^{16}, 8.5×10^{16}, and 3.3×10^{17} cm^{-3} for 10^{-4}, 10^{-2}, and 1.2 GPa, respectively.

From the maximum values of the capacitance in the *C-V* curves we can extract the effective thickness of the buried oxide. The results are presented in Fig. 3(a). It is observed that an increase in the applied pressure leads to a decrease in the thickness of the buried oxide. We note that the XTEM images do not demonstrate the real thinning of the buried oxide.

Another parameter, which can be extracted from the *C-V* curves, is the charge in the insulator layer at both interfaces. We have used the flatband voltage[8] for the charge estimation. Charge values for the top Si/buried oxide interface Q_f, and for the buried oxide/substrate interface, Q_s, as a function of pressure, for the sample treated at 1130 °C, are shown in Fig. 3(b). We observe that for oxygen-implanted samples the interface charges slightly change with pressure. This charge does not only include the positive fixed charge in the buried oxide but also the negative charge at the interface traps. If we use V_{mg} for the charge estimation, the fixed charge in the buried oxide is extracted. It allows us to calculate the density of the interface traps, D_{it}, as a difference between charges extracted from *C-V* with the use of V_{mg} and V_{FB}. The D_{it} values [$\sim(6-8)\times10^{11}$ cm^{-2}] were found for

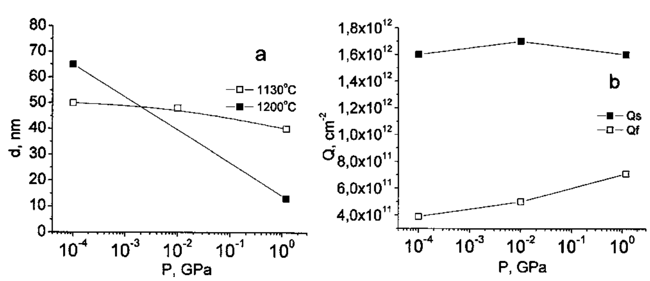

FIG. 3. Thickness of the buried oxide extracted from the maximum values of the capacitance in the *C-V* curves for anneals at 1130 and 1200 °C (a) and charge values for the top Si / buried oxide interface, Q_f, and for the buried oxide/substrate interface, Q_s, for anneals at 1130 °C (b), as a function of pressure for samples implanted with O^+ ions of 6×10^{17} cm^{-2} dose.

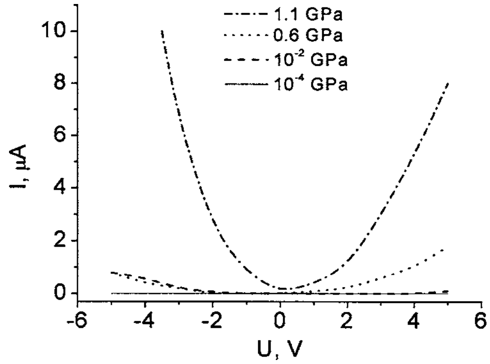

FIG. 4. *I-V* characteristics for silicon implanted with oxygen with a dose of 2×10^{18} cm^{-2} and treated at 1130 °C for 5 h at different pressures.

both interfaces of $Si/SiO_2/Si$ structures to be independent on the pressure applied during anneals of the structures. An increase in the oxygen dose leads to a weak increase in the fixed charge in the buried oxide and the interface trap density.

The *I-V* characteristics for the Si:O samples implanted with a higher dose of 2×10^{18} cm^{-2} and subjected to the same thermal treatments (1130 °C, 5 h) show an increase in the current through the buried oxide with pressure (Fig. 4).

B. The structures prepared by nitrogen implantation

The depth distribution of nitrogen and oxygen in the as-implanted and annealed samples measured by SIMS is presented in Fig. 5 (compare Ref. 11). The presence of oxygen in the as-implanted sample was caused by gettering of oxygen on radiation defects most likely due to the heating of the samples during implantation. The sheet concentrations of oxygen in the nitrogen-rich layer were 3×10^{15} and 5×10^{15} cm^{-2} for samples treated at 1130 °C for 5 h under atmospheric pressure and 1.4 GPa, correspondingly. According to Fig. 5 the utilization of HP leads to (a) a slightly narrow peak in distribution of nitrogen, (b) the disappearance of the additional peak near the surface and "tail" in nitrogen profile, and (c) an increase in oxygen concentration in the nitride layer.

C-V characteristics for nitrogen-implanted samples, for a dose of 8.5×10^{17} cm^{-2} annealed at $T=800-1130$ °C at different pressures, are given in Fig. 6. In the case of annealing at atmospheric pressure *p*-type conductivity is found in the top silicon layer [Fig. 6(a)], whereas in the case of samples

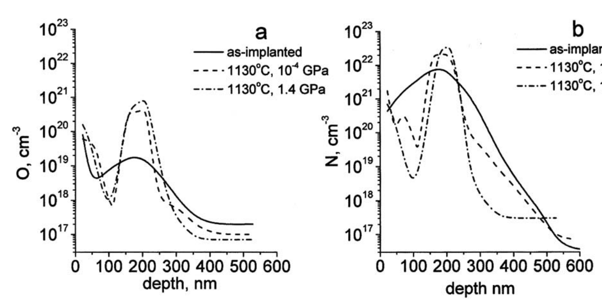

FIG. 5. Depth distribution of nitrogen and oxygen atoms measured by SIMS in the as-implanted and annealed samples (1130 °C, 5 h) for the case of implantation of N_2^+ ions with a dose of 1×10^{17} cm^{-2}.

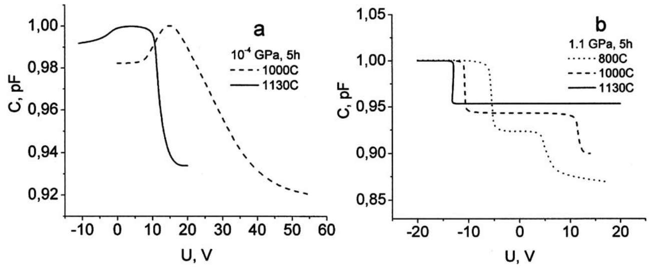

FIG. 6. C-V characteristic for samples implanted with N_2^+ ions with a dose of 8.5×10^{17} cm^{-2} and treated at different temperatures and pressures, for 5 h.

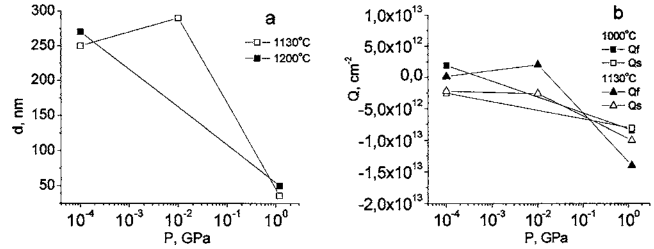

FIG. 8. Thickness of the buried oxide extracted from maximum values of the capacitance in the C-V curves (a) and charge values for the top Si/buried oxide interface, Q_f, and for the buried oxide / substrate interface, Q_s, (b) as a function of pressure for samples implanted with N_2^+ ions with a dose of 8.5×10^{17} cm^{-2} and treated at different temperatures.

treated at 1.1 GPa the top silicon layer has an n-type conductivity [Fig. 6(b)]. The concentrations of electrons extracted from the C-V curves for both cases are higher than 10^{18} cm^{-3} (Fig. 7). Formation of acceptor centers is observed in the top silicon layer of samples nitrogen implanted with the highest dose and also in the substrate of the samples implanted either with nitrogen or oxygen, with doses of $\sim 10^{18}$ cm^{-2} (Fig. 7).

The pressure dependence of the effective thickness of the nitride and the charge values at both interfaces extracted from C-V curves are presented in Fig. 8(a). The effective thickness of the nitride increases as the pressure increases, for low values of the pressure, until a maximum is reached, and then decreases. In the case of oxygen-implanted samples the effective thickness decreases gradually with pressure. At this stage a reasonable explanation for this difference in the observed behavior cannot be put forward. The comparison with SIMS data (Fig. 5), for a dose of 1×10^{17} cm^{-2}, demonstrates that the real thickness of the dielectric layer slightly decreases with pressure and is equal to 60–65 and 45–50 nm, for samples annealed at atmospheric and high pressure, correspondingly. The charge values at the Si_3N_4/substrate interface are negative and they strongly increase with pressure [Fig. 8(b)]. Only in the case of annealing of the nitrogen-implanted samples at low pressure (10^{-4}–10^{-2} GPa) the charge at the top Si/Si_3N_4 interface is positive. The interface trap density in these structures is varied in a wide range of $(3-20) \times 10^{11}$ cm^{-2} dependently on the temperature of the treatment and the applied pressure. Any prediction of what would happen if a higher pressure is used cannot be made at the present stage. A decrease in the nitrogen dose leads to a decrease in the fixed charge in the buried oxide and the interface trap density and to a lower pronounced effect of pressure.

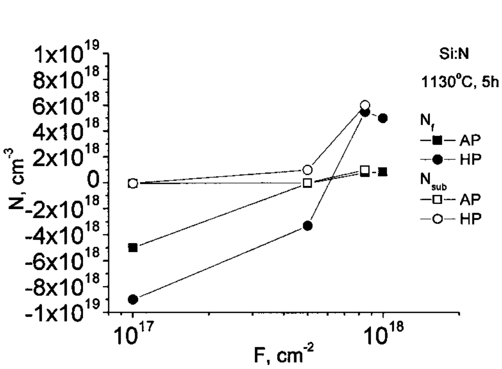

FIG. 7. Carrier concentration in the top silicon layer (N_t) and substrate (N_{sub}) vs nitrogen dose, for nitrogen-implanted samples after annealing at 1130 °C, for 5 h, at high and atmospheric pressures. The negative values correspond to n-type conductivity, whereas the positive values correspond to p-type conductivity.

IV. DISCUSSION

The removal of radiation defects from the top silicon layer is a common effect of the application of pressure on implanted Si:O and Si:N structures. As a result, dislocation loops are not observed in the top silicon layer and the substrate. The relation between the lattice parameters of silicon and the nitride differs from that for silicon and the oxide. The misfit at the silicon/insulator interface for the case of annealing under pressure can be estimated from the expression[12]

$$\varepsilon = \varepsilon_0 + \frac{K_d}{3K_d + 4G_{\mathrm{Si}}}\left[P\left(\frac{1}{K_{\mathrm{Si}}} - \frac{1}{K_d}\right) + \Delta T(\beta_d - \beta_{\mathrm{Si}})\right],$$

where K_{Si} and K_d are the elastic moduli of the silicon and the dielectric material, β_{Si} and β_d are the thermal expression coefficients, G is the shear modulus, P is the pressure, and ΔT is the change in temperature. $K_{\mathrm{Si}}=94.4$ GPa, $K_{\mathrm{SiO_2}}=40.7$ GPa, $K_{\mathrm{Si_3N_4}}=310$ GPa (we have taken values for bulk materials), $G=79.9$ GPa, $\beta_{\mathrm{Si}}=1.6\times 10^{-6}$ K^{-1}, $\beta_{\mathrm{SiO_2}}=1.3 \times 10^{-5}$ K^{-1}, and $\beta_{\mathrm{Si_3N_4}}=3.3\times 10^{-6}$ K^{-1}. The change in the misfit $(\varepsilon-\varepsilon_0)$ as a function of the applied pressure for temperatures of treatment in the range of 1000–1300 °C is given in Fig. 9.

As follows from Fig. 9, in the case of $Si/SiO_2/Si$ structures for $P > 0.3$ GPa and $T > 1000$ °C, an attractive elastic potential for interstitials due to the highly reduced SiO_2 volume is formed. As a result, the Si/SiO_2 interface serves as a very effective getter for interstitial defects and impurities. Gettering causes an increase in the strain at the Si/SiO_2 interface after HT-HP treatments. An analogous effect was ob-

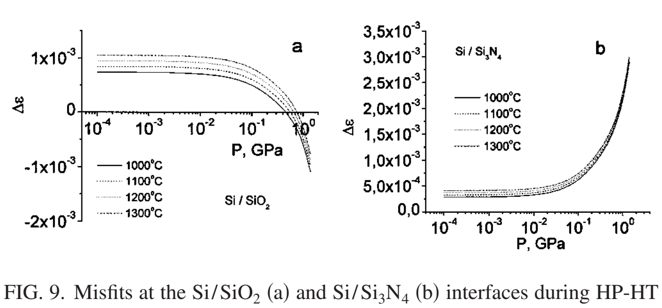

FIG. 9. Misfits at the Si/SiO_2 (a) and Si/Si_3N_4 (b) interfaces during HP-HT treatments as a function of pressure for different temperatures.

served for oxygen precipitates in HT-HP-treated silicon.[13] The perfect top silicon layer in the samples treated at ~1 GPa is the result of interstitial defect gettering at the Si/SiO_2 interface.

Another situation occurs in the case of $Si/Si_3N_4/Si$ structures. The increase in the misfit at the Si/Si_3N_4 interface with pressure leads to gettering of vacancy defects at the interface. The large pressure-induced gradient of mechanical stress in the top silicon layer during HT-HP treatments causes the removal of interstitial defects to the sample surface. The vacancy gettering at the interface together with the rejection of interstitials towards the surface contribute to the creation of a perfect top silicon layer. Actually, TEM images of Si:N sample treated under high pressure reveal a perfect top silicon layer.[5,14] A set of defects in the buried nitride seems different from that in the buried oxide. Consequently, interstitial defects are removed from the silicon layer towards the surface in the case of Si:N structures. We note that in the case of Si:O structures the interstitial defects move towards the interface with oxide due to the different signs of misfit at the Si/buried insulator interface.

Central to the above, we mention that previous studies[15] of pressure-induced effects in silicon demonstrate a pronounced increase in the vacancy concentration. Annihilation between pressure-induced vacancies and interstitials also leads to the removal of interstitial defects from the top silicon layer. As a result of this annihilation process and the pressure-induced gradient of mechanical stress, a perfect top silicon layer is formed both in the Si:O and Si:N samples.

Defect and impurity getterings at Si/SiO_2 interface under HP-HT treatment have been clearly demonstrated for the case of oxygen precipitates in silicon.[13] The same effect is suggested to occur in the case of Si:O structures. It is very interesting that in spite of this "dirty" Si/SiO_2 interface (due to the gettering) formed under high pressure, the interface charge is not practically changed [Fig. 3(b)] in Si:O structures. In the case of Si:N structures, another type of defects and impurities has to getter at the Si/Si_3N_4 interface. In the latter case, the interface charge strongly increases with pressure.

The average thickness of the buried oxide in the $Si/SiO_2/Si$ structures according to TEM results is equal to 140–160 nm and according to our data and Ref. 16 it does not practically change with pressure. The effective thickness of the oxide extracted from *C-V* characteristics is lower (about 50–65 nm for samples annealed at atmospheric pressure) than that extracted from TEM measurements and decreases with pressure. It means that, due to defect accumulation in the buried oxide, its effective thickness for electrical measurements becomes lower, in the case of HP treatments. According to TEM data, the buried oxide of structures created by HP-HT treatments contains numerous silicon inclusions and dislocation loops or half loops and other defects. Thus, the low value of the effective thickness extracted from *C-V* characteristics can be attributed to the formation of a thick transient layer between the top silicon layer and the buried oxide. This layer does not act as a dielectric layer due to the high defect density present.

In $Si/Si_3N_4/Si$ structures the effective thickness of the buried dielectric extracted from the maximum capacitance values at *C-V* characteristics is higher than that extracted from SIMS measurements, for low pressure and they are practically the same for high-pressure treatments. This experimental result is connected: (1) with a relatively high concentration of the oxygen in the buried dielectric, possibly responsible for the high value of the buried dielectric thickness, (really, oxynitride is formed and the decrease in the dielectric constant of oxynitride in comparison to that of the nitride is not taken into account) and (2) with the presence of a depletion area in silicon (top silicon layer or/and substrate), which does not disappear by the application of the voltage to the structure. As follows from Figs. 6 and 8(b) in the samples treated at both low and high pressures the substrate has a *p*-type conductivity and a negative charge in the buried dielectric and at the interface states. This situation leads to an accumulation regime at the Si_3N_4/substrate interface. In the samples annealed at atmospheric pressure the top silicon layer has a *p*-type conductivity and there is a positive charge at the Si_3N_4/Si interface. The depletion area is present at this interface after annealing at atmospheric pressure. In the case of treatment at high pressure there is an *n*-type conductivity in the top silicon layer and a negative charge in the buried dielectric. The depletion area is again formed at this interface. But the carrier concentration is higher in the samples treated at high pressure and the thickness of the depletion layer is smaller in comparison with that of the samples annealed at atmospheric pressure. The formation of a depletion layer near the surface is also possible. Thus, the high values of the buried insulator thickness, extracted from *C-V*, are most likely connected with a partial depletion of the top Si due to the charges in the buried insulator and/or the band bending at the surface.

The formation of donor centers was found after annealing of the oxygen-implanted samples at relatively low temperature (1130 °C). The electron concentration increases with the applied pressure during treatment. The values are 3.3×10^{16}, 8.5×10^{16}, and 3.3×10^{17} cm^{-3}, for treatments at atmospheric pressure (10^{-4} GPa) and enhanced pressures (10^{-2} and 1.2 GPa), respectively. The thickness of the top silicon layer for treatment at 1130 °C according to XTEM is ~180 nm. For the concentration value of 3×10^{16} cm^{-3} the maximum width of the depletion area is equal to 160 nm. For the concentration value of 3×10^{17} cm^{-3} the maximum width of the depletion area has to be ~70 nm. This allows us to state that the electron concentrations in the top Si layer given above are the real values in the silicon layer and are not restricted by the thickness of the top layer. Thus, the concentration of donors formed in the top silicon layer increases with the applied pressure during the postimplantation treatments.

The carrier concentrations extracted from *C-V* characteristics for the nitrogen-implanted samples treated at high pressures are very high (~10^{18} cm^{-3}). According to SIMS data the thickness of the top silicon layer for a nitrogen dose of 1×10^{17} cm^{-2} is about 150 nm. Thus, the thickness of the depletion layers is essentially lower than that of the top silicon layer.

Concerning the origin of the shallow donor centers formed in the top silicon layer the following could be stated: Introduction of donors was found in the cases of (1) oxygen implantation (dose of 6×10^{16} cm^{-2}) and thermal treatment at 1130 °C, at any pressure and (2) nitrogen implantation and thermal treatment only under high pressure. In the first case, the donor concentration increases with pressure. Notice that the formation of donors in implanted silicon has been observed in SIMOX structures.[17] The origin of these donors is unknown. The pressure-stimulated formation of donors was observed also after implantation with high-energy ions of inert gas[18] as well. In the latter case the donor centers were assumed to be oxygen-related new thermal donors and they were stable only up to 1050 °C. The formation of donor centers in the top silicon layer was also observed in SOI structures, formed by wafer bonding and hydrogen slicing.[18] The hydrogen stimulates (or participates in) the formation of donors.[19] The common element in the processes given above is the implantation. Most likely some defect complexes are the origin of donor centers. The presence of hydrogen, oxygen, or the utilization of high pressure seems to stimulate the formation of donors. It is necessary to emphasize that the interval of temperature stability of the defect complexes changes in the case of a thin silicon film due to the interfaces. The origin of the acceptor centers found after a high dose oxygen or nitrogen implantation is most likely connected with radiation-related defects[20] or process-induced acceptor impurities.[21]

V. SUMMARY

Two common pressure-induced effects are found for Si:O and Si:N SOI-like structures: (1) the removal of radiation defects from the top silicon layer and the formation of a perfect layer with simultaneous degradation of the buried insulator (increase in current through the buried insulator), and (2) a pressure-induced formation of the electrically active centers in the top silicon layer and the substrate. Different pressure effects are revealed for the fixed charge in the buried insulator. Our result indicated a practically constant value of the charge at the Si/SiO_2 interface and a strong increase with pressure of the negative charge at the Si/Si_3N_4 interface.

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