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Microstructure of silicon implanted with high dose oxygen ions

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Buried implanted oxide layers have been formed by high dose implantation of oxygen ions (3×10^{18} ions cm^{-2}) into ⟨100⟩ silicon wafers, at a constant temperature of 500 °C. The implanted layers were studied by cross-sectional transmission electron microscopy and secondary ion mass spectroscopy. The defects at both the top Si/SiO_2 and the SiO_2/bulk Si interfaces are shown to be SiO_2 precipitates. The precipitates are unstable and can be eliminated by heat treatment, and a homogeneous top silicon layer with a low density of dislocations can be obtained.

There is increasing interest in making silicon-on-insulator (SOI) structures by high dose oxygen implantation. Although these structures have been studied by several authors[1–6] and there is considerable knowledge about the structure of the buried oxide layer and the silicon layers on either side of it, the defect structures of the SiO_2 interfaces have not been analyzed to a degree that allows their complete understanding. Some authors attribute these defects to SiO_2 precipitates, since the oxygen concentration in these regions is above the solubility limit.[3,7,8]

The purpose of this work is to investigate the effects of high dose implantation at an elevated wafer temperature, to characterize the nature of defects created under those conditions, and to suggest means of eliminating them.

The buried structure was produced by implanting O^+ ions at 200 keV. During the implantation, the wafer temperature was kept constant at 500 °C by a specially designed heating system. Similarly Holland *et al.*[9] used a heated sample holder, and showed that the quality of SOI structures strongly depends on the substrate temperature at the beginning of the implantation. The implanted samples were examined by cross-sectional transmission electron microscopy (XTEM) and by secondary ion mass spectroscopy (SIMS) for complementary analysis. Although we obtained our optimum results with lower doses, we give here results from a sample implanted at 500 °C with a very high dose (3×10^{18} ions cm^{-2}), and consequently the most heavily damaged one, in order to show the important role that a proper post-implantation heat treatment plays.

As-implanted specimens display in XTEM a demarcated buried oxide layer, adjacent to which are highly defective Si monocrystalline regions, as seen in Fig. 1(a). The top layer of thickness 180 nm $\pm$ 10 is single crystalline of the same orientation as the substrate. Observations under higher magnification of this area reveal the existence of numerous defects [see inset of Fig. 1(a)] that may be attributed to ion implantation damage. These can be classified as small dislocation loops, large rodlike defects [probably interstitial Frank dipoles[10] located in the (111) planes] and characteristic speckles with a diameter of about 2 nm. The latter has been identified by Holland *et al.*[9] as SiO_2 precipitates in the as-implanted samples. The layer is heavily damaged over its entire width up to the surface. The buried SiO_2 layer is 650 nm thick. The silicon substrate is also heavily damaged to a depth of approximately 200 nm below the oxide.

After annealing at 1150 °C for 2 h in a nitrogen ambient, with a 600-nm-thick SiO_2 capping layer, the top Si layer is divided into two regions: the upper region, which is 60 nm thick, appears to have an improved single crystalline structure in which the only defects are threading dislocations at a density of about 10^{10} cm^{-2}, whereas the lower region adjacent to the SiO_2 layer contains many defects [Fig. 1(b)]. The defective region was previously attributed[7,11] to high oxygen concentration which is manifested in the SIMS findings. In particular, Fathy *et al.*[8] mentioned the existence of amorphous oxide inclusions, with a density decreasing towards the free surface of silicon, but information about the shape, the orientation, the dimension of these precipitates and the strain around them was lacking.

Concentrating on the defected region, our observations reveal that the shape of the faults is polyhedral and their diameter varies from 10 to 50 nm. The upper and lower Si/SiO_2 interfaces have the same characteristic defects (Fig. 2). Tilting experiments under different diffraction conditions reveal contrast features which, according to Van Landuyt *et al.*[12] correspond to cavities filled with noncrystalline material, with almost no strain in the silicon matrix around them, which means that the surface tension in the matrix is balanced by the interior of the cavities. In our samples we attribute these findings to voids filled with amorphous SiO_2 since the presence of gas in the cavities is very improbable after the high-temperature treatment. Such SiO_2 filled voids have also been observed in the Cu-Si system by Ashby and Brown.[13] Precipitates of amorphous SiO_2 have been previously identified in bulk silicon,[14–16] placing the surrounding matrix in a compressive state leading to the formation of extrinsic stacking faults.[17] On the contrary, in the implanted silicon, the lattice in the region surrounding the precipitates is almost free of strain. Nevertheless, recently, precipitates with polyhedral shapes and no strain have also been observed by Ponce *et al.*[15] in annealed bulk CZ silicon. Indeed a rough estimation of the expected misfit strain due to the difference in the Si-Si distance in amorphous silica and in crystalline silicon is given by Maher *et al.*[17] as being in the order of = 0.07. Taking this value into account we may estimate the expected image width due to the distortion of the lattice matrix.[18] Thus for particles 50 nm in diameter the expected image width would be 50% of the background in-

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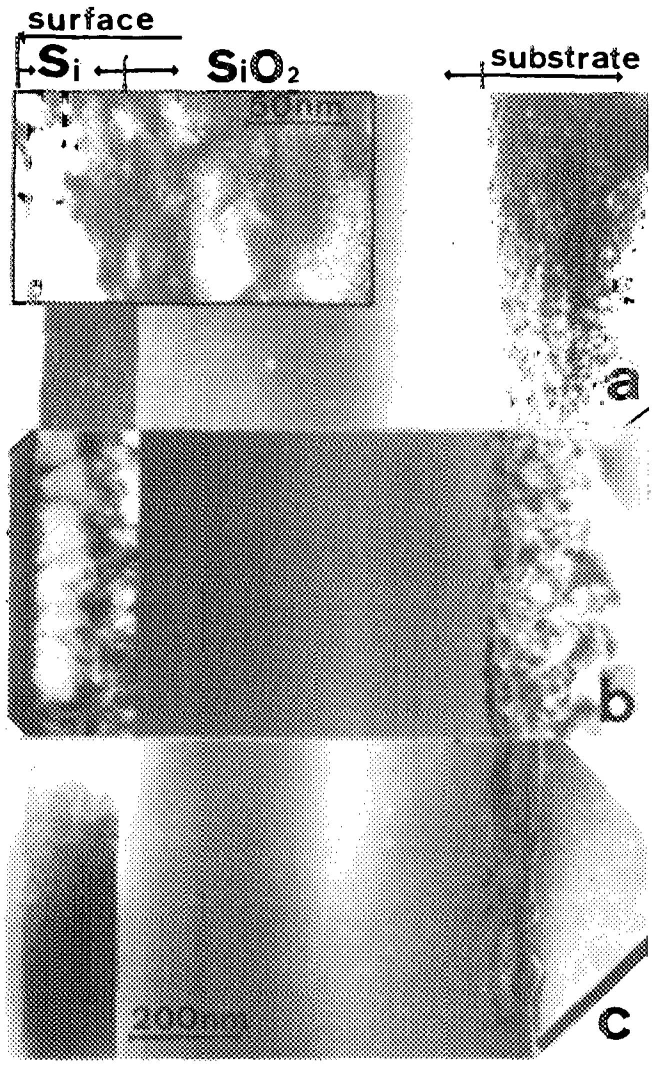

FIG. 1. Cross-sectional TEM micrograph near ⟨110⟩ orientation. (a) As implanted (200 keV, 3×10^{18} cm^{-2}, 500 °C). The inset is a high magnification image of the top silicon layer; characteristic speckles appear in this area. (b) Annealed for 2 h at 1150 °C. (c) After a second annealing for 6 h at 1210 °C.

tensity at a distance of 100 nm from the center of the particle, when the g_{111} reflection is operating. Densitometer measurements on our images show a contrast of 2% or less, implying a strain less than 0.01. This means that a very important migration of interstitial silicon atoms or vacancies has to take place in order to reduce the misfit.

The mechanism of formation of these precipitates is not known. It is very likely the same as that involved in bulk silicon,[15] but in implanted silicon there is formation of a larger number of precipitates because of the higher oxygen concentration, which far exceeds the limit of solubility, and this large number of small SiO_2 precipitates[9] act as nucleation centers for the formation of bigger precipitates. Due to this high density of precipitates, the area of the silicon/silicon oxide interface dramatically increases, resulting in an increase of the total surface free energy. Under these conditions these faults should be highly unstable.

To test the above hypothesis we performed a second annealing of our samples at 1210 °C for 6 h in a N_2 atmosphere without capping layers. The results are shown in Fig. 1(c). The highly defective layer adjacent to the buried silicon dioxide layer disappeared completely, with only threading dislocations remaining, but with a lower density. Only a few silicon islands remained trapped in the oxide, near the SiO_2/bulk interface. While annealing of such layers under nitrogen without capping leads to the formation of pits,[19,20] a detailed examination of our samples by scanning electron microscopy showed the absence of pits. This difference is very likely due to the two-step annealing (1150 °C with an SiO_2 capping plus 1210 °C without capping, both under nitrogen) that we used.

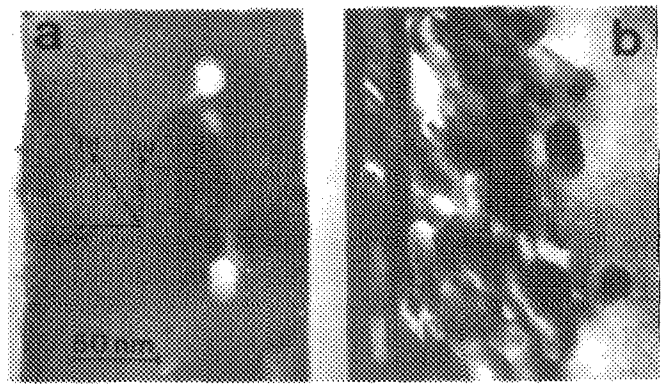

FIG. 2. High magnification micrograph of polyhedra defects as they appear after annealing for 2 h at 1150 °C. (a) In the top silicon area. (b) At the SiO_2/substrate interface.

SIMS profiles of the oxygen concentration are shown in Fig. 3. They were obtained with xenon as primary ions associated with an electron bombardment, and O^+ ions were used as secondary ions. In these conditions we observe an O^+ signal only when oxygen comes from SiO_2. This O^+ formation from SiO_2 has been explained by Juliet[21] as the result of Auger interatomic transitions which can occur only in SiO_2. A thin layer of SiO_2 developed on the surface of the sample during the 1210 °C annealing. The origin of the layer is under investigation. Annealing at 1150 °C for 2 h causes the appearence of a hump in the profile (Fig. 3). After annealing at 1210 °C for 6 h this hump disappears. The behavior of the hump corresponds very closely to that of the cavities seen in TEM, and this, together with the results from XTEM, leads us to conclude, in agreement with other authors,[3,8,9] that the cavities are filled with SiO_2. Similarly, by comparing

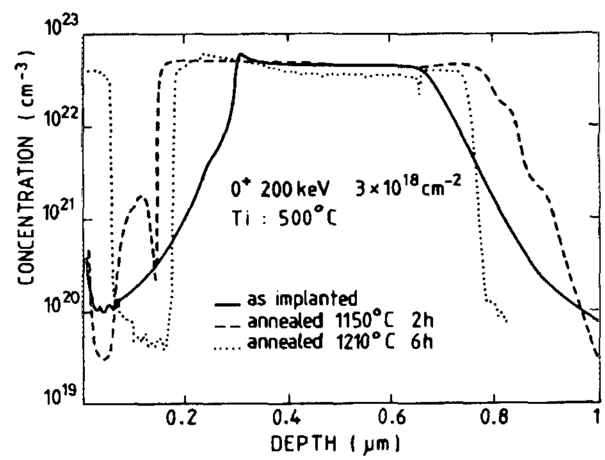

FIG. 3. SIMS profiles of oxygen.

samples implanted at different temperatures, Tuppen *et al.*[3] related the evolution of the oxygen profile measured by Auger electron spectroscopy to that of the microstructure observed by XTEM.

In conclusion, under our experimental conditions, the only defects in the top silicon layer are threading dislocations and SiO_2 precipitates. With an appropriate thermal annealing it is possible to reduce the concentration of these precipitates and even in some cases to eliminate them. This enables the thickness of the nearly defect-free silicon to be increased, which could enable the energy of the implanted ions to be reduced, reducing in turn the dose required to form a buried insulating layer.

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Direct observation of an enhanced concentration of the principal deep level EL2 at single dislocations

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Regions of low dislocation density ($\sim 5\times 10^2$ cm^{-2}) in 2-mm-thick {001} wafers from indium doped, liquid encapsulated Czochralski grown, 2-in-diam GaAs ingots have been examined by transmission infrared microscopy. Discrete dislocations were located and identified by their etching behavior in the calibrated A/B etchant. Comparison of identical regions before and after etching showed that increased absorption at 1 μm occurred at the sites of each dislocation. The results demonstrate directly that enhancement of [EL2], the concentration of the deep donor level EL2, occurs at single dislocations.

Correlation between variations in dislocation density and variations in [EL2], the concentration of the deep donor level EL2, in undoped, liquid encapsulated Czochralski (LEC), semi-insulating GaAs was originally reported by Martin *et al.*[1] Long range "W" or "U" shaped fluctuations across ⟨110⟩ diameters of {001} specimens were observed. Subsequently, Brozel *et al.*[2] demonstrated that fine scale ($\sim 100\,\mu$m) inhomogeneities in [EL2] were superimposed on the long range variations, and they could be correlated with fine scale inhomogeneities in dislocation distributions across {001} and {110} specimens. These correlations were established by a combination of quantitative near-infrared absorption measurements at 1 and 2 μm to determine [EL2], infrared microscopy to display the absorbing regions as a qualitative distribution throughout entire specimen volumes, and use of the Abrahams–Buiocchi[3,4] (A/B) etch to reveal dislocation distributions across entire specimen surfaces.[5,6] As Martin *et al.*[7] have indicated, it is difficult to find a point-to-point correspondence between both distributions because dislocations usually can only be studied in thin slices