Thermal annealing behavior of an oxide layer under silicon

A. H. Hamdi\textsuperscript{a} and F. D. McDaniel
\textit{Department of Physics,\textsuperscript{b} North Texas State University, Denton, Texas 76203}

\textit{Texas Instruments,\textsuperscript{c} Central Research Laboratories, Dallas, Texas 75265}

(Received 2 August 1982; accepted for publication 27 September 1982)

High resolution Rutherford backscattering spectrometry and ion channeling have been employed to evaluate the crystallinity of the surface silicon layer in oxygen implanted silicon. The quality of the top surface layer was determined by measuring the minimum yields along (110) directions in channeling spectra. Single crystal (100) silicon was implanted with 300 keV O\textsuperscript{2+} to a dose of $1.06 \times 10^{18}$ O\textsuperscript{2+} /cm$^2$. Measurements of residual damage of the top layer were made after annealing the samples at 1150 °C for times ranging from 10 to 240 min in either Ar or N\textsubscript{2}. Under the implantation conditions used in this experiment, a uniform oxide layer 0.52 μm thick was buried under a top silicon layer 0.17 μm thick. The buried oxide layer has abrupt silicon to oxide interfaces. The highest quality silicon surface layer was produced after 3-h annealing in an Ar ambient. A lesser quality silicon surface layer was produced by annealing for shorter times or for equivalent times in N\textsubscript{2} ambient.

PACS numbers: 61.70.Tm, 68.55. + b, 81.10.Jt, 85.40.Ci

Silicon-on-insulator (SOI) structures are currently being evaluated for use in very large scale integrated circuits (VLSI). The advantages of devices using SOI instead of bulk single crystal silicon are low parasitic capacitance, superior hardness to transient radiation, and low power dissipation. Buried oxide production by high dose oxygen implantation is a promising technique in SOI technology.\textsuperscript{1,2} Dielectric isolation by ion implantation provides abrupt Si/SiO\textsubscript{2} interfaces with charge densities near that of thermal oxide.\textsuperscript{3} Furthermore, when the maximum oxygen doses are chosen such that the Si/O atomic ratio exceeds the stoichiometric ratio of 1:2 at the distribution peak, the buried oxide layer is still SiO\textsubscript{2}. In buried oxide SOI post-implantation annealing homogenizes the buried oxide and recrystallizes the top surface layer.\textsuperscript{5} In this letter we report for the first time the quality and the recrystallization of the top surface layer as a function of annealing time at 1150 °C in Ar and N\textsubscript{2} ambient.

The substrates were p-type (100) Czochralski-grown silicon wafers, 76 mm in diameter with resistivities of 6–8 Ω cm. The wafers were cleaned using standard IC fabrication techniques. A 100-nm-thick layer of SiO\textsubscript{2} was thermally grown and removed by chemical etching before the wafers were loaded into the ion implanter. Molecular oxygen was used as the incident ion to reduce the implantation time. The beam energy was 300 keV and the beam current was 200–350 μA. The substrate temperature is estimated to be between 300 and 600 °C. To achieve a thicker top surface layer and to minimize the implantation damage in that layer, ion implantation was done along (100). The oxygen dose in all wafers was $1.06 \times 10^{18}$ O\textsuperscript{2+} /cm$^2$, equivalent to $2.1 \times 10^{18}$ O\textsuperscript{2+} /cm$^2$ with an energy of 150 keV. This dose was chosen so that the buried SiO\textsubscript{2} layer has abrupt Si/SiO\textsubscript{2} interfaces.\textsuperscript{2} After implantation, wafers were divided into 1×1 cm samples. The samples were annealed in flowing gas at 1150 °C. One set was annealed in Ar and the other in N\textsubscript{2} for 10, 20, 30, 60, 120, 180, and 240 min. Rutherford backscattering spectrometry (RBS) and ion channeling were performed using a 2-Mev He\textsuperscript{+} beam. The analyzing beam current was typically 30 nA with a beam divergence < 0.005°. An ORTEC 7050/1150 data acquisition and analysis computer was used with an energy calibration of 2.28 keV/channel. A surface barrier detector with 13-keV energy resolution [full width at half-maximum

\textsuperscript{a}Robert A. Welch Foundation Fellow.
\textsuperscript{b}Supported in part by Robert A. Welch Foundation and NTSU Organized Research Committee.
\textsuperscript{c}The HVEM was performed at the Argonne National Laboratory supported by the Department of Energy. This work was supported in part by RADC Contract F19628-81-C-0136, P. Vail, contract monitor.

FIG. 1. Cross-sectional weak beam HVEM photomicrograph of the buried oxide layer in an as-implanted sample illustrating the heterogeneous nature of the unannealed top silicon layer.

(FWHM) detected scattered ions at an angle of 154° with respect to the incident ion direction. Channeling along the most open axial direction, (110), was found by tilting the sample 45° from the incoming beam direction. Angular scan across (110) were made for each sample by increasing the tilted angle in 0.1° steps from 42.5° to 47.5°. Similar scans were done by rotating the sample about an axis parallel to the surface normal. Spectra were taken at several spot locations to avoid damage caused by the analyzing beam. Random spectra off the channeling direction were taken during a continuous rotation of the sample about the surface normal.

Cross sectional, weak-beam HREM microscopy (Fig. 1) of the as-implanted sample shows that the top silicon layer has a high defect density. The buried oxide region is almost stoichiometric SiO$_2$. A thin surface oxide layer approximately 350 Å thick was observed in all the annealed samples. Samples analyzed with RBS were etched with a 10% HF solution for 70 s to remove the surface oxide.

Figure 2 shows typical RBS random and (110) aligned spectra for 2-MeV He$^+$ ions scattered from Si implanted with $2.1 \times 10^{16}$ O$^+$ cm$^{-2}$ at 150 keV (equivalent energy) and annealed for 3 h at 1150°C in an Ar ambient. Thickness calculations based on tabulated stopping cross sections showed that the top surface layer is 0.17 μm thick and the buried oxide layer is 0.52 μm thick. These values agree with LSS theory to within 10% and with high resolution scanning electron microscopy measurements to within 5%. The peak between 1.1 and 1.2 MeV corresponds to the top surface silicon layer. The dip in the yield between 0.8 and 1.1 MeV represents silicon in the buried oxide layer. The lower yield in this region is due to the lower atomic density of silicon in SiO$_2$. The channeling spectrum shows that the minimum yield in the top surface layer is 12.3% compared to 2.2% in unimplanted single crystal silicon.

Figure 3 shows (110) aligned spectra for as-implanted and annealed samples for various times at 1150°C in an argon ambient. One can see the improvement in crystallinity of the top Si layer and the sharpening of the Si/SiO$_2$ interface. This could be seen by monitoring the decrease in the back-scattered yield. As the annealing time is increased, the yield at 1.18 MeV decreases and the minimum occurs at lower energy. The decrease in half-width at half-maximum of the peak at 1.15 MeV indicates the sharpening in the Si/SiO$_2$ interface.

Figure 4 shows the minimum yield $\chi_{\min}$ as a function of annealing time for channeling spectra of samples annealed in Ar and N$_2$. The minimum yield is a qualitative measurement of the crystallinity. Annealing in an Ar ambient is consistently better than annealing in a N$_2$ ambient for equivalent times. The minimum yield for samples annealed in Ar decreased from ~33% to 12.3% in 180 min and remains about the same for longer anneal times. The Si/SiO$_2$ interface also...
improves with increasing anneal time, as shown in Fig. 3. The apparent improvement of the top surface layer by annealing the samples in Ar over those annealed in N₂ is not fully understood at this time, but may be related to differences in the point defect concentrations in the top surface layers caused by the ambient. A similar ambient effect has been reported for oxygen precipitation in silicon. The top silicon surface layer of the sample annealed for 240 min in a nitrogen ambient cracked during annealing and could not be analyzed.

After annealing, the surface layer was found to be capable of supporting epitaxial growth with a minimum yield as good as that found in virgin single crystal silicon. This fact is remarkable because (1) the top surface layer shows no channeling in as-implanted samples, (2) analyses of channeling show that the top Si near the buried oxide layer contains substoichiometric oxide, as indicated in Fig. 2 by the reduction in the Si signal in the random spectrum at 1.14 MeV, and (3) the recrystallization proceeds from the surface into the top layer.

In summary, we have reported for the first time the quality of the top surface Si layer for buried oxide as a function of anneal time at 1150 °C in both nitrogen and argon ambients. In general, we have found that longer anneal times produce improved crystallinity of the top surface Si layer and sharpen the interface between the top Si layer and the buried oxide. Annealing in an Ar ambient is slightly better than annealing in a N₂ ambient. In both N₂ and Ar ambients, the top Si layer recrystallizes from the front surface toward the buried oxide.