Low-dislocation-density silicon-on-insulator material produced by sequential oxygen implantation and low-temperature annealing

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(Received 27 December 1991; accepted for publication 3 April 1992)

Lattice strain and defect formation in oxygen-implanted silicon (SIMOX) were investigated by high-resolution x-ray diffraction and transmission electron microscopy. At doses of 1×10^{17} and 3×10^{17} cm^{-2} a high density of vacancy-type defects formed in a uniaxially compressed layer at the surface of the as-implanted wafer. Annealing at 900 °C for 0.5 h reduced this negative strain as the defects coarsened into observable cavities. The development of cavities upon annealing was used in a sequential-implantation and low-temperature-annealing process to produce low-threading dislocation density SIMOX. This new process offers several advantages over other methods of producing low-dislocation-density material.

High-dose oxygen-ion implantation (SIMOX) has proven to be a successful means of producing silicon-on-insulator materials. A continuing problem, however, has been the high density ($\sim10^9$ cm^{-2}) of threading dislocations (TDs) that form above a critical dose of 5×10^{17} cm^{-2}.[1] Low-TD densities have been reported for multiple low-dose implants with sequential high-temperature anneals,[2–5] when the implantation conditions were carefully controlled to nucleate cavities in the near-surface region during the implant[6–9] and when a superlattice-like array of oxide precipitates was formed with constant and very low-current-density channeled implants.[10,11]

In this letter, we present high-resolution x-ray diffraction (HRXRD) measurements and cross-sectional (XTEM) and plan-view (PTEM) transmission-electron-microscopy observations that have led to a new method of producing low-dislocation-density SIMOX. This new process utilizes some features of the above methods, while avoiding some of their disadvantages.

160 keV $^{16}O^+$ was implanted into (100) silicon wafers at doses from 1×10^{16} to 9×10^{17} cm^{-2} with a 10 μa/cm^2 beam-current density at 500 °C. An oxide cap was deposited on samples before furnace annealing in flowing N_2 for 0.5–6 h at 900–1300 °C. TEM observations were performed on a JEOL 200CX operating at 200 keV. HRXRD rocking curves (RCs) were obtained from a Phillip's 5-crystal diffractometer. In order to correctly interpret these RCs, dynamical x-ray simulations using a series of trial strain distributions were performed with the RADS computer program.[12]

RCs of the 1×10^{16} and 3×10^{16} cm^{-2} samples indicated the presence of a buried layer in unidirectional tension perpendicular to the surface. We have interpreted this lattice expansion in terms of an excess of Si and O interstitials near the projected range of the ions.[13]

As the dose was increased to 1×10^{17} and 3×10^{17} cm^{-2}, a surface layer in unidirectional compression perpendicular to the surface became evident. The (004) RC taken from a 3×10^{17} cm^{-2} as-implanted sample, together with a calculated RC and corresponding strain distribution is shown in Fig. 1. An extensive series of such RC simulations confirmed that the essential features of the strain distribution are correct, even though a detailed match was not sought. In particular, simulations of buried layers showed many additional features not evident in the experimental RC. Thus, a surface layer has undergone an average perpendicular lattice contraction of -2680 ppm extending to a total depth of about 2100 Å, while the maximum strain extends to a depth of about 1350 Å.

Additional RCs using the asymmetric (044) reflection at glancing incidence and exit angles indicated that the average strain parallel to the surface was only 8 ppm. Thus, the strain was accommodated by a tetragonal distortion of the lattice with virtually no relaxation, in agreement with other x-ray measurements on ion-implanted silicon.[14,15]

After annealing the 3×10^{17} cm^{-2} sample at 900 °C for 0.5 h, the average perpendicular lattice contraction decreased to -525 ppm while the average parallel strain remained very low at only 11 ppm. Further annealing at 1150 °C and above completely removed all measurable x-ray strain from the sample.

TEM observations of these samples shed considerable light on the origin of this surface lattice contraction. Figure 2 shows XTEM micrographs of both the as-implanted and 900 °C, 0.5 h annealed 3×10^{17} cm^{-2} samples. In the as-implanted state, the top silicon layer is largely defect free except for large faults at the buried oxide interface. Upon annealing, a high density of 25–35-Å-diam cavities or voids develops in the top 1120–1370 Å of the wafer, which corresponds to the depth of the strain-layer maximum. Samples annealed at 1150 °C showed no defects of any kind in the top 1200 Å, while below this depth, oxide precipitates formed.

These observations strongly suggest that the surface lattice contraction in the as-implanted condition is due to vacancy clusters, which would not be visible in conventional TEM images. These defects then coarsen into ob-

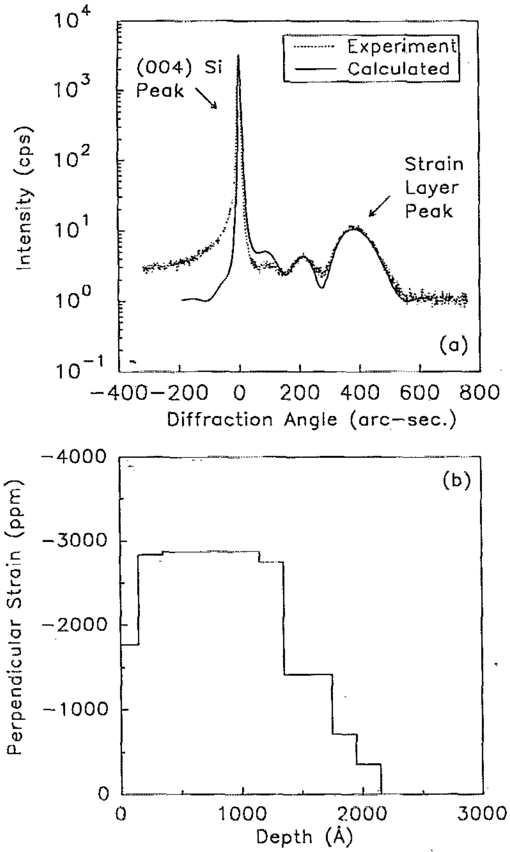

FIG. 1. HRXRD data for the 3×10^{17} cm^{-2} as-implanted sample showing (a) experimental and calculated RCs about the (004) reflection and (b) the trial strain distribution used for the calculation in (a).

servable cavities upon annealing, with a corresponding decrease in x-ray strain. It should be noted that carbon, a common impurity in very-high-dose SIMOX, could also give rise to a surface lattice contraction provided it occupied substitutional sites. However, we estimate that this would require almost 0.5 at. % C. Even if such a large quantity of C could be substitutionally dissolved in the as-implanted condition, substantial SiC precipitation would have to occur to account for the strain decrease upon annealing. Since we do not observe such SiC precipitation, but rather, the appearance of cavities upon 900 °C annealing, we conclude that vacancy clusters are more likely responsible for the surface lattice contraction.

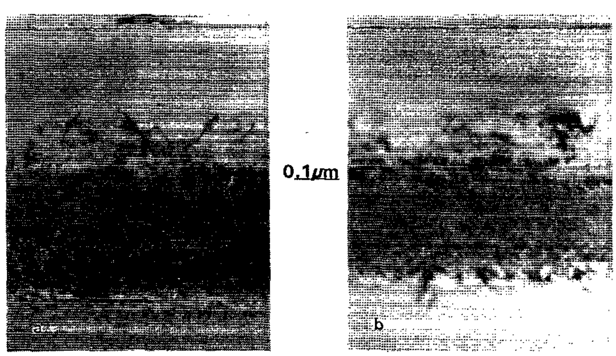

FIG. 2. XTEM micrographs of the 3×10^{17} cm^{-2} sample (a) as-implanted and (b) after annealing at 900 °C, 0.5 h.

As noted earlier, cavities have been observed in as-implanted SIMOX[6–9,16] but, to our knowledge, this is the first report of their formation after annealing. This is probably due to the emphasis in most SIMOX annealing studies on temperatures >1100 °C, where our observations indicate that the vacancy clusters become unstable instead of coarsening into cavities. This instability may be related to a lack of stabilizing oxygen gas and the presence of vacancy sinks due to extensive SiO_2 precipitation at >1100 °C.

The presence of cavities in as-implanted SIMOX has been correlated with low-TD densities.[6–9] Unfortunately, *in situ* cavity nucleation and stabilization apparently requires high implant temperatures (>600 °C) which can lead to poor buried oxide layers[17] and substantial surface roughness.[18] In addition, the *in situ* cavity-nucleation process is also quite sensitive to the implantation conditions. El-Ghor *et al.*[6] noted that, in analogy, to void nucleation in irradiated metals, only a narrow range of implant temperatures for a given scan rate, beam current, etc., will nucleate cavities. Our observations indicate that even outside of this narrow window for *in situ* cavity formation (i.e., at lower implant temperature), a high density of vacancy-type defects can be attained in the as-implanted condition. Moreover, low-temperature annealing (<1000 °C) will then cause these defects to coarsen into cavities, as shown.

These observations suggest a new means of producing low-dislocation-density SIMOX, which offers the advantage of being less sensitive to the implantation conditions and can be performed at lower implant temperatures. One has only to ensure that the implantation conditions are within the broader range for vacancy (as opposed to cavity) stabilization, implant to 3×10^{17} cm^{-2}, anneal for only 900 °C, 0.5 h to nucleate cavities, and repeat the cycle until the desired dose has been obtained. The brief low-temperature anneals in this process would also be a substantial improvement over the many high-temperature anneals used in the usual multiple-implant/anneal method[2–5] because of reduced contamination and faster processing. As in every SIMOX processing scheme, a final high-temperature anneal would be required to form a useful buried oxide layer.

In order to test this hypothesis, we performed two such cycles to a total dose of 6×10^{17} cm^{-2} with a 900 °C, 0.5 h anneal between implants. A final anneal of 1300 °C, 6 h was used to form a continuous buried SiO_2 layer. Figure 3 shows representative XTEM and PTEM micrographs of the multiple-implant sample and, for comparison, a sample with a single 6×10^{17} cm^{-2} dose followed by the high-temperature anneal. Some planar defects were evident at the buried oxide interface in the multiple-implant sample. However, no TDs were observed in the multiple-implant PTEM samples, indicating that the threading dislocation density was less than the PTEM detection limit of about 10^5 cm^{-2}. This is a substantial reduction over the single-dose sample where the TD density was 10^8 cm^{-2}. Clearly,

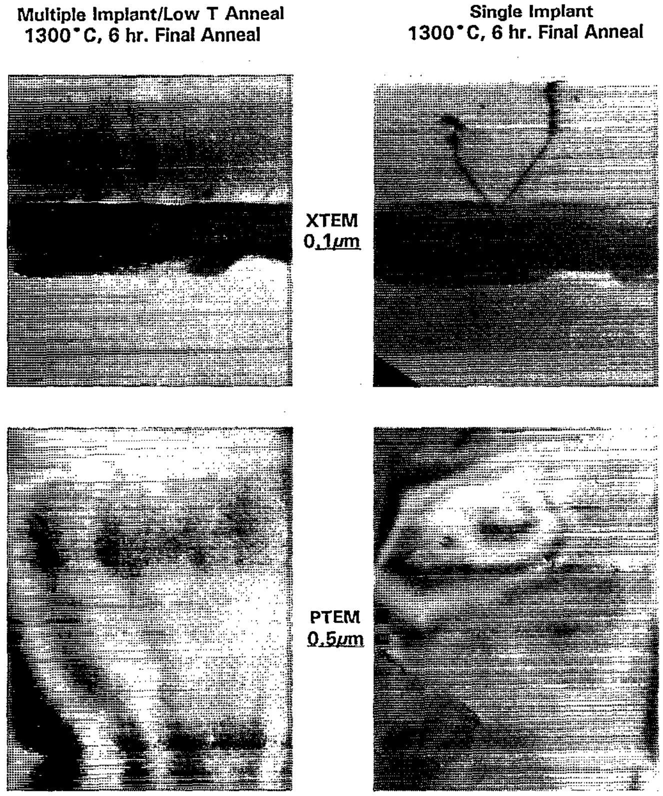

FIG. 3. XTEM and PTEM micrographs of a multiple-implant/low-temperature-anneal sample and a single-dose implant to the same total dose of 6×10^{17} cm^{-2}.

the new method was successful in producing low-dislocation-density SIMOX. We note that this method might be further improved be performing the cavity-forming anneals in the implanter as part of a multiple-implant/low-temperature *in situ* annealing process.

Despite the success of this and other methods in avoiding high-TD densities, no definitive explanation for the efficacy of these methods has emerged. The central problem lies in the fact that the mechanism by which TDs form is still uncertain. Our working hypothesis has been that TDs could arise from either implantation-induced point defects (type I damage)[19] or from precipitation-related defects (type V damage).[19] Multiple implants with high-temperature anneals remove both types of defects from the surface silicon layer. Our results indicate that it is not necessary to remove all defects if, instead, one can cause the dominant point defect in the surface silicon layer (i.e., vacancies) to aggregate as cavities rather than, perhaps, as extended defects. Cavities can also reduce precipitation-related defects by acting as a strain-free means of precipitating oxygen.[6] Thus, cavities also effectively prevent both TD formation mechanisms from operating.

In summary, we have shown that oxygen implantation of silicon can lead to a high density of vacancy-type defects in a layer at the surface of the as-implanted sample. These defects are not visible in conventional TEM images but are detectable by HRXRD due to the negative lattice strain they produce. Annealing at 900 °C, 0.5 h significantly reduces this negative strain as the defects coarsen into observable cavities. This microstructural development can be used in a sequential-implantation and low-temperature annealing process to produce low-threading dislocation-density SIMOX. The new process substantially reduces the annealing time and temperature necessary for the multiple-implant/anneal method, while it offers a relaxation of the potentially stringent conditions necessary for *in situ* cavity formation as a means of dislocation-density control.

We would like to thank Janette Manke, formerly of SPIRE Corp. for experimental assistance. One author (D. V.) would also like to thank V. Krishnamoorthy of the University of Florida for many valuable and stimulating discussions. This work was partially funded by the NSF-PYI program.

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