

## Reduction of Threading Dislocations and Oxide Precipitates in SIMOX Material

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Investigation was done to reduce defects in SIMOX (Separation of silicon by IMplantation of OXYgen) material. Reduction of threading dislocations was demonstrated by utilizing misfit dislocations formed at the interface of  $\text{Si}_{1-x}\text{Ge}_x/\text{Si}$  heterostructure grown onto SIMOX substrate. Threading dislocations were reduced to  $4 \times 10^7 \text{ cm}^{-2}$  from  $1 \times 10^9 \text{ cm}^{-2}$  by this technique. To reduce oxide precipitates, two-step anneal was adopted: hydrogen ambient anneal at  $850^\circ\text{C}$  followed by standard inert ambient anneal at  $1250^\circ\text{C}$ . The oxygen bump seen in an oxygen depth profile of superficial silicon layer was almost eliminated. Linewidth of Raman scattering spectra was also improved from  $4.45 \text{ cm}^{-1}$  by conventional anneal to  $4.19 \text{ cm}^{-1}$  by this technique.

### 1. Introduction

SIMOX (Separation of silicon by IMplantation of OXYgen) is one of the most promising techniques for preparing SOI (silicon-on-insulator) substrates because of its uniform thickness over an entire wafer and its process compatibility to the existing Si process technologies. Successful fabrication of high speed CMOS<sup>1)</sup> and radiation-hardened<sup>2)</sup> devices from SIMOX wafers have been reported. In spite of much effort in the past several years, however, SIMOX wafers still contain many defects such as threading dislocations and oxide precipitates. A typical dislocation density is in the range of  $10^9 \text{ cm}^{-2}$ , which limits its application for bipolar devices.

In this experiment, investigation was done to reduce those defects by several methods: (i) multiple implant/anneal cycle,<sup>3)</sup> (ii) gettering of threading dislocations by  $\text{Si}_{1-x}\text{Ge}_x/\text{Si}$  heterostructure, and (iii) two-step anneal technique to reduce oxide precipitates.

### 2. Experimental

Substrates used in this study were all (100)-oriented silicon wafers 100 mm in diameter. In the multiple implant/anneal cycle experiment, sequential implantation with an oxygen dose of

$5 \times 10^{17} \text{ cm}^{-2}$  and thermal anneal at  $1285^\circ\text{C}$  for 2 h were repeated three times. Implantation energy and temperature were 200 keV and  $600^\circ\text{C}$ , respectively. For experiments of dislocation gettering by  $\text{Si}_{1-x}\text{Ge}_x/\text{Si}$  heteroepitaxy and oxygen precipitates reduction by two-step anneal, a single implant SIMOX was used. Implantation dose, energy, and temperature were  $2 \times 10^{18} \text{ cm}^{-2}$ , 180 keV, and  $550^\circ\text{C}$ , respectively.

A layer of  $\text{Si}_{1-x}\text{Ge}_x$  and then a layer of pure Si were grown onto SIMOX wafers including Ge-doped SIMOX (see section 3.2), which had been annealed under standard conditions;  $1250^\circ\text{C}$ , 2 h in Ar/2%  $\text{O}_2$  ambient. The epitaxial growth was done by an Applied Materials (AMC-7810) reactor, at  $1000^\circ\text{C}$ , 55 Torr. The Si layer was deposited from  $\text{SiH}_2\text{Cl}_2$  after  $\text{Si}_{1-x}\text{Ge}_x$  was deposited from the mixture of  $\text{SiH}_2\text{Cl}_2$  and  $\text{GeH}_4$ . Dislocation density was measured by counting Secco-etch pits observed from a Scanning Electron Microscope (SEM). Dislocation densities reported in this study were only ones which terminated on the top of a  $3.1 \mu\text{m}$  epitaxial layer deposited either directly on SIMOX substrate or on  $\text{Si}_{1-x}\text{Ge}_x/\text{SIMOX}$  substrate.

Two-step anneal was adopted for other single implant SIMOX wafers. A low temperature anneal at  $850^\circ\text{C}$  for 16 h in a hydrogen ambient was done prior

to the standard anneal at 1250°C.

The Raman spectrometer used in this study was a model NR-1100 from Japan Spectroscopic Co., Ltd. operating with the excitation wavelength of 488 nm.

### 3. Results and Discussion

#### 3.1 Multiple implant/anneal cycle

Multiple implant/anneal cycle technique<sup>3)</sup> is very effective in reducing dislocation density. We have found very few threading dislocations when a three-step implant/anneal cycle was applied using the implantation energy of 200 keV and a total dose of  $1.5 \times 10^{18} \text{ cm}^{-2}$ . Figure 1 shows a cross-section TEM image of SIMOX material prepared in this way. It can be seen that many huge oxide precipitates existed in this material ranging in size from 50 to 100 nm in diameter located above the interface between superficial silicon and buried oxide layers. In contrast, however, the dislocation density was  $1.3 \times 10^8 \text{ cm}^{-2}$  using 180keV of energy and  $1.8 \times 10^{18} \text{ cm}^{-2}$  of dose. In this case, fewer oxide precipitates were observed.

#### 3.2 Gettering of threading dislocations by $\text{Si}_{1-x}\text{Ge}_x/\text{Si}$ heterostructure

Kamins et al.<sup>4)</sup> have studied the gettering of threading dislocations in epitaxial SIMOX wafers by using a  $\text{Si}/\text{Si}_{1-x}\text{Ge}_x$  superlattice heterostructure. The strain fields associated with the heterojunction of the superlattice were used as a gettering agent. They found little reduction of dislocation density with this method. We have studied an alternative technique using the misfit dislocations associated with the heterostructure instead of a strain field as a gettering agent. The planar misfit dislocations can intercept the vertical threading dislocations and force them to bend over along with misfit dislocations and terminate at the wafer edge. The misfit dislocations can be generated when the  $\text{Si}_{1-x}\text{Ge}_x$  layer thickness is greater than its critical value.<sup>5)</sup> We have found that misfit dislocations were formed with a 0.4  $\mu\text{m}$  thick  $\text{Si}_{1-x}\text{Ge}_x$  with an x value greater than 0.02. The density of misfit dislocations increases with the value of x. When the x value of 0.06 and 0.09 are used, the density

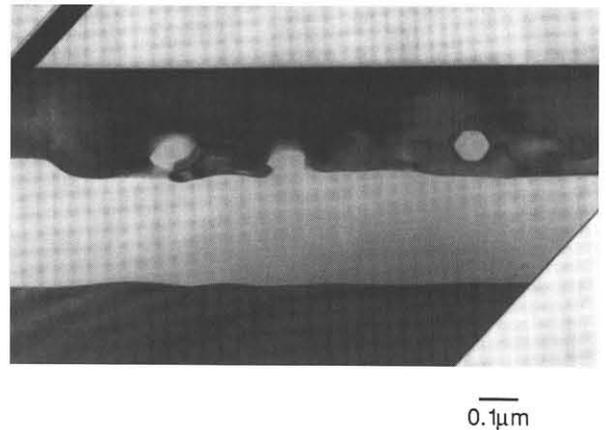


Fig. 1 A cross-section TEM image of SIMOX obtained by multiple implant/anneal cycle.

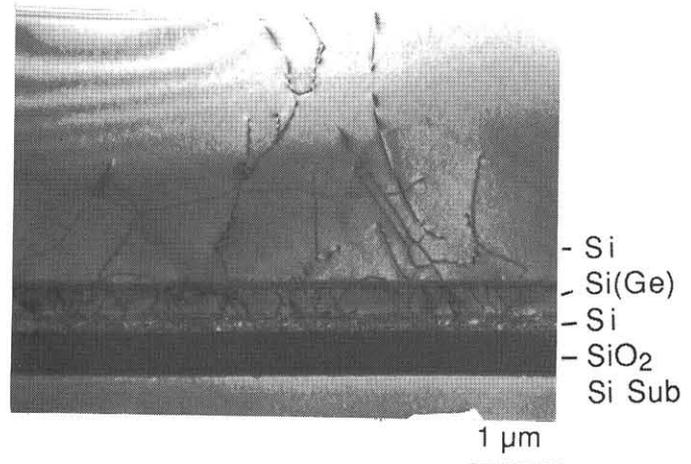


Fig. 2 A cross-section TEM image showing some gettering of threading dislocations by  $\text{Si}_{1-x}\text{Ge}_x/\text{Si}$  heterostructure on SIMOX ( $x = 0.06$ ).

can reach to  $10^8$  and  $10^9 \text{ cm}^{-2}$ , respectively. We have found that these high misfit dislocation densities are needed in order to reduce threading dislocations significantly.

We have deposited 0.4  $\mu\text{m}$  thick  $\text{Si}_{1-x}\text{Ge}_x$  layer on SIMOX followed by deposition of a 3.1  $\mu\text{m}$  of pure silicon. Figure 2 shows a cross-section TEM image for the sample with  $x = 0.06$ . It clearly shows that a considerable amount of threading dislocations terminate and bend over at the  $\text{Si}/\text{Si}_{1-x}\text{Ge}_x$  interface. The dislocation density counts were made for three values of x. It was found that the threading

dislocation densities are reduced to  $1.7 \times 10^8$ ,  $1.4 \times 10^8$ , and  $1.3 \times 10^8 \text{ cm}^{-2}$  from approximately  $10^9 \text{ cm}^{-2}$  for  $x$  values of 0.03, 0.06 and 0.09, respectively.

We have also used the double gettering method by applying the heterostructure twice. In this case a  $0.4 \text{ }\mu\text{m}$  thick of  $\text{Si}_{1-x}\text{Ge}_x$  and  $0.1 \text{ }\mu\text{m}$  thick of pure silicon were sequentially deposited on a bulk silicon substrate. Oxygen was implanted into such substrate and the "Ge-doped" SIMOX was formed after thermal anneal. A  $0.4 \text{ }\mu\text{m}$  layer of the  $\text{Si}_{1-x}\text{Ge}_x$  layer was again deposited followed by deposition of a  $3.1 \text{ }\mu\text{m}$  of pure silicon on the "Ge-doped" SIMOX. The double gettering has reduced the dislocation density to  $4.1 \times 10^7$ ,  $5.3 \times 10^7$  and  $4.5 \times 10^7 \text{ cm}^{-2}$  for  $x$  value of 0.06, 0.09, and 0.16, respectively.

We have shown reductions of threading dislocation densities up to  $10^8 \text{ cm}^{-2}$  by single gettering, and to  $10^9 \text{ cm}^{-2}$  by double gettering. It seems that an  $x$  value at 0.06 is an optimum. The lower the  $x$  number, the fewer misfit dislocations were created and greater portions of threading dislocations were able to propagate through the heterostructure. The higher the  $x$  number, the greater the ability of  $\text{Si}_{1-x}\text{Ge}_x/\text{Si}$  interface to intercept dislocations. However, new threading dislocations can be generated from the heterointerface when a high  $x$  value is used, and thus the final threading dislocations were never reduced to

less than  $10^7 \text{ cm}^{-2}$ .

A disadvantage of this gettering technique for the SIMOX wafers is that it results in a relatively thick SOI structure which is not ideal for a high speed devices. However, it may potentially be adopted for use in power devices. An advantage of this method is that the built-in misfit dislocations underneath the epitaxial layer can be used for gettering of metallic impurities as they have been shown by Kikuchi et al.<sup>6)</sup>

### 3.3 Precipitate elimination by two-step anneal

Another major defect that should also be addressed is oxide precipitates in the superficial silicon. Figure 3 (A) indicates the depth profile of oxygen and silicon concentration measured by sputtering Auger Electron Spectroscopy (AES) in a single implant SIMOX wafer annealed with the standard conditions ( $1250^\circ\text{C}$ , 2 h in Ar/2%  $\text{O}_2$  ambient). There is a bump and a dip in the oxygen and silicon concentration profiles, respectively, which is indicative of oxide precipitates. On the contrary, the bump and a dip reduce in Fig. 3 (B), which shows the depth profile in a sample with two-step anneal: a 16 h  $850^\circ\text{C}$  anneal in hydrogen ambient plus the standard Ar/2%  $\text{O}_2$  anneal. This reduction was not observed when we tried the two-step anneals both in Ar/2%  $\text{O}_2$  ambient.

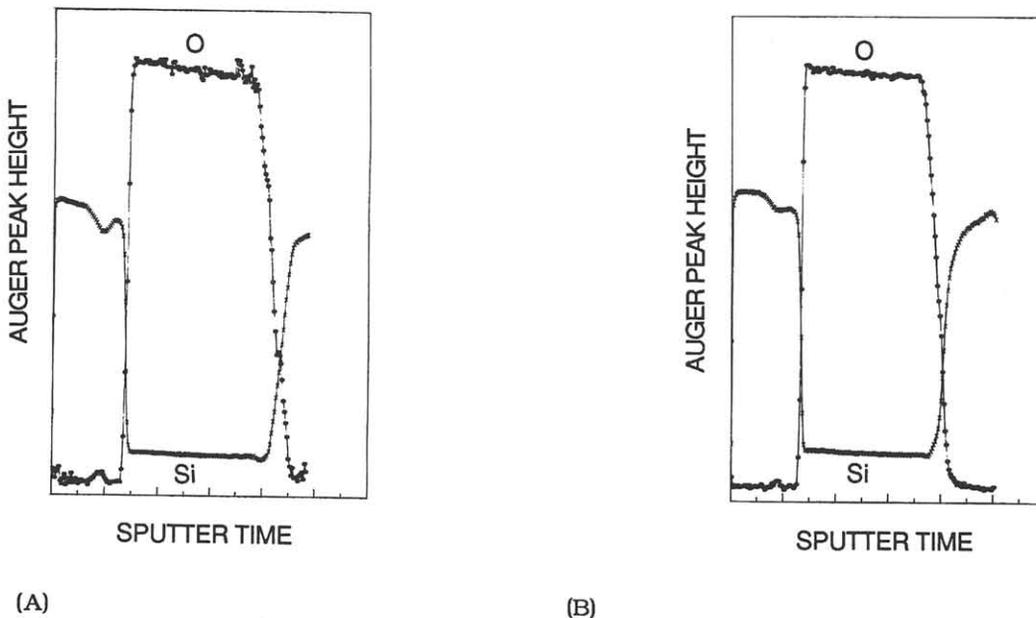


Fig. 3 Oxygen and silicon AES depth profile of SIMOX samples with (A) standard anneal and (B) low temperature hydrogen ambient plus standard anneal.

Table I. Summary of Lorentzian curve fitting for Raman scattering spectra of two step annealed SIMOX. Signals indicated in italics are those from the superficial silicon layer.

anneal	520cm <sup>-1</sup> peak		513cm <sup>-1</sup> peak	
	peak	width	peak	width
as-implant	520.0	3.56	<i>513.0</i>	<i>10.08</i>
standard anneal	<i>519.3</i>	<i>4.45</i>	-----	-----
850°C H <sub>2</sub> anneal	519.8	3.81	<i>513.3</i>	<i>9.31</i>
850°C H <sub>2</sub> anneal + standard anneal	<i>519.2</i>	<i>4.19</i>	-----	-----

This improvement was confirmed from linewidth changes of Raman scattering spectra. Result of the measurement and Lorentzian curve fitting analysis for observed spectra is summarized in Table I. In this table, signals indicated in italics are those from the superficial silicon layer. Those signals show that the linewidth of 10.08 cm<sup>-1</sup> for the as-implanted sample decreases to 4.19 cm<sup>-1</sup> with two-step anneal, and only to 4.45 cm<sup>-1</sup> by the standard anneal.

These experimental results suggest the reduction of oxide precipitates in SIMOX through an interaction between the precipitates and hydrogen atoms during the low temperature anneal stage, although the Raman scattering linewidth after this stage is not so narrow as compared to that of as-implanted sample. The same reduction of precipitates occurs with hydrogen implantation before the anneal.<sup>7)</sup>

#### 4. Conclusion

In conclusion, the following items were found. First, huge oxygen precipitates still exist in the superficial silicon layer of SIMOX material obtained by multiple implant/anneal cycle technique, although it is very effective in reducing dislocation density. Then, as an alternative technique, threading dislocations can be gettered by misfit dislocations formed at the interface of Si<sub>1-x</sub>Ge<sub>x</sub>/Si heterostructure grown onto SIMOX material. Measured dislocation density was reduced to 4.1x10<sup>7</sup> cm<sup>-2</sup> when x = 0.06,

using a heterostructure twice. Finally, two-step anneal is effective in reducing oxide precipitates in the superficial silicon layer.

By combining these methods, we believe that SIMOX would become a material able to be widely used even for bipolar devices where low defect densities are required.

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