Effect of surface proximity on end-of-range loop dissolution in silicon

R. Raman, M. E. Law, V. Krishnamoorthy, and K. S. Jones
SWAMP Center, Department of Electrical and Computer Engineering, University of Florida, Gainesville, Florida 32611-6130

S. B. Herner
Applied Materials, Santa Clara, California 95054

(Received 11 November 1998; accepted for publication 19 January 1999)

The effect of surface proximity on the dissolution of end-of-range dislocation loops in silicon was investigated by transmission electron microscopy (TEM). A layer of dislocation loops was formed at a depth of 2600 Å by annealing a Si wafer amorphized by a 10^{15} cm^{-2}, 120 keV, and a 10^{15} cm^{-2}, 30 keV dual Si⁺ implant for 30 min at 850 °C. The wafer was diced into 1 cm × 1 cm pieces and polished by a chemical–mechanical polishing technique to decrease the loop depth to 1800 and 1000 Å. The samples were then furnace annealed at 900 and 1000 °C in N₂ gas. Quantitative TEM analysis revealed that the density of small loops decreases as the loop band is brought closer to the surface. The flux of interstitials to the surface varied inversely with loop depth, indicating that the loop dissolution is diffusion limited. Assuming that the loops maintain a supersaturation of interstitials (C_{IL}) around them, and by integrating the measured interstitial flux from the loop layer to the surface, the relative supersaturation of interstitials near the loop layer (C_{IL}/C_{F}^+) was extracted 900 and 1000 °C. © 1999 American Institute of Physics. [S0003-6951(99)02611-X]

Ion implantation is the preferred method of introducing dopants into silicon for the manufacture of integrated circuits (ICs). Frequently, amorphization is used to prevent channeling of light elements. However, this results in the formation of end-of-range (EOR) dislocation loops below the amorphous/crystalline (a/c) interface after the regrowth of the amorphous layer during a high-temperature anneal. The dislocation loops affect the dopant distribution by capturing or releasing point defects during a subsequent thermal cycle. The growth of dislocation loops has been determined to be governed by a bulk diffusion mechanism, whereas the loop coarsening process, which is the redistribution of point defects within the loop band, follows Ostwald ripening.¹ The enhancement of dopant diffusivity due to the excess interstitials caused by the implantation process results in transient-enhanced diffusion (TED). Recent studies have shown that the distance between the silicon surface and the ion-implant profile plays a key role in the evolution of point and extended defects. Reduction of the transient-enhanced diffusion has been observed both when the surface is brought closer by variable etching² and when the implant profile is made deeper due to higher implant energy.³ In order to better understand and model TED, a greater level of fundamental knowledge about the evolution of loops and the role of the surface in controlling their behavior is necessary.

We have studied the effect of surface proximity on the evolution end-of-range dislocation loops in silicon. A 150 mm (100) Czochralski (CZ) -grown n-type (8–30 Ω cm) Si wafer was implanted with 10^{15} cm^{-2}, 120 keV and 10^{15} cm^{-2}, 30 keV Si⁺ on an Eaton NV/GSD 200E implanter to produce a continuous surface amorphous layer. Postimplantation annealing was performed in a furnace with flowing N₂ gas (99.999% purity) at 850 °C for 30 min. Cross-sectional transmission electron microscopy (XTEM) revealed that a band of EOR loops approximately 300 Å wide were centered at a depth of 2600 Å. The wafer was then diced into 1 cm × 1 cm pieces. A chemical–mechanical polishing (CMP) procedure⁴ that removes measured amounts of silicon and leaves the surface smooth was used on the pieces to bring the loop band closer to the surface. The depths were verified by XTEM. The three loop samples were then subjected to a second anneal for either 30 and 120 min at 900 °C or 15 and 30 min at 1000 °C. These times and temperatures were chosen based on prior studies in the growth and coarsening mechanisms of dislocation loops.¹ The control sample did not undergo any further anneals. The procedure for counting interstitials in loops from plan view transmission electron microscopy (PTEM) images and a detailed account of the counting error encountered has been reported.⁵

For the PTEM micrographs shown in Figs. 1(a)–1(d), it was found that the area density of the loops was 1 × 10^{11} cm^{-2} for the 2600 Å loop depth and 5 × 10^{10} cm^{-2} for the 1000 Å loop depth sample. Figures 2(a)–2(c) show that the size distribution for the 1000 °C, 30 min anneal gets skewed toward the larger sizes as the loops get closer to the surface. The density of the loops of diameters less than 400 Å decreases from 16×10^9 to 4×10^9 cm^{-2}, going from a loop depth of 2600 Å to a depth of 1000 Å. The loop mean diameter increased as the loop band was brought closer the silicon surface.

The number of interstitials bound by the dislocation loops in the loop control sample was found to be 4.5×10^{14} atoms. The number of interstitials lost from the loops during the anneal was determined by using the control sample as a reference and subtracting the number of interstitials trapped in loops at a subsequent time step from the reference. The
interstitials bound by loops as a function of time with respect to the control are shown in Figs. 3(a)–3(b) for the 900 and 1000 °C anneals, respectively. At 900 °C the loop sample with the band centered at 1000 Å lost $2 \times 10^{14}$ interstitials in the first 30 min of annealing as compared to $0.75 \times 10^{14}$ interstitials lost from loops in the 2600 Å sample. The curves flatten out with increasing anneal time to 120 min, indicating that the rate of loss declines with time. Similar trends are observed in the 1000 °C anneal sample. The interstitial flux is determined by the slope of a straight line fit between the control and the sample annealed for 30 min at 900 °C and a straight line fit between the control and the sample annealed for 15 min at 1000 °C. These numbers were extracted from the above plots as a function of the loop depth.

The loop dissolution is analyzed by assuming a diffusion-limited model. The loop band, at a distance $d$ from the surface, has an interstitial concentration of $C_{IL}$ in the loop boundary, which is the concentration of interstitials in the periphery of the loop layer. The equilibrium is denoted by $C^*$. The Si surface, which is assumed to be a perfect sink, maintains the interstitial concentration at the surface at $C^*$. The interstitial concentration in the vicinity of the loops, defined as $C_{IL}$, is pinned at some value near the loop band. The loops dissolve at higher temperatures to maintain $C_{IL}$, generating a flux to the surface. With a high enough thermal budget, the interstitial profile from the loops to the surface is a straight line, i.e., it is diffusion limited.

With these assumptions, the interstitial flux equation to the surface is defined as

$$\frac{dn_I}{dt} = D I C^* \left( \frac{C_{IL}}{C^*} - 1 \right),$$

where $dn_I/dt$ is the interstitial flux to the surface and $D I$ is the diffusivity of the interstitials. The $dn_I/dt$ values are determined from the loop dissolution rates described above. Experimentally determined values for $D I C^*$ are in good relative agreement with each other compared to either $D I$ or $C^*$ in which there are great variations.

Using the $D I C^*$ values from the literature, it is possible to extract $C_{IL}/C^*$ from the rate of loss of interstitials from the loops. Figure 3 shows that the rate loss changes in time. This is to be expected, because the loop distribution is also changing in time. $C_{IL}$ is a function of the loop radius, and therefore, changes in time as the loops dissolve and ripen. Consequently, the best place to investigate the surface effect $C_{IL}/C^*$ is at the shortest time, since the loop distribution starts the same for all depths. As the loops evolve in time, we can no longer assume that $C_{IL}$ is the same for all depths. Since we cannot directly measure the rate loss, we have to measure the number of interstitials at some later time. Figure 3 shows that the rate loss changes in time.
point in time, and this introduces an error into comparing the different depths to one another. If this error is small and the surface controls dissolution, we would expect to see a good match in the computed values of \( C_{IL} / C_I^* \).

The flux values can then be inserted into Eq. (1) to extract the \( C_{IL} / C_I^* \) values. Table I shows a spreadsheet of the extracted flux and the interstitial supersaturation values. The \( C_{IL} / C_I^* \) was found to be \( \sim 17 \) at 900 °C and is fairly constant with loop depth, as expected. At 1000 °C, the \( C_{IL} / C_I^* \) value drops to \( \sim 2 \). This result compares well with previously used values in the literature. It was found that the flux varies inversely with the loop depth for both the annealing temperatures. This result indicates that the dissolution of dislocation loops is diffusion limited to the surface. It also indicates that \( C_{IL} \) is not changing significantly in the loops over the initial time period.

In conclusion, it was found that the proximity to the silicon surface significantly affected the dissolution kinetics of dislocation loops. The flux of interstitials to the surface was measured using quantitative TEM analysis and was found to be varying inversely with loop depth, indicating that the loop dissolution is diffusion limited to the silicon surface. By integrating the measured interstitial flux from the loop layer to the surface, the relative supersaturation of interstitials near the loop layer \( (C_{IL} / C_I^*) \) was extracted. This work demonstrates that the loop dissolution is diffusion limited to the surface, since identical loop layers were annealed with the only difference being the amount of surface polishing. Since the dissolution rate has a nearly exact inverse depth dependence, the surface is the controlling sink.

The authors acknowledge SRC and NSF for funding this work.

---

TABLE I. A spreadsheet showing the extracted flux and the interstitial supersaturation values.

<table>
<thead>
<tr>
<th>Depth (Å)</th>
<th>Temperature (K)</th>
<th>( D_I C_I^* )</th>
<th>( dN/dt )</th>
<th>( C_{IL} / C_I^* )</th>
</tr>
</thead>
<tbody>
<tr>
<td>2600</td>
<td>1173</td>
<td>( 7.43 \times 10^4 )</td>
<td>( 4.71 \times 10^{10} )</td>
<td>17.46</td>
</tr>
<tr>
<td>1800</td>
<td>1173</td>
<td>( 7.43 \times 10^4 )</td>
<td>( 5.02 \times 10^{10} )</td>
<td>17.45</td>
</tr>
<tr>
<td>1000</td>
<td>1173</td>
<td>( 7.43 \times 10^4 )</td>
<td>( 1.22 \times 10^{11} )</td>
<td>17.40</td>
</tr>
<tr>
<td>2600</td>
<td>1273</td>
<td>( 3.19 \times 10^4 )</td>
<td>( 1.73 \times 10^{11} )</td>
<td>2.48</td>
</tr>
<tr>
<td>1800</td>
<td>1273</td>
<td>( 3.19 \times 10^4 )</td>
<td>( 2.38 \times 10^{11} )</td>
<td>2.54</td>
</tr>
<tr>
<td>1000</td>
<td>1273</td>
<td>( 3.19 \times 10^4 )</td>
<td>( 3.22 \times 10^{11} )</td>
<td>2.00</td>
</tr>
</tbody>
</table>

---