Copper gettering by aluminum precipitates in aluminum-implanted silicon

G. A. Petersen and S. M. Myers

Sandia National Laboratories, Dept. 1112, Mail Stop 1056, P.O. Box 5800, Albuquerque, NM
87185-1056, USA

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Copper in Si is shown to be strongly gettered by Al-rich precipitates formed by implanting Al to supersaturation and followed by annealing. At temperatures ranging from 600 to 800°C a layer containing Al precipitates is found to getter Cu from Cu silicide located on the opposite side of a 0.25-mm Si wafer, indicating a substantially lower chemical potential for the Cu in the molten-Al phase. Cu gettering proceeds rapidly until an atomic ratio of approximately 2 Cu atoms to 1 Al atom is reached in the precipitated Al region, after which the gettering process slows. Redistribution of Cu from one Al-rich layer to another at low Cu concentrations demonstrates that a segregation-type gettering mechanism is operating. Cu gettering occurs primarily in the region containing the precipitated Al rather than the region where the Al is entirely substitutional.

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I. Introduction

Transition-metal impurities can degrade Si-based devices by introducing deep electronic levels into the Si band gap, and by precipitating at critical Si / SiO₂ interfaces, causing electrical breakdown.¹ These effects are enhanced by the rapid diffusion of transition-metal impurities in Si, which allows metal-silicide precipitates to grow by drawing metal atoms from the macroscopic wafer volume during processing.^{2,3} In order to control this degradation, Si device manufacturers generally supplement stringent clean-room procedures with gettering, whereby transition-metal impurities are removed from the device region of the wafer by transport to remotely located sinks. Gettering is usually implemented using one or more of three approaches³: 1) formation of a region with preferred nucleation sites for metal silicide precipitation, such as point defects or SiO₂ precipitates (relaxation-induced gettering),^{1,3} 2) creation of a zone with increased impurity solubility (segregation-induced gettering), such as Al-backside gettering⁴, or 3) P-diffusion gettering, which involves dynamic processes arising from interstitial-defect gradients (injection-induced gettering).^{1,5}

While relaxation-induced gettering is effective, it is limited by the fact that the solution concentration of a metal impurity can be reduced to the thermodynamic solid solubility and no further. This solubility exceeds acceptable impurity levels at the elevated temperatures of device processing, requiring subsequent gettering at lower temperatures. Also, if a metal silicide particle precipitates at a critical device location, such as the base of a gate oxide, no driving force exists to dissolve the particle, since the metal chemical potentials at the particle and the silicide sink are the same. In contrast, segregation-induced gettering and injection-induced gettering remain active at all metal impurity concentrations.⁶

Gettering of several transition metals by Al in Si has been investigated previously.^{4,7-9} Evaporated Al films were found to getter Fe⁴ and Co⁷ as well as Cu and Ni below the Al-Si eutectic temperature, 577°C.⁸ Also, an Al doped Si layer with dopant concentrations below the solid solubility was found to getter Cu, Ni, and Au.⁹

In the present paper, we describe the gettering of Cu by Al precipitates within the Si matrix at temperatures from 600 to 800°C, above the eutectic where the Al-Si phase is molten. The precipitates were formed by implanting Al to levels above the solid solubility and then annealing. Aspects investigated include the stability of the gettered state relative to that of the Cu_3Si phase, the maximum concentration of strongly gettered Cu in the precipitated Al phase, the segregation character of the gettering mechanism, and the dominance of the Al-precipitation gettering over gettering by substitutional Al.

II. Materials and Experimental Techniques

Two types of experiment were carried out in order to characterize Cu gettering by Al precipitates. In the first, 0.25 mm-thick, double-side polished float-zone (111) Si samples were implanted on one side with 1×10^{16} Al/cm² at an energy of 180 keV, with an average projected range of 300 nm. The specimen was then annealed at 800°C for 1 hour to induce Al precipitation and activation of substitutional Al. All anneals were done in a continuously ion pumped quartz furnace tube. The pressure during the anneals was $\sim 10^{-7}$ Torr and the anneal was concluded by removing the tube from the furnace and allowing the samples to cool to room temperature under vacuum. It has previously been found that when Al is ion-implanted into Si at levels above the solubility and then annealed, the Al precipitates into a double-peak structure in the region where the Al concentration exceeds its solid solubility, ($\sim 10^{19}$ cm⁻³ at 700°C)¹⁰ with a diffusion tail of substitutional Al extending to greater depths.¹¹⁻¹⁴ Similar behavior was observed in our experiments. The 800°C anneal was followed by an implant of 1×10^{17} Cu/cm² on the opposite side of the wafer at an energy of 150 keV, giving an average projected range of 125 nm. As shown previously, this treatment leads to the formation of Cu_3Si during subsequent annealing at temperatures of 600°C or greater.¹⁵ As a result, the silicide acted as a continuous source of metal atoms to solution, thereby stabilizing the adjacent solution concentration of Cu at the solid solubility during the vacuum gettering anneals at 600, 700, and 800°C. During the

anneals, the redistribution of the Cu from the Cu_3Si to the Al precipitated region was measured as a function of anneal time.

The second type of experiment also used 0.25 mm double-side polished float-zone (111) Si samples. These samples were implanted with $1 \times 10^{16} \text{ Al/cm}^2$ at an energy of 180 keV on both sides and annealed at 800°C for 1 hour to form the Al precipitates at a range of 300 nm. Copper was then implanted on one side to a dose of $3.4 \times 10^{15} \text{ Cu/cm}^2$ at an energy of 150 keV, locating the Cu in a range between the surface and the precipitated Al layer and centered at depth of 125 nm. Subsequent vacuum annealing at 700°C caused the Cu to diffuse to the Al regions, first to the closest Al layer and then across the wafer to the second layer.

Rutherford backscattering spectrometry (RBS) and secondary-ion mass spectrometry (SIMS) were used to measure the diffusive redistribution of metal atoms in both the Cu silicide-to-Al layer experiment and the Al layer-to-Al layer experiment. The SIMS concentration measurements were calibrated using low-dose ion-implanted Si standards; the sensitivity was $\sim 10^{15} \text{ cm}^{-3}$ for both Cu and Al. An inherent limitation of SIMS is the difficulty of measuring high ($\geq 1.0 \text{ at.}\%$) concentrations of the metal impurity atoms in a Si matrix due to non-linearities in sputtering. This is illustrated in Figure 1a, where a comparison of the depth profiles from both SIMS and RBS of Cu gettered to an Al layer in Si is shown. The two profiles have similar shape, however, when calculating the areal density, the SIMS result gave a 20% higher value than the RBS result. Thus, RBS is better suited to the task of measuring the Cu in these samples containing higher Cu concentrations. Figure 1b shows the RBS versus SIMS concentration profiles for Cu in an Al layer where the concentration of Cu in Si is less than 0.5%. Here, the resulting RBS and SIMS data are much more comparable. Because of the limited sensitivity of RBS, SIMS data was better suited for measurements of the Cu impurity concentrations in the concentration regime below 0.5 at.%. In all cases the peak Al concentrations were on the order of 1.0 at.% in Si, and since RBS is not well suited to measure Al in Si, the Al concentrations were taken from SIMS.

III. Results

The first type of experiment involved the redistribution of metal atoms from a Cu silicide layer to a layer containing molten Al-Si precipitates. Figure 2 shows the areal density of the gettered Cu in the Al region for 600, 700, and 800°C versus anneal time as measured by RBS. Also shown is the calculated accumulation rate for steady-state diffusion of Cu from Cu_3Si to a strong sink. This flux is given by:

$$\Phi \cong C_{\text{sol}} D / \Delta x, \quad (1)$$

where C_{sol} is the metal solid solubility, D is the metal diffusion coefficient, and Δx is the average interlayer diffusion distance. At each temperature, the values of C_{sol}^{16} and D^{17} were obtained from the literature. For the three temperatures studied, the amount of gettered Cu accumulated rapidly to an areal density of approximately $2 \times 10^{16} \text{ cm}^{-2}$, or about twice the implanted dose of Al. This was followed by continued gettering, but at a slower rate. Note that the observed rate of redistribution agrees quite well with the rate calculated for diffusion-limited flow, until the point where the gettering slowed. Rutherford backscattering spectrometry after the final anneal showed that Cu_3Si still remained on the source side of the samples, so that the solution concentration of Cu in this region remained close to the solid solubility.

Figures 3a and 3b show the concentration profiles of the Al and Cu in the gettering layer at 600°C and 800°C, respectively, as measured by SIMS after the final anneals. The precipitated Al double-peak structure described by others in previous works is seen.¹¹⁻¹⁴ The SIMS data show a proportionality between the concentrations of precipitated Al and the gettered Cu at depths where the Al is precipitated. At greater depths where the Al concentration is below the solubility, and therefore not precipitated, the gettering of Cu is much less pronounced. This indicates that the precipitated Al gettered Cu much more strongly than substitutional Al. In a separate experiment, increasing the implanted dose of Al by a factor of three resulted in a

proportional increase in the amount of gettered Cu. At the highest temperatures, 800°C, there was about a 30% loss of Al from the Al precipitated region. This was probably due to surface segregation of the Al to form Al_2O_3 in the near surface region. An examination of the SIMS data in Figure 3b shows a large near-surface Al peak, presumably arising due to this effect.

The second type of experiment examined the redistribution of metal atoms from one precipitated Al layer to another. Figure 4 shows the measured areal density of Cu in the second Al layer after annealing at 700°C. The dashed line is experimental data from Figure 2 showing the Cu redistribution from a Cu silicide to an Al-Si layer at 700°C and is plotted for comparison. During the first 0.5 hour anneal, the initially implanted Cu moved quickly into the nearest Al layer and then diffused to the second layer on the backside of the sample. Approximately 2×10^{15} Cu/cm² remained in the near-side precipitated Al layer, while the remainder, 1.4×10^{15} Cu/cm² went to the second layer. After annealing for one hour, two-thirds of the initially implanted Cu had diffused across the wafer to the second Al layer. Subsequent annealing showed a reduced, yet still non-zero, gettering rate to the second layer.

Following the final 700°C anneal, SIMS analysis was performed to examine the Al and Cu concentration profiles on both the front and the backside of the sample. Again there was a direct proportionality between the precipitated Al and the gettered Cu, with very little Cu being gettered to the substitutional Al region. A large variation between the initially implanted amount of Al in the first layer (1×10^{16} Al/cm²) and the amount remaining after the final anneal (4×10^{15} Al/cm²) was seen. This apparent loss of Al from the sample will be discussed in the following section. Very little Al was lost from the second precipitated Al layer, with 9.4×10^{15} Al/cm² remaining. The amounts of Cu gettered to the front and back layers after 2 hours at 700°C were 1.3×10^{15} Cu/cm² and 2.0×10^{15} Cu/cm², respectively.

III. Discussion

Precipitated Al strongly gettered Cu from Cu_3Si at all of the investigated temperatures. In the initial stage of gettering, the Cu went into solution from the bound state of the silicide and diffused to the precipitated Al layer on the opposite side of the wafer. The dashed lines in Figure 2 show the predicted rate of redistribution for an infinitely strong sink. The correspondence between these lines and the experimental data suggests that the chemical potential of the gettering sink formed by the Al precipitates is initially much lower than that of the silicide. When the amount of the Cu in the sinks rose above a certain level, the process slowed, indicating a weaker driving force or similar chemical potential between the Cu in the silicide and the Cu in the precipitated Al phase. Since the final areal density of Cu gettered to the Al precipitated region was more than a factor of two greater than the implanted Al dose, a strong possibility exists that in addition to the liquid Al-Cu-Si phase, other Al-Cu phases precipitated. This would cause a reduction in the gettering rate if the Cu in the new phase or phases has a chemical potential only slightly lower than that of the Cu in the Cu_3Si . By studying the phase diagrams of both the binary Al-Cu¹⁸ system and the ternary Al-Cu-Si^{19,20} system, a detailed explanation of the reduction of the gettering rate can be proposed.

Initially, at the elevated temperatures investigated, there exists Cu_3Si on the source side of the sample, as shown by previous investigations of implanted Cu in Si.¹⁵ On the gettering side of the sample, the implanted Al exists as a molten Al-Si phase as observed in previous studies¹¹ and as expected at temperatures above the binary Al-Si eutectic.²¹ As Cu is gettered to the Al region, a molten Al-Cu-Si mixture develops. As still more Cu is gettered to the Al precipitated region, solid phases will precipitate out of the molten liquid. Examination of the ternary phase diagram shows that these phases will essentially consist of binary Al-Cu phases with a small amount of Si included. Based on the Al-Cu binary phase diagram, the initial solid Al-Cu phases will precipitate at Cu:Al ratios of approximately 0.5 at 600°C and 1.0 at 800°C. As the Cu concentration increases still further, a succession of single-phase and multi-phase conditions can occur. In this investigation, at all investigated temperatures, the gettering

proceeds rapidly to a Cu:Al ratio of about 2.0-2.5, at which point the gettering process slows dramatically. The 600°C case, shown in Figure 2, allows the most accurate determination of the Cu:Al ratio because there is little continued gettering after the gettering rate slows, and there is no significant loss of Al from the gettering layer. A Cu:Al ratio of 2.3 is measured by SIMS after a total anneal time of 50 hours. The determinations at the higher temperatures are less accurate due to the evolution of the system after the slowdown of the gettering process, and the loss of some Al from the precipitated Al layer. However, by examining Figure 2 at the point where the initial gettering slows, and assuming a nominal Al areal density of 10^{16} Al/cm² (no Al loss), a Cu:Al ratio of approximately 2.3 for both the 700 and 800°C cases can be extracted. According to the binary phase diagram, this concentration corresponds closely to the γ_1 -phase of the Al-Cu binary, which has a nominal composition of Al₄Cu₉, or a Cu:Al ratio of 2.25. The reduction in the gettering rate is then believed to occur when the Cu:Al ratio rises above that of the γ_1 -phase of the Al-Cu alloy, where still more Cu-rich phases are expected to form. In this region, the difference in the Cu chemical potential between the Al-Cu alloy and the Cu₃Si source phase has become small.

Figures 3a and 3b display the near proportionality between the concentrations of the precipitated Al and the gettered Cu. The Cu was gettered much more strongly to the precipitated Al phase than to the substitutional Al, as evidenced by the Cu:Al ratio being much greater in the region containing the precipitated Al than in the region containing substitutional Al alone. Moreover, as was mentioned earlier, when the implanted Al dose was increased by a factor of three, a proportional increase in the amount of gettered Cu resulted. While gettering by substitutional Al has been reported elsewhere,⁹ substitutional gettering was not seen in the present experiments where a precipitated Al phase was present, suggesting that gettering by substitutional Al is less energetically preferred.

In the second type of experiment performed in this study, the redistribution of Cu from one precipitated Al layer to another was examined. The total amount of Cu implanted into the system was sufficiently small to prevent precipitation of Al-Cu solid phases in addition to the

molten Al-Cu-Si phase. When annealed, the initially implanted Cu, located near the first precipitated Al layer, moves quickly into that layer. Within the precipitated Al layers, as the Cu concentration increases, the chemical potential of the Cu also increases. The chemical potential difference between the two layers results in a driving force to redistribute the Cu from the first layer containing a higher Cu concentration to the second layer containing a lower Cu concentration. So, as the anneal continues, Cu is getterred to the second precipitated Al layer from the first until the redistributive flow stops and the chemical potential between the two gettering layers becomes equal. Upon completion of the redistribution, the Cu:Al ratio in the two precipitated layers should be equal.

After annealing the sample at 700°C for two hours, SIMS analysis of both sides of the sample showed a 60% loss of Al from the first layer and little to no loss from the second layer, resulting in sink layers that were no longer identical. The second layer, with more precipitated Al, getterred more Cu than the first layer. Measuring the getterred amount of Cu in the first and second precipitated Al layers yielded Cu:Al ratios of 0.32 and 0.22, respectively. Examination of the Al-Cu binary phase diagram at 700°C showed that for the above Cu:Al ratios, the gettering phase is still entirely liquid, suggesting that the redistribution was occurring as described above. However with the simultaneous loss of Al from the first precipitated layer, full equilibration between the two layers may not have been achieved, thereby accounting for the greater Cu:Al ratio in first layer.

In principle, the redistribution of Cu between the two molten Al-Si sinks can be used to determine a value for the activation energy associated with the transfer of one Cu metal atom from its bound state in the precipitated Al into interstitial solution. However, due to complications arising from the loss of Al from the first Al-Si layer during the gettering anneal, quantification of the activation energy was not performed. The Al loss occurred only on the side of the wafer which contained the initially implanted Cu and not from the second Al layer. Possibly, the loss of Al from the first layer is due to an enhanced surface-segregation of the Al due to near-surface implant damage caused by the Cu implantation that followed the formation

of the Al precipitates. Further annealing at the gettering temperatures might allow for enhanced-diffusion of the Al to the surface.

A more qualitative conclusion was obtained by comparing the 700°C experimental data in Figure 2, showing the Cu redistribution from Cu_3Si to a molten Al-Cu-Si layer, to the data in Figure 4, showing the Cu redistribution at 700°C between molten Al-Cu-Si layers. For a more direct comparison, the dashed line of Figure 4 is the 700°C data of Figure 2 re-plotted. In both cases, the gettering layer consisted of Al-Si precipitates on the opposite side of the sample. A key point is that the Cu redistribution from one Al-Si layer to another is more than a factor of 3 times slower than the Cu redistribution from Cu_3Si to an Al-Si layer. Assuming that the diffusion coefficient for Cu in Si at 700°C for both experiments is equal, then the statement can be made that the solution concentration of Cu in the vicinity of the Al-Si precipitates is lower than the solution concentration of Cu near the Cu_3Si , implying the corresponding Cu chemical potential difference. This is as expected given that the first set of experiments displayed the ability of the Al-Si precipitates to getter substantial amounts of Cu from Cu_3Si .

As mentioned earlier, at impurity concentrations <1.0 at.% in Si, SIMS is more quantitative than at higher concentrations and provides higher sensitivity than RBS. Figure 5 shows the concentration profiles of the Cu and the Al in the second layer after annealing at 700°C for 2 hours as measured by SIMS. Examination of the Cu:Al ratios in the precipitated and the substitutional regions reveals that the ratio in the precipitated phase is more than 10 to 100 times greater than the ratio in the substitutional Al region. This again shows that the Cu is getterred much more strongly to the precipitated liquid Al-Si phase.

V. Conclusions

Implanted and annealed Al in Si formed precipitates when the Al concentration exceeded the solid solubility. These precipitates strongly getterred Cu at elevated temperatures ranging from 600°C to 800°C, dissolving Cu_3Si located on the opposite side of a 0.25-mm Si wafer, and

gettering more than 2 Cu atoms per atom of implanted Al. At all investigated temperatures, the gettering proceeded relatively rapidly to a Cu:Al ratio of about 2.3, at which point the gettering process slowed. This transitional atomic ratio corresponds to the γ_1 -phase of the Al-Cu binary system. When precipitated Al was present, gettering of Cu to substitutional Al sites was negligible compared to the gettering of Cu to the molten Al-Si phase. It was determined that the Cu chemical potential in the precipitated Al region increases with increasing Cu concentration, and that the binding free energy of Cu in the liquid Al-Si precipitates relative to solution was much larger than that of Cu in Cu_3Si . These results suggest that the use of Al precipitates is technologically attractive for applications requiring the removal of Cu impurities from Si over extended diffusion distances in order to improve the material's electronic properties. Examples of such applications would include improved efficiencies and minority carrier diffusion lengths in silicon solar cells, and reduced impurity levels in Si used for microelectronic device processing, resulting in higher device yields.

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Figure Captions

- FIG. 1. Comparison of the RBS and SIMS profiles of Cu in a precipitated Al layer: (a) after annealing at 600°C for 50 hours resulting in a Cu concentration >1.0 at.%; (b) after annealing at 700°C for 2 hours resulting in a Cu concentration <1.0 at.%.
- FIG. 2. Diffusive redistribution of Cu from Cu_3Si layer to a precipitated Al-Si layer at three temperatures. The predicted redistribution of Cu from Cu_3Si to a strong sink at each temperature is also shown (dashed lines).
- FIG. 3. SIMS depth profiles of Al and gettered Cu in Si after annealing (a) at 600°C for 50 hours, and (b) at 800°C for 8 hours, where the Cu was gettered from Cu_3Si located on the opposite side of a 0.25-mm wafer.
- FIG. 4. Diffusive redistribution of Cu from a precipitated Al-Si layer to another precipitated Al-Si layer at 700°C. The redistribution of Cu from Cu_3Si to an Al-Si layer at 700°C (from Fig. 2) is also shown (dashed line) for comparison.
- FIG. 5. SIMS depth profiles of the second precipitated Al layer in Si showing the Al and gettered Cu after annealing at 700°C for 2 hours, where the Cu was transferred from the first precipitated Al layer on the opposite side of a 0.25-mm wafer.

3a

3b

5