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Use of a buried loop layer as a detector of interstitial flux during oxidation of SiGe heterostructures

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The injection of interstitials from oxidation of Si and SiGe has been investigated quantitatively using transmission electron microscope (TEM) to monitor the growth of a layer of implantation induced dislocation loops. The layer of loops was introduced via a 50 keV P implant at a dose of $2 \times 10^{14}/cm^2$ followed by annealing at 750 °C. Subsequently, silicon–germanium containing heterostructures, consisting of a 5 nm silicon cap on top of either a 20 nm $Si_{0.7}Ge_{0.3}$ layer or 25 nm Si layer were grown on the implanted wafers. The wafers were then oxidized, and the trapped interstitials in the dislocation loops were determined via quantitative plan view TEM. It is shown that the SiGe layer and the inherent epitaxial interfaces are fully transparent to a flux of interstitials arising from the oxidizing interface. As expected, oxidation of the Si control and Si on SiGe result in strong interstitial injection. However, for the latter sample, as the oxidation front proceeds into the SiGe layer, interstitial injection is reduced and eventually halts as the Ge accumulates at the oxidizing SiGe interface. At 900 °C after 2 h in dry O_2, the oxidizing interface injects $3 \times 10^{14}/cm^2$ of interstitials and this value drops to below $1 \times 10^{13}/cm^2$ after oxidation of SiGe. These finding are consistent with the concept that the presence of Ge decreases the strain at the interface, reducing interstitial injection. © *2016 American Vacuum Society*.
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I. INTRODUCTION

Silicon germanium alloys play an important and growing role in the microelectronics industry, where they are commonly used for both bandgap and strain engineering in multiple device types.[1,2] Efforts toward larger scale integration of different SiGe technologies and device types on the same die has placed an increased emphasis on understanding the role of thermal processing subsequent to epitaxial growth,[3] and novel processes involving the thermal oxidation of SiGe alloys is still an active area of ongoing research.[4] Thermal oxidation of silicon is well known to generate a flux of interstitial atoms at the oxidizing interface, some of which then travel into the bulk of the material contributing to oxidation enhanced diffusion (OED) of impurities and extended defect growth.[5–7] The presence of germanium at the oxidizing interface greatly reduces this phenomenon.[8] Buried dislocation loops produced from ion implantation damage serve as an efficient trap of silicon interstitial atoms, and can be used to quantify the flux of interstitials through a material.[9–11] In this work, we combine these two phenomena to precisely quantify the effect of a SiGe layer on interstitial injection during dry oxidation and demonstrate the use of loops as a viable method for determining a dynamic change in the point defect behavior of a material during oxidation of a Si-SiGe heterostructure.

II. EXPERIMENT

Czrochralski (100) silicon wafers were implanted with 50 keV P^+ ions at a dose of 2×10^{14} cm^{-2} and subjected to a furnace anneal of 750 °C for 30 min under argon ambient. Although this implant dose is on the verge of the amorphization threshold for silicon, the implant was verified to be nonamorphizing via cross-section TEM (XTEM). This nonamorphizing implant and subsequent anneal served to form a buried defect layer centered at a projected range, R_p of ~70 nm deep and well into the loop stage of defect evolution. This anneal also served to repair any surface damage prior to subsequent epitaxial growth, and minimize the impact of any thermal treatment during the growth process. Following this anneal, a low temperature epitaxial growth process was performed by Global Foubdries. SiGe samples grown consisted of the growth of a 5 nm Si buffer layer, followed by 20 nm of $Si_{0.7}Ge_{0.3}$ and a 5 nm Si capping layer. All layers were confirmed to be pseudomorphic (defect free) by TEM. A set of control samples were also grown which consisted of a uniform Si layer 30 nm thick grown on top of the implanted layers. The Si thickness grown was selected to ensure that the buried loop layer was at the same depth for all samples. Postgrowth, samples were annealed for times ranging 15 min to 2 h at 900 °C, in a tube furnace under an ambient of argon or pure dry oxygen. Plan-view TEM (PTEM) specimens were prepared via a method of mechanical polish and chemical etch, while XTEM samples were prepared using a focused ion beam system. Implantation defects were imaged in PTEM using g_{220} and g_{040} weak beam dark field imaging conditions on a JEOL 200CX or

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JEOL 2010F TEM. The bound interstitials were quantified using a well established method of measuring the area of the loops and assuming the areal density of bound interstitials is 15.6 nm^{-2}.[12] Measurements were performed using OSIRIX image processing software (nonmedical version)[13,14] and error determined by quantifying four images taken from separate areas of the sample and calculating the standard deviation of the bound interstitials and loop density.

III. RESULTS AND DISCUSSION

PTEM and XTEM of the grown SiGe initial experimental material structure are shown in Figs. 1(a) and 1(b). The buried loop layer is clearly present at a depth of ∼100 nm, which is sufficiently deep to avoid being consumed by the oxidation process. The density of the loops can be seen in Fig. 1(b) and is enough to serve as an efficient trap of interstitials.

After thermal oxidation, similar thicknesses of oxide formed on both SiGe and silicon control samples as shown in Figs. 2(a) and 2(b). The germanium was rejected from the oxide to form a concentrated pileup layer in Fig. 2(b), as expected from literature.[15–17] It is worth noting that the quantity of oxide grown exceeds that predicted by the Deal–Grove model. The furnace was carefully calibrated by thermocouple, but it is possible that a very small quantity of moisture could have contaminated the oxygen supply, which is well known to dramatically enhance the oxidation rate even at very low levels. However, any moisture that might have been present was not sufficient to enhance the rate of germanium oxidation relative to silicon oxidation, which is a well understood phenomenon.[18,19]

Despite identical oxide growth, only the oxidized silicon sample showed substantial loop growth at longer oxidation times, while the loops in the SiGe sample under oxidation appeared almost identical to those of samples annealed under inert ambient. This suggests total or near total suppression of interstitial injection, in agreement with literature.[8,20] However, in this study, we show that the magnitude of suppression can be readily quantified dynamically using the buried loop detector method.[9–11] Figure 3(a) shows that the bound interstitials in the layered SiGe sample under oxidation closely matches those in the pure silicon sample until a time of 0.5 h, after which it rapidly drops off to the same number as found in the unoxidized samples. This initial parity is due to the presence of the 5 nm silicon capping layer, which injects interstitials as it oxidizes.

The consumption of 5 nm of silicon at 900 °C under dry oxidizing ambient occurs in roughly 20 min according to the modified Deal–Grove model utilized in the FLORIDA OBJECT ORIENTED PROJECT SIMULATOR software,[21] which is in good agreement with the trend of bound interstitials in our data. This transient could also partially be due to the initial pileup of germanium required before complete shutoff occurs.[20] The measured transitory flux of interstitials prior to suppression of interstitial injection when the oxidation front arrives at the SiGe layer also shows that epitaxial interfaces within the SiGe are not trapping interstitials prior to their arrival and capture at the loop layer. Previous literature has reported that a Si/SiGe interface can act as a trap for interstitials, but this is not observed in our work as the interstitials must pass through two such interfaces prior to reaching the loop layer.[22] As soon as the 5 nm capping layer is consumed, interstitial injection shuts off completely and both the bound interstitial population and loop density return to an equilibrium value due to coarsening that is virtually identical to that seen under inert conditions for similar times. One potential downside of using loops as a detector is that they undergo thermal evolution (coarsening) during annealing. However, once formed, these loops are extremely stable even at very long times and high temperatures and as such the total loop area is a better indicator of bound interstitials than the total loop density provided that the loop density remains above $1 \times 10^{10}/\mathrm{cm}^2$ and can thus serve as an efficient trap for any passing flux of point defects.[9,23] The loops do not dissolve under annealing at these temperatures, but rather undergo a thermal coarsening effect. For this reason, it is important to have a high starting density of loops and also important to have inert control samples to account for this coarsening effect. Since loops grow or shrink by a climb mechanism, their trapping efficiency could potentially decrease if they become too large, but in all our samples, sufficient space was available between loops for climb to occur. Because

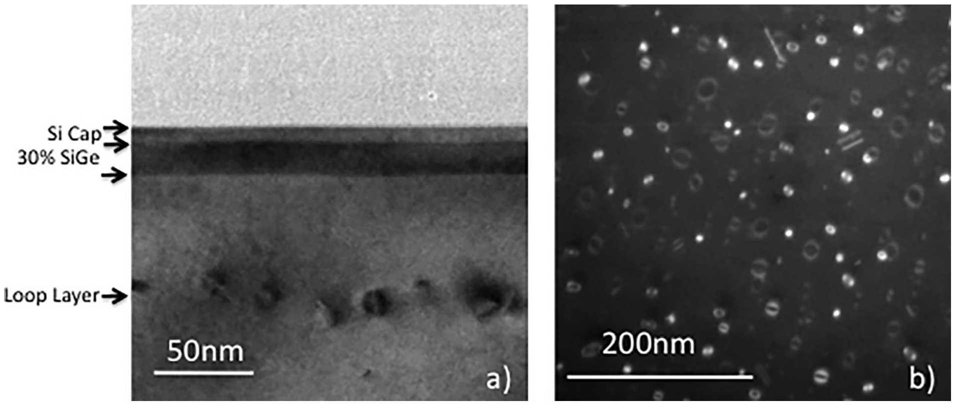

FIG. 1. Starting structure of samples in (a) XTEM and (b) PTEM. Silicon samples were identical to the one pictured here except without the SiGe layer. Defect morphology and depth was identical for both sets of samples.

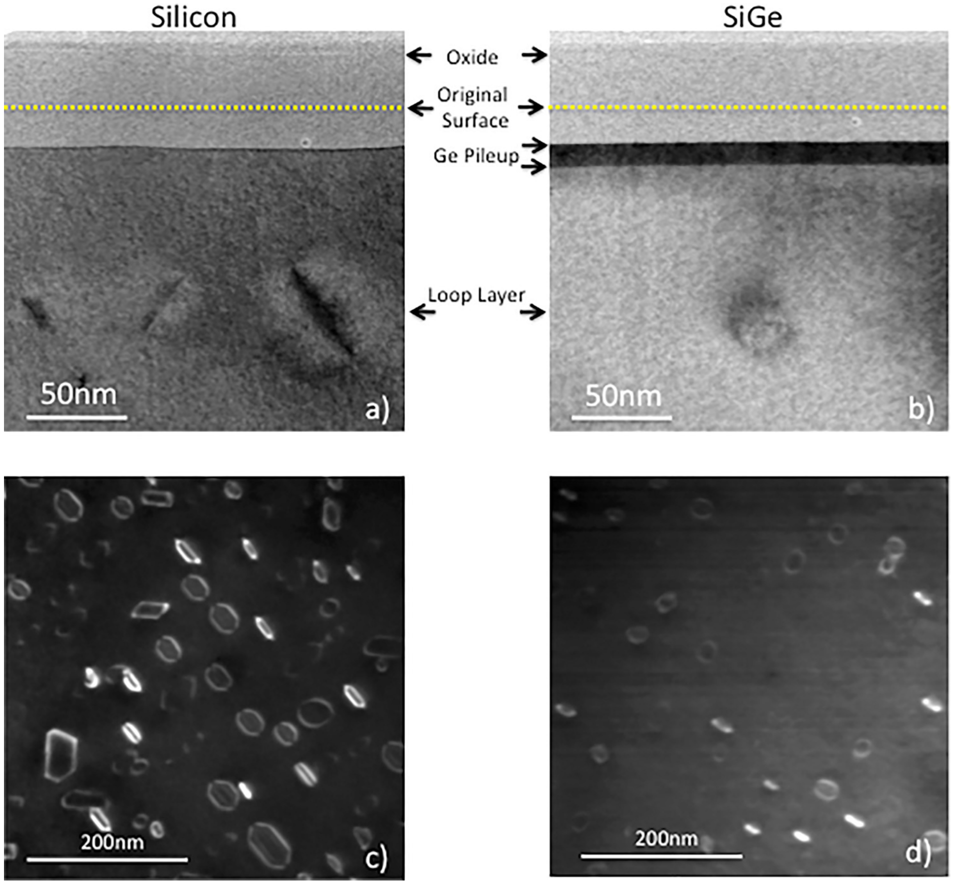

FIG. 2. (Color online) Samples after 2 h oxidizing anneal at 900 °C. Oxide thicknesses for both (a) Si and (b) SiGe samples were found to be identical. Loops are seen to have grown dramatically in samples (c) without germanium, whereas the loops in the (d) germanium sample have decreased in both size and density relative to their starting value in Fig. 1 due to thermal coarsening effects.

loops grow or shrink through a climb mechanism active at the edge, it is important to have a sufficiently high density of edges to serve as an efficient trap.[9]

Figure 3(a) also shows that the rate of interstitial injection by the silicon control sample is roughly linear over the investigated oxide growth regime, when taking into account the initial coarsening effects. It is also readily possible to determine from the oxide thickness and the loop growth the fraction of oxidized silicon atoms that are injected and captured. The total fraction of oxidized atoms injected as interstitials after 2 h in the silicon control is 0.029 interstitials/oxidized atom, which is in agreement with literature, suggesting that only a very small fraction of the excess atoms arising from volume mismatch between the oxide and

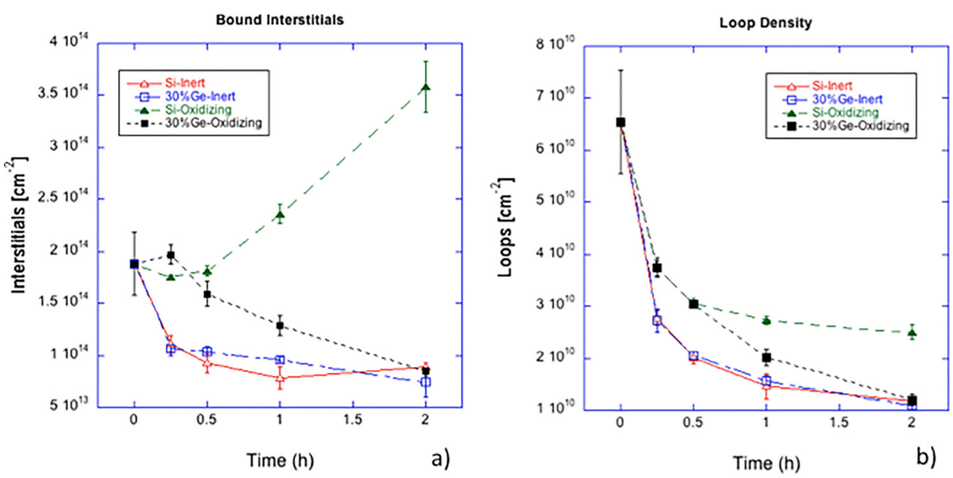

FIG. 3. (Color online) (a) Interstitial and (b) loop density as a function of annealing time for Si/SiGe and Si control samples under oxidizing and inert ambient.

silicon get injected into the substrate.[24,25] Despite the same number of silicon atoms being consumed in the SiGe containing sample, any injection taking place was below the detection limit, giving an effective injected fraction of zero. One possible mechanism for the Ge suppressing the injection to a level below the detection limit is the larger Ge atom not participating in the oxidation reaction helps to alleviate the strain induced by the volume mismatch.[24,26] Another is that the piled-up germanium could serve to alter the fraction of generated interstitials getting injected into the bulk while the reaction still generates the same number at the interface.[20]

IV. SUMMARY AND CONCLUSIONS

These results prove the viability of using a buried loop layer as a detector for specifically determining the point of interstitial shutoff during an oxidation of layered materials containing SiGe, and quantifying the number of interstitials injected at each point. This method can also be used to investigate properties of epitaxial interfaces and layered structures to determine their transparency to a flux of interstitials. This method could also prove viable to other systems or heterostructures where the transparency of an intermediate layer to a flux of interstitials needs to be quantified, or where point defect injection is expected to start and stop at different points during processing. Using control samples, we determine the precise fraction of oxidized atoms being injected as interstitials. By applying this method to a SiGe containing heterostructure, we were able to determine that the interstitial injection drops below the detection limit once the Ge containing layer was reached, and persisted upon further oxidation.

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