Evolution of the defect structure in helium implanted SiGe/Si heterostructures investigated by *in situ* annealing in a transmission electron microscope

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The evolution of the He-implantation induced defect structure in SiGe/Si heterostructures is observed during *in situ* annealing at 650 and 800 °C within a transmission electron microscope. The He implantation and annealing results in the formation of He precipitates below the SiGe/Si interface, which at first show a platelike shape and subsequently decay into spherical bubbles. The coarsening mechanism of the He bubbles is revealed as coalescence via movement of entire bubbles. The nucleation of dislocation loops at overpressurized He platelets and their propagation into the heterostructure could be observed as well. We found distinctly different velocities of the dislocations which we attribute to glide and climb processes. The *in situ* experiments clearly show that the He platelets act as internal dislocation sources and play a key role in the relaxation of SiGe layers.
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Substantial improvement of the performance of Si-based microelectronic devices can be achieved by the implementation of strained silicon as high-mobility channel material.[1] Since strained silicon is generally realized by epitaxial deposition of Si on relaxed SiGe layers, relaxed buffers in an adequate quality are required. Efficient relaxation of thin SiGe films with a thickness of 100–200 nm and Ge contents of up to 30% was demonstrated by ion implantation of H or He and subsequent annealing.[2–7] Its mechanism, however, is still under debate. Recently, Schwarz[8] simulated for a similar model system the dislocation dynamics during strain relaxation by assuming solely dislocation glide processes. Here, we follow an analytical model proposed by Trinkaus *et al.*[9] where H/He implantation and annealing induces the formation of dislocation loops created at overpressurized He precipitates located underneath the SiGe/Si heterointerface. These loops glide to the interface where subsequently one part unlaces up to the surface and creates two threading segments, whereas the lagging part in the interface forms a strain-relieving misfit dislocation segment. Driven by the misfit strain the two threading segments move apart and eventually annihilate with threading dislocations of opposite Burgers vector. As a consequence, efficient and "healthy" strain relaxation is observed. Measurements of the pressure within the platelike shaped He precipitates, which represent the initial stage of He precipitates,[10,11] revealed the shear stress to reach the critical value of dislocation formation.[12] This indicates that He platelets may be capable to emit dislocations. In order to gain deeper insight into the relaxation mechanism of SiGe layers, we studied the evolution of the helium bubble structure as well as its interplay with nucleation and growth of dislocation loops utilizing *in situ* annealing investigations by transmission electron microscopy (TEM).

$Si_{81}Ge_{19}$ layers with a thickness of 170 nm were deposited onto Si (001) substrates by chemical vapor deposition. Helium was implanted with an energy of 37 keV at a dose of 1×10^{16} cm^{-2} resulting in a helium concentration profile with its maximum in a depth about double the layer thickness. These implant conditions are optimum with respect to high degree of relaxation and low threading dislocation density achievable after annealing at 850 °C for 10 min.[7] In order to start the *in situ* experiment with a defined defect structure, the formation of overpressurized He filled platelets was provided by an *ex situ* annealing step at 420 °C for 1 min.[11,12] Subsequently, TEM plan-view samples were prepared by mechanical polishing and subsequent argon ion milling.

The *in situ* annealing experiments were performed in JEOL 4000FX transmission electron microscope operated at 400 kV equipped with a GATAN-heating holder: The temperature was raised up to 510 °C within 20 s and subsequently in intervals of about 30 °C/min up to the desired observation temperature of 650 and 800 °C, respectively. For imaging an in-column GATAN type 673 TV camera in combination with a digital video recording system enable observations with 25 frames/s at a discretization of 720 $\times$576 pixels.

The evolution of the defect structure is observed by *in situ* annealing of thin TEM samples. Besides the possibility of enhanced helium outdiffusion due to the additional surface, strain relaxation within the thin TEM foil has to be considered. By choosing preannealed material a well-defined precipitate layer is created as starting condition of the experiments, such that most of the He is confined to the precipitates prior to *in situ* annealing. Therefore, the mechanism of dislocation formation and coarsening of the bubble structure can be assumed to comply with the situation in the bulk.

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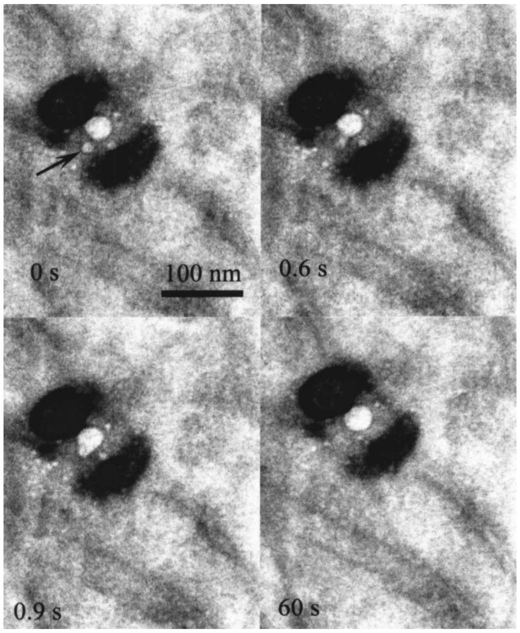

FIG. 1. Time evolution in the *in situ* TEM recorded under kinematical underfocused imaging conditions at 800 °C in a plan-view specimen. Coarsening of the planar bubble arrangements by coalescence takes place until mostly single bubbles remain after longer annealing times. The dark contrast regions induced by minor diffraction effects due to the high pressure inside the precipitates decrease at longer annealing times.

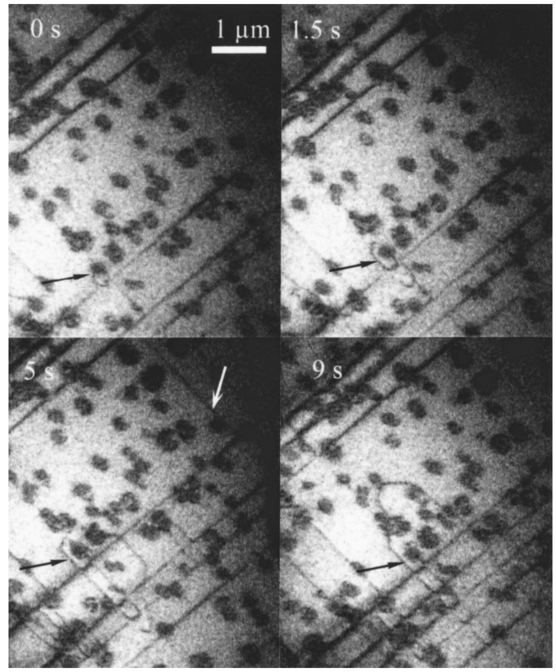

FIG. 2. Time evolution in the *in situ* TEM recorded under dynamical $g = [400]$ bright field imaging conditions at 650 °C in a plan-view specimen. At helium filled platelets, which lie underneath the heterostructure interface and are visible here as dark areas, dislocation loops nucleate (marked with black arrows) and enlarge in all directions within the interface for distances of several micrometers. In addition threading dislocations cross the area of observation leaving behind misfit segments (marked with white arrow).

Indeed, both cases, i.e. *ex situ* and *in situ* anneals, qualitatively show the same typical defect structure. However, quantitatively differences are observed, for instance with respect to the final misfit dislocation density.

The preannealing step at 420 °C results in the formation of He platelets, which show similar characteristics as observed for implantation studies in Si.[12] The platelets with typical diameters from 40 to 120 nm are uniformly distributed below the SiGe/Si interface and dominatingly show {100} habit planes with preferential orientation parallel to the wafer surface. According to the large He pressure of up to 13 GPa the surrounding Si matrix is heavily strained. No misfit dislocations can be observed in this initial stage.

During annealing the He platelets decay into planar arrangements of bubbles as reported previously.[10,13] Having undergone the shape transformation, the bubbles show a coarsening behavior upon annealing at higher temperatures, which is demonstrated by video frames obtained at 800 °C (Fig. 1). The bubbles show bright contrast due to the kinematical, underfocused imaging condition adjusted. A large central bubble is surrounded by smaller ones. All bubbles move in stochastical manner during the experiment. Clearly, a coalescence of two bubbles can be observed. The dark contrast lobes indicate that the bubble arrangement causes strain in the surrounding Si matrix. These contrasts diminish during several minutes of annealing.

In previous investigations it could not be clarified whether the coarsening mechanism in silicon is driven by coalescence or is dominated by Ostwald ripening,[4,13,14] which implies that He diffusion through the bulk is the major mechanism to transport He from small precipitates to larger ones. This investigation shows that entire bubbles move stochastically within the bubble arrangement and finally coalesce. Therefore, surface diffusion, i.e., Si diffusion on the bubble's surface, is the fundamental process of the movement.

To monitor the formation and evolution of the dislocation structure a temperature of 650 °C was adjusted, which represents the onset of relaxation. According to the (400) two-beam imaging condition chosen in Fig. 2 local strain fields surrounding the platelets and the dislocations show dark contrast. The platelet marked with the black arrow shows an ejected dislocation loop, which expands in ⟨110⟩ directions as time proceeds. Obviously, misfit dislocations have already formed, which are identified by the dark, straight contrast lines. During the course of the experiment additional misfit dislocations are created by curved threading segments moving with a velocity of 11.2 μm/s through the area of observation leaving straight misfit segments behind, which is indicated in the third frame of Fig. 2 by a white arrow.

The considerable propagation of the dislocation loop segments seen in Fig. 2 seems to be influenced by the strain field of the He platelets lying underneath the SiGe/Si interface. The last picture of the video clearly shows bowing of the dislocation loop around the underlying platelets, which obviously hinder the movement of the dislocation. This pinning behavior due to the strain fields around the platelets is frequently observed in our experiments. The propagation speed of different loop segments is not uniform due to forces imposed by pinning, adjacent dislocations, and thin foil effects. The highest velocity in the example shown in Fig. 2 (indicated by a black arrow) is found to be 0.32 μm/s.

The role of He precipitates as internal dislocation sources is clearly proven by the *in situ* annealing experiment showing the emission of a dislocation loop from a He platelet (Fig. 2). Two processes proceeding on distinctly different time scales can be observed: (i) The formation of misfit dislocations. The fast movement of short bent dislocation segments, which cross the area of observation and leave straight lines behind, can be ascribed to gliding of threading dislocation segments. (ii) The growth of the dislocation loops. During the experiment the loops enlarge parallel to the sample surface. This implies that they must lie within a plane parallel to the interface or even within the interface, because otherwise one loop segment would have to encounter the surface of the TEM sample and, as a consequence, would vanish. However, this process was not observed in 60 different runs. Therefore, we conclude that the loops grow within (001) planes. As a consequence, climb processes must be involved for the movement observed. Since such a loop does not contribute to the relaxation of the SiGe layer, we can only speculate about the driving force of the climb process parallel to (100) planes being a supersaturation of point defects created by the implantation.

The velocity of the climb process is more than one order of magnitude slower than the glide of threading dislocations. This difference in velocity is significant since both values are measured at the same observation area in one experiment within a few seconds. However, the measured absolute speed values are strongly affected by every particular TEM experiment. For instance, the stress imposed on the dislocations in the thin TEM foil varies locally and differs from the situation in the bulk. In addition the local temperature at the area of observation may deviate from the nominal temperature due to the geometry of the TEM sample. Therefore, we abandon the idea of quantitatively analyzing the dislocation velocities.

These results clearly demonstrate that it is not sufficient to consider solely glide processes in the dislocation dynamics. Instead climb processes have to be taken into account, which may be facilitated by the implantation defects in the He implanted SiGe/Si. This is of particular importance regarding the efficiency of annihilation of threading dislocations which requires climb processes to allow the annihilation of dislocations on adjacent glide planes. Therefore, climb processes may be essential to achieve relaxed buffer layers with low threading dislocation densities.

In conclusion, spherical precipitates which result from a shape transformation of the helium platelets, coarsen at higher temperatures by movement and coalescence of entire bubbles. The dislocation sources in He implanted SiGe/Si heterostructures are unambiguously identified to be He filled platelets. Thus, in agreement with the model proposed by Trinkaus *et al.*[6] the relaxation of the SiGe films grown onto Si substrates is governed by dislocations expelled from the He precipitates. Dislocation glide mechanisms are observed to proceed by more than one order of magnitude faster than climb processes. In contrast to standard dislocation relaxation models, besides glide climb processes also have to be taken into account.

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