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On the nature of cross-hatch patterns on compositionally graded $Si_{1-x}Ge_x$ alloy layers

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The effect of strain relaxation on the surface morphology of compositionally graded $Si_{1-x}Ge_x$ layers grown at 550 °C has been investigated by a combination of transmission electron and atomic force microscopy. By annealing unrelaxed graded layers, we have found that shear displacements caused by dislocation glide roughen the surface dramatically. This effect is attributed to the formation of a network of dislocation clusters which give rise to the pronounced slip-band pattern on the surface of the graded layers. It is shown that the surface plastic displacements produced by such a network during growth of the graded layer contribute significantly to the formation of the cross-hatch patterns.

Compositional grading provides a unique possibility of growing relaxed low-dislocation-density $Si_{1-x}Ge_x$ alloy layers on a silicon substrate.[1,2] These structures have already received a great deal of attention due to their potential use for the fabrication of novel devices,[1,2] as well as for studies of bulk properties of unstrained $Si_{1-x}Ge_x$ alloys.[3] Although full relaxation and very low densities of threading dislocation have been achieved by the grading technique, the surface of the samples is not smooth and is characterized by a gradual undulation, referred to as a cross-hatch pattern.[2] Such surface features are deleterious to a number of electronic components and can interfere with device geometries defined by lithography.

Surface morphologies of the $Si_{1-x}Ge_x$ alloys grown on compositionally graded buffers at high temperatures (700–900 °C) have been studied recently by Fitzgerald *et al.*[2] and by Hsu *et al.*[4] They proposed that the cross-hatch patterns in these structures arise from spatially nonuniform growth rates caused by inhomogeneous strain fields associated with misfit dislocations. While this formation hypothesis can be conceptually accepted, it can be only a part of the "truth" about the formation of the cross-hatch patterns. It is well known that the introduction of dislocations in a crystal will produce steps on its surface, simply due to the plastic shear displacements of the surface. In the case where the lateral distribution of dislocations is uniform, no large-scale roughening of the surface occurs. If, however, the dislocations accumulate on the same or closely separated parallel slip planes, visible surface roughness seen as slip bands will develop. This effect has to our knowledge not been studied as a possible source of the development of the cross-hatch patterns in the Si/SiGe system. In the situation, where the strain relaxation occurs during film growth, the surface morphology is expected to arise from a combination of both dislocation-induced mechanical shears of the surface and from growth phenomena controlled by the surface kinetics and energetics. In order to separate pure mechanical effects we have grown strained graded $Si_{1-x}Ge_x$ films at 550 °C and then annealed them at 620 °C to introduce the misfit dislocations. Before and after annealing the samples were characterized by a combination of transmission electron microscopy (TEM) and atomic force microscopy (AFM). We have also studied an early stage (just at the beginning of strain relaxation) of the cross-hatch pattern formation during the epilayer growth.

The samples used in this study were grown by molecular beam epitaxy on 100 mm (100) Si wafers in a VC Semicon V80 system fitted with an Inficon Sentinel III flux controller. A silicon buffer layer of thickness 1 μm was first grown followed by the growth of the graded layer with a starting Ge content of about 2%. The total (Si plus Ge) growth rate of 5 Å/s was kept constant throughout the growth procedure. The TEM investigations were done with a Philips CM 20 microscope operating at 200 kV. The AFM studies were performed in air with a fully automated commercial instrument.[5] The z calibration of AFM scans was made by the self-imaging technique described in Ref. 6.

The display of composition vs thickness shown in Fig. 1 characterizes the samples used in this study. The final structure (sample E) consists of a 2 μm layer linearly graded to 30% of Ge and followed by a 2 μm uniform $Si_{0.7}Ge_{0.3}$ layer.

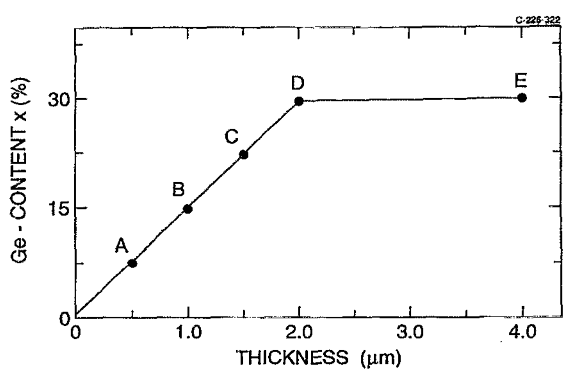

FIG. 1. Germanium content vs thickness in the $Si/Si_{1-x}Ge_x$ structures used in this study. The letters A, B, C, D, and E represent points at which the growth was stopped and the samples were removed from the growth chamber for investigations.

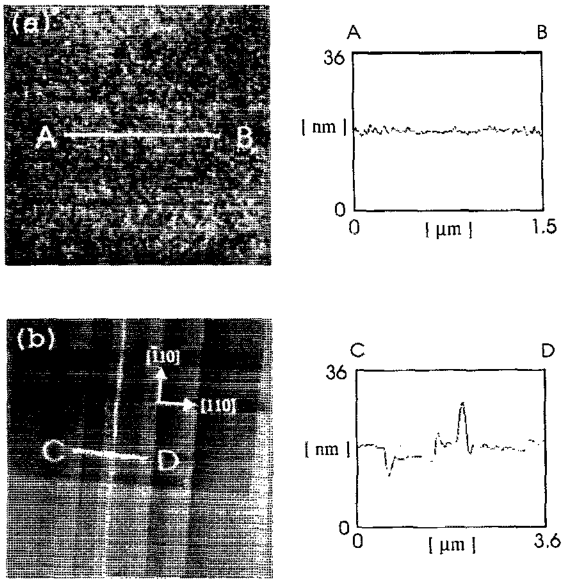

FIG. 2. AFM images and height profiles (along the displayed lines AB and CD) of the surface morphology of (a) sample B as-grown, and (b) sample B after annealing at 620 °C for 60 min. The scanned area and the z scale (black to white) are 2.6×2.6 μm^2 and 3.6 nm, respectively, in topography (a), and 13×13 μm^3 and 36 nm, respectively, in topography (b).

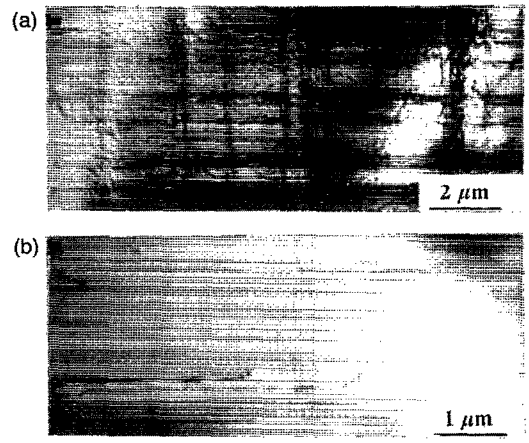

FIG. 3. Plan-view bright-field TEM micrographs of (a) sample B after annealing at 620 °C, (b) micrograph illustrating the beginning of strain relaxation during growth of the graded layer (sample C).

In order to obtain the unrelaxed graded layers and to study the development of the surface morphology during growth of the graded layer we also prepared intermediate structures by terminating the growth at the points A, B, C, and D. In samples A and B no relaxation was observed after growth and sample B was further annealed in a furnace at 620 °C to follow the main idea of the experiment. In sample C the beginning of the strain relaxation (an appearance of the first misfit dislocations) was revealed. An AFM study of sample B as-grown revealed no morphological features characteristic of the cross-hatch pattern. The surface is quite smooth [Fig. 2(a)] and is characterized by a root-mean-square (rms) roughness equal to ~0.58 nm. Dramatic changes of both the structure and the surface morphology were found in sample B after annealing. Misfit dislocations grouped in high-dislocation-density clusters are now seen by TEM [Fig. 3(a)]. These clusters are separated by regions of a low dislocation density and form a rectangular cluster network. Although different types of 60° dislocations were identified in a single cluster, dominant parts of the dislocations in each cluster form bunches of dislocations which have the same Burgers' vector and are distributed on parallel {111} glide planes throughout the graded layer. Some of the dislocations penetrate into the substrate. The formation of the network of dislocation clusters in sample B is accompanied by a dramatic roughening of the surface. In contrast to the unannealed sample, the surface morphology is composed of rectangular blocks with edges oriented along ⟨110⟩ directions. The blocks are displaced relative to each other in the direction parallel to the surface normal [Fig. 2(b)]. The rms roughness measured on an area of 13×13 μm^3 was found to be ~2.6 nm. The relation of this morphology to the underlying dislocation structure is obvious. The development of such a large-scale surface morphology during annealing cannot be attributed to any known surface reconstruction or elastic relaxation by surface rippling[7] process. On the other hand, the block-shaped morphological features observed after annealing correlate perfectly to the geometry of the network of dislocation clusters. The width of the flat bands in the AFM topography was found to be between ~200 and 3000 nm which corresponds to the distances between the dislocation clusters in the TEM pictures. Hence, the AFM topography shown in Fig. 2(b) is an image of the slip bands caused by the dislocation glide. Several conclusions can be drawn from a detailed analysis of this image.

(1) The relative vertical displacements of the rectangular regions of the surface were found to be in the range from ~1 to 5 nm. The step produced by the single dislocation at the surface is ~0.3 nm. Hence, a glide of about 4–20 of the 60° dislocations grouped in the same single or close-separated parallel glide planes is needed to provide the appearance of such displacements.

(2) The striking feature of Fig. 2(b) is the long range coherency of the bands of displaced material. Although these bands traverse each other, the coherency is conserved on areas of at least 30×30 μm^2 (maximum scanned area). This means that the dislocation cluster network is long range coherent.

(3) The AFM map and the height profile in Fig. 2(b) show that the sharp features seen as troughs and ridges are also formed by the dislocation glide during relaxation. The appearance of these features indicates the existence of closely spaced dislocation bunches with different preferential glide planes. These conclusions are all consistent with the results of the TEM observations.

Now let us consider what happens in the situation where the strain relaxation occurs during the growth of the graded layer (sample C). The beginning of the relaxation manifests itself as an appearance of dislocations shown in Fig. 3(b). We see that already at this early stage, the distribution of dislocations in the projection on the interface plane is highly nonuniform: the dislocations are grouped into clusters. A typical

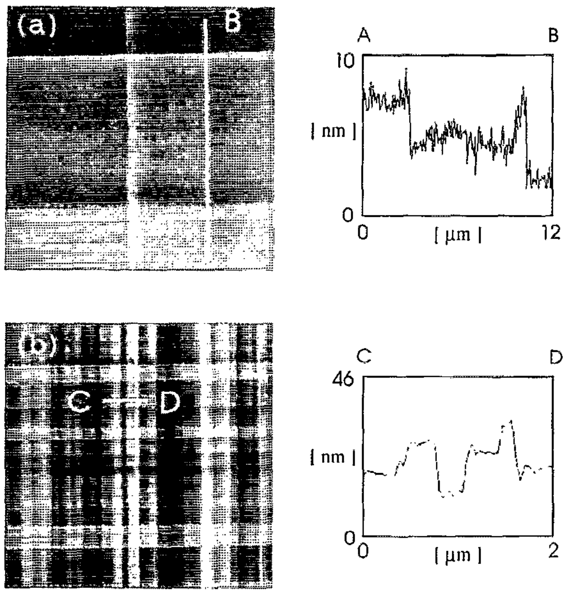

FIG. 4. AFM images and height profiles of the surface morphology of (a) sample C as-grown, and (b) sample C after annealing at 620 °C for 60 min. In both topographies the scanned area is 13×13 μm^2. The z scale is 10 nm in (a) and 46 nm in (b).

cluster is composed mainly of 60° dislocations with the same Burgers' vectors and these dislocations glide on parallel closely separated glide planes. The arrangements of this type, referred to as dislocation pileups, are usually attributed to an operation of regenerative dislocation sources such as the Frank–Read-type or spiral sources [see Ref. 8 and references herein]. The AFM investigation of sample C as-grown [Fig. 4(a)] shows that the dominating morphological features of the surface are almost the same as for the annealed sample B [Fig. 2(b)]. The surface is built with rectangular blocks displaced relative to each other in directions parallel to the macroscopical growth direction. This result is important as it represents an early stage of the formation of the cross-hatch pattern and, thus, indicates clearly that mechanical effects contribute significantly to the initial surface patterning during growth of the graded layer. In addition, we observe narrow regions of elevated material located at the edges of the rectangular blocks. The drop in the surface height from the top to the bottom of a ridge at the edges of some blocks reaches a value of about 6 nm [see the cross-section A-B in Fig. 4(a)]. This value is too high to be explained only by mechanical displacements produced by the dislocation pileups observed by TEM in sample C. That is why we propose that the formation of such features is caused mainly by preferential growth.

To investigate a potential effect of the full relaxation in sample C on its surface morphology we further annealed this sample at 620 °C. It is seen in Fig. 4(b) that the dislocation glide during the strain relaxation increases the roughness of the surface dramatically. The rms roughness is increased from ~1.4 nm before annealing to ~7.9 nm after annealing. The relative vertical displacement of some of the blocks reaches a value of ~14 nm [see the cross-section C-D in Fig. 4(b)]. By comparing the rms roughness of the annealed sample B (2.6 nm) to the rms roughness of the annealed sample C (7.9 nm), we can see that an increase in the thickness of the graded layer results in a larger mechanical roughening of the surface. It means that the number of dislocations gliding in parallel slip planes in each cluster increases with the thickness of the graded layer.

The cross-hatch pattern on the surface of the uniform cap layer (sample E) was found to be similar to the patterns observed by AFM in Ref. 4 and is characterized by a gradual undulation giving a rms roughness of ~5 nm. Although this value is comparable with the rms roughness produced by mechanical displacements in the graded layers, such an undulation-like morphology differs remarkably from the block-shaped patterns observed on the surface of the graded layers (sample C). This result shows that the surface morphology developing during the growth of the graded layer is modified significantly by the surface growth processes during the growth of the uniform cap layer.

Perhaps the most important result of this study has been its ability to educe a pure mechanical effect of dislocation introduction on the surface morphology of the graded $Si_{1-x}Ge_x$ films. By annealing of unrelaxed graded layers, we have found that shear plastic displacements caused by the dislocation glide process result in dramatic roughening of the surface. The rms roughness produced by these mechanical displacements is shown to be comparable with the roughness of the cross-hatch patterns seen on the surface of a uniform cap layer. This effect is directly attributed to the formation of the pronounced slip-band surface morphology arising from the network of dislocation clusters. It is demonstrated that, at low growth temperatures (~500 °C), the formation of the slip bands during the growth of the graded layer plays a significant role in the development of the cross-hatch patterns.

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