Characterization of dislocations in germanium substrates induced by mechanical stress

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Dislocations are observed in germanium crystals (9° off axis toward the [011] direction) that have undergone plastic deformation. Optical microscopy reveals that the substrates exhibit a crosshatch pattern, consisting of ridges and trenches that extend in the [011] and [011] directions. Further characterization of these features with scanning tunneling microscopy shows that they consist of bands of steps. These bands are created when a group of dislocations emerge onto the crystal surface from the bulk. The dislocations are determined to be type $(a/2)\langle 011\rangle$. © *1998 American Institute of Physics.* [S0003-6951(98)04334-4]

Germanium is currently used as a substrate for fabricating compound semiconductor solar cells used in satellite power stations. Germanium is the substrate of choice, because it can be made much thinner and lighter than gallium arsenide substrates, while at the same time, providing a suitable template for GaAs epitaxy. At room temperature, germanium is a brittle material, but as one heats the substrate, it becomes increasingly ductile.[1] When stress is applied to a Ge wafer held at elevated temperature, it will undergo plastic deformation, and eventually break. Plastic deformation occurs through the generation and propagation of dislocations.

Plastic deformation results from the slip, or glide, of a crystallographic plane along a well-defined direction. For the diamond crystal structure, slip occurs between adjacent (111) planes.[2] When the dislocation emerges at the surface, it generates a step whose structure is determined by the Burger vector of the dislocation. Therefore, one can study the formation and propagation of dislocations in a crystal through analysis of the step structure on the surface. Shown in Fig. 1 are the ball-and-stick models of vicinal Ge(100) surface with and without dislocations.[3] Figure 1(b) shows a dislocation of type $(a/2)[011]$ that results in a step two atomic layers in height. Figure 1(c) shows the type $(a/2)[211]$ that results in a step one atomic layer in height. These types of steps have been seen in previous studies. For example, glide steps of two atomic layers in height in the $\langle 011\rangle$ directions have been reported on Si(111) wafers that have undergone plastic deformation.[4]

In this letter, we report on the formation of dislocations in Ge (100) crystals that are induced by mechanical stress. These dislocations are examined by optical and scanning tunneling microscopy. Our results indicate that dislocations of type $(a/2)\langle 011\rangle$ form in Ge (100) substrates that are oriented 9° off axis toward the [011] direction. In addition, we observed a ridgelike structure on the arsenic-passivated Ge (100) surface that is oriented 13° away from the [011] and [011] directions. This structure probably arises from additional stress induced by the smaller size of the adsorbed As atoms relative to the Ge substrate atoms.

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The experiments were carried out in an ultrahigh vacuum (UHV) system combined with a metalorganic chemical vapor deposition (MOCVD) reactor, which is described in detail elsewhere.[5] Vicinal Ge (100) substrates were obtained from Tecstar, and sliced into rectangles 1 cm wide by 2 cm long. These samples were rinsed in acetone, methanol, and de-ionized water, then sequentially dipped three times in baths of concentrated HF, de-ionized water, and concentrated H_2O_2.[6] The samples were held in the HF and H_2O_2 baths for 30 s, whereas they were rinsed with water for 2 min. After cleaning, each Ge rectangle was placed on a holder made from thin molybdenum foil that was 120 μm thick by 1 cm wide by 5 cm long. The crystal was

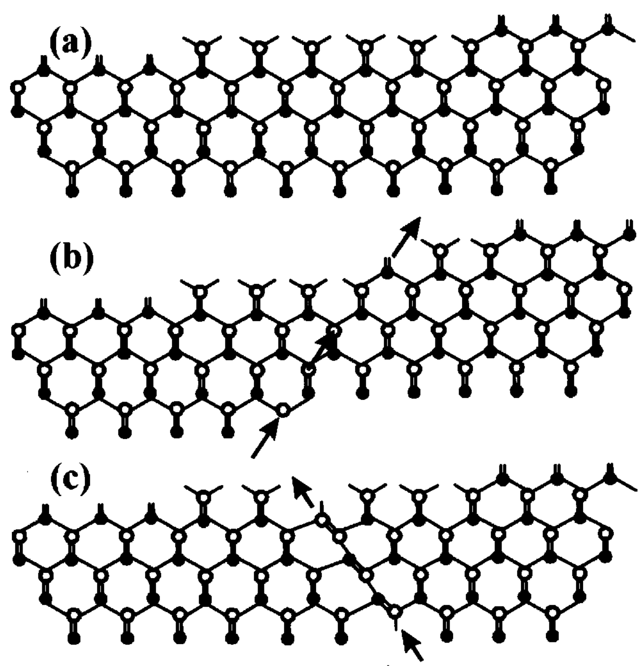

FIG. 1. Ball-and-stick models of the unreconstructed Ge (100) surface, as viewed from the [011] direction: (a) one domain with three terraces separated by steps one atomic layer in height; (b) two domains with five terraces created by an $(a/2)\langle 011\rangle$ dislocation; and (c) two domains with five terraces created by an $(a/2)\langle 211\rangle$ dislocation. The open and filled circles represent Ge atoms with their tetrahedral bonds oriented parallel and perpendicular to the plane of the paper, respectively.

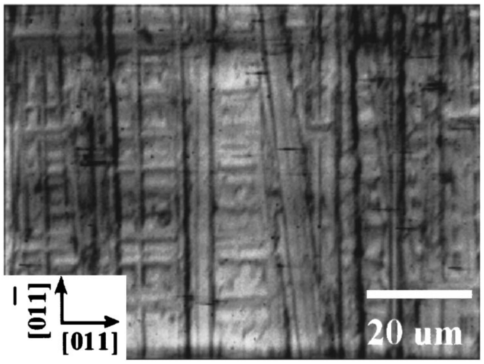

FIG. 2. Optical micrograph of the surface of a mechanically stressed Ge (100) crystal (image size = 90×70 μm^2).

held in place by two metal flaps folded over its long edges, while the short edges remained free. Upon heating, the Mo foil bent slightly along the long axis, thereby applying stress to the germanium crystal.

After cleaning, the samples were loaded into the UHV system and transferred to the MOCVD reactor through an interface chamber. They were annealed in 2.0 Torr tertiarybutyarsine (TBAs) and 97.0 Torr hydrogen (H_2) at 650 °C for 30 min to passivate the germanium surface with arsenic. The flow rates of TBAs an H_2 were controlled at 41 and 600 sccm, respectively. After the annealing step, the samples were cooled to 100 °C in flowing TBAs and H_2, then cooled further in flowing H_2 to 30 °C, and finally transported to the UHV system for storage.

The As-passivated Ge (100) substrates were next subjected to annealing cycles in either ultrahigh vacuum, or in 99.0 Torr of H_2 in the MOCVD reactor. The temperatures the samples were heated to ranged from 300 to 700 °C. After this step, the crystals were cooled to 30 °C and transferred to the scanning tunneling micrograph (STM) chamber for imaging. The STM was operated in constant current mode with a current of 0.5–1.0 nA and bias voltages of −1.0 or +1.2 V. After these tests, the sample surfaces were also analyzed *ex situ* by optical microscopy.

An optical micrograph of a germanium wafer that has been mechanically stressed is shown in Fig. 2. This sample was subjected to 15 cycles of heating to 650 °C and cooling to 30 °C in the UHV system. A crosshatch pattern is observed, with ridges and trenches extending in [011] and [011] directions. It is interesting to note that the exact same pattern is seen for strained SiGe/Si alloys surfaces.[7,8] These patterns result from the plastic deformation of the Ge and SiGe/Si substrates during high-temperature annealing. In order to study the development of the surface morphology as a function of annealing temperature, we have subjected a series of Ge wafers to heating cycles ranging from 500 to 700 °C. The crosshatch patterns always appear when the crystals are heated to 650 °C and above.

The rides and trenches on the stressed Ge (100) crystals have been examined by scanning tunneling microscopy. On samples without the crosshatch patterns, the STM images do not exhibit any unusual morphological features. The surfaces are smooth, consisting of uniform arrays of (2×1) reconstructed terraces, separated by step two atomic layers in height. The terraces are on average 20 Å wide. Dramatic changes in surface morphology are found on samples that show the crosshatch patterns. Presented in Fig. 3 is a large-scale STM image of the surface of a mechanically stressed Ge (100) crystal. One sees light and dark lines running across the image in the [011] and [011] directions. The white arrows in the image indicate two lines that are separated by 0.8 μm. Examination of many STM pictures reveals an average separation of 2.0 μm. Each line delineates a slab of the surface that is displaced up or down relative to its neighbors. As will be discussed below, the lines are comprised of band of steps formed by the emergence of a group of closely spaced dislocations on the surface.

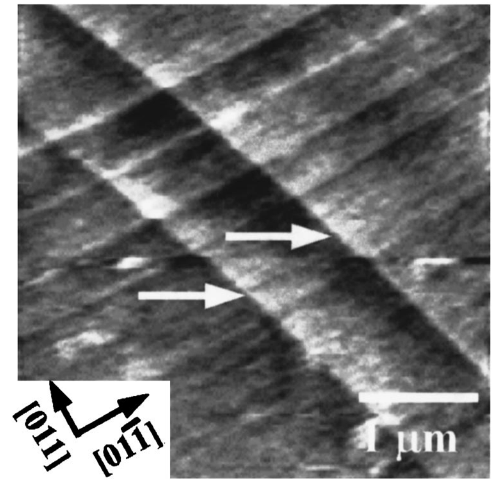

FIG. 3. Scanning tunneling micrograph of the surface of a mechanically stressed Ge (100) crystal (image size = 4×4 μm^2).

Presented in Fig. 4 is an STM image of the As-terminated Ge (100) surface after the substrate had been mechanically stressed. The x-ray photoelectron spectroscopy (XPS) As/Ge *2p* ratio equal 5.0%, corresponding to an As coverage of about 0.11 monolayers. This sample was subjected to 15 cycles of heating to 650 °C and cooling to 30 °C in the UHV system. This image shows a series of lines running in the [011] and [011] directions, as is also seen on the clean Ge (100) surface. However, in addition to these features, another series of lines are observed that are displaced 13° away from the [011] axis. These lines are coupled to the As coverage, since they disappear upon desorption of the

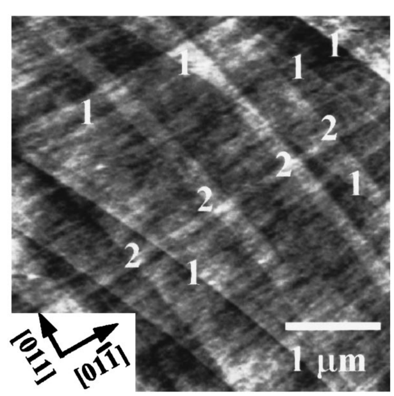

FIG. 4. Scanning tunneling micrograph of the As-terminated Ge (100) surface. Dislocation lines parallel to the ⟨011⟩ directions are labeled ''2,'' while dislocation lines offset 13° with respect to the former are labeled ''1'' (image size = 4×4 μm^2).

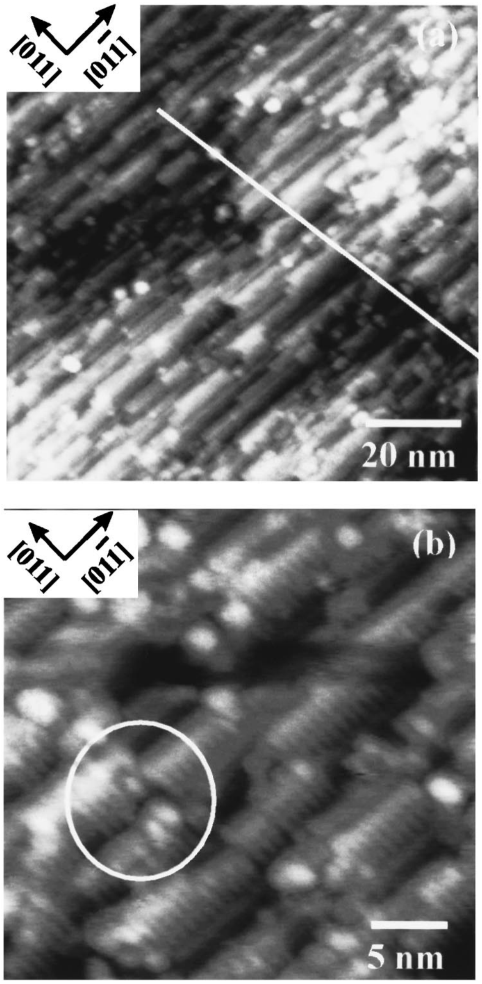

FIG. 5. Scanning tunneling micrographs of the As-terminated Ge (100) surface, providing close-up views of a dislocation line extending along the [011] direction [the white line in (a)]. The white circle in (b) shows where three terraces merge at a single dislocation. The image sizes are 1000 $\times$1000 Å^2 in (a) and 320$\times$320 Å^2 in (b).

arsenic. The origin of the 13°-offset lines is unclear at this time. We speculate that they may be related to the stress generated by terminating the Ge surface with a layer of smaller arsenic atoms.

In Fig. 5, atomic resolution STM images are shown of one of the lines seen on the As-terminated Ge (100) surface. The white stripe in Fig. 5(a) is parallel to the [011] direction, and marks a location on the surface where a dense band of [011]-facing steps are found. These steps cut off the As-terminated terraces. The band of steps is randomly distributed over a region of the surface about 100 Å wide. This band is easily recognized by the strong change in contrast of the image from light gray on the right-hand side of the white stripe to black on the left-hand side of the white stripe. The contrast change corresponds to a vertical height of about 16 Å, and it is responsible for the lines seen in the large-scale STM images (refer to Figs. 3 and 4).

In Fig. 5(b), an atomic-resolution image is shown of a region within the band of steps. The terraces are covered with arsenic dimers, and produce short rows, about 8 Å apart, that run parallel to the [011] direction (i.e., across the terraces). The reconstruction on these terraces is obviously (2×1), which is also confirmed by low-energy electron diffraction. Within the white circle in Fig. 5(b), three terraces merge. Each terrace exhibits the same (2×1) structure, indicating that the step down from one terrace to the next is two atomic layers in height, equal to 2.8 ± 0.1 Å. According to the models presented in Fig. 1, a step change of two atomic layers corresponds to an $(a/2)\langle011\rangle$ dislocation. The band of steps, yielding a height variation ranging from 3.0 to 22.0 Å, is due to a group of $(a/2)\langle011\rangle$ dislocations that have emerged onto the surface.

In summary, we observe crosshatch patterns on plastically deformed Ge (100) crystals. These crosshatch patterns are due to the groups of $(a/2)\langle011\rangle$ dislocations. Scanning tunneling microscopy reveals that these dislocations generate a band of steps, covering about 100 Å of the surface, and producing a height change of between 3 and 16 Å. On As-terminated Ge (100), new step bands are observed that are oriented 13° away from the $\langle110\rangle$ directions.

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