

Formation of step structures by As deposition on a double-domain Si(001) substrate

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We report the rich variation in the evolution of step structures of vicinal Si(001) upon As deposition, studied by scanning tunneling microscopy. Clean 1° vicinal Si(001) exhibits a typical double-domain surface that consists of single-monolayer-height steps called S_A and S_B . On the other hand, As-deposited surfaces show drastic changes in the step structures depending on the substrate temperature during the deposition and the As coverage: At relatively low temperatures (500°C), the surface becomes nearly a single domain by the deposition of a half monolayer and two-dimensional islands appear after more As is deposited. At medium temperatures (600°C), however, one-monolayer As deposition makes the surface nearly a single domain, consisting of double-monolayer-height steps called D_B . At high temperatures (700°C), a double-domain surface is formed containing triple-monolayer-height steps where the dimer rows in the upper terrace are perpendicular to the step edge. These various surface step structures are explained by kinetic effects of atomic movement during deposition and desorption and by the energetically stable shapes of Si(001)-As in equilibrium.

I. INTRODUCTION

The surface structures of the As-covered Si(001) surface have been intensively studied because of its importance in the heteroepitaxy of III-V semiconductors, especially GaAs, on Si. Recently, this surface has also become of interest to the surfactant-mediated epitaxial growth of Ge on Si.¹ In epitaxial growth the morphology of the starting surface, and changes to it during the initial stages of growth, plays a crucial role in determining the growth process itself. For instance, antiphase disorder in the epitaxial film, one of the major problems in GaAs epitaxial growth on Si(001), depends on the step structure of substrates.² In surfactant-mediated epitaxial growth, not only the growth mode of the epitaxial layers but also interfacial mixing related to segregation of the As layer have been the subject of recent studies.^{3,4}

The step structures of Si(001) are well understood as the result of many experimental⁵⁻⁷ and theoretical⁸⁻¹¹ studies. Si(001) surfaces with small misorientations show a double-domain structure of 2×1 and 1×2 reconstructions separated by single-layer steps called S_A and S_B . On the other hand, surfaces with large misorientations show a single domain having double-layer steps labeled D_B . The step notation S_A , S_B , D_A , and D_B was defined by Chadi⁹ to represent (S) single- or (D) double-layer height and the direction of dimer rows in the neighboring upper terrace running (A) parallel or (B) normal to the step edges.

Several groups¹²⁻¹⁵ had studied As adsorption on Si(001) substrates. Recently, it was found that As not only grows on top of the Si substrate but also sometimes displaces the top Si layer of the substrate. Becker, Klitsner, and Vickers¹² investigated this phenomenon by scanning tunneling microscopy (STM) using a vicinal Si(001) substrate on which the single-domain surface is

constructed by an array of the double-layer steps (D_B). In their investigation, they observed As-covered single-domain surfaces constructed by D_B steps, which means that substitution of a Si dimer layer by an As dimer layer was taking place. Alerhand *et al.*¹³ explained this by the difference of total energy on single-domain surfaces constructed by D_A and D_B steps based on their calculations.

Furthermore, Bringans, Biegelsen, and Swartz¹⁴ studied the domain-rotation induced by As adsorption on single-domain Si(001) substrates carefully. Results of their experiments showed that As deposition on heated bare Si substrates results in the displacive adsorption, while the As deposition at room temperature and the succeeding annealing results in on-top adsorption. They tried to explain this by the exchange of Si dimers at the step edge by As dimers during heating. Low-energy electron microscopy (LEEM) examination carried out by Tromp and Reuter,¹⁵ however, revealed that the exchanges take place on the terrace of double-domain substrates on nonvicinal (001) samples.

Thus, the consensus in the previous studies are that As adsorption sometimes takes place as the substitution of Si dimer layers by As dimer layers. In the case of the highly stepped surface, the As-covered single domain constructed by D_A steps are energetically metastable compared with that constructed by D_B steps. As atoms displace Si dimers on the terrace.

Due to its application to the GaAs growth on Si(001) surface, most of the studies about the effect of As on step structures have concentrated on the substrate with a large misorientation, i.e., on the single-domain surface. On the other hand, there have been few examinations of step structures on the substrate with a small misorientation. Therefore, we had little knowledge about the step structure formed by As deposition on double-domain substrates, which might be very important in the mi-

croscopic investigation of surfactant-mediated epitaxial growth. In this paper, we present a STM investigation of the formation of step structures by As adsorption on Si(001) double-domain substrates. The STM observations reveal the variations in step structure that depend on substrate temperature during As deposition. Additional annealing after As deposition present clues to the mechanism of the evolution of these step structures. As a result, the various surface step structures are found to be explained by the kinetics of deposition and desorption and the energetics of domain formation, depending on the substrate temperature.

II. EXPERIMENT

The instrumentation used in this study is an ultrahigh vacuum (UHV)-STM system combined with a molecular-beam-epitaxy (MBE) chamber. Silicon samples were cut from (001) wafers (Shin-Etsu Handotai Co, CZ boron doped 0.01–0.02 Ω cm) with a miscut angle of 1° toward the $\langle 110 \rangle$ direction. After rinsing with alcohol and acetone, the samples were cleaned by annealing above 1200 $^\circ\text{C}$ in UHV. Heating of the sample was done by passing direct current through it. Arsenic was deposited using a Knudsen cell and the flux was estimated via a flux gauge mounted in the sample position. Typical As pressure in the flux used in this study was about 2×10^{-8} Torr and MBE background pressure in the chamber was kept below 4×10^{-10} Torr. Away from the As flux after annealing above 1200 $^\circ\text{C}$ for cleaning, the sample was cooled down to 500, 600, or 700 $^\circ\text{C}$. Then the sample was exposed to the As flux by transferring to the front of the As cell. As soon as deposition was stopped by transferring the sample out of the As flux again, the sample was radiatively cooled down to room temperature.

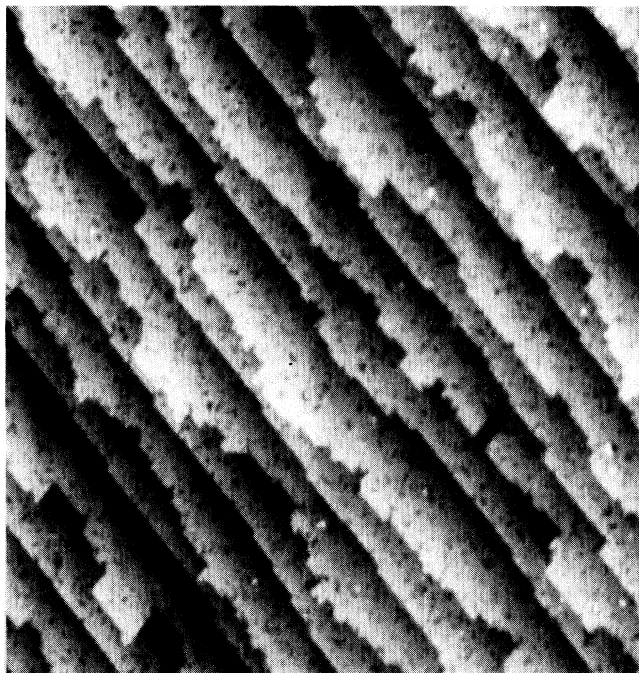
All STM observations were carried out at room temperature. All images shown in this report were obtained with a negative sample bias (i.e., tunneling from filled surface states). The As coverage on the surface was measured after STM observations by Auger electron spectroscopy calibrated by regarding the saturated As coverage on Si(001) as 1 ML.

III. RESULTS

A. Clean 1° miscut Si(001)

First, the step structure of the Si(001) clean surface tilted about 1° toward the $\langle 110 \rangle$ direction was checked, Fig. 1. The normal double-domain surface is clearly evident, consisting of 1×2 and 2×1 domains divided by the single-layer steps called S_A and S_B . The area covered by one domain is obviously larger than that of the other domain, in agreement with a diffraction study.⁷ The

(a)



(b)

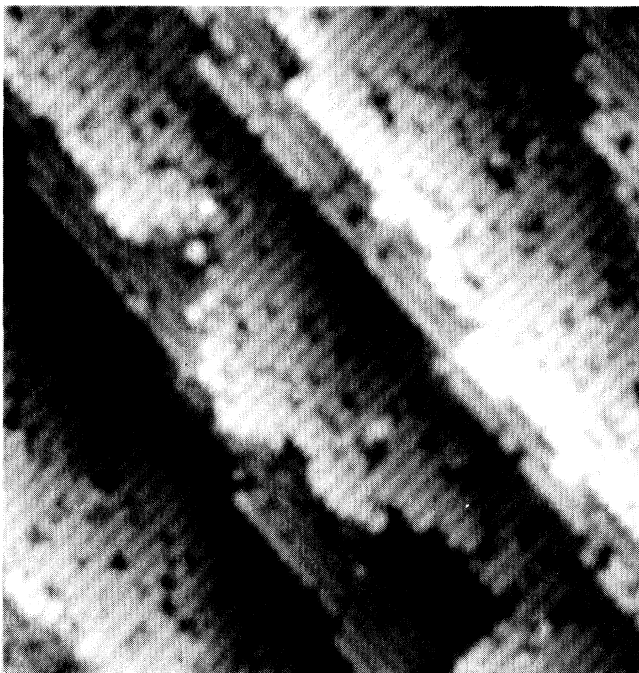


FIG. 1. STM images of a clean vicinal Si(001) surface tilted 1° towards $[110]$. The scan areas are approximately (a) $1000 \times 1000 \text{ \AA}^2$ and (b) $300 \times 300 \text{ \AA}^2$.

typical proportion of the minor domain where the dimer rows run parallel to the step edge is $40 \pm 5\%$. On these samples I have observed neither large fluctuations in the arrangement of steps nor the obviously wavy steps that have been observed by LEEM on surfaces with extremely small misorientations.¹⁵

B. As deposition at 500 °C

Figure 2 shows a series of As depositions onto Si(001) substrates held at 500 °C. The As coverages of these surfaces were about (a) 0.6 and (b) 1.0 ML, respectively. Islands form for surface of As coverage above half monolayer. The islands elongate in the direction normal to the dimer rows on the substrate. Below the half monolayer, S_B steps gradually become closer to the neighboring lower S_A steps. At the half monolayer, as the result, the surface becomes nearly a single domain. This step evolution indicates that S_B steps are much more reactive than S_A steps. In addition, I also observed bias dependence of the STM image suggesting that displacive adsorption is taking place in the terrace, which is in agreement with the report by Tromp *et al.*³: At larger bias only regular arrangements of dimer rows were visible

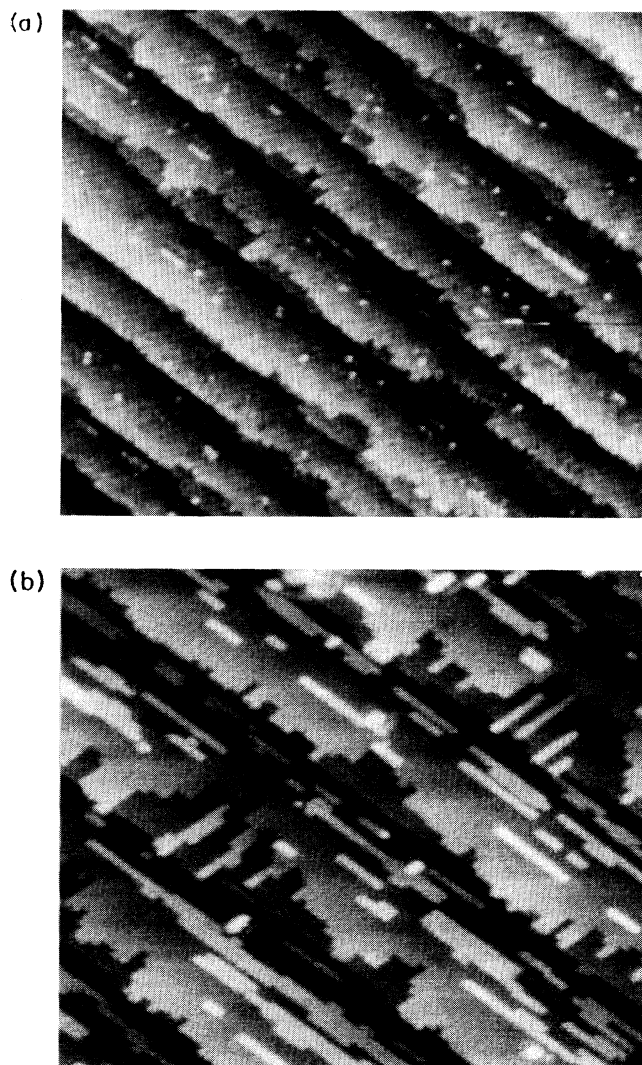


FIG. 2. STM images of Si(001)-As surfaces. These surfaces were exposed to As flux at 500 °C. As coverages were (a) 0.6 ML and (b) 1.0 ML. The scan areas are approximately (a) $1000 \times 1000 \text{ \AA}^2$ and (b) $500 \times 500 \text{ \AA}^2$.

in the terrace, while at smaller bias many corrugations were observed located irregularly in the terrace, which suggests coexistence of different chemical species in the terrace.

C. As deposition at 600 °C

Figure 3 shows the surface after As deposition at 600 °C. The samples shown in Figs. 3(a) and 3(b) were exposed to the As flux for 50 s and for 3 min, respectively; however, both resulted in a saturated As coverage of 1 ML. As seen in the micrograph, a 3 min exposure made a nearly single-domain surface. In fact, the area of the minor domain, in which the As dimer rows run parallel to the step edge, is only $10 \pm 5\%$ [Fig. 3(b)]. In most of the surface, double-layer steps form by pairing neighboring S_A and S_B steps. As with the single-domain surface of clean vicinal Si(001), such double-layer steps

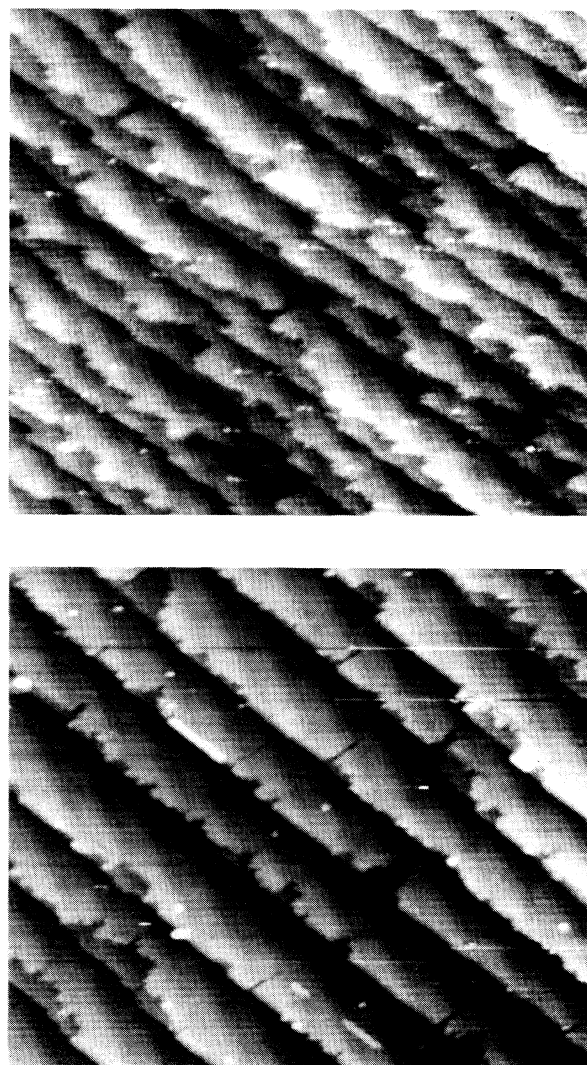


FIG. 3. Si(001) surfaces covered by 1-ML As. These surfaces were exposed to As flux at 600 °C for (a) 50 s and (b) 180 s. The scan area is $1000 \times 1000 \text{ \AA}^2$.

are always D_B , in which the dimer rows on the upper terrace run perpendicular to the step edge.

On the other hand, after exposure for 50 s, the surface remains double domain rather than single domain. In fact, the area of the minor domain in Fig. 3(a) is still $30 \pm 5\%$. This means that the surface changes to single domain during overexposure, after enough As has been deposited to saturate the surface. I have also observed a surface exposed for 20 min, which exhibits the same nearly single-domain surface. Thus the single-domain surface can be regarded as the equilibrium configuration of Si(001):As.

D. As deposition at 700 °C

Figure 4 shows the surface after As deposition at 700 °C. The step structures of this surface are quite dif-

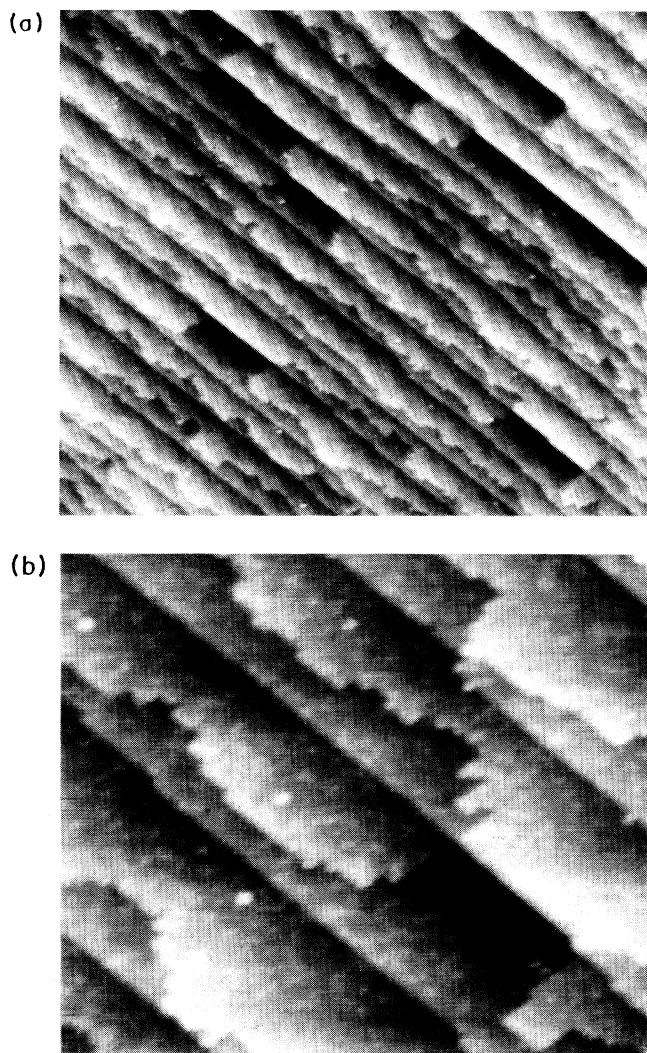


FIG. 4. Large and small area STM images of a Si(001)-As surface. The surface was exposed to As flux at 700 °C. As coverage on the surface was 0.7 ML. The scan areas are approximately (a) $2000 \times 2000 \text{ \AA}^2$ and (b) $300 \times 300 \text{ \AA}^2$.

ferent from those shown above. In this image, triple-layer steps are visible. In Fig. 4 the triple-layer steps are displayed as steps with distinctly high contrast, e.g., steps in the upper right-hand side of Fig. 4(a) and a step in the lower right-hand side of Fig. 4(b). Those steps were observed equally everywhere on this surface. In the neighboring upper terrace of this triple-layer step, dimer rows always run normal to the step edge. Thus, extending the usual step notation, we can name this the T_B step. These T_B steps are fairly straight, suggesting that the formation of kinks is energetically rather unfavorable. The coverage of As on this surface is about 0.7 ML even after sufficiently long deposition. The coverage of 0.7 ML means that the saturation coverage of 1 ML can never be achieved by deposition at this temperature due to simultaneous evaporation, as confirmed by post-deposition annealing described below.

E. Annealing after the deposition

Figure 5 shows the effect of annealing after 1-ML deposition. Figure 5(a) shows the surface annealed at 500 °C for 10 min after deposition at the same temperature. It is quite similar to the unannealed surface [Fig. 2(a)]. The coverage of As on this surface stayed at 1 ML. These results suggest that the rearrangement of atoms during such an anneal is negligible.

Figure 5(b) shows a surface annealed at 600 °C for 5 min after 1-ML deposition at 500 °C. On this surface the number of two-dimensional (2D) islands are much decreased and their shapes are less anisotropic. There still remains nearly 1-ML of As on this surface. This means that the topographic change was driven by the rearrangement of atoms diffusing on the surface. Thus, during As deposition at 600 °C the surface gradually approaches its equilibrium shape due to frequent rearrangement of atoms. The shapes of the islands suggest that the difference in formation energies of the two types of steps is not so large as would be inferred from the extreme shape anisotropy of the islands just after deposition [Fig. 2(b)]. I believe that the latter results from the large difference in sticking reactivities at the edges of the islands.

Figure 5(c) shows a surface annealed at 700 °C for 5 min after deposition at the same temperature. The coverage of As immediately before annealing is 0.7 ML, whereas the coverage on this surface decreased into 0.2 ML. This indicates the evaporation of As atoms during annealing at 700 °C. In this image there remain no triple layer steps, indicating that they are transient structures during evaporation. These T_B steps also gradually appeared upon annealing at 600 °C for 60 min after 1-ML deposition at the same temperature [Fig. 5(d)]. During this anneal the As coverage gradually decreased as well, to 0.3 ML.

IV. DISCUSSION

In this study, I examined the evolution of step configuration by As adsorption on the double-domain substrate.

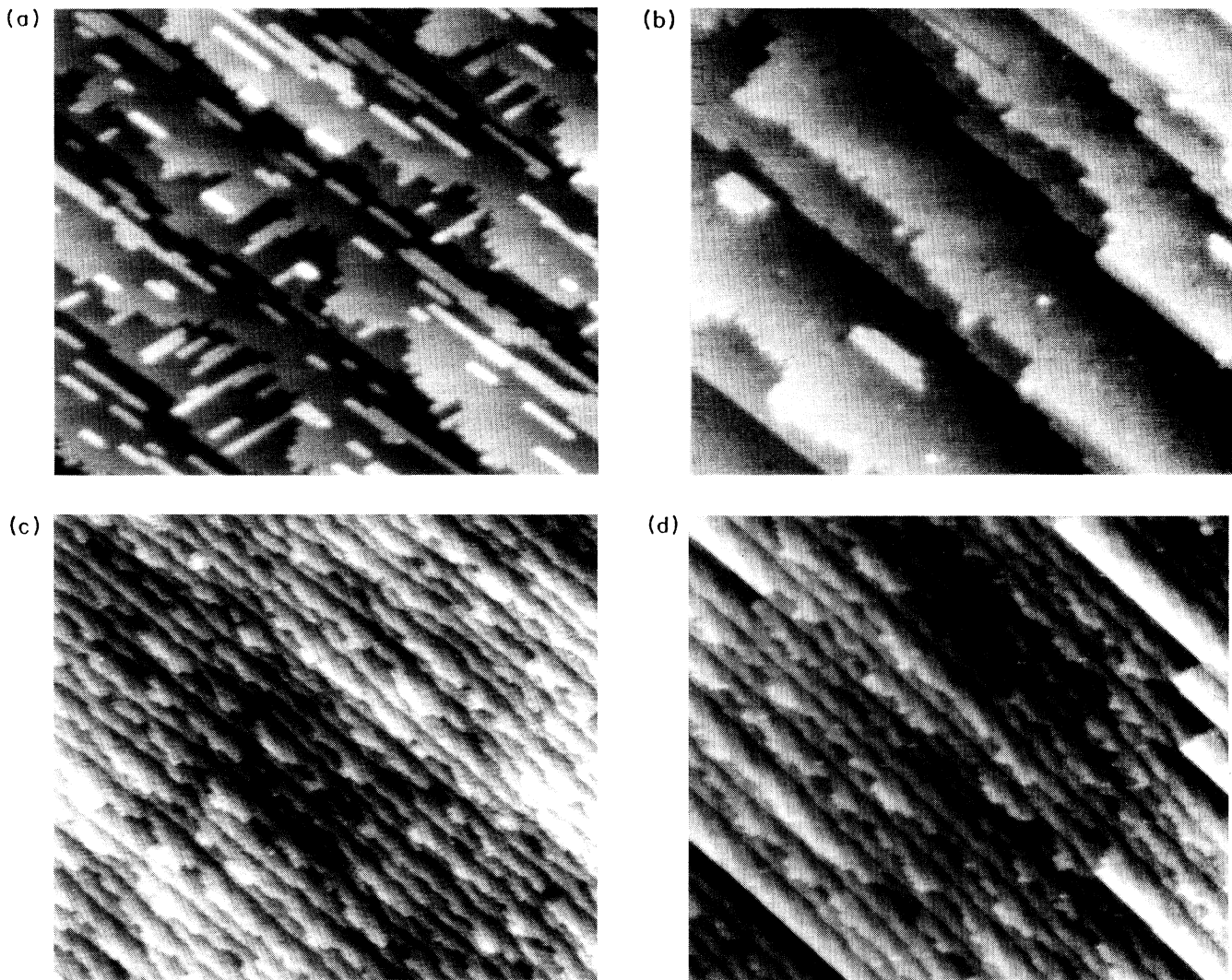


FIG. 5. Effect of annealing after the deposition. (a) Surface annealed at 500 °C for 10 min after 1-ML deposition at 500 °C. (b) Surface annealed at 600 °C for 5 min after 1-ML deposition at 500 °C. (c) Surface annealed at 700 °C for 5 min after 0.7-ML deposition at 700 °C. (d) Surface annealed at 600 °C for 60 min after 1-ML deposition at 500 °C. The scan areas are approximately (a)–(c) $1000 \times 1000 \text{ \AA}^2$ and (d) $2000 \times 2000 \text{ \AA}^2$.

As a result, I found phenomena that, to my knowledge, have been unknown so far, e.g., formation of a single-domain substrate from a double-domain substrate. In the following part we will discuss the mechanisms of formation of those surface configurations.

The evolution of the surface configuration during deposition at 500 °C can be roughly explained by a kinetic picture. At first, As atoms exchange with Si atoms on the terrace. Those Si adatoms diffuse on the terrace and stick to step edges. According to STM observations of the denuded zone of Si growth on Si(001), the S_B step was found to be a good sink for the Si adatom, whereas S_A was not.¹⁶ Judged from the shape anisotropy of 2D Si islands, the sticking coefficient at the S_A step is much smaller than at S_B .¹⁷ Therefore, most Si adatoms, including those once approached to S_A steps, stick mainly

at S_B steps. When a one-half monolayer of As is deposited, many D_B steps are formed as the result of coalescence of S_A and S_B steps. Similar single-domain surfaces are formed by one-half monolayer deposition in the growth of Si on a Si(001) substrate¹⁸ as well.

We observed the formation of 2D islands by As deposition above a one-half monolayer. There are two possible driving forces for the formation of 2D islands. One is the lowering of reactivity at the step edge. In the growth of Si on a Si(001) substrate, D_B steps are less reactive for the Si adatoms and 2D islands easily develop on the substrate constructed by D_B steps.¹⁹ In As adsorption the D_B step could be less reactive as well. The other driving force is a local dimer exchange of the Si adatom with the surface As dimer which was proposed by Tromp and Reuter¹⁵ for the growth of Ge on As-covered Si(001). This phe-

nomenon was recently calculated by Yu and Oshiyama²⁰ and found that Ge adatoms exchange with As dimers on the terrace. As well as the case of exchange of Ge on the As-covered surface, Si adatoms could exchange with As dimers. Thus as the As coverage increased, the exchange could take place in the terrace and that would form a 2D island like Ge adsorption.

During deposition at 600 °C, we observed the formation of a single domain as the equilibrium surface morphology. On the originally single-domain Si(001) surfaces, previous researchers found the formation of the same type of single-domain surface as a result of the displacing of the top layer. Becker *et al.*¹² ascribed the stability of this surface constructed by D_B steps to the parallel strain induced by dimers with a strain field inherent to the vicinal Si(001) surface. First-principles calculations¹³ on such a highly stepped surface revealed that the displaced surface is stabilized by the relaxation of dimer stress at the D_B step, which is in agreement with the explanation by Becker *et al.* In this study we found the formation of a single domain on a substrate with the small misorientation of 1° where the bare Si surface had been double domain. To discuss the transformation from double domain into single domain, the energetics of surface constructed by single-layer steps S_A and S_B should be also compared with that constructed by D_B steps.

Generally, for a clean Si(001) surface with a small mis-cut, the double-domain surface is the most energetically stable. It is known that the single domain is more energetically favorable only in surfaces with a large mis-cut angle above 4°. These stabilities are explained by a balance between strain relaxation energies and step formation energies.¹⁰ The strain comes from the anisotropic stress caused by dimer reconstruction.²¹ The stress is tensile along the dimer bond direction because the dimer atoms move closer to form a dimer bond, while the stress is compressive along the dimer row direction. The compressive stress is, I believe, driven by the dimer π bond. Due to the π bond, which would have p -orbital-like character, the hybridization could be between sp^2 and sp^3 , which makes the bond angle larger than that of sp^3 in the bulk. The anisotropic strain induced by those stresses can be relaxed by the coexistence of two orthogonal domains of 2×1 and 1×2 . Thus the strain relaxation energy favors double domain. On the other hand, step formation energies favor the single domain because the D_B step has a lower formation energy than the combination of S_A and S_B steps. Of these two contributions the strain relaxation energy is dominant on the surface with small misorientation, while the step formation energy is dominant on the highly stepped surface.

The present experimental results indicate that the single domain is energetically more favorable for the Si(001):As surface even with a small mis-cut angle of 1°. First-principles calculations²¹ revealed that surface stress induced by As dimers on Si(001) is qualitatively different from that by Si dimers. To stabilize the dangling bonds by the s^2 configuration of the lone pair, the As atom prefers to hybridize with subtetrahedral bond angles. This leads to tensile stresses in both directions, which is nearly isotropic according to the calculation results.²¹

The formation of two orthogonal domains does not help relieve the isotropic stress. The trends in step formation energies on Si(001):As are apparently similar to those on Si(001) because the As-covered Si(001) vicinal surfaces display a single domain. Thus the transformation of the stable surface configuration from double domain to single domain by As deposition results from the balance between the strain energies and the step formation energies.

STM observations of the formation of multiple layer steps on Si(001)-As had been reported by Becker *et al.*¹² on a highly stepped substrate. In that report they just said “steps with heights of up to five layers” can be observed in the case of As-saturated deposition on 4° vicinal at 700 °C. In our experiments, steps with heights of up to three layers were formed and every triple layer step is T_B . This difference could be the smaller vicinal angle than that used in their experiment. We believe that the step configuration with the height of four layers found by Pukite and Cohen²² should be a combination of these T_B and S_A steps. A predominant formation of one type of triple-layer step T_B among two would probably cause such four times periodicity. As described in the report by Becker *et al.*, multiple-layer steps can be stable on As covered surfaces owing to the stability of the 1×1 reconstruction of Si(111):As which is locally formed at the step edge of the (001) surface. In order to explain the predominant formation of T_B steps, we have to further discuss the mechanism of the formation of steps.

A possible reason for the formation of T_B steps could be different kinetic pathways for the composition and decomposition of steps during evaporation. Assuming there are no simultaneous multilayers stripping off, i.e., only a monolayer can be stripped off at the same time, during evaporation, the kinetic pathways in the composition and decomposition of triple-layer steps are different. The T_B step can be combined from the upper D_B step and the lower S_B step by stripping off the terrace between their steps. The desorption reactivity at the S_B step could be higher by analogy with the high sticking probability at the S_B step. As seen in the formation of the single-domain, the D_B step is frequently formed. Thus, this combination pathway into the T_B step would be likely during evaporation. Moreover, when the T_B step is destroyed, the energetically unfavorable D_A step is formed, which would be unlikely. Therefore, the T_B step could be a stationary state. On the other hand, the T_A step can be combined from the upper D_A step and the lower S_A step. In contrast to the case of the T_B step, the D_A step is very rare and the reactivity at the S_A step could be smaller, which makes the combination pathway into the T_A step unlikely.

V. SUMMARY

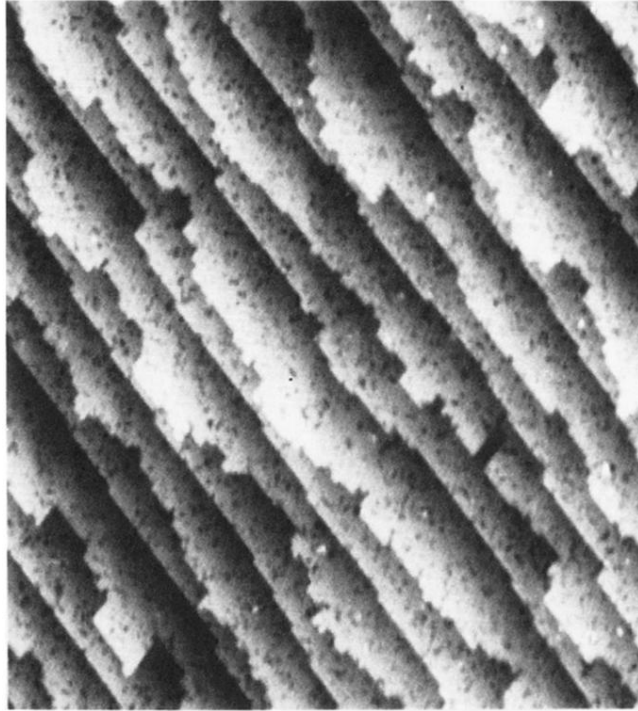
The evolution of the surface configuration upon As deposition on double-domain Si(001) was investigated using STM. The configurations of As covered Si(001) surfaces

show a rich variation in step and domain structures. For As deposition at 500 °C, S_B steps grow dominantly and needle like 2D islands are formed. Formation of these structures can be described chiefly by movement of Si atoms, i.e., diffusion on the surface and sticking at step

edges. For As deposition at 600 °C, a single-domain surface is found to be the stable configuration formed by the rearrangement of surface atoms. For As deposition at 700 °C, T_B steps are formed by simultaneous evaporation.

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(a)



(b)

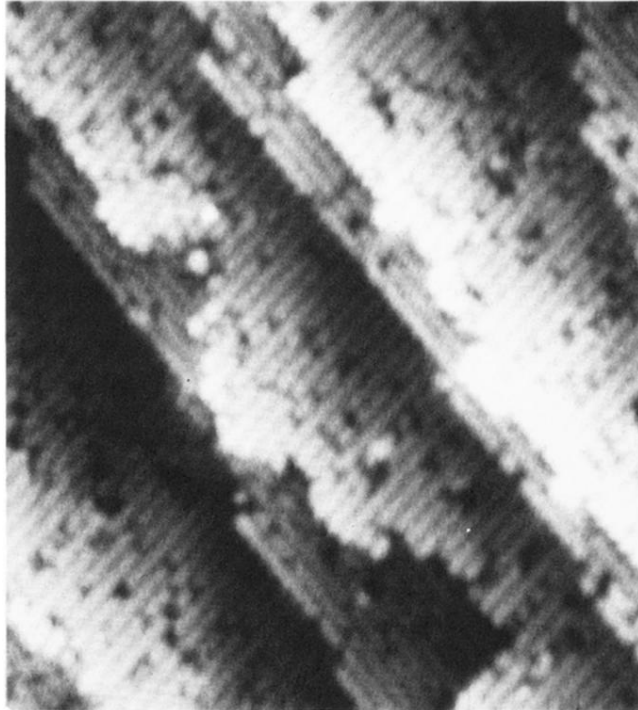


FIG. 1. STM images of a clean vicinal Si(001) surface tilted 1° towards [110]. The scan areas are approximately (a) $1000 \times 1000 \text{ \AA}^2$ and (b) $300 \times 300 \text{ \AA}^2$.

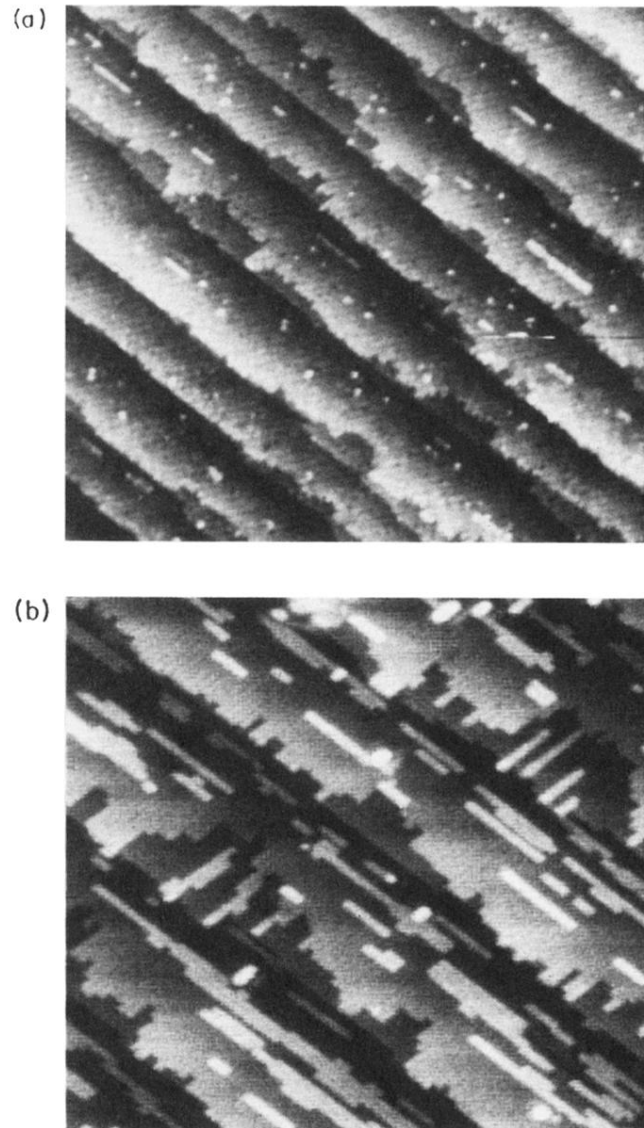


FIG. 2. STM images of Si(001)-As surfaces. These surfaces were exposed to As flux at 500 °C. As coverages were (a) 0.6 ML and (b) 1.0 ML. The scan areas are approximately (a) $1000 \times 1000 \text{ \AA}^2$ and (b) $500 \times 500 \text{ \AA}^2$.

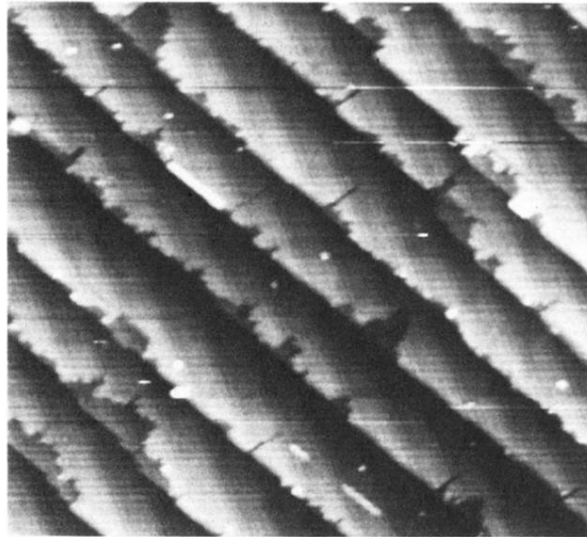
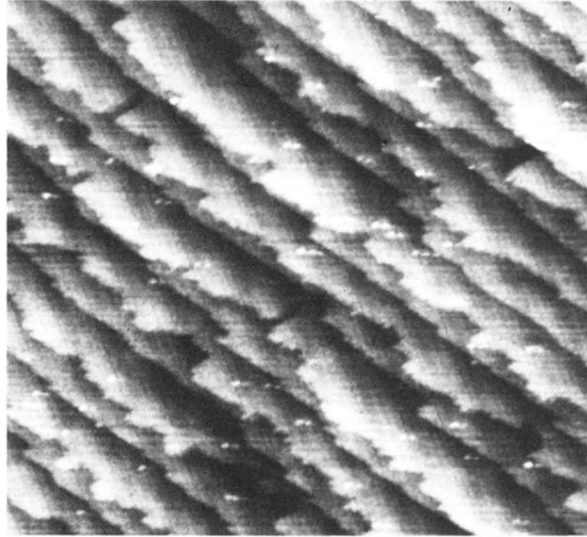


FIG. 3. Si(001) surfaces covered by 1-ML As. These surfaces were exposed to As flux at 600 °C for (a) 50 s and (b) 180 s. The scan area is $1000 \times 1000 \text{ \AA}^2$.

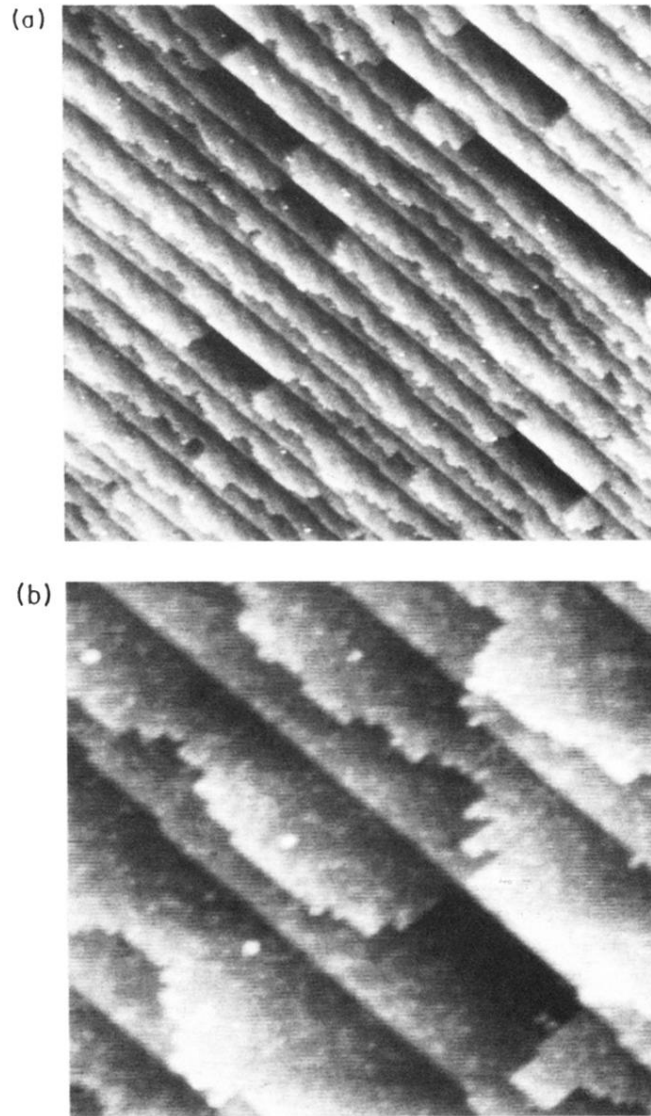


FIG. 4. Large and small area STM images of a Si(001)-As surface. The surface was exposed to As flux at 700 °C. As coverage on the surface was 0.7 ML. The scan areas are approximately (a) $2000 \times 2000 \text{ \AA}^2$ and (b) $300 \times 300 \text{ \AA}^2$.

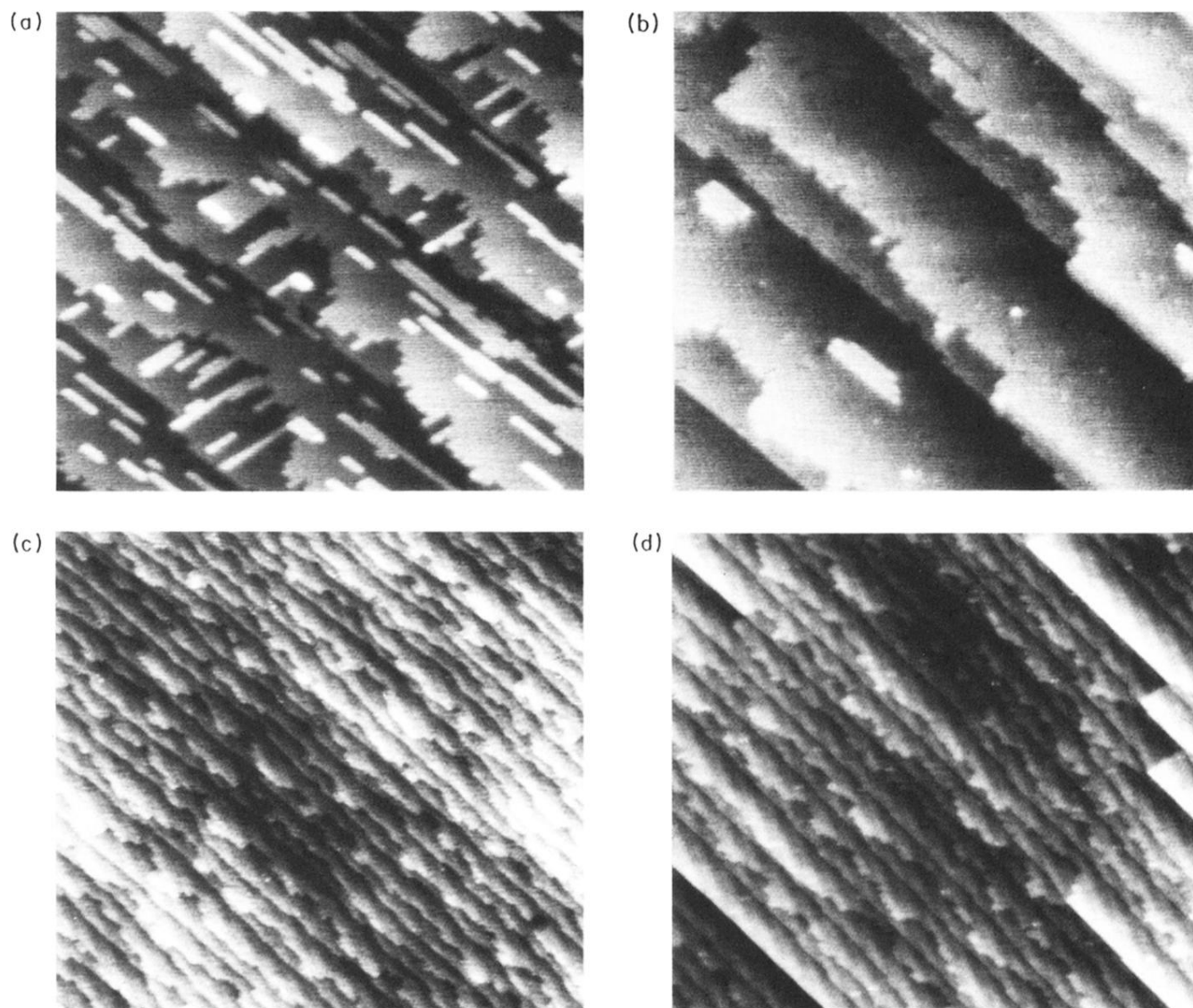


FIG. 5. Effect of annealing after the deposition. (a) Surface annealed at 500 °C for 10 min after 1-ML deposition at 500 °C. (b) Surface annealed at 600 °C for 5 min after 1-ML deposition at 500 °C. (c) Surface annealed at 700 °C for 5 min after 0.7-ML deposition at 700 °C. (d) Surface annealed at 600 °C for 60 min after 1-ML deposition at 500 °C. The scan areas are approximately (a)–(c) $1000 \times 1000 \text{ \AA}^2$ and (d) $2000 \times 2000 \text{ \AA}^2$.