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STM study of In nanostructures formation on Ge(001) surface at different coverages and temperatures*

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Different In/Ge(001) nanostructures have been obtained by annealing the samples at 320°C with different coverages of In. Annealing a sample with a critical coverage of 2.1 monolayer of In, different In/Ge(001) nanostructures can be obtained at different temperatures. It is found that thermal annealing treatments first make In atoms form elongated Ge{103}-faceted In-clusters, which will grow wider and longer with increasing temperature, and finally cover the surface completely.

Keywords: scanning tunnelling microscopy, surface structures, Ge, In
PACC: 6116P, 6820, 6822, 6865

1. Introduction

During the last decades much attention have been paid to the fabrication of nanostructures such as one-dimensional metal wires for potential applications in building nanoscale microelectronic and optoelectronic devices.[1−3] Two basic approaches are adopted to fabricate nanoscale structures in a controllable and repeatable manner, i.e. 'top-down' and 'bottom-up' techniques.[1,4−6] The (001) surfaces of Si and Ge have similar dimer row structures, and are good templates for fabricating one-dimensional metal nanowires by using 'bottom-up' technique.[7−9] In previous experimental or theoretical studies, by considering the compatibility with current semiconductor industry, most of them were on Si(001) substrate.[9−15] Recently, fabrications of metallic Au and Pt nanowires on Ge(001) surface have renewed the activities of Ge(001) substrate-based nanostructures.[16−18] More and more researches are focused on the metal/Ge(001) system, among which In/Ge(001) system is an ideal case for investigating the metal–semiconductor interface because no In–Ge compounds has been found to exist in their binary system and no In–Ge immiscibility has been reported.[19] Sub-monolayer In on Ge(001) surface has been intensively studied on the formation of In-rows or In-clusters structures.[20−25] When a saturation coverage of one monolayer (ML) In was deposited on a Ge(001) surface, Nielsen *et al* reported parallel stripes of 'hut' clusters formed by Ge atoms and In atoms, and gave a simple atomic arrangement model.[26] In addition, annealing a Ge(001) sample with 1.2ML coverage of In at above 200°C could result in the Ge(001)-(5×4)-In reconstruction.[25]

In this paper, In was fabricated on Ge(001) surface with different coverages of 0.85 ML, 1.4 ML, 2.1 ML, 2.8 ML, and annealed at a temperature of 320°C. Different In/Ge(001) nanostructures were observed using scanning tunnelling microscopy (STM). Meanwhile, the sample with 2.1 ML coverage of In at different annealing temperatures was also investigated by means of STM, and various In/Ge(001) nanostructures were demonstrated.

2. Experimental details

Our experiments were performed using an OMICRON STM apparatus equipped with low energy electron diffraction (LEED) Auger electron spectroscopy (AES), which was operated in an ultrahigh vacuum (UHV) chamber with a base pressure of $\sim 5\times10^{-9}$ Pa.

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An n-type polished Ge(001) sample ($\sim 12 \times 6 \times 0.38\,\mathrm{mm}^3$, $0.1\Omega\cdot$cm resistivity at room temperature, highly-doped with Sb) was introduced into the UHV chamber and used as a substrate. The detailed experimental setup and procedures of cleaning Ge(001) surface have been described elsewhere.[27] A large scale almost defect-free Ge(001) surface was obtained, on which high purity (99.99999%) In was deposited several coverages at room temperature. During In deposition, the pressure was kept at $\sim 3 \times 10^{-5}$ Pa and the evaporation temperature was set at 595°C. The deposition rate was calibrated by calculating the percentage of In coverage on the STM images obtained at different initial adsorption processes and the AES signals accordingly. In this paper, 1 ML is equal to 6.24×10^{14} atoms/cm^2, which is the site number density for the unreconstructed Ge(001) surface. STM imaging was all conducted at room temperature using a tungsten tip etched in NaOH solution.

3. Results and discussion

At room temperature, 0.85 ML In was deposited on a clean Ge(001) surface and then annealed at 320°C for 1 h, and the related STM image is shown in Fig.1.

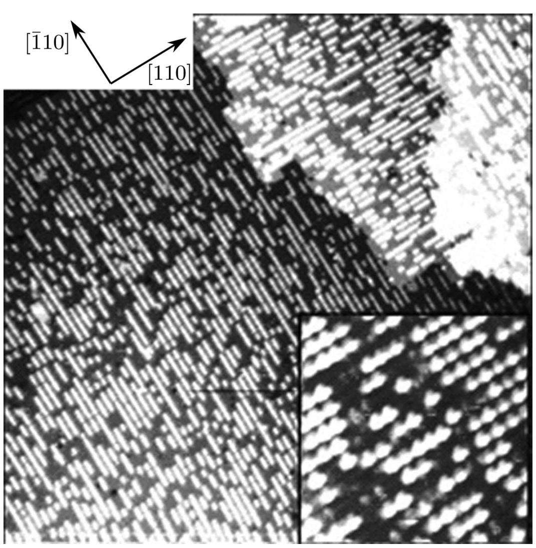

Fig.1. STM image (106×106 nm^2, 1.50 V, 0.40 nA) of 0.85 ML In on Ge(001) surface, deposited at room temperature and annealed at 320°C for 1 h. The inset is its high-resolution (20×20nm^2) STM image (-1.10 V, 0.20 nA) on one terrace.

Bright rows run in one direction on each terrace. From the high resolution STM image (inset), we can see the rows lie between the substrate Ge dimer rows. These bright rows are In-rows, which are made up of In-dimers. As we know, on Ge(001) substrate, Ge dimer rows of adjacent terraces run perpendicularly in two directions, [110] and [$\bar{1}$10]. If the Ge dimer rows of the ith layer terrace run along [110], then those of the (i+1)th or ($i-1$)th layer terraces will run along [$\bar{1}$10]. So the In-rows are also arranged in two directions, [110] and [$\bar{1}$10].

In Fig.2, an STM image is shown, with 1.4 ML In deposited on a clean Ge(001) surface at room temperature and subsequently annealed at 320°C for 1 h. The substrate is completely covered by In-rows with several round-like In-clusters. It indicates that most In atoms make up In-rows and the rest atoms accumulate into the round-like clusters during the high-temperature annealing process. In-rows in one direction such as [110] ([$\bar{1}$10]) direction can be seen as a local domain and defined as domain A (B).

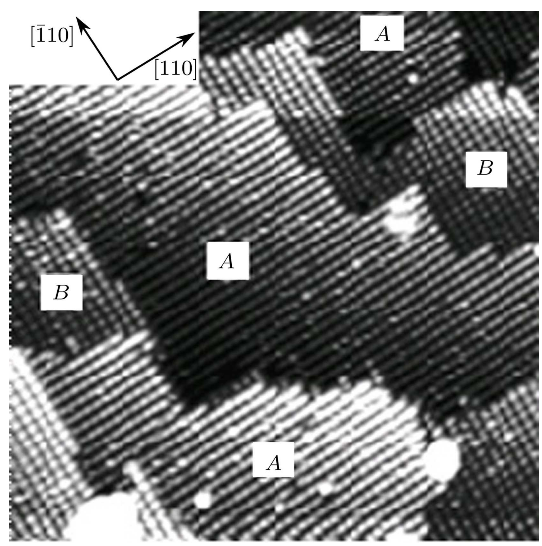

Fig.2. STM image (71×71nm^2, -1.40 V, 0.30 nA) of 1.4 ML In on Ge(001) surface, deposited at room temperature and annealed at 320°C for 1 h.

We increased the In coverage to 2.1 ML and annealed it at 130°C for 1 h. The related STM images are shown in Fig.3. As shown in Fig.3(a), the substrate terraces cannot be observed, but uniform In rows are clearly displayed, which orientate in two directions, [110] and [$\bar{1}$10]. In addition, there are a lot of elongated clusters (indicated by white arrows) and small clusters with irregular shape (indicated by black arrows) distributing among the A and B In-row domains. During 130°C annealing process, firstly the In atoms in the domain A preferentially nucleated into small clusters with irregular shape and size, and subsequently one side grew more rapidly than the other side and elongated clusters were formed. It can be determined in the high-resolution STM observation of Fig.3(b) that the elongated clusters are oriented in [010] or [$\bar{1}$10], running 45° counterclockwise from the direction of In-rows as shown in Fig.3(c). These STM images confirm that In atoms in domain $A(B)$ incline to nucleate along [010] ([$\bar{1}$10]) steps, formed in the two-dimensional In-layers during growth, into elongated clusters. This is an efficient way to relieve strain induced by lattice mismatch. The importance of these kind of clusters for the relaxation of strain was first revealed on Ge/Si(001) system by Mo *et al.*[28,29] The four surfaces of the elongated clusters correspond to Ge ($\bar{1}$03), (103), (0$\bar{1}$3) and (013) facets, which means that the elongated clusters are Ge{103}-faceted In-clusters.[22,26] The annealing temperature of 130°C is not high enough for all the In atoms to diffuse, rearrange, conglomerate, and nucleate, so small In-clusters, elongated Ge{103}-faceted In-clusters and In-rows coexist on the surface. From the height profiles shown in Fig.3(d), the average width of the elongated Ge{103}-faceted In-clusters is 2.5 ± 0.1 nm, and the separation of In-rows is 1.2 ± 0.03 nm (about $3a$, where a denotes the 1×1 substrate lattice constant).

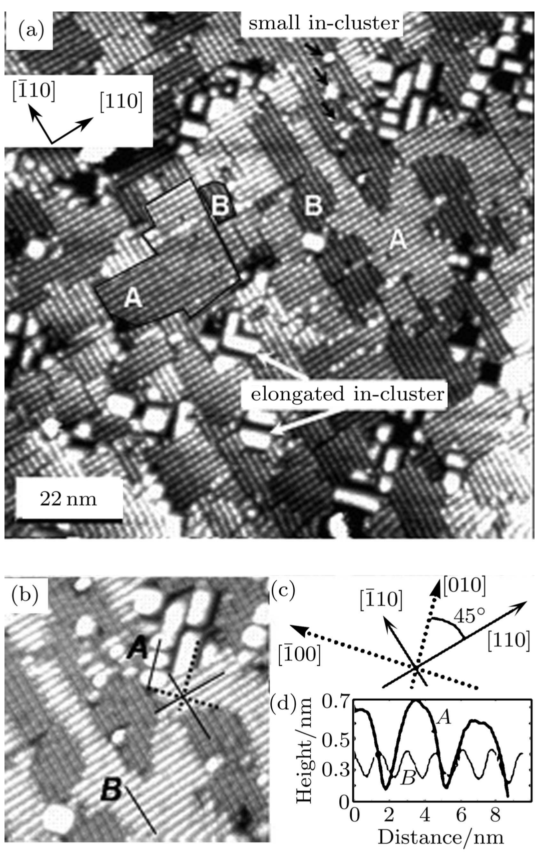

Fig.3. (a) and (b) STM images (1.55 V, 0.30 nA) of the sample with 2.1 ML coverage of In after 130°C annealing for 1 h. Scan scale is $110 \times 110\,\text{nm}^2$ in (a) and $48 \times 48\,\text{nm}^2$ in (b). (c) The relative orientations of the In-rows and the Ge{103}-faceted In-clusters. The solid lines indicate the directions of In-rows and the dotted lines indicate the directions of Ge{103}-facets In-clusters as shown in (b). (d) Height profiles measured along line A (Ge{103}-faceted In-clusters) and line B (In-rows).

Then the sample was further annealed at 240°C for 1 h, and a large-scale STM image of it is shown in Fig.4. It can be found that the surface is completely covered by Ge{103}-faceted In-clusters. This kind of topography is invariable at different bias voltages ranging from −3.0 to 3.0 V. However, detailed atomic structure of Ge{103}-faceted In-clusters cannot be obtained at any bias voltages. In addition, this structure can stably exist in the UHV chamber for several weeks.

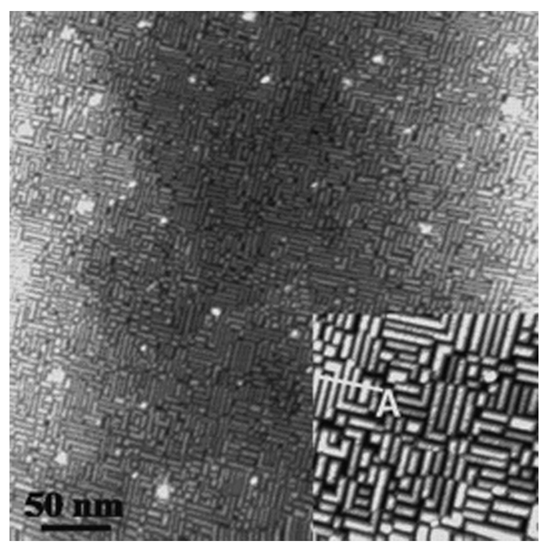

Fig.4. Large scale Ge{103}-faceted In-clusters topography ($400 \times 400\,\text{nm}^2$, 1.40 V, 0.34 nA) of the sample with 2.1 ML coverage of In after annealing at 240°C for 1 h. (inset) Zoomed in STM image ($100 \times 100\,\text{nm}^2$, 1.40 V, 0.34 nA).

Figure 5(a) shows that STM topography on the same sample obtained after further 320°C annealing for 1h has a similar topography. From the height profiles shown in Fig.5(b), with the annealing temperature increasing from 240°C to 320°C, the average width of Ge{103}-faceted In-clusters increases from 3.1 ± 0.1 nm to 4.3 ± 0.2 nm and their length also increases. It means that further higher-temperature (320°C) annealing continuously makes more In atoms located on the surface of the Ge{103}-faceted In-clusters, which causes them to grow wider and longer.

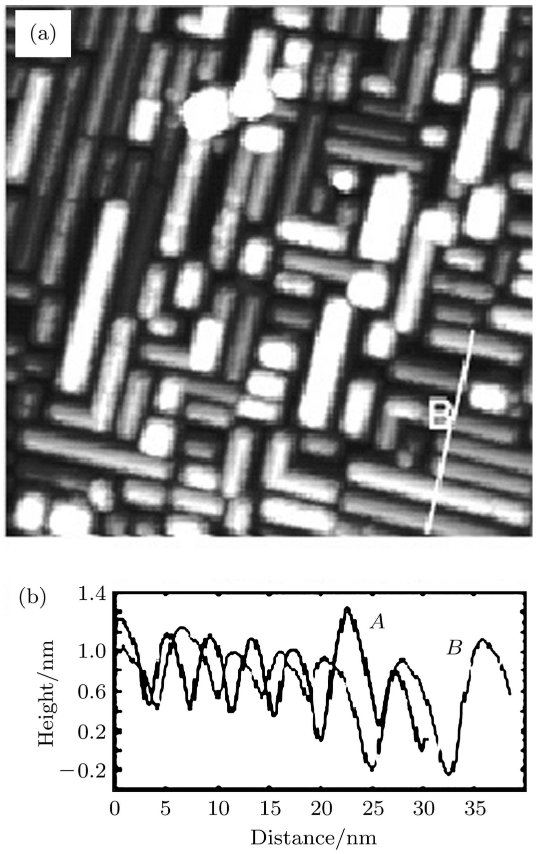

Fig.5. (a) Typical STM image ($100 \times 100\,\mathrm{nm}^2$, 1.61 V, 0.34 nA) of the sample with 2.1ML coverage of In after annealing at 320°C for 1 h. (b) Height profiles measured along lines A and B indicated in the inset of Fig.4 and (a), respectively.

The formation of Ge{103}-faceted In-clusters is coverage dependent. When 0.85 ML and 1.4 ML In were deposited on Ge(001) surface and annealed at 320°C, no Ge{103}-faceted In-clusters formed as shown in Figs.1 and 2. Further increasing the coverages up to 2.0 ML and annealing at 320°C, still no Ge{103}-faceted In-clusters formed (the related STM results are not shown here). Until 2.1 ML In was deposited, Ge{103}-faceted In-clusters can be formed after a suitable annealing process. These results reveal that 2.1 ML is the critical coverage for Ge{103}-faceted In-clusters completely covering the surface after high-temperature annealing.

The formation of Ge{103}-faceted In-clusters with increasing annealing temperature is schematically shown in Fig.6. As shown in Fig.6(a), In-row domains A and B are formed on the substrate terrace. At an annealing temperature of 130°C, irregular small In-clusters (precursors of elongated Ge{103}-faceted In-clusters), elongated Ge{103}-faceted In-clusters and In-rows coexist on the substrate as shown in Figs.6(b) and 3(a). Higher-temperature (240°C) annealing makes more In atoms located on the surface of the precursors, which will saturate all the Ge dangling bonds, and the precursors grow wider and longer as shown in Fig.6(b). When increasing the annealing temperature to 320°C, Ge{103}-faceted In-clusters completely cover the surface as shown in Fig.6(c).

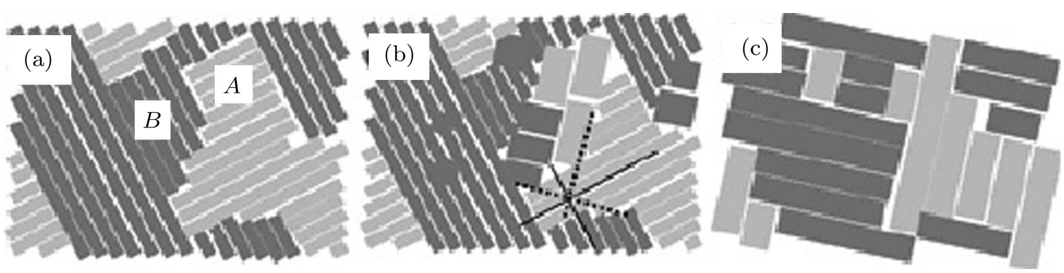

Fig.6. Schematic model of the structural evolution with temperature. (a) The surface is covered by In-rows structure, (b) irregular small In-clusters, Ge{103}-faceted In-clusters and In-rows coexist on the surface, (c) Ge{103}-faceted In-clusters completely cover the surface.

Finally, after 2.8 ML In was deposited on a clean Ge(001) surface and subsequently annealed at 320°C for 1 h, Ge{103}-faceted In-clusters structures were observed in a large scale, as shown in Fig.7(a). The inset shows its zoomed in STM image. From the height profile shown in Fig.7(b), the average width of the Ge{103}-facets increased from 4.3 ± 0.2 nm to 6.7 ± 0.2 nm with the coverage of In increasing from 2.1 to 2.8 ML.

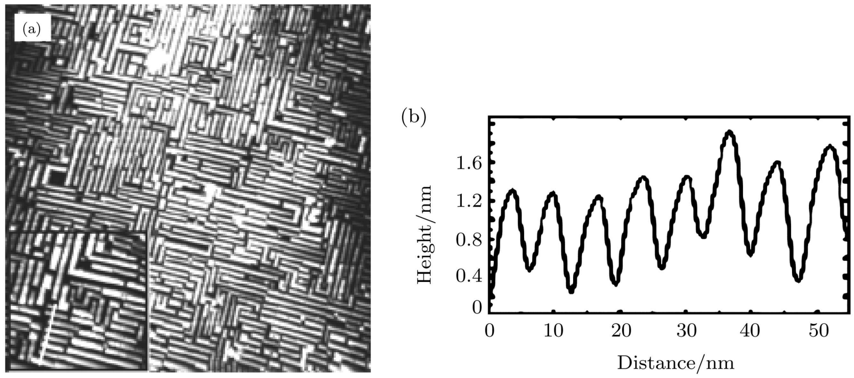

Fig.7. (a) STM image ($367\times367\,\text{nm}^2$, -2.10 V, 0.47 nA) of 2.8 ML In on Ge(001) surface annealed at 320°C for 1 h. The inset of (a) is its zoomed in STM image ($117\times117\,\text{nm}^2$, -2.10 V, 0.47 nA). (b) Height profile along the white line across Ge{103}-facets In-clusters indicated in the inset of (a).

4. Conclusions

In conclusion, we study various In nanostructures on Ge(001) surface, at different temperatures and In coverages using UHV-STM. With increasing annealing temperature, In atoms first form precursors of Ge{103}-faceted In-clusters along [010] ([$\bar{1}$10]) steps, then more In atoms are gathered on their surfaces and saturate all the dangling bonds, and then Ge{103}-faceted In-clusters grow wider and longer, and completely cover the substrate finally. The length and width of the Ge{103}-faceted In-clusters increase with increasing coverage and annealing temperature.

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