Density behaviors of Ge nanodots self-assembled by ion beam sputtering deposition*

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Self-assembled Ge nanodots with areal number density up to 2.33×10^{10} cm^{-2} and aspect ratio larger than 0.12 are prepared by ion beam sputtering deposition. The dot density, a function of deposition rate and Ge coverage, is observed to be limited mainly by the transformation from two-dimensional precursors to three-dimensional islands, and to be associated with the adatom behaviors of attachment and detachment from the islands. An unusual increasing temperature dependence of nanodot density is also revealed when a high ion energy is employed in sputtering deposition, and is shown to be related to the breaking down of the superstrained wetting layer. This result is attributed to the interaction between energetic atoms and the growth surface, which mediates the island nucleation.

Keywords: Ge nanodot, self-organization, ion beam sputtering deposition, adatom behavior

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1. Introduction

Ge quantum dots on Si substrate show promise for the fabrication of highly efficient Si-based light emitters and detectors,[1,2] and can be prepared by self-organization with the Stranski–Krastanov (SK) growth mode in the heteroepitaxy of strained thin films.[3] The specifics of self-assembly are that the accumulated strain in a critically thick two-dimensional (2D) wetting layer acts as the driving force for a transition towards an energetically favored morphology of three-dimensional (3D) islands. Many epitaxial techniques, such as molecular beam epitaxy (MBE) and chemical vapor deposition (CVD), have realized the self-organization of Ge nanodots at deposition conditions close to the thermal equilibrium state.[4,5] The prepared Ge nanodots exhibit a bimodal size distribution, where small dots are square-based pyramids with four (105) facets, and larger ones are polygon-based domes enclosed by (105), (113), and (15 3 23) steeper facets.[5–7] To provide quantum efficiency high enough for application in optoelectronic devices, the nanodots must be prepared with an areal number density higher than 10^{10} cm^{-2} and a narrowly distributed small size.

Previous reports have shown that the island size and density are inversely related, and the density behavior is mainly determined by the island nucleation and growth evolution.[8] As a direct function of the kinetic parameters, the nanodot density has been observed to increase with decreasing temperature and increasing deposition rate in growth by MBE or CVD.[7,9] Based on the behaviors of surface adatoms, a mean-field theory is employed to analyze the time dependence of the nanodot density and how the density varies with external growth conditions.[10] Two possible processes have been proposed to account for the island nucleation and growth: adatoms captured by islands and adatoms escaping from islands.[10,11] The density variations are shown to be the results of kinetic behaviors restricted by the thermodynamic limitation. Since all the processes of the nucleation and growth are dominated mainly by the strain field, the decomposition of the superstrained wetting layer also has a significant effect on the growth evolution.[12]

It has been reported that the energetic atom bombardment to the growth surface can easily induce a superstrained state of the wetting layer during growth by ion beam sputtering deposition (IBSD).[13] The great compressive strains retained in the epilayer can enhance the lateral elastic coupling between the islands.[14] This implies that IBSD as a novel powerful technique for self-organization should favor the growth of small nanodots with high density. Since the sputtered atoms have high kinetic energy, the surface phenomena always exhibit discrepancies off equilibrium.[14–16] The adatom behaviors in IBSD processes are different from those in growth by MBE or CVD, and the kinetic energy should play an important role in the kinetics-defined deposition. By varying the incident ion

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energy and flux density independently, the kinetic behaviors of the energetic atoms can be manipulated,[13,17] which suggests a potential way to control the growth of Ge nanodots. In this paper, the Ge nanodots with a very high density and small size are prepared by IBSD. Nucleation and growth of the islands is investigated by measuring nanodot densities at various deposition conditions. The results reveal a bizarre behavior of the surface adatoms related to the interaction between the energetic atoms and the growth surface.

2. Experiment

The ion beam sputtering system employed an Ar-ion beam generated by a Kaufman-type ion source. The target was water cooled. The sputtered atoms were deposited on an n-type (100)-oriented Si substrate with a resistivity of 1 Ω·cm–2 Ω·cm. The base pressure and Ar gas pressure during deposition were 1.0×10^{-5} Pa and 2.0×10^{-2} Pa respectively. The substrate was first chemically cleaned and dipped in 2.5% HF solution to achieve the hydrogen-passivated surface. After being loaded into the IBSD system, the substrate was kept in the growth chamber at 900 °C for 10 min with a background pressure of 1.0×10^{-5} Pa to desorb some surface-terminated contaminants. After that, a 50-nm-thick Si buffer was deposited at 700 °C and annealed *in situ* for 10 min. Then the Ge nanodots were deposited at the predefined sputtering conditions. After annealing *in situ* for 3 min, the prepared sample was immediately cooled down to room temperature. IBSD offered a large degree of freedom in ion energy and ion flux independently. During the sputtering, the Ar pressure was maintained at 2.0×10^{-2} Pa, while the ion energy was regulated in the range of 0.50 keV–2.5 keV by changing the working voltage of the ion source, and the ion accelerating voltage. The deposition rate was mainly determined by the ion beam current. For the deposition of the Si buffer layer, the selected ion beam current was 7 mA and the deposition rate was 4.2 ML/min. Deposition coverage was controlled by sputtering time. Ge nanodots were prepared at various deposition conditions (temperature, rate, and Ge coverage). The surface morphology was measured by an atomic force microscopy (AFM) system, operating in tapping mode at room temperature in air. As a result of statistical averaging on the AFM image of 5 μm×5 μm, the areal number density, average base width, and height were obtained.

3. Result and discussion

Ge nanodots with small size and high-density on the Si (001) substrate were prepared by IBSD as shown in Fig. 1. The areal number density is up to 2.33×10^{10} cm^{-2} after depositing 15 ML Ge coverage at 600 °C. The height H of the nanodots ranged from 3 nm to 12 nm and the base width W varied between 30 nm and 60 nm as shown in Figs. 1(b) and 1(c). The statistical data reveal that the unimodal size distribution for the height peaked at 7.4 nm and the base width at 47.3 nm, which can be fitted by Gaussian expressions. It has been demonstrated that island formation beyond a critical thickness is energetically favored since the energy to form an island is less than that to form another flat epilayer.[18] Therefore, the size distribution, as a result of the growth evolution of the islands, is determined by the competition of the surface energy and the strain energy. Shchukin *et al.*[19] presented

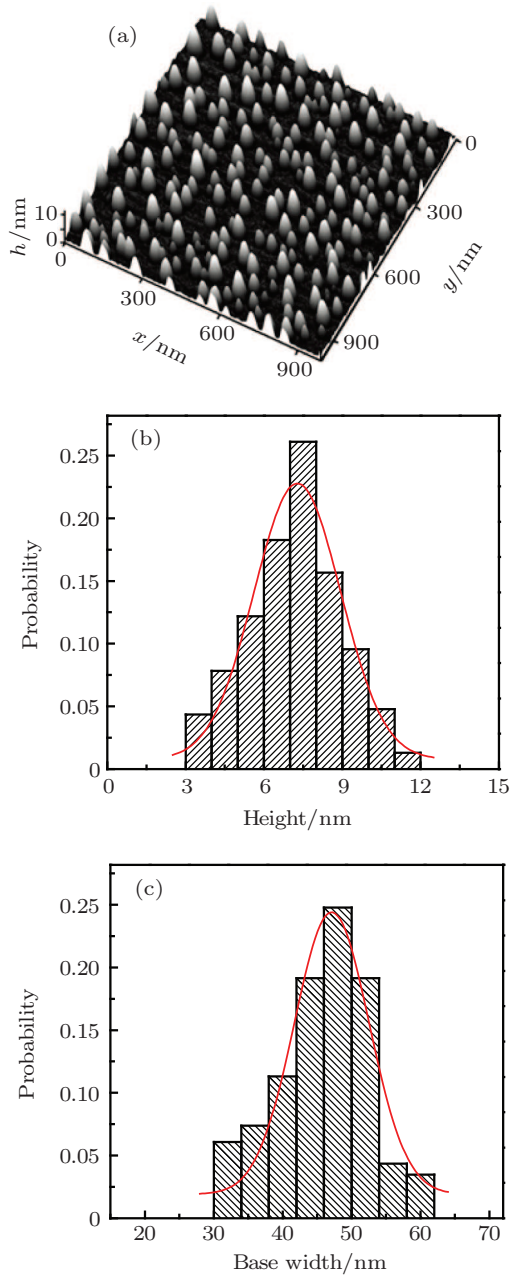


Fig. 1. (color online) (a) 3D AFM image of the Ge nanodots prepared by IBSD. The histograms of the size distribution probability, (b) for the nanodot height, and (c) for the base width, with Gaussian fits given by the solid lines. The nanodots were grown by depositing 15 ML Ge coverage on Si (001) substrate at 600 °C. The ion energy was 0.6 keV and the deposition rate was 4.6 ML/min during the sputtering.

an energy model for lattice-mismatched heteroepitaxy in the Ge/Si system that stabilizes the island size distribution at some ideal value in an energy minimum of island ensembles. The existence of a minimum-energy size was evidenced by the fact that we did not observe Ostwald ripening, that is, the average and deviation of the island size increase linearly with time. Rather, a narrow size distribution of self-assembled Ge nanodots was observed after holding the as-grown Ge islands at a constant temperature for a number of minutes.[20] We also observed that the peak island size and the distribution width for the prepared Ge nanodots are nearly independent of the amount of Ge coverage. These results indicate that the formations have reached a thermodynamically stable state, and that the self-assembling growth of the Ge nanodots by IBSD is dominated by the minimization of free energy.

Compared with other conventional approaches, one distinct characteristic of IBSD is the energy transfer through high-energy ion bombardment, which enhances the adatom mobility on the growth surface. Only when surface diffusion is fast, compared with the incident flux rate of Ge atoms, can we expect the growth to be limited thermodynamically and the distribution of island sizes to have an approximately Boltzmann form.[6,18] Therefore, it can be concluded that the islands reach their stable states through kinetic evolution pathways via the high surface diffusion of adatoms.

Detailed observations of island shape and size distribution made using scanning tunneling microscopy reveal that the Ge quantum dots prepared by MBE or CVD always show a bimodal size distribution, with one peak corresponding to pyramids at small sizes and another representing dome shapes at large sizes.[6] The coexistence of two different islands was attributed to the two stable states corresponding to the local minima of island free energy, which are separated by an energy barrier.[6,15] Unlike previous reports of growth with MBE and CVD, we obtained only a unimodal size distribution of Ge nanodots with a single shape of a dome by IBSD. An AFM image of a typical single dome is shown in Fig. 2(a). The dome displays a complex texture of edges that characterizes the various facets of the structure. The cross-sectional AFM analysis was performed to measure the facet contact angles. The line scan taken along the $\langle 100 \rangle$ direction was obtained perpendicular to the intersection of the facet plane with the substrate plane as shown in Fig. 2(b). The major dome facets we observed in AFM topographs are (105), (113), and (15 3 23) planes, which form contact angles of 11°, 26°, and 34°, respectively. Meanwhile a small but distinct (001) plane appears on the top surface. Because of the introduction of steep facets at the island edges, the aspect ratio $\alpha = H/W$ always shows a large value. The results of α, from hundreds of the nanodots prepared by IBSD, as a function of base width, are plotted in Fig. 2(c). The measured values of α, ranging from 0.12 to 0.22, are determined by the area ratio of the facets. The nanodots not only have an areal density higher than 10^{10} cm^{-2}, but also have an aspect ratio larger than 0.12. This result is difficult to realize by other conventional growth approaches. We did not observe a discontinuity such as that jump of α from 0.1 to 0.17 which is regarded as a marker for the structural transition from pyramids to domes during deposition by CVD.[7] The phenomenon that the sputtered atoms with high kinetic energy tend to grow along the steep facet has also been observed by Chung *et al.*,[15] and can be ascribed to elastic energy relaxation by the strain-driven atomic detachment from the island perimeter.[14,15,21]

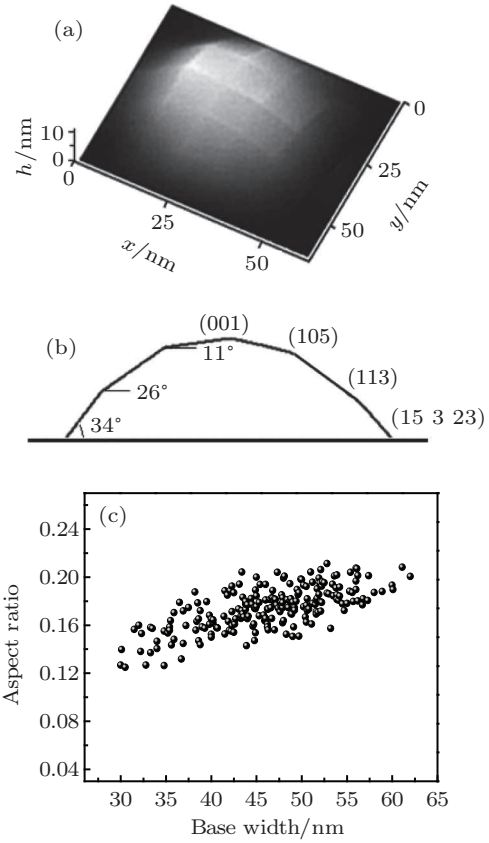


Fig. 2. (a) 3D AMF image. (b) Profile of $\langle 100 \rangle$ cross section of a single dome. The contact angles of the steeper facets with respect to (001) facet are denoted. (c) Aspect ratio of hundreds of nanodots shown as a function of the base width.

The island growth evolution is related to the behaviors of sputtered atom deposition, surface diffusion, and adatom attachment or detachment from the nanodots. The mean-field theory yields the time dependence of nanodot density with respect to external deposition conditions.[10] The nucleation and growth evolution of the islands can be described as follows: First, atoms that are arriving at and adsorbed to the growth surface can be considered as delocalized adatoms. These mobile adatoms diffuse over the surface and eventually meet and bond together to form small 2D clusters. Afterward, those clusters larger than critical size i, as the precursors of the 3D islands,

grow by capturing atoms from both the vapor and the substrate, whereas clusters with size less than i break apart due to thermal fluctuation. Driven by the strain and depending on the size, adatoms detach from edges and migrate onto the top of the island. Finally when a sufficient density of atoms is present on the top of the island to nucleate a new island at its center, the 2D precursor will convert into a 3D dot to release the strain. Because some elastic energy has been partially relaxed in 3D islands, by reducing the rate of atom detachment, the 3D nanodots will rapidly achieve their equilibrium shape and size determined by minimizing the island free energy. We designed the deposition rate of less than 12 ML/min intending that the areal number density of the 3D islands would be controlled by the density of 2D precursors that nucleate on the wetting layer. Based on mean-field theory, the rate of conversion from 2D precursors to 3D islands γ_0 is related to the growth temperature, the diffusion constant, the binding energy of the critical nuclei composed of i atoms, and the size-dependent energy barrier $E_{\rm d}(r)$ for the detachment of atoms from a 2D precursor that has a radius of r.[11] Since $E_{\rm d}(r)$ decreases as r increases, for the exponential function of γ_0, there exists a critical size below which the conversion rate is nearly zero and beyond which the 2D precursors convert very rapidly.[10] The measured areal density of the 3D nanodots versus Ge coverage is plotted in Fig. 3. The deposition was completed with the growth rate of 4.6 ML/min at 650 °C. As the coverage increases, the density initially rises very rapidly due to the fast conversion of 2D precursors to 3D dots. After the formation of 3D islands, they can easily trap atoms detached from the clusters. As a result, the average size of the 2D precursors decreases below the critical value for the transition from 2D clusters to 3D dots, so that the nucleation of 3D islands becomes less frequent and the density tends to saturate. In fact, we previously observed that islands grown by IBSD go through two stages with Ge coverage increasing.[22] When Ge coverage increases from 6 ML to 10.5 ML, domes with small aspect ratios can be obtained. When coverage is greater than 11.5 ML, vertical growth dominates the islands' evolution, and thus the prepared domes always have larger aspect ratios. We show the dot density only as the coverage increases from 10.5 ML to 18 ML, because a very high dot density can be obtained in this range and the same variation characteristics were observed with coverage increasing from 6 ML to 10.5 ML. The areal density of the 3D nanodots is shown as a function of deposition rate in Fig. 4. The Ge coverage of 15 ML was deposited at 650 °C. For the low growth rates, the density is an increasing function of the growth rate. Although the number of 2D islands continues to increase with increasing growth rate, the average size of these islands eventually falls below the value at which the 2D–3D conversion rate is appreciable. Consequently, the nanodot density comes to decrease as the growth rate exceeds 7.7 ML/min. An analogous characteristic has also been observed in the growth of InP/GaAs by metal-organic vapor phase epitaxy.[23]

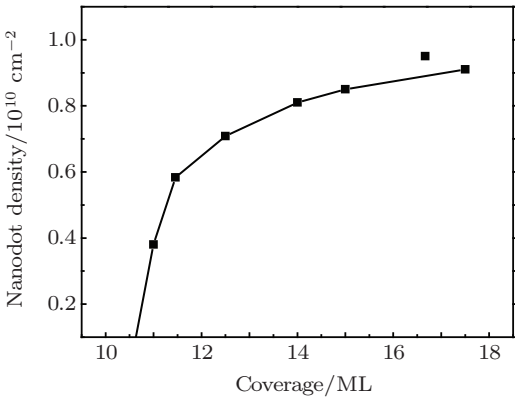


Fig. 3. Nanodot density as a function of deposition coverage θ. Deposition coverages from 10.5 ML to 18 ML are just shown. The nanodots were grown on Si (001) substrate at a temperature of 650 °C. The ion energy was 1.1 keV and the deposition rate was 4.6 ML/min during the sputtering.

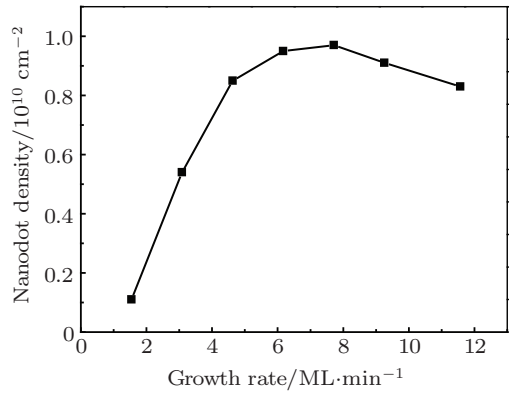


Fig. 4. Nanodot density as a function of the deposition rate. Ge coverage of 15 ML was deposited at 650 °C by IBSD. Ion energy was maintained at 1.1 keV during the sputtering.

Island density is always a decreasing function of deposition temperature in growth by MBE and CVD, a phenomenon which has been ascribed to an intermixing of Ge and Si atoms as well as simultaneous coarsening at high temperature.[9,24–26] However, in the Ge dot growth by IBSD, this characteristic was observed only when the ion energy is less than 0.6 keV, as shown in Figs. 5(a)–5(c). When the ion energy is larger than 1.1 keV, on the contrary, island density shows an increasing trend with increasing deposition temperature, as can be seen in Figs. 5(d)–5(f), which has also been reported previously.[14] The ion energy mainly determines the kinetic energy of the sputtered atoms as the flux rate is kept constant. Considering the interaction between the sputtered atoms and the growth surface, increasing the kinetic energy of sputtered atoms would reinforce the related effects on island growth evolution.

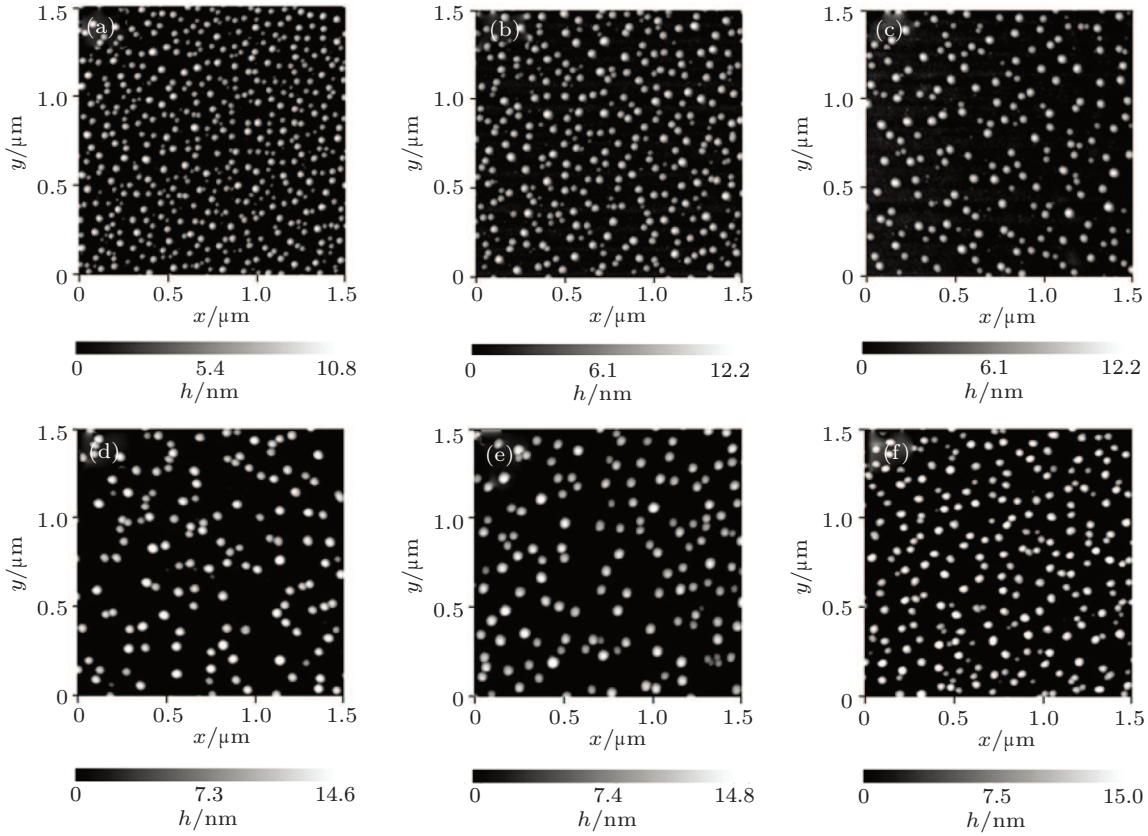


Fig. 5. The 1.5 μm×1.5 μm AFM topographic images of Ge nanodots grown by IBSD at various deposition temperatures. When ion energy of 0.6 keV is employed in the sputtering, the nanodot density decreases as the deposition temperature increases from (a) $T = 600$ °C to (b) $T = 630$ °C and to (c) $T = 700$ °C. However, when the ion energy is 1.1 keV, the nanodot density increases as the deposition temperature increases from (d) $T = 600$ °C to (e) $T = 630$ °C and to (f) $T = 700$ °C. The deposition rate was 4.6 ML/min and the deposition coverage was 15 ML. All samples have gone through annealing *in situ* for 3 min after deposition.

Several researchers have reported that high quality $Si_{1-x}Ge_x$ films can be prepared by IBSD over a wide range of growth temperature.[13,16,27] The films were observed to retain compressive strains greater than those measured in a layer prepared by MBE under similar deposition conditions.[13,16] At the same time, the critical wetting layer at the transition from 2D to 3D growth was also observed to be about 6 ML, which is much thicker than that in growth by MBE. These phenomena are assumed to be the result of high intensity surface bombardment with fast particles.[13,27] Sasaki *et al.* calculated the energy profiles of sputtered atoms by Monte Carlo method, showing a single peak roughly at 25 eV and few atoms with energy greater than 100 eV.[16] The mean free path of the sputtered atoms is about 25 cm, while the distance between the target and substrate is only 15 cm in our case, therefore the growing film is supposed to be exposed to bombardment by the sputtered atoms in addition to the recoiled Ar atoms. In this condition, the initial binary collision between the energetic ion and a surface atom should be considered since only one or two displacements can be made by the bombardment of the atoms with energy lower than 100 eV. Energy will be transferred to adatoms on the collision of fast atoms with small 2D clusters, which is an effective way for either flattening the 2D cluster by moving a top-layer atom down or detaching an adatom from the cluster laterally.[17] For a cluster with size close to or smaller than the critical size to be thermodynamically stable, the removal of adatoms will result in the cluster being reduced below the critical size and dissociating spontaneously. Decrease in the number of 2D islands will make the number of nuclei small, and consequently the density of the nanodots will be reduced when high ion energy is used in the growth by IBSD at a low deposition temperature. This hypothesis is in accord with the comparison of the results shown in Figs. 5(a) and 5(d), or in Figs. 5(b) and 5(e), in which the nanodots are prepared at the same deposition temperature but at different ion energies. Choi *et al.*[17] also demonstrated that low-energy (< 30 eV) Ar ion irradiation can enhance surface diffusion during InAs heteroepitaxy on Si and prolong the layer-by-layer stage of SK nucleation by several monolayers. Thus we conclude that energetic atom impacts create an additional energy barrier E_b to nucleation and suppress island formation.

Ge nanodots have been observed to grow on a wetting layer thinner than that at the onset of nucleation.[28] The wet-

ting layer, being a metastable state of superstrain, is easy to break down since a large amount of strain energy has been accumulated in it. Atoms escaping from a partial wetting layer also take part in the island nucleation and growth to relax the excess strain energy, and ultimately reach equilibrium.[29] The nucleation rate γ of the 3D nanodots is not only thermally activated with activation energy or nanodot formation energy, but also promoted by the superstress of the wetting layer.[12] Taking the ion-induced reduction of island nucleation into account for growth by IBSD, we may obtain

$$\gamma = \gamma_0 \exp\left(\frac{E\Delta\theta - E_b}{K_B T}\right), \quad (1)$$

with the constant E related to the long-range van der Waals forces[30] and nonlinear elastic effects[31] in the wetting layer, T is the growth or annealing temperature, and K_B is the Boltzmann constant. The term $E\Delta\theta = E(\theta_i - \theta_e)$ plays the role of island formation energy, whose coverage dependence might be comprehended by a change of precursor.[12] Here, θ_i and θ_e correspond to the thickness of the wetting layer at the onset of nucleation and that at growth equilibrium, respectively. Because energetic atom bombardment induces the dissociation of island precursors that favors the 2D growth, $\Delta\theta$ is an increasing function of the ion energy in growth by IBSD. The effect of the super-strained wetting layer on the island nucleation rate is crucial when the ion energy is high, while it can be omitted reasonably if the ion energy is very low. In the second case, the growth behavior in island preparation by IBSD can be considered to be somewhat similar to that in growth by MBE. The increasing size and decreasing density of islands can be observed during the IBSD deposition and *in situ* annealing at higher temperature, as shown in the upper part of Fig. 5. These may result from more severe coarsening via a combination of Si interdiffusion and conventional ripening as reported for MBE growth.[24,25] On the contrary, the growth behavior will be mediated by concurrent energetic particle bombardment when sputtered atom energy is high. As shown in the lower part of Fig. 5, the island density increases and the island size decreases with increasing deposition temperature when a high ion energy of 1.1 keV is employed during sputtering. These results suggest that the island density may be determined by a supersaturation-driven nucleation. In this condition, higher deposition temperature results in the reduction of θ_e, and the subsequent increase of $\Delta\theta$ enhances the island nucleation rate and promotes decomposition of the super-strained wetting layer. In thermodynamically limited self-assembled growth, the smaller islands are favored at higher temperature since there are more possible arrangements of the atoms, and hence higher entropy for small islands than for large ones.[18] Therefore, the atoms released from the wetting layer would nucleate to form new islands rapidly, giving rise to the growth of islands with a higher number density. This may contribute to the unusual temperature dependence of island density when high ion energy is used in the growth by IBSD.

4. Conclusion

In conclusion, we have shown that the self-assembled Ge nanodots with the areal number density higher than 10^{10} cm^{-2} and the aspect ratio larger than 0.12 have been prepared by IBSD on Si substrate. The density evolution reveals a kinetic growth process mediated by the interaction of energetic atoms and the growth surface, which enhances the surface diffusion and changes the island nucleation. The unimodal size distribution combined with the morphology of only dome-shaped islands indicates that the assemblies have reached their thermodynamically stable state through kinetic evolution driven by the high surface diffusion of adatoms. The dependence of island density on the deposition rate and the Ge coverage is demonstrated to be limited mainly by the transformation from 2D precursors to 3D nanodots and to be associated with the adatom behaviors of attachment and detachment from the islands. The nanodot density is an increasing function of temperature when the sputtering deposition employs high ion energy, which differs from the growth at the thermal equilibrium state. We argue that this phenomenon is due to the island formation accompanied by the consumption of the metastably superstrained wetting layer, which is mainly caused by the striking of sputtered atoms, and we conclude that the concurrent energetic particle bombardment can be used to modulate the self-assembling growth of Ge nanodots.

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