Shape, size, and number density of InAs quantum dots grown on the GaAs$(\overline{113})$B surface at various temperatures

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InAs quantum dots (QD's) were grown on the GaAs$(\overline{113})$B surface by molecular-beam epitaxy at temperatures between 435 and 490 °C. Their shape, size, and number density were investigated by *in-situ* scanning tunnelling microscopy. The shape of the QD's is given for the most part by {110}, (111)B, and vicinal (001) bounding facets, and does not change significantly with growth temperature. The diameter at the base and the height of the QD's increase monotonously from 25 to 54 nm, and from 3.5 to 9.8 nm, respectively, whereas the number density decreases as temperature increases. This is explained by assuming a slight decrease of the number density of critical growth nuclei with increasing temperature. The size distribution is bimodal: besides the coherent QD's, some larger and probably incoherent islands are observed that are extended along $[33\overline{2}]$. Post annealing increases the diameter and the height by about 30% and decreases the number density, but does not change the shape significantly.

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I. INTRODUCTION

Self-assembled quantum dots (QD's) have been extensively studied in recent years because of their potential for technological applications.[1] Self-assembling occurs by taking advantage of the Stranski-Krastanow (SK) growth mode that often applies for heteroepitaxy in systems with lattice mismatch ⩾2% such as InAs/GaAs (7.2%): Three-dimensional (3D) dislocation-free (so-called coherent) islands form on the top of a wetting layer that is largely composed of the deposited material. These islands can be completely overgrown with substrate material establishing an ensemble of 10^9–10^{11} QD's cm^{-2} and, can be used for quantum-optoelectronic devices. In the SK growth mode, islands form when the strained heteroepitaxial film reaches a critical thickness because the material can better relax in slightly strained 3D islands than in a heavily strained film. Thereby, the gain in elastic energy compensates the energy cost due the increase in surface area.

For InAs QD's on GaAs(001), it is well known that experimental growth parameters such as temperature,[2–9] rate,[9–13] value of As_2 (As_4) pressure (or As/In flux ratio),[6,11,12] total amount of deposited InAs material,[3,11–17] and post annealing[18] influence the size distribution and the number density. Although there are some discrepancies among these reports, there is a general trend that the QD's increase in size and decrease in number density, when the growth temperature increases, or the growth rate or the As pressure decreases. For an increasing amount of deposited InAs material, the number density of the QD's increases largely, whereas the size of the QD's increases only slightly up to a certain thickness. Thereafter, the QD's tend to coalesce and form incoherent dots.[3,11–16] Incoherent dots—as opposed to coherent dots or QD's—relax strain through incorporation of dislocations at the interface. Annealing after growth (post annealing) may also increase the average size of the QD's to some degree, but it usually induces decomposition of the QD's.[9,18] The change of the size of the QD's is explained so far by kinetics,[2,3,6,8,9,11,12] i.e., mainly by a change of the In diffusion length on the surface.

Recently, the GaAs(113)B surface was studied in some detail[19–24] because of its potential as a substrate for self-organized growth of InAs quantum dots (QD's). We have already reported that InAs QD's grown on the GaAs(113)B surface are composed of a main part sitting on a flat base.[25] The bounding facets of the main part are formed by low-index surfaces, namely, {110} and (111)B, and a rounded region from vicinal (001) surfaces, while the flat base consists of high-index {135}B and (112)B surfaces. This shape is quite different from that of InAs QD's grown on the GaAs(001) surface whose main bounding facets are {137} facets.[26] The (137) surface was understood as a part of the reconstruction of the (2 5 11) surface that was discovered recently as a stable surface within the stereographic triangle.[27–29]

Considering the previous studies, it cannot be excluded a priori that the growth parameters influence also the shape of the QD's. Actually it was reported that the shape of QD's on GaAs(001) varies with growth temperature (or with QD size).[7] In the present study, therefore, we investigated the shape, the size distribution, and the number density of the QD's grown on GaAs(113)B at different temperatures by *in situ* scanning tunneling microscopy (STM) from which we also could determine the surface structure by atomically resolved images. Our paper is organized as follows: After giving some experimental details in Sec. II, we will present the results and discussion in Sec. III, which is followed then by our conclusions in Sec. IV.

II. EXPERIMENT

The experiments were carried out in a multichamber ultrahigh-vacuum system consisting of a surface analysis chamber, a small molecular-beam epitaxy (MBE) chamber, and an STM chamber (Park Scientific Instruments, VP2).[30] Samples with a typical size of ≈5×10 mm^2 were cut from a GaAs(113) wafer (*n*-type, Si-doped, carrier concentration

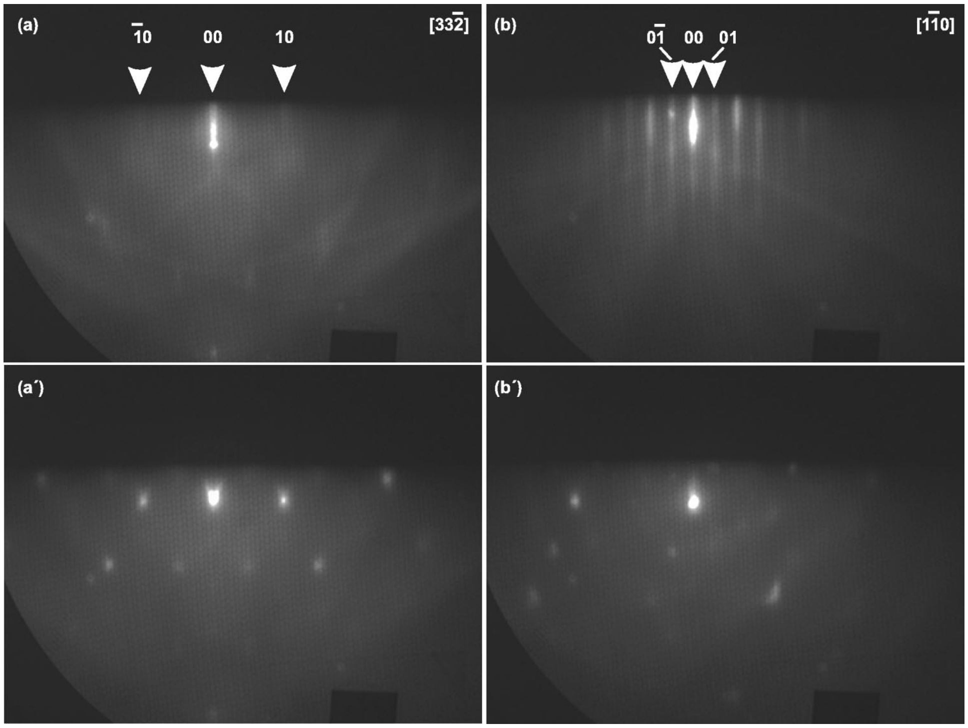

FIG. 1. RHEED patterns before (a,b) and after (a′,b′) InAs deposition of 2.5 ML at 435 °C. The electron beam was incident along [$33\bar{2}$] for (a) and (a′), and along [$1\bar{1}0$] for (b) and (b′).

$1.4–4.8\times10^{18}$ cm^{-3}, Wafer Technology). The samples were cleaned by several ion bombardment and annealing cycles. Afterwards, a GaAs buffer layer about 50 nm thick was deposited using MBE at 530 °C. The temperature was measured by a pyrometer that was calibrated against the GaAs(001) $c(4\times4)$ to $\beta2(2\times4)$ transition at 465 °C. Then the samples were cooled down to a growth temperature between 435 and 490 °C, and InAs was deposited. The sample heater and the In- and As-Knudsen cells were shut off immediately, as soon as the reflection high-energy electron-diffraction (RHEED) pattern along [332] changed from streaky to spotty. For 1-min post annealing, only the In-Knudsen cell was shut off simultaneously with the changing RHEED pattern. Then the samples were transferred to the STM chamber within less than 1 min without breaking the vacuum. The nominal amount of InAs deposited onto the surface was 2.5–2.9 monolayer (ML), 1.4–2.0 ML, 2.1–2.5 ML, and 1.8–3.4 ML for 435 °C, 450 °C, 470 °C, and 490 °C, respectively. For the {113} surfaces the ML is 0.17 nm high. The growth rate of the InAs was about 0.005 nm/s. Beam equivalent pressure ratio of As_2/In was 40–50 at an As_2 pressure of $\sim7\times10^{-7}$ mbar.

III. RESULTS AND DISCUSSION

Figure 1 shows RHEED patterns before and after deposition of 2.5 ML of InAs at 435 °C. For the pattern before InAs deposition [Fig. 1(a) and 1(b)], arrowheads indicate the positions of the zero- and first-order diffracted beams, which corresponds to periodicities of 0.4 nm and 1.3 nm perpendicular to the [332] and [110] directions, respectively. Superstructure spots are not observed. Accordingly, the surface structure of the bare GaAs(113)B surface exhibits (1×1) symmetry assuming a face-centered unit cell for the bulk-truncated surface. This agrees with a previous report on a mixed (2×1)+(1×1) structure of the face-centered unit cell forming at As-rich condition.[23] Lowering the sample temperature, a phase transition between the Ga-rich (8×1) and the As-rich (1×1) structure occurred at 470–490 °C that is in agreement with approximately 470 °C reported earlier.[23] All InAs QD's shown below were grown on the (1×1) reconstructed (113)B surface.

After InAs deposition, the RHEED pattern changed from streaky to spotty as shown in Figs. 1(a′) and 1(b′), which indicates the formation of 3D islands. Moreover, it is noted that the RHEED pattern along [332] shows V-like streaking of the reflexes, so called chevrons, which indicates that flat facets are formed on the 3D islands. These chevrons are not observed in the [110] azimuth in Fig. 1(b′). The InAs deposition was stopped as soon as the change from 2D to 3D was recognized by eye on the fluorescent screen. The nominal amount of deposited InAs could not be kept accurately constant in the present study. This variation is considered to be in part due to differences of the 2D-3D transition on growth temperature and in part also to the sensitivity of the operator.

Figure 2 shows overview STM images of InAs QD's grown at different temperatures between 435 and 490 °C. The number densities are 6×10^{10}, 3×10^{10}, 1×10^{10}, and 3×10^{9} cm^{-2} for 435, 450, 470, and 490 °C, respectively. However, as the deposited amount of InAs is not the same for all four growth temperatures, the number densities are considered to be only a rough measure. Two types of 3D islands or dots are clearly seen in these STM images. There are many relatively small islands of remarkably uniform size and some larger islands that vary significantly in size, i.e., the size distribution is bimodal. We assign the smaller islands to coherent dots, which presumably are QD's, and the larger islands to incoherent islands, as described in detail below.

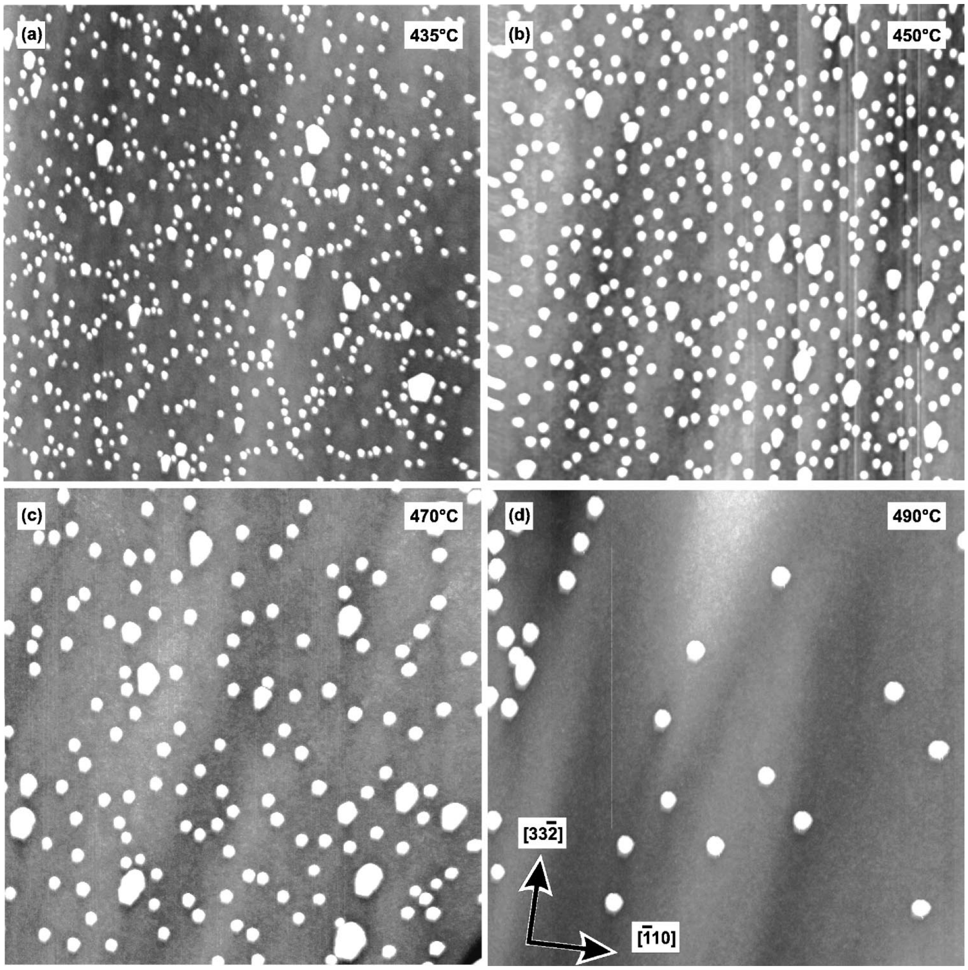

FIG. 2. Overview STM images of InAs QD's grown on the GaAs(113)B surface at (a) 435 °C, (b) 450 °C, (c) 470 °C, and (d) 490 °C ($U=-3$ V, $I=0.1$ nA). The nominal amount of deposited InAs is (a) 2.5 ML, (b) 1.4 ML, (c) 2.1 ML and (d) 3.1 ML. The size is 1×1 μm^2.

The QD's seem to be more round at the interface, while the larger islands exhibit some elongation along [332]. The size and the height distributions of the QD's and the larger islands will be discussed in detail below. However, from the overview STM images, it is already obvious that the size of the QD's increases and the number density decreases as growth temperature increases.

Large-magnification 3D STM images of the QD's grown at indicated temperatures are shown in Fig. 3. For all images except Fig. 3(d), atomic resolution is achieved, which allows determining the orientation of the bounding facets.[25] It is obvious from these images that the shape of all four QD's is the same: A main part with steeper facets sits on a flat base with flatter facets—flat and steep with respect to the (113) substrate. A model is given in Fig. 3(e). The main part consists of a frontal (111)B facet, two {110} side facets, and a rounded back part that may be composed of vicinal (001) surfaces; the flat base is terminated by two {135}B high-index facets and (less certain) a (112)B facet.[25] The {135}B facets connect the two {110} side facets with the substrate; (112)B does the same for the (111)B facet. From these images it is evident that the shape of the QD's does not change for growth temperatures between 435 and 490 °C.

One may ask whether the flat base is a stable part of the QD or only metastable, induced by growth kinetics. In order to answer this question we performed post annealing experiments for InAs QD's grown at 435 °C. Figure 4(a) shows an overview STM image and Fig. 4(b) a 3D STM image of a single QD post-annealed for 1 min. Comparing the overview STM image with Fig. 2(a) one recognizes that there is no significant change: there are still many QD's and some larger islands. However, the relative number of large islands increased, the size of the QD's slightly increased, and the number density (2×10^{10} cm^{-2}) decreased, which are considered to be due to creation of additional large islands by coalescence and some growth of the QD's at the expense of the decomposition of some of them.[18] Also the shape of the QD's does not change significantly as seen in Fig. 4(b). The flat base composed of the high-index facets still exists as well as the main part, which suggests that the flat base is not a frozen-in transitory structure appearing only during growth, but is an intrinsic part of the InAs QD's on GaAs(113)B.

A larger (presumably incoherent) island and a smaller (presumably coherent) QD prepared at the same conditions as for the QD's depicted in Fig. 4 are shown in Fig. 5(a). The incoherent island, marked by an arrow in Fig. 5(a), is reproduced in Fig. 5(b) in 3D format. It is more elongated along [332] than the QD. It is important to note that the incoherent island has a shape similar to that of the QD. The main part and the flat base is confined by the same bounding facets found for the QD's. This is quite different from GaAs(001)

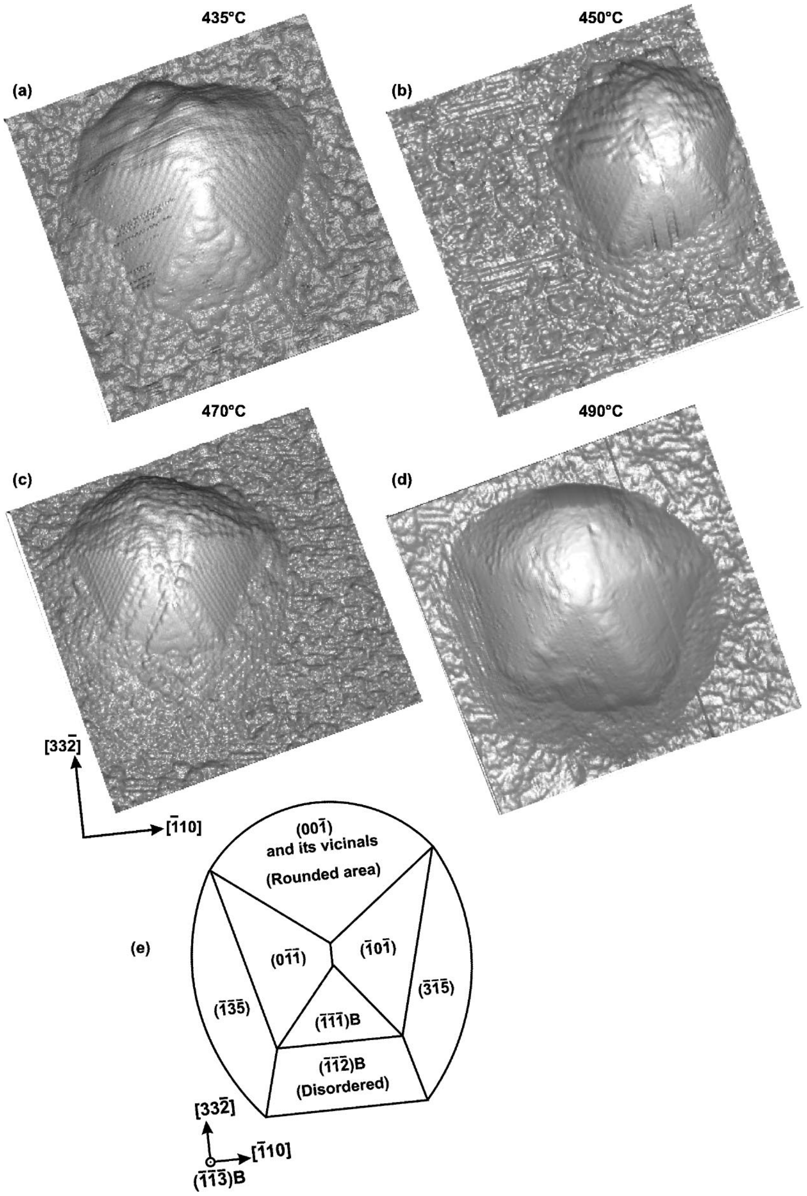

FIG. 3. High-resolution 3D STM images of InAs QD's grown on the GaAs(113)B surface at different temperatures ($U=-3$ V, $I=0.1$ nA). The size is (a) 28×28 nm^2, (b) 36×36 nm^2, (c) 42×42 nm^2, (d) 65×65 nm^2, and (e) model derived from the STM images.

substrates, for which the incoherent islands exhibit irregular shape.[13,15,16]

The question of whether the large islands are incoherent or not has to be answered by photoluminescence measurements on our samples in a future study. This question is connected with the basic understanding of the strain-induced Stranski-Krastanov growth. In this model a bimodal distribution of QD's is difficult to understand: Larger objects are more strained and grow more slowly than smaller ones inducing then a rather sharp distribution centered at one size. Two exceptions may be imaginable: First, it is known from GaAs(001) that the QD shape changes from more flat to more steep if the amount of deposited material increases.[7] Thus, in a narrow coverage range both types may be observable. However, this does not apply to our case as the shape of QD's and larger islands is nearly the same. Second, in the wetting layer one may imagine areas of reduced strain, e.g. around step edges, which may allow for growth of larger dots. Such a case in which the distribution became bimodal was discussed for annealing of QD assembles.[31] In our case we did not observe any correlation between steps and large islands. Thus, from the structural observations we conclude that the larger islands are incoherent.

Figure 6 shows the length distribution along $[33\bar{2}]$ (including the flat base) and the height distribution of the QD's and larger islands grown at 470 °C. The distributions for QD's grown at the other temperatures (not shown here) are similar to those of Fig. 6. There are relatively sharp peaks at about 35 nm in (a) and 6 nm in (b). The standard deviation is less than 15% in size and 20% in height for all temperatures. The sharpness of the distributions indicates that the small dots are real QD's, i.e., objects whose sharp size distribution is induced by an appreciable strain. This strain is expected to reduce the growth rate for the larger dots, thus sharpening the size distribution. In addition to the main peaks in Fig. 6, there are weak and broad side peaks or tails between 50 and 85 nm in (a), and between 9 and 14 nm in (b). Therefore we call the distributions bimodal. The side peaks are due to the

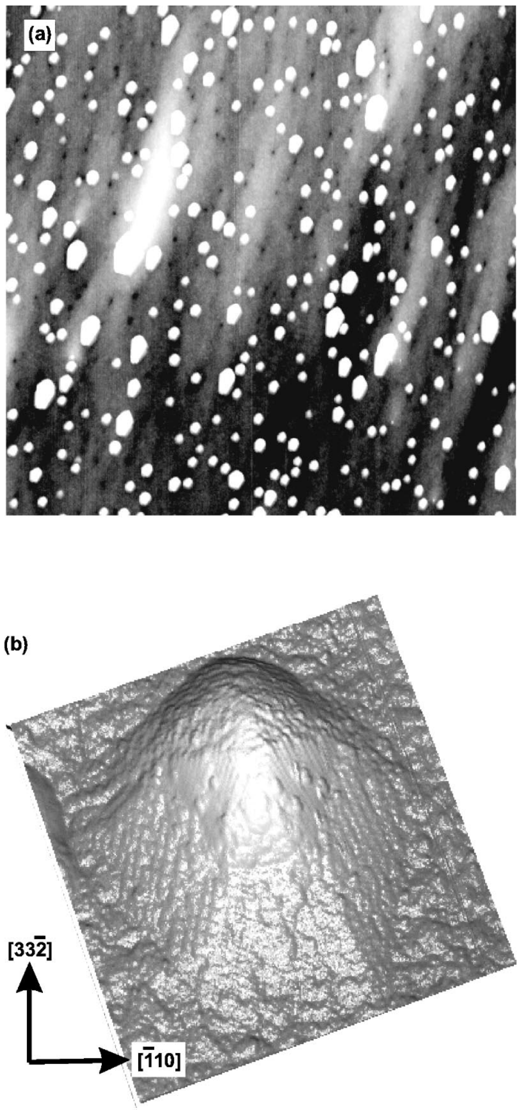

FIG. 4. (a) An overview STM image of InAs QD's after growth at 435 °C ($U=-3$ V, $I=0.1$ nA) post annealed by 1 min and (b) a 3D STM image of a single QD after the same procedure. The nominal amount of deposited InAs is 2.6 ML. The size is (a) 1×1 μm^2 and (b) 37×37 nm^2.

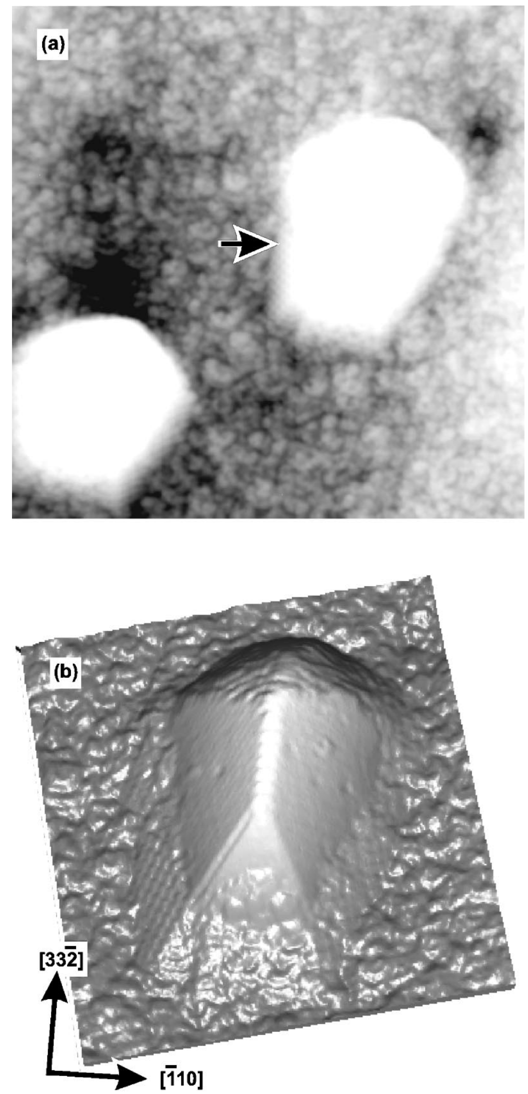

FIG. 5. (a) An STM image of a larger (incoherent) island and of a QD prepared by the same condition as the islands shown in Fig. 4 ($U=-3$ V, $I=0.1$ nA). The incoherent island, indicated by an arrow in (a), is reproduced in (b) in 3D form. The size is (a) 78×78 nm^2 and (b) 43×43 nm^2.

larger islands. Since they do not have uniform size, they are considered to be incoherent islands, in which strain is relieved by incorporation of dislocations and the further growth is not limited by strain.[32,33]

Figure 7 shows the growth-temperature dependence of (a) the size and (b) the height of the QD's. Triangles at 435 °C in (a) and (b) indicate those values measured from samples post-annealed for 1 min. The size and the height increase with temperature monotonously from 25 to 54 nm and from 3.5 to 9.8 nm, respectively. The post annealing increases the size and the height by about 30%. A similar dependence on growth temperature has been found for the growth of InAs QD's on GaAs(001).[2–9]

The dependence of the QD size and number density on the growth temperature has been explained so far by kinetics, as pointed out in the Introduction. QD's are formed when a critical thickness of the deposited InAs layer is achieved. After the nucleation, larger strain concentration at the edge of the larger QD's makes it more difficult for the incoming adatoms to be attached to the larger QD's than to the smaller ones, which explains the rather uniform size distribution in terms of kinetics.[34–36] In the scheme of kinetics, one nucleation site collects InAs material from a circular area whose radius is proportional to the diffusion length of the In atoms. Therefore, when the growth temperature (and the diffusion length of the In atom) increases, the average size of the QD's increases and the density of the QD's decreases. In kinetics, the shape of the QD's is considered to be determined mainly by the growth speed of each bounding facet.[36,37] However, no theoretical calculation or confirmation of the shape of the QD's in kinetics has been reported so far. This explanation seems quite straightforward, but a closer look discloses a severe problem: As recognized from Fig. 2, the mutual dis-

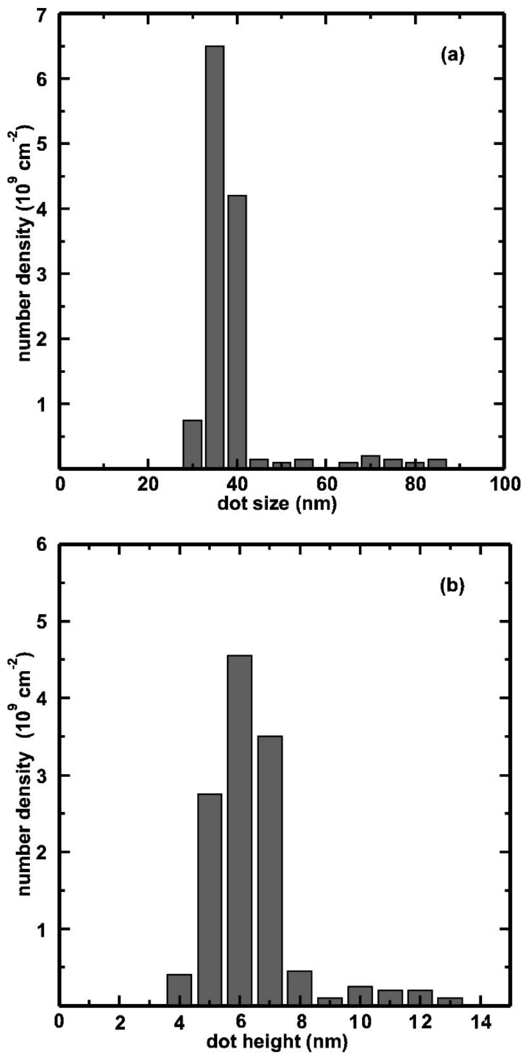

FIG. 6. (a) Size and (b) height distributions of the QD's and the larger islands grown on the GaAs(113)B surface at 470 °C. The size is taken as the length at the base along [332] including the [112]B facet of the flat base.

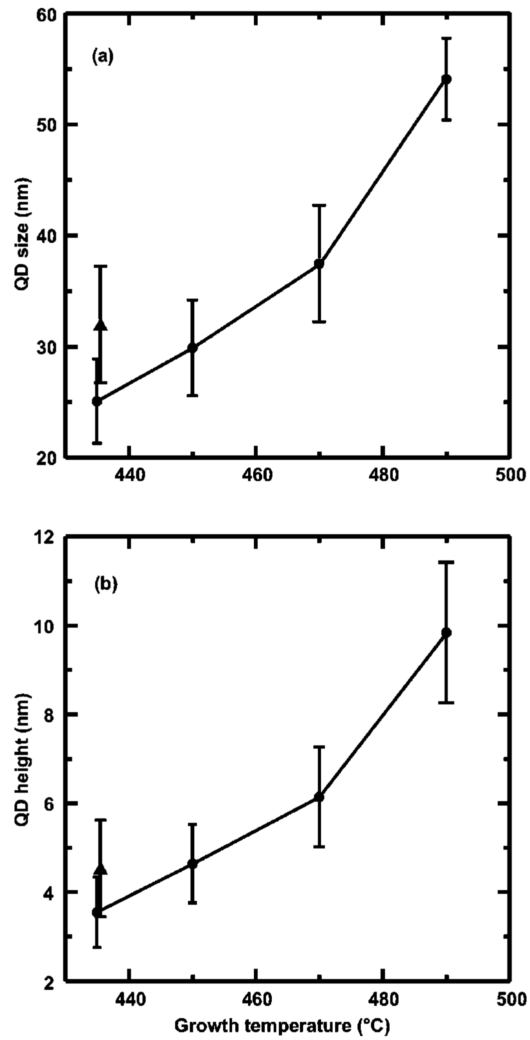

FIG. 7. Growth-temperature dependence of QD size and height. Triangles at 435 °C in (a) and (b) indicate size and height measured from samples post annealed by 1 min. Error bars are given.

tances between the QD's exhibit a rather large distribution. This indicates that the distance between the QD's is not dominated by a diffusion zone, out of which the material is collected for each dot. Instead, our observation makes it likely that the site distribution of the dots is governed by the statistics in developing growth nuclei. The increase in dot size with temperature may, therefore, indicate a decrease of the number density of critical nuclei, in line with the well-known fact that the size of the critical nucleus increases with temperature. The amount of material in the second InAs layer is then distributed quite equally among the given nuclei including some probability for the growth of larger incoherent islands.

Meanwhile, the QD formation was also modeled on thermodynamic grounds.[38,39] Here, the QD's are supposed to be in thermal equilibrium. The QD size is calculated from a balance between reduced strain energy and enhanced surface energy, which explains the uniform size of the QD's. The shape of the QD's could also be derived by energetics. It was reported that mainly the surface energy of each bounding facet determines the shape.[38] However, we note that the shape of the QD's on the GaAs(001) surface calculated in energetics was composed largely of the {011} and the {111} facets,[40] which is not in correspondence with a recent experimental result in which {137} bounding facets were observed.[26] Moreover, opposite to the experiments, the average QD size calculated by energetics decreases as the growth temperature increases due to entropy contribution.[41] For a 2D model it was shown by the same authors that the size distribution, immediately after growth, is controlled by kinetics, whereas the size distribution changes to thermal equilibrium after long-time annealing.[41] However, the question of, whether the growth mechanism is governed by kinetics or energetics, is still open. A discussion of further results concerning this topic can be found in a recent paper.[42]

IV. CONCLUSION

For MBE-grown InAs QD's on the GaAs$(\overline{113})$B surface, the size distribution is bimodal: Many small, coherent QD's of remarkably uniform size and some larger, presumably incoherent islands that are extended along [332] and vary significantly in size, are observed. Our earlier result on the shape of the QD's is fully reproduced here: {110}, (111)B, and vicinal (001) surfaces act as the main bounding facets. The shape of the QD's does not change significantly for growth temperatures between 435 and 490 °C. Post annealing increases the size and the height by about 30% and decreases the number density, but does not change the shape significantly. The incoherent islands have a similar shape as the QD's. The size and the height of the QD's increase monotonously from 25 to 54 nm and from 3.5 to 9.8 nm, respectively, whereas the number density decreases as the temperature increases. This is tentatively explained as due to a decreasing number density of critical growth nuclei with temperature.

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