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Increased InAs quantum dot size and density using bismuth as a surfactant

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We have investigated the growth of self-assembled InAs quantum dots using bismuth as a surfactant to control the dot size and density. We find that the bismuth surfactant increases the quantum dot density, size, and uniformity, enabling the extension of the emission wavelength with increasing InAs deposition without a concomitant reduction in dot density. We show that these effects are due to bismuth acting as a reactive surfactant to kinetically suppress the surface adatom mobility. This mechanism for controlling quantum dot density and size has the potential to extend the operating wavelength and enhance the performance of various optoelectronic devices.

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Self-assembled InAs quantum dots (QDs) on GaAs (001) have enabled important advances in optoelectronic devices.^{1–4} Further progress in QD-based devices will require improved control over the QD size, uniformity, and density, all of which influence the QD electronic states. The QD density and size are affected by a variety of growth conditions including the growth rate, substrate temperature, growth interruptions, and surrounding InGaAs quantum wells.^{5–10} For example, decreasing the InAs growth rate or increasing the substrate temperature during QD growth typically results in larger QDs due to an increase in the In adatom diffusion length. However, there is a concomitant reduction in QD density with these methods. An additional method to control QD size and density during growth is the use of a surfactant.¹¹ Antimony (Sb) has successfully been used as a surfactant during the growth of InAs QDs on GaAs, typically leading to increased QD densities and smaller QD sizes.^{12,13} Here, we have used bismuth (Bi) as a surfactant, because the larger Bi atoms do not incorporate into the InAs QDs or the surrounding GaAs matrix. Prior studies on surfactant-mediated epitaxial film growth have revealed that at typical growth temperatures, Bi segregates to the surface without incorporating into the epilayer, and acts as a reactive surfactant to kinetically limit the surface adatom mobility and improve surface smoothness.^{14–17} For example, the addition of Bi during the molecular beam epitaxial (MBE) growth of GaAs_{1–x}N_x and InGa_{1–x}As_x has been shown to reduce epilayer surface roughness and improve photoluminescence efficiency.^{18–20} For QD growth, in one case, the use of trimethylbismuth (TMBi) during the metalorganic vapor phase epitaxy (MOVPE) growth of InAs QDs in InGaAs quantum wells (QWs) was found to increase QD uniformity and size without Bi droplet formation or incorporation.²¹ Another MOVPE study showed an increase in QD density and a reduction in QD size with the introduction of Bi.²² For previous MBE-grown InAs QDs, introducing Bi simultaneously with InAs during QD growth resulted in improved QD uniformities, but lowered QD densities.²³ Thus, although the Bi surfactant has been found to consistently improve QD uniformity, the effects of Bi on QD densities and sizes have not been consistent.

Here, we reconcile these apparent contradictions by comparing QD growth with and without a Bi surfactant for a wide range of InAs depositions (2.3 ML–3.3 ML). We find that for lower InAs depositions (<2.6 ML), the Bi surfactant leads to an increase in QD size and uniformity, but a decrease in QD density. However, for InAs depositions ≥ 2.6 ML, using Bi as a surfactant enables an increase in QD dimensions and an improvement in QD uniformity, without the concomitant decrease in QD density as reported in prior studies. Correspondingly, there is a red-shift in the emission wavelength, a narrowing of the linewidth, and an improvement in the luminescence, suggesting that the Bi surfactant-mediated QDs have the potential to improve the performance of QD-based optoelectronic devices. We also demonstrate that this observed increase in QD density is due to the Bi surfactant decreasing the In adatom surface diffusion length during MBE growth, as is expected for a reactive surfactant.¹¹

Samples were grown by solid-source molecular beam epitaxy in a Varian Gen II system on semi-insulating (001) GaAs substrates. The As₂:Ga beam equivalent pressure (BEP) ratio was 15:1 and the As₂:In BEP ratio was 50:1. Buffer layers of 200 nm GaAs were grown at a nominal substrate temperature of 540 °C determined with band edge thermometry. The substrate temperature was then ramped down to 500 °C during the last 50 nm of GaAs buffer growth, followed by the growth of the first layer of InAs QDs at an InAs growth rate of ~ 0.05 ML/s. The QDs were immediately overgrown with GaAs, followed by a second layer of nominally identical QDs that was left uncapped to facilitate atomic force microscopy (AFM) studies of dot morphology. The two QD layers were separated by at least 50 nm of GaAs to prevent dot stacking and coupling.²⁴ For QDs grown with the Bi surfactant (Bi BEP $\sim 5 \times 10^{-8}$ Torr), the Bi shutter was opened during the last 5 Å of GaAs growth prior to InAs deposition in order to cover the growth surface with ~ 0.1 ML Bi, and remained open during all of the InAs QD growth. During this time, there was no observable difference in the reflection high-energy electron diffraction (RHEED) surface reconstruction between the samples grown with and without Bi.

The QD optical emission characteristics were investigated with room temperature photoluminescence (PL), at a pump power of 112 mW and a laser spot size of $\sim 32 \mu\text{m}$. The QD density and dimensions were characterized by tapping-mode AFM. A Veeco Dimension ICON AFM was used to obtain surface topography images at room temperature and in ambient atmosphere. A silicon probe tip with an average spring constant of 42 N/m, a resonance frequency of 320 kHz, and typical tip radius of 8 nm was used for proximal probing. Since AFM images are a convolution of the probe tip and the QD itself, the measured widths can be $\sim 8\text{--}10$ nm larger than the actual diameters of the QDs.^{25,26} To minimize tip-induced differences in comparing the island sizes and densities, all the samples in this study were scanned using the same AFM tip, and tip broadening was not observed after extensive scanning. It should be noted that the dimensions of the buried QDs will be different from those of the uncapped QDs that are presented here; unlike the surface QDs, buried QD dimensions are influenced by surface segregation of the In atoms and alloying of the InAs in the QD with the surrounding GaAs.^{27–29} Furthermore, the uncapped QDs could ripen even after growth has stopped, during the time it takes for the substrate to cool, unlike the buried QDs, which are immediately capped with GaAs. However, since the different sets of uncapped QDs were grown under nominally identical growth conditions, a comparison of AFM images can illuminate general trends in the QD distribution, under the influence of the Bi surfactant. To examine the structure of buried QDs, stacks of nominally identical QDs separated by 50 nm GaAs were grown, and the structure was examined using cross-sectional transmission electron microscopy (XTEM) to confirm the effect of the Bi surfactant on capped InAs QD growth. A Tecnai Osiris 200 kV microscope was used to image the stack of InAs QDs under the bright field $g = (002)$ two-beam condition.

Prior investigations of InAs QD growth in the absence of surfactants revealed that following the 2D-3D Stranski-Krastanov (S-K) growth mode transition, the QD density increased with increasing InAs deposition, followed by considerable mass transport between the QDs.^{30,31} Plan-view scanning tunneling microscopy images of uncapped QDs and XTEM images of buried QDs have suggested that with increasing InAs deposition, the combination of Ostwald ripening and diffusion of detached adatoms on the surface enables the growth of larger, dislocated islands at the expense of smaller QDs.^{32–37} These islands are typically asymmetric, and the number of dislocations in the islands increases with island size.^{38,39}

Topographical AFM images of the QDs grown without and with a Bi surfactant are shown in Fig. 1 for varying depositions of InAs, ranging from 2.3 ML to 3.3 ML. The bright circular features correspond to the InAs QDs. In addition to the QDs, the AFM images of samples grown without Bi reveal irregularly shaped features with diameters larger than 40 nm, which appear with a higher prevalence for the larger depositions of InAs, such as in Figs. 1(b)–1(d). These large features are dislocated islands of coalesced InAs.^{32,33} The AFM images of QDs grown without Bi revealed that increasing InAs deposition resulted in a decrease in QD density and an increase in the density of large, dislocated InAs islands

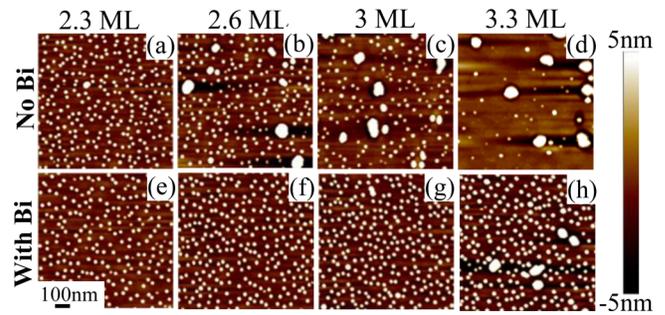


FIG. 1. Plan-view AFM topography of QDs formed from various depositions of InAs, without a Bi surfactant on the left, and with a Bi surfactant ($\sim 5 \times 10^{-8}$ Torr BEP) on the right. (a), (e) 2.3 ML InAs; (b), (f) 2.6 ML InAs; (c), (g) 3 ML InAs; and (d), (h) 3.3 ML InAs. The bright circular features correspond to the InAs QDs, and the large asymmetrical features correspond to dislocated islands formed from InAs coalescence. The gray-scale range displayed is 10 nm.

(Figs. 1(a)–1(d)). However, when Bi was present during QD growth, as shown in Figs. 1(e)–1(h), the formation of large islands was suppressed, indicating that the Bi surfactant significantly modifies the growth kinetics.

To quantify the influence of the Bi surfactant on the QD dimensions and densities, several AFM images of the QDs grown with and without a Bi surfactant were analyzed, and the results are summarized in Fig. 2. The densities of QDs (Fig. 2(a)) and dislocated islands (Fig. 2(b)) are plotted separately as a function of deposited InAs. Without Bi, increasing InAs deposition resulted in a higher density of dislocated islands and, as required by mass conservation, a correspondingly lower density of QDs. However, when Bi was used as a surfactant, increasing the InAs deposition resulted in a gradual increase in the QD density, with the density dropping slightly after ~ 3 ML InAs. These results suggest that the Bi surfactant is suppressing QD coalescence and ripening.

To illuminate the effects of the Bi surfactant on the In adatom surface diffusion, we compared InAs QDs grown with and without a 30 s growth interruption (under an arsenic flux) following 3 ML InAs deposition (Fig. 2(e)). Typically, growth interruptions during QD growth increase the In adatom surface diffusion length, thereby promoting QD coalescence and decreasing the QD density.^{8,9} In Fig. 2(e), the overall QD density decreases after a 30 s growth pause, but the reduction in density is limited when Bi is present during growth. More specifically, the density decreased by $\sim 31\%$ in the absence of Bi, but the reduction in density was limited to $\sim 15\%$ when Bi was present during growth, indicating that the Bi surfactant suppresses the diffusion of In adatoms during growth. It should be noted that the densities of the 3 ML InAs QDs grown without growth interruptions in Fig. 2(e) are higher than those in Fig. 2(a), because they were grown on a different MBE system. However, the above comparison of samples grown with and without growth interruptions still illuminates the general effect of the Bi surfactant on the In adatom surface diffusion.

In addition to enabling a high density of QDs, by suppressing In surface diffusion during InAs QD growth, the Bi surfactant also increased the typical QD size. The average QD diameters and heights are plotted in Figs. 2(c) and 2(d),

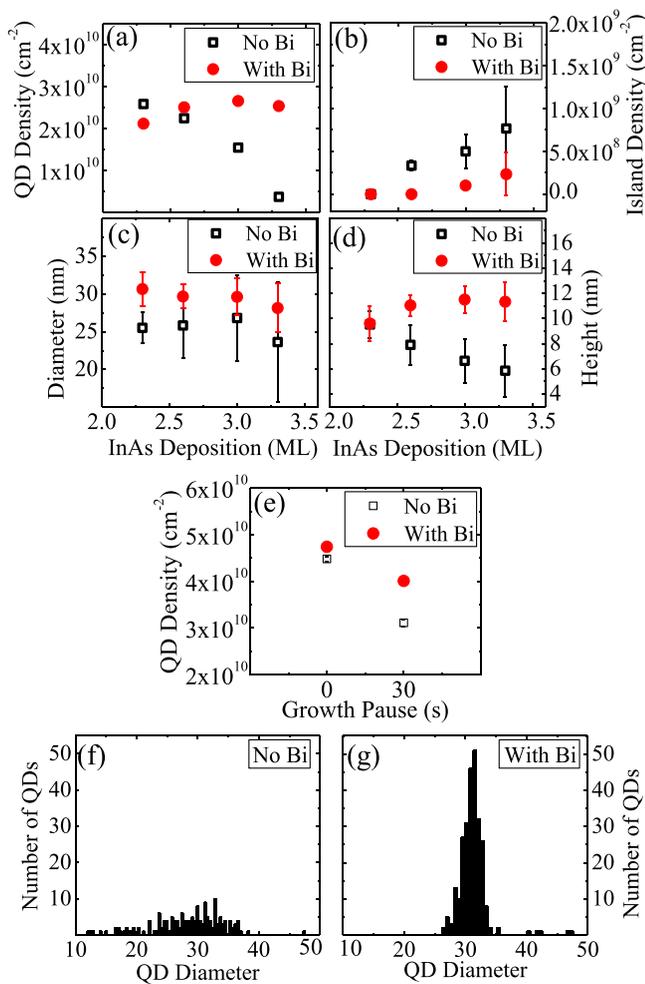


FIG. 2. Measured average (a) QD density, (b) coalesced InAs island density, (c) QD diameter, and (d) QD height, as functions of InAs deposition, both without and with a Bi surfactant during growth. The black open squares correspond to QDs grown without Bi and the red filled circles correspond to QDs grown with Bi ($\sim 5 \times 10^{-8}$ Torr BEP). Without Bi, the QD density drops due to an increase in the coalesced island density with increasing InAs deposition, but with Bi, island coalescence is suppressed so a high QD density can be maintained. The diameters and heights of the QDs are also typically larger when QDs are grown with Bi. The average QD densities with and without growth pauses are plotted in (e). Histograms of the size distribution for 3 ML InAs QDs are plotted in (f) for QDs grown without Bi and in (g) for QDs grown with a Bi surfactant, showing a significant improvement in QD size uniformity with Bi.

respectively. As shown in the figures, QD dimensions were larger when grown in the presence of Bi, for all of the InAs depositions under investigation. For example, for 3 ML InAs QDs, the presence of a bismuth flux increased the average QD diameter from 27 ± 6 nm to 30 ± 3 nm, and increased the average QD height from 7 ± 2 nm to 12 ± 1 nm. This is in contrast to QDs grown with Sb as a surfactant, which typically had smaller dimensions than QDs grown without the Sb surfactant.^{12,13} Indeed, the surfactant effects of Sb and Bi appear to differ dramatically.

To analyze the effects of the Bi surfactant on QD uniformity, histograms of the diameters of 3 ML InAs QDs grown without and with a Bi flux are shown in Figs. 2(f) and 2(g), respectively. The diameters were measured from representative $1 \mu\text{m} \times 1 \mu\text{m}$ AFM images containing a total of 170 QDs in Fig. 2(f) (no Bi) and 264 QDs in Fig. 2(g) (with Bi). QDs grown without Bi exhibited a wider distribution in the

diameters; for QDs grown with Bi, the size distribution was much narrower, with the majority of QDs having diameters in the range of 28–34 nm. Thus, the Bi surfactant can be used to fabricate larger and more uniform QDs, which is particularly useful when incorporating QDs in devices where a high density of homogeneous QDs is beneficial.

The suppression of large island formation in the presence of Bi was also observed in XTEM images, shown in Fig. 3. For both the 3 ML and 3.3 ML InAs QDs, the density of large, relaxed islands is reduced for QDs grown with Bi. Consequently, a higher density of QDs is maintained in the presence of Bi. In Fig. 3(a), the heights of the QDs grown with Bi are noticeably larger than the heights of the QDs grown without Bi. Thus, although the QD size and shape are significantly influenced by the capping process,^{27–29} the XTEM images reveal an improvement in QD density and size when the QDs were grown with Bi. Several XTEM images collected from different locations on the QD sample showed the same trends.

We have shown in Fig. 2 that the Bi surfactant increases the average QD dimensions and densities for most InAs depositions, but there are some deviations to the trend for InAs depositions lower than 2.6 ML (Fig. 2(a)). This deviation can be explained by the Bi surfactant suppressing In adatom surface diffusion during growth. Quantum dot formation is influenced by the structure of the underlying buffer layer,^{40,41} and since Bi modifies the surface structure of the GaAs buffer prior to InAs deposition,¹⁷ we can expect a difference in initial QD density. Typically, during S-K growth, InAs first forms a two-dimensional wetting layer (WL) on the growth surface, and as the thickness of the WL increases with the deposition of additional InAs, the strain in the film increases until it is relieved by the formation of QDs. However, when Bi is introduced prior to InAs deposition, the

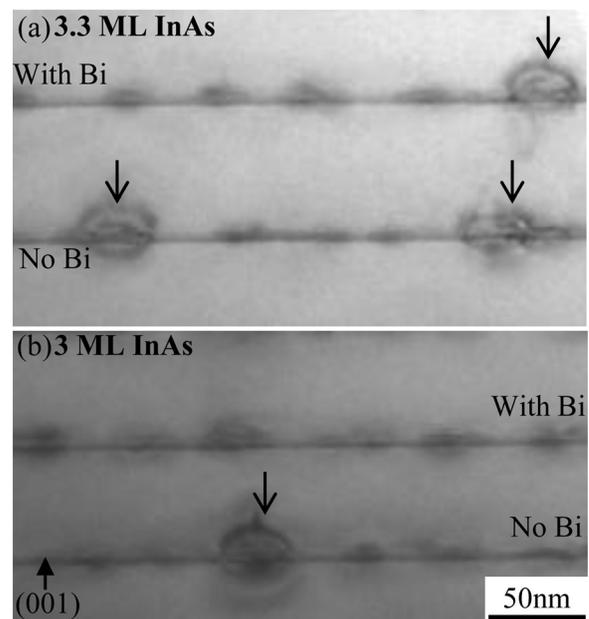


FIG. 3. Cross-sectional TEM images of (a) 3.3 ML InAs and (b) 3 ML InAs QDs grown with and without a Bi surfactant present during growth. Arrows mark the locations of large relaxed islands. Without Bi, the QD density drops due to the formation of relaxed islands, but with Bi, island coalescence is suppressed and a high QD density is maintained.

Bi atoms covering the growth surface suppress In adatom diffusion and WL formation, and QDs can form only in regions with a significant thickness of InAs. Thus, for low (<2.6 ML) depositions of InAs, the QD density is lower with Bi. However, with further increases in the InAs deposition, the QD density increases with the availability of additional InAs combined with the suppression of island coalescence.

The use of a Bi surfactant during QD growth also resulted in improved optical properties of capped QDs, as demonstrated by the PL results summarized in Fig. 4. For each sample, following multiple measurements under the same pump power, we found that, the variation in PL peak intensity was <10%, the variation in the PL emission wavelength was $<\pm 2$ nm, and the variation in FWHM was < 4 meV. The room-temperature PL intensity decreased with increasing InAs deposition for QDs grown without Bi (Fig. 4(a)). However, with Bi, the PL intensity increased with increasing InAs deposition (Fig. 4(b)). Furthermore, the PL spectra for QDs grown with a Bi surfactant showed enhanced emission from both ground- and excited-states, consistent with improved optical quality. These trends were observed for the entire range of investigated InAs depositions. As shown in Fig. 4(c), the PL emission intensity for QDs grown without a Bi surfactant decreased as the deposited InAs increased, likely due to the increase in the number of dislocated islands with increasing InAs deposition (Fig. 2(b)). On the other hand, with Bi, the emission intensity increased with increasing InAs deposition, likely due to the suppression of

QD coalescence that results in large, dislocated, islands. Furthermore, as shown in Fig. 4(d), the emission wavelength was in the 1.0–1.15 μm range, as expected for QDs grown under these conditions without Bi, but the emission wavelength was red-shifted with the use of a Bi surfactant during growth. This can be attributed to the increase in QD height with Bi (Fig. 2(d)), which is the strongest contributor to quantum size effects. The narrowing of the PL linewidth, observed in Fig. 4(e), can be attributed to the improvement in QD size uniformity with Bi, which is evident from the AFM size histograms shown in Figs. 2(f) and 2(g).

In conclusion, we experimentally demonstrated that with all other growth conditions held constant, surfactant-mediated epitaxy using Bi enabled an increase in InAs QD density, dimensions, and uniformity, due to the suppression of dislocated island formation with Bi. Correspondingly, there were increases in the PL intensity and emission wavelength and decreases in the PL linewidth. These trends were consistent for a wide range of InAs depositions, with surfactant effects becoming more prominent at higher InAs depositions. Future work will examine the role of the bismuth surfactant on the QD capping process in more detail. The mechanism presented here for increasing QD density and uniformity has the potential to improve the performance of a variety of QD optoelectronic devices. For example, an improvement in QD density can increase the differential gain and modulation bandwidth of QD lasers. For QD-based photodetectors, an improvement in QD density and uniformity has the potential to increase the intraband and intersubband absorption strength.

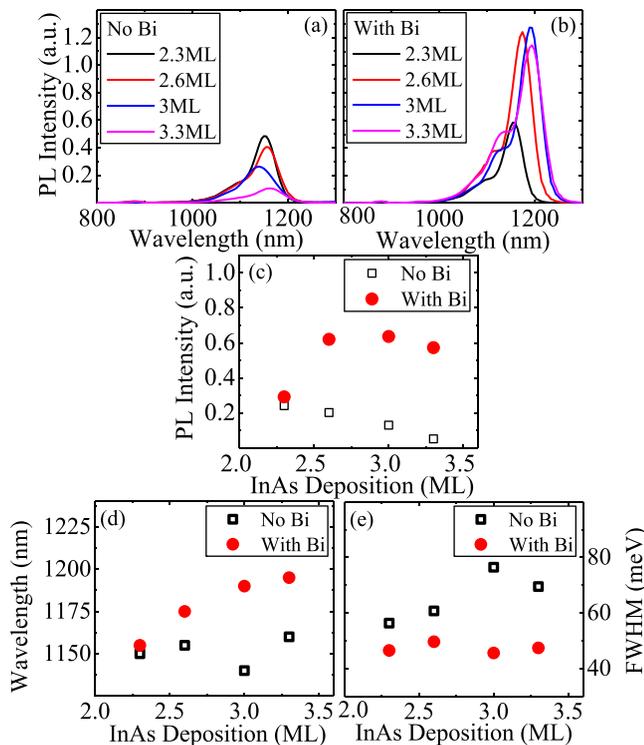


FIG. 4. Measured room-temperature photoluminescence spectra from InAs QDs grown (a) with and (b) without the Bi surfactant during QD growth, showing an increase in the intensity and emission wavelength combined with a narrowing of the FWHM with Bi. (c) PL intensity, (d) PL emission wavelengths, and (e) PL linewidths, for 2.3 ML, 2.6 ML, 3 ML, and 3.3 ML InAs QDs grown with and without the Bi surfactant. The black open squares correspond to QDs grown without a Bi surfactant and the red filled circles correspond to QDs grown with Bi ($\text{Bi} \sim 5 \times 10^{-8}$ Torr BEP).

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