The effect of two-temperature capping on germanium/silicon quantum dots and analysis of superlattices so composed

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It is well documented that quantum dots (QDs), grown and subsequently buried under silicon at temperatures greater than 400 °C, flatten into a pancakelike shape. Although QD arrays in Si superlattices have been studied for more than a decade, the process of flattening is not well understood. Here, we examine the process by which flattening occurs, using a two-temperature capping technique. Briefly, a 300 °C cold-cap layer is deposited, conformally coating the QD, followed by deposition of hot Si at 750 °C. Through this process, full or partial shape retention of the buried QDs can be selectively maintained. Interestingly, we found that QDs grown with this technique do not flatten in the traditional way. In fact, the QD truncates without any associated base spreading. The material from this truncation fills in the valleys, thereby flattening the surface of the sample. We analyzed this truncation by growing a series of samples under conditions of varied cold cap thicknesses and base growth temperatures. In addition, we found annealing at or above 650 °C sufficient to induce this truncation, while adatom flux was not required for truncation. We examined the samples using cross-sectional transmission electron microscopy (TEM) to determine the degree of shape retention and location of the truncated material. This technique also provided the optimal growth conditions for two additional studies: photoluminescence (PL) analysis, which is used to determine how shape retention affects recombination efficiency. In sharp contrast to earlier studies, PL data indicated that while shape retention could be maximized by low temperature overgrowth, this was accompanied by an unacceptable loss in luminescent intensity. This decrease suggested quenching by QD surface and/or overlayer traps. To retain both QD shape and PL intensity, a low/high temperature procedure was identified. These samples were then analyzed by TEM for the appearance of dislocations, degree of shape retention, and alignment/self-segregation of upper layers compared to lower layers. The findings from all of the above techniques help to elucidate the properties of QDs in complicated structures and imply possible techniques to refine current technological practices. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2203203]

I. INTRODUCTION

In order to generate functional quantum dot (QD)-based integrated circuits (ICs), many challenges need to be overcome. For example, following growth, SiGe QDs require capping with a layer of Si before further processing can occur, a procedure that has been shown to alter QD function.[1] In fact, regardless of the requirement for further processing, the QDs must be capped in order to prevent contaminants and oxides from altering their function. Here, we define "function" as the ability to quantum mechanically confine the charge carrier, which can be measured by photoluminescence (PL).

In order to study how growth conditions affect quantum confinement, we grew a series of superlattice structures. A superlattice is a periodic, multilayered structure constructed by the growth of one material on a substrate, followed by burial in yet another material. Growth of QDs in a series of successive layers results in their self-ordering. Seminal theoretical work in the 1990s predicted that this type of QD growth would result in nearly identical placement of QDs in each successive layer due to strain and slow readjustment of position in order to minimize interaction energies.[2,3] Given enough layers, this process leads to a neatly ordered set of QDs. Further, interdot spacing was predicted to be governed by the thickness of the spacer layer between the QDs. This model has been proven in part, through experiments on tens of layers.[2,3] It is well documented, however, that Ge QDs flatten when they are capped with layers of hot Si (>550 °C).[4] This process not only disturbs the degree of QD shape retention but also generates an intermixing between the Si and Ge. Once intermixed, the QDs no longer retain all of their confinement properties; instead, their behavior

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becomes more akin to quantum wells.[1] This transformation significantly decreases the efficiency of any device based on these structures. Shape retention and clearly delineated regions of Si and Ge are required for maximum device efficiency. In addition, we observe the reordering of buried QDs in superlattice structures to the lowest energy state, as well as their alignment with lower layers.

Kubler *et al.* were the first to show that shape retention could be achieved.[5] They did this by burying QDs with a layer of Si that was deposited at a temperature significantly less than the growth temperature. Hereafter this will be referred to as a cold cap. In their experiment, Kubler *et al.* used a cold cap of 100 °C to bury hut clusters. The resultant structure was examined by transmission electron microscopy (TEM), where it showed full shape retention in the buried layer. Rastelli *et al.* found that Si capping of QDs at a temperature less than 450 °C also resulted in shape retention.[4] More specifically, they grew cold caps over QDs at 300 °C, examined the resultant restructure with TEM, and found the deposited Si conformally coated the QDs. When QDs were capped with 30 nm of Si at either 300, 450, or 550 °C, followed by addition of a 30 nm Si hot cap at 550 °C, shape was either fully retained, partially retained, or flattened depending upon the cold cap temperature, respectively. This illustrates the association between capping temperature and shape retention of buried QDs. Similar experiments carried out by Stoffel *et al.* using a PL analysis technique revealed a temperature-dependent redshift in emission wavelength.[6] They concluded that this dependence resulted from intermixing of Ge with the capping layer. Here, we examine the effects of two-temperature capping under a variety of conditions. In contrast to most earlier work, our goal was to retain QD shape without the optical degradation often associated with lower temperature growth.

II. EXPERIMENT

In this work, Ge and Si films were grown via molecular-beam epitaxy (MBE) on (001) Si substrates. The experiments were carried out using a custom-built VG 90S double-chamber UHV-MBE system at the University of Virginia (UVa). Before sample growth, a modified piranha clean procedure[7] was employed to clean and hydrogen passivate the substrates. The resulting hydrogen-terminated layer, along with any surface oxide, was desorbed at 775 °C. A typical sample growth used in this study involved the deposition of a 1000 Å Si buffer layer at a starting temperature of 775 °C. The substrate temperature was progressively lowered to the Ge growth temperature of 650 or 750 °C during deposition of the buffer layer. This procedure allows for immediate growth of the Ge epilayer, thereby eliminating any chance of contamination buildup. Following Ge deposition, the sample was cooled to the cold-capping-layer temperature, generally 300 °C, and the cold cap was deposited. The wafer was then reheated to the temperature of the original Ge deposition (650 or 750 °C) for annealing or deposition of a hot cap. This process was subsequently repeated when growing superlattice structures, restarting with the deposition of Ge. The base pressure in the chamber prior to growth was typically 2×10^{-10} Torr. Once removed from the MBE, the sample was cleaved into pieces for analysis by atomic force microscopy (AFM) for uncapped samples or TEM and PL for capped samples. AFM was performed on a Digital Instruments Dimension 3100 Nanoprobe AFM.

For the PL arrangement, excitation was provided by a chopped Ar^+ laser operating on 514 nm. The Si/Ge QD samples are inserted into the cryostat (Optistat CF1204, from Oxford Instruments) and lowered to a temperature of ~3 K. Lenses L1 and L2 focus the PL signal to the grating monochromator (model: SPEX 270M). A filter is used to block the unwanted laser light. Photoluminescence was measured using a Ge liquid nitrogen-cooled detector (model: EO-817L) from North Coast Scientific Corp. using lock-in techniques.

III. RESULTS

A. Size and shape uniformity of uncapped QDs

A high degree of QD shape and size uniformity is required in order to assess changes due to capping conditions. By varying growth temperature and analyzing the resultant QDs by AFM and PL, we determined the optimal conditions for achieving uniformity. As shown in Figs. 1 and 2, as growth temperature increases, the average QD size and degree of uniformity also increase. Although uniformity was increased by annealing for 30 min following growth at 650 °C [Figs. 1(b) and 2(b)], the samples created at 750 °C [Figs. 1(c) and 2(c)] were superior. PL of QD samples confirmed this, showing that the 750 °C samples demonstrate a higher average energy and significantly reduced full width at half maximum PL response than that observed for samples grown at 650 °C (Fig. 3). These observations led us to use a 750 °C growth temperature for our studies.

B. QD shape retention following capping

Rastelli *et al.* showed that domes, cold capped with varying Si thicknesses at 300–350 °C, fully retain their shape. It was unknown, however, whether or not these QDs also retained their functionality. To address this, we first confirmed the shape retention results. As shown in Fig. 4, QDs capped with a conformal coating of Si under similar conditions to those employed by Rastelli *et al.* (cold cap of 20 nm and growth at 300 °C) demonstrated full shape retention. We then grew samples as above but followed by a hot cap of Si at 750 °C. As was the case for Rastelli *et al.*, we observed full shape retention under these conditions.

Rastelli *et al.*, however, only examined a limited set of growth conditions. We were interested in determining whether similar shape retention would be observed under a variety of cold cap thicknesses. Thus, we next explored the effects of varying cold cap thickness, from 0.86 to 200 nm, on QD shape retention. Figure 5 shows that, as the capping-layer thickness decreases, the QDs truncate. This truncation process is different from the flattening process observed for non-cold-capped QDs in that the bases do not spread when the QDs become truncated. Instead, the nontruncated portions of the QDs are completely stabilized by the cold cap. The height at which the truncation happens appears to be determined by the combined layer thickness of the Ge and

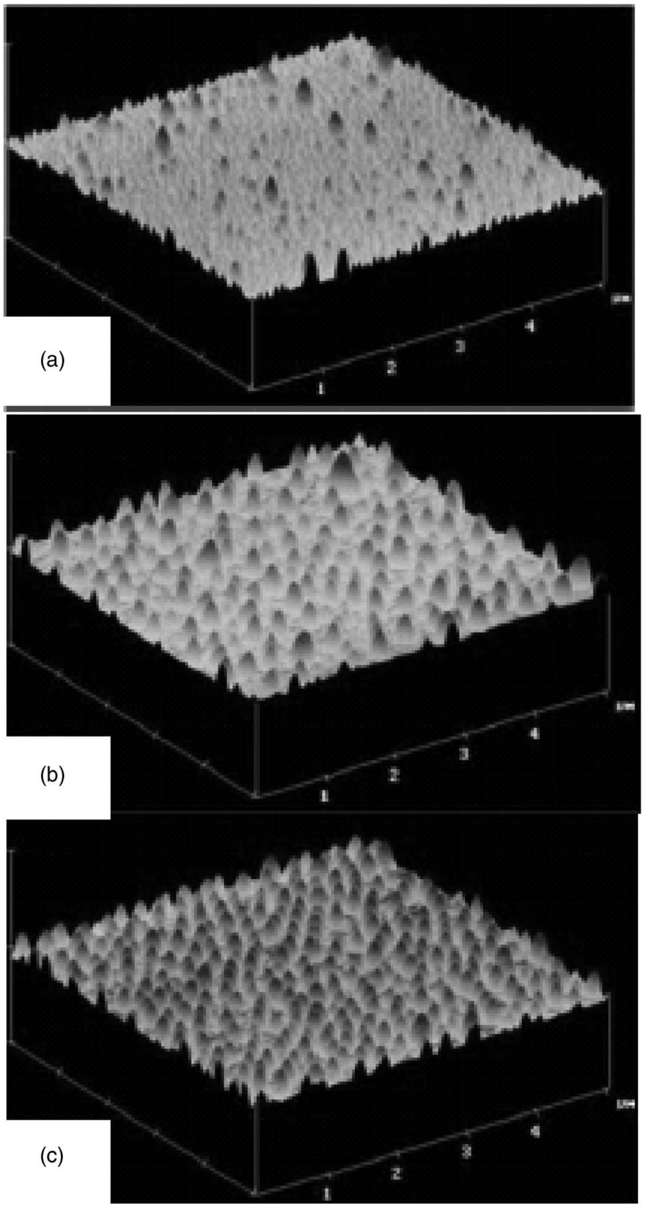

FIG. 1. AFM-generated 3D topology plots of the surface of a sample under conditions of varying growth temperature. The sample grown at 750 °C (c) had a greater degree of size and shape consistency than those grown at 650 °C (a), even after a 30 min annealing cycle (b).

cold cap layers, regardless of topology. For example, 1.4 nm of Ge plus 10 nm of cold Si yields a truncation 11.4 nm above the substrate. This was further confirmed by experiments where the samples were annealed after cold cap deposition (i.e., truncations still occur at the predicted location), indicating that the deposition of hot Si is unrelated to the flattening process (Fig. 6). Further, the Ge dispersed by the truncation spreads out along the interface, intermixing with Si. These observations led us to hypothesize that as temperature increases for hot capping or annealing, the upper capping layers of Si relax into valleys in order to lower strain energy (Fig. 7). This process continues to expose new layers to surface diffusion, which also relax, until the surface planarizes or the Ge is exposed. If the surface height reaches equilibrium prior to Ge exposure, the truncation stops, leaving a relatively planar surface. If the Ge is exposed, however,

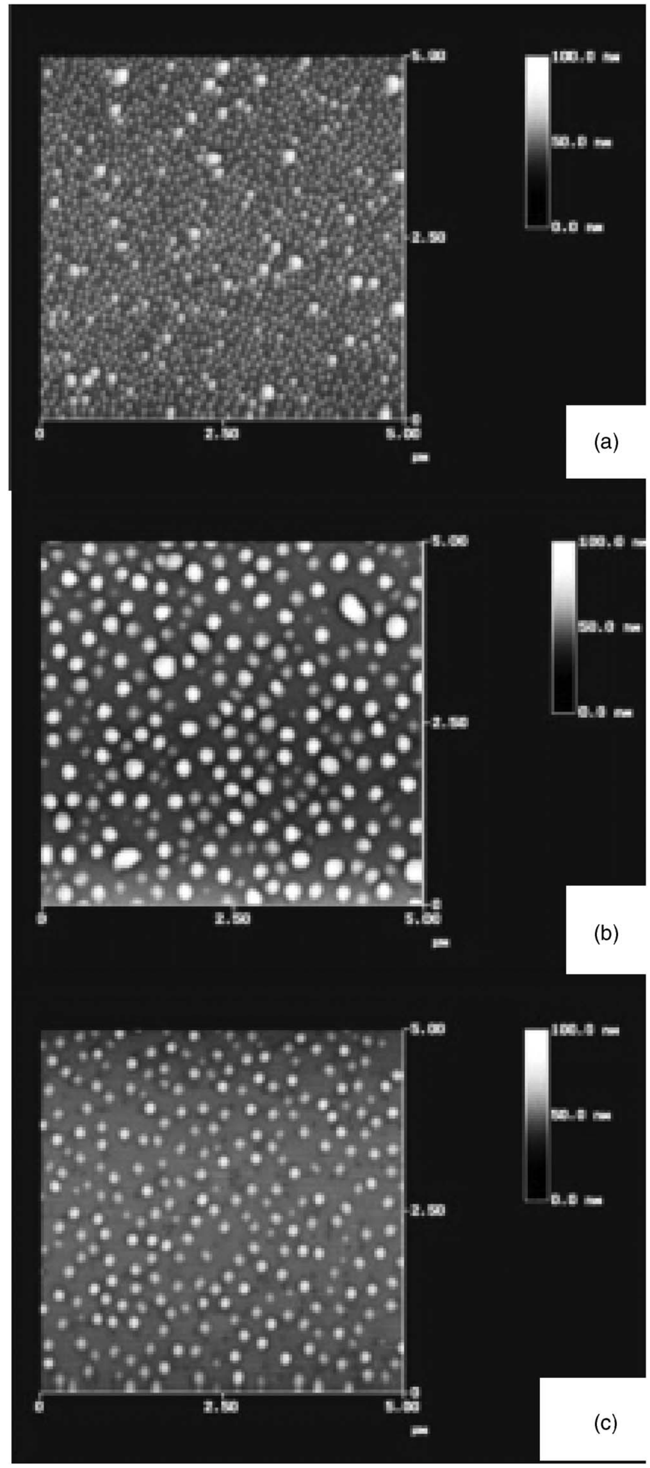

FIG. 2. AFM-generated 2D topology plots of the surface of a sample under conditions of varying growth temperature. The sample grown at 750 °C (c) had a greater degree of size and shape consistency than those grown at 650 °C (a), even after a 30 min annealing cycle (b).

then the energetics of intermixing also contribute, leaving the characteristic plumes (diffuse Ge along the interface) observed in some TEM images (data not shown). This intermixing could play an important role in lowering strain energy by both widening the width of the quantum well and decreasing the slope of its walls. When these structures with intermixed Ge are incorporated into superlattices, they allow for higher Ge-loading fractions.

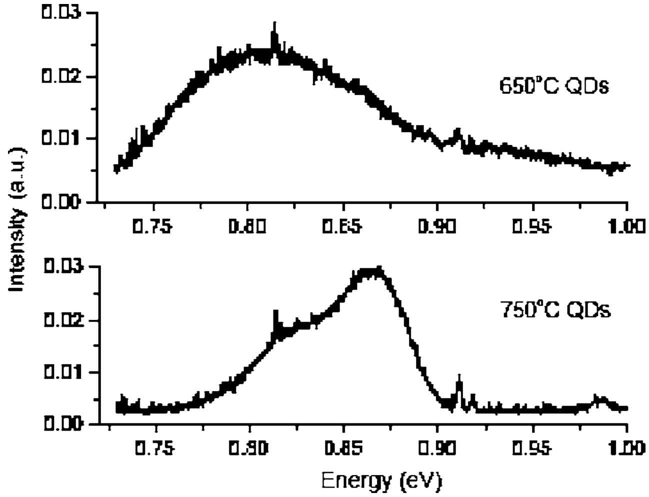

FIG. 3. PL response for similarly prepared QD samples grown at 650 and 750 °C. The sample grown at 750 °C has a greater intensity and larger average energy PL response, indicating that the dots are more uniform in size and distribution than that observed for the samples grown at 650 °C.

C. Two-temperature superlattices and QD self-ordering

While uniformity is increased by higher growth temperatures, self-ordering observed in superlattice structures may be required to achieve the QD density and degree of uniformity needed for QD architectures. As discussed earlier, without a cold cap, pancake-shaped QDs demonstrate a high degree of intermixing. This leads to a lower amount of strain energy to guide nucleation on subsequent layers. As noted earlier, however, superlattice structures, where self-ordering was observed, have been extensively studied. In these superlattice structures, the QDs reorder to minimize energy. This process involves shifting of positions within columns (as described below) into more regular arrays. If the columns are too close, they may either bend away from each other or one will disappear. If the columns are too far apart, however, a new column will form. Figure 8 depicts a superlattice where the initial layer is comprised of tightly packed QDs. With increasing layers, the QDs rearrange, moving farther apart when close together and closer together when far apart. One column disappears entirely within this cross section. Figure 9 shows the opposite scenario, where the QDs originate at distant locations. The QDs rearrange such that they become more evenly spaced and new columns arise in areas where the QDs are too distant.[8]

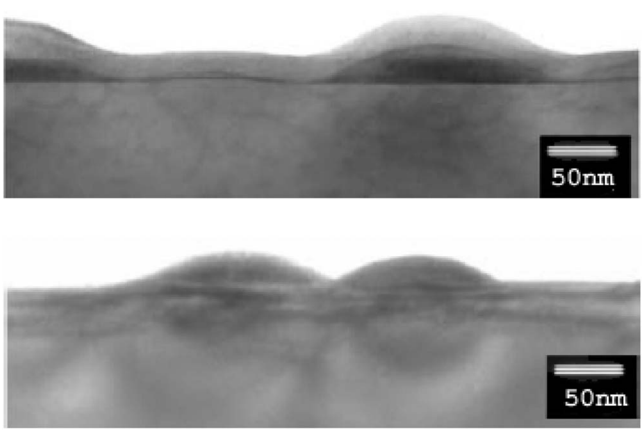

FIG. 4. (Upper panel) Cold capping with Si conformally coats Ge dots. Ge dots (dark domes) retain their shape despite capping with 20 nm of Si at 300 °C. The thin Ge wetting layer is also visible in this image (thin dark line that appears between dots). (Lower panel) Uncapped Ge dots exhibit a domelike shape.

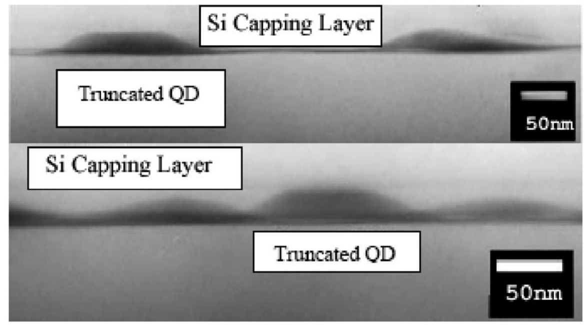

FIG. 5. The tops of taller QDs are more likely to truncate. In the upper TEM image, the QD on the left, which was originally taller than the one on the right, has been truncated at the height of the cold/hot cap interface. The dot on the right is shorter than the interface line, so it maintains its rounded top. In the lower image, the larger QD, which was in the center, became truncated.

D. Photoluminescence of buried quantum dots

As mentioned earlier, Rastelli *et al.* demonstrated conservation of shape under cold-capping conditions but failed to examine the functionality of these buried QDs. This functionality can be tested by examining the optical properties of the buried QDs. The QD confinement potential and the sample's crystalline quality, as determined by PL, indicate the optical utility of these QDs.

We, therefore, grew QDs under several capping conditions (single-layer study) or superlattices varying in layer number (multilayer study) and analyzed these samples for functionality by PL. The single-layer study was designed to observe the effect of capping layers, both cold and hot, on shape retention and QD confinement potential. Nine types of samples were grown for this study: no cap, cold cap only, 20 nm cold cap+20 min anneal, 50 nm cold cap+20 min anneal, hot cap only, 1 nm cold cap+hot cap, 10 nm cold cap+hot cap, 20 nm cold cap+hot cap, and 30 nm cold cap+hot cap. All cold caps were grown at 300 °C and all hot caps and anneals were performed at 750° C.

As shown in Fig. 10, uncapped QDs demonstrated a very poor PL response, which was presumably due to the availability of nonluminescent recombination sites at the surface of the dots. As previously mentioned, a QD must be buried to prevent contaminants, which can act as recombination sites,

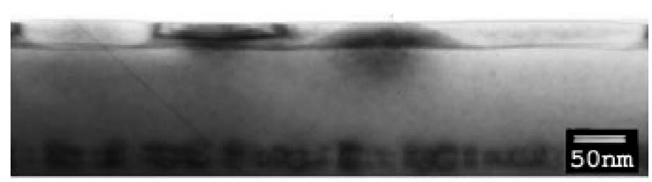

FIG. 6. Samples annealed after cold capping are also truncated if the QD height is greater than that of the average layer thickness.

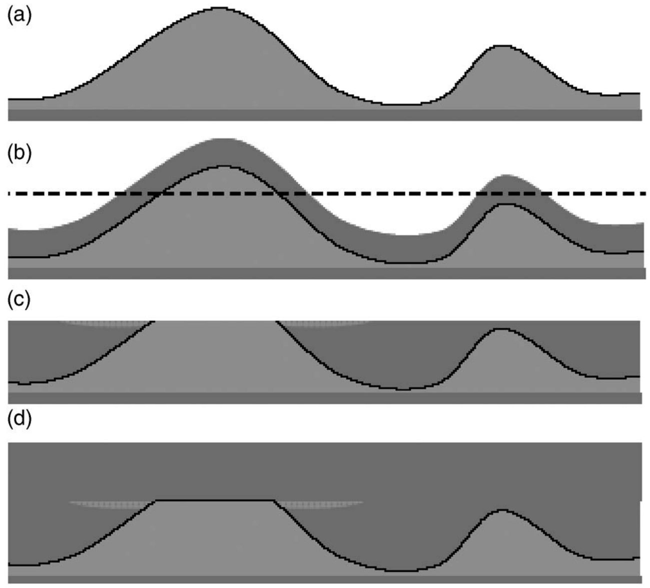

FIG. 7. Truncation model. (a) Two QDs of unequal size. (b) Cold capping of the QDs, which conformally coats them. The dashed line represents the average surface height of the deposited materials. (c) Planarization to the average surface height (dashed line) of the deposited material from annealing after cold capping. QDs taller than this line are truncated. (d) Truncation of QDs at the same spot as shown in (c) resulting from hot capping after cold capping.

from accumulating on the surface. Cold capping of dots failed to improve the PL response over what was observed in the absence of a cap altogether (Fig. 10). This suggested that the cold cap itself was of poor crystalline quality and, therefore, had a large number of recombination sites. The crystalline quality of the cold cap was improved by annealing the sample after the deposition. This annealing only provided a marginal improvement in PL response, however, with the thicker cold cap, producing a slightly better response (Fig. 10). This arises because cold caps thinner than the height of the dots result in QD truncation, leaving part of the QD surface exposed, thus enabling recombination. PL intensity increased upon deposition of hot Si on top of the QDs, indicating that, despite some intermixing, the high-quality crystalline hot cap does not posses a significant number of recombination sites (Fig. 10). When QDs are first cold capped, followed by a hot cap, the intensity improved over what was observed for just the hot cap, suggesting removal of the cold-cap recombination sites (Fig. 10). Additionally, we found that the average confinement energy in the QDs to be increased in these samples, indicating a reduction in intermixing. When hot capped, we observed that as the thickness of the cold cap layer decreases, the PL intensity increases (Fig. 10). This can be explained by several possible mechanisms: First, the repair of defects that occurs during hot capping of the cold caps may not be as effective for thicker cold caps, leaving a greater number of recombination sites inside the thicker samples. Alternatively, the average density of recom-

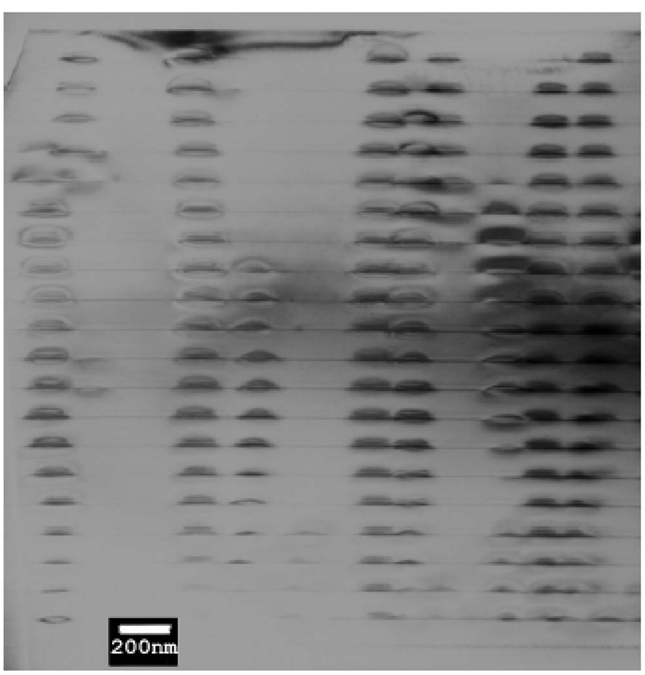

FIG. 8. TEM cross section of a QD superlattice where the template layer has a high density of QDs. As the number of layers progresses, columns realign to distance themselves; when this is not possible, they cease to replicate. Each successive layer of QDs aligns itself to the previous while trying to reorder into a lower energy configuration.

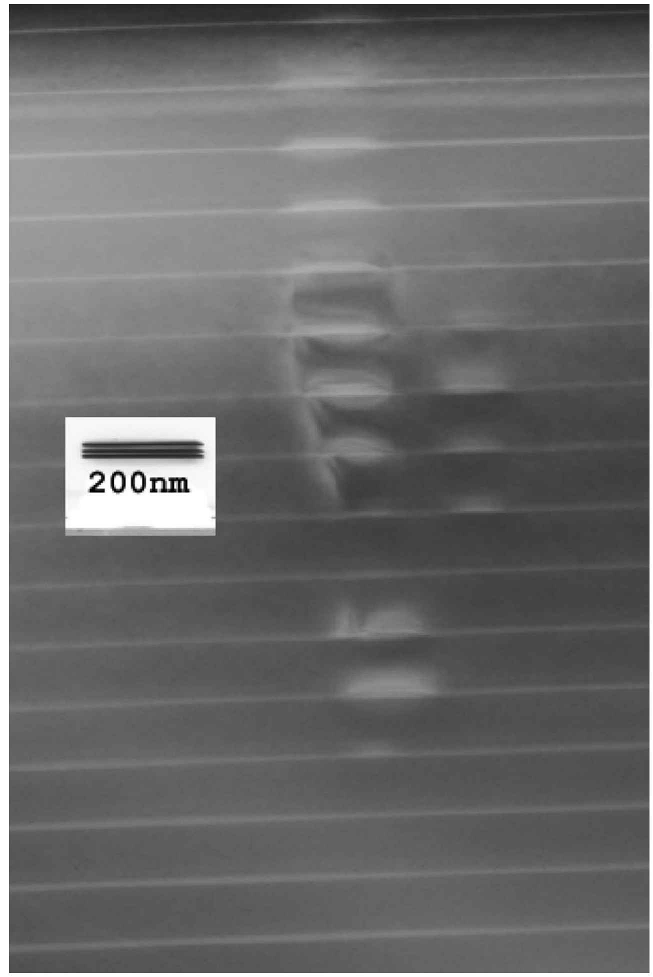

FIG. 9. TEM cross section of a QD superlattice where the template layer has a low density of QDs. As the number of layers progresses, new columns of islands form to lower the surface energy. There was not a QD at this position in the template, but it was energetically favorable for a new column to form. Once a new column is established, each successive layer of QDs aligns to the previous while trying to reorder into a lower energy configuration.

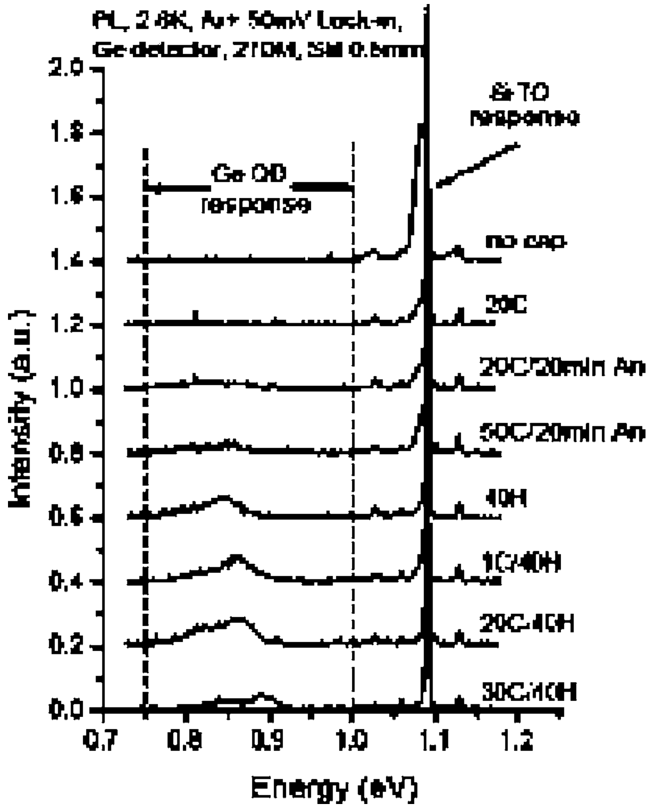

FIG. 10. Comparison of PL response of samples grown with a single Ge layer and capped under various conditions. PL response for dots without a cap or a cold cap is negligible. Annealing a cold-capped sample slightly revives PL response, but it is still less than a QD sample that is hot capped. When cold capped and then hot capped, the thickness of the cold cap is inversely proportional to the PL response. The PL response is greatest for the sample with a 1 nm cold cap topped with a 40 nm hot cap. The numbers next to each plot indicate the thickness of a layer, while C indicates a cold cap, H indicates a hot cap, and An indicates an anneal for the times noted.

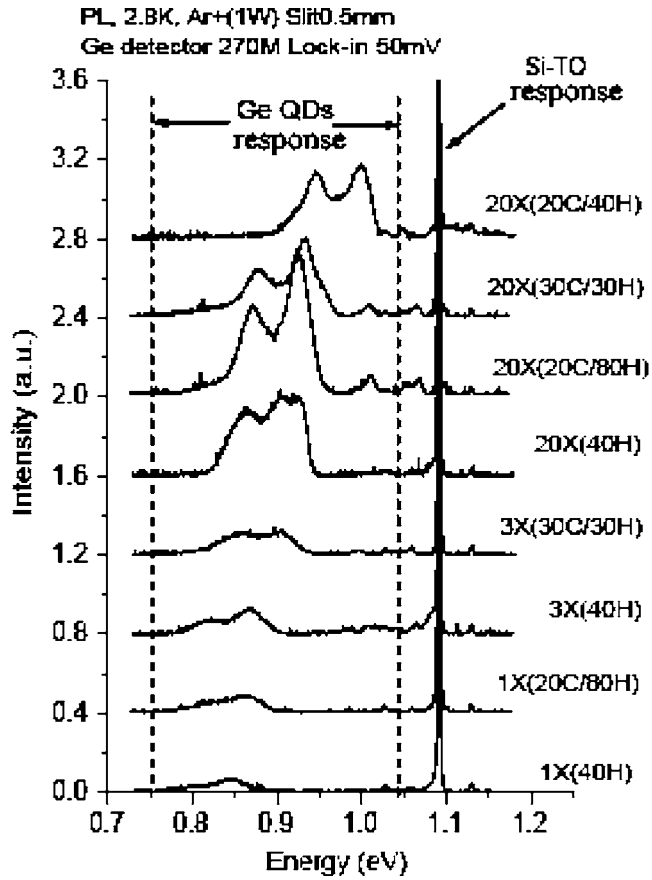

FIG. 11. Comparison of PL response of multilayered samples grown with or without cold and hot caps. Increasing the number of layers increases the PL response. Having a cold and a hot cap always yields a higher PL response than that obtained with a hot cap alone. The 20-layer sample without dislocations, 20C/80H, demonstrates the greatest PL response. The sample with the thinner total layer thickness and a low dislocation density, 20C/40C, has the smallest dots and, therefore, the highest average energy PL response. 1X, 3X, and 20X represent 1, 3, and 20 layers; the numbers before the C and H represent the thickness of the cold and hot caps, respectively.

bination sites may remain constant throughout the material, suggesting that as thickness increases, the possibility for recombination increases proportionately. On the other hand, this increased PL response for thinner cold caps might result from a greater size uniformity induced by the truncation of the QDs, shown in Fig. 6. This figure illustrates the situation where a large QD is truncated while a smaller QD is not, thus increasing QD size uniformity. This uniformity decreases the spread of the energy emissions from the QDs, increasing the PL intensity.

The self-organization described in the literature for superlattice structures should yield PL responses of increasing intensity and decreasing energy spread as the number of layers increases, because there are more dots to respond and the dots are more uniform in both size and shape. We next sought to test this by growing samples of multiple layers and analyzing the resultant superlattices by PL. The base conditions for these studies were a 20 nm cold cap and a 40 nm hot cap, except where noted. In addition to increases in PL due to enhanced uniformity induced by superlattice formation, intensity also increases proportionally with the number of layers, as these additional layers of dots luminesce as well. In order to separate these two mechanisms of increasing PL, inherent in superlattice formation, from that which results from two-temperature capping, we grew superlattices with only hot caps as control. For specific numbers of layers—whether 1, 3, or 20 layers—the intensity of the QD PL response increased and the average emission energy increased for samples grown with cold and hot caps, when compared to those grown with hot caps only (Fig. 11). As the number of layers increased, the QD PL response broke into two distinct peaks. This differentiation of peaks is due to the two possible recombination paths for the excitons. The higher energy peak is the no-phonon recombination and the lower energy peak is the so-called phonon-replica peak, which represents the phonon-assisted recombination of the exiton. This is further evidenced by the energy spacing of the peaks, as seen in Fig. 12, which corresponds to the reported Ge–Si transverse-optical (TO) phonon energies.[9] We have done both pump-power and temperature dependence PL measurements on one of the multiple Ge layer samples where the lower energy band is a phonon replica of the no phonon (NP), as shown in Figs. 13 and 14. Note that these are two-layer samples. For both the power dependence and temperature dependence, the ratios of NP to phonon replica are independent of pump power or temperature, ruling out the explanation as ground

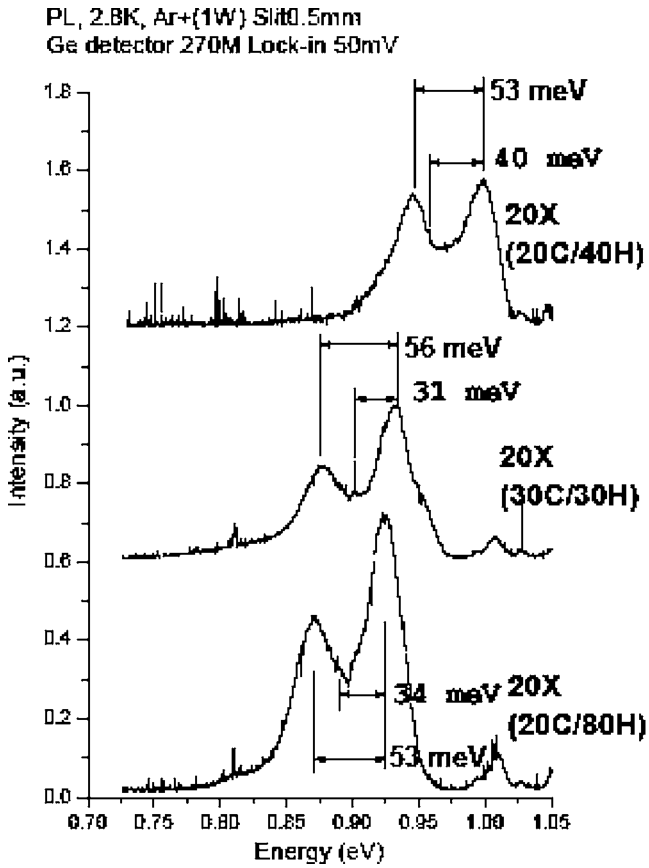

FIG. 12. The photoluminescence response from the QDs indicates two types of recombination events: one purely photonic at a higher energy and one that is phonon assisted at a lower energy. This effect is called phonon replica. Shown on the three plots in blue is the energy spacing between the two main peaks. This spacing compares well with the previously observed Ge–Si TO phonon energy of 50 meV, see Ref. 9. Additionally, small peaks can also be seen in some of our data. The energy spacing for these small peaks to the no-phonon peaks is indicated in green and correspond well to the previously observed energy of 36 meV for the Ge–Ge TO phonon, see Ref. 9.

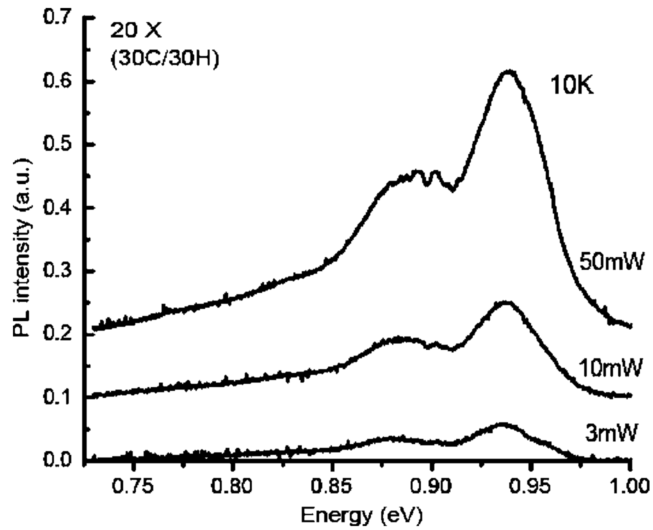

FIG. 13. The temperature dependence PL measurement, on one of the multiple Ge layer samples, illustrates that the lower energy band is a phonon replica of the no-phonon (NP) band and not due to temperature-dependent phenomena. Note that these are two-layer samples. The ratios of NP to phonon replica are independent of temperature, ruling out the explanation as ground and excited states in dots.

and excited states in dots. Additionally, Fig. 15 shows a cross section of a typical top and bottom layer in one of the stack structures, illustrating the lack of bimodality in the layers, thus removing that potential alternate explanation. If smaller QDs were grown, the radiative recombination efficiency should increase; therefore, the no-phonon peak should increase in size and the phonon-replica peak should diminish. Upon growth of infinite layers, the QDs would be expected to settle into one size, producing sharp peaks.

In addition, we examined the effect of alterations in the total capping-layer thickness, which changes the strain energy in the superlattice. For these studies, samples of 20 layers were grown under three different conditions: (1) 20 nm cold cap+80 nm hot cap, (2) 30 nm cold cap +30 nm hot cap, and (3) 20 nm cold cap+40 nm hot cap (Fig. 11). The sample grown under conditions of 20 nm cold cap+80 nm hot cap elicited the most intense PL response but also demonstrated the lowest average energy. This was expected, since the total layer thickness is greatest for this sample and, thus, the dots are larger and more distanced from each other. The QDs in this sample were slightly truncated and the entire lattice was dislocation-free. The sample grown under conditions of 30 nm cold cap+30 nm hot cap demonstrated a lower PL intensity but a slightly higher average energy. This sample resulted in dots with full shape retention but a large dislocation density. The third sample, grown under conditions of 20 nm cold cap+40 nm hot cap had slightly higher PL intensity and significantly increased average energy. This type of sample had a minor truncation, similar to what was observed for the first sample, but a low dislocation density. The second and third samples had the same total thickness, which implies that QD density should also be the same. However, dots tend to nucleate at dislocations. Therefore, in the second sample, the dislocation density was the limiting factor in the degree of QD organization, resulting in the different average energies observed for these

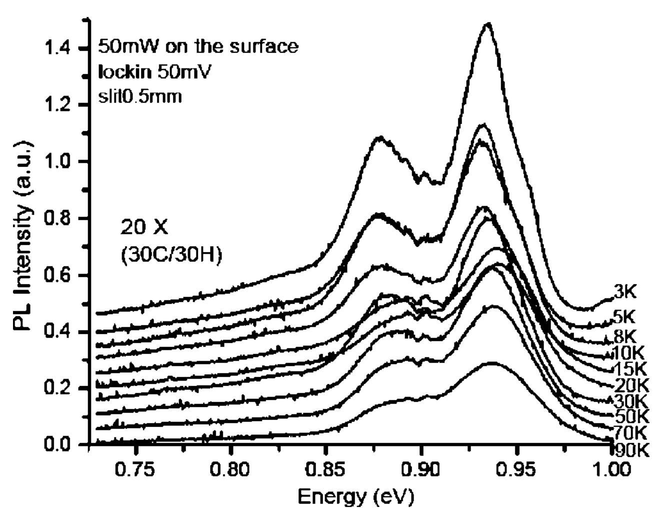

FIG. 14. The pump-power dependence PL measurement, on one of the multiple Ge layer samples, illustrates that the lower energy band is a phonon replica of the no-phonon (NP) band. Note that these are two-layer samples. The ratios of NP to phonon replica are independent of pump power, ruling out the explanation as ground and excited states in dots.

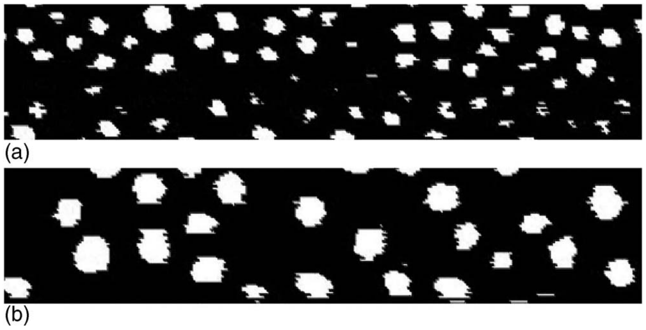

FIG. 15. Cross sections of the lower and upper layers of a QD super lattice under conditions of an initial high-density template. (a) The lower layer has a high density of randomly sized and spaced dots. (B) After 20 layers of growth, the QDs have become more uniform in size and are showing signs of self-organization. This figure indicates that there is no bimodal distribution in the upper layers, therefore indicating that this is not the cause of the double peak observed in the PL measurement.

two samples. An interesting example of this phenomenon was found in a sample where the QD density in the first layer was low, but individual dot size was very large. Dislocations spread out from the template-layer dots, nucleating successive layers of new dots in a treelike pattern (Fig. 16).

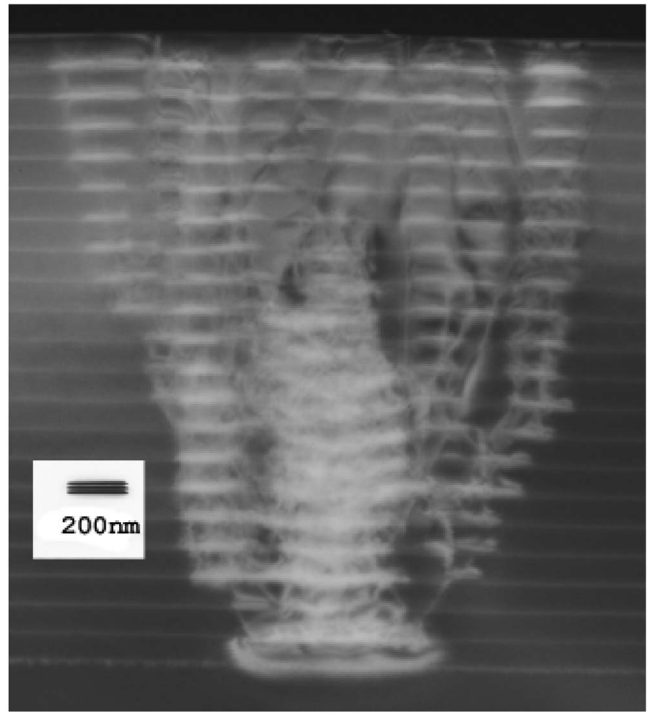

FIG. 16. Dislocations branching out from a large initial dot act as preferential nucleation sites for new layers of QDs, creating a treelike pattern.

IV. CONCLUSIONS

In order to integrate QDs into ICs, burial is required for further processing and protection from contaminants. Previous burial techniques resulted in intermixing of the QD Ge and the capping-layer Si, thus lowering the QD confinement potential and reducing its functionality. This intermixing was previously found to be avoidable by two-temperature capping, thus achieving full shape retention of the dots. The functionality of these dots, however, remained unknown. We found that full or partial shape retention of buried QDs could be achieved by varying the thickness of a deposited cold cap layer prior to the addition of hot Si. These layers of buried dots formed superlattice structures, demonstrating a remarkable degree of self-ordering by decreasing and increasing column number density, depending upon the density of the template layer. These results were further confirmed through three-dimensional (3D) tomographic reconstruction; results are shown elsewhere.[10]

The functionality of these buried structures was tested by PL, which showed that intensity increases proportionally with the numbers of layers, while the energy full width at half maximum decreases. The sizes of the QDs demonstrate a bimodal distribution, and truncation of dots increases PL response and decreases the energy spread. Additionally, we found that dots grown at higher temperatures have a tighter, higher energy response.

Capping is a critical procedure in device fabrication. These results illustrate that this process can be completed with full QD shape retention and maintenance of functionality. This technique allows one to opt for conditions that maximize shape retention or optical response, each of which have their benefits. Additionally, tomographic analysis of superlattices allows us to gain a greater understanding of self-ordering mechanisms that may prove crucial in the design of commercially viable, self-assembled, QD-based architectures.

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