Local structure of Ge quantum dots self-assembled on Si(100) probed by x-ray absorption fine-structure spectroscopy

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The local structure of Ge quantum dots (QD's) self-assembled on Si(100) has been probed by extended x-ray absorption fine-structure and x-ray absorption near-edge structure spectroscopies. We found that in the uncapped QD's, Ge is partially oxidized (~35%) while the other part (~40%) alloys with Si leaving only ~25% as a pure Ge phase. In the Si-capped dots the structure strongly depends on the growth temperature. For QD's grown at a rather high temperature of 745 °C, Ge is strongly intermixed with silicon forming a Ge-Si solid solution. The fraction of Ge atoms existing as a pure Ge phase does not exceed 10%. In the QD's grown at a lower temperature (510–550 °C), on the other hand, the Ge-rich phase clearly exists. The Ge-Ge bond length in the uncapped dots is close to the bulk value of Ge, indicating elastic relaxation of the misfit strain. The Ge-Si bond length in the capped QD's grown at 745 °C approaches the bulk value of Si, revealing compressive strain in the buried Si/Ge dot structures. In QD's grown at lower temperatures the Ge-Ge bond length equals 2.42 Å indicating a small compressive strain. We also found that the structural disorder is higher in the uncapped samples.

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I. INTRODUCTION

Fabrication of self-assembled quantum dots (QD's) due to the lattice mismatch of the substrate and the grown overlayer has been recently attracting much attention.[1] This growth mechanism, known as the Stranski-Krastanov mode, has been successfully applied to the fabrication of Ge/Si QD's which are of special interest due to the compatibility with Si technology.[2,3] Various groups reported that, depending on the growth conditions, QD's of different shape can be formed, such as pyramids, domes, and hut clusters.[4–9]

While the formation of the nanostructures has been unequivocally detected by scanning force microscopy and transmission electron microscopy, the local structure of the QD's is still a matter of controversy. While generally such QD's are referred to as Ge QD's, there has been growing evidence of intermixing of Ge with Si,[10–13] as in the case of Ge-Si superlattices.

Application of Raman scattering which is routinely used for the structural studies is, in the particular case of Ge/Si QD's, not very effective. Due to the presence of the two-phonon acoustic peak of silicon at almost exactly the same frequency as the main peak of (bulk) germanium, unambiguous determination of the Ge-Ge peak is usually difficult[14,15] although a peak due to Ge-Si intermixing is commonly observed.[16,17]

An alternative technique which allows for unambiguous determination of the local structure around the Ge species is x-ray absorption fine-structure (XAFS) spectroscopy. An advantage of this method is that the absorption edge of each of the constituent species can be probed separately even without long-range order. This technique has been successfully used to study Ge/Si superlattices.[18,19] We have now applied this technique to study the local structure of self-assembled Ge/Si QD's. In this paper, we report the microscopic structure and composition of QD's grown on Si(100) under different growth conditions.

II. EXPERIMENT

The samples were grown by solid source molecular-beam epitaxy (MBE) on Si(100) substrates under various condi-

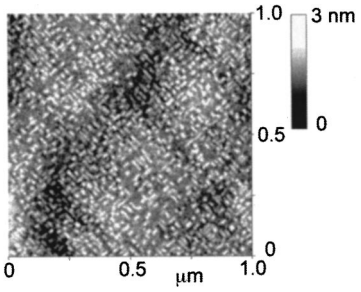

FIG. 1. A typical AFM image of a Ge/Si(100) QD's (the sample grown at 510 °C).

tions. The formation of Ge dots is ensured for all studied samples by *in situ* reflection high-energy electron diffraction (RHEED) showing clear spotty patterns due to the dot formation. The formation of Ge dots and their structural and optical properties for all growth conditions used were also studied by atomic-force microscopy (AFM), low-temperature photoluminescence (PL),[20] and, in part, by transmission electron microscopy (TEM). A typical AFM image is shown in Fig. 1.

Two samples for this work were grown at a temperature of 745 °C. On a 150-nm Si buffer, 6 monolayers (ML's) of Ge were deposited at a rate of 0.2 Å/s. On a Ge wetting layer of about 5-monolayer nominal thickness, Ge islands form in the Stranski-Krastanov growth mode. The islands are about 180 nm in diameter, about 12 nm in height, and with an areal density of 1×10^9 cm^{-2}. One sample with uncapped islands was cooled down immediately, the other sample was capped by 90-nm Si deposited at a rate linearly increasing from 0.05 to 0.5 Å/s. The capped sample reveals island-related photoluminescence at low temperature at an energy of 0.89 eV which indicates a Ge content of about $x=0.5$ in the center of islands.[20]

In order to investigate the role of the growth conditions, we have also studied the structure of the samples grown at lower temperatures. One sample was grown at 510 °C and had a nominal thickness of 7-ML Ge, the dots have the hut shape and are ~20 nm in diameter and 2 nm in height with 6×10^{10} cm^{-2} density. The other sample with a nominal thickness of 8.5-ML Ge was grown at 550 °C. The islands are larger (70 nm in diameter and 6 nm in height) and have a density of 5×10^9 cm^{-2}.

All samples with capped Ge islands reveal photoluminescence (PL) at low temperature with energies in the range of 0.79–0.89 eV. It is attributed to an indirect recombination of electrons within Si and holes in the ground state within Ge islands. Si alloying, strain relaxation, and/or quantum confinement of heavy hole states in growth direction result in transition energy much higher than the energy of about 0.5 eV estimated from the strained Si/Ge band offset values. Due to the similar influence of these effects, however, PL offers little information on the actual Ge content within the different types of Ge islands. A discrete level scheme of hole states localized in three dimensions and a level separation of about 37 meV was observed by admittance measurements from structures with small Si/Ge islands deposited at 510 °C.[21] In larger islands, the much lower energy separation of discrete levels was not resolved so far.

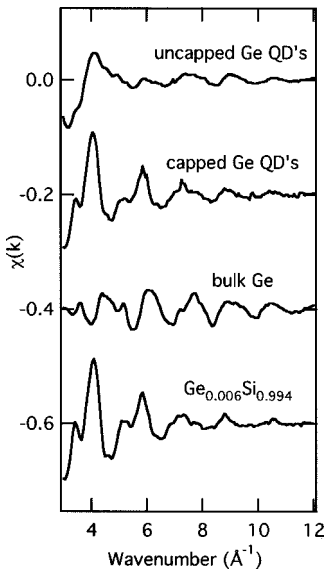

FIG. 2. Raw EXAFS oscillations of the uncapped and Si-capped Ge QD's compared with those for bulk Ge and dilute solid solution of Ge in Si.

As references, we have used a 300-nm-thick Ge film MBE grown on Si(100), a 6-nm-thick $Si_{0.7}Ge_{0.3}$ quantum well (QW) capped by 480-nm Si, and a dilute solid solution of $Ge_{0.006}Si_{0.994}$.

X-ray absorption measurements [EXAFS and x-ray absorption near-edge structure (XANES)] have been performed at beamline 13B of the Photon Factory at room temperature in a fluorescence mode. (The samples were exposed to air prior to EXAFS measurements.) For the fluorescence x-ray detection, we have used a 19-element pure Ge detector.[22] In order to increase the surface sensitivity, a grazing-incidence geometry has been used. More details on the experimental equipment can be found in Ref. 23. The data analysis was performed using FEFFIT of the UWXAFS3 (Ref. 24) and USTCXAFS2.0 (Ref. 25) codes. Theoretical amplitudes and phase shifts have been calculated using FEFF8.[26]

III. RESULTS

Figure 2 shows raw EXAFS oscillations for the uncapped and Si-capped QD's grown at 745 °C. Also shown at the bottom of the same figure for comparison are EXAFS oscillations for bulk Ge and for a very dilute solid solution of Ge in silicon ($Ge_{0.006}Si_{0.994}$).

One can immediately see that both measured spectra have very little in common with the bulk Ge EXAFS spectrum which has a maximum amplitude around 8 Å^{-1}. Much higher amplitude of the EXAFS oscillations in our QD's samples at lower k and rapid damping of the amplitude at higher k is a manifestation of high concentration of light elements as the first-nearest neighbors of Ge. One can also see a striking similarity between the spectra of the Si-capped Ge QD's and that of the dilute Ge solid solution which suggests that very strong Ge/Si intermixing occurs.

In order to analyze the data quantitatively, the k-weighted

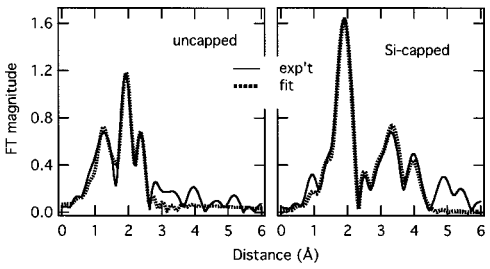

FIG. 3. Fourier transformed spectra for the uncapped QD's (left) and Si-capped QD's (right).

EXAFS oscillations $[k\chi(k)]$ were Fourier transformed (FT) into R space using a Hanning window and the k range from 3 to 12 $Å^{-1}$. The FT spectra are shown in Fig. 3. The two curves differ in several aspects. The uncapped sample (left) has a three-peak feature in the first shell contribution indicating that at least three different kinds of atoms are located around Ge atoms. The Si-capped sample (right), on the other hand, shows a characteristic FT pattern of a tetrahedral diamond structure. The positions of second- and third-nearest neighbors match with the simulated FT results as will be discussed later.

We first describe briefly the fitting results for the uncapped sample. The three features in the 1.5–2.5-Å range have been fitted using three shells or three phases, i.e., Ge-Ge, Ge-O, and Ge-Si first-neighbor correlations. The results of the fitting are also shown in Fig. 3. The obtained values for the average partial coordination number of Ge are: $N_{\text{Ge-Ge}}=1.4\pm0.9$, $N_{\text{Ge-O}}=1.5\pm0.9$, and $N_{\text{Ge-Si}}=1.8\pm0.8$. The disorder parameters (mean-square relative displacement, MSRD) are, $\text{MSRD}_{\text{GeGe}}\sim0.005$ Å^2, $\text{MSRD}_{\text{GeO}}\sim0.009$ Å^2, and $\text{MSRD}_{\text{GeSi}}\sim0.002$ Å^2, respectively. The bond lengths are $\text{BL}_{\text{GeGe}}=2.46\pm0.02$ Å, $\text{BL}_{\text{GeO}}=1.70\pm0.04$ Å, and $\text{BL}_{\text{GeSi}}=2.44\pm0.06$ Å, respectively. The large number of parameters in the fitting results in rather large uncertainties.

For the Si-capped samples, the strong similarity of the EXAFS oscillations for the capped sample and that of dilute Ge-Si solid solution suggests strong intermixing. Fitting of the first peak with both Ge-Ge and Ge-Si correlations yielded the Ge-Si fractional coordination number of 3.9 ± 0.3 and the Ge-Si bond length $\text{BL}_{\text{GeSi}}=2.37\pm0.01$ Å.

We have also performed a curve fitting for shells higher than the first-nearest neighbors and obtained the following values for the bond lengths: Ge-Si(1)$=2.37\pm0.02$ Å, Ge-Si(2)$=3.83\pm0.02$ Å, and Ge-Si(3)$=4.50\pm0.02$ Å. These values agree very well with the interatomic distances in the silicon crystal which are equal to: 2.35, 3.84, and 4.50 Å, respectively.

We now proceed to the dots grown at lower temperatures. Figure 4 (top) shows raw EXAFS oscillations for the two samples grown at lower temperature and their comparison with the spectrum for the reference QW. Figure 4 (bottom) shows the Fourier transforms of those spectra. The amplitude of the first oscillation (at $k\sim4$ $Å^{-1}$) and the amplitude of the first-neighbor peak decrease in the following sequence: QW, $\text{QD}_{550\,^\circ\text{C}}$, $\text{QD}_{510\,^\circ\text{C}}$ indicating a decrease in the fraction of Si surrounding the Ge atoms. The first shell (both Ge-Ge and Ge-Si correlations included) has been analyzed using

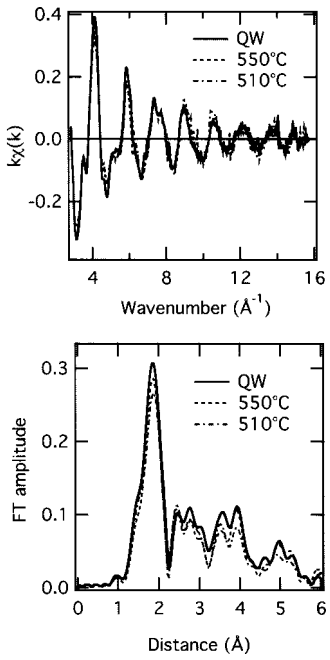

FIG. 4. Raw EXAFS oscillations for Ge QD's grown at 510 and 550 °C compared with those for the $Si_{0.7}Ge_{0.3}$ QW (top) and Fourier transforms of the EXAFS spectra (bottom).

USTCXAFS2.0 code and fitting was performed in k space (Fig. 5). The numerical results are summarized in Table I.

For the samples grown at lower temperatures, where the Ge-rich phase clearly exists, we have also performed polarized EXAFS measurements with the electric vector of the x-ray beam being either parallel or perpendicular to the growth direction. (This experiment was performed at BL12C at the Photon Factory[27]). The obtained values for the Ge-Ge bond lengths were identical within the experimental uncertainties.

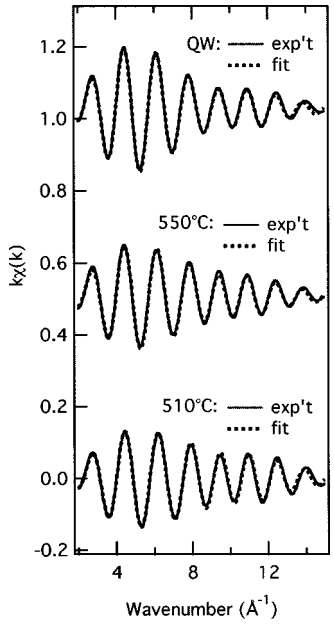

FIG. 5. The fitting results for Ge QD's grown at 510 and 550 °C compared with those for the $Si_{0.7}Ge_{0.3}$ quantum well (QW).

TABLE I. Fitting results for the Si-capped Ge QD's.

Growth T	N_{GeSi}	BL_{GeGe} Å	BL_{GeSi} Å	$MSRD_{GeGe}$ Å^2	$MSRD_{GeSi}$ Å^2
510 °C	2.7 ± 0.1	2.42 ± 0.01	2.37 ± 0.01	0.0030	0.0025
550 °C	2.9 ± 0.1	2.41 ± 0.01	2.38 ± 0.01	0.0032	0.0022
745 °C	3.9 ± 0.3		2.37 ± 0.01		0.0021

In addition to EXAFS we have also analyzed XANES spectra of both uncapped and Si-capped samples. The measured spectra are shown in Fig. 6. We have fitted the measured spectra with the simulated spectra formed as a weighted sum of the reference samples: bulk Ge, Ge-oxide, and a dilute solid solution $Ge_{0.006}Si_{0.994}$. The spectrum for the uncapped QD's was fitted by a linear combination of the three references with the partial weight of each component being as follows: bulk Ge, 25%; Ge-oxide, 30%; $Ge_{0.006}Si_{0.994}$, 45% (Fig. 3 of Ref. 28). The fit reproduces all the features of the experimental spectrum quite well and the obtained numerical values agree with the results of the EXAFS data analysis.

The spectra for the capped QD's are quite similar among themselves and also similar to that of the $Si_{0.7}Ge_{0.3}$ quantum well and of the dilute Ge-Si solid solution. We obtained reasonably good fits for the average Ge concentration being in the range of 5–30%.

IV. DISCUSSION

We start the discussion with the local structure of Ge QD's in the uncapped sample. Although the large number of the fitting parameters used to fit the three features in the experimentally observed sample leads to rather large uncertainties, their number is smaller than the number of independent points in the data and several definite conclusions can be made. First of all, the result clearly shows that the uncapped QD's are partially oxidized. The remaining Ge is partially intermixed with silicon but a part of it (about ~25%) remains as a pure Ge phase. This corresponds to an average Ge content of about 40% in the crystalline SiGe alloy.

The obtained Ge-Ge bond length in the uncapped QD's of 2.46 ± 0.02 Å is close to the value for bulk Ge which means that the dots are essentially relaxed. Interestingly, the Ge-Si bond length 2.44 ± 0.06 Å is also very close to this value. The result suggests that the QD's are relaxed as a result of intermixing and/or oxidation which are conducive to the more flexible network formation while most of the misfit strain is accommodated by the alloyed region. Most likely this fact accounts for why the remaining Ge is stable within the Ge phase.

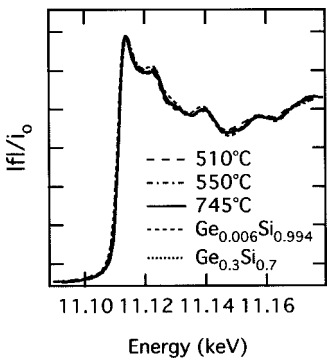

FIG. 6. XANES spectra for Si-capped Ge QD samples grown at different temperatures together with those for the references.

It is interesting to note that, while in the capped QD's higher shells can be seen up to 6 Å, in the uncapped QD's only the first shell (consisting of Ge-O, Ge-Ge, and Ge-Si correlations) is observed. Higher shells have the amplitude of the peaks at least twice lower than in the capped sample. We believe that suppression of higher-shell intensities is caused by the fact that intermixing and oxidation induce extra disorder in the second- and higher nearest-neighbor correlations which results in smearing out of the higher shells. Interference among the higher shells of three phases could also apparently reduce the amplitude of FT peaks.

The numerical values for the partial fraction of Ge-Ge, Ge-O, and Ge-Si bonds estimated independently from XANES agree very well with the values obtained from the EXAFS analysis.

We now proceed to the local structure of the Si-capped QD's. For the sample grown at 745 °C, both the similarity between the raw EXAFS oscillations of the capped QD's and those for dilute Ge solid solution and the fitting results ($N_{Ge\text{-}Si}=3.9\pm0.3$) demonstrate that Ge strongly dissolves in Si. The Ge atoms are located at the Si sites, replacing silicon atoms, and are subject to compressive local strain. The Ge-Si bond length in the capped sample is obtained to be 2.37 ± 0.01 Å. This value is in an agreement with the Ge-Si bond length obtained for $Ge_{0.006}Si_{0.994}$, namely, 2.38 ± 0.01 Å.[23]

The uncertainty in the obtained Ge-Si coordination number $N_{Ge\text{-}Si}$ does not allow us to completely exclude the presence of the Ge phase in the Si-capped dots. However, we can definitely claim that the fraction of Ge existing as the Ge phase is less than 10% of the total amount.

For the samples grown at lower temperatures the situation is different. The Ge phase clearly exists. A somewhat larger fractional Ge-Ge coordination number observed for small dots grown at the lowest studied temperature of 510 °C is easy to understand since intermixing at lower temperature is much slower.

It should be kept in mind that EXAFS, being a species-sensitive tool, is not site sensitive. In EXAFS experiments we probe *all* Ge atoms, including those in the wetting layer, in the QD's, and in the segregated layer and the obtained values are the average. The Ge atoms at the interface with

the substrate, with the Si cap, and the surface-segregated Ge atoms necessarily have Si neighbors while Si neighbors around the Ge atoms in the QD's result from intermixing. Estimation of the fraction of each group of Ge atoms and the Ge concentration in the QD's *per se* is, for this reason, not straightforward.

For the case of the sample grown at the higher temperature (745 °C) where experimentally we only detect Ge-Si bonds the conclusion is the simplest: Ge strongly intermixes with Si. The fraction of Ge in Ge-rich phase is determined entirely by the experimental uncertainties and is not more than 10%. Since intermixing is more likely to occur in a thin wetting layer (the typical thickness of the wetting layer being around 4 monolayers[6,29,30] and the intermixing taking place on a 2–3-monolayer scale on each side of the interface[31]) than in large dots, we believe we can ascribe the Ge-rich region (if it exists in this case) to the QD's. Taking into account the relative fraction of Ge atoms in the wetting layer and in the QD's we can estimate the average Ge concentration in the QD's not to exceed 30%. This result agrees with the conclusion drawn previously from optical studies of similar samples.[20]

For the samples grown at lower temperatures where Ge phase is clearly detected this estimate is more difficult. Taking the QD's size and density into account, we can estimate that the amount of Ge (and intermixed Si) in the QD's is about 40% of the total amount of deposited Ge atoms. Again, assuming that most of Ge-Si bonding is due to interfaces, we suggest that the Ge-rich phase exists as a core of the QD's. Taking the observed average coordination number into account we can estimate the average Ge concentration in the QD's grown within the 510–550 °C range to be ~70%.

Using this value for the average Ge-Si coordination in the QD's region and the size of the QD's obtained from the AFM measurements, we can roughly estimate, using the approach suggested in Ref. 31, the thickness of the intermixed region. The obtained values are ~5 and ~15 Å for the QD's fabricated at 510 and 550 °C, respectively. Such a big difference in the thickness of the intermixed region cannot be explained simply by thermal activation of the diffusion between the QD's and the cap. We believe that it results from intermixing occurring during the QD's growth stage. Assuming that the larger dots are formed at the expense of smaller ones, which are already intermixed, this possibility is not unreasonable. Alternatively, one can imagine that Ge adatoms arriving at the wetting layer do not diffuse to the QD's surface inreactively, but interact and site exchange with the intermixed wetting layer. Yet another alternative is lateral site exchange with Si during the overgrowth of Ge(Si) islands with Si. Si fills up the valleys between islands. Lateral diffusion of Si and Ge within the surface layer during the overgrowth results then in intermixed edges of islands. For ΔT of 40 K, we estimate an increase of lateral diffusion constant D of Si to be about a factor of 3 [based on an activation energy of 1.4 eV (Ref. 32)]. Finally, since the intermixing is driven by strain, it should increase at larger strain values and the local strain at the edges of larger islands should be stronger than that at smaller islands. All mentioned growth mechanisms result in the formation and growth of the intermixed region surrounding the Ge-rich ''core'' of the QD's.

The fact that the Ge-Ge bondlength for the Ge-rich phase is isotropic suggests that the mismatch strain is accommodated by the bond angles rather than by the bond lengths.

A similarity of the XANES spectra for the Si-capped Ge QD's with those for $Si_{0.7}Ge_{0.3}$ QW and $Ge_{0.006}Si_{0.994}$ provides an independent evidence that the capped Ge QD's strongly intermix with silicon. The surface segregation of Ge and site exchange at the Ge-Si interface are the major sources of Ge-Si intermixing.[33] Both may be affected by local strain surrounding the partially relaxed Ge dots. Since XANES spectra of GeSi alloys are quite similar in the Ge concentration range from 0.6 to 30%, the estimate of the Ge content from XANES is not accurate. Their similarity, on the other hand, provides very significant information. Since XANES, being dependent on the local structure, primarily contains information on the local density of unoccupied states, the similarity of the spectra for the Ge content in the 0.6–30% range demonstrates that the density of states depends on the *total amount* of Ge atoms in the alloy rather than on their concentration.

The results for the uncapped Ge QD's demonstrate the formation of the Ge oxide together with intermixing with Si. The existence of the relaxed Ge core suggests that the formation of an oxide layer on top of the QD's is a self-limiting process.

Our results on Ge/Si intermixing in the Si capped QD's agree well with recent x-ray photoemission studies[12] and also with XAFS experiments by other groups.[11] While AFM, TEM, and PL studies clearly demonstrate the formation of dots and quantum confinement in the samples studied in this work and in similarly prepared structures, the generally used term ''Ge QD's'' can be misleading: the QD's in most cases do not consist of a pure Ge phase and when the growth is performed at higher temperatures the concentration of Ge in the QD's may actually be very low.

V. CONCLUSIONS

We have probed the local structure of Ge QD's self-assembled on Si(100) by EXAFS and XANES. Independent analysis of both kinds of measurements yields very similar results.

We have found that in the uncapped Ge QD's, about 35% of Ge is oxidized while the rest partly intermixes with silicon. The fraction of Ge remaining in the Ge phase is on the order of 25%. The Ge-Ge bond length in the remaining parts of the QD's is $BL_{GeGe}=2.46\pm0.02$ Å, i.e., very close to the value for the bulk Ge. The Ge-Si bond length in the intermixed region has a similar value, $BL_{GeSi}=2.44\pm0.06$ Å.

In the case of Si-capping at high temperatures (about 745 °C), Ge QD's dissolve in silicon. The Ge-Si bond length equals $BL_{GeSi}=2.37\pm0.1$ Å and the distances to the second and third-nearest neighbors are, 3.83 ± 0.02 Å and 4.50 ± 0.02 Å, respectively, all these values being, within the experimental accuracy, identical to the Si-Si distances in the Si crystal. The partial coordination numbers obtained from the

curve-fitting indicate that Ge is almost completely dissolved in silicon. The uncertainties of the fitting cannot completely rule out the existence of the Ge phase. Nevertheless, our results allow us to claim that the fraction of Ge atoms which may exist as pure Ge does not exceed 10%. In the QD's grown at lower temperature (510 and 550 °C) Ge atoms have, on the average, 1.2-Ge neighbors. We estimate the fraction of Ge in the QD's to be about < 30 and $\sim 70\%$ for the cases of higher- and low-temperature growth, respectively. We suggest that the Ge/Si intermixing primarily takes place during the QD's formation rather than being the result of interdiffusion after the Si capping.

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