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Formation and ordering effects of C-induced Ge dots grown on Si (001) by molecular beam epitaxy

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Abstract

The deposition of sub-monolayer coverages of C on Si (001) prior to Ge growth leads to the formation of small, irregularly shaped Ge islands well below the critical thickness of Ge on Si. We studied the nucleation of these Ge dots on Si and the ordering of these dots in stacks of dot layers by in-situ scanning tunneling microscopy (STM) and transmission electron microscopy (TEM). It is found that island formation already starts at a sub-monolayer deposition of Ge on these C covered Si surfaces. Ge islands 2–3 nm wide with a height of a few monolayers are obtained by STM after the deposition of 0.1 monolayer of C and 0.5 monolayer of Ge. Apparently the Stranski–Krastanov mode of growth, typical for the formation of Ge hut clusters on Si, does not occur. Instead a Volmer–Weber type of growth is responsible for the island formation. It is noteworthy that no wetting layer is observed for these small C-induced Ge dots. TEM investigations of multiple dot layers containing 10–20 nm wide C-induced Ge dots reveal vertical alignment of the dots for Si barriers of less than 10 nm. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Formation; Ordering effects; C-induced Ge dots; Molecular beam epitaxy

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1. Introduction

Self assembled quantum dot structures of compound semiconductors have attracted much research activity due to their potential to improve the characteristics of opto-electronic devices such as semiconductor lasers [1]. Consequently, most of the research was focused on III–V compounds. Si based group IV compounds are indirect bandgap materials with very limited application potential for optically active devices [2]. Embedding Ge dots in Si may open new paths towards the ambitious goal of Si based opto-electronics [3]. Recently it has been found that the density of Ge dots can be drastically increased and the size substantially decreased at deposition temperatures around 500°C by depositing Ge on a C alloyed Si surface [4]. In particular Ge dot formation was observed after the deposition of two monolayers (ML) of Ge, which is clearly below the critical thickness of 4–5 ML for Ge dot formation on clean Si (001) surfaces. These C-induced Ge islands show significantly enhanced photoluminescence [5]. In previous work we analysed the structural details of the C-alloyed Si surface [6] as well as those of the dots after depositing 2.5–6 ML of Ge [7]. It was found that the deposition of C on Si (001) surfaces leads to areas exhibiting a c(4 × 4) reconstruction. A structure containing clusters of six C atoms sitting in the two topmost ML was identified as causing this reconstruction. The deposition of 2–4 ML of Ge on this surface leads to the formation of irregularly shaped islands consisting of stacked Ge ML. At higher coverages again pyramidal shaped islands are found, but they are smaller and appear with a higher density [7]. However, from these relatively thick Ge layers, where at a coverage of 2.5 ML islands are readily developed, no definitive conclusions can be drawn about the actual nucleation process. One could only state that island formation starts at lower coverage than on bare Si (001) and that the island shape differs from the anticipated hut cluster shape. At which coverage the nucleation of the GeC islands exactly starts remained rather speculative at that stage. In this study we unravel the details of the initial nucleation of Ge dots on the C-alloyed Si (001) surface as well as strain induced ordering effects in stacks of dot layers.

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2. Experimental

The structures were grown by molecular beam epitaxy (MBE) using e-beam evaporation for Si and Ge. The C was evaporated from a pyrolithic graphite filament. Typically the deposition rate for Si was kept at 0.1 nm s^{-1}, whereas the Ge flux was adjusted to 0.013 ML s^{-1} for samples containing submonolayer coverages of Ge and 10 times higher for the samples containing more than 2 ML of Ge per dot layer. The Ge and Si fluxes were controlled by regulating the focus of the e-beam using the signal of a mass spectrometer [8]. The amount of C and Ge deposited was adjusted by data obtained from secondary ion mass spectroscopy (SIMS) and X-ray diffractometry on separate samples. The growth temperature was adjusted in the temperature range from 350 to 500°C. The growth temperature was verified by a thermocouple welded to the front side of a p-type Si wafer.

The substrates were wet chemically cleaned and then transferred to the MBE chamber. The samples were baked at 600°C for 10 min and at 950°C for 20 min prior to the growth of a 100 nm thick Si buffer layer deposited at 750°C. The temperature was reduced to 500°C after the growth of the buffer layer. The growth was interrupted and subsequently C was deposited followed by the deposition of Ge.

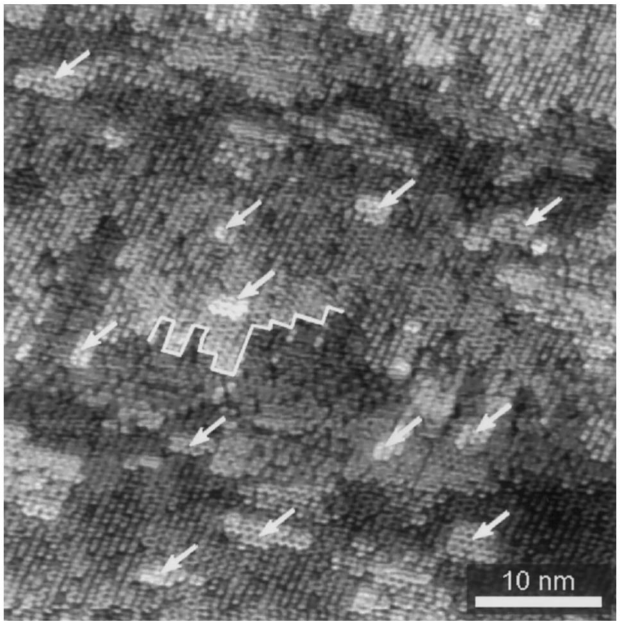

Fig. 1. STM image (-2 V, 0.17 nA) of 0.1 ML Ge deposited onto 0.11 ML C/Si (001) at a substrate temperature of 350°C and a flux of 0.01 ML s^{-1}. The arrows mark small single-layer Ge islands on the terraces surrounded by buckled dimer reconstruction. Islands are never found on top of carbon rich c(4 × 4) areas. A substantial amount of Ge is incorporated at step edges. A S_B step, where Ge might have gathered, is outlined.

The Ge dot formation on the Si alloyed surface was analysed by in-situ scanning tunneling microscope (STM) measurements. The samples were transferred from the MBE chamber into the STM chamber at ultra high vacuum conditions (10^{-10} mbar). A detailed description of the UHV-STM is given elsewhere [9]. The stacks of dot layers were analysed by cross sectional high resolution transmission electron microscopy (HR-TEM) using 300 kV electrons.

3. Results and discussion

3.1. Nucleation

Fig. 1 shows a 50 × 50 nm STM image of 0.1 ML Ge deposited onto 0.11 ML C/Si (001) at 350°C. Several small Ge islands of single layer height have nucleated on terraces (indicated by arrows). These islands are always surrounded by a certain number of buckled dimer rows in the terrace layer, which are assumed to be silicon. Although smaller islands of about 8 × 6 atoms dominate, larger islands are also found. The aspect ratio, however, is small compared to the more elongated islands reported on bare Si (001), although narrow islands are also present. The diffusion anisotropy on the surface seems to be reduced due to the presence of the c(4 × 4) regions. Ge ad-layer islands are not found directly on top of a c(4 × 4) area. The RMS roughness of this surface corresponds to 0.10 ± 0.01 nm and an island density of $8 \pm 1 \times 10^{11}$ cm^{-2} is deduced at this coverage. If all Ge ad-atoms were contained in the islands these would have an average size of 8 × 11 atoms or about 3.0 × 4.5 nm, but the islands are in fact smaller. This implies, that a substantial amount of Ge (approximately 50%) is incorporated at step edges. The incorporation at S_B steps in places where the lower terrace is 2 × 1 reconstructed is favorable. It has been shown that the energy barrier for ad-atoms to cross S_B steps downwards, the Ehrlich–Schwoebel barrier [10,11] is small [12]. Presumably it is also energetically favourable for Ge atoms impinging onto a c(4 × 4) domain near an S_B step to leave the domain by migrating step downwards. The outlined step edge in Fig. 1, where the terrace area behind exhibits c(4 × 4) symmetry, is, therefore, assumed to consist of Ge. A second layer island then prefers to nucleate on top of this Ge area because the lattice mismatch is small here.

The observation, that the germanium atoms do not nucleate on the carbon rich c(4 × 4) reconstruction is in perfect agreement with the finding of a repulsive interaction of Ge and C in this SiGeC material system [13]. It can be understood in terms of bond lengths and lattice constants. In the c(4 × 4) areas the Si surface is compressively strained due to the high carbon content. Consequently, the average lattice constant here is

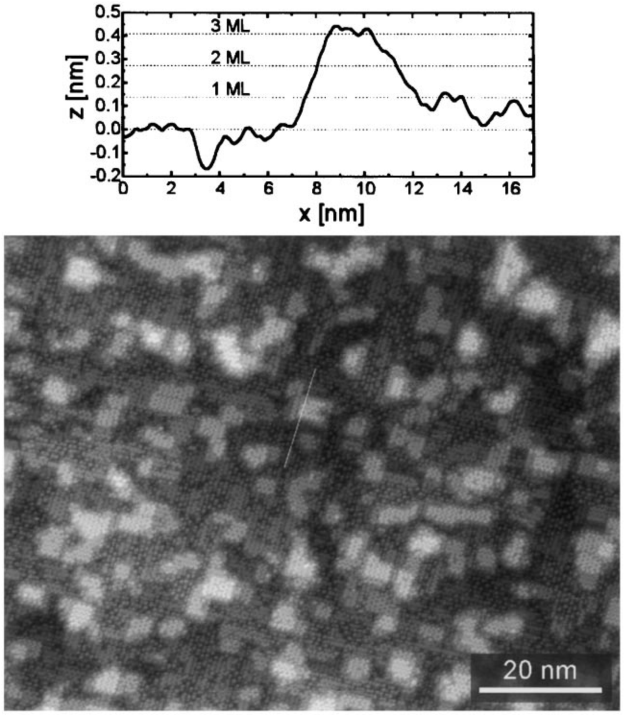

Fig. 2. STM image (−2.3 V, 0.2 nA) of 0.5 ML Ge on 0.1 ML C /Si (001), image size 100 × 75 nm. Islands started to pile up to 3–4 ML height. They are laterally restricted by the Ge-repelling c(4 × 4) patches. The line scan follows the white line from the lower left to the upper right. High islands are predominantly found at steps.

smaller than that of Si, which discourages the larger Ge atoms from nucleating in these areas. Furthermore, as there is also C present directly at the surface, the formation of Ge–C bonds would be very costly in energy, involving severe amounts of strain due to the difference in bond length of −37%. Instead, the Ge tends to wet the Si(2 × 1) regions first, since they are unstrained or even tensilely strained in between the carbon-containing areas. The resulting lattice mismatch of less than 4% favors Ge nucleation on the Si(2 × 1) areas. In addition, at this low growth temperature the ad-atom diffusion is sufficiently slow to form islands on the terraces despite the small terrace width of about 10–15 nm.

Increasing the Ge coverage to 0.5 ML, the island density rises only slightly to a value of $9.5 \pm 1.0 \times 10^{11}$ cm^{-2}. The RMS roughness has increased to 0.15 ± 0.01 nm simultaneously. Consequently the islands grow in size and height, but their lateral extensions are somewhat restricted by the ubiquitous c(4 × 4) areas. Still, the Ge atoms seem to avoid the formation of Ge–C bonds and therefore start nucleating on top of the existing islands, resulting in 3-dimensional (3D) island growth. The restructuring of the Si surface due to the C pre-deposition obviously forces Ge to grow in a Volmer–Weber mode rather than a Stranski–Krastanov mode at the given temperature. This 3D growth mode is demonstrated in Fig. 2 where many of the Ge islands clearly consist of two or even three layers. The line scan underlines this experimental finding. The area between the islands is mainly c(4 × 4) reconstructed. Islands have most frequently a rectangular shape with a low aspect ratio. The edges are aligned along $\langle 110 \rangle$.

An interesting observation is that most of the 3D islands are located at step edges. They virtually form the border between adjacent terraces in those places where the c(4 × 4) reconstruction does not reach the step edge. In Fig. 2 the terraces descend from the top of the image to the bottom. The line scan starts on a lower terrace across the island and ends one ML above on the upper terrace, as indicated by the white line in Fig. 2. The highest islands are predominantly found at the steps having $\mathrm{S_B}$ character, where we already assumed the agglomeration of the Ge not seen as islands at a coverage of 0.1 ML. Islands in the centre of a terrace remain somewhat smaller and flatter. A possible explanation is the following: due to the enhanced surface diffusion along dimer rows on the upper terrace Ge ad-atoms migrate towards the step edge, descend and are incorporated there, which corresponds to the initial stages of a step flow mode. But the lateral extension of this first monolayer of an island is restricted when it approaches the c(4 × 4) regions on the lower terrace, because of the reluctance to form Ge–C bonds. From then on the presence of c(4 × 4) areas on the lower terrace acts as a barrier that prevents new Ge ad-atoms from descending the Ge step and from growing further in step flow mode. Instead, the Ge atoms start to pile up by preferential nucleation on top of these existing Ge areas, where they can most easily accommodate to the lattice constant. So the 3D growth is driven by strain relaxation because the carbon enriched Si surface areas have a smaller average lattice constant. Note that some of the larger flatter islands exhibit the missing dimer rows known from the relief of strain in thin Ge ad-layers on Si (001).

So far we demonstrated, that using a substrate temperature of 350°C the Ge is completely repelled by the C rich areas on 0.11 ML C/Si (001) surfaces and grows three-dimensionally in a Volmer–Weber mode. In a previous paper we detected for thicker Ge films such as 2.5 ML and more, grown at 520°C, islands with a density of 1×10^{11} cm^{-2} whereby the c(4 × 4) reconstructed areas have disappeared [7]. Consequently, Ge is assumed to be intermixed with the $\mathrm{Si}_{1-x}\mathrm{C}_x$ areas at this temperature and coverage. The evolution of Ge growth below one monolayer is further investigated to uncover the process of intermixing and island formation at 520°C.

Fig. 3 represents a 100 × 75 nm STM image of 0.5 ML Ge on 0.11 ML C/Si (001) grown at 520°C. At first glance the Ge growth is now dominated by steps flow. The image is stepped downwards from the right to the left. It has been shown for the C-alloyed surface, that the c(4 × 4) areas are formed preferentially at the step edges, so that these are usually terminated by the c(4 × 4) pattern [6]. After the deposition of 0.5 ML Ge the areas around S_A steps are mostly terminated by (2 × 1) reconstructions. This area is expected to consist of Ge indicated by the characteristic missing dimer superstructure. It adjoins the c(4 × 4) reconstructed areas, which are believed to be located at a terrace edge before Ge deposition.

In contrast to the S_A steps, the S_B steps are still often formed by c(4 × 4) rows. All in all one still finds a lot of c(4 × 4) areas, either at step edges or surrounded by Ge or Si. That means, even at this substrate temperature of 520°C Ge is repelled by the C rich areas. A certain amount of intermixing between Ge and C, however, cannot be ruled out, since some of the (2 × 1) reconstructed areas contain — in addition to the strain relieving perpendicular missing dimer rows — rows with a periodic train of missing dimer defects. In these rows only every third or fourth dimer is visible, a structure not usually observed in Ge or SiGe adlayers. Furthermore, small c(4 × 4) patches are found in the direct vicinity or even on top of the areas believed to consist of Ge.

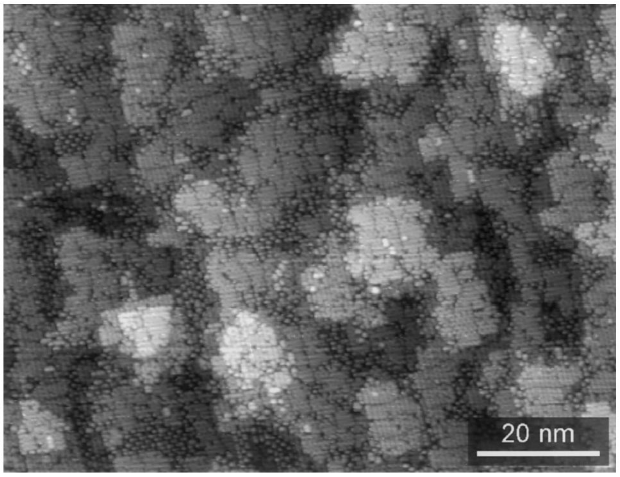

Fig. 3. STM image (− 2.4 V, 0.18 nA; size 100 × 75 nm), of a surface covered with 0.5 ML Ge deposited at 520°C on top of 0.11 ML C/Si (001) at a rate of 0.1 ML s^{-1}. The growth is rather 2- than 3-D, but two ML high islands are also formed. Still Ge is basically repelled by the C-rich regions, but some intermixing cannot be ruled out, indicated by the arrows that mark unusual structure within the Ge adlayer.

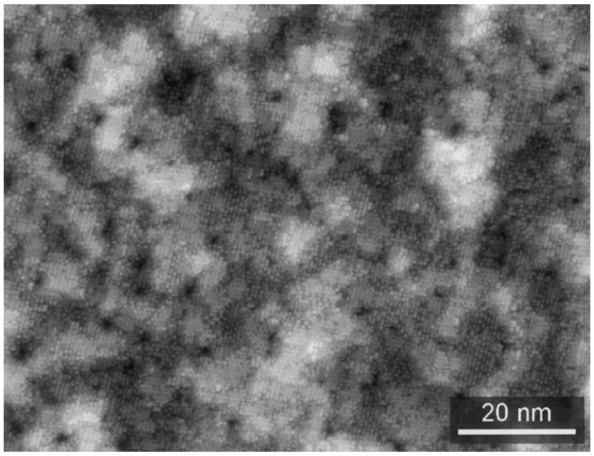

Fig. 4. STM image (− 1.5 V, 0.2 V; 100 × 75 nm) of 1 ML Ge on 0.11 ML C/Si (001). Some large and high islands and many smaller and flatter islands not higher than three atomic layer are present The c(4 × 4) areas are partly disordered, indicating some Ge incorporation.

The growth does, however, not proceed in pure step flow mode. On top of the islands a second layer has nucleated. The islands reach relatively large sizes, but small ones are also present. Islands with extensions of about 20 nm are dominant, having rather isotropic shape, i.e. without preferential directions of elongation. On all of them the Ge missing dimer rows are developed. Large islands exist predominantly in the vicinity of steps. The RMS roughness is determined to be 0.10 ± 0.02 nm.

It should be mentioned that the nucleation of a third layer on top of the large islands is rarely observed, indicating, that the diffusion length of Ge ad-atoms on these large islands is higher than the island diameter and the activation barrier for diffusing down the island edges is overcome at this growth temperature. This observation may be caused by different diffusion lengths on terraces and on islands. If they were identical one should find pure step flow growth, especially for the small terrace width of the substrate. Therefore, intuitively, one can conclude, that the diffusion on the terraces is hindered by the presence of the C-rich c(4 × 4).

Counting the islands on this surface by a gray scale discrimination and thresholding procedure an island density of $3 \pm 1 \times 10^{11}$ cm^{-2} is derived. This value is only a factor of three lower compared to the island density at 350°C. This difference in island density upon the large temperature difference of 200 K is remarkably small. From Si [14] or Ge nucleation on bare Si (001) surfaces one expects a drop of the island density by several orders of magnitude, because diffusion is a thermally activated process [15]. Even though the procedure for island counting, which involves leveling of the stepped surface, is not very accurate for large flat islands, because it leads to an average inclination of the terraces and eventually also protruding parts of step edges are counted, the order of magnitude of the island density remains without doubt correct. Hence, it is the presence of the C-rich c(4 × 4) reconstructed areas that modifies the fundamental process in Ge growth.

Fig. 4 shows an STM image of the surface after one monolayer Ge deposition at 520°C. Note, the surface

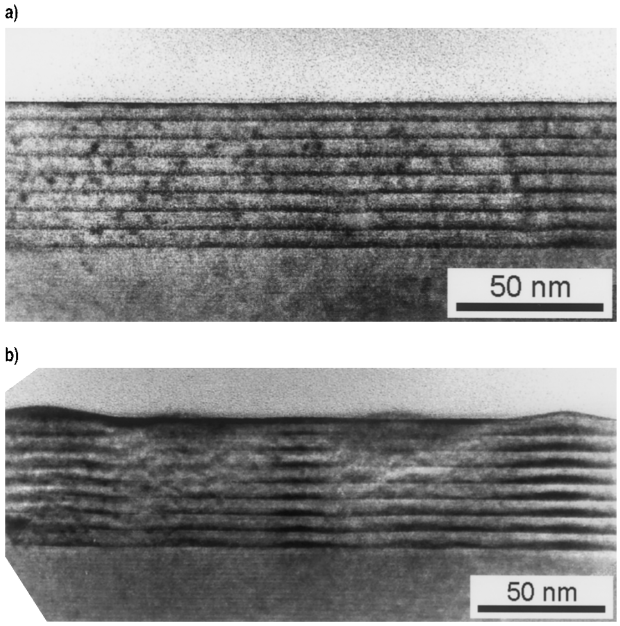

Fig. 5. TEM cross section of stacked multiple sheets of Ge containing (a) 0.05 ML C and 4 ML Ge in the first and 4 ML of pure Ge in the subsequent sheets, and (b) 0.05 ML C and 6 ML Ge in the first as well as 6 ML of pure Ge in the subsequent sheets. For 5 nm wide Si spacer layers vertical alignment is only observed if the Ge thickness exceeds the critical thickness of dot formation

exhibits substantial amounts of c(4 × 4) reconstructed areas. Even at 520°C the Ge does not wet the whole surface but is still essentially repelled by the C rich areas. The island growth is more pronounced for monolayer coverage than for 0.5 ML. The RMS roughness has increased considerably to 0.17 ± 0.02 nm. Some islands have a height up to 6 ML. The largest islands have sizes of 10–15 nm. The majority of islands, however, are flat, rarely exceeding a height of three monolayers. The island density remains constant at a value of $3 \pm 1 \times 10^{11}$ cm^{-2}. The large islands are expected to grow on top of the large two layer high islands found at 0.5 ML, whereas the smaller islands do not pile up further. This is understandable since the growth of the smaller islands, which is probably laterally restricted by the strain in surrounding areas, would lead to steeper sides, which is unfavorable in terms of surface energy. Furthermore, the supply of the small islands with ad-atoms is kinetically restricted as they have a smaller capture area.

Intermixing of Si and Ge depends primarily on the substrate temperature, but as for any thermodynamic mixing (diffusion) process the particle density is also involved. Therefore at higher Ge coverage intermixing is expected to become more important. Consequently the c(4 × 4) areas become more disordered compared to 0.5 ML Ge. In some places the same unusual rows with periodically missing dimers are found as for 0.5 ML Ge. This could be interpreted as a sign for a partial intermixing of Ge and Si in C rich areas.

In conclusion, there is no wetting layer of Ge even after 1 ML at 520°C. So even at this substrate temperature the growth mode is of a Volmer–Weber type. It is definitely not Stranski–Krastanov growth as observed on bare Si (001).

For a realistic description it should be mentioned that the step density of the substrates is relatively high. The terrace lengths of about 15–20 nm corresponds to an arbitrary miscut of 0.4–0.5°, which, in addition, can vary in direction from wafer to wafer, although they originate from the same series. Hence, the terrace width is comparable to the lateral size of the 3D islands. Undoubtedly the steps do affect the island nucleation and, therefore, alter the island size and density. Nevertheless, the general finding of the non-wetting of the c(4 × 4) areas resulting in Volmer–Weber growth will also be true on larger terraces.

3.2. Vertical ordering

In the previous section we discussed the effect of C induced modification of the strain, i.e. the lattice constant, at the surface on the nucleation of Ge islands. A related effect causes the vertical alignment of islands in multi-sheet arrays of islands. The second sheet of islands grows in the strain field created by the buried islands of the first sheet. This type of vertical correlation has been observed in various material systems including Si/Ge [16–19]. In the Si–Ge–C system the situation appears more complex and it is unclear whether the C pre-deposition will be affected by a sheet of buried dots. Another appealing experiment is the vertically self-organised replication of small C-induced Ge dots in the first sheet with pure Ge dots in subsequent sheets. For the latter case some initial work has been published [20] showing vertical alignment in a sample containing 0.16 ML C and 3 ML Ge in the first layer and 4 ML of Ge and no C in the subsequent layers and Si spacer layers in between the island sheets of only 2 nm. Remarkably no ordering was observed if C is pre-deposited in every island sheet.

In our experiments we kept the spacer thickness constant at 5 nm and reduced the amount of C to 0.05 ML. The amount of Ge was varied from 4 ML, which is below the critical thickness of Ge for island formation, to 6 ML, which is above the critical thickness. Fig. 5 shows TEM cross sections of the two samples with 4 ML (Fig. 5a) and 6 ML (Fig. 5b). The C pre-deposition leads to the formation of small islands and high densities at 4 ML. The density is about $2\text{–}3 \times 10^{10}$ cm^{-2} and thus is at least an order of magnitude higher than for the hut clusters found on bare Si under the same growth conditions. However, the replication of dots is only obtained in the two subsequent dot sheets, but already showing a gradual extinction of dots. Above these dot sheets the Ge transfers into a smooth 2D layer, as would occur in a single 4 ML Ge layer grown on Si. This result is consistent with the previous work, showing ordering only for Si spacer layers as narrow as 2 nm [20].

At 6 ML (Fig. 5b), above the critical thickness, the Ge dots are faceted, as in the case of pure Ge on Si, but the dots are smaller and the density is 10 times as high [7]. In this case the vertical self-organised replication of the dots is clearly visible. The pure Ge dots remain relatively small and in the top layer are about the same size (40–50 nm) as in C-induced first layer. The same experiment without the C predeposition in the first dot layer would produce islands with diameters in the range 70–100 nm.

In the next set of samples we put down a C pre-deposition prior to every Ge dot sheet, growing stacks of C-induced dots. Fig. 6 depicts a cross sectional TEM of the same structure as shown in Fig. 5a but with a deposition of 0.05 ML of C prior to every Ge growth. The islands of subsequent sheets are clearly aligned. Also the effect of island coalescence can be observed [21] leading to an increase in island size towards the surface. Some of the top islands are of the faceted cluster type although the initial layer consists of non-faceted C-induced Ge dots. The increase in size due to island coalescence presumably drives the cluster size beyond the critical size for facet formation, thus even enforcing the strain driven correlation. These results are somewhat contradictory to the literature data [20] where no self alignment for a stack of 0.16 ML C/3 ML Ge dot sheets separated by 5 nm Si spacers is reported. Possibly this is due to the smaller amount of C pre-deposited in our samples. At high C coverages the strong effects of the C on the surface may erase the inferior effects of the strain fields of the small buried dots.

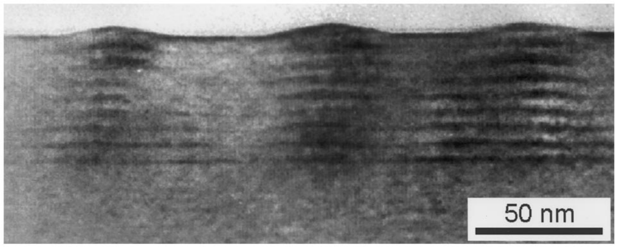

Fig. 6. Cross sectional TEM image of a stack of eight 0.05 ML C/4 ML Ge dot layers separated by 5 nm Si spacers. Note the change of dot shapes from the initial layer, where they are a non-faceted DeC dot type, to the top layer that shows pyramidal faceted cluster shapes.

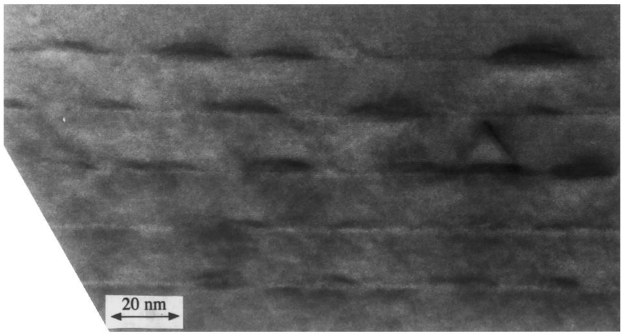

Fig. 7. TEM image indicating anti-correlation in a stack of five C-induced Ge dot layers separated by 16 nm Si spacer layers. Each dot layer contains 0.29 ML of C and 3.4 ML of Ge.

Rising the Si spacer layer thickness from 5 to 15 nm destroys the vertical alignment and every dot sheet consists of non-faceted C-induced Ge dots. In distinct areas of the sample the dots appear to align in an anti-correlation structure, in other areas no correlation is detected. Anti-correlation is expected to occur if the spacer layer thickness is comparable to the dot diameter [22], and has been found in the InAs/GaAs system for very small InAs dots. We found a clear anti-correlation as a result of growth instabilities in C rich Si/SiC multiple quantum well structures grown around 500°C [23]. To explore the possibility of anti-correlation in the C-induced Ge dot system we grew several samples with variations in the amount of C and Ge deposited and with Si spacer layer thickness between 12 and 20 nm, which is in the range of the dot diameter. Fig. 7 shows a cross sectional TEM of a sample containing a stack of 5 C-induced Ge dot sheets. The growth temperature was 460°C. Each dot layer contains 0.29 ML C and 3.4 ML Ge. The individual dot sheets are separated by 16 nm Si spacer layers. Some anti-correlation is obtained in the three topmost Ge layers, however this anti-correlation is only found in parts of the sample (left side of Fig. 7). Other parts in the sample show disordered arrays of dots and due to the high C concentration some dislocations, which may have disturbed the anti-correlation. At lower C concentrations or thinner Si spacer layers the tendency for anti-correlation weakens.

In general it appears that at low C concentrations and thin Si spacer layers clear vertical alignment can be achieved, even for Ge depositions of 4 ML, which is below the critical thickness of islanding in the pure Si/Ge system. At high C concentrations the C induced strain fields on the surface layer overlays the strain fields of the buried island which destroys the vertical correlation. However, at high C concentrations, above 0.2 ML, and thick Si spacer layer, the spacer layer thickness are about the same thickness as the Ge dot diameter and some indications for anti-correlation is found.

4. Conclusions

The basic observation of this study is that on the C-restructured Si (001) surface the Ge starts forming 3D islands right from the beginning of deposition. Hence the growth mode is Volmer–Weber growth and no Ge wetting layer is formed. Especially, the formation of high-density but isolated ultra-small Ge islands at a lower growth temperature is a promising result offering good prospects for potential light emitters based on SiGe technology. However, the evaluation of their optical properties and of Si capping remain topics for future work.

The irregular islands at higher coverage (above 2.5 ML) arise from the small islands by coalescence and a gradual intermixing or coverage of the C-rich areas with germanium. The patches with high C-concentration are thus positioned in between the 3D-islands. Possibly they are covered with a very thin Ge layer that interconnects the island, or have intermixed into a SiGeC alloy. This layer will then become thicker with increasing Ge coverage and, hence, reduce the well defined carrier confinement of electrons in the C-rich Si areas and of holes in the Ge-dot. So, finally, this can explain the rapid decay of PL emission with increasing Ge deposition observed in the previous study [7], that has its maximum around 2.5 ML Ge and almost vanishes at 4 ML already.

Replication of C-induced dots in subsequent layers of pure Ge has been found for the deposition of 6 ML Ge and 5 nm wide Si spacers. The vertical alignment of islands in stacks of C-induced Ge dots depends on the exact relationship between the amount of C and Ge as well as of the Si spacer layer. For low C concentrations and thin Si spacers vertical alignment of the dots was clearly observed. For high C concentrations and thick Si buffer layers indications of an anti-correlation of C-induced Ge dots were found.

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