Adsorbate induced self-ordering of germanium nanoislands on Si(113)

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Abstract. The impact of Ga preadsorption on the spatial correlation of nanoscale three-dimensional (3D) Ge-islands has been investigated by low-energy electron microscopy and low-energy electron diffraction. Submonolayer Ga adsorption leads to the formation of a 2D chemical nanopattern, since the Ga-terminated ($2 \times 2$) domains exclusively decorate the step edges of the Si(113) substrate. Subsequent Ge growth on such a partially Ga-covered surface results in Ge 3D islands with an increased density as compared to Ge growth on clean Si(113). However, no pronounced alignment of the Ge islands is observed. Completely different results are obtained for Ga saturation coverage, which results in the formation of (112) and (115) facets regularly arranged with a periodicity of about 40 nm. Upon Ge deposition, Ge islands are formed at a high density of about $1.3 \times 10^{10}$ cm$^{-2}$. These islands are well ordered as they align at the substrate facets. Moreover, the facet array induces a reversal of the Ge islands’ shape anisotropy as compared to growth on planar Si(113) substrates.

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1. Introduction

The growth of nanoscale three-dimensional (3D) germanium islands on silicon has attracted a large interest because of potential technological applications in opto-electronic devices [1]–[3]. For many of these applications, a uniform size distribution of the nanoislands is important. This is closely related to a uniform distribution of the islands’ distance distribution, because via growth kinetics, island nuclei with regular spacing will grow at similar rates. This correlation between regular arrangement and size distribution has been demonstrated for stacked layers of Ge islands with Si spacer layers in between [4]–[6]. An explanation for the spatial ordering of nanoscale islands in stacked hetero-epitaxial structures has been given in terms of strain [7]. After overgrowth with a spacer layer of the host material, the lattice mismatch leads to strain fields emerging from the islands. This results in a strain modulation at the spacer layer surface and, thus, to heterogeneous nucleation conditions. For a proper choice of the spacer layer thickness, this approach does not lead to a reproduction of the nucleation sites in the next layer (strong vertical ordering), but to a reduced vertical ordering which allows for an increase of the lateral ordering with increasing number of island layers, as has been demonstrated for many different material systems like InAs/GaAs(001) [8, 9], InP/InGaP(001) [10], PbSe/PbTe(111) [11, 12] and CdSe/ZnSe(001) [13, 14].

In comparison to artificially introduced ordering, e.g. by electron beam lithography [15] or atomic probe manipulation [16], the above-mentioned approach of growing stacked nanoisland structures employs self-organization and, thus, is much better suited for technological applications, since it enables parallelized fabrication. However, this self-ordering approach has a major drawback: in the bottom layers, the islands are still quite randomly distributed. Different self-organized growth techniques have been employed in order to produce regularly spaced nanostructures in a single layer, i.e. without (or prior to) stacking. Most of these techniques again employ a strain modulation of the substrate in order to induce heterogeneous, spatially periodic nucleation conditions. For instance, this can be achieved by using dislocated [17] or rippled, pseudomorphically strained [18, 19] Ge\textsubscript{x}Si\textsubscript{1−x} buffer layers. In these cases, the lateral periodicity is coupled to the Ge content $x$, therefore this parameter can hardly be used for additional tailoring of the electronic properties. This limitation might be overcome by using adsorbate-induced self-ordering. Recently, we were able to show that submonolayer...
deposition of Ga on Si(111) at high temperature leads to the decoration of step edges and domain boundaries of the initial Si(111)-(7 × 7) domains [20, 21]. As a consequence, a 2D nanopattern of Ga-terminated and Ga-free surface regions can be achieved, and subsequent Ge deposition on such a chemically modulated surface results in the growth of 3D nanoscale Ge islands aligned at step edges and domain boundaries [22]. In the present study, we explore the possibilities to use the approach of adsorbate-induced ordering for the alignment of Ge islands on the Si(113) surface. As will be shown in the following, this approach can also be applied here, but the mechanism, as compared to Si(111), is completely different, allowing for even more pronounced Ge island ordering.

In contrast to many other high-index Si surfaces, the Si(113) surface has a low surface free energy [23] and is, therefore, a stable facet of Si. From a technological point of view [24], Si(113) might become an alternative to Si(001), the latter still being the substrate for most device applications. In contrast to Si(111), the lack of rotational symmetry of the Si(113) surface facilitates the growth of highly anisotropic Ge islands. In a narrow temperature and coverage range, nanowires extending in [33 2] direction can be grown [25, 26]. It is believed [27] that this strong anisotropy is stabilized by a reduction of the strain energy originating from the lattice mismatch between Ge and Si. However, this is controversially discussed [28]. It is also reported that the nanowires are metastable and can be transformed into elongated islands by ripening at growth temperature [29], hence growth kinetics and especially diffusion anisotropy might also play an important role. Regarding the spatial correlation of such Ge nanowires (as well as that of similar Ge$_x$Si$_{1-x}$ nanowires), vertical ordering in stacked heterostructures has been observed [30]–[32]. However, no reports on lateral ordering of Ge nanostructures on Si(113) are available so far. In the following, we present low-energy electron microscopy (LEEM) results in order to address that issue. This experimental method has already been shown to be very well suited for the study of the evolution of Ge nanostructures during growth on Si [22, 33].

2. Experimental

The experiments discussed in the following were performed in the ultra-high vacuum set-up at the spectroscopic photoemission and low-energy electron microscope [34, 35] located at the undulator beamline 1.2 at the Elettra synchrotron light source, with a base pressure in the low 10$^{-10}$ mbar range.

The Si(113) substrates were degassed at 600 °C for at least 12 h. The native oxide was removed by 2–3 short flashes to about 1200 °C for up to 30 s. This procedure results in the formation of the (3 × 2) surface reconstruction. Subsequently, the samples were exposed to different deposits of Ga at a temperature of about 500 °C. Finally, Ge was grown at temperatures ranging from 460 to 500 °C, without Ga codeposition. All preparation steps as mentioned above were monitored in situ by low-energy electron diffraction (LEED) and LEEM. The temperature was measured with an infrared pyrometer as well as with a thermocouple attached to the sample holder. The absolute accuracy is estimated to be about ±40 °C. Both Ga and Ge were evaporated from effusion cells heated by electron beam bombardment, at deposition rates of about 0.17 and 0.24 ML min$^{-1}$ for Ga and Ge, respectively. One monolayer (ML) corresponds to 4.09 × 10$^{14}$ atoms cm$^{-2}$. Experiments without Ga preadsorption were performed to calibrate the Ge evaporator flux. For this purpose, we used the onset of the formation of Ge Stranski–Krstanov islands [36] at 4.4 bilayers (BL; 1 BL = 2 ML), as previously reported by Yasue et al [37]. As the Ge sticking coefficient can be expected to be very close to unity,
the Ge exposures as given in the following will reflect the actual coverage. The situation is different for Ga, since at elevated temperature the Ga sticking coefficient decreases with increasing coverage [38], leading to a Ga saturation of the Si(113) surface: even after prolonged Ga exposure, we could not observe the formation Ga droplets in the temperature range investigated here. Since there is no reliable value from the literature for the local Ga coverage of the Ga:Si(113)-(2×2) which we observe (see section 3.3), we tentatively attribute the complete evolution of this superstructure at 500 °C to a Ga exposure of 0.5 ML, complying with previous work by Suzuki et al [39]. It should be emphasized again that the values given in the following refer to the Ga exposure and do not reflect the Ga coverage of the surface.

3. Results and discussion

3.1. Submonolayer Ga coverage

Figure 1 shows bright-field LEEM images of the Si(113)-(3×2) surface during submonolayer Ga adsorption at 500 °C. At the beginning, there is only a faint contrast visible originating from step edges of the substrate, running from the top left to the bottom right in figure 1(a). From the LEEM images, we determine an average terrace width of about 250 nm. Upon Ga adsorption, the contrast is enhanced, as can be seen for Ga exposures of Θ_Ga = 0.24 and 0.30 ML in figures 1(b) and (c), respectively. This is explained by the formation of Ga-terminated domains at the step edges which exhibit a (2×2) reconstruction in contrast to the (3×2) reconstruction of the bare
Figure 2. Bright-field LEEM images obtained during Ge deposition (a)–(e) at 460 °C on a partially Ga covered Si(113) surface and, for comparison, (f) at 440 °C on bare Si(113). The Ge coverage is indicated in each frame. Note the different scale bar in image (f).
experiment, further Ge deposition does not lead to a noticeable change in LEEM contrast, until small bright spots appear at a Ge coverage $\Theta_{Ge}$ between 4 and 5 BL, as depicted in figure 2(d). These bright spots are identified as 3D Ge islands. At $\Theta_{Ge} = 6.4$ BL (cf figure 2(e)), the nucleation stage of these 3D islands has been finished, i.e. the islands grow in size, whereas their density remains constant. For comparison, a LEEM image recorded from a Ge film of similar thickness grown at similar temperature on bare Si(113) [36] is shown in figure 2(f). In both cases, i.e. with and without submonolayer Ga preadsorption, the 3D islands are elongated along the [332] direction. Also, the onset of 3D island formation [37] (between 4 and 5 BL) is very similar in both cases. The most apparent difference between both surface orientations is the reduced 3D island density for growth on bare Si(113). As we reported previously [36], the Ge 3D island density on the bare Si(113) surface shows a clear Arrhenius behaviour, from which an island density of $6 \times 10^7$ cm$^{-2}$ is deduced for a growth temperature of 460$^\circ$C. The corresponding value for Ge growth on partially Ga-terminated Si(113), as determined from images like the one shown in figure 2(e), is almost two orders of magnitude larger and amounts to about $4 \times 10^9$ cm$^{-2}$. This clearly proves that Ga step-edge decoration of Si(113) leads to a reduction of the surface diffusion length for subsequently deposited Ge. When comparing the surface morphology found here to that observed for Ge grown on a partially Ga-terminated Si(111) surface [22], the alignment of the Ge 3D islands—if any, cf figures 2(a) and (e)—with respect to the step edges is much less pronounced for the (113) surface orientation. One reason might be the small diffusion length for Ge already at the initial stages of growth. From figure 2(b) this can be estimated to be only a few tens of nanometres at the growth temperature used here. This limited surface diffusion could be due to island nucleation at surface defects like domain boundaries. In comparison, e.g. to Si(111)-(7 × 7), the domain boundary density of Si(113)-(3 × 2) is much larger. On clean Si(113), however, these domain boundaries do not act as heterogeneous nucleation centres, and step flow growth has been observed above 400$^\circ$C [41]. Therefore, the low Ge diffusion length between the decorated step edges observed here can most likely be attributed to mobile Ga atoms on the terraces which may promote 2D Ge island nucleation.

Owing to the low diffusion length, the density of the 2D Ge islands is high, offering many additional step edge sites which are likely to be energetically attractive to Ga atoms. This probably leads to a redistribution of Ga all over the surface and hence may spoil the chemical surface modulation. The assumption of a redistribution of Ga is further supported by the fact that after Ge growth no contrast from the former Ga-rich domains is visible with LEEM, opposed to respective results on Si(111) reported previously [22].

In order to verify the scenario proposed above, further investigations would be necessary with the Ge diffusion length tuned to be at least comparable to the mean terrace width, by increasing either or both the Ge growth temperature as well as the step edge density, e.g. via the miscut angle. Then, a significant alignment of Ge 3D islands at step edges could be expected. But instead of pursuing this approach, we present a completely different scheme in the following, which enables a well-ordered arrangement of Ge 3D islands.

3.3. Ga saturation coverage

The evolution of the surface reconstruction during Ga adsorption at 500$^\circ$C up to saturation coverage is shown in the LEED patterns in figures 3(a)–(c). At $\Theta_{Ga} = 0.42$ ML, the initial (3 × 2) pattern has changed to a superposition of contributions from (3 × 2) and (2 × 2) domains,
Figure 3. LEED patterns obtained during saturation of the Si(113) surface with Ga at 500 °C. The Ga exposure is indicated in each frame.

Figure 4. LEED pattern (top) obtained after Ga saturation at 500 °C. Line scans along the direction of the red, green and blue arrows in the LEED pattern (at 0, 1/6 and 1/4 of the Brillouin zone (BZ)), are shown as a function of the vertical scattering vector component \( k_\perp \) in the three frames at the bottom, with respective colour maps. (See also movie 1.)

as already discussed in section 3.1. After exposure to about 0.6 ML, the surface is completely (2 × 2) reconstructed. Based on the results discussed in section 3.1, this means that the whole surface is Ga-terminated at this stage. Nevertheless, the evolution of the surface structure is not yet complete. Upon further Ga deposition, the LEED pattern changes again, as shown in figure 3(c) for \( \Theta_{\text{Ga}} = 1.1 \) ML. The nature of this rather complex LEED pattern is revealed in figure 4. Reflections occur at lines parallel to the [332] direction, i.e. the (almost) vertical
Figure 5. Left: bright-field LEEM image after Ga saturation at 500 °C. The contrast arises from facets. Right: line scan along [332] through the 2D autocorrelation function (shown in the inset at the upper left; the red line marks the line scan) of the LEEM image. The inset at the upper right shows a cross-sectional sketch of the surface morphology.

direction in the LEED pattern in figure 4. The [110]-position of these lines, i.e. the position along the (almost) horizontal direction, is either \(p/6\) or \(q/4\) of the surface BZ, with \(p\) and \(q\) being integer numbers. Line scans have been taken as a function of electron energy along the [332] direction for (i) \(p = q = 0\), (ii) \(p = 1\) and (iii) \(q = 1\). Three reciprocal space maps in the \(k_{∥} - k_{⊥}\) plane have been compiled from these data and are shown at the bottom of figure 4. Remarkably, there are no reciprocal lattice rods running along the [113] direction, as would have been expected for a flat surface. Instead, all reciprocal lattice rods are inclined. This can only be observed for a completely facetted surface. For \(p = q = 0\), two different types of rods are found with opposite inclination angles of 9.8° ± 0.5°. From these inclination angles, the crystallographic orientation of the facets can be determined. The best agreement is obtained for (112) facets and (115) facets. With respect to the (113) surface plane these facets are inclined by 10.0° and 9.4°, respectively. Similar results have also been obtained previously [39, 42]. However, the presence of (116) facets, as proposed by Li et al [42], seems very unlikely here, because this would lead to an inclination of 12.0°. For \(p = 1\) and \(q = 1\), only one facet orientation is found in each of the corresponding reciprocal space maps (cf figure 4). This is explained by different surface reconstructions on each facet type, a \((6 \times 1)\)-reconstruction on the (112) facets, and a \((4 \times 1)\)-reconstruction on the (115) facets.

With the information from reciprocal space, the interpretation of the LEEM images obtained after Ga saturation, such as shown in figure 5, becomes straightforward. The dark and bright stripes extending along [110] are identified as (112) and (115) facets. From real-space images, it is also possible to directly obtain the average spacing between facets of common orientation, i.e. the periodicity of the surface morphology along the [332] direction. Here, we find a value of 40 ± 6 nm. Already at first glance, the facet array in the LEEM image in figure 5 looks quite regularly arranged. This is confirmed by the autocorrelation along the [332] (also shown in figure 5), in which correlation peaks up to eighth order are clearly resolved.

Our results are in good agreement with those obtained in a scanning tunnelling microscopy study by Suzuki et al [39], as far as orientation and reconstruction of the facets is concerned. Interestingly, the facet arrangement found by Suzuki et al shows weaker ordering and, most
important, an average period length of only about 10 nm, that is a factor of 4 smaller as compared to our results. Although the reason for this difference is not yet fully understood, we suggest that the facets’ average spacing and their long-range ordering depend on surface kinetics and, hence, on details of the sample preparation. More specifically, in their systematic study of the dependence of surface reconstruction and morphology on Ga deposit and substrate temperature, Suzuki et al always used a quite large Ga flux of about 1 ML per minute and quenched the samples to room temperature immediately after Ga adsorption. In the work presented here, the Ga flux was much lower and, accordingly, the deposition times were longer. This may lead to conditions closer to thermodynamic equilibrium, and thus to coarser structures and enhanced ordering.

3.4. Ge growth on facetted Ga:Si(113)

The facet structure as characterized in the previous section has a strong impact on the surface morphology after Ge deposition. As can be seen from the LEEM image in figure 6, a very high density (about $1.3 \times 10^{10} \text{cm}^{-2}$) of small Ge 3D islands is formed at 500 °C. Again, the Ge
islands have an anisotropic shape, but now they are elongated along [\bar{1}10], that means they have an opposite shape anisotropy as compared to Ge growth on bare or partially Ga covered Si(113). This is confirmed by the quantitative analysis of the average Ge island diameter, which amounts to 25 ± 6 nm in [\bar{3}3\bar{2}] direction, in contrast to 40 ± 15 nm along [\bar{1}10], as can be seen from the histograms in figure 6. The same type of anisotropy is observed for the Ge island spacing. From the autocorrelation along [110], as depicted in figure 6, an average nearest-neighbour distance of 100 ± 10 nm is derived, whereas in [\bar{3}3\bar{2}] direction, a value of 50 ± 6 nm is found. Within the experimental uncertainty, the latter value matches the facet periodicity of 40 ± 6 nm. In addition, more pronounced maxima are observed in the autocorrelation along [\bar{3}3\bar{2}] as compared to that along [110] (cf figure 6), pointing to a longer-range order in [\bar{3}3\bar{2}] direction.

Both the spacing and the ordering anisotropy are strong indications for an alignment of the Ge islands at the facets. This is further supported by the above-mentioned value of the Ge island width in [\bar{3}3\bar{2}] direction (25 ± 6 nm), as this value is in good agreement with the average facet width, i.e. half the facet array periodicity. Due to different surface free energies of the different facets, one can expect that the Ge islands preferentially nucleate at one type of facet (which, however, could not be resolved in our experiments). The facet boundary then acts as a growth barrier for the Ge islands, thus the width of these islands is expected to be limited by the facet widths. In addition to this impact from surface energetics, surface kinetics also favours the reversal of the shape anisotropy. This can be seen from figure 7, where a surface region with a large contaminant island has been selected for LEEM imaging. In the vicinity of this contamination along the \langle110\rangle directions, almost no Ge islands are found within a distance of
Figure 8. Left: LEED pattern obtained after deposition of 6.7 BL Ge at 500 °C on a Ga-saturated, faceted Si(113) surface. Middle and right: reciprocal space maps in $k_{\perp}$-$k_{\parallel}$ planes with $k_{\parallel}$ parallel to [33$\bar{2}$] and with a constant $k_{\parallel}$-offset in [1$\bar{1}$0] direction of 0 % (middle) and 20 % (right) of the surface BZ.

about 300–400 nm. The presence of these denuded zones show that the contamination has an attractive chemical potential for Ge species. The fact that the width of the denuded zones along $<$33$\bar{2}$$>$ is much smaller proves that the Ge diffusion along the facet trenches in [110] direction is much faster than in the perpendicular direction.

Figure 8 illustrates LEED results obtained after Ge growth on faceted Ga:Si(113). Similar to the analysis described in the section 3.3, reciprocal space maps have been extracted from the energy dependence of the LEED pattern. From these reciprocal space maps (cf figure 8), it follows that after Ge growth the surface is still faceted. The inclination of the facets has been determined to be $11.3° \pm 1.0°$, which slightly deviates from the angles expected for (112) and (115) facets ($10.0°$ and $9.4°$, respectively). On the one hand, this might indicate the transition of (115) facets to (116) facets (with an expected angle of $12.0°$). On the other hand, a deviation of the facet inclination from the expected values might also be caused by strain due to lattice mismatch. In contrast to the facet reconstructions observed prior to Ge deposition, the LEED pattern in figure 8 reveals that there is only one additional periodicity attributed to a $(5 \times 1)$ reconstruction of the (112) facets. The reciprocal lattice rods with opposite inclination are found only in reciprocal space maps through integer reflections. Therefore, the corresponding facets, with either (115) or (116) orientation, do not exhibit a well-ordered superstructure.

The change of the facet surface reconstructions shows that not only 3D Ge islands have formed, but that there is also a 2D Ge wetting layer on the facets. It might be noteworthy that, although there are many examples for facetting of high-indexed Si and Ge surfaces after adsorption of group-III elements [43]–[45], no such adsorbate induced facetting has been observed for Ga on bulk Ge(113) [44].

4. Conclusion

Different regimes of Si(113) substrate prepatterning by Ga adsorption have been investigated and explored for their suitability for the growth of aligned Ge 3D nanoislands. In the case of submonolayer Ga exposures, the adsorption leads to step edge decoration, as has already been observed for Si(111). In contrast to the (111) orientation, however, the resulting nanoscale
chemical surface modulation cannot be used for a pronounced heterogeneous nucleation of Ge 3D islands. This is most likely due to the limited diffusion of Ge, which is reflected by the homogeneous nucleation of 2D Ge islands already in the initial growth stage and, as compared to Ge growth on bare Si(113), by an increased density of Ge 3D islands upon further Ge deposition. Our results suggest that the approach of submonolayer Ga adsorption might only be appropriate if the terrace width can be reduced down to the order of the diffusion length, e.g. by using vicinal Si(113) substrates.

For Ga saturation coverage, the Si(113) surface becomes unstable, and (112) and (115) facets extending along the [110] direction are formed, with (6 × 1) and (4 × 1) reconstruction, respectively. For the preparation conditions used here, the facet arrangement is very regular, with a period length of about 40 nm. Subsequent Ge deposition leads to the selective growth of nanoscale Ge 3D islands which align at the facets. The shape anisotropy of these islands is reversed with respect to that found for growth on bare or partially Ga-covered Si(113). One consequence of this change in anisotropy is that the approach presented here might also enable the growth of Ge nanowires extending in the [110] direction.

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