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## SURFACE MORPHOLOGY AND STRAIN RELIEF IN SURFACTANT MEDIATED GROWTH OF GERMANIUM ON SILICON(111)

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### Abstract

The growth of Ge on Si is strongly modified by adsorbates called surfactants. The relevance of the stress on surface morphology and the growth mode of Ge on Si(111) is presented in a detailed *in situ* study by high resolution low energy electron diffraction (LEED) during the deposition. The change from islanding to layer-by-layer growth mode is seen in the oscillatory intensity behaviour of the 00-spot. As a strain relief mechanism, the Ge-film forms a microscopic rough surface of small triangular and defect-free pyramids in the pseudomorphic growth regime up to 8 monolayers. As soon as the pyramids are completed and start to coalesce, strain relieving defects are created at their base, finally arranging to the dislocation network. Without the driving force for the micro-roughness, the stress, the surface flattens again showing a much larger terrace length. The formation process of the dislocation network results in a spot splitting in LEED, since the periodic dislocations at the interface give rise to elastic deformation of the surface. Surprisingly the Ge-film is relaxed to 70% immediately after 8 monolayers of coverage, which is attributed to the micro rough surface morphology, providing innumerable nucleation sites for dislocation.

**Key Words:** Heteroepitaxy, surfactant, dislocation-network, low energy electron diffraction (LEED), strain-relaxation, silicon, germanium, antimony, micro-roughness, surface-undulation.

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### Introduction

The heteroepitaxial growth of lattice mismatched semiconductors has been a challenge in materials science for a long time since the benefits for the semiconductor technology are numerous [3]. But islanding of the growing film, threading defects and a high number of point defects in the grown film are some of the problems in this field. The reason for the difficulties are the different lattice constants, which usually drives the system into the Stransky-Krastanov growth mode (layer growth followed by islanding) without any control of the generation of the misfit adjusting defects. Growing in the kinetically limited regime (low temperature and high fluxes) results in a continuous film but at the expense of a high number of defects and dislocations.

In this paper, we will characterize the modification of the heteroepitaxial growth by surfactants (*surface active species*), which show a way out of the dilemma demonstrated above. A surfactant is an adsorbed monolayer of a third element, changing the surface properties and therefore, the growth behaviour without getting incorporated, but floating as an adsorbate on the growing surface. The mobility of the deposited semiconductor atoms is strongly hindered by the surfactant and results in a layer-by-layer growth mode instead of islanding at these quite high substrate temperatures. The reduction in surface free energy by the surfactant drives the strong segregation to the surface. The doping materials Sb [5, 9] and As [1, 2] have been tested to be qualified as surfactant for the Si/Ge-epitaxy. In this paper, we will focus on the Si(111)/Ge/Sb system, where a periodic dislocation network is formed at a Ge film thickness of 8 ML [1 ML (monolayer) =  $7.21 \cdot 10^{14}$  atoms/cm<sup>2</sup>], confined to the Si/Ge-interface and exactly matching the different lattice constants [5]; no threading defects have been found [8], any Ge film thickness could be grown.

In order to study these effects *in situ*, if possible also during the growth process, a surface sensitive method has to be used. In this study, we have used spot profile analyzing low energy electron diffraction (SPA-LEED) because it provides quantitative and qualitative information about the surface morphology. In contrast to the scanning tunneling microscopy (STM), the

measurements are not only possible after the film growth but also at elevated substrate temperature during the deposition.

The determination of the parallel and vertical (layer distance) lattice constants with an accuracy of 0.005 Å allows the control of the strain relaxation of the growing film. Not only the kind of superstructure, but also size distributions of islands or terraces are available on a length scale from a few atoms up to 2000 Å. The surface roughness is determined by the energy dependence of the spot positions (facets) or the spot profile (islands) in a quantitative way. We also present the direct observation of the generation and development of the misfit adjusting dislocation network at the interface. In contrast to STM, which provides local information, LEED provides the overall information of an surface area in the mm<sup>2</sup> range. Using Si(111)/Ge/Sb as a model system for heteroepitaxial growth, we describe the whole growth process in detail by SPA-LEED, especially the influence of the stress on the surface structure and the formation of the dislocation network during growth. A new model for the generation of the misfit adjusting defects, based on the special surface morphology, is presented.

### Materials and Methods

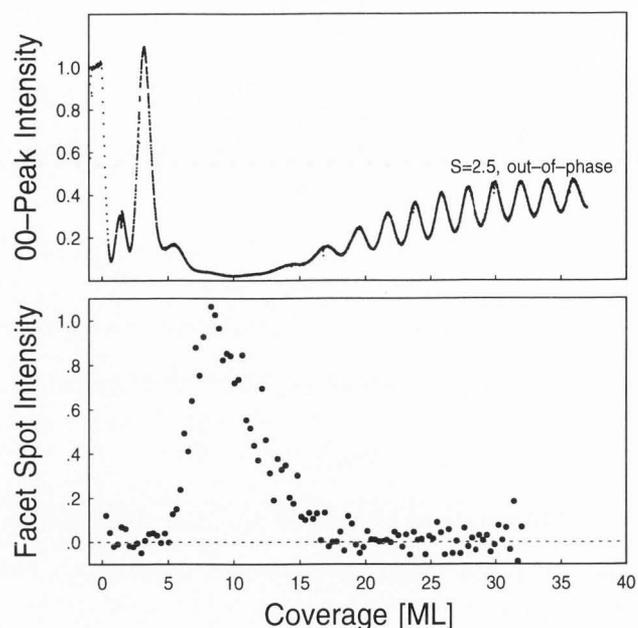
The experiments were performed in a standard ultra high vacuum chamber equipped with a SPA-LEED [13] (spot profile analyzing), a cylindrical mirror analyzer for Auger measurements and a quadrupole mass spectrometer. Using a grazing angle electron gun in a geometry similar to a reflection high energy electron diffraction (RHEED) experiment [4], the films could be grown *in situ* in the same chamber.

The Ge-films were usually grown following the same scheme: 1 ML Sb is adsorbed at a substrate temperature of 670°C, no more than 1 ML Sb sticks at this temperature. The (7x7)-superstructure of the Si(111) surface is changed to a ( $\sqrt{3}\times\sqrt{3}$ )R30-superstructure [12], covering the whole surface in large domains. The temperature was decreased to the growth temperature of 580°C during 5 minutes under Sb flux. This procedure establishes a flat surface with a well ordered ( $\sqrt{3}\times\sqrt{3}$ )-structure, since adsorption of the Sb at 580°C results in a strongly distorted mixture of (2x1)- and ( $\sqrt{3}\times\sqrt{3}$ )-superstructure domains.

During the evaporation of Ge an Sb flux of 0.1-0.2 ML/min was maintained to compensate for Sb desorption, which occurs significantly during the growth of the first 20 ML Ge. We attribute this to a rough intermediate surface structure, occurring between 5 and 20 ML Ge film thickness, as shown in the "Micro Facet Formation". An Sb coverage of at least 0.5 ML is necessary to prevent islanding of the Ge-film [6].

### Growth Oscillations

Evidence of epitaxial layer-by-layer growth may be seen in the intensity oscillations of the central spike of the 00-spot at the out-of-phase condition with  $S =$



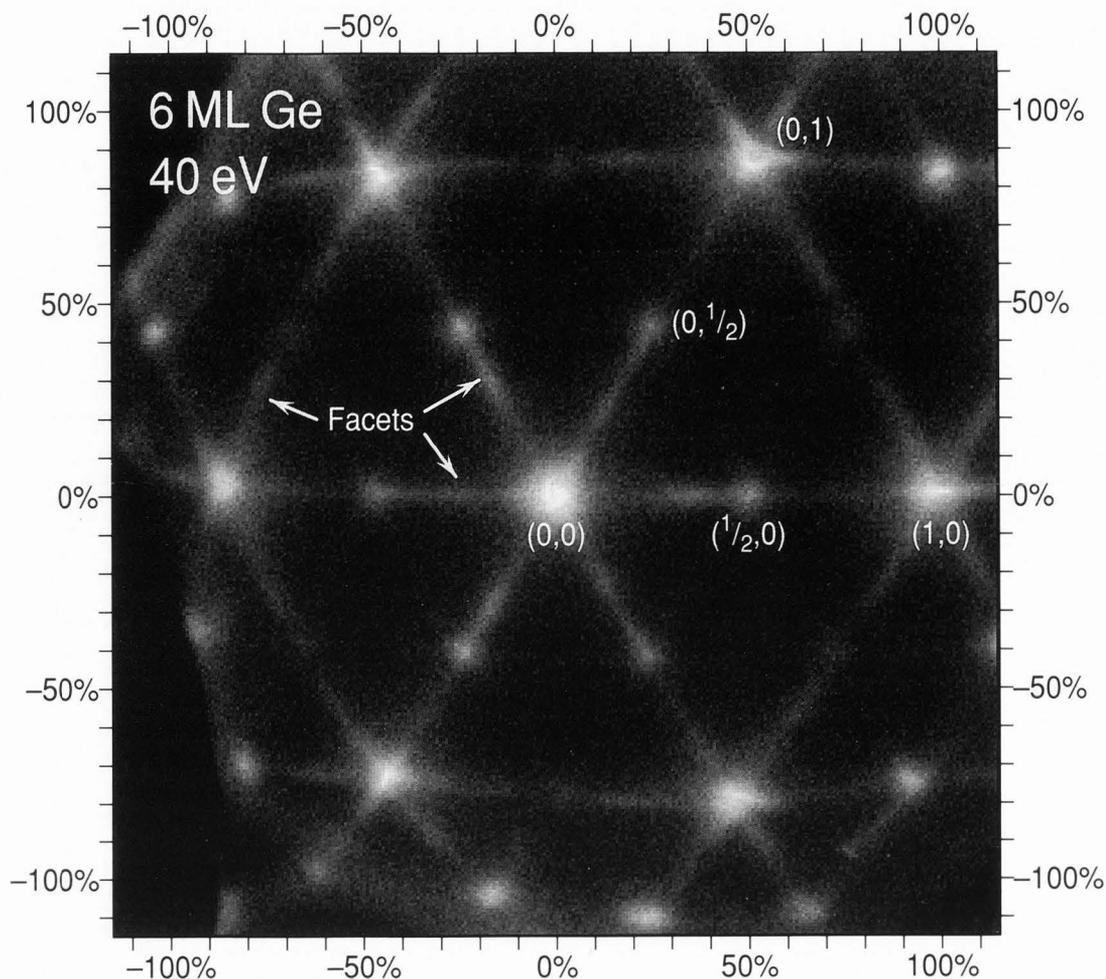
**Figure 1.** (a). Central spike intensity oscillations of the 00-spot during growth at out-of-phase scattering geometry. The later oscillations show a bilayer ( $14.4 \times 10^{14}$  atoms/cm<sup>2</sup>) period. (b). The facet spot intensity shows a sharp peak at 8 ML Ge corresponding to the nucleation of the strain relieving defects. Without the strain as driving force for the micro roughness, the surface flattens again, resulting in a decreasing facet intensity.

2.50 in Fig. 1a (93 eV and 25° incidence angle, electrons scattered from neighbouring terraces interfere destructively, thus reducing the spike intensity for a stepped surface). The occurrence of the pseudomorphic layer of ~3 ML Ge is clearly seen in the intensity maximum at 3 ML. Ge usually grows on the (111) face in a bilayer mode (the layer with three dangling bonds per atom is never forming the surface). Here we observe the formation of such a layer (Ge with three bonds toward the surface), which must be stabilized by the Sb which results in a (1x1) reconstruction [9] as for example As on Si(111). With increasing coverage, the intensity drastically decreases, reflecting a more and more rough surface. Surprisingly, the oscillations return after growth of 10 ML Ge with regaining intensity in the 00-spot, indicating a more and more flat and smooth surface. The period of the later oscillations corresponds to a bilayer growth mode of the Ge-film (1 period =  $14.4 \times 10^{14}$  atoms/cm<sup>2</sup>). After the growth of 100 ML Ge, a sharp and brilliant (2x1)-LEED pattern reflects a perfect layer-by-layer growth of Ge, the terrace size larger than 100 atoms and only kinetically limited.

### Micro Facet Formation

The LEED-pattern of the rough stage after growth of 6 ML is shown in Fig. 2 as a two-dimensional-scan covering the 00-spot, the next neighbored

Surfactant mediated growth of germanium on silicon(111)



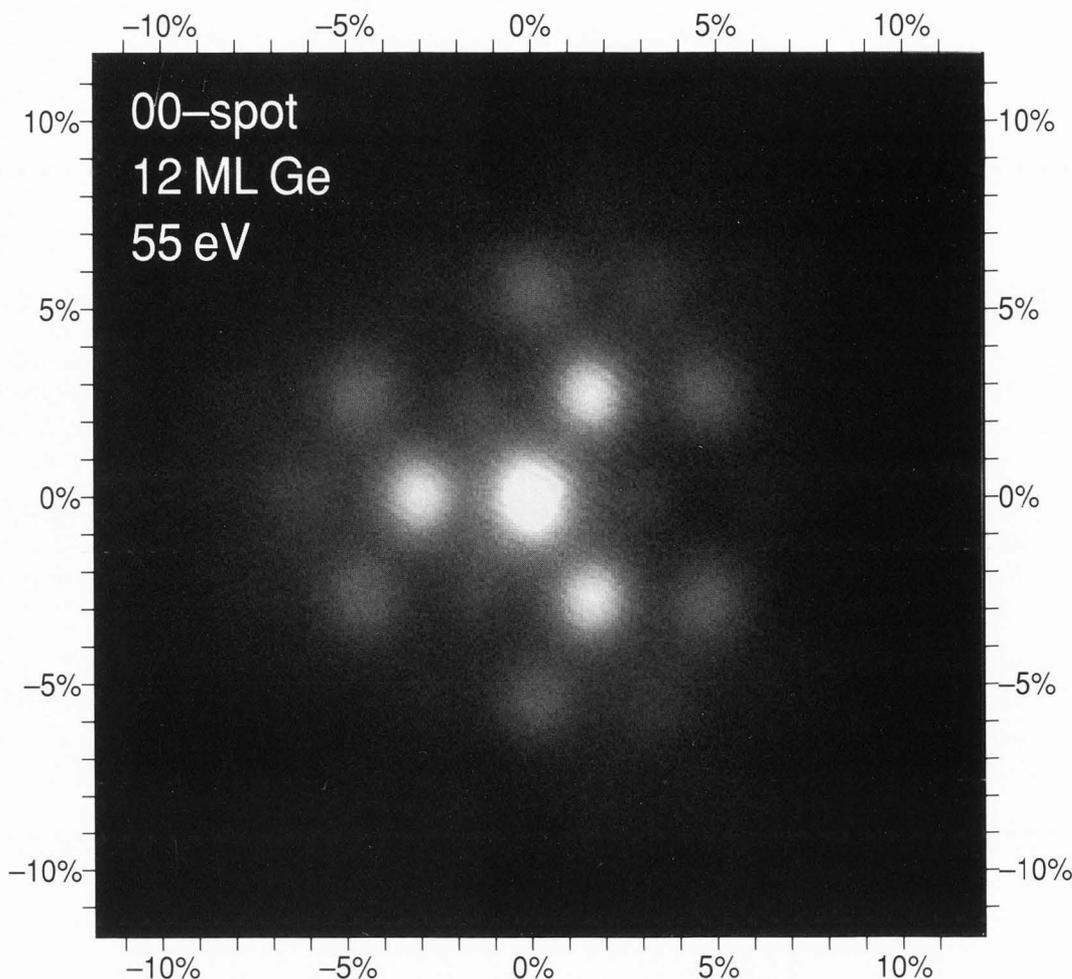
**Figure 2.** LEED-pattern after growth of 6 ML Ge showing the integral order spots, the (2x1)-superstructure spots and very broad and elongated spots resulting from [113]-type micro facets. A logarithmic intensity scale has been used to demonstrate all features.

integer order spots, and a number of (2x1)-spots at 40 eV. The high background reflects a surface with a large portion of defects and irregularities. Between the integer order spots, very broad and elongated spots are also visible. All spots on one straight line move with increasing electron energy into the same direction indicating facets with a [113] type orientation at the surface. Thus, the surface must be composed of facets tilted in only three directions, as a triangular pyramid.

The very elongated form of the spots in k-space reflects a similar but rotated structure in real space. The narrow width corresponds to a long extension on the surface, the broad direction results from a very short extension of the facet. Thus, this elongated form of the facets could easily be understood assuming triangular pyramids constructed of three facets showing the three possible orientations. These pyramids are irregularly arranged over the surface, having different sizes ( $\sim 60$  Å) and heights ( $\sim 8$ -10 ML), they may even be truncated with a flat top for coverages below 8 ML. But,

nearly the whole surface is covered with these faceted areas, since most of the intensity of the Brillouin zone is confined in the facet spots. Meyer *et al.* have, for the same system, observed the formation of a rough surface structure at 6 ML coverage [9]. From those STM results, it is not clear whether at 8 ML Ge coverage the pyramids are still truncated at the top or not.

The formation of this quite rough surface structure is a very efficient way to relieve some of the strain in the Ge film in this still pseudomorphic growth regime. The small islands allow the Ge in the outer layers of the pyramids to relax partially towards its own lattice constant. This process is most effective for small island sizes. The size of  $\sim 60$  Å of the triangular pyramids is of the same order as the coincidence distance ( $\sim 75$  Å) of the Si and Ge lattice. It would be difficult for the islands to grow larger, since more and more strain would be accumulated. The formation of smaller pyramids would allow a larger strain relief, but only for much smaller amounts of coverage.



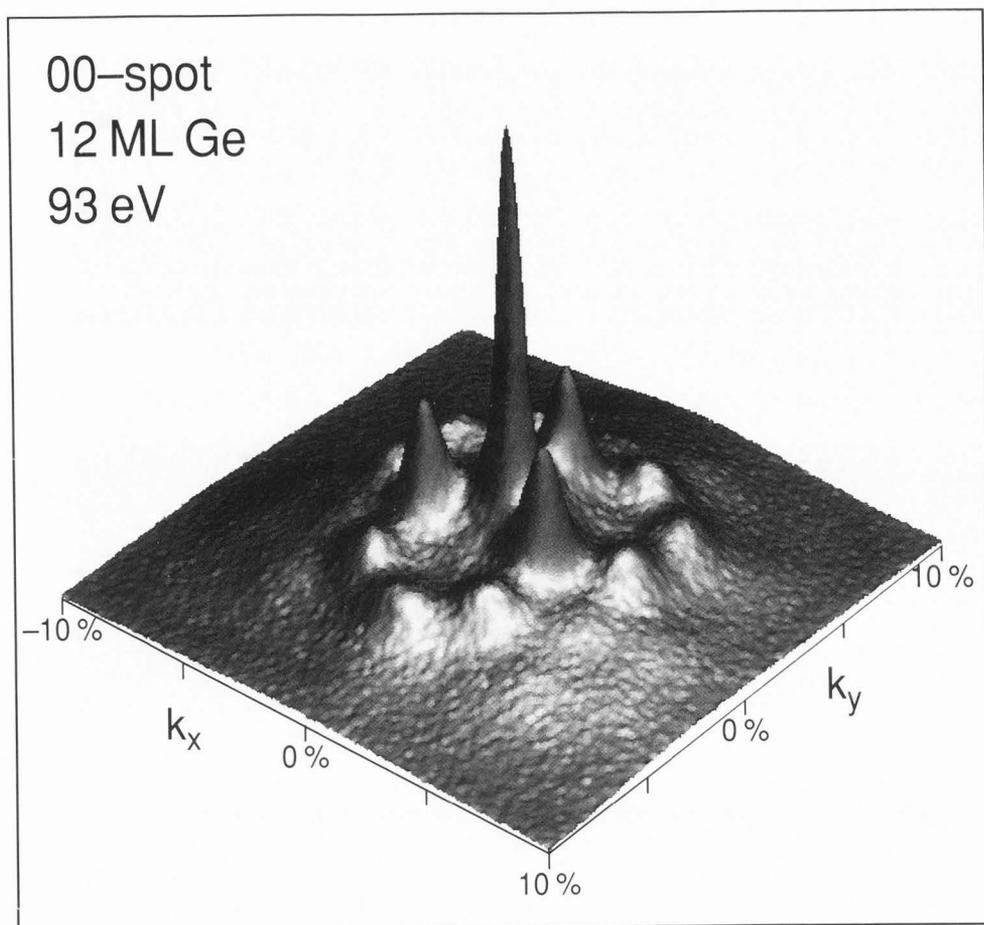
**Figure 3.** Spot splitting of the 00-spot resulting in a network of satellite spots with a mesh-length of 3.20% BZ. The surface is elastically deformed by the interfacial dislocation network, resulting in a periodic undulation with an amplitude of 0.6 Å, acting as a two-dimensional phase grating.

The development of the micro pyramids during growth is studied by recording the intensity of one of the facet spots as a function of coverage (Fig. 1b). The facet spot intensity steeply arises after 5 ML of Ge, with a maximum at 8 ML (i.e., the coverage, where misfit relieving defects were introduced), and vanishes at ~20 ML. The growth of the pyramids is limited by the distance to each other (~60 Å) and their facet orientation. A maximum volume is reached after the total amount of 8 ML Ge, corresponding to a base length of ~60 Å unit cells and a height of ~10 ML. With additional coverage, the pyramids coalesce by filling up the trenches between them. But these lattice sites show the highest stress and are the most unfavorable growth sites. So it is not surprising to find the nucleation of misfit adjusting defects in this stage of growth. Partial dislocations are gliding from the base of the pyramids beneath them, creating a stacking fault and relieving strain. The formation of the so called *hut-cluster* [10] in the Si(100)/Ge-system is a quite similar process of strain relaxation.

As soon as the defects are generated, the driving force for the roughening of the growth front is lost and the surface starts to smooth by filling up the trenches between the islands as seen in the decreasing intensity of the facet spot intensity (in Fig. 1b) and the increasing oscillating intensity of the 00-spot (Fig. 1a).

#### Dislocation Network Formation

The formation of the strain relieving and misfit adjusting dislocation network at the interface is directly observed by the elastic deformation of the Ge film resulting from the dislocations themselves. The network is composed of three dislocation "lattices" consisting of alternating parallel rows with and without a stacking fault connected by Shockley partial dislocations, each rotated by 120° [5, 8]. The 90°-Shockley partial dislocation elastically deforms the Si and Ge layers close to the interface [11]. The surface also follows this vertical undulation of the lattice planes. Electrons reflected



**Figure 4.** Up to five orders of satellite spots are visible in this semi three-dimensional plot of the LEED intensity. A linear intensity/height scale has been used. Most of the intensity is confined in the satellite spots at the expense of the central spot.

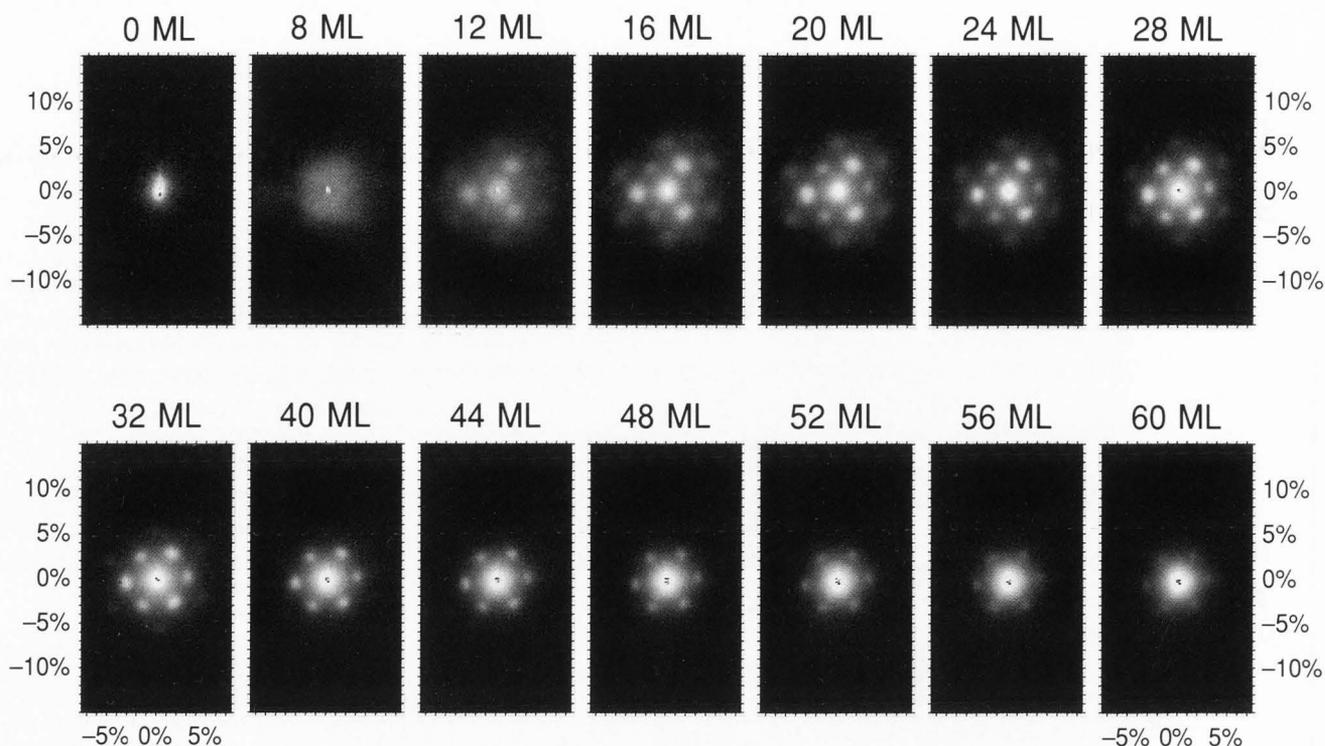
from this surface undergo a phase shift due to this vertical displacement of less than 1 Å. Thus, the surface forms a two dimensional periodically warped face resulting in spot splitting.

This spot splitting of the 00-spot into a set of satellite spots is shown in Fig. 3, resulting after growth of 26 ML Ge. The satellite spots are arranged on a hexagonal net with a mesh-length of  $3.20\% \pm 0.10\%$  BZ (100% BZ is defined as the length between the integer order spots, i.e., the length of the Brillouin zone). Up to 5 orders of satellite spots with a threefold symmetry can be seen at some energies, as visible in Fig. 4 in a semi three-dimensional image of the LEED intensity. These satellite spots are not only surrounding the 00-spot, but also all other integral order spots as well as the extra spots of the Sb-reconstruction.

The intensity of the satellites change very slowly with electron energy. The other integer-order spots as well as the superstructure spots show all the same intensity-behaviour in the satellites as the 00-beam. It is therefore concluded that just a small vertical displacement of the unit cells at the surface without any lateral component are responsible for the splitting. Due to the very weak but nevertheless distinct dependence on energy, the corrugation has to be small; we estimate a height variation of the surface in the order of 1/5 of the step

height  $d$  of 3.27 Å of Ge. A detailed analysis of the satellite spot intensity as well as the derivation of the shape of the surface corrugation will be further addressed in a forthcoming publication [7].

The evolution of the satellite spots with coverage is seen in Fig. 5 in a series of LEED-images during the growth. The first intensity in the satellite spots is seen after 8 ML, the network of spots is well developed at 12 ML with an intensity maximum at 26 ML and is still visible up to 60 ML. The integral intensity in the spots (as the sum of intensity in equivalent spots) is plotted as a function of coverage in Fig. 6a. The satellite spots have their intensity maxima at different coverages, reflecting a change in shape of the elastic deformation. The early decrease of the higher order spot,  $I_{-11}$ , reflects the faster weakening of the steeper parts of the deformation. The deformation gets more and more six-fold as seen by the late maximum of the  $I_{10}$  spot and the approach to the curve of the  $I_{01}$  spot. The flattening of the surface by filling up the trenches between the micro pyramids is seen in the increase of the sum over all satellite spots (including the 00-spot),  $\Sigma I_{ij}$ , to its final value at  $\sim 30$  ML. The steep decrease of  $\Sigma I_{ij}$  around 5 ML reflects the formation of the micro pyramids, which cover the whole surface at 8 ML.



**Figure 5.** The evolution of the dislocation network is reflected in this series of two-dimensional scans of the satellite spots during deposition. The network starts to form at 8 ML and is completed at 18 ML. The surface undulation vanishes with increasing coverage. Nearly no variation of the spot distance could be seen. The spots get sharper with increasing coverage (compare 12 ML with 44 ML, a logarithmic scale has been used to demonstrate all features).

One of the most interesting questions is the coverage needed to complete the dislocation network to a full adjustment of the lattice constants. This information is expressed in the distance of the satellite spots to the 00-spot, i.e., the distance of the dislocations at the interface. The distance  $\Delta k_{\text{sat}}$  of the satellite spots for a complete relaxation of the film to the Ge bulk lattice constant is determined by the lattice mismatch  $\Delta a_0$ , the number of identical dislocation nets  $n$  (depending on the kind of symmetry of the surface orientation, here three-fold, thus three identical dislocation nets), the sine of the angle of the Burger's vector of the dislocation to the dislocation line (the full dislocation formed by the two Shockley partials is a  $60^\circ$  dislocation) and the number  $\text{dim}$  of dimensions to be relaxed (here the two directions of the surface):

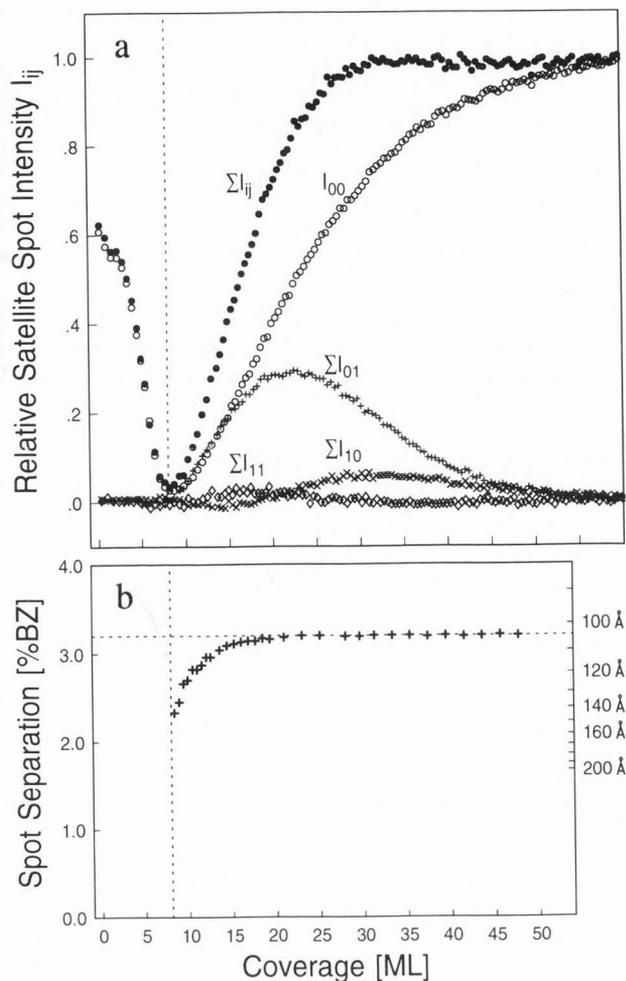
$$\Delta k_{\text{sat}} / k_{10} = \Delta a_0 / a_0 \cdot \text{dim} / n \sin 60^\circ = 3.20\% \text{ BZ}$$

The spot separation plotted in Fig. 6b shows the astonishing result, that already at 8 ML, which is the thickness where the first dislocations are generated, the part of the Ge film contributing to the 00-spot is immediately relaxed to 70% of the Ge bulk lattice constant. Within only 4 additional monolayers of Ge a relaxation of 90% and a full compensation of the lattice mismatch is achieved at a total coverage of only 18 ML of Ge with

a dislocation distance of 104 Å, i.e., a spot separation of 3.20% BZ. Thus, most of the dislocation network is at least partially completed after only  $\sim 12$  ML Ge.

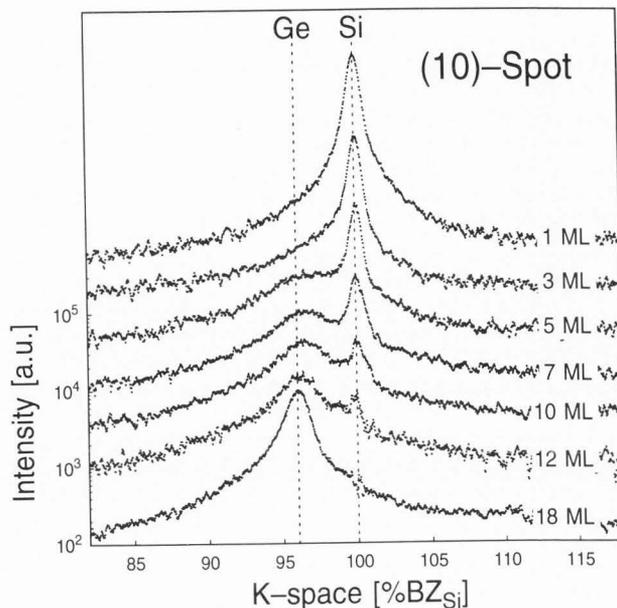
Very outstanding is the very short period for the complete relaxation of the Ge-film. It is hard to imagine the driving force for this process, since already by the creation of the first loops or half loops of dislocations, the lattice strain is reduced and needs to be accumulated again by the growth of additional layers to create more dislocations. Following this process, it is not even expected to fully relieve the lattice mismatch for thick films. We attribute the influence of the surface morphology to be responsible for this effectiveness in strain relief. At 8 ML coverage, the small pyramids provide a larger number of equivalent nucleation sites for the dislocations than needed for a complete relaxation. Due to the more or less non-continuous, rough surface, no lateral dispersion of the strain relaxation over more than one pyramid is possible, thus all the dislocations are nucleated independent of each other during the coalescence of the pyramids. So, the full strain relief is achieved as soon as all facets are overgrown, i.e., at 18-20 ML.

Additional information is available from the spot profile of the satellites, which is described by a sum of Lorentzian functions. The full width at half maximum



**Figure 6.** (a) Integral intensity of the strongest satellite spots during deposition (scattering phase  $S = 3.0$ ). The sum of intensity in equivalent spots is plotted. The formation of the dislocation network starts at 8 ML, as seen at the steep rise of  $I_{01}$ . The change in shape of the surface undulation shifts the maxima of  $I_{10}$ ,  $I_{01}$  and  $I_{11}$  to different values. The increase of  $\Sigma I_{ij}$  reflects the flattening process of the surface up to a coverage of 30 ML. (b) The satellite spot separation is determined by the average distance of the dislocations in the network. The Ge-film is immediately relaxed to 70% after the formation of the first dislocations. A complete adjustment of the lattice constants is achieved after 18 ML, resulting in a spot splitting of 3.20% BZ, i.e., an average distance of the dislocations of 104 Å.

(FWHM) of the satellite spots increases with the order of the spots and the scattering phase  $S$ , thus describing the irregularities in the dislocation network arrangement. A detailed analysis of the spot broadening is addressed in a forthcoming paper [7]. The meandering and diffusion of the dislocations during the formation of the dislocation network may be seen in the decrease of the FWHM of the satellite spots especially in the range



**Figure 7.** Radial scans through the 10-spot show the change in lattice constant from Si toward the Ge value. A shift is already apparent in the still pseudomorphic growth regime below 8 ML caused by the partial relaxation of the Ge in the micro pyramids toward its own lattice parameter.

from 8 ML (1.7% BZ) to 18 ML (0.8% BZ), thus a process toward higher regularity of the network (compare in Fig. 5 the image at 12 ML and 44 ML).

#### Lattice Constant

The change of the lattice constant of the Ge-film has been observed in the change of the position of the 10-spot. In Fig. 7, radial scans through the Si-10-spot are plotted in a logarithmic intensity scale as a function of coverage. The positions of the Si- and Ge-10-spots are marked by dashed lines. With increasing coverage, the intensity of the Si-10-spot decrease strongly to zero (the electrons do not penetrate deeper than  $\sim 12$  Å into the film), while a second very broad (FWHM 6% BZ) spot arises at a position closer to the 00-spot resulting from the Ge film. In the range from 5 to 12 ML, the total intensity is strongly decreased due to the faceted areas on the surface as already seen for the 00-spot.

Already in the still pseudomorphic growth regime-beginning at 3 ML, a very broad spot arises at  $\sim 2\%$  BZ continuously moving to larger distances with increasing coverage. We attribute this broad peak to the effort of the Ge to relieve the strain in the pseudomorph, defect free and micro faceted pyramids by a partial relaxation toward the Ge-bulk lattice constant. The distance of the Ge atoms at the top of the pyramids is  $\sim 2.5\%$  larger than the Si lattice constant of the substrate. Thus, the mechanism resulting in the strain relief by the micro roughness is directly observed.

At ~8-10 ML, when the first dislocations are generated, the position of the Ge-10-spot is determined by the dislocation distance of ~150 Å as shown in Fig. 6b. At a coverage of 18 ML, the Ge-10-spot appears at a position consistent with the Ge-bulk lattice constant and gets more and more intense. This is the already relaxed Ge-film with nearly the Ge bulk lattice constant. The 10-spot also shows the additional satellite spots resulting from the interfacial dislocation network (not visible in the radial scans).

### Conclusions

We have demonstrated in this work the manner in which the strain in surfactant mediated heteroepitaxial growth is changing the surface morphology and thus changing the way strain relieving dislocations are generated. The surface free energy as well as the lattice mismatch are the dominant parameters controlling the growth mode close to the thermodynamical equilibrium. Ge is able to wet Si due to the lower surface energy, but the stress due to the 4.2% lattice mismatch drives the system into islanding after forming the 3 ML thick Stransky-Krastanov film. Selective change of the growth kinetics by reducing the mobility of the evaporated species without changing the high substrate temperature allows layer-by-layer growth with excellent bulk properties. As soon as a Ge atom has taken in a lattice site, it is bonded not only to the Ge substrate, but with all four electrons either to the Ge lattice or to the Sb monolayer, thus strongly reducing the probability of desorbing again from a kink or step site.

The strain is relieved by a special mechanism of creating a rough surface, composed of very small defect free, triangular pyramids. The Ge in this pyramid is now able to extend its lattice spacing toward its own bulk lattice constant, thus partially relieving strain. This process is not possible for a flat layer or for islands much larger than the coincidence distance (~75 Å) of the Si and Ge lattice. To maximize the effectiveness of this process, nearly all the pyramids have the same size. This stage of growth seems to contradict the "island-free"-layer-by-layer growth which is claimed for the surfactant mediated epitaxy. But the islands we have observed are much smaller and free of defects than the islands that usually occur in the heteroepitaxial growth without surfactants.

As soon as the pyramids are completed with definite facets, Ge has to be placed into the lattice sites with the highest stress at the bottom of the trenches between the pyramids. This is connected with the generation of a dislocation parallel to the surface, which dissociates into two Shockley partial dislocations gliding on the (111) plane beneath the pyramid, creating the stacking fault and strongly relieving strain. Since the surface is still very rough in this stage, the strain relaxation could not disperse over a larger region, thus nearly all dislocations are generated at the same coverage, determined by the size of the pyramids.

The formation of the dislocation network is observed by the elastic deformation of the lattice due to one of the Shockley partial dislocations of the network. This undulation of the lattice is also seen at the surface and detectable with LEED. This is the first time the dynamics of the formation of a dislocation network has been observed *in situ* during the growth process. The dislocation network is completed during a range of coverage of only 10 ML, with a relief of 90% of the strain already after 4 additional ML of Ge coverage after generation of the first dislocations. Without the stress as the driving force, the surface is smoothed again by preferred filling of the trenches (the pyramids itself are not dissolving since Ge atoms, which

have a lattice site, are no longer likely to diffuse due to the extra bond to the surfactant).

In this system, the change of surface morphology is the dominant factor in determining the final microstructure of the Ge-film. Instead of three-dimensional clustering of the Ge with an uncontrolled defect structure, we are now able to obtain films with all the strain-relieving defects confined in a network at the interface. The final product is a fully relieved defect-free Ge film with a flat surface on Si(111), a model system for perfect heteroepitaxial growth.

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**Editor's Note:** All of the reviewer's concerns were appropriately addressed by text changes, hence there is no **Discussion with Reviewers**.