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STRAIN DISTRIBUTION DURING GROWTH OF Ge/Si(001) AND THE EFFECT OF SURFACTANT LAYERS

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Abstract

Grazing incidence X-ray diffraction has been employed to determine directly the distribution of strain in the plane of the interface during deposition of Ge onto Si(001). The corresponding strain distribution has also been deduced for a relaxed island whose atomic structure has been determined by molecular dynamics. The results illustrate the central role of elastic deformation of islands in the initial stage of strain relief. The results are also compared with those for growth with a Sb surfactant layer which suppresses island formation. An investigation of surfactant-like behaviour is also presented for homoepitaxial growth of Ag on Ag(111), where submonolayer coverages of Sb promote a layer-by-layer growth mode over a wide temperature range.

Key Words: Strain, epitaxial growth, germanium, silicon, surfactant, molecular dynamics, X-ray diffraction, growth modes.

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Introduction

Epitaxial growth is usually considered to proceed in one of three classical growth modes. These are conventionally named Frank-van der Merwe or layer-by-layer growth, Volmer-Weber growth, where the epilayer islands grow immediately, and Stranski-Krastanow growth, where the film grows initially in a layer-by-layer fashion followed by islanding of the surface. The growth mode occurring in a particular system is governed by the surface energies of the substrate and epilayer material and the energy of the interface between the two materials. It has long been recognised that the growth mode may be modified by reducing the growth temperature, thus preventing the system attaining equilibrium during growth, but this usually has a detrimental effect on the material quality. More recently, it has been shown that the growth mode of Ge on Si(001) may be altered from the usual Stranski-Krastanow mode to a layer-by-layer mode by deposition of a monolayer or less of As or Sb prior to the growth of Ge (Copel *et al.*, 1990). The group V element continually segregates to the surface during deposition and hence this effect is often referred to as surfactant growth. In this paper, we report on the strain distribution within Ge/Si(001) during deposition with and without a surfactant layer, determined using grazing incidence X-ray diffraction (GIXRD). The strain distributions are compared with calculated strain distributions from molecular dynamics simulations. Surfactant growth has been employed in semiconductor growth; here we describe its use in homoepitaxial metal growth, where a similar change in growth mode is observed.

Strain Distribution in Ge/Si(001)

The epitaxial growth of Ge on Si has been extensively studied from a wide range of viewpoints, yet despite this, it continues to raise further questions on the physics of heteroepitaxial growth. The effect of strain in a lattice mismatched system has been known for a relatively long time. Through an elastic distortion of the overlayer, the strain energy is allowed to increase as the layer grows pseudomorphically. Eventually, however,

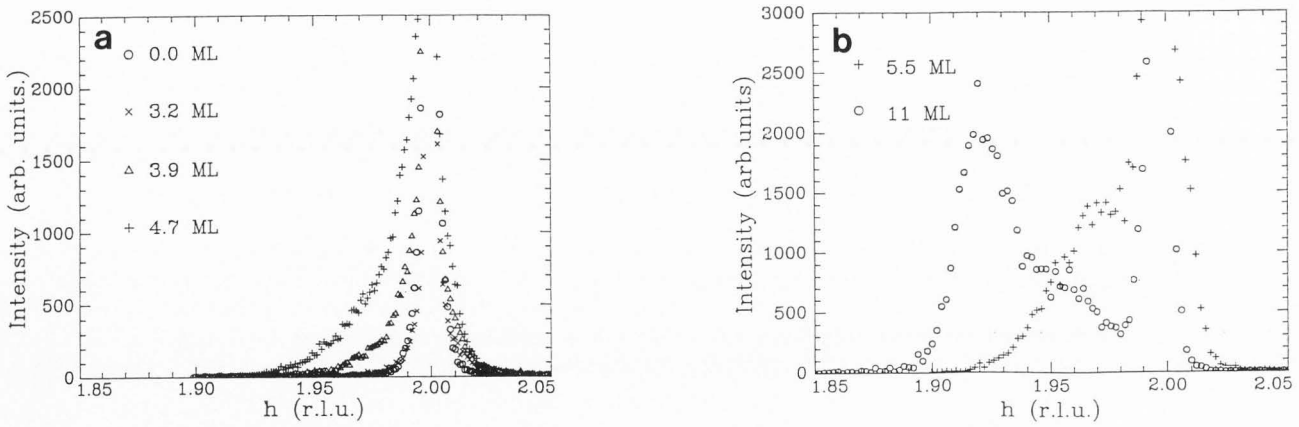

Figure 1. Radial scans through the (2,0) Bragg peak [corresponding to (220) in bulk notation] for Ge coverages (**a**) up to 5 ML, and (**b**) above 5 ML. The onset of relaxation is observed at 3.9 ML.

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the strain energy becomes too large to sustain, and at some critical thickness, the layer is able to overcome a reordering energy barrier, and thereby, reach a lower energy, more relaxed state.

In relatively lightly strained systems, the critical thickness may be many micrometers, at which point the film becomes substantially relaxed through dislocation. The prediction of the critical thickness of such systems is now fairly accurate, and has been described by various models based on the existence and propagation of dislocations in the overlayer (Matthews and Blakeslee, 1975). In a more highly strained system like Ge on Si, which has a 4% lattice mismatch, the initial strain relaxation mechanism has been found to be rather different. This makes an accurate prediction of critical thickness rather more difficult, since it requires an in-depth understanding of the overlayer growth processes.

The Ge/Si system normally grows in the Stranski-Krastanow growth mode at conventional growth temperatures of about 500°C, whereby three dimensional (3D) growth of islands occurs after only about 3 monolayers (ML) of Ge have formed. Even though this growth mode has been known for many years, the exact nature of the islanding still remains open to question. It appears that the initial islands are coherent with the substrate, and only become dislocated when they reach a critical size (Eaglesham and Cerullo, 1990). Under specific growth conditions, it also appears as though some elastic deformation of the Si substrate takes place in association with these islands.

Grazing incidence diffraction provides a sensitive probe of strain relaxation in monolayer-thick films (Macdonald *et al.*, 1991). The scattering vector **Q** lies in the plane of the sample surface and hence the distribution of in-plane lattice spacings is measured directly in radial scans in reciprocal space. The intrinsic peak width parallel to the interface $\Delta\mathbf{Q}_{\parallel}$, is much narrower than that perpendicular to the interface $\Delta\mathbf{Q}_{\perp}$, due to the much larger extent of the epilayer parallel to than normal to the interface, thus providing good resolution for ultrathin layers. The Ge films were grown at a deposition rate of 1 ML per 18 minutes at a substrate temperature of 550°C. Scans were performed *in-situ* on the grown surface at the surface diffraction facility at the Daresbury synchrotron radiation source (Fig. 1). The peak profile remains unchanged for a coverage $\theta \leq$ 3 ML due to the coherent epitaxial nature of the Ge layer. The wings of the peak are not substantially broadened indicating the high crystalline quality of the overlayer. At 4 ML, a weak shoulder appears on the Bragg peak due to the onset of strain relaxation. At $\theta \approx$ 6 ML, a substantial increase in the amount of relaxed material is observed. The epilayer contains almost fully-relaxed Ge at θ = 11 ML, but lattice constants intermediate between those of Ge and Si are also observed (Williams *et al.*, 1991).

Insight into the mechanism of strain relaxation may be obtained from comparison with transmission electron microscopy (TEM) and scanning tunnelling microscopy (STM) results. The onset of relaxation at 3-4 ML coincides exactly with the coverage at which islanding of the Ge overlayer occurs (Mo *et al.*, 1990). Furthermore, the STM images show that, in the range 3-6 ML, the islands are small (few tens of nm in length and about 3 nm high), rectangular clusters having well-formed <105> facets. At 6 ML, these small islands coalesce to form large islands having <113> facets. TEM images indicate that the macroscopic islands occurring at about 8 ML coverage are dislocation-free, even though the island height is ≈ 50 nm, well in excess of the calculated critical thickness of ≈ 1.5 nm (Matthews and Blakeslee, 1975). Thus, it appears that strain relaxation occurs when islands are formed on the Ge surface. However, these islands are dislocation-free and thus, the question arises as to how the islands aid the relaxation process.

The critical thickness can be thought of as the number of layers of film that can be deposited above which it is energetically favourable to either form 3D islands rather than form pseudomorphic layers; or create

misfit dislocations to relieve the strain in the film. To perform the energy calculations, within a molecular dynamics framework, the Si substrate was modelled by a crystal block with the (001) face representing the surface and atoms interacting through an empirical potential of the Stillinger and Weber (1985) or Tersoff (1989) form. The pseudomorphic Ge epilayer was represented by Ge atoms having the same in-plane lattice coordinates as the substrate, but expanded perpendicular to the surface. Strain-relief was modelled by introducing a dislocation at or near the interface. Edge, screw and 60° glissile dislocations were considered. However, although there is some dependence on the type of dislocation and the interatomic potential used, the critical thickness in all cases was found to exceed 10 ML.

To determine the formation energy of 3D clusters, islands with (111) facets were constructed on the Si(001) substrate and the system allowed to relax as before. A comparison of the island formation energies with those of strained layers having the same number of atoms results in the former being lower for coverages in excess of 2.6 ML, in good agreement with the observation of islanding with STM and TEM at coverages of 3 ML and with the onset of strain relief at about the same thickness. The agreement between these values and also the fact that dislocation formation would be unfavourable up to a coverage greater than 10 ML does indeed confirm that the initial onset of strain relaxation is caused by islanding of the surface rather than by generation of dislocations.

The mechanism for the onset of strain relief by islanding may be visualised from a scaled diagram of the atomic coordinates in the relaxed island of Ge, as shown in Figure 2. The atoms around the edges of the island are seen to relax as a result of the unconstrained surfaces. This results in very slight bowing of atomic planes within the islands and in the topmost atomic layers of the substrate underneath the island. This bowing has also been suggested from the observation of strain contrast in TEM images (Eaglesham and Cerullo, 1990). The in-plane strain distribution may also be deduced from the atomic coordinates resulting from the molecular dynamics calculation. Hence, the scattering function may be calculated for the relaxed island (Fig. 3) and compared with the radial scans at grazing incidence shown in Figure 1. Thus, it is seen that the islanding leads to a shoulder on the lower side of the Bragg peak, which is similar to that observed in the X-ray diffraction scans for coverages around 3-5 ML. In Figure 3, the intensity falls off monotonically away from the Bragg peak, which would be intuitively expected for an elastically deformed island. The observation of a double-peaked structure in the strain distribution at 11 ML would be consistent with the existence of dislocations, which are likely to form as the islands increase in size.

Strain Distribution During Surfactant Growth

Copel *et al.* (1990) have shown that deposition of about a monolayer of As or Sb onto the Si surface prior

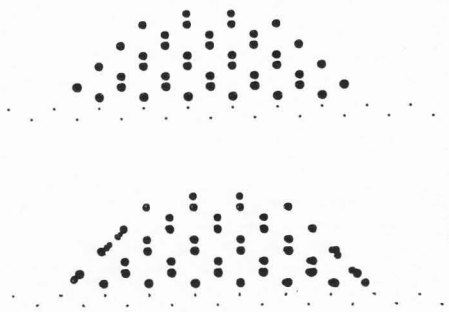

Figure 2. A cross-sectional view through the island in the molecular dynamics simulation (**a**) before relaxation, and (**b**) after the island was allowed to relax. The atoms around the edges of the island, which have <111> faces, are seen to undergo elastic distortion.

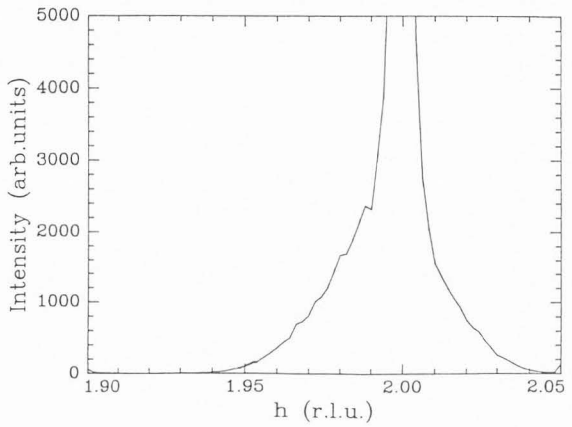

Figure 3. The scattered intensity for a radial scan corresponding to Figure 1, calculated for the relaxed island shown in Figure 2. A shoulder is observed on the lower side of the Bragg peak in agreement with the data for the early stages of strain relief.

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to deposition of Ge suppresses the growth of islands. We again used GIXRD to determine the strain distribution in a Ge film grown with a 0.7 ML Sb surfactant layer, as described in detail Thornton *et al.* (1992). The early depositions of both Sb and Ge onto the Si substrate resulted in no change to the profile of the radial scan, and hence in the distribution of in-plane lattice spacings. This is indicative of pseudomorphic growth, where the overlayer is completely strained to the lattice parameter of the substrate. After a deposition of ~ 11 ML of Ge, however, a small shoulder becomes apparent on the Bragg peak, which is attributable to the onset of strain relaxation of the Ge overlayer (Fig. 4). The absence of strain relaxation up to 8 ML coverage confirms the role of islanding in the onset of relaxation without the surfactant. With such a mechanism unavailable as a means of achieving strain relaxation, the Ge film continues to increase in thickness, with the strain energy increasing correspondingly. This must continue until another

mechanism for strain relief becomes favourable over further pseudomorphic growth.

Beyond the observed onset, the development of strain relaxation in the overlayer was monitored as a function of coverage. As can be seen in Figure 4, the shoulder to the Bragg peak gradually shifts further away and increases in intensity with overlayer coverage. After ~ 21 ML, a distinct feature becomes apparent at h = 1.975 reciprocal lattice units (r.l.u.), which suggests at least some meta-stability in the overlayer structure. At higher coverages, another feature gains intensity at h = 1.94 r.l.u. as further strain relaxation occurs. The growth of this peak in the distribution of lattice spacings seems to occur at the expense of the remainder, showing that a more complete relaxation is achieved with increasing coverage. The overall view, therefore, is one where a second stage of strain relaxation takes place over a stable, partially relaxed layer of ~ 30 ML in thickness. By comparing the scans for angles of incidence above and below the critical thickness, it could be deduced that the more relaxed material was situated near the surface.

The precise mechanism for the suppression of islanding and of eventual strain relief raise several interesting possibilities. Clearly, the suppression of the islanding is linked to the surface segregation of the As or Sb atoms. Comparison of the energies of Si/As/Ge with Si/Ge/As show that the latter structure has the lower energy by about 1.4 eV per dimer (Copel *et al.*, 1990). Thus, the most likely mechanism is that a site exchange occurs rapidly between the incoming atom and a Sb or As atom, thus burying the Ge atom and hence drastically reducing its surface diffusion. The mechanism for strain relaxation at about 10 ML seems to be caused by the formation of novel V-shaped trenches in the Ge overlayer, as observed by STM (Jusko *et al.*, 1992), which are later filled in with Ge at much higher coverages, resulting in the defects observed by TEM (LeGoues *et al.*, 1990).

Other Surfactant-Related Systems

The growth of Ge on Si has been the subject of several recent studies of the effects of surfactant layers on the growth mode. Sub-monolayer coverages of As, Sb and Te (Copel *et al.*, 1990, Higuchi and Nakanishi, 1991) have all been shown to suppress islanding of the Ge film. In the overgrowth of an embedded layer of Ge on a Si substrate with a Si cap with chemical vapour deposition (CVD), adsorbed H atoms on the growing surface helps maintain an abrupt Ge/Si interface (Copel and Tromp, 1991). The use of a Sb surfactant layer during growth of Ge/Si superlattices has also been shown to yield more abrupt interfaces as demonstrated by secondary ion mass spectroscopy (SIMS) studies (Fujita *et al.*, 1990). Whereas, most studies of the effect of surfactants have concentrated on the Ge/Si system, we now describe briefly other studies of possible surfactant-related effects in homoepitaxial metal growth and III-V systems.

Homoepitaxial metal growth

Surfactant layers may also be used to modify the mode of homoepitaxial growth as reported recently (van der Vegt *et al.*, 1992). Specular reflectivity of X-rays may be used to give detailed quantitative information on the surface morphology during crystal growth as well as defining the growth mode (Vlieg *et al.*, 1988, van Silfhout *et al.*, 1989). Reflectivity measurements were performed as a function of time during deposition of Ag onto a clean well-oriented (miscut < 0.05°) Ag(111) surface. The incident angle was 5.5°, corresponding to a (1/2,1/2,1/2) reflection, where scattered radiation from neighbouring (111) planes interfere destructively. For a growth temperature of 575°K, the reflected signal did not decrease with deposition time, indicating a step flow mode of growth (Fig. 5a). At lower growth temperatures, the intensity decreases monotonically, indicating roughening or islanding of the surface. At temperatures below 225°K, the intensity decay followed closely the exponential behavior expected for Poisson statistics i.e., growth without inter-layer diffusion. Oscillations in the reflected intensity, which would indicate layer-by-layer growth, were not observed throughout the investigated temperature range of 175-575°K.

Similar measurements were repeated following deposition of 0.2 ML of Sb onto the surface, in order to examine the effect of Sb on the growth mode. The variation of the specular reflected intensity with time was dramatically modified, displaying strong oscillations for growth temperatures down to the lowest temperature investigated of 225°K (Fig. 5b). Thus, the sub-monolayer coverage of Sb has induced layer-by-layer growth, which did not occur without the Sb for the temperature range under investigation.

Whereas the X-ray data do give a clear picture of the growth mode, they do not give direct information on the underlying microscopic mechanism. The contrast between the morphology with and without the Sb surfactant indicates that the Sb atoms promote diffusion of Ag between atomic layers. Field-ion microscopy studies of W and Ir (Wang and Erlich, 1991), indicate that surface adatoms experience potential barriers at descending steps. One possible model for the observed behaviour is that the Sb atoms decorate the island edges and hence reduce the energy barrier to inter-layer diffusion. The suppression of islanding has also been observed in deposition of Fe or Ni onto Cu(100) surfaces as a result of adsorption of 0.5 ML of oxygen onto the surface. However, in this case, such behaviour may be attributed to the respective heats of formation of NiO, FeO and CuO (Egelhoff and Steigerwald, 1989). Such chemical effects have long been known to modify bulk crystal growth. In the case of homoepitaxial growth of Ag, no such chemical effects can occur and hence the Sb atoms must affect the growth in a more subtle way.

III-V systems

Whereas, group V and VI atoms act as surfactants in growth of Ge/Si, there have been no examples of surfactant behaviour in III-V growth. This is hardly surprising in view of the presence of anions and cations at the surface. Nevertheless, it is well known that dopants,

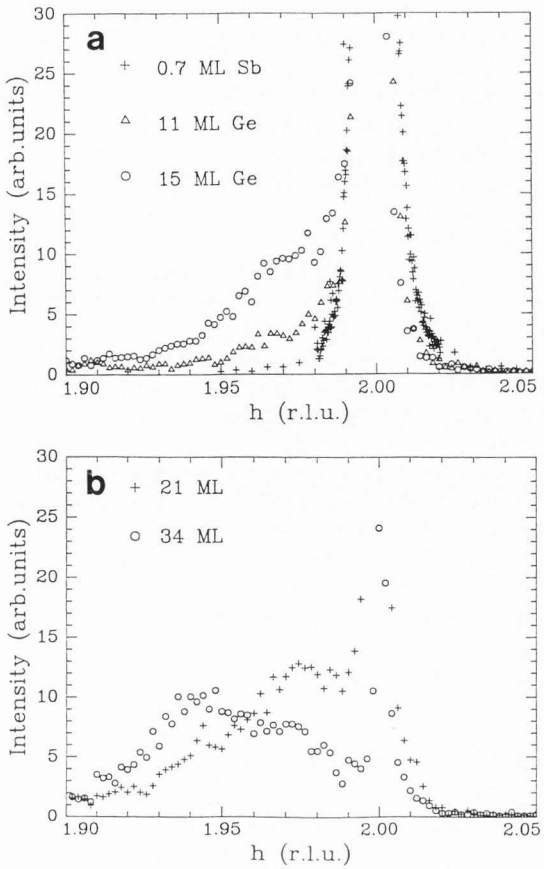

Figure 4. Radial scans through the (2,0) Bragg peak for Ge deposition in the presence of a 0.7 ML Sb surfactant layer, for Ge coverages (**a**) up to 15 ML, and (**b**) above 15 ML.

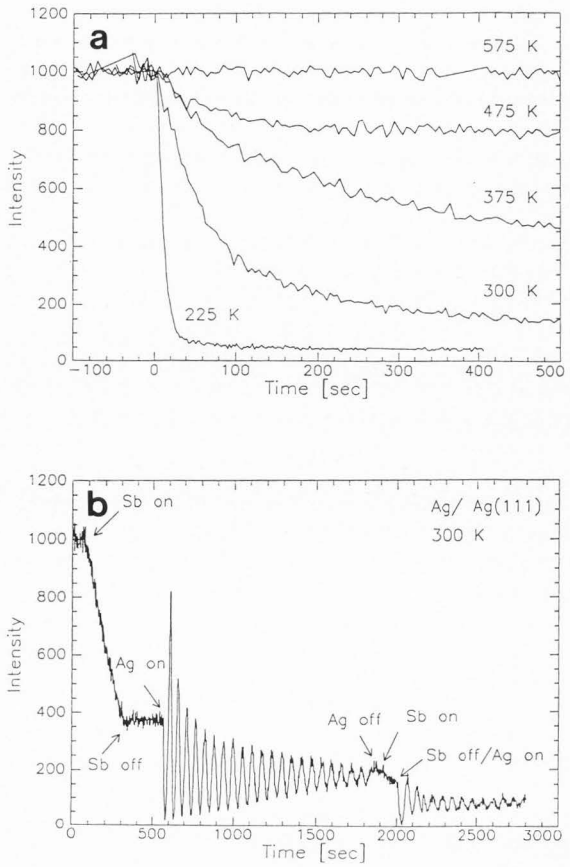

Figure 5. The intensity of the (1/2,1/2,1/2) reflection during Ag deposition onto (**a**) the clean Ag(111) surface, and (**b**) the Ag(111) surface after deposition of 0.2 ML Sb.

such as Sn, do tend to segregate to the surface during growth of GaAs, resulting in surface to bulk concentration ratios of about 1000:1 (Alexandre *et al.*, 1980). In attempting to answer whether Sn could act as a surfactant in such a system, reflection high energy electron diffraction (RHEED) studies of the growth of InAs and $In_{0.36}Ga_{0.64}As$ on GaAs were performed (Petrich *et al.*, 1991). The transition coverage from two dimensional (2D) to 3D growth was unaffected by Sn in both cases. However, although Sn does not act as a surfactant during growth, it was found that sub-monolayer coverages of Sn do reduce significantly the meandering of terraces on vicinal GaAs(001)B surfaces. This effect is also due to Sn atoms migrating to and decorating step edges.

Conclusions

The in-plane strain distribution has been probed with grazing incidence X-ray diffraction during growth of Ge onto a clean Si(001) surface and also in the presence of 0.7 ML Sb which acts as a surfactant. For deposition onto the clean surface, strain relief sets in at 3-4 ML, which coincides with islanding of the surface. TEM images indicate that these islands are dislocation-free for island heights up to 50 nm. A more detailed understanding is obtained from energy minimisation within a molecular dynamics framework. The central role of islanding in strain relief is confirmed by comparing the energies of dislocated films and islanded films with that for a strained structure. The islanded film is energetically favourable at coverages greater than 2.6 ML, whereas, dislocated films have higher energies than fully strained films up to coverages in excess of 10 ML. The atomic coordinates in the relaxed island show elastic deformation around the edges of the islands, inducing bowing of planes in the island and in the underlying substrate. The scattered intensity distribution calculated from the simulation is similar to the observed distribution in the coverage range 3-5 ML.

The strain distribution is very different during deposition of Ge in the presence of a Sb surfactant layer. Strain relief set in at about 10 ML, coinciding with the formation of trenches in the Ge film as observed with STM. Subsequent strain relief indicates further stages in relaxation, some of which may be tied in with TEM images. The mechanism for the prevention of islanding probably involves site exchange between the Sb and Ge

atoms, thus suppression Ge diffusion.

Sb atoms modify the growth rather differently in homoepitaxial growth of Ag on Ag(111). Layer-by-layer growth is observed over a wide range of temperatures, in contrast to growth onto the clean surfaces. In this case, it seems that the Sb affects inter-layer diffusion by decorating the edges of islands. Although still speculative, this is a quite different mechanism from that for Ge/Si. The ordering of 2D layers by adatoms may have a one dimensional equivalent in the ordering of terraces on vicinal GaAs(001)B surfaces in the presence of Sn adatoms. These various systems demonstrate the important role of adatom species on the growth mode, even though the term 'surfactant' appears to be an increasingly inappropriate description of the phenomenon.

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Discussion with Reviewers

D.J. Lockwood: Is the transition to three-dimensional growth or to the relaxed state depend on the growth temperature and the Ge deposition rate?

Authors: We have not investigated in detail the effect of these parameters on the surface morphology. However, the onset of strain relaxation seems to occur at a coverage of 3-4 ML irrespective of the growth temperature between 350°C and 500°C, although the detailed strain distribution after relaxation does differ. A 500-fold increase in the growth rate at 500°C seems to have little effect on the relaxation process.

D.J. Lockwood: In the Si-Ge experiments, how is the Sb coverage measured and is its magnitude critical for maintaining two-dimensional growth?

Authors: Excess Sb was desorbed thermally at 700°C as calibrated by ion scattering. The coverage of 0.7 ML Sb was confirmed by an initial X-ray reflectivity measurement. We did not monitor the effect of varying the Sb coverage and the diffraction results are not sensitive to low-level doping effects.