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# Balancing Surface Energy Terms for Stable Growth of Planar Surfaces

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## BALANCING SURFACE ENERGY TERMS FOR STABLE GROWTH OF PLANAR SURFACES

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### Abstract

We investigate the driving forces that determine the growth mode of heteroepitaxial Ge layers grown from solution on Si substrates with orientations (001), (011) and (111) by transmission electron microscopy (TEM) and atomic force microscopy (AFM). Using liquid phase epitaxy, we can study the influences of strain and surface energy terms independently on effects due to limited surface diffusion. In (001) and (011) orientated layers, {111} faceted islands form (Stranski-Krastanov growth). In contrast, (111) orientated layers grow in a two-dimensional step flow growth mode (Frank-van der Merwe growth).

We model these investigations in terms of energy minimisation considering surface energy reduction by formation of faceted islands and elastic strain energy relaxation by island formation. The strain energy relaxation by island formation is calculated by the finite element method. According to our considerations, two-dimensional growth is obtained by selective increase of the free surface energy of the low indices facet planes to a value higher than that of the substrate surface. Formation of faceted islands thus would increase the total surface energy; as a consequence, island formation is suppressed. By choosing the appropriate solvent and temperature in solution growth, we can provide for thermodynamically stable two-dimensional growth.

**Key Words:** Surface energy microscopy, heteroepitaxial growth, germanium, silicon, transmission electron microscopy, atomic force microscopy, surfacants, solution growth, step flow growth mode, Stranski-Krastanov growth mode, liquid-solid interface energy.

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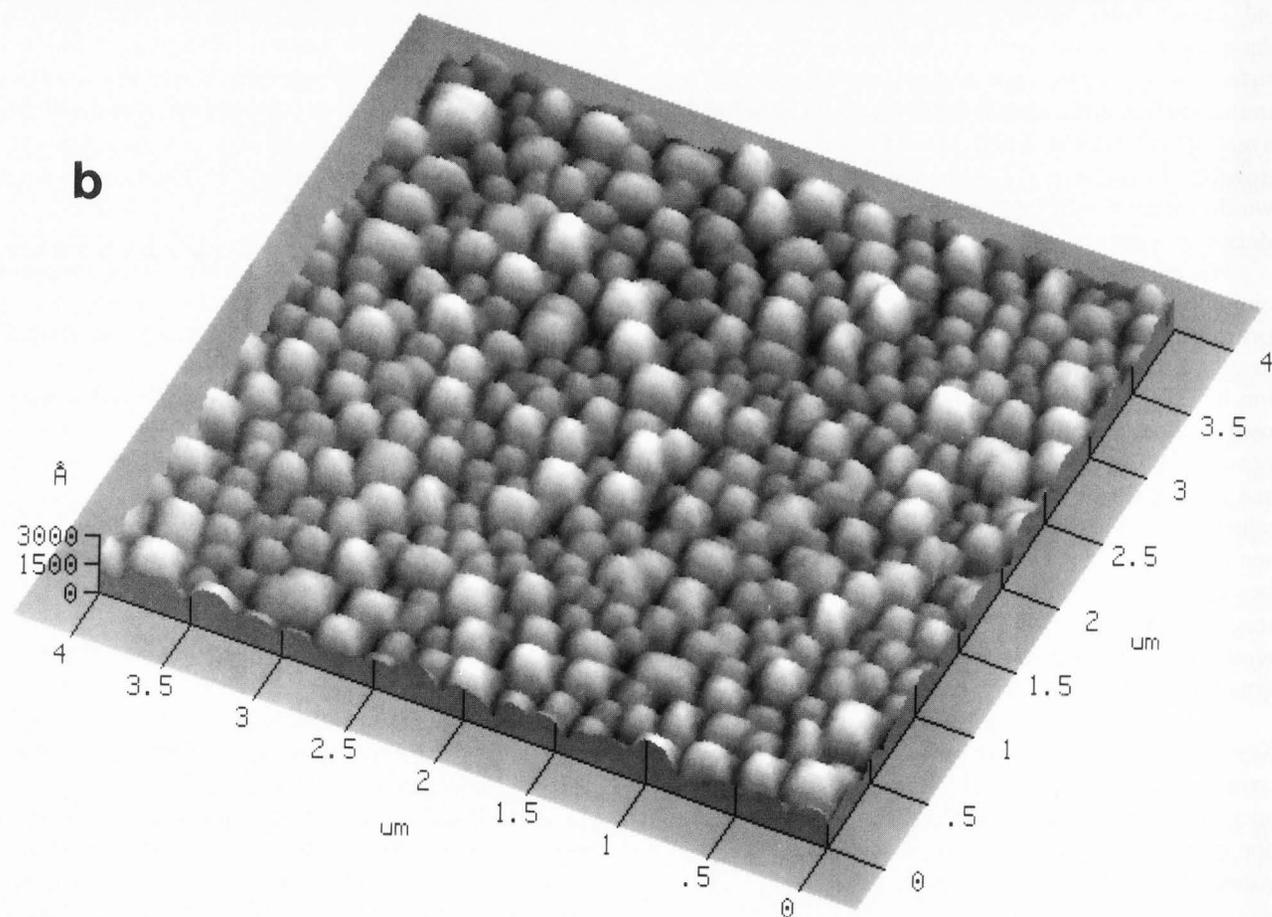
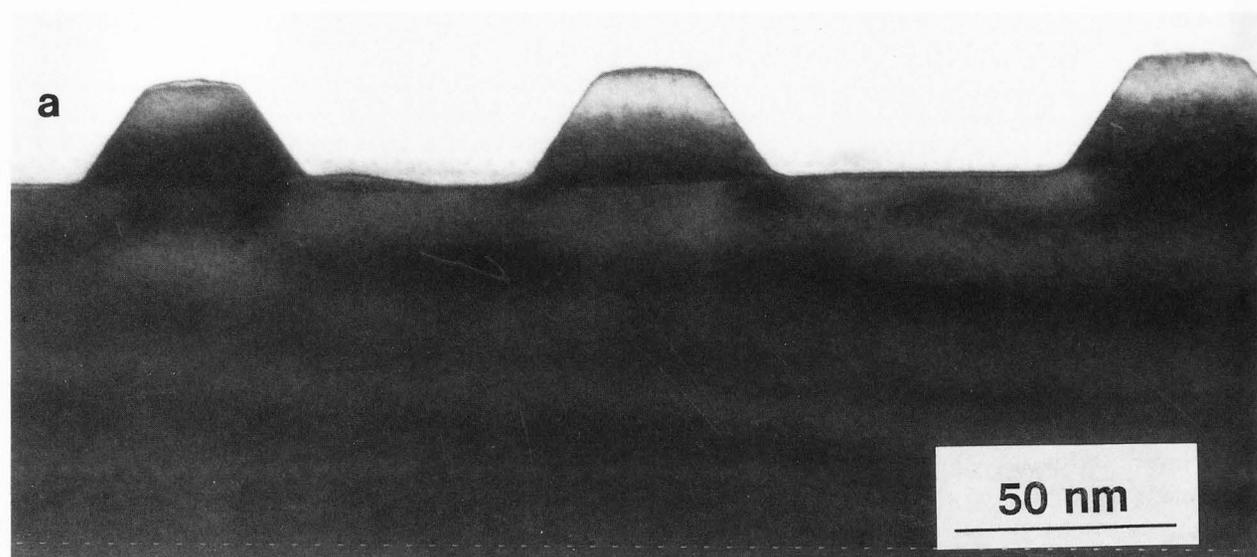
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### Introduction

The growth mode of heteroepitaxial layers on a substrate under conditions of lattice mismatch is energetically determined by the balance between the surface free energy of the epitaxial layer and the substrate, and by the strain energy, which rise within the layer with increasing layer thickness (Bauer, 1958; Bauer and van der Merwe, 1986; Matthews *et al.*, 1987; Marée *et al.*, 1987; Pintus *et al.*, 1987). In general, Ge on Si has been observed to grow in Stranski-Krastanov growth mode (Stranski and Krastanov, 1939) (island form on top of a 3-4 monolayer thick continuous Ge Layer). Island growth can be suppressed by applying surfactants at the growth front (Copel *et al.*, 1989, 1990; LeGoues *et al.*, 1989, 1990). However, whether a change in the surface energy balance due to the surfactant (Copel, 1989; Grandjean *et al.*, 1992) or reduced diffusion of adatoms at the growth surface (Tromp and Reuter, 1992; Orr *et al.*, 1992; Snyder *et al.*, 1992) alters the epitaxial growth mode from three-dimensional to two-dimensional growth is still under discussion.

Using liquid phase epitaxy, we can study the influence of the surface energy terms on the growth mechanism independent of effects due to limited surface diffusion: within the liquid solvent, adatoms have a diffusion constant of about  $2 \times 10^{-8} \text{ m}^2\text{s}^{-1}$  at the growth temperature of 800°C according to self-diffusion in the liquid. The solution atoms completely cover the solid surface during growth, thus influencing the liquid/solid surface energy (Hansson *et al.*, 1993).

In this paper, we investigate the influence of surface energy terms on the growth of Ge layers onto Si substrates that have (001), (011) and (111) orientations. The results of these investigations are analysed in terms of an energetic equilibrium analysis considering elastic strain relaxation due to island formation and surface energy reduction due to formation of low energy facets. From this analysis, we obtain conditions for stable two-dimensional growth by liquid phase epitaxy. The consequences of our analysis in understanding the function of surfactants will be pointed out.



**Figure 1.** Stranski-Krastanov growth of Ge(Si) on Si(001) from Bi solution. (a) Cross-sectional transmission electron micrograph of islands. The pseudomorphic islands exhibit the form of truncated tetragonal pyramids. They are bound by {111} side facets. (b) Distribution of islands on the substrate surface; atomic force microscopy.

#### Experimental

Thirty and 300 nanometer thick  $\text{Ge}_x\text{Si}_{1-x}$  ( $x = 0.85$ ) layers (in the following termed as GeSi) are grown by liquid phase epitaxy from Bi solution at around  $800^\circ\text{C}$  (specified below) on (001), (011) and (111)-orientated Si substrates. Corresponding to the Ge concentration of  $x$

= 0.35 these layers have a lattice mismatch of 3.6%. The Si substrate is initially cleaned by an RCA treatment, followed by a (2.5%) HF-dip and *in-situ* oxide desorption. The solvent Bi is saturated with Ge and Si at the growth temperature of 810°C. To realise growth times as short as one second, the solution is transported on and off the substrate by gravitational force in a computer-controlled graphite-boat. The nominal growth rate is about 2 nm/s. We characterise the epitaxial layers by plane-view and cross-sectional transmission electron microscopy (TEM) using a Philips EM 400 operated at 120 kV. Additional information on the growth topology is obtained by atomic force microscopy (AFM).

## Results

In the following, we present TEM and AFM results of the initial pseudomorphic stages of epitaxial growth with respect to the growth mode of Ge(Si) layers grown on Si substrates with orientations (001), (011) and (111).

After a few seconds of growth on a (001) orientated substrate, the epitaxial layer consists of truncated tetragonal islands grown onto a continuous 4 monolayer thick GeSi layer (Fig. 1a). The islands are bound by {111} side facets and have a base width of 50 nm. Contrast analysis shows the continuous layer and the islands to be free of dislocations, i.e., pseudomorphic. Atomic force micrographs (e.g., in Fig. 1b) show the islands to be statistically distributed on the sample surface. The surface coverage of the thin pseudomorphic layer by islands is 0.5 as obtained from both AFM and TEM plane-view investigations.

The corresponding growth stage on (011) substrates, e.g., in Figure 2a, consists of elongated prisms, which have an aspect ratio (width  $w$  to length  $l$ ) of  $w/l > 10$ . The islands are bound by {111} side facets and (contrary to the expected low energy {111} facets) by {100} front facets. TEM plane-view investigations show the islands to extend in  $\langle 011 \rangle$  directions up to several  $\mu\text{m}$  in length. An atomic force micrograph, see Figure 2b, shows that the islands cover the sample surface completely. TEM contrast analysis reveals the islands to be pseudomorphic up to a height of 40 nm.

In contrast to the results obtained in (001) and (011) orientated layers, growth in (111) orientated layers proceeds in a two-dimensional layer-by-layer growth mode. Figure 3 shows a cross-sectional micrograph of a layer grown from Bi solution at 820°C. The layer in Figure 3a is pseudomorphic and has a thickness of 15 nm. In a TEM plane-view micrograph taken from this layer (cf. Fig. 3b), an arrangement of surface steps can be revealed. According to AFM measurements, these steps have elementary (0.32 nm) and double height. No indication of islanding or faceting has been found throughout

TEM and AFM of these and related samples. From this, we conclude that Ge grows on Si(111) substrates in a Frank-van der Merwe step flow mode.

## Discussion

Our observations on the initial growth stages show that the epitaxial growth mode is essentially influenced by the anisotropic behaviour of surface energies: (i) Frank-van der Merwe growth in a step-flow growth-mode is observed in the case of substrate orientation (111), which is known to exhibit rather low surface energy, (ii) Stranski-Krastanov growth and formation of faceted islands are observed in the case of (001) and (011) substrate orientations, which have higher surface energies. In the following, we will discuss these results in terms of minimisation of the total energy of the epitaxial system. Here, we follow the approach first formulated by Matthews *et al.* (1975) and later modified by several other authors (Marée *et al.*, 1987; Pintus *et al.*, 1987). The total energy  $E_{\text{tot}}$  of the epitaxial system is:

$$E_{\text{tot}} = E_{\text{str}} + E_{\text{surf}} \quad (1)$$

Here  $E_{\text{surf}}$  is the surface energy of the epitaxial system, given as  $E_{\text{surf}} = \gamma (A_s + A_f)$  where  $\gamma$  is the surface energy of the epitaxial layer,  $A_s$  is the surface area, and  $A_f$  is the additional free surface exposed when islands form.  $E_{\text{str}}$  is given by  $E_{\text{str}} = E_C + E_I$  in Matthews *et al.* (1975), where  $E_C$  is the elastic energy stored in the continuous layer and  $E_I$  is the elastic energy of the islands and of the misfit dislocations beneath the island. Below, we modify the approach of Matthews *et al.* for the case of growth of pseudomorphic faceted islands, considering:

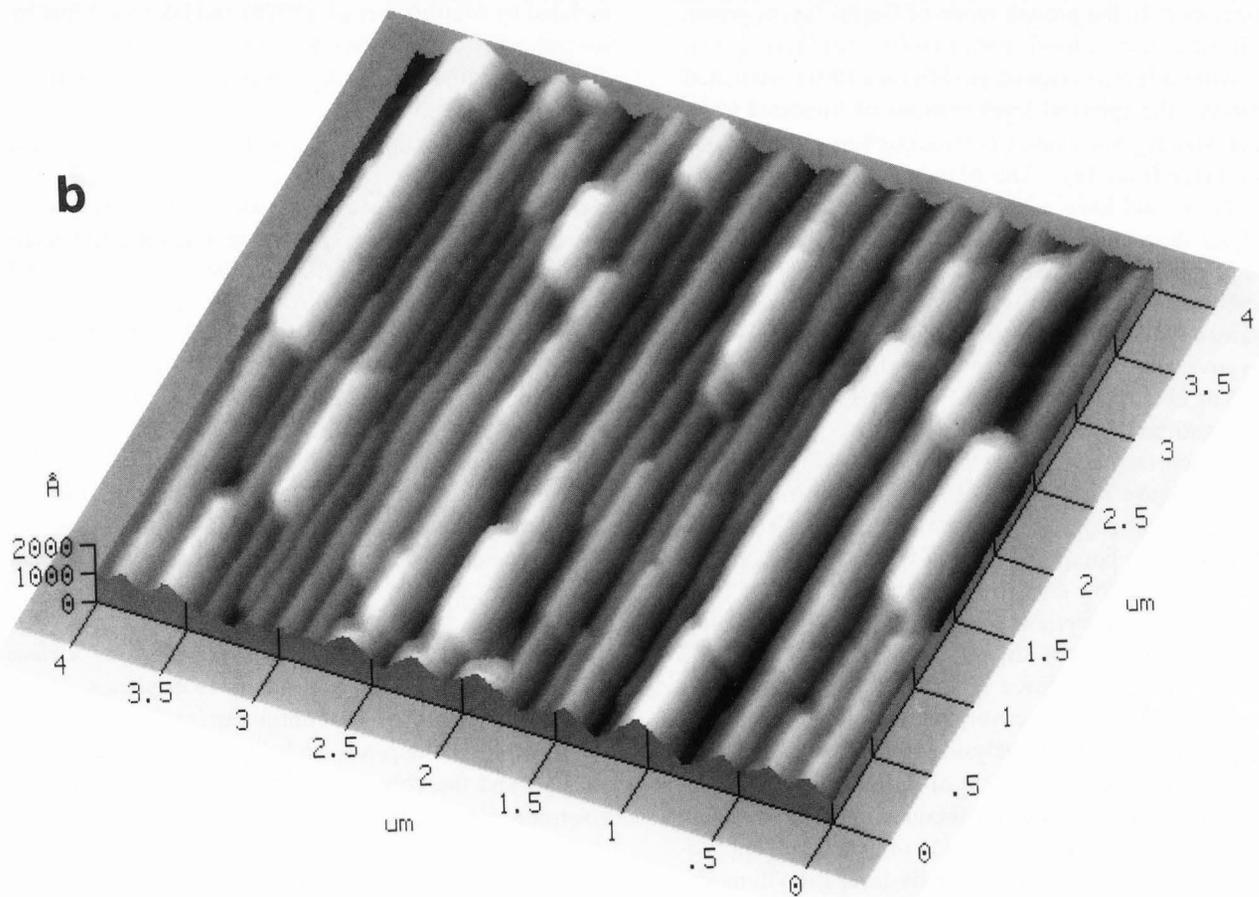
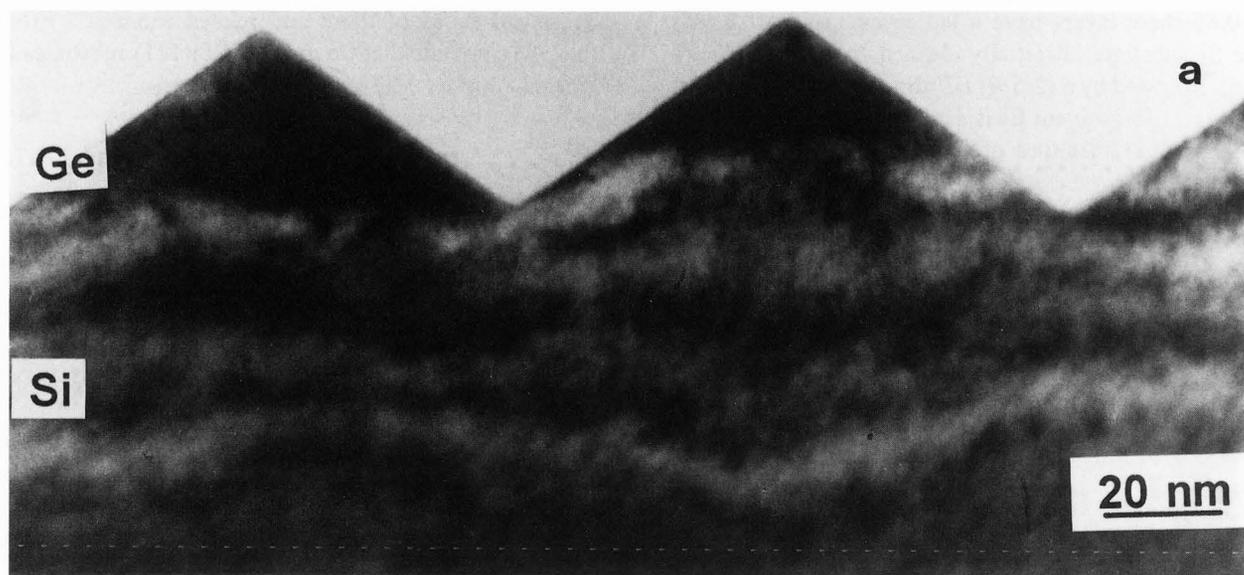
(i) that island formation reduces the strain elastically instead of by formation of dislocations at the island's interface; and

(ii) that island formation reduces the total surface energy by formation of low energy facet planes.

The strain energy of a continuous, pseudomorphically strained layer depends exclusively on the lattice mismatch  $f$  and the volume  $V$  of the layer and is given by (isotropic elasticity):

$$E_c = 2G \{(1 + \nu) / (1 - \nu)\} f^2 V \quad (2)$$

where  $G$  is the shear modulus of the epitaxial layer and  $\nu$  is Poisson's ratio. In this approach of plane strain, the strain energy density within the layer is constant and independent of areas. In contrast to a continuous layer, in a three-dimensionally confined island, part of the misfit strain is relaxed elastically. One approximation to account for this strain relaxation is to adopt the strain

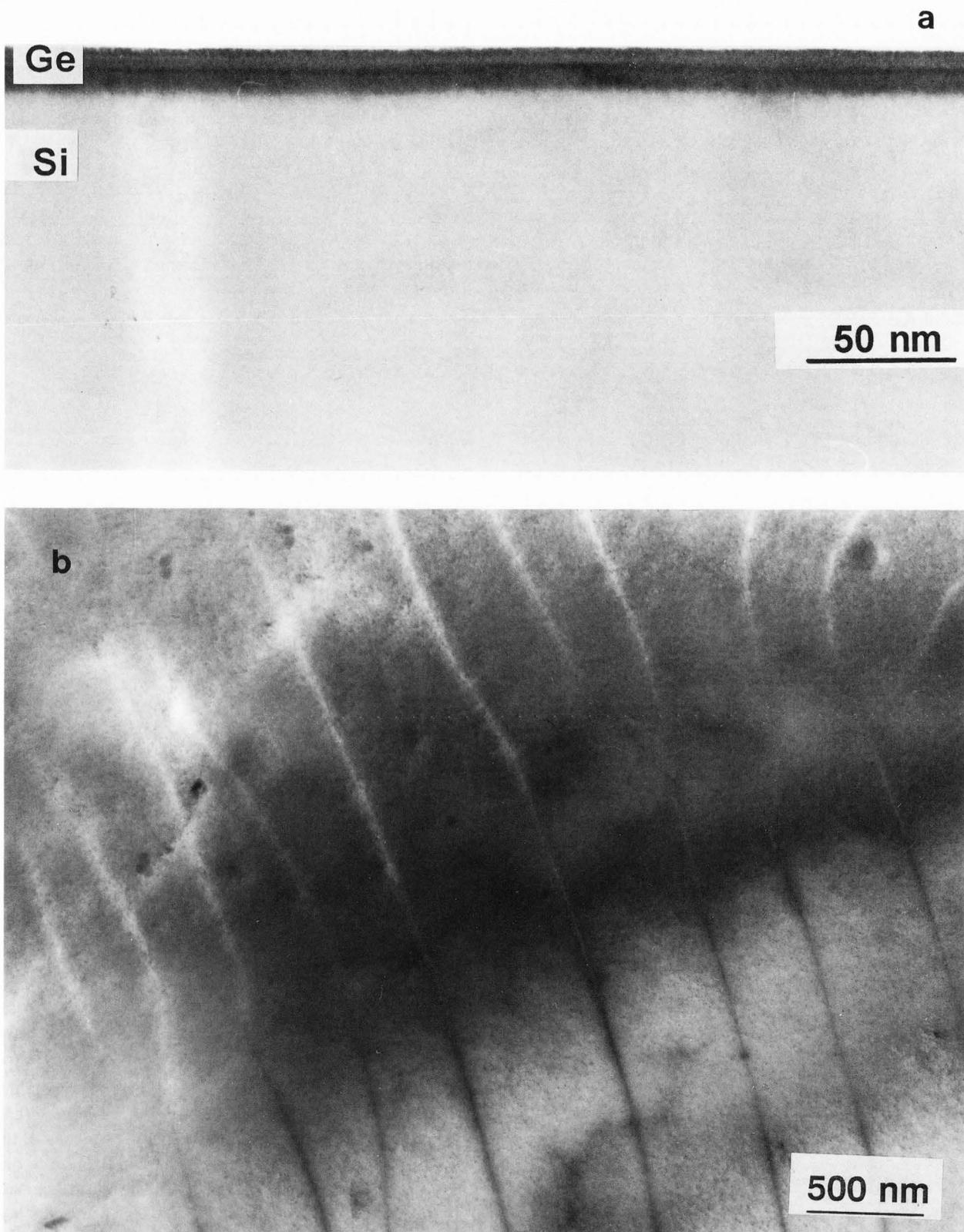


**Figure 2.** Stranski-Krastanov growth of Ge(Si) on Si(011) from Bi-solution. (a) Distribution of islands on the substrate. The islands are elongated along 2-3  $\mu\text{m}$ . (b) Cross-sectional transmission electron micrograph of the same sample in [1-10] multibeam conditions. The side faces are formed by  $\{111\}$  side faces. The substrate is completely covered by islands.

energy of a two-dimensional layer, eq. 3, by substituting the misfit  $f$  by a reduced effective misfit  $f_{\text{eff}}$  within the island:

$$f_{\text{eff}} = f_{\Phi} \quad (3)$$

Theoretical approaches to determine  $f_{\text{eff}}$  are based on



**Figure 3.** Frank-van der Merwe growth in Ge/Si(111). (a) Cross-sectional transmission electron micrograph of a continuous 15 nm thick layer. The layer is pseudomorphic. (b) Transmission electron plane-view bright-field micrograph. The dark lines are monatomic and biatomic steps indicating step flow growth mode.

analytical calculations in the framework of linear elasticity (Cabrera, 1964; Jesser and Kuhlmann-Wilsdorf, 1967; Luryi and Suhir, 1986; Hull and Fischer-Colbrie, 1987), semi-analytical approaches using atomic potentials (Ashu and Matthai, 1991; Ratsch and Zangwill, 1993), and finite element calculations (Christiansen *et al.*, 1994, 1995). Experimentally,  $f_{\text{eff}}$  has been determined using convergent beam electron diffraction (CBED) (Christiansen *et al.*, 1994).

Cabrera (1964) and Jesser and Kuhlmann-Wilsdorf (1967) determined  $\Phi$  analytically for a spherically shaped island. Luryi and Suhir (1987) determined  $\Phi$  for square patterned islands dependant on their aspect ratio width  $w$  to height, showing an exponential decay of the lattice strain (and thus of the strain energy density) with decreasing aspect ratio in three-dimensional islands (Luryi and Suhir, 1986; Hull and Fischer-Colbrie, 1987). Three-dimensional islands with less than 5000 atoms, were treated by calculations based on the Frenkel-Kontorova model (Ratsch and Zangwill, 1993) and on atomic potentials (Ashu and Matthai, 1991) such as Stillinger-Weber-, Keating-, or Tersoff-potential.

For larger islands, the strain energy distribution, and thus the total strain energy stored in the island, can be obtained from calculations by finite element method (FEM) (Christiansen *et al.*, 1994, 1995). From FEM calculations, we obtain  $\Phi$  by calculating the strain energy of the islands with different aspect ratios  $l/h$  and dividing it by the strain energy of a homogeneously strained layer with the respective volume.  $\Phi$  is then given by the correction function (Christiansen *et al.*, 1995):

$$\Phi = (1 - e^{-1/12h})^{1/2} \quad (4)$$

In the approach of Matthews *et al.* (1975), the surface energy density is isotropic and thus, increases proportionally with increasing surface area per unit substrate area. As our results above show, the anisotropy of the surface energy essentially influences the growth mode by formation of faceted islands. The total surface energy of a faceted epitaxial layer is given by the following expression:

$$E_{\text{surf}} = \sum_{\text{hkl}} A_{\text{hkl}} \gamma_{\text{hkl}} \quad (5)$$

where  $\gamma_{\text{hkl}}$  is the free surface energy of a  $\{\text{hkl}\}$  facet plane and  $A_{\text{hkl}}$  is the corresponding surface area consisting of  $\{\text{hkl}\}$  facets. To our knowledge, experimentally determined surface energy values for the different surfaces of semiconductors are not available. Therefore, we use, as an approximation, surface energies that are calculated by the "broken bond model" (Mutaftschiev, 1988). According to this model, the surface energy  $\gamma_{\text{hkl}}$  of a  $\{\text{hkl}\}$  facet plane is the energy, necessary to break

**Table 1.** Free surface energies of different facet planes for Ge according to the broken bond model (Mutaftschiev, 1988). The energy per broken bond  $\epsilon$  is taken from (Mercer and Chou, 1993);  $a_{\text{hkl}}$  is the surface unit area,  $N$  is the number of broken bonds per surface unit and  $\gamma$  is the resulting free surface energy.

	(001)	(011)	(111)
$\epsilon$ /[eV]	0.79	0.98	0.98
$a_{\text{hkl}}$	$a^2/2$	$a\sqrt{2}$	$(3/4) \cdot a^2$
$N$	2	2	1
$\gamma$ /[Jm <sup>-2</sup> ]	1.57	1.42	1.12

the  $N$  bonds of a unit cell surface area  $a_{\text{hkl}}$ , corresponding to the respective  $\{\text{hkl}\}$  facet plane. From this, we calculate the surface free energy  $\gamma_{\text{hkl}}$ :

$$\gamma_{\text{hkl}} = \epsilon (N/a_{\text{hkl}}) \quad (6)$$

The energy  $\epsilon$  of one broken bond in the framework of this model is given by the cohesive energy  $\Psi_c$  of an atom in bulk material divided by the coordination number  $n$  of this atom. Theoretical *ab initio* calculations, as well as tight binding calculations of the broken bond energies  $\epsilon$  on (001) and (111) surfaces of Ge and Si (Mercer and Chou, 1993), show the broken bond model to be a good approximation in calculating surface energies. In Table 1, the surface energies of low indices facets of Ge obtained from the broken bond model are summarised.

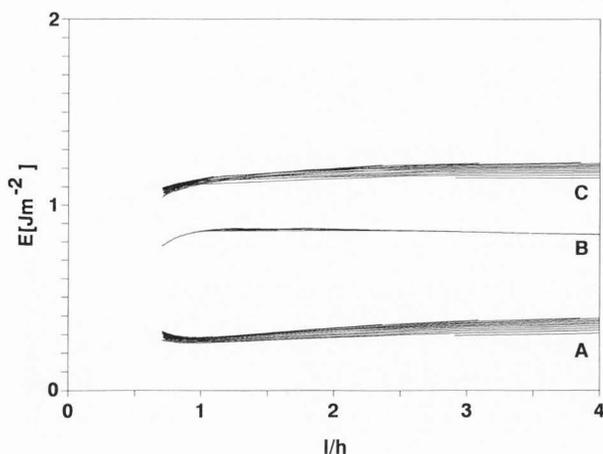
In the following, we present calculations, based on eqs. 2-6, of the total energy of the epitaxial systems for substrate orientations (001) and (111). The total energy of a pseudomorphic epitaxial island on a thin continuous pseudomorphic layer is given by:

$$E_{\text{tot}} = E_{\text{surf}} + E_I + E_C \quad (7)$$

To apply this equation we have to appropriately define the entries: (a) the type of facet planes used to calculate the total surface energy, (b) the dependence of the volume and surface area of the islands on substrate orientation, (c) the coverage  $C$  of the substrate surface with islands, and (d) the thickness of the pseudomorphic layer, on which the islands form.

These assumptions are the following:

- (a) Based on our observations we only consider the low indices facet planes  $\{111\}$ ,  $\{011\}$  and  $\{001\}$ .
- (b) We assume polyhedral pyramids bound by these



**Figure 4.** Energy terms for growth of Ge on Si(001) as dependent on the aspect ratio  $l/h$ . The pyramidal islands are bound by  $\{111\}$  facets, (island coverage  $c = 0.5$ ). A: total surface energy; B: strain energy; C: sum of A and B. Strain energy and surface energy as well as the total energy have a minimum for tetragonal islands.

facet planes. The base planes of the polyhedra are triangular in the case of the (111) orientation and quadratic in the case of (001) orientation according to the substrate symmetry. The volume and the surface areas of these islands follow from simple trigonometry.

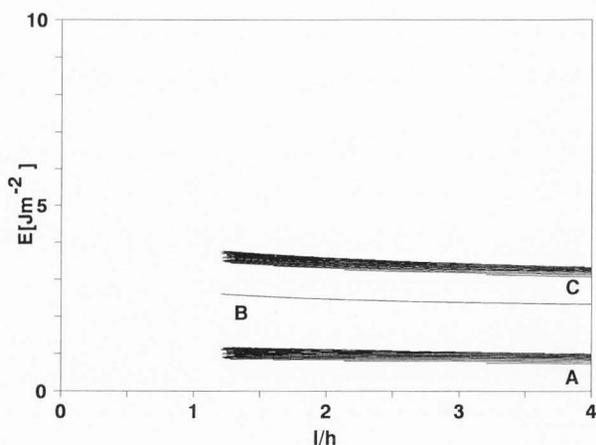
(c) The coverage  $C$  of the substrate with islands is given by the surface area covered with islands divided by the total surface area of the substrate. We compare total energies for coverages  $C = 1$  (complete coverage) and  $C = 0.25$ .

(d) The thickness of the pseudomorphic continuous layer is assumed to amount four the monolayers according to our observations (Hansson *et al.*, 1992a).

To make surface, strain and total energies comparable for all growth morphologies, we standardize these energies in the substrate surface unit area that corresponds to the coverage with islands. Therefore, we divide  $E_{\text{tot}}$  by the unit area, which has  $1/C$  times the edge length of the island.

#### (001) substrate

Figure 4 shows the energy contributions for (001) substrate orientation with a coverage of  $C = 0.5$  (as observed by TEM (Albrecht *et al.*, 1993)). The energy terms for a certain volume are plotted as dependent on the aspect ratio  $l/h$  of the island. The surface energy (curve set A) has a minimum at  $l/h = 1/\sqrt{2}$ , i.e., if the island is a tetragonal pyramid. The formation of  $\{111\}$  facets reduces the total surface energy. The strain energy in curve B has a minimum in the case of a tetrahedral pyramid as well. The strain energy increases with



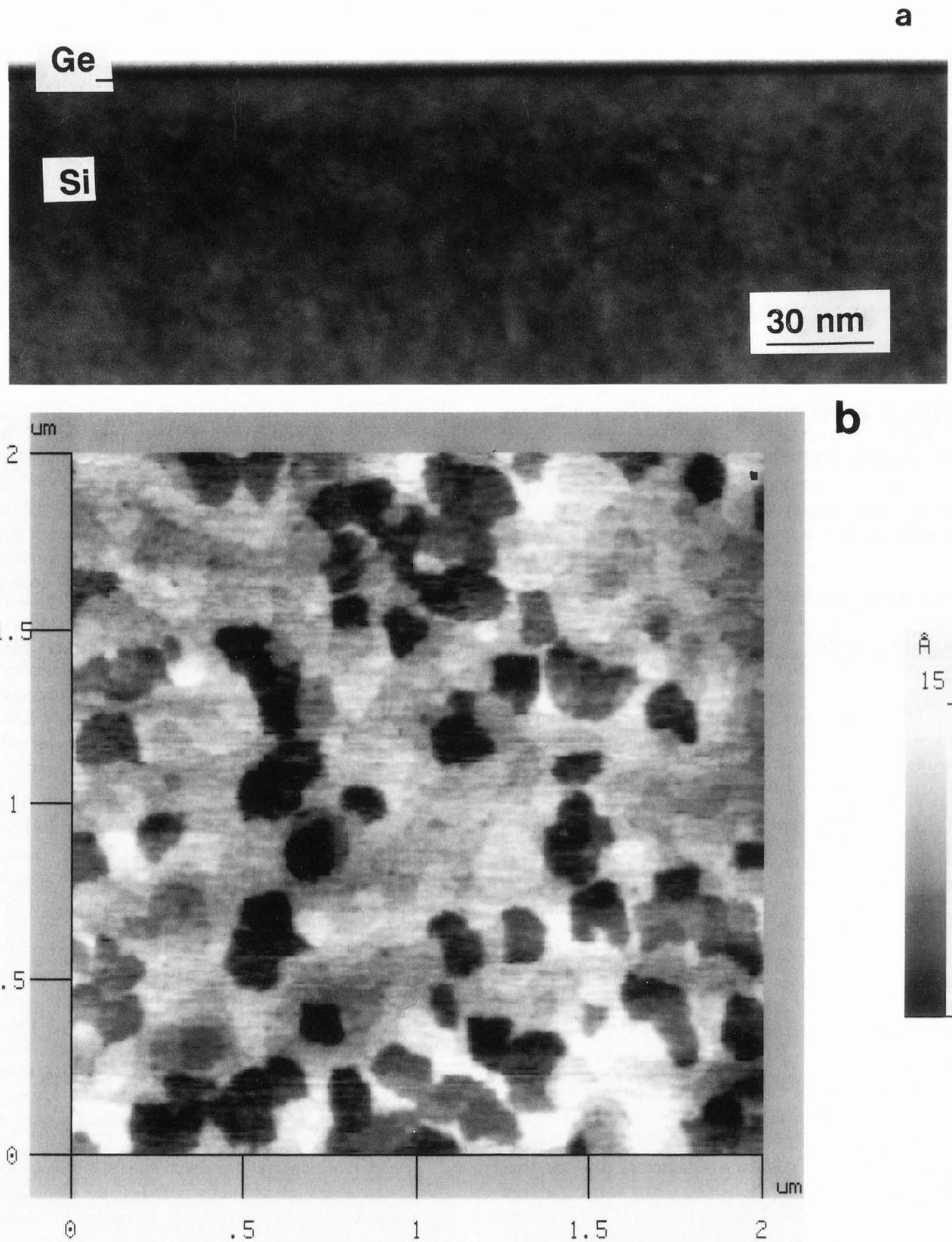
**Figure 5.** Energy terms for growth of Ge on Si(111) as dependent on the aspect ratio of tetrahedral islands. A: total surface energy; B: strain energy; C: Total energy. The strain energy reduction is a maximum for the formation of tetrahedral islands corresponding to the (001)-orientation. In contrast, the surface energy is a minimum for  $l/h = \infty$ . The total energy is dominated by the surface energy. Thus, two-dimensional growth takes place.

increasing volume. As a consequence, the total energy of the epitaxial system has a minimum for tetragonal islands. Calculations for different coverages show the total energy to be a minimum for  $C = 0.5$  as observed.

#### (111) substrate

The situation is in fact completely different from that of (001) orientation. The (111) surface has the lowest surface free energy and formation of any islands leads to an increase in surface energy. This is shown in curve A in Figure 5. The lowest surface energy is obtained for an aspect ratio  $l/h$  versus reaching  $\infty$ , i.e., for a plane surface. In contrast, the strain energy is a minimum for islands in the form of tetrahedral pyramids (curve B in Fig. 5). However, the total energy is dominated by the surface energy term, and thus has a minimum for a continuous layer with a plane surface (curve C in Fig. 5). As experimental investigations show (Hansson *et al.*, 1992b), the misfit beyond a thickness of 6 nm is relaxed by formation of dislocations instead of islands.

From these energetic considerations, we conclude that elastic stress relaxation by islands is the main driving force for island formation; the formation of islands in any case reduces the total strain energy of the epitaxial layer. Formation of islands with low energy facets (facets with surface energies lower than that facet which corresponds to the substrate surface) may further reduce



**Figure 6.** Two-dimensional growth of Ge on Si(001) from In solution. (a) Cross-sectional transmission electron micrograph. The layer is 10 nm thick and pseudomorphic. (b) Atomic force micrograph of the same sample. The surface contains islands of monatomic height.

the total energy, and thus stabilise island formation and especially control the island geometry. In other words, an epitaxial layer grows two-dimensionally in a layer-by-layer mode when faceting by island formation would increase the total surface energy by more than is gained by reduction in elastic strain energy. Thus, the only possibility to obtain stable two-dimensional growth is to selectively increase the energy of those facet planes that have lower free surface energies than the substrate surface. This contrasts with models concerning surfactant mediated growth related to molecular beam epitaxy. These models propose that a general lowering of the surface energy of the strained overlayer was the main effect in suppressing island formation (Copel *et al.*, 1989; Grandjean *et al.*, 1992). According to our model, a general lowering of the surface energy would enhance island formation instead of inhibiting island growth. However, our model is in good agreement with experimental results obtained in the InGaAs/GaAs (001) system by Snyder *et al.* (1993) which show that increasing the surface tension by cation stabilized growth conditions inhibits island formation. An investigation of surfactant mediated molecular beam epitaxy (MBE) growth in the GeSi/Si system by Eaglesham (1993) showed that the morphology of epitaxial films can be attributed to the changes of surface energy anisotropy by different surfactants.

In liquid phase epitaxy, we can provide for two-dimensional growth conditions by choosing appropriate solvents. An excellent guideline is the wetting/non-wetting condition of the liquid solvent for a certain facet plane. In the case of wetting, the liquid/solid interface energy  $\gamma_{ls}$  is the difference between the free surface energy of the solvent  $\gamma_l$  and the solid  $\gamma_s$  (Mutaftschiev, 1988). In the case of non-wetting, the liquid/solid interface energy is the sum of the free surface energy of the solvent and of the solid.

Thus two-dimensional growth in (001) orientated layers is expected if the solvent wets the (001) plane but not the (111) lattice planes. These conditions are fulfilled for growth from In solution at a temperature of  $T = 500^\circ\text{C}$  (Hansson *et al.*, 1993). Figure 6a shows a cross-sectional transmission electron micrograph of a 6 nm thick layer grown under these conditions. The corresponding AFM micrograph in Figure 6b indicates layer-by-layer growth. It proceeds by nucleation and lateral growth of two-dimensional islands which contrasts to the pure step flow growth mode as in the (111) oriented layers.

### Conclusion

In conclusion, we have demonstrated that two-dimensional growth can be obtained under conditions of

thermodynamical equilibrium and without any restrictions of surface diffusion when the energy of those facet planes that have lower free surface energies than the substrate surface is increased by more than is gained by reduction in elastic strain energy. In liquid phase epitaxy, we can provide for these conditions by choosing an appropriate solvent.

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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.**