Predictability of plastic relaxation in metamorphic epitaxy

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Theoretical models and experimental data on plastic relaxation are reviewed. It is concluded that neither equilibrium nor kinetic effects are responsible for the relaxation observed under conditions of high growth quality in III–V epitaxy; instead dislocation multiplication mechanisms and the space they require to operate constitute the determining factor. Residual strain is then given by $\varepsilon(h) = (0{\cdot}8 \pm 0{\cdot}1\ \text{nm})/h$ for a layer of thickness h. Deviations from this rule are indicative of poor crystallinity. Silicon–germanium and II–VI materials are briefly discussed. MST/3328

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Introduction

Until recently, lattice matched growth was mainstream in semiconductor epitaxy, and strained layer growth was a scientific curiosity. It was commonly thought that even if strain could be incorporated into layers during growth, it would not survive device processing, or would lead to rapid degradation of the device in service. This has been strongly refuted by the success of the strained layer laser and it is probable that in the future strain will be incorporated into semiconductor heterostructures routinely. If strained layers do not normally relax and degrade during processing and device operation, the conditions under which they do relax require investigation, in addition to whether relaxation is predictable (like plastic deformation generally) or whether it is intrinsically unpredictable (like cracking).

In the present paper, the theoretical background is first considered and then the experimental evidence for predictability. Plastic relaxation is shown to be predictable provided growth is well controlled and of the highest quality.

Theoretical background

It is apparent that the large strains, of the order of 10^{-2}, that can be grown into semiconductor layers are only possible because the layers are very thin. Bulk semiconductors at growth temperatures (in the region of 600°C) have elastic yield strengths in the region of 10 MN m^{-2}, corresponding to strains of only 10^{-5}. The thickness required has been studied extensively for approximately 40 years (*see* e.g. Refs. 1–3, and *see* Ref. 4 for a review of theory and experiment). Both equilibrium and kinetic effects have been considered, are summarised briefly below, and are compared with the empirical behaviour, particularly of III–V strained layers. It is concluded that neither the equilibrium theory nor kinetic effects account for the observed behaviour.

EQUILIBRIUM EFFECTS

The physics of the increased elastic yield strength of a sufficiently small object is well established. If the elastic strain energy integrated over the volume is insufficient to produce a dislocation, no plastic relaxation occurs. There are some difficulties in calculating exactly the strain and dislocation energies, but reasonable approximations lead to the Matthews equation,[2] which is sufficiently accurate at low strains (below about 10^{-3}) and predicts a maximum strain of

$$\varepsilon_{max} \approx \frac{0{\cdot}1\ \text{nm}}{h} \quad (1)$$

for a layer of thickness h. At higher strain values the approximations in the derivation of the Matthews equation become more severe, but numerical calculations[5] show that equation (1) can continue to be used for strains above 10^{-2} (within the limits permitted by growth).

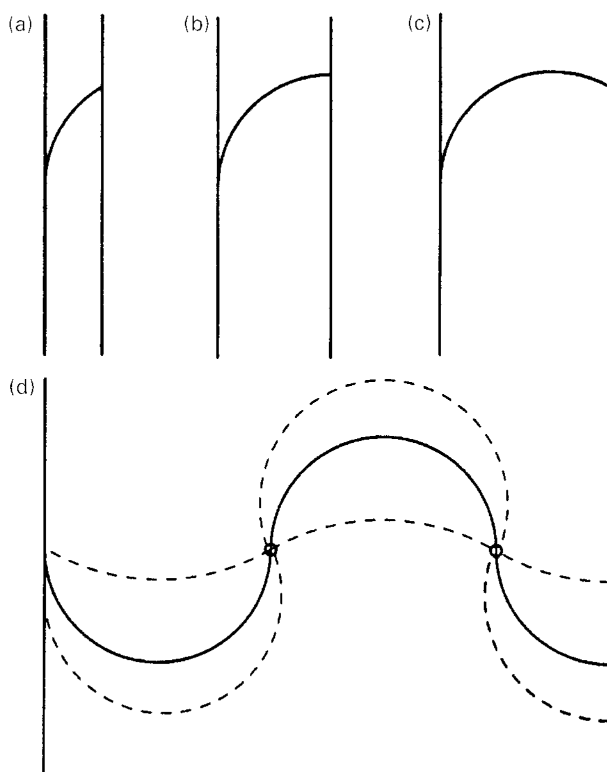
In the derivation it is assumed that dislocations already exist (for example, threading dislocations originating from the substrate). In practice there are usually far too few existing dislocations to give significant relaxation, and dislocation generation or multiplication mechanisms must be involved. To analyse this problem, it is helpful to recast the Matthews derivation in terms of dislocation curvature. Cottrell[6] gave the minimum radius of curvature R_{min} of a dislocation in a stress σ as

$$R_{min} = \frac{T}{b\sigma} \quad (2)$$

where T is the line tension of the dislocation and b is the relevant component of the Burgers vector. The Matthews model identifies the maximum thickness for a given strain (the critical thickness h_c) with R_{min}, so that a misfit dislocation makes a quarter circle from the interface to the free surface (Fig. 1*b*). Dislocation multiplication mechanisms, however, require layer thicknesses of several times R_{min}. A spiral source, for example, requires $4R_{min}$ (Ref. 7), whereas a Frank–Read source requires $5R_{min}$ (Fig. 1*d*). It was also noted by Freund[8] that the creation of the two orthogonal arrays of misfit dislocations required for relaxation in both directions requires about twice the critical thickness. Thus significant relaxation of a strained layer is not expected until a thickness which is a simple multiple (of the order of ×5 to ×10) of the critical thickness given by equation (1).

KINETIC EFFECTS

Figure 1 shows dislocations which move by glide. Glide is a process with an activation energy in the region of 1–2 eV (Ref. 9), and it becomes significant at the elastic–plastic transition, at approximately 300°C in the III–V compounds. There is then a wide temperature interval before dislocation climb occurs at temperatures sufficiently high for significant diffusion, for which the activation energy is about 3·5–4 eV (given as 3·7 eV by Bradley *et al.*[10]) in the III–V compounds.



a layer is below equilibrium critical thickness h_c with $h < R_{min}$ from equation (2); *b* layer is at h_c; *c* layer is above h_c; *d* thickness required for Frank-Read source to operate between substrate and free surface – solid curve shows dislocation at R_{min}, dotted curves show larger radii occurring before and after, and minimum layer thickness from substrate to free surface can be seen to be $5R_{min}$, which by comparison with *b* is $5h_c$

1 Misfit dislocation which terminates by curving to surface with radius given by equation (2): substrate interface is to left, free surface to right

requiring 700–800°C. Most growth is carried out in the range 500–650°C, so it is apparent that glide occurs freely whereas climb is insignificant. Consequently, no significant change of relaxation with growth temperature or growth time is expected, i.e. there are no kinetic constraints.

This differs from Si based systems, for which growth is carried out near or below the elastic–plastic transition and kinetic constraints are observed.[4,11] Fitzgerald,[4] however, notes that Si–Ge growth carried out at temperatures much higher than normal results in relaxation behaviour similar to that of the III–V compounds.

Empirical relaxation behaviour

A wide diversity of results may be found in the literature, possibly implying that many parameters are involved, some perhaps uncontrolled, or even implying that relaxation is essentially unpredictable. Most data are available for the InGaAs ternary alloy, which can be grown mismatched on GaAs or on InP. The present authors have therefore concentrated on this system, and other material systems are considered briefly below.

Considerable diversity also originates from the use of different techniques to measure relaxation.[4] The present discussion is restricted to results obtained from double crystal X-ray diffraction (DCXRD), in which measurement of the tetragonal distortion and of the size of the unit cell gives both strain and composition without any assumptions. Similarly, it is preferable to use only thickness data from cross-sectional transmission electron microscopy (TEM), rather than nominal values from growth.

EXPERIMENTAL RELAXATION OF InGaAs

Whaley and Cohen[12] measured the lattice parameter of InGaAs layers during growth, varying the composition and the substrate temperature, and fitted the results to the Dodson and Tsao[11] model. Good agreement was found, which suggests the importance of kinetic constraints, except for one very significant discrepancy. When growth was interrupted, the lattice parameter should have continued to evolve with time, but did not. Further evidence of the absence of kinetic constraints is provided by the annealing experiments of Drigo *et al.*[13] and of Lourenço and Homewood,[14] which show that significant further relaxation of partially relaxed layers can only be achieved by very severe annealing at temperatures and for times appropriate for interdiffusion to occur.

Drigo *et al.*[13] present their own data for the strain in relaxing layers as a function of the thickness, together with some data from previous authors. A square root dependence was found with $\varepsilon \propto h^{-1/2}$ for strains from $1{\cdot}5 \times 10^{-3}$ to $1{\cdot}5 \times 10^{-2}$ and for compositions of $In_xGa_{1-x}As$ with x from 0·035 to 0·25. This relationship is only approximate, with a scatter around the curve within a factor of two, but a similar trend can be detected in other data. For example, much of the present authors' data (*see* Fig. 3 in Ref. 15) can be shown to be consistent with this trend.

Dunstan *et al.*[16] have reported a systematic relaxation behaviour with three regimes. In regime I, above the equilibrium critical thickness of equation (1), a few misfit dislocations may form but no significant relaxation occurs. In regime II, predictable relaxation occurs, obeying[16]

$$\varepsilon(h) = \frac{0{\cdot}83\ \text{nm}}{h} \qquad (3)$$

Finally, at small strains below about $0{\cdot}1\varepsilon_0$, residual strain remains which is essentially independent of thickness (regime III). Work hardening occurs and the layer becomes able to support its strain without the benefit of small thickness effects.[16]

This behaviour was deduced from a set of results for a few $In_{0{\cdot}2}Ga_{0{\cdot}8}As$ layers and an $In_{0{\cdot}05}Ga_{0{\cdot}95}As$ layer, with strains from $5{\cdot}6 \times 10^{-4}$ to $8{\cdot}3 \times 10^{-3}$, grown under normal molecular beam epitaxy (MBE) conditions. Subsequently, similar behaviour was observed in $In_{0{\cdot}2}Ga_{0{\cdot}8}As$ grown at the much lower temperature of 400°C.[17] Up to about 50% relaxation these samples followed equation (3), and two samples at greater thickness showed some evidence of kinetic constraint (at 200 nm thickness the strain was $5{\cdot}3 \times 10^{-3}$ whereas $4{\cdot}2 \times 10^{-3}$ is expected, and at 400 nm it was $3{\cdot}2 \times 10^{-3}$ whereas $2{\cdot}1 \times 10^{-3}$ is expected). All these samples were grown in the same MBE machine by the same growers. More recently, some $In_{0{\cdot}1}Ga_{0{\cdot}9}As$ samples grown by MBE in a different laboratory were presented and they were also shown to obey equation (3) to good accuracy.[18]

It was shown theoretically that multilayer samples could be expected to obey a generalisation of equation (3) (*see* 'Multilayer structures' section below). Using two and three layer structures from several laboratories, it was found that the average strain of samples and their total thickness were indeed related by equation (3).[18] In the present context, the significance of this observation is that the multilayer structures were grown by atomic layer MBE (ALMBE) at 350°C, by MBE at 500°C, and by metalorganic chemical vapour deposition (MOCVD) at 650°C.[18] This is direct evidence that equation (3) does not represent a kinetic constraint on relaxation towards the equilibrium value of equation (1).

In summary, much of the experimental data appear to demonstrate a square root variation (equation (2)) for large sets of data, and to lie accurately on the curve of equation (3) when small, carefully selected sets of data are analysed. For the former, the relaxation of an individual

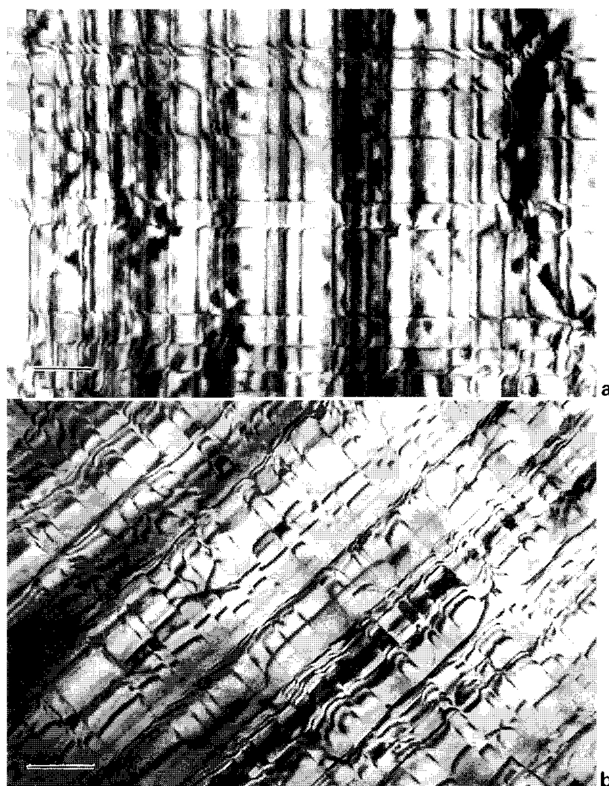
layer is not entirely predictable, whereas for the latter it is. These two trends will be reconciled below.

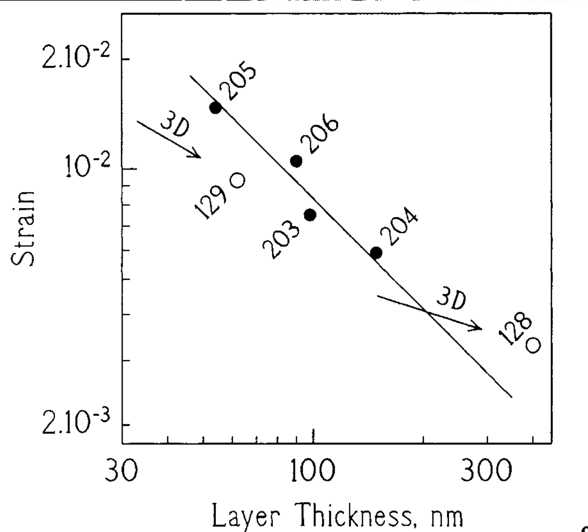
DISCUSSION

Initially, it should be noted that the samples of Ref. 16, for which equation (3) was first demonstrated, came from a well characterised and well controlled growth programme.[19] They were interspersed at intervals among a large number of quantum well samples grown for demanding device programmes, in which any deviation from high quality growth was rapidly detected. Routine characterisation also included Nomarski microscopy and DCXRD and a large number of samples were analysed by plan view and cross-sectional TEM. Samples grown when the MBE reactor was operating below optimum performance were not used for further study and therefore did not feature in Refs. 16–18. In contrast, larger sets of data, which included, for example, the entire output of a MBE machine, inevitably contain samples grown under less than perfect conditions. These data have therefore been analysed under the assumption that equation (3) will hold for samples of the highest growth quality only. The following (non-exhaustive) list gives reasons for which a sample may deviate from equation (3) and is represented by an open circle in Figs. 2, 3, and 4:

(i) it is grown to below relaxation critical thickness h_{cr} (in regime I)
(ii) it is grown to above work hardening thickness, $\sim 10h_{cr}$ (in regime III)
(iii) it exhibits defective surface morphology (i.e. other than smooth, striated, or cross-hatched)
(iv) cross-sectional TEM shows an unusually high density of threading dislocations
(v) plan view TEM shows a disordered array of misfit dislocations
(vi) DCXRD rocking curves show unusually broad peaks
(vii) there is a severe discrepancy between nominal and measured thicknesses or compositions (indicative of a suspect growth run)
(viii) if the samples grown immediately before or after are defective, the growth run is suspect
(ix) absolute perfection, which can be approached by Si–Ge only
(x) only if none of the above criteria apply then a sample may be expected to obey equation (3); in this case a sample is represented by a solid circle in Figs. 2, 3, and 4.

Some of these criteria are to be expected, and some require further discussion. Some correspond to evident growth problems, such as three-dimensional growth, cracking (in tensile layers), incomplete desorption of the oxide layer on the substrate, etc., which prevent the two-dimensional nature of the strained layer from supporting the strain. Although MBE, in particular, seems a conceptually very simple growth technique, it is well known that there are occasions when the MBE machine performs satisfactorily and occasions when it does not. There is not always a clear reason for this and workers must often repeat their procedures with considerable care in the hope that growth quality will improve. For this reason there are two criteria, (vii) and (viii), relating to the growth run rather than to the individual sample. Criterion (ix) recognises that crystal perfection may prevent relaxation if there is a sufficient absence of defects to produce dislocation sources. Spontaneous symmetry breaking, such as plastic relaxation, may be very slow when the original symmetry is close to perfect. This is supported by the experiments of Higgs *et al.*,[20] in which sudden relaxation of a Si–Ge layer was observed on annealing after evaporation of a submonolayer of Cu on to the surface to break the translational symmetry of the crystal.

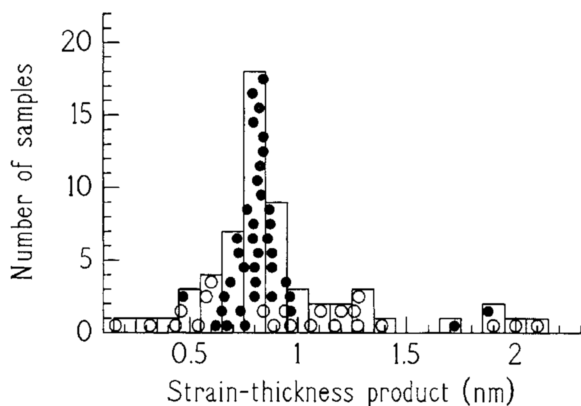




a sample 129, from earlier growth run (including samples 128 and 129) which gave only samples that did not fulfil criterion (x), showing abnormalities (*see* text); *b* sample 204, from later growth run (samples 202–206) which gave samples fulfilling criterion (x), showing normal orthogonal dislocation arrays only; *c* graph showing equation (3) as solid line – as expected, points for samples 203–206 (solid circles) are close to curve, whereas points for samples 128 and 129 (open circles) follow trend expected for e.g. three-dimensional growth, shown by arrows marked 3D

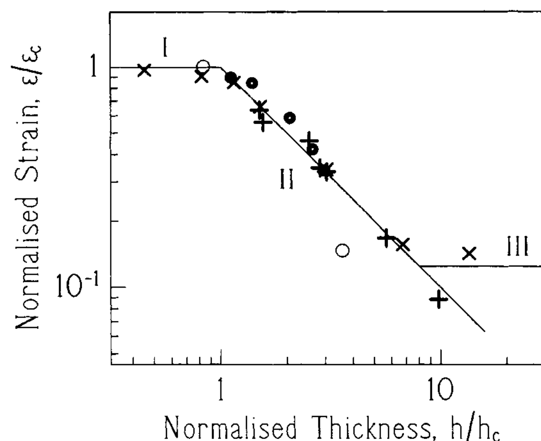
2 Results from two growth runs in same molecular beam epitaxy (MBE) machine: scale bars represent 200 nm (TEM)

It is worth noting that, in the light of the criteria for the attribution of open or solid circles to samples, there is a scarcity of data in the literature which can be used to test equation (3). Most authors report only those data and results of characterisation relevant to their thesis. Unfortunately most of the literature does not give sample numbers, so it is not possible to determine whether the samples in a set were grown contiguously or in what order. There is also often little comment on the quality of interspersed samples.



samples were grown by MBE, metalorganic chemical vapour deposition, atomic layer MBE, and chemical beam epitaxy – most are undoped, but some ● samples are doped – most are single layers, and a few ● samples are multilayer structures (also shown in Fig. 4)

3 Strain-thickness products for about 50 InGaAs/GaAs samples plotted to form frequency histogram peaking sharply at 0·8 ± 0·1 nm: each sample is represented by ○ or ● according to criteria given in text



solid line represents expected behaviour; in region I, layers are below relaxation critical thickness and are expected to have full misfit strain; in region II layers relax according to equation (3); in region III work hardening sets in and layers become capable of supporting residual strain without small size effects; crosses represent some previously published data for single layers of MBE InGaAs[16] (×) and for multilayers[18] (+); circles (○) represent data of Maigné *et al.*[22] for AlInSb layers; solid and open circles have been allocated as described in text and value of 0·83 nm in equation (3) has been replaced by 0·95 nm in proportion to lattice constant of InSb

4 Present and previous data for normalised strain, or fraction of misfit strain, as function of thickness normalised by relaxation critical thickness defined by equation (3)

In Fig. 2 some results are shown from an MBE growth programme to show the typical behaviour of solid and open circle samples. The two open circle samples, 128 and 129, came from an early growth run. The plan view TEM image of sample 129 (Fig. 2*a*) shows a normal array of misfit dislocations. However, it also shows regions of lighter and darker contrast, which could be due either to compositional fluctuations or to interfacial roughness. Compositional fluctuations are supported by the observation that other samples in this growth run showed In droplets. In contrast, the later samples, 203–206, were quite normal in every respect and are therefore expected to obey equation (3).

Taking a much larger set of data, equation (3) is best tested by plotting a frequency graph of the strain–thickness products of the layers. From equation (3), this is expected to have the value $\varepsilon h = 0{\cdot}83$ nm. Figure 3 shows results for approximately 50 samples, from MBE, ALMBE, chemical beam epitaxy, and MOCVD growth programmes, grown during good and bad growth runs. Included are single layers and multilayers, doped samples and undoped, compressive layers and tensile. It can be seen that samples fulfilling criterion (x) (solid circles) are sharply concentrated in a peak centred on 0·8 nm, whereas samples condemned by criteria (i)–(ix) (open circles) are scattered over a wide range of values of εh with no preferred value.

Even ignoring the concept of open and solid circles, this frequency plot shows the significance of the value of $\varepsilon h = 0{\cdot}8$ nm, with more than half the samples lying within $\varepsilon h = 0{\cdot}8 \pm 0{\cdot}1$ nm (the mean of this set of samples obeying equation (3), ignoring the three outlying samples, is 0·80 nm and the standard deviation is 0·08 nm). However, the clear division of the scattered samples which are represented by solid circles and the highly concentrated samples represented by open circles, allocated according to the criteria given above, does confirm the relevance of these criteria to the predictability of plastic relaxation.

It may also be possible to identify mechanisms whereby samples can deviate from equation (3). Consider, for example, the many growth phenomena which can result in three-dimensional or islandlike growth. Among these is excessive mismatch (e.g. in InGaAs/GaAs, a mismatch exceeding about 0·017).[21] When island like growth occurs, the stresses at the island edges are much higher and relaxation can occur much more readily than in a continuous two-dimensional layer. Therefore initially the strain in the layer is expected to decrease below region I (*see* Fig. 4). As growth continues, the islands coalesce to form a continuous layer, but with a high density of misfit dislocations already formed within each island and also formed where the islands join. This results in a very high density of threading dislocations which propagate upwards as growth continues. Dislocation–dislocation interactions, i.e. work hardening, may then prevent relaxation occurring readily so that the layer crosses region II of Fig. 4 and is eventually situated above the curve of equation (3) (*see* Fig. 2*c*).

RELAXATION IN OTHER SYSTEMS

As noted above, there are few results in the literature which can be used to verify equation (3). There are, however, some data consistent with equation (3), and many data not in agreement with it but which can be understood in terms of growth quality.

AlInSb

Maigné *et al.*[22] have recently investigated the residual strain in $Al_xIn_{1-x}Sb$ layers grown on InSb. The mismatch is tensile, and is $0{\cdot}053x$. In six layers of thickness 100 nm with x varying from 0·15 to 0·5 the strain–thickness product is in the range 0·8–1·2 nm (*see* Fig. 4). One sample ($x = 0{\cdot}15$) is below critical relaxation thickness, and Maigné *et al.* comment that the DCXRD {115} peaks of a further sample ($x = 0{\cdot}64$) were very weak. These two samples are therefore represented by open circles in Fig. 4. Since strain–thickness products would be expected to scale with lattice constant, the 0·83 nm in equation (1) would be about 1 nm in InSb based materials. Figure 4 shows that this is in good agreement with the data of Maigné *et al.*[22] Thus although they conclude from their data analysis that the mechanism of strain relief may be different from that in the InGaAs system,[22] their results may also be taken as evidence that equation (3) is generally true in the III–V compound systems (with the value of 0·83 nm adjusted for lattice constant).

AlGaAsSb

Lazzari *et al.*[23] presented results for a large number of samples of AlGaAsSb grown on GaSb with both tensile and compressive strains. Whereas the tensile samples have strains less than those predicted from equation (3), in contrast with the tensile AlInSb layers mentioned above, the compressive layers are scattered on and above the curve of equation (3). Insufficient information is given to determine whether these samples may be best represented by solid or open circles, but it is significant that some of them lie on the curve of equation (3).

II–VI compounds

Most of the present data are from large misfit systems, such as ZnS or ZnTe grown on GaAs. Generally, layers are almost fully relaxed even at moderate thicknesses below the relaxation critical thickness of equation (3). For example, Giannini *et al.*[24] report that ZnS layers of thickness $\geqslant 0{\cdot}42$ μm grown on GaAs are almost fully relaxed, with residual strain accounted for by thermal mismatch.

Multilayer structures

Rarely would a useful metamorphic structure consist of a single layer only. Usually, prediction of the relaxation of graded layers, superlattices, and other multilayer structures is required and consequently knowledge of the interaction of different strained layers is necessary.

THEORY OF MULTILAYER STRUCTURES

Theoretically, the analysis of multilayer structures presents little problem. The Matthews theory can be expressed in terms of the force on a threading dislocation segment given by $F=\sigma bh$. This force is compared with the line tension of the misfit dislocation to yield the Matthews equation.[2] Within this theory, the variation of σ across the thickness is not important, the main concern is the integrated force $F=\int \sigma(h) b \, dh$.[18] During growth, if this force reaches the dislocation tension when integrating from the free surface down to any depth, then misfit dislocations will form at that depth and the layers above will be relaxed accordingly.

The Matthews theory is an example of a class of models of plastic relaxation for which analysis is particularly straightforward. These are the models in which relaxation is due to properties of the strained layer only, and does not depend on parameters such as growth rate. A quantity S, which is a function of state, may therefore be calculated for each layer and summed (or integrated) over a complex structure. Equation (3) shows that the present model is of this type, with S representing the strain–thickness product which can be summed or integrated over the structure.[18] It can be shown that in all these models, relaxation occurs at one plane only and that this plane can move upwards (towards the free surface) during growth but never downwards.[25] The proof is beyond the scope of the present work, but the results are significant since they ensure the stability of underlying layers which have already relaxed when further layers are grown over them.

The model of the relaxation of linearly graded layers presented by Tersoff[26] is consistent with this result. It is predicted that the layer should relax to zero strain, except for an unrelaxed region at the top. With further growth, the parts at zero strain remain at zero strain.

EXPERIMENTAL RELAXATION OF MULTILAYER STRUCTURES

It was shown in Fig. 2 that multilayer structures do appear to obey equation (3). Many authors have reported linearly graded layers which behave in agreement with the Tersoff[26] model, i.e. in accordance with equation (3). It is perhaps more important to highlight the discrepancies between the observed behaviour of multilayers and the theory, showing areas where further experimental work is required.

For simple layers of a single composition, equation (3) predicts that to achieve zero strain would require infinite thickness and the concepts of work hardening and intrinsic elastic yield strength explain the deviation from equation (3) at low strains (region III of Fig. 4). In contrast, the theory for multilayers and graded layers predicts that zero strain can be achieved in a layer at a finite thickness, by growing a more highly strained layer on top. Whether this prediction is valid or not is not yet apparent. Data on double and triple layer structures[18] show that residual strain can remain in underlying layers even when the structure as a whole obeys equation (3). In contrast, several authors report that the relaxed portion of a graded layer does indeed relax completely,[27,28] in accordance with the Tersoff[26] model and with equation (3). Further work is required to establish the conditions under which residual strain occurs in layers that should be at zero strain, and the effect such residual strain may have on the relaxation of higher layers.

ACCURACY OF PREDICTION

It is worth commenting on the accuracy with which plastic relaxation may be predicted and indeed measured. In unrelaxed epitaxial structures, microscopic uniformity may be assumed and material characteristics such as lattice parameter may be measured to several significant figures. A different approach is required for relaxed layers because the random and non-uniform nature of plastic relaxation requires a statistical approach. The breadth of the DCXRD rocking curve peaks does not correspond to measurement error but to a distribution of values of lattice spacing and tilt. Only the average value can be predicted. In addition, it is not yet possible to determine whether the width of the distribution of solid data points in Fig. 3 represents real differences between the samples or whether it merely represents the accuracy with which the DCXRD rocking curves can be interpreted.

Conclusions

It is concluded that plastic relaxation is predictable, to the accuracy of measurement, at least for III–V growth of the highest quality. The predictability of relaxation may indeed serve as a measure of growth quality in the III–V and II–VI systems. However, neither equilibrium considerations nor kinetic effects are responsible for the extent of relaxation; rather, dislocation multiplication mechanisms and the space required for them to operate account for the empirical strain thickness relationship.

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