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STRAIN RELAXATION IN COMPOSITIONALLY GRADED InGaAs/GaAs HETEROSTRUCTURES

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Abstract

Epilayer strain relaxation in the InGaAs/GaAs system occurs via two mechanisms, plastic deformation and/or surface roughening. Under conditions of two-dimensional growth, we find that compositionally graded InGaAs/GaAs (001) multi-layer buffer structures will plastically deform with <110> misfit dislocations approaching 100% strain relaxation. At higher growth temperatures, large-amplitude roughening is observed preferentially along the [110] direction, and the strain relaxation becomes asymmetric in the <110> directions. In single epilayers, the symmetry of the strain relaxation is dependent on the magnitude of the substrate offcut angle. In all cases, the epilayers develop a tilt about an in-plane axis in proportion to and opposite in direction to the substrate offcut. With roughening, there is also a change in the orientation of the tilt axis such that only the dislocations with $[1\bar{1}0]$ line directions develop a preferred tilt component. These results illustrate the importance of surface steps and morphologies to strain relaxation and perhaps offer clues to the identification of the dislocation formation mechanisms at these interfaces.

Key Words: InGaAs, GaAs, epitaxy, strain, X-ray diffraction, dislocation, lattice mismatch, tilt, surface roughening.

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Introduction

It is well known that epilayer strain at lattice-mismatched semiconductor interfaces is relieved via the generation of misfit dislocations when the thickness exceeds a certain critical value. However, the exact mechanism has not been conclusively identified. In the InGaAs/GaAs system for mismatch less than about 1% (x = 0.15), when the growth is two-dimensional (2-D), the predominant type of misfit dislocation that forms is 60° a/2<110>. These reach the interface by glide on {111} planes. Substrate dislocations probably provide the first misfits in this compound semiconductor system but their density ($<10^5$/cm^2) is too low to explain strain relaxation greater than about 0.1% (x = 0.02). Hence, it is fair to say that most misfits originate by multiplication or nucleation at heterogeneous sources.

A second relaxation mechanism that occurs at higher growth temperatures and/or at very large mismatch is surface roughening. Both ternary and binary semiconductor systems will develop rough surfaces that relieve some strain before further relaxation occurs via misfit dislocations (Cullis *et al.*, 1992; Tersoff and LeGoues, 1994). In the extreme case, such as InAs/GaAs, islands form immediately and sessile edge dislocations are generated at the edges of these islands (M. Chisholm, private communication, 1987; Zhang *et al.*, 1993). In the single epilayer InGaAs/GaAs system, the growth conditions and interfacial strain which result in a transition from 2-D to three-dimensional (3-D) growth have not been clearly outlined for all compositions, and the consequences of large-amplitude roughness on device properties is not understood.

We have been studying the growth and characterization of strain-relaxed InGaAs single (Kavanagh *et al.*, 1988; Chang *et al.*, 1992a; Goldman *et al.*, 1994a; Raisanen *et al.*, 1994) and multi-layer compositionally graded buffer structures (Chang *et al.*, 1992b, 1993a, 1993b; Goldman *et al.*, 1994b) grown by molecular beam epitaxy (MBE) on GaAs substrates. By increasing the composition gradually, keeping the mismatch low, strain relaxation in the x = 0.3 - 0.5 In mole fraction

Table 1. Summary of results from double crystal X-ray diffraction on the strain relaxation of the **surface** epilayer of single, step-graded and linearly graded InGaAs/GaAs heterostructures. Listed are the sample structure, the In mole fraction x, the thickness t, the nominal growth temperature T, the substrate offcut angle nominally flat to a certain tolerance or cut towards (110) about an axis [010] or [100] θ, residual in-plane strain ε^r, strain relaxation with respect to the substrate R, tilt angle and axis δ, surface roughness amplitude and reference.

Sample Structure	x	t	T	θ	ε^r		R		δ		Surface Roughness Amplitude	Ref.
		nm	°C	°	$\times10^{-3}$ [110]	$[\bar{1}10]$	% [110]	$[\bar{1}10]$	° Mag.	Axis		
Single Layer	0.06	300	535	< ±0.05	3.4±0.2	3.4±0.2	13 ± 1	12 ± 1	0.01	[010]+10°	Small	Goldman 1994b
	0.06	300	535	2	4.1±0.2	3.5±0.2	8 ± 1	20 ± 1	0.02	[010]	Small	
	0.10	300	535	<±0.5	3.0±0.1	2.1±0.1	60±6	71±7	0.07	[010]	Small	new
	0.10	300	535	2	1.1±0.1	4.8±0.3	86 ±9	37 ±4	0.14	[010]	Small	new
3-step graded	0.31	300	535	2	0.44	3.33	98	85	0.55	$[\bar{1}10]$	Large along [110]	Chang 1992a
	0.33	300	535	2	0±.03	8.5±0.4	~100±10	63±6	0.50	[010]	Large	new
4-step graded	0.4	300	500	2	0.57	16.6	98	42	1.2	$[\bar{1}10]$	Large along [110]	Chang 1993a
	0.40	300	470	2	1.7±1	1.7±1	98±10	98±10	1.0	$[\bar{1}10]$	Small	Goldman 1994a
linearly graded	0.52	500	450	< ±0.5	1.1	2.2	97 ±3	94 ±3	0.27		Small	Chang 1993c

range can be studied. We find that these compositions exhibit asymmetric strain relaxation, surface roughing and epilayer tilting that are affected by the grading, substrate offcut angle and growth conditions including substrate temperature. Correlation of the structural properties and the electrical properties of bulk single layers, quantum wells and modulation doped test structures grown on GaAs is one of the motivations for this work (Chen *et al.*, 1992). This paper will review our structural results towards an understanding of the strain relaxation mechanisms in this system.

Experiment

Single and step-graded InGaAs layers were grown by solid-source MBE on (001) GaAs wafers, nominally flat or cut 2° towards [010]. For these growths, the As_4/group III beam equivalent pressure ratios ranged from 30-40, and GaAs growth rates were approximately 0.9 μm/hr. Growth temperatures were varied depending on the In composition and ranged from 580 to 470°C as measured by a thermocouple in contact with the backside of each molybdenum block. The step-graded samples consisted of composition steps of x = 0.1, each 300 nm thick. The total thicknesses for a 3 or 4-step buffer layer were 900 or 1200 nm, respectively, with surface In mole fractions of 0.3 or 0.4. In most cases, a modulation doped InAlAs/InGaAs heterostructure was grown on the surface of the multi-layer buffers consisting of 10 nm of undoped spacer layer and 30 nm of Si-doped InAlAs supply layer capped with 10 nm Si-doped GaAs. Uniform composition $In_{.53}Ga_{.47}As$ layers were grown on a linearly graded InGaAs buffer layer on nominally flat (001) GaAs using chemical beam epitaxy with an As_2 source and solid-source Ga and In. The growth temperature was varied from 600 to 450°C as the In concentration was gradually increased at an overall grading of about x = 0.1/380 nm.

The strain relaxation, epilayer tilt about an in-plane axis, and In compositions were measured with double crystal X-ray diffraction using Cu K_α radiation monochromated by four Ge (220) crystals. Samples were also investigated with transmission electron microscopy (TEM) at an accelerating voltage of 300 keV.

Results

The results of X-ray diffraction from the surface epilayer of single and multi-layer InGaAs/GaAs heterostructures are summarized in Table 1. Listed is the In composition, thickness, the nominal growth temperature, the magnitude of the substrate offcut angle (flat to a given tolerance or 2°→[010] about a [100] axis), the residual in-plane strain and percentage relaxation with respect to the substrate in [110] and $[1\bar{1}0]$ directions, its tilt magnitude and axis, the surface morphology, and a reference where more information can be obtained about the sample.

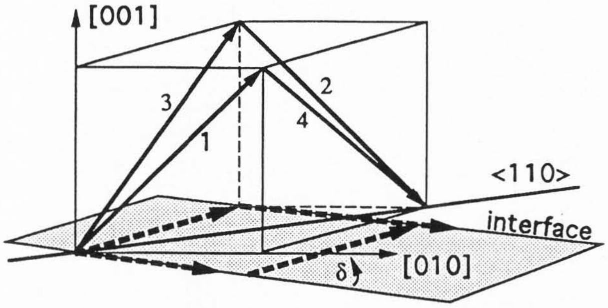

Figure 1. Interface components of the Burger's vectors of the four 60° misfit dislocations for a (001) interface off-axis towards [010] by angle δ about the [100] axis.

Consider first the single heterostructures. The strain relaxation is symmetric, to within experimental error, if the layer is grown on a wafer flat to a tolerance of ±0.05, (x = 0.06) otherwise it is asymmetric. The results indicate that the offcut has increased the relaxation in one direction at the expense of the other, while the average relaxation remained approximately constant. The strain relaxation by a given misfit dislocation is in proportion to the magnitude of the perpendicular component of its Burger's vector on the interface. The offcut causes the interface to be tilted off the exact (001) surface by a given angle and axis. There are four 60° a/2<110> dislocations that form at these interfaces as shown in Figure 1. For the [100] offcut axis, Burger's vector 1 (2) in the figure will have a slightly smaller (larger) component on the interface and hence will be slightly less (more) efficient at strain relief. Meanwhile, the interface components of Burger's vector 3 and 4 will be unaffected by the offcut. Ayers *et al.* (1991) argue that this difference changes the driving forces, resulting in a higher preference for the formation of dislocations with Burger's vector 1. However, the α and β dislocations are affected equally. Therefore, if we can assume perfectly smooth surfaces, asymmetric strain relaxation must be related simply to the intrinsic differences between the core structures of α and β dislocations.

In n-type GaAs, the dislocations with $[1\bar{1}0]$ line directions (α dislocations) are reported to have higher glide velocities (Kuesters *et al.*, 1986; Yonenaga and Sumino, 1993). However, in our case, the direction of maximum strain relaxation was sometimes [110] and other times $[1\bar{1}0]$ for nominally identical growth conditions. Since we see a preference for α dislocations in some cases and β dislocations in others, the intrinsic characteristics cannot be the only determining factor in the formation mechanism.

Another explanation may be that the simple presence of interface steps, surface steps or asymmetric step morphologies affects the dislocation formation mechanism in some way. Surface steps, like dislocations, have different characteristics in orthogonal directions on (001) surfaces. For higher mismatched single epilayers, for example, x = 0.2 (Werner *et al.*, 1993) and x = 0.38 (Hara *et al.*, 1993), an asymmetric dislocation density is observed that is independent of the magnitude of the offcut towards [010]. In their samples, surface roughening is perhaps changing the dislocation formation mechanism as is true for our step-graded multi-layer results to be discussed below.

The offcut is also expected to affect the choice of out-of-plane Burger's vector component. A preference for an up or down direction will cause the epilayer to develop a tilt during relaxation (Ayers *et al.*, 1991). This tilt is expected to increase in proportion to the offcut angle and degree of strain relaxation, i.e., the number of misfit dislocations; it is expected always to act in a direction such as to reduce the surface offcut towards a perfectly singular surface. These are precisely the results we observe. The epilayer tilt increase of the single layers with In composition and offcut is entirely consistent with these simple arguments. Similar results are reported for the SiGe system (Mooney *et al.*, 1994).

Somewhat of a surprise, however, is the fact that the tilt axis remained the same as the nominal substrate offcut axis even though the strain relaxation in the single epilayers was asymmetric. This means that the density of dislocations with a preferred tilt component was the same in the two orthogonal line directions. This result also means that the factors that determine asymmetric strain relaxation and hence, asymmetric dislocation formation rates, do not apparently affect the choice of tilt component to the same degree.

When two or three layers of higher In composition are added forming 3 or 4-step buffer structures the situation changes. The strain asymmetry on 2° offcut substrates (about an in-plane <010> axis) became a function of growth temperature and associated with large-amplitude surface roughening (30-50 nm). For an In mole fraction of 0.3, a growth temperature of 535°C was associated with surface roughening preferentially along the [110] direction such that grooves are observed along the $[1\bar{1}0]$ direction spaced roughly 1 μm apart. The degree of roughening increased for the x = 0.4 layer. For this composition, a lower growth temperature of 510°C developed an even greater severity of grooving along the $[1\bar{1}0]$ direction than the x = 0.3 layers at 535°C. TEM work on this 4-step buffer showed that the grooves contained vertical grain boundaries with a parallel network of [110] dislocations that extended into the lower layer of the buffer (Chang *et al.*, 1993a).

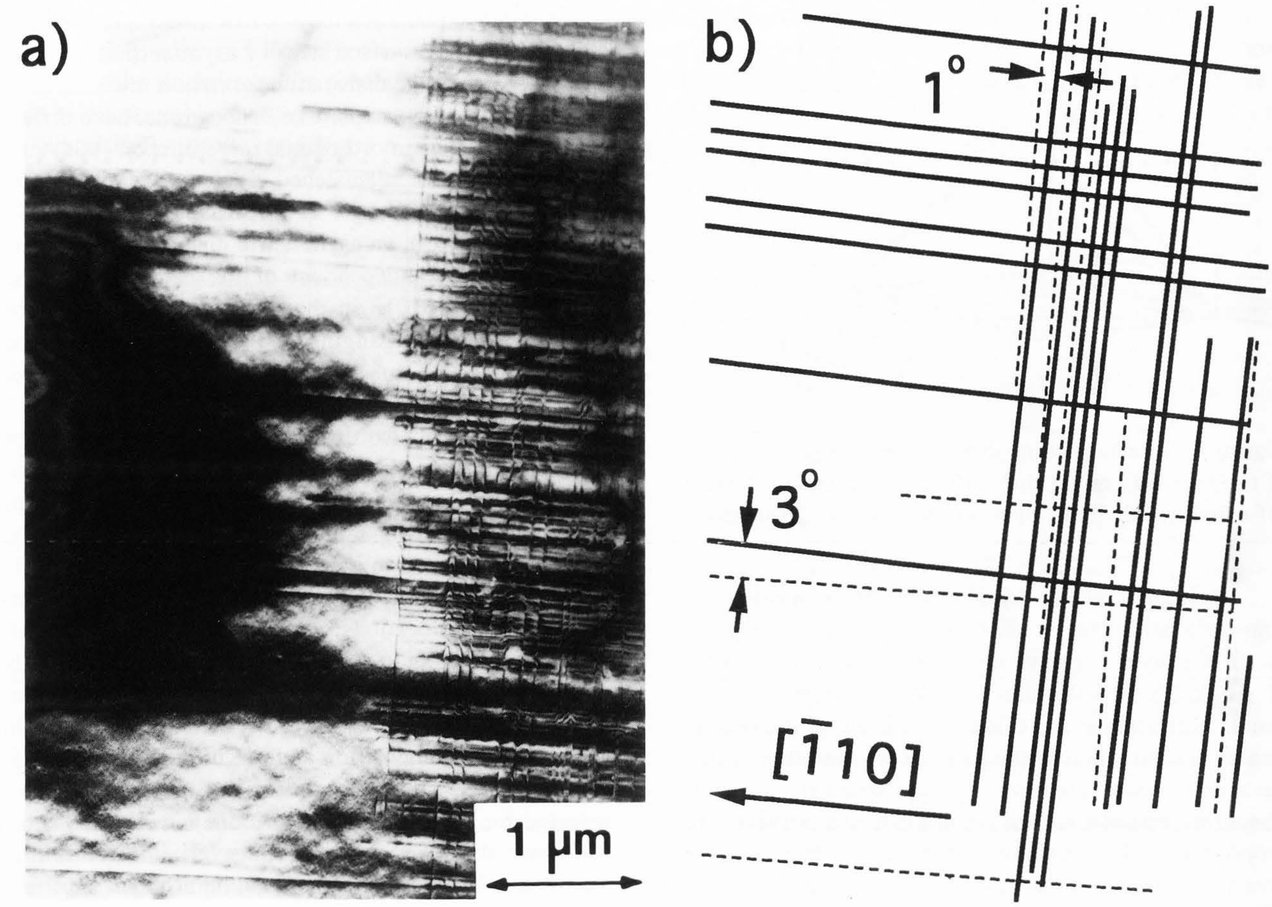

Figure 2. (a) Plan-view transmission electron micrograph of a 3-step compositionally graded $In_xGa_{1-x}As$ buffer layer grown on GaAs. The sample is wedge-shaped and therefore misfit dislocations from the surface modulation doped structure and the top interface of the buffer layers are distinguishable. (b) Diagram of the misfit dislocation lines showing the effect of surface off-cut on their directions.

When surface grooving was observed, the strain relaxation was asymmetric, and the epilayer tilt axis changed to [1$\bar{1}$0].

Note that the direction of maximum strain relaxation [110] was also the direction of roughening. In other words, the α dislocations were parallel to the direction of the surface grooves. Also, the asymmetry in the percentage relaxation is all the more dramatic if it is remembered that, with respect to the underlying buffer epilayer, the difference in the two directions is much larger. The change in tilt axis to [1$\bar{1}$0] indicates that only the generation of α dislocations is affected by the substrate offcut. The β dislocations had no tilt component preference, although the same driving force, i.e., a substrate offcut angle of 1.4°, was in effect. This result is also seen from plan-view TEM images. Figure 2 shows a plan-view image of the 3-step buffer sample with the large strain asymmetry and [110] tilt axis. The TEM sample was prepared by etching the backside until a small hole developed. Around the edge of the hole, the thickness is wedge-shaped. In the micrograph, the thinner regions show only the surface modulation doped interfaces where some misfits are observed (low density). As the wedge gets thicker, the sample now also contains the deeper 0.2/0.3 In interface. As shown in the diagram (Fig. 2b), the dislocations are not parallel but there are two sets of dislocations that are a certain angle apart. This angle is created by the vicinal surface and is equal to twice the offcut angle in the line direction (Kightley *et al.*, 1991). In the [1$\bar{1}$0] line direction, the angle is about 3° indicating an offcut of about 1.5° while in the [110] line direction, the angle is only 1° or an offcut of about 0.5°. Both are consistent with the X-ray measurements. From X-ray measurements, this sample had a tilt of 0.85° about the [1$\bar{1}$0] axis reducing the surface offcut from 1.4 to 0.55°, in the [110] orientation. The overall surface offcut angle has been reduced to 1.52°, and it is now oriented away from the [010] axis towards the [110]. These results are shown in a polar plot in Figure 3.

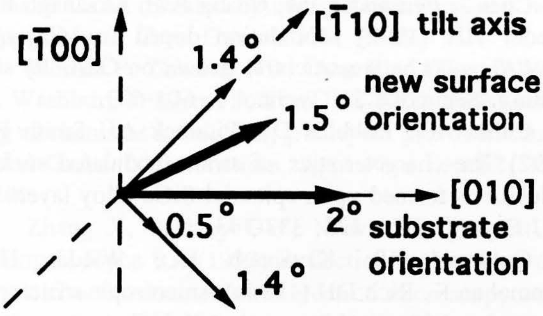

Figure 3. Polar plot of the surface offcut angle for a 3-step InGaAs buffer layer grown on a GaAs(001) substrate offcut 2° towards [010] that has undergone a tilt about the $[1\bar{1}0]$ axis of 0.9°.

When the substrate growth temperature was reduced, the surface morphology showed only the familiar small-amplitude (≥5 nm) roughness ("cross-hatched") visible with Nomarski interference microscopy, and the strain relaxation became symmetric; for example, the 4-step buffer layer grown at 470°C. However, the epilayer tilt axis still changed to $[1\bar{1}0]$ in this case. It is possible that an earlier roughness was buried after relaxation (Jesson *et al.*, 1993). We need to quantify the degree of roughness as seen with Nomarski.

The linearly graded sample grown on a nominally flat substrate had a symmetric relaxation and a tilt axis the same as the substrate. The tilt was small since the wafer was nominally flat but the finite tilt measured revealed the actual offcut and orientation of the substrate. The surface of this sample was symmetrically cross-hatched, and hence, the growth temperature or strain was sufficiently low to prevent large-amplitude roughening from developing.

Discussion

Definitive conclusions from this data regarding the question of whether the dislocation source is heterogeneous, multiplicative or some combination of both cannot be made. However, the results show that strain asymmetry is apparently independent of tilt asymmetry. The epilayer tilt axis remained the same as the substrate offcut axis for single layers and step and linearly graded multi-layers grew with no large-amplitude surface roughening. This was in spite of the fact that the strain relaxation in the single layers was asymmetric. When the tilt axis remains constant, this means that the rates at which tilts are generated in orthogonal directions are identical. There need not be equal densities of dislocations in the two directions (as for symmetric relaxation), but the difference in the density of tilt up and tilt down dislocations must be the same in both directions. The dislocation generation source consistent with these data may simply be sensitive to the local step density or heights. When these are equal in both directions, as is likely the case with a relatively smooth surface, then perhaps if the source is dependent on this density then the degree of tilt in each <110> dislocation line direction is identical. This would also explain why when the surface becomes very rough in a particular direction and when the local density of steps is asymmetric, the degree of tilt differs dramatically in the two directions.

A further conclusion from this is that if we are to have symmetric tilt with asymmetric relaxation, as was observed with the single layers, then a single bi-directional source is not feasible. A bi-directional multiplicative source that has been proposed for SiGe, the so-called modified Frank-Read loop source, generates a corner shaped misfit dislocation with ends running in both directions (LeGoues *et al.*, 1992). The source is associated with apparent pile-ups of loops into the substrate which we observe in the linearly graded InGaAs buffers (Chang *et al.*, 1993b). With only this source operating and equal glide velocities, the tilt axis remains as the substrate, however, the strain relaxation is then necessarily equal in the two directions. For single layers with unequal glide velocities, this source would have to operate in combination with a uni-directional dislocation source of some kind.

The fact that the multi-layers grown under continuous 2-dimensional growth conditions develop symmetric strain relaxation, although the lower composition single layers do so only on flat wafers, also needs to be explained. First of all, since the multi-layers are close to 100% relaxed, any asymmetries in the rates of relaxation in perpendicular directions in the lower layers are buried. The only layer that could conceivably still display asymmetric relaxation is the top layer which is not completely relaxed. The surface layers of one of the four-step-graded samples and the linearly graded sample had symmetric relaxation. Symmetric strain relaxation on an offcut wafer means that dislocation nucleation and velocity in the higher In compositions may be less sensitive to the core structure and to surface steps (at least under the growth conditions we used). This also means that when strain relaxation is asymmetric at these concentrations, a serious change in the step morphology has occurred. This apparently causes an impediment to either nucleation or glide, precisely consistent with our observations on samples with severe roughening.

Conclusions

In conclusion, we have investigated the strain relaxation in single and multi-layer step and linearly graded

InGaAs/GaAs(001) heterostructures. In single epilayers, the strain relaxation is asymmetric in the two in-plane <110> directions unless the wafer is flat to high tolerances (±0.05°). For offcut wafers, the maximum relaxation in n-type layers grown under nominally identical growth conditions can occur in either direction, inconsistent with dislocation velocity arguments. In-plane tilting of the epilayers occurs in proportion to the offcut and relaxation and is symmetric in single epilayers in spite of asymmetric relaxation occurring at the same time. Thus, the mechanism for strain asymmetry is independent of tilt asymmetry. At higher In compositions in multi-layer structures, the strain relaxation is close to 100% and symmetric if the growth is 2-dimensional and only low amplitude cross-hatched roughness occurs on the surface. If the growth temperature is too high, severe surface roughening occurs preferentially in the [110] directions creating deep grooves along the $[1\bar{1}0]$ direction. This is associated with inhibited relaxation in the $[1\bar{1}0]$ direction and a change in the tilt axis to $[1\bar{1}0]$. The effects of offcut and, therefore, step morphologies and surface roughening on strain relaxation need to be investigated more closely.

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Discussion with Reviewers

R. Beanland: It seems clear that there is a correlation between surface morphology, dislocation structure and relaxation. It is stated in **Results** that "Another explanation may be that the simple presence of interface steps, surface steps or asymmetric step morphologies affects the dislocation formation mechanism in some way." Is it not possible that the converse may equally be true: that the dislocations may influence the surface structure, or that both of these effects may be influenced by some other factor?
Authors: Certainly, the 60 degree dislocations affect the surface roughness as they each introduce a surface step. The tilt tells us the ratio of steps up to steps down, and this could conceivably generate a coarser roughness than for a surface with the same number of 60 degrees but without tilt. The dislocations also cause roughening when surface diffusion responds to the pattern of residual strain in the epilayer. Whether there is a feedback process going on here would depend on whether the dislocations are nucleating at the surface or interface, a fact which we do not know at this time.

R. Beanland: The amount of relaxation and the tilt measured for the different samples are compared in order to draw inferences on the mechanisms which produce these effects. This requires that the variability in each measurement is less than the variability between samples. Is it certain that this requirement is satisfied? For example, it is mentioned that similar constant composition samples can have strongly asymmetrical relaxation, some being relaxed most by dislocations lying parallel to [110] and others by dislocations parallel to $[\bar{1}10]$; also, the table of measurements shows that the relaxation of virtually identical 3-step graded samples can vary by over 30%. A related point is the variability of relaxation and tilt for each sample; are the same results obtained, within experimental error, for different regions of the same sample?
Authors: We have not checked the uniformity of every sample but in those that have been measured and remeasured on different areas of the same wafer, there is an absolute variation in relaxation or composition of some amount. But the overall trend, including the direction of relaxation, is reliable. The real problem is essentially that there is much less repeatability in the growth temperature from MBE growth to MBE growth. This lack of control in the MBE system is what we believe accounts for the variability in X-ray results between samples that were apparently grown under the same conditions.

P. Mooney: The effects of dislocation glide have not been mentioned here. In SiGe/Si heterostructures, glide results in significant annihilation of threading segments due to self-aligned nucleation sources. Is annihilation observed in these structures or would it only occur in the case of corner dislocations (bi-directional sources)?
Authors: Annihilation occurs when two threading dislocations with the same Burger's vector traveling in opposite directions encounter each other. The probability of this occurring is greatest if sources are aligned in some manner such as with multiplicative sources at misfit dislocation crossing points. Annihilation must be occurring if the nucleation rate of the dislocations exceeds that of the final number inferred by our X-ray measurements or from TEM. However, we do not know what source is operating in our InGaAs/GaAs, and we currently do not know the nucleation rate. A number for the nucleation rate in the SiGe/Si system has been deduced from measurements of the tilt rate as a function of offcut (LeGoues *et al.*, 1993). We are endeavoring to carry out similar experiments with InGaAs/GaAs.

P. Mooney: Even if the same number of dislocations are nucleated in the two perpendicular directions, would differences in glide result in asymmetric relaxation? The number of misfit segments would be the same but their lengths would be different, making the density of misfit segments different and, thus, the strain relieved in the two directions unequal.
Authors: You are correct, but the minute you have different densities or different total lengths of dislocations in the perpendicular in-plane directions, the tilt orientation is also automatically determined and will be equally asymmetric.

G. Salviati: Some authors (Kidd *et al.*, 1993; Ferrari *et al.*, 1994) have found that epilayer in-plane tilts can occur in $In_xGa_{1-x}As/GaAs$ double layers ($0.05 < x < 0.2$) (which follow the same relaxation law as observed for single layers) grown on flat substrates (±0.2-0.5°). In these cases, the difference among the offcut axis

Burger's vector components on the interface is extremely small, and it is not so evident that it can affect the epilayer tilt in a significant way. Could the authors comment on these results on the basis of the hypothesis that the substrate miscut provides the driving force for the epilayer tilt?

Authors: The first reference you cite deals with reciprocal space maps of a double layer structure. The tilt that is mentioned is not the overall epilayer tilt but rather the local mosaic tilt of the lattice planes created by the dislocations. This cannot be obtained from our rocking curves but it would be interesting to do such a comparison given the data. We would not be surprised if the local tilts were larger than the average that we measure. The second paper reports on data showing a tilt of 300-400 arcsecs even though the substrate was nominally singular. We would question the accuracy of the wafer flatness. A tilt of 300-400 arcsecs corresponds to only 0.1° of tilt, a very small offcut. However, one assumption that has been made here is that the dislocation source is capable of providing a potentially even distribution of Burger's vectors. If this is the case, then the final tilt is determined by the offcut and the magnitude of its effect on the source nucleation rate. However, one way that a flat substrate might produce tilted epilayers would be if there was an uneven distribution of Burger's vectors in the substrate dislocations. If the source is depending on a multiplication at crossed misfits, for example, and if there are only a limited selection of Burger's vectors available, then tilt will develop with any offcut angle. Alternatively, if the source is related to a surface growth morphology, this can be completely independent of the offcut.

Additional References

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