Title

Rapid Variation in Epilayer Threading Dislocation Density near x = 0.4 in Ge{sub x}Si{sub 1-x} on (100) Si

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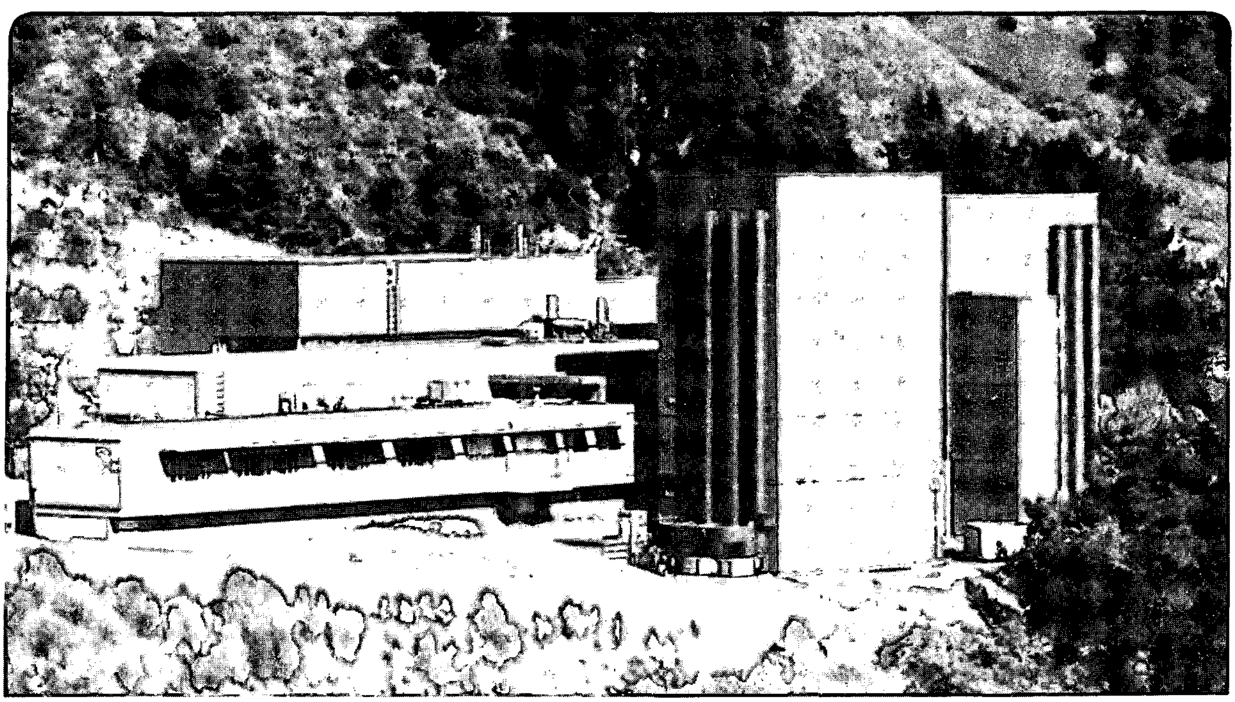

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DISCLAIMER

Rapid Variation in Epilayer Threading Dislocation Density near $x = 0.4$ in Ge_xSi_{1-x} on (100) Si

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Abstract

A sharp increase in the number of dislocations threading from the heterointerface to the growth surface has been observed when Ge content is increased (and consequently critical thickness decreased) at about $x = 0.4$ in Ge_xSi_{1-x} single epilayers grown on (100) Si. This increase by a factor of 60X seems to be due to the change in glide behaviour of epithreading dislocations associated with interfacial misfit dislocations with thickness. The threading density difference persists in layers of equal thickness but differing Ge content to thicknesses well above critical.

Introduction

Si-Ge alloys have received much attention lately as potential candidates, via band-gap engineering and heterostructure growth, for HBT and optical device materials. A critical step in many of the potential applications of these electronic or photonic materials is the growth of a thick epilayer or superlattice of high ($x = 0.5$) average Ge content, but low dislocation density in the device region. Substantial effort has gone into growing low-dislocation density 'effective substrates' of $x = 0.5$, for example (E.H. Kasper, H.-J. Herzog, H. Daembkes, and G. Abstreiter (1986)), but as yet these have not been as successful as desired.

Hull and Bean (1989 I) have recently shown that the average dislocation length should decrease with decreasing critical thickness due to blockage of slip in extending misfit dislocations by intersection with prior, perpendicular misfit dislocations. As a consequence, the number of misfit dislocations, for a given degree of relaxation, should increase, and this should result in an increase in the number of unreacted interfacial dislocation ends, which ultimately constitute epithreading dislocations. An increase in the epithreading dislocation density should therefore be expected in the region where the Hull and Bean model begins to apply, namely the thickness at which the repulsive force (i.e. the interaction stress integrated along the entire glissile epithreading segment) exerted on a moving dislocation by a perpendicular-lying dislocation becomes an appreciable fraction of the excess misfit stress-induced force (J.Y. Tsao and B.W. Dodson (1988)) which is available to extend misfit dislocations by motion of epithreading ends.

We have examined a series of Ge_xSi_{1-x} alloy single epilayers grown on (100) Si, both near and substantially above their critical thicknesses, and quantified the change in threading dislocation density as a function of x for a given thickness.

Experimental Procedure

The epilayer growth was by MBE at 550°C upon double Si buffer layers, each of 100nm thickness, grown at 750 then 550°C. The epilayer was grown on an intentionally unrotated single wafer, in masked bands of nominal 10, 20, and 100nm nominal thicknesses. The masking was arranged to maximise the concentration gradient across the wafer, leaving the composition constant in the direction perpendicular to the mask edges. The target central composition was x = 0.5.

Specimens for TEM were prepared by mechanical thinning followed by, for plan view, chemical thinning in HNO_3:HF 15:1 and/or Ar^+ ion milling, or, for cross sections, simply by milling. Imaging was done in conventional bright- and dark-field, but principally in weak-beam dark-field conditions.

Cross-sectional microscopy revealed that the epilayer thickness was constant across the width of each masked band, with measured thicknesses of 9.5, 18, and 85nm. High spatial resolution EDX was performed on material from the central region of the thickest (85nm) layer using a VG HB501 STEM, at probe size of 1nm. This gave a Ge content at the heterointerface of x = 0.42, which rose rapidly (in <10nm) to x = 0.47, remaining constant thereafter. Semiquantitative EDX (using a Philips 400 and a probe size of 30nm) was also performed on the 85nm band cross sections from the centre and both extrema, and showed a relative variation of 0.20x (i.e. 20% relative change in Ge content) from the central composition, in line with quantitative measurements made on similarly grown materials. Hence the low and high Ge contents are taken here as x = 0.38 and x = 0.57.

Results

Misfit dislocation length just above the critical thickness was seen to agree qualitatively with the observations of Hull and Bean (1989 I). The thinnest epilayer band showed an orthogonal array of predominantly edge dislocations only in the x = 0.57 region. These dislocations were randomly arrayed and of very variable length, with a mean length of 350nm (standard deviation of 310nm). They were very commonly observed to have terminated in the vicinity of a perpendicular-lying dislocation. Misfit dislocations appeared in the central region (x = 0.47) in the 18nm thick band, again in an orthogonal array, but in a ratio of edge to 60° type of 2:1. These were generally longer at the same interdislocation spacing, with an average length of about 1 micron, but again of very variable length. These morphologies are shown in Figure 1. The change from 60° to edge type is the subject of another publication (E.P. Kvam, D.M. Maher, and C.J. Humphreys (1989)).

For the lowest composition ($x = 0.38$), the dislocation content near critical thickness could not be observed in these materials. For comparison, however, it may be noted that in $x = 0.20$ epilayers near critical thickness (200nm thick), 60° dislocations were universally observed, with an average length of at least several tens of micrometres (D.J. Eaglesham, E.P. Kvam, D.M. Maher, C.J. Humphreys, and J.C. Bean (1989)).

The morphologies of the 85nm thick epilayer at low, central, and high compositions are shown in Figure 2. The micrographs were taken in weak-beam conditions to optimise the visibility of both the misfit dislocation array (horizontal and vertical arrays) and the epithreading dislocations. The epithreading dislocations in the low-x case (arrowed) seem to be in the glissile configuration observed at similar or lower compositions (e.g. R. Hull, J.C. Bean, D.J. Werder, and R.E. Liebenguth (1988); Eaglesham, et alia (1989)). The configuration is not so straightforward in the other cases; although many epithreading dislocations appear similar to the low-mismatch examples, several appear to wander from these configurations. This may be due to a number of reasons, e.g. glide may carry on at a distance from the heterointerface nonuniformly when prior dislocations create an impediment very near the interface, or because the epithreading segments immobilised at an early stage have not continued to grow on their glide plane(s) as epi growth proceeded. It was also indeterminant whether the epithreading segments observed were screw type, which could only be produced by 60° interfacial dislocations, or 60° type, which could be associated with either edge or 60° misfit dislocations.

The epithreading density is plotted for these materials in Figure 3; also included for comparison, is the density measured in a low-x epilayer substantially beyond its critical thickness ($x = .20$, 1μm thick) grown under the same conditions. There is a slow rise in epithreading density to about $x = .35$, then a rapid increase, by about 60X, as Ge concentration increases to about 0.45, which then returns to a slow rise again. As seen in fig. 2, there is some difficulty in reliably quantifying the epithreading density in the higher concentration materials, so the absolute values must not be taken as perfectly reliable; however, they are probably lower bounds rather than upper, as the epithreading dislocations having <100> traces were readily identified, but those with traces near <110> (e.g. lying in <211> inclined directions) would be difficult to deconvolute from the misfit dislocation array, and unlikely to be counted.

Discussion

The rapid increase in epithreading dislocation density shows not only the blockage of glissile threading dislocation movement, with the consequence of extra misfit dislocation introduction, as previously reported by Hull & Bean (1989 I), but also illustrates that these blockages are, by some process, made permanent, since they persist to thicknesses well above their initial occurrence. These processes could be the forementioned growth into sessile configurations, entanglements with other epithreading dislocations, or simply the loss of excess misfit stress (by introduction of sufficient misfit dislocations) needed to drive dislocation motion having occurred prior to growth to thicknesses where blocking would be ineffective. If the latter is the case, the dislocation epithreading line density would be directly related to the initial line length near critical thickness.

We can compare two extreme examples at a similar degree of misfit relaxation: the 9.5nm thick, $x = 0.58$ layer here, and a 300nm thick, $x = 0.20$ layer previously examined (Eaglesham, et alia). The latter had dislocation spacings of about 1 micrometre, and line lengths generally well over 10 micrometres; if we estimate the average length as about 20 μm, the epithreading density thus established at this relatively early stage was $\approx 2\times10^{7}$ cm^{-2}. Similarly, for the thin, high-mismatch layer, we use the measured spacing and length averages of 350 nm and 310 nm, which yield an epithreading density of $\approx 4\times10^{9}$ cm^{-2}. In both cases it has been assumed that there is one epithreading dislocation per dislocation termination.

Each of these is a factor of 10X below the measured density at much larger thicknesses, implying that substantial further dislocation introduction is responsible for both further epithreading density increase and misfit strain relief. However, this also establishes that there is a probable pattern of final epithreading density dependence upon initial dislocation introduction.

The obvious further question arises as to the source of the misfit dislocation density in higher mismatch materials, i.e. a large number of short dislocations as opposed to a far lower number of very long dislocations. Hull and Bean (1989 II) have shown that, at high mismatch, surface nucleation may be induced by the very high misfit stress, aided by both local variations in composition found naturally in a random alloy, and by an alloying effect to reduce the core energy of the dislocations. We note here that this is in conjunction with a probable further core energy reduction induced by the higher stress in the epilayer, which would cause a reduction in the core width.

While for low-mismatch materials pre-existing dislocations, dislocation interactions, oxide particles, and epi growth-induced defects have been seen to be heterogeneous dislocation sources, no such sources were observed to operate in these materials. There does exist a roughness at the interface of about 2nm and with characteristic length about 40nm (which seems to be the cause of the 'rumple' contrast seen as a general background in Fig.1),but this was not associated with any crystal discontinuities (no dislocations and very few stacking faults were observed in the 9.5nm, $x = 0.47$ material, for example), the interface region could only provide a nearly homogeneous nucleation site for dislocation loops. As such full loops would be far more energetic than half-loops the surface half-loop nucleation source seems very likely.

In short, although dislocation glide blockage by perpendicular dislocations seems to be the reason for dislocation length being quite short, it is not an explanation for the large number of misfit dislocations. The large number requires a well-distributed source, and surface nucleation at local inhomogeneities of composition fulfills this requirement. The large number of short dislocations being introduced near critical thickness is not alone sufficient to account for all the dislocations in thick materials which are substantially stress-relieved, but it seems that a consistent pattern is established at an early point, dependent upon the mode of dislocation introduction. This pattern is reflected in a sharp increase in epithreading density at about x = 0.45, which is the point where both glide blockage and surface sources become relevant factors.

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FIGURE CAPTIONS

Figure 1

Dark field plan view images (**g** = {220}) of misfit dislocations at the hereointerface near critical thickness. Dislocations lying parallel to g are 60° type, perpendicular to g either 60° or edge type. (a) x = 0.57, 9.5 nm epilayer (b) x = 0.47, 18 nm thick epilayer.

Figure 2

Weak beam dark field plan view images (**g** = {220}) of 85 nm thick epilayer structures (a) x = 0.38 (b) x = 0.47 (c) x = 0.57

Figure 3

Plot of measured epithreading density vs. composition for thick films (considerably beyond critical thickness).

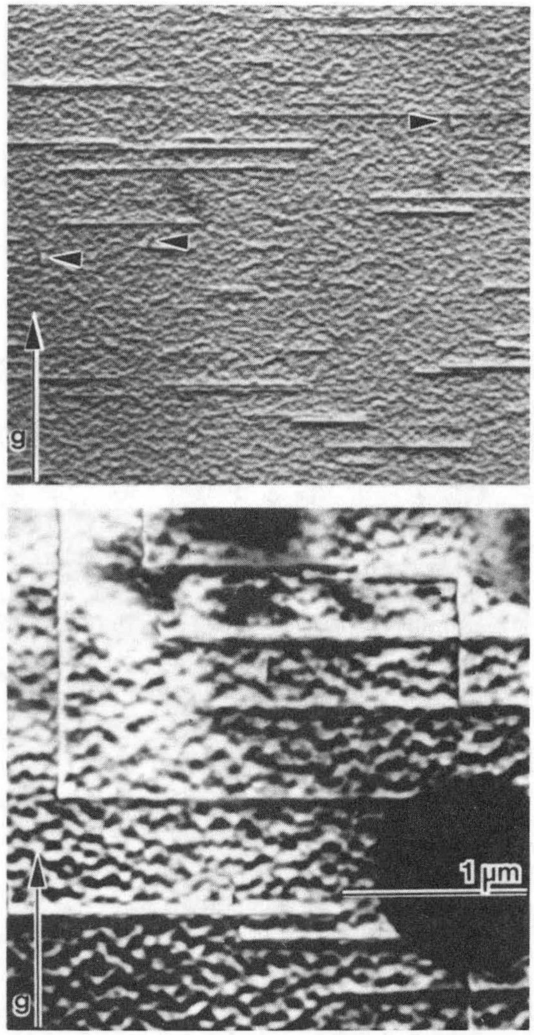

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Figure 1

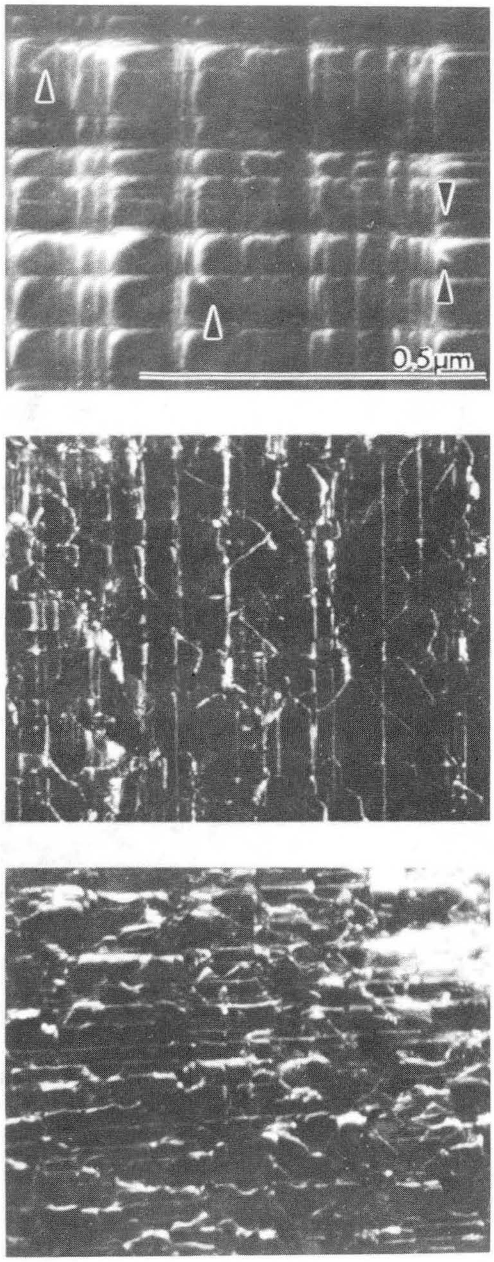

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Figure 2

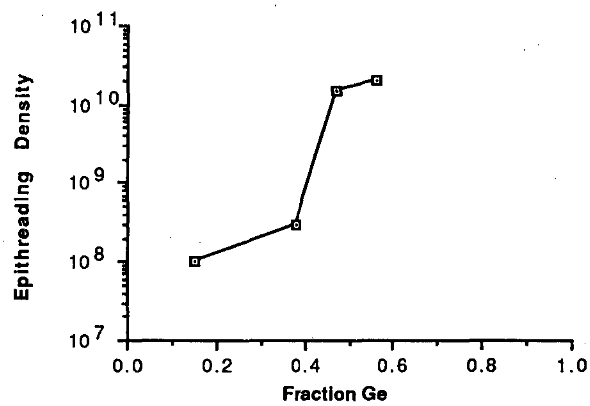

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Figure 3

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