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DISLOCATION BEHAVIOUR IN Ge_xSi_{1-x} EPILAYERS ON (001)Si

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ABSTRACT

We have observed that the nature of misfit dislocations introduced near the critical thickness in Ge_xSi_{1-x} alloys on (001)Si changes markedly in the region $0.4 \leq x \leq 0.5$. At or below the lower end of this compositional range, the observed microstructure is comprised almost entirely of 60° type dislocations, while at the high end, the dislocation structure is almost entirely Lomer edge type. Concurrent with this change, the dislocation density at the top of the epilayer varies by a factor of about 60X. Similarly, several other observables (e.g. dislocation length and spacing) also change appreciably.

Part of the reason for the morphological variation seems to be a change in the source for dislocation introduction, in conjunction with a change in glide behaviour of dislocations as a function of film thickness. Evidence will be presented that indicates strain, as well as thickness, has a critical value for some dislocation introduction mechanisms, and that these together determine the resulting microstructure.

Furthermore, it appears unlikely that the edge-type Lomer dislocations which appear at about x = 0.5 are either introduced directly, by climb, or grown in, as in the three-dimensional island growth and coalescence which occurs when x approaches unity. Instead, a two-step mechanism involving glissile dislocations is proposed and discussed.

INTRODUCTION

It is well known [1] that as misfit strain increases, critical thickness, h_c (the point of initial misfit dislocation introduction) decreases. Recent work [2] has shown that for sufficiently small critical thicknesses (associated with strains on the order of 0.015 or greater), the glissile misfit dislocations introduced can be glide-stopped by the repulsive forces exerted by perpendicular-lying dislocations. This must cause either (i) a slowing of misfit strain relaxation until the epilayer thickness grows beyond the point where pinning is effective, or (ii) introduction of new dislocations, to continue the strain relaxation process.

It has been shown that at sufficiently low strains, the nucleation energy to introduce new dislocations at the surface is high [3,4], and that all dislocations may be accounted for by existing internal sources, such as pre-existing threading dislocations [5] or growth defects [3,4]. However, at higher mismatches (above some critical strain), it has been shown that surface nucleation may be allowed, possibly assisted by alloy distribution microvariations and changes in the dislocation core energy [6]. This critical strain level was estimated to be of the order 0.02, i.e. about the same as at the inception of glissile dislocation pinning.

It is probably not coincidental, as we shall show, that the interfacial misfit morphology changes near this strain level from 60° type to Lomer edge type [7], and that the epithreading density (density of dislocations threading through the epilayer) suddenly increases.

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 $x = 0.56$.

RESULTS AND DISCUSSION

Similar to previously reported work [7], the interfacial dislocation morphology was seen to change from essentially all 60° type dislocations, with lengths of tens of microns (at $x \leq 0.38$) to essentially all Lomer edge type, with submicron average lengths (at $x \geq 0.56$). These microstructures are illustrated in Figure 1. This figure illustrates typical two-beam images of (a) low mismatch, (b) transition, and (c) high-mismatch microstructures very near critical thickness. In each case the dislocations lay in the two interfacial <110> directions, and had full lattice ($^1/_2$ <110> type) Burgers vectors. Materials of lesser thicknesses in each case showed no dislocations whatsoever in TEM, indicating good two-dimensional growth (and the limit of onset of h_c).

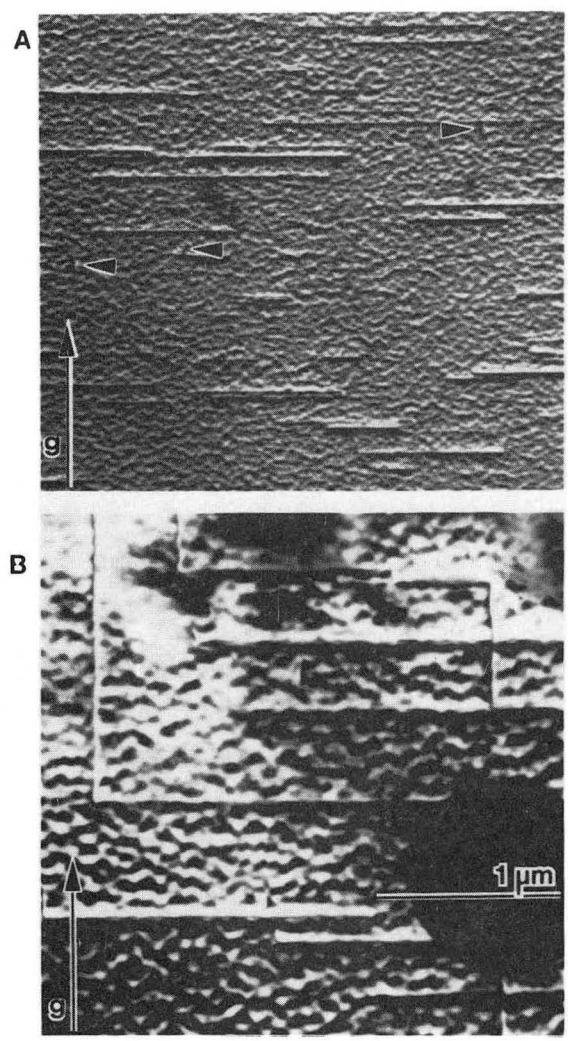

Figure 1.
Typical (a) high misfit and (b) intermediatemisfit microstructures. Imaging condition allows visibility of both edge and 60° dislocation lines lying horizontally, but only 60° type lines lying vertically. The intermediate structure shows several long 60° lines, while the high misfit has only a few, and those very short (arrowed). XBB 890-8783

Within this range of strains ($0.37 \leq x \leq 0.56$), the epithreading morphologies in well-relaxed layers also change appreciably, as seen in Figure 2. For the purpose of clearly showing the epithreading dislocations, the micrographs were taken from thick (85nm) layers, and in weak-beam condition. Figure 3 shows a plot of epithreading density versus Ge content, illustrating the sharp jump in the region near $x = 0.40$ (strain of 0.017), in comparison to the slow rise through the rest of the range.

All these changes in microstructure do not occur immediately and simultaneously, however. As seen in Figure 1(b), there is a range where a transition microstructure exists. The transition microstructure (at $x = 0.47$) is comprised of a mixture of edge and 60° dislocations. The line lengths are greater than those seen in high mismatch materials, but far shorter than seen in low mismatch. Similarly, the epithreading density (related to the number of interfacial dislocation endings) can be seen to have risen substantially. This implies that glide dislocation blockage has already begun to have an effect at this level, and can be seen clearly by the high density of epithreading dislocations in the 18nm, $x = 0.47$ layer (fig.2(b)).

Figure 2.
Weak beam dark field {220} images of (a) low, (b) intermediate, and (c) high misfit microstructures, illustrating heavy epithreading density (easily distinguished by non-Cartesian directions) in the latter two.
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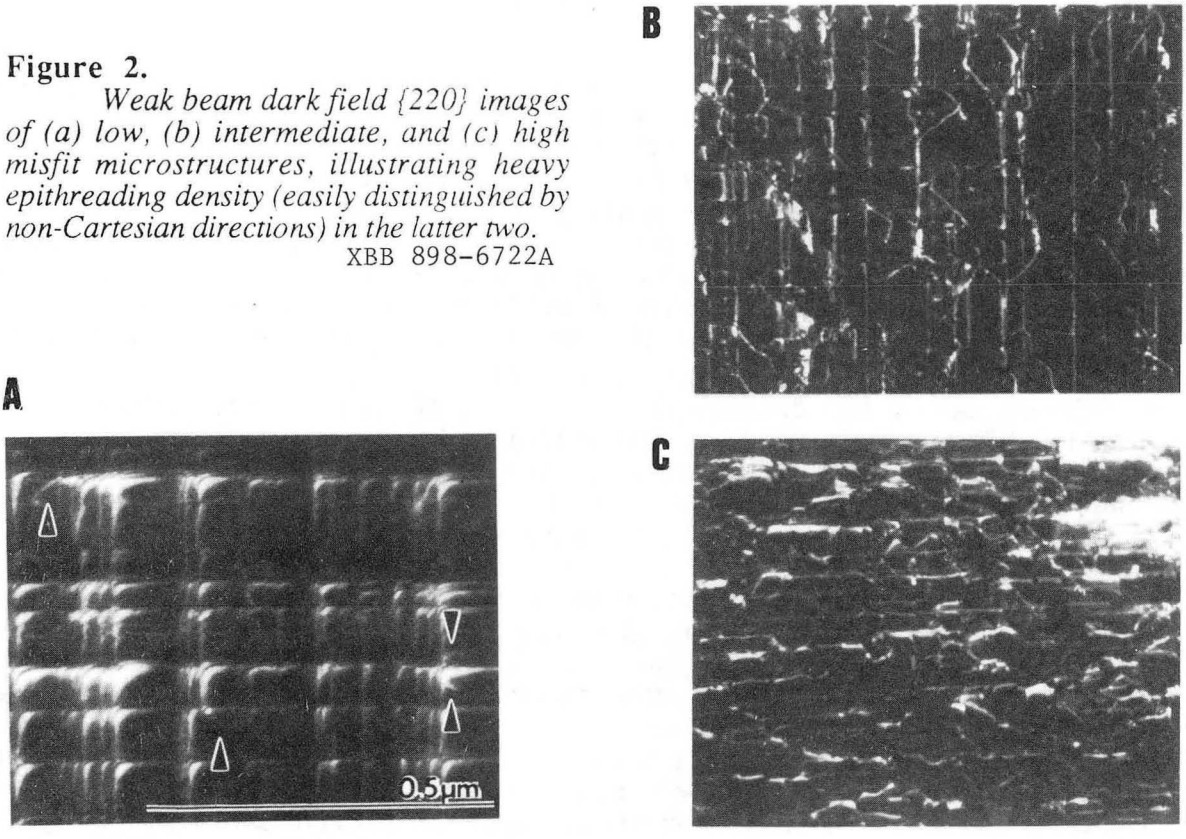

Second, the dislocations in the transition layer could be imaged over their entire length, but none were observed to be associated with any visible defect. This implies that the dislocations have appeared via surface nucleation (since surface nucleation is so much less energetic than internal). If so, the critical bulk mismatch for surface nucleation can be approximated as ≤ 0.019. (This critical strain, however, must be viewed in light of the Hull and Bean model [6], wherein local strains may be higher than those of the overall bulk mismatch.)

A third point is the coexistence of 60° and edge dislocations in the transition material. Our observation was that about 1/3 of the dislocations were 60° type, the remainder edge type. It seems that the nucleation mechanism is either approximately equally likely to produce 60° or edge dislocations, or that one mechanism (for production of 60° dislocations) begins operating but is quickly overrun by a second (edge producing) mechanism. We suggest the latter case, in slight modification, applies, and that the edge dislocations observed in two dimensional growth have 60° type dislocations as precursors.

The mechanism we suggest is essentially one of strain-induced formation of pairs of 60° dislocations of complementary type, i.e. which can combine to form a Lomer edge

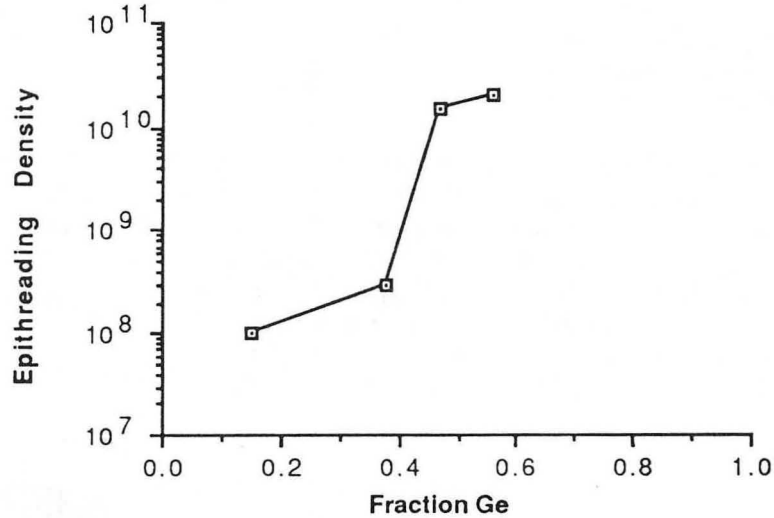

Figure 3.
Plot of epithreading density against Ge content for layers well above critical thickness. A rapid rise is seen near x = 0.40.

dislocation. At the onset of surface nucleation, glissile 60° dislocations begin to appear, expanding towards, then lengthening at, the heterointerface. When the local stress is high enough, the complementary dislocation may be nucleated at the surface as well, gliding down to the heterointerface to combine with the initial 60° dislocation and form a Lomer lock. Simple Peach-Koehler calculations (assuming long, straight dislocations and taking epilayer stress into account) show that an initial 60° dislocation will stabalise the epilayer against further introduction of parallel dislocations in the near region, with the exception of the complementary 60° dislocation. This effect would be greater in higher mismatch materials, as the closer proximity of the initial dislocation to the surface (at smaller h_c) could more strongly help induce nucleation at the surface of the complementary dislocation.

Having formed both dislocations (initial 60° misfit at the heterointerface, newly nucleated half-loop at the surface), the complementary dislocation would glide toward the heterointerface, and the initial dislocation could move slightly to the line of intersection of the two differently inclined glide planes (since exact matching at the interface is unlikely), the Lomer edge being formed at this line when the dislocations meet and combine. This is illustrated schematically in Figure 4.

That Lomer dislocations are seen less in transition microstructures may be due to the point above (greater distance from the heterointerface, resulting in less influence upon subsequent nucleation events), and also to the fact that, in transition microstructures, introduction of a single dislocation releases proportionally more of the initial misfit strain, leaving less potent sites for further nucleation.

Another possibility, less likely but not negligible (in consideration, e.g., of the diamond defect), is that nucleation of the complementary dislocation could occur directly upon the initial 60° dislocation, the first dislocation serving as the nucleation site. Again, as above, the second nucleation event is more likely as strain increases, leaving a gap in which the first event only may occur.

It is also possible that initially perpendicular 60° complementary dislocations may meet, with one turning 90° to react and form an edge dislocation, as suggested by Dodson and Hull [8], but a distinct minority of dislocation intersections (in fact almost none) show the morphology which might be expected to be characteristic of this reaction, namely two 60° dislocations and an edge dislocation all emanating from a common intersection point. Climb of edge dislocations is also possible, especially in consideration of new measurements of diffusion in Si-Ge alloys [9[and the fact that the dislocation line itself would provide a fast diffusion path. However, the calculated nucleation energy for edge dislocations has been shown to be appreciably higher than that of 60° dislocations [6]; the combined greater nucleation rate and mobility of 60° dislocations should swamp the direct entry of edge dislocations, so 60° dislocation nucleation could be expected to dominate the kinetics of morphological development.

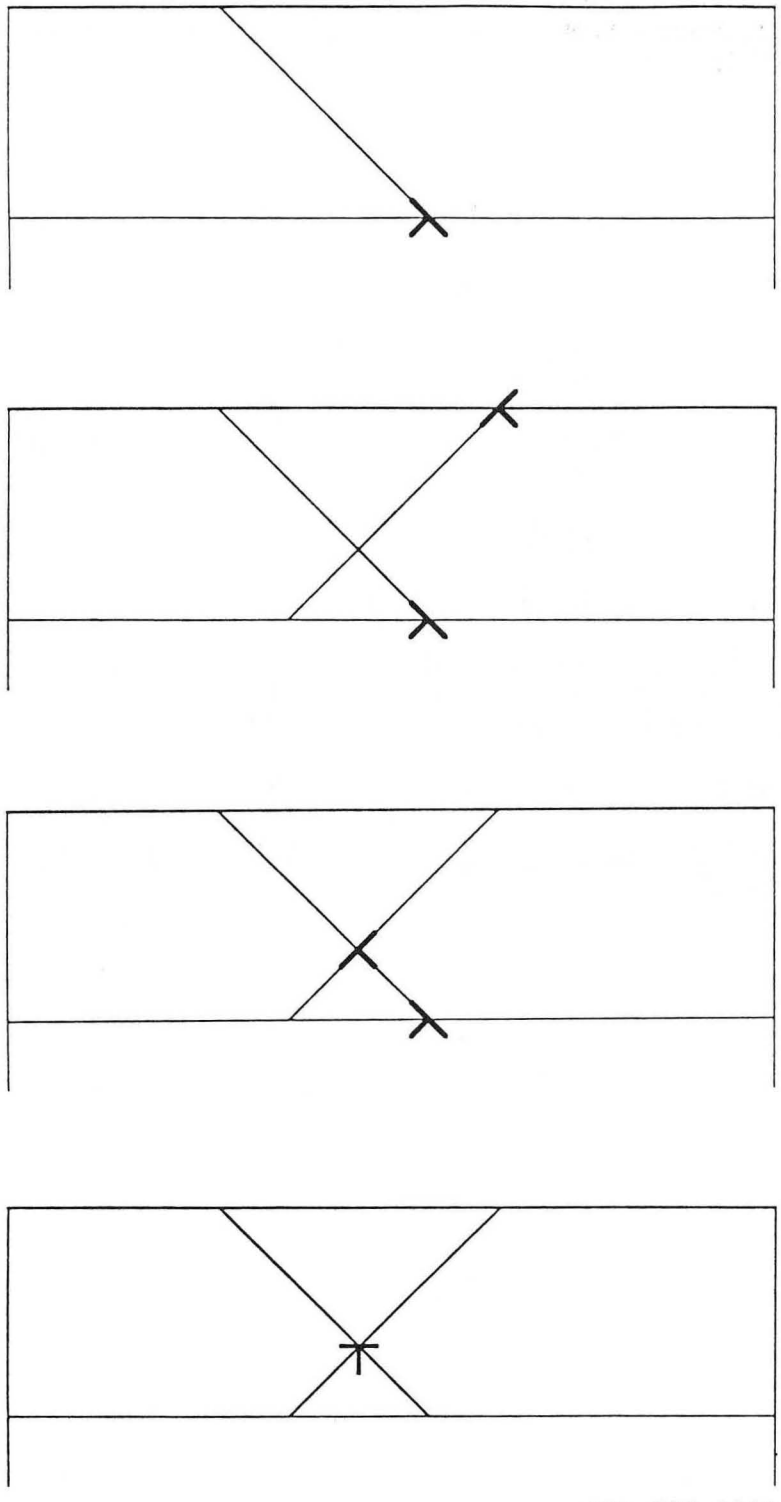

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Figure 4.

Stages of proposed edge dislocation formation mechanism: (i) formation of initial 60° surface-nucleated dislocation, which glides to heterointerface, (ii) initiation of nucleation of comlementary dislocation, also at the surace, (iii) glide of complementary dislocation toward heterointerface, (iv) slight glide of initial dislocation to meet complementary dislocation, reacting with complementary dislocation, resulting in Lomer lock.

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REFERENCES

1. J.W. Matthews, S. Mader, and T.B. Light, J. Appl. Phys., 41, 3800 (1970)

2. R. Hull and J.C. Bean, Appl. Phys. Lett., 54(10), 925 (1989)

3. D.J. Eaglesham, D.M. Maher, E.P. Kvam, C.J. Humphreys, and J.C. Bean, **Phys. Rev. Lett.**, 62(2), 187 (1989)

4. D.J. Eaglesham, E.P. Kvam, D.M. Maher, C.J. Humphreys, and J.C. Bean, **Phil. Mag. A**, 59(5), 1059 (1989)

5. E.A. Fitzgerald, **J. Vac. Sci. Tech.**, B7(4), 782 (1989)

6. R. Hull and J.C. Bean, **J. Vac. Sci. Tech.**, A7(4), 2580 (1989)

7. E.P. Kvam, D.J. Eaglesham, D.M. Maher, C.J. Humphreys, J.C. Bean, G.S. Green, and B.K. Tanner, in Defects in Electronic Materials, edited by M. Stavola, S.J. Pearton, and G. Davies (Mater. Res. Soc. Proc. 104, Pittsburgh, PA 1988) p. 623

8. B.L. Dodson, R. Hull, and J.C. Bean to be submitted (**Appl. Phys. Lett.**)

9. S.J. Chang, K.L.Wang, R.C. Bowman, Jr., and P.M. Adams, **Appl. Phys. Lett.**, 54(13), 1253 (1989)

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