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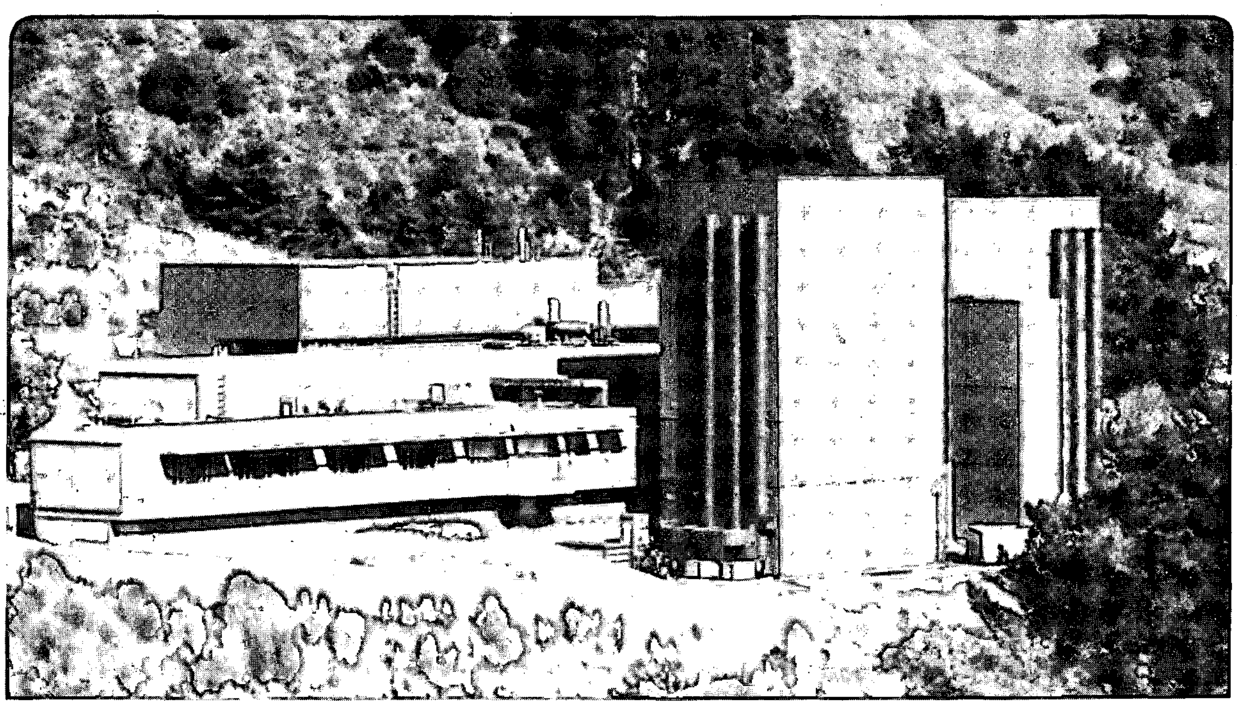

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ABSTRACT

A change in microstructure, including dislocation Burgers vector, length, and behaviour has been observed to occur when the epilayer mismatch is varied in Ge_xSi_{1-x} layers grown on (100) Si. At low mismatches (<1.5%), there is an orthogonal array of very long 60° misfit dislocations. At higher mismatches (>2.3%) there is an orthogonal array of short edge dislocations. At intermediate mismatches (1.5 to 2.3%) there is a mixture of 60° and edge dislocations. The nature of the microstructure has a pronounced effect on the density of threading dislocations in the epilayer, which increase by a factor of ~60X through a relatively small range of mismatch (1.7 to 2.1%, corresponding to x ranging from 0.4 to 0.5). These morphologies are discussed in the light of recent work on the sources of misfit dislocations. While mechanisms for the introduction and propagation of dislocations at low mismatch have recently been observed and explained, the high misfit case is clearly very different, i.e. surface nucleation seems to be likely in the latter case as opposed to operation of an internal source in the former. A mechanism for edge dislocation formation is proposed.

INTRODUCTION

The initial introduction of misfit dislocations near the critical thickness of strained epilayers of Ge_xSi_{1-x}/Si has been a subject of much recent scrutiny[1-11]. However, low (<1.5%) mismatch heterostructures have been much more intensely studied with respect to dislocation structures than have microstructures of coherent two-dimensionally grown high (>2%) mismatch heterostructures. There are clear and distinct differences between these microstructures[6], with the implication that they may derive from different modes of initial misfit dislocation introduction.

In each case, essentially all dislocations introduced have one of the six possible $^1/_2$<110> Burgers vectors, and lie in the two orthogonal <110> interfacial directions. At low misfits, the dislocations observed have been shown to be almost uniformly of 60° character, very long (from 10 µm to millimetres), and irregularly spaced, often appearing in large clusters. At higher misfits they were seen to be predominantly of pure edge character, mostly of submicron length, and randomly arrayed. Recent work[8,9,10] has shown that the low misfit microstructure may be explained by the operation of an internal source to produce misfit dislocations as well as by source mechanisms extrinsic to the growth process (e.g. oxide particles), while suggesting that at higher mismatch (beyond some critical mismatch, estimated to be several percent) classical surface half-loop nucleation[12-14] may operate. Hull and Bean[10] have recently suggested that random clustering of the Ge alloy may produce regions of locally high stress concentrations, and that the alloying may also reduce the dislocation core energy, both of which would notably reduce the critical nucleus size and energy. However, this is insufficient to explain fully the observed high-mismatch microstructures in that only 60° dislocations, not pure edge, would be produced by simple operation of such a surface nucleation/glide mechanism.

Hull has also shown that one of the reasons for the shortening of dislocations is the thinner films associated with the critical thickness at higher strains[11]. As an epilayer becomes progressively thinner, gliding dislocations are proportionally more sensitive to external forces, such as strain inhomogenieties and back-stresses from other dislocations, and thus will more often encounter impassable obstacles, e.g. they will be stopped by the stress field of perpendicular-lying dislocations. This is clearly one of the reasons why dislocations are shorter, and consequent threading density higher, for materials with smaller critical thicknesses (higher misfits). A further step is necessary if the sessile dislocation ends are not to become glissile again as growth proceeds, though.

Most of the materials discussed here were MBE-grown on unrotated (100) Si substrates. Initial Si buffers of 100nm each were grown at 750, then 550°C, followed by the single-layer alloy growth at 550°C. The growth was in masked bands of 9.5, 18, and 85 nm thicknesses, and the intentional non-rotation allowed a continuous variation in composition across each band from 35% Ge (1.5% mismatch) to 55% Ge (2.3% mismatch) as measured by EDX. Some other materials which were grown on rotated substrates have also been examined, but no differences in microstructure were observed from the unrotated materials. The starting wafers were in all cases nominally dislocation free.

This paper will concentrate on high mismatch and transition region (mixed) microstructures, but a brief note on low-mismatch epitaxy is useful for comparison.

LOW MISFIT

The production of misfit 60° dislocations in the low-mismatch materials examined was shown to be due to an internal faulted loop (the diamond defect) operating as a modified Frank-Read source[7,8]. (In other SiGe alloy epilayers, instances have been found of dislocation generation from strain centres such as oxide inclusions[4,5].) The resultant microstructure was very inhomogeneous, with dislocations highly clustered (in comparison to the random spacing of the array of edge dislocations at high mismatch). Further, despite extensive dislocation interaction, very little of the dislocation content at low mismatch was edge type. A typical low-mismatch epilayer microstructure is shown in Figure 1.

In all materials examined, the interfacial dislocations had <110> line vectors. For 60° type dislocations in thick, low-mismatch epilayers, the Burgers vector was seen to be of inclined 1/2<110> type, leaving the dislocation glissile on a single {111}; such dislocations typically ended in screw-type epithreading dislocations connecting the interfacial misfit dislocation to the growth surface. Such screw dislocations would be also glissile on a second {111}, allowing, e.g. L-shaped morphologies[1-3,9] and reactions such as the Hagen-Strunk mechanism[2,3]. Detailed analysis shows that those few edge dislocation segments found in low-mismatch materials were, in fact, obviously the result of combination of two vicinal 60°-type dislocations. Almost no stacking faults (except for the diamond defect itself) were observed in low-mismatch epilayer materials.

HIGH MISFIT

As mismatch increased through about 2%, the microstructure was seen to change from 60°-type to edge-type misfit dislocations. This can be seen in Figure 2, which shows a series of micrographs using **g** = {220}, in which all 60° dislocations, but only one of the orthogonal sets of edge dislocations (those lying perpendicular to **g**), are in contrast, and **g** = {400}, in which all misfit dislocations (edge or 60° type) are in contrast. The micrographs corresponds to an area only slightly above the critical thickness. We observed in these materials a small proportion (numerically 1 to 2%) of very short 60°-type dislocations.

In the thinnest epilayers (9.5 nm) there is no direct evidence of what type of threading dislocations connect the interfacial dislocation to the growth surface; the contrast from the very short epithreading dislocations is masked both by contrast from the ends of the interfacial dislocations and by background contrast which, for reasons that are not definitely determined, was much more severe for high misfit structures than for low misfit structures (compare figures 1 and 2). For slightly thicker epilayers these effects are diminished but definite identification of threading dislocations could only rarely be made.

In thick (85 nm) high-mismatch epilayers, however, the observed threading dislocations are of the inclined $^1/_2$<110> **b** type normally associated with interfacial 60° dislocations. The threading dislocation density in thick films was seen to change rapidly with only small changes in mismatch approaching the 45% Ge (1.9% mismatch) level. Figure 3 illustrates this, with a threading density in the epilayer of (a) 4 x 10^8 cm^{-2} in 35% Ge (1.5% mismatch) material, (b) 1.5 x 10^{10} cm^{-2} at 45% Ge, and (c) 3 x 10^{10} cm^{-2} at 55% Ge.

INTERMEDIATE MISFIT

The transition region at about 45% Ge (1.9% mismatch) carries considerable information, and may help to infer the dislocation introduction source for the high misfit case. In this region the dislocation morphology was neither predominantly edge nor 60° type, but a mixture of the two. Figure 4 illustrates this transition morphology. The transition regime can be seen to have elements of both of the other regimes.For instance, i) dislocations were sometimes seen to turn a 90° angle by themselves (not by intersecting other dislocations, but in 'clear' material), ii) react with each other, and iii) some dislocations were of very long lengths, all of which are typical of low-misfit materials, while iv) dislocations were unassociated with visible internal defects, v) often ended at their perpendicular intersection with another dislocation, and vi) isolated submicron 60°-type half-loops were observable, all typical of high-mismatch materials.

Points iv and vi are indicative of surface nucleation, with a larger density of such small 60°-type half-loops seen in intermediate mismatch material than in higher or lower mismatched epilayers. These half-loops tended to be larger than those seen in the highest mismatched materials, possibly because of their larger critical nucleation size, possibly for other reasons (e.g. being more stable against reactions to create edge dislocations than in more highly strained materials). At higher misfit stress, the excess stress would presumably allow the dislocation to grow more quickly[14,15] and/or react to form an edge dislocation, while at lower stresses surface nucleation to produce such half-loops could not occur.

In transition morphology cases (with mismatch of about 1.9%), there are some indications of edge dislocations ending at pairs of epithreading dislocations lying on inclined (presumably the {111} glide) planes rather than at single edge dislocations lying in the {110} climb plane (i.e. along or above the edge dislocation), as expected from a climb mechanism. This is illustrated in Figure 5 at **A**. Also, instances are readily found of reactions between edge and 60° type dislocations, such as a 60° dislocation segment beginning at a perpendicular edge dislocation terminus, in an L-shaped structure, as seen in Figure 5 at **B**. These points suggest that at least some of the edge dislocations seen in high mismatch materials end at non-edge (inclined Burgers vector) epithreading dislocations, implying that they initially appeared as a result of some interaction between two 60° dislocations and not by climb alone, which is very significant in terms of the implicated nucleation mechanism.

NUCLEATION OF EDGE DISLOCATIONS

Although diamond defects were observed in the high mismatch materials, they were not associated with any change in density of the edge dislocations, and visibly not responsible for dislocation production; in fact, the length of the $^1/_6$<411> dislocation within the epilayer, and thus operated upon by the stress was not enough for the proposed Frank-Read mechanism to reach critical radius. Essentially no other defects were observed. Also note that the interfacial dislocations were evenly distributed at densities of up to 10^9 cm^{-2}, c.f. diamond defect areal densities of 10^4-10^5 cm^{-2}. Further, the (edge) dislocations did not extend, except by chance, into the region near diamond defects, opposite to the low-mismatch case where (very near critical thickness) every dislocation whose entire length could be observed was associated with a diamond defect. Since interfacial or epilayer defects other than interfacial waviness were not observed (oxide particles, for instance, were only at the original buffer/substrate interface), a different and intrinsic dislocation source must be responsible.

The model for edge dislocation production we propose is a two step process: first an initial 60° character dislocation is produced as a half-loop at the surface, which then reacts to form an edge dislocation. The initial nucleation is induced by the large misfit stress, probably acting at a stress concentrating inhomogeneity such as a random high-Ge cluster[11] and/or a surface multiple step (note that this step need not be stable, only transient). This half-loop could propagate on a {111} down to the heterointerface and lengthen by glide of the end epithreading segments, as originally proposed by Matthews, et al.[12,13]. As noted, small 60° type half-loops have been observed (amongst the predominantly edge-type network) in the highest misfit materials, and many larger ones in transition morphology materials.

These 60° dislocations were, in high mismatch materials, invariably quite short, none seen being longer than 300 nm (c.f. supermicron lengths in transition and low mismatch materials), and were not glide-stopped by perpendicular dislocations as often as the longer edge dislocations. These short 60° dislocations are visible in Fig. 2.

The second (and less documented) step in the process is the transformation of the initial 60° dislocation into an edge dislocation. Nucleation of a second 60° dislocation with opposite screw and surface-normal edge components, required to transform the initial dislocation into a perfect edge misfit dislocation (hereafter referred to as the complementary 60° type dislocation), would be stabilised by the existence of the initial 60° dislocation, to the detriment (in part by reduction of misfit strain) of other nucleation events. The screw and surface-normal edge components of a 60° type dislocation, of course, do nothing to take up misfit stresses, and represent excess energy. The longer the initial dislocation has become, the greater will be the strain energy stabalisation of the complementary dislocation. This is perhaps the cause of 60° dislocations happily existing in such great size and number in transition materials and not higher mismatch materials, where the excess misfit strain (required to nucleate and propagate the complementary dislocation) has not been relatively as effectively relieved by the initial dislocation introduction.

The complementary 60° dislocation, propagating on a {111} toward the heterointerface (if not on exactly the "right" plane, i.e. to meet the initial dislocation at its interfacial position) would induce a slight movement of the initial dislocation so as to combine to form a pure edge dislocation,with no screw or interface-normal edge components along the line of intersection of the two slip planes. It is easily seen that the two 60° dislocations would necessarily have different glide planes. This mechanism is shown schematically in Figure 6, and has been described as formation of a super-sessile dislocation. Such a cooperative movement of two 60° dislocations was seen by Fitzgerald, et al.[17,18] to result in formation of edge dislocations slightly into the buffer layer in InGaAs/GaAs heterostructures. It should also be noted that even if such a mechanism resulted in formation of an edge dislocation slightly away from the interface, short-range climb could move it a few atomic spacings toward the heterointerfacial plane. By contrast, climb would be far more difficult very near the surface because of the image force opposing downward movement of the edge dislocation.

A second, related possibility for edge dislocation formation is the nucleation of the complementary dislocation directly upon the initial 60° dislocation at the interface; that is, the initial 60° dislocation breaking into two perfect 1/2<110> type dislocations, one the complementary 60° dislocation, the other the edge type interfacial misfit dislocation. This would require the generation of a second 60° dislocation in the opposite operation to that described by Fitzgerald, et al., and would be fairly energetic. It is not, however, entirely unlikely, since just such a 'partialisation' of existing dislocations is the mechanism by which the diamond defect produces 60° dislocations in low misfit materials[7,8].

It is interesting to note that a mechanism for edge dislocation motion was proposed for P diffusion-induced dislocation movement in (001) Si[19]. In that work, a network of edge dislocations was seen to break into 60° type dislocations, which moved downward on the inclined {111} glide planes, recombining at deeper levels to keep up with the diffusional front. The mechanism is very similar to tha second possibility mentioned above, in that on lattice dislocation splits into a pair of lattice dislocations at the strain interface.

For cooperative nucleation to occur, several criteria must be met. Obviously, surface (or internal) nucleation must be possible, i.e. there must be some minimum "critical" stress in the epilayer. This critical stress must remain high enough for the production of <u>both</u> dislocations, which is why such a process would not be expected (and is not seen) in lower misfit materials. The strain relief of the first dislocation must not too greatly reduce the mismatch strain energy (the probable reason for 60° stabilisation in the intermediate mismatch transition epilayers), and, if double surface nucleation is involved, the initial dislocation must be close enough to the surface to strongly induce nucleation of the complementary dislocation. The complementary dislocation, near the depth at which it has become a stable nucleus, (i) must not have moved away from the initial dislocation, or the stabilising effect of the first will diminish before the critical depth for the second dislocation is reached, and (ii) the two dislocations must initially be close enough for the strain-field stabilisation to be strong. These affect the possible lateral positioning of the second dislocation such that final edge dislocation should be between the midplane of the epilayer and the heterointerface. It should be noted that sub-interface combination to edge type of independently derived 60° dislocations, such as observed by Fitzgerald, et al., was not definitely observed here, though could not be ruled out.

Either reaction would produce an interfacial edge dislocation attached to the surface by four epithreading dislocations, two at each end. If the epithreading segments at the terminus of the edge do not combine, they will both be glissile dislocations (of inclined Burgers vector type), and the edge dislocation can lengthen by glide; if they do combine, the epithreading dislocation will be sessile, and unable to lengthen except by climb. Paired epithreading segments have been observed in this work (Fig. 5), and Hull has observed some slip in high-mismatch materials[20], but such observations are not necessarily representative of all, or even the majority, of cases. Dislocations having locked into a sessile configuration would explain the need for introduction of further dislocations to relieve mismatch, with the consequence that many more epithreading dislocations are generated. If the locking mechanism is time dependent (e.g. thermally activated), it would account for the observation that most edge dislocations end at perpendicular intersections, where they had stalled awaiting further growth. Alternatively, if epithreading dislocations continue to grow other than on their slip planes, or to develop Cottrell atmospheres which severely inhibited glide (unlikely for screw dislocations), this also would cause them to become sessile.

SUMMARY

The gradual change from 60° to edge type interfacial dislocations with increasing mismatch has been documented, as has the concurrent rapid increase of epithreading dislocation density.

A two-stage process of dislocation introduction is proposed to explain the transition in misfit dislocation morphology which occurs in Si-Ge on Si(001) epilayers, with predominantly 60° type at low mismatch to predominantly edge type above about 45% Ge (~2% bulk mismatch). This transition seems to be associated with a point of critical strain for surface nucleation of 60° misfit dislocations, followed by a cooperatively-induced nucleation and propagation of a second, complementary dislocation to form a pure sessile edge dislocation. The subsequent locking up of dislocations in a sessile state results in a rapid increase in the number of threading dislocations in the epilayer.

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Figure Captions

Figure 1

Low-mismatch misfit dislocation microstructure ($x = 0.18$, 270 nm thick epilayer) near critical thickness. Microstructure is completely dominated by very long single or tightly clustered 60° dislocations, with (at small relaxations) large areas of 'good' material between. **g** = (022)

Figure 2

High mismatch microstructure near critical thickness ($x = 0.55$, 9.5 nm thick epilayer). In (a) **g** = (022), edge dislocations parallel to the arrowed g direction are out of contrast; dislocations visible in this direction are 60° type. In (b) **g** = (004), all dislocations are visible (N.B. Although **g•b** may be 0 for some of the 60° dislocations, **g•bΛu** contrast still remains for the {400} reflections.)

Figure 3

Weak-beam (**g** = (022)) micrographs of 85 nm thick Ge_xSi_{1-x} epilayer in plan view. The orthogonal dislocation network (horizontal and vertical) is that of the misfit dislocations lying at or near the heterointerface; the others are epithreading dislocation segments lying predominantly along inclined <110> directions, with <100. traces. a) $x \sim 0.35$ b) $x \sim 0.45$ c) $x \sim 0.55$ d) Plot of dislocation density vs. mismatch over the range examined. (N.B. Point for 0.7% mismatch is from 270 nm thick material, similarly grown, for comparison.)

Figure 4

Intermediate msimatch morphlogy, contrast conditions (a) **g** = (022), (b) **g** = (040), (c) **g**= (02-2). Notice the long 60° dislocations which appear (c.f. Figure 2), as well as the complex interactions. Misfit dislocations are not uniformly of a single type, but rather substantially assorted between edge and 60° types.

Figure 5

At [**A**], a V-shaped pair of epithreading segments extend upwards from the one end of end edge-type misfit dislocation, while at [**B**] a 60°-type misfit dislocation extends orthogonally from the end of another edge dislocation. Both effects are indicative of a glide rather than climb source for the origin of some the edge dislocations. Weak beam dark field images, (a) **g** = 022), (b) **g** = (004).

Figure 6

Possible mechanism for edge dislocation formation by combination of a pair of surface-nucleated 60° type dislocations.

(a) Initial 60° dislocation (e.g. 1/2 [101], lying along [-110] and slipping on (-1-11)) is nucleated at the surface and glides to the heterointerface.

(b) Initial dislocation at heterointerface in thin epilayer induces surface nucleation of complementary 60° dislocation (e.g. 1/2 [011] along [-110] and slipping on (111)).

(c) Complementary dislocation slips toward heterointerface under combined influence of epilayer misfit stress and initial dislocation stress.

(d) In the general case, both dislocations need to slip slightly to combine, forming an edge dislocation along line of slip planes' intersection. If the new dislocation line does not lie exactly in the heterointerfacial plane, short-range diffusion may allow it to climb slightly toward the heterointerface.

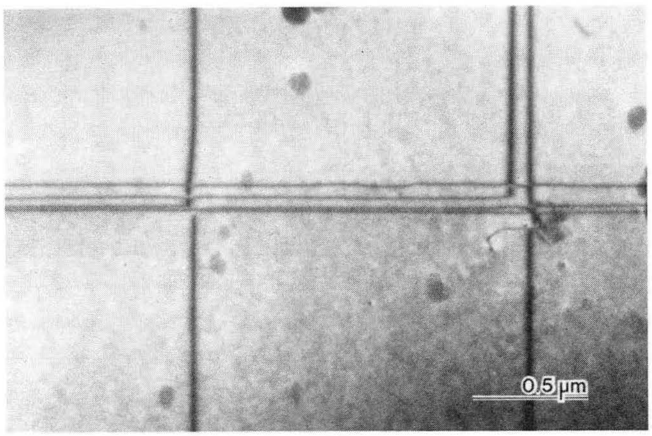

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Figure 1

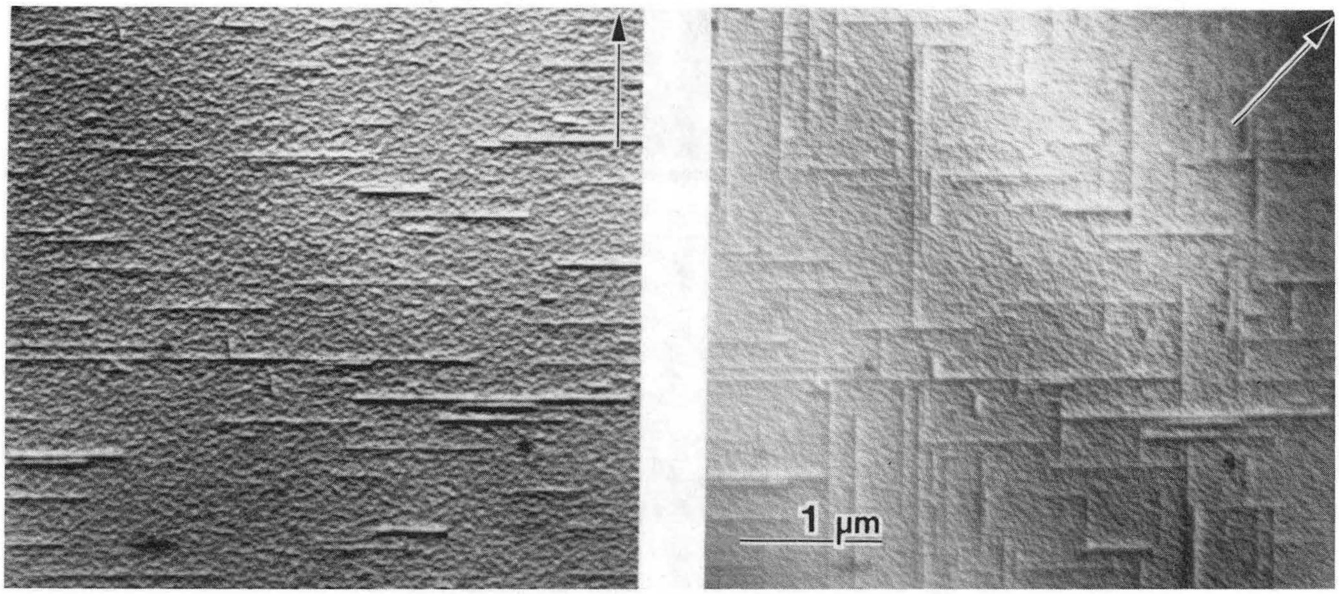

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Figure 2, a & b

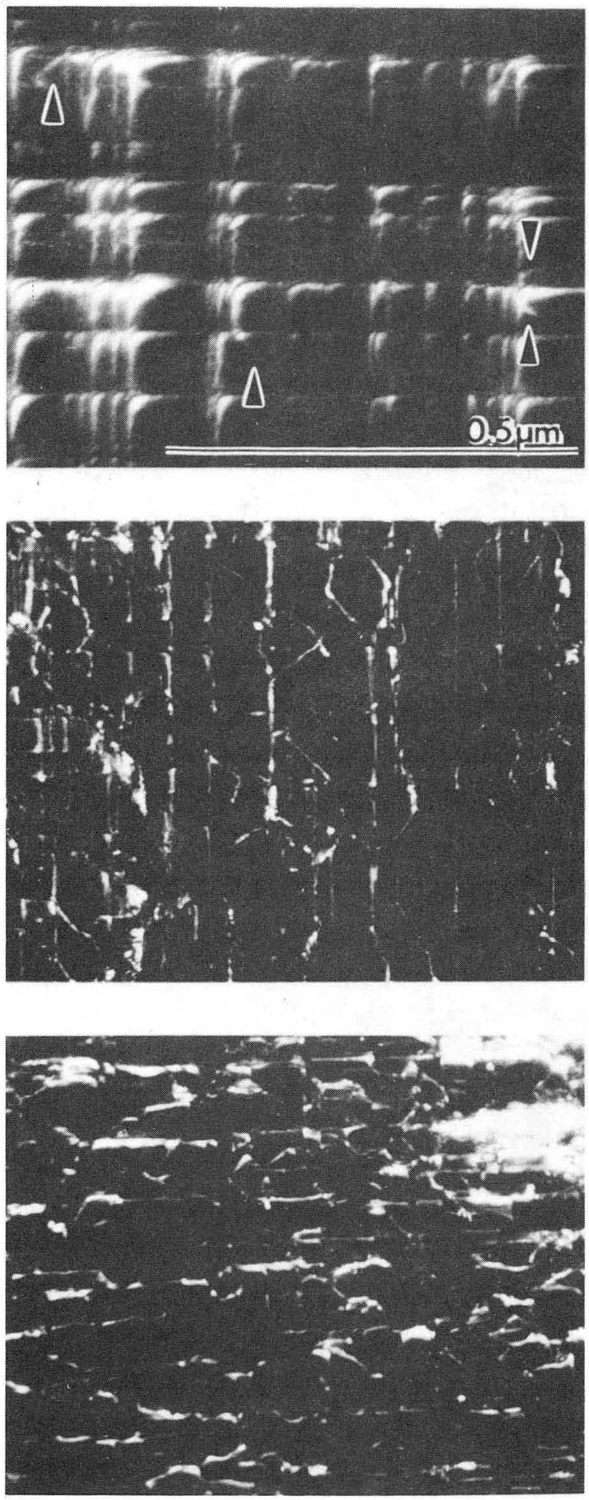

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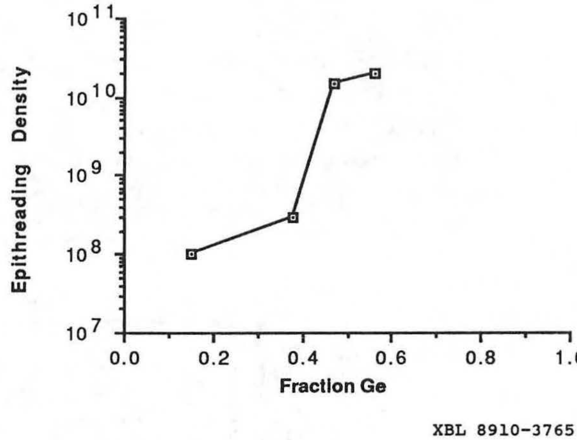
Figure 3 a-c



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10^{10}
10^{9}
10^{8}
10^{7}
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0.2
0.4
0.6
0.8
1.0
Fraction Ge
XBL 8910-3765

Figure 3d

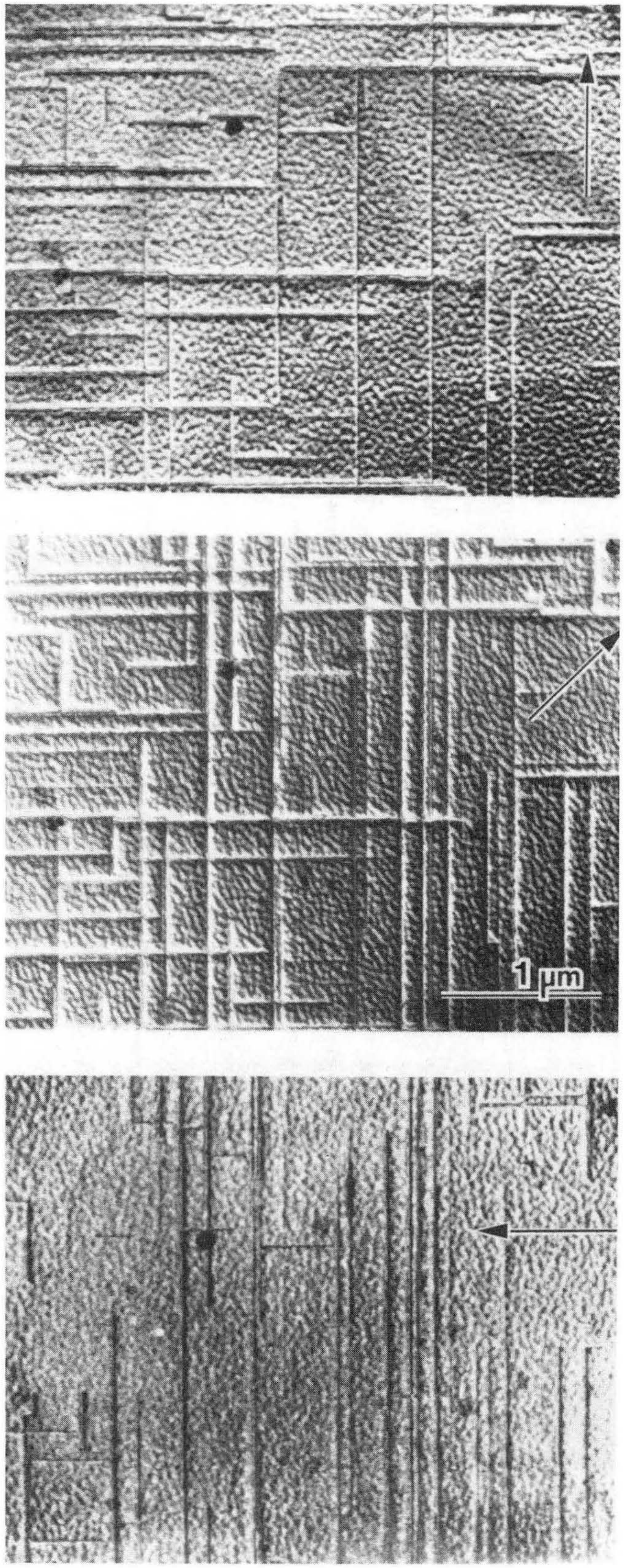

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Figure 4 a-c

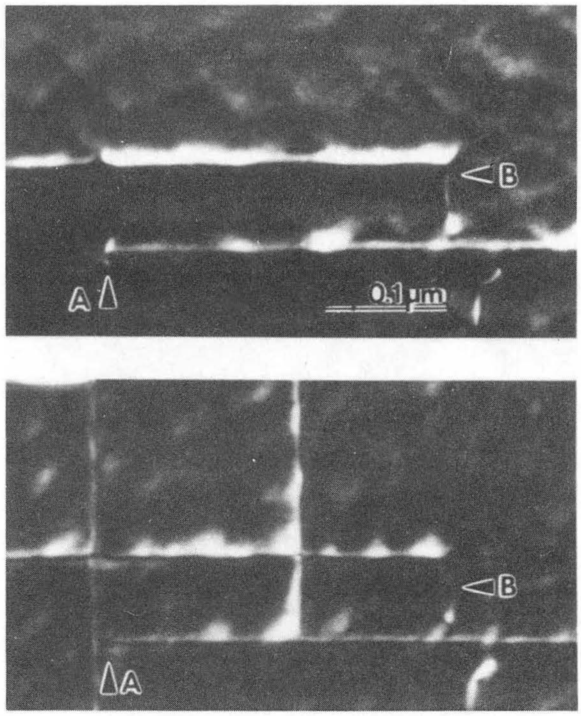

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Figure 5 a & b

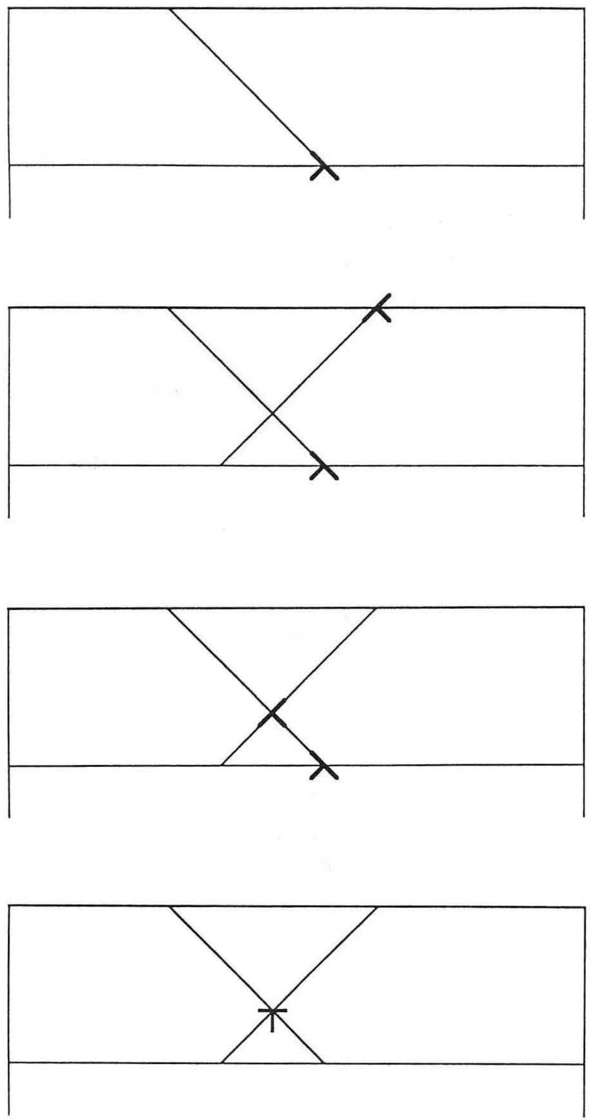

Figure 6 a-d