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DEFECTS IN EPITAXIAL MULTILAYERS

II. DISLOCATION PILE-UPS, THREADING DISLOCATIONS, SLIP LINES AND CRACKS*

J. W. MATTHEWS and A. E. BLAKESLEE

IBM Thomas J. Watson Research Center, Yorktown Heights, New York 10598, U.S.A.

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Multilayers composed of many thin layers of GaAs and $GaAs_{0.5}P_{0.5}$ were grown on GaAs substrates by chemical vapor deposition. They were examined by optical microscopy, electron microscopy and scanning electron microscopy. Slip lines, dislocation pile-ups, threading dislocations, and cracks were found. These defects were made to relieve elastic stresses generated as a result of misfit between the multilayer taken as a whole and its substrate. The roles of dislocation pile-ups and superkinks in the propagation of dislocations through multilayers are discussed.

1. Introduction

In part I[1]) we described multilayers composed of many (60 to 120) thin (75 to 700 Å) single-crystal films of GaAs and $GaAs_{0.5}P_{0.5}$. Also described were dislocations that accommodated part of the misfit between individual GaAs and Ga(As, P) layers. In addition to this obvious interlayer misfit there is another misfit not so generally recognized, namely that between the GaAs substrate and the multilayer considered as a single entity. The purpose of part II is to discuss dislocation pile-ups, slip lines, and microcracks generated as a result of the misfit between the multilayer taken as a whole and its substrate. These defects are of interest partly because of the adverse effects that they would be expected to have on the performance of a "superlattice" device[2]) and partly because investigation of them has revealed a mechanism for the motion of dislocations through composite materials. Dislocations find it difficult to move through composites for two reasons[3,4]). Coherency strains give rise to stresses that aid the motion of a dislocation through one of the materials present but oppose its motion through the other[3]). Also, if the elastic constants of the two materials are different, a stress is needed to move a dislocation out of the soft material into the hard one[4]).

* A summary of this work, as well as of Part I (ref. 1), was presented at the Conference on Vapor Growth and Epitaxy, Jerusalem, May, 1972.

2. Observations

2.1. Dislocation pile-ups

All multilayers contained straight dislocation lines which bore a superficial resemblance to the misfit dislocations described in I. In common with the misfit dislocations of I, the straight dislocations described here had Burgers vectors of type $\frac{1}{2}a\,\langle 110\rangle$ which were inclined at about 45° to the almost (001) specimen plane. Their line directions were approximately parallel to the $\langle 110\rangle$ directions in (001) and they were arranged in arrays on $\{111\}$ slip planes. The two types of dislocation differed in that the Burgers vectors of adjacent dislocations in the arrays considered here were parallel, whereas the Burgers vectors of adjacent dislocations in the arrays of I were antiparallel. Another important difference concerns the dislocation separation. The projected separation of the dislocations discussed here did

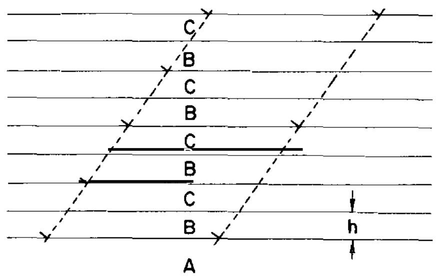

Fig. 1. Arrays of dislocations on $\{111\}$ slip planes. The dislocations are separated by two layers on the left and by four on the right.

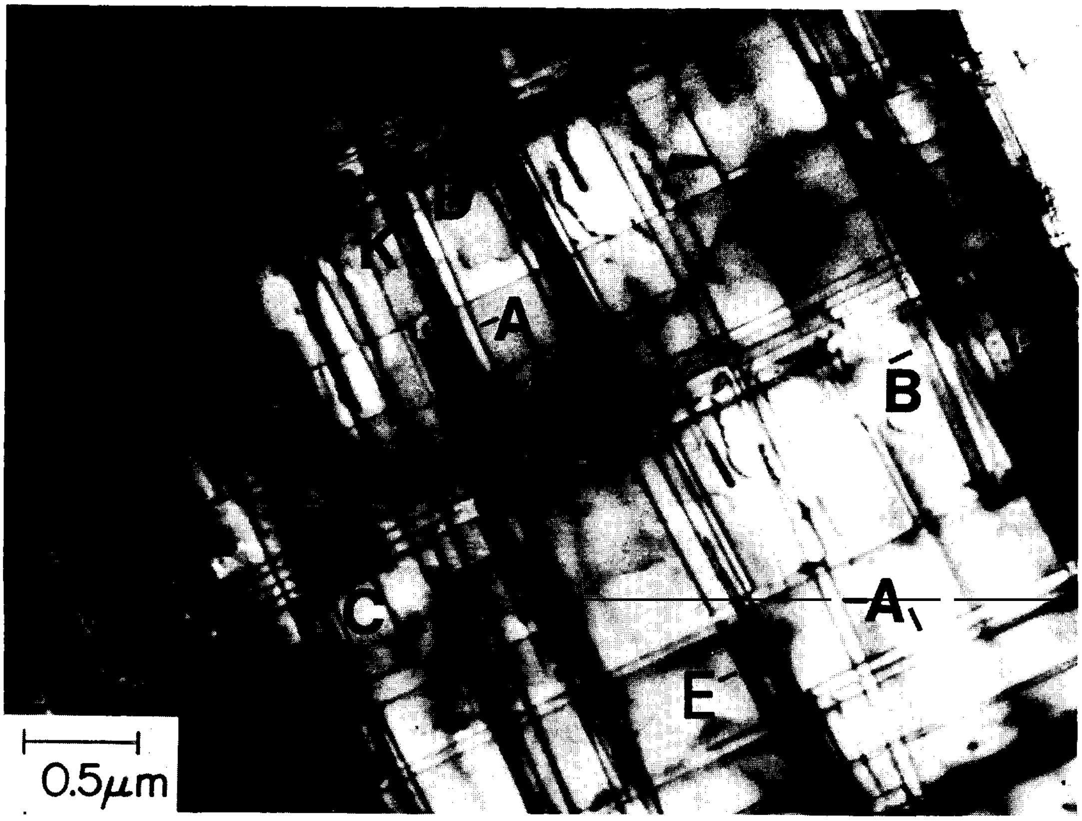

Fig. 2. Micrograph of a multilayer composed of 350 Å layers. The layer plane is approximately perpendicular to the incident electron beam. Dislocations separated by three layers are labelled A and B. The dislocations in the arrays near C and D are separated by two layers. Superkinks are labelled K.

not obey eq. (1) of I but was given by

$$S = 2nh \cot 55°, \qquad (1)$$

where $n = 1, 2, \ldots$, and h is the thickness of individual layers. This result indicates that the dislocations lay on {111} planes and that their lines were separated by even numbers of layers as illustrated in fig. 1. The arrays in fig. 1 are a form of dislocation pile-up. They differ from conventional pile-ups in that S has discrete values[5,6]). This is discussed in section 3.5.

The number of arrays that obeyed either eq. (1) above, or eq. (1) of part I, was very large. Indeed, the presence of dislocations arranged in uniformly spaced arrays was the most striking feature of the samples. However, not all arrays were regular. There were many arrays composed of dislocations on different, and sometimes on non-parallel, {111} slip planes. Examples of these can be seen at E in fig. 2 and in the lower portion of fig. 4.

An example of a pile-up in which the dislocation lines are separated by two 350 Å layers is labelled C in fig. 2. Another more distorted example is labelled D. The dislocations labelled A and B in fig. 2 are examples of the misfit dislocations described in I. They are paired, have antiparallel Burgers vectors, and are separated from one another by three 350 Å layers.

The arrays at C and D in fig. 1 were almost certainly portions of much larger arrays. It is probable that portions of the arrays lay above and below those visible in the micrograph and that they were removed when the sample was thinned.

Evidence that adjacent members of dislocation pile-ups have parallel Burgers vectors is provided by the way in which members of a pile-up change interfaces.

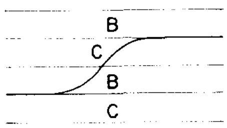

Fig. 3. A superkink.

Fig. 4. Micrograph of a multilayer composed of 350 Å layers. Dislocations two and four layers apart are labelled A and B. K is a superkink that threads two layers.

When the misfit dislocations described in I changed interfaces they bent back on themselves to give a defect similar to a dislocation dipole. When a member of a pile-up changes interfaces it continues along in the same direction as illustrated in fig. 3. The portion of dislocation line that threads the B and C layers in fig. 3 is a superkink. Examples of superkinks in a multilayer composed of 350 Å layers are labelled K in fig. 2. A clearer example is present in fig. 4.

Evidence that kinks like those in figs. 2 and 4 do thread two layers has been obtained by stereo-electron microscopy. A stereopair of a superkink in a multilayer made up of 500 Å layers is present in fig. 5. The inclination of the kink to the interface plane is demonstrated by the large change in projected width of the kink from one image to the other.

The "S" bend in the kink in fig. 5 results from the bowing of portions of its line to make short lengths of misfit dislocation as described in I. The presence of positively and negatively curved parts results from the fact that the kink threads two layers and the sign of the stress in one layer is opposite to that in the other.

The existence of pile-ups in multilayers indicates that there are dislocation sources in the multilayers. These emit a succession of dislocations on the same or on nearby {111} slip planes. A succession of dislocations made by a source is illustrated in fig. 6. This figure is drawn on the assumption that the thickness of the B

(a) (b)

Fig. 5. Stereo-electron images of a superkink in a multilayer composed of 500 Å layers.

and C layers lies below h_{crit}, the value of h at which it becomes energetically favorable for misfit dislocations to be made.

Fig. 6 does not show the source itself. This is because its geometry is not known. We have seen several defects that could have been sources but we have not yet found any that we could confidently interpret as sources. However, trains of dislocations similar to those we

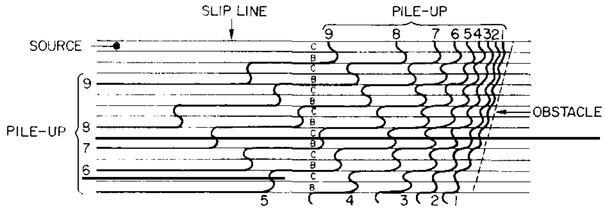

Fig. 6. Dislocations emitted by a source in the upper surface of a multilayer. It has been assumed that layer thickness lies below h_{crit} and that the lateral motion of the dislocations is prevented by an obstacle. The diagram is not intended to be quantitative; the dislocation spacings in the pile-up on the right do not follow the predictions of Eshelby et al.[6]).

have observed could have been made by the Frank–Read mechanism or by sources similar to those that Mader and Blakeslee[7]) have found in Ga(As,P) thin films.

2.2. Slip lines

Optical micrographs of the upper surfaces of multilayers revealed slip lines. Examples found on the surface of the specimen made up of 700 Å layers are seen in fig. 7. The height of surface steps associated with slip lines has been measured on transmission electron micrographs of surface replicas. The measurements were approximate but showed that steps as high as 300 Å were present. These steps could not have resulted from the processes discussed in I. The processes in I give rise to monolayer steps but cannot be responsible for steps many layers in height. High steps are made when sources like the one in fig. 6 emit a hundred or so dislocations.

Fig. 7 shows that the multilayer surface is divided into patches of two kinds. In one, the slip lines lie along [110], and in the other they lie along [1$\bar{1}$0]. The existence of these patches is evidence that slip on planes that intersect (001) along [110] is prevented by slip on planes that intersect (001) along [1$\bar{1}$0]. Electron micrographs of the border between two patches would be expected to reveal threading dislocations impacted against dislocations on intersecting planes. The threading dislocations would connect the ends of the slip lines to the pile-ups described in 2.1 (see fig. 6).

2.3. Threading dislocations

Dislocations that threaded the multilayers and were impacted against obstacles on intersecting slip planes were common. A micrograph of a portion of a pile-up of these dislocations in a specimen composed of 500 Å

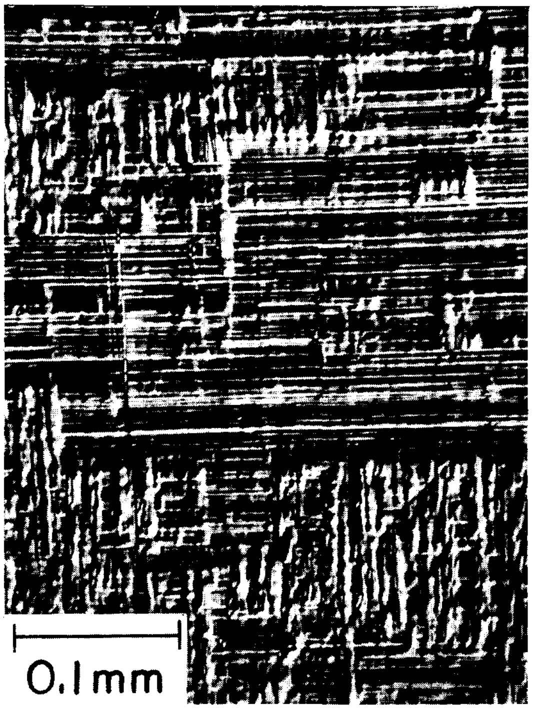

Fig. 7. Optical micrograph of slip lines on the surface of a multilayer composed of 700 Å layers.

layers is seen in fig. 8. The specimen was tilted to bring the {111} plane containing the pile-up more nearly perpendicular to the incident electron beam. This increased the projected length of the dislocation lines. The wavy appearance of the dislocations is not an electron optical artifact. It is present because the dislocations bent back and forth under the influence of the coherency strain in individual layers (cf. fig. 6 and fig. 4 of I).

The bowing of the dislocations in fig. 8 has not (with the exception of the dislocation that is arrowed) resulted in the formation of long misfit dislocations of the type discussed in I. This is so in spite of the fact that the thickness of the layers was greater than h_{crit}. Part of the explanation for this seems to be that the

Fig. 8. Micrograph of piled-up threading dislocations in a multilayer composed of 500 Å layers. The specimen was tilted so as to increase the projected length of the dislocations. The arrowed dislocation has bowed out to form short lengths of misfit dislocation as discussed in I. The positions of the end points of the dislocations indicate that some members of the pile-up did not lie on precisely the same slip plane.

bowing of a dislocation in a pile-up like that in fig. 8 is opposed by the other members of the pile-up.

A factor which may have contributed to the geometry of the dislocations in fig. 8 is diffusion along the dislocation lines. Pipe-diffusion along the threading dislocations in fig. 8 may have alloyed the layers sufficiently to raise h_{crit} above 350 Å (see 3.2 in I) in the vicinity of the dislocations. One of the effects that one might expect from enhanced diffusion along threading dislocations is electrical shorting of the superlattice device[2]). Electrical measurements made on the multilayers described here revealed an ohmic I–V characteristic rather than the predicted non-linear one. This could have been due to electrical short circuits associated with enhanced diffusion.

2.4. Relief of misfit stress by fracture

Multilayers sometimes contained cracks on the {110} planes almost perpendicular to the interface plane. A pair of cracks in a multilayer composed of 250 Å layers is seen in fig. 9. This figure is a scanning electron image of the multilayer seen from the side. One of the cracks extends through the multilayer. The other does not; it terminates on an array of dislocations. Etch pits formed at the emergence points of these dislocations are discernible. Although the pits do not give us the signs of the Burgers vectors of the dislocations there is little doubt that one sign predominated. There are two reasons for this. (i) The thickness of the layers in fig. 9 is below that at which arrays of dislocations with alternating Burgers vectors are stable (see 3.2 and 4.3 in I). (ii) Dislocations that emerge from the tip of an expanding crack are expected to have the same sign[8]).

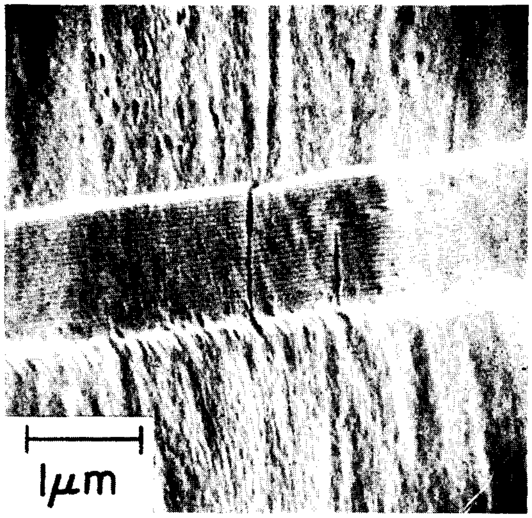

Fig. 9. Scanning electron micrograph of a specimen composed of 250 Å layers. The imaged surface is parallel to (110). A pair of cracks on $(1\bar{1}0)$ are visible. One of the cracks extends through the multilayer. The other does not; it terminates on an array of dislocation lines. Etch pits associated with the dislocations are visible.

3. Discussion

3.1. Role of dislocation pile-ups

Dislocation pile-ups are difficult to interpret as misfit dislocations between layers. This is because insertion of a pile-up into a thin multilayer, that is supported by its substrate but not elastically strained by it, increases the elastic energy of the system. The only satisfactory explanation for the presence of pile-ups seems to be that they are made to relieve elastic stresses generated as a result of the misfit between the multilayer taken as a whole and its substrate. The elastic energy of a thin multilayer can be reduced by a dislocation pile-up if the sign and strength of the pile-up are such that they reduce the elastic stresses generated in the multilayer by its substrate.

Although a pile-up on a plane inclined to the plane of a multilayer can accommodate part of the misfit between the multilayer and its substrate, a pile-up with this geometry is clearly not ideal for the purpose. The elastic energy of a multilayer with inclined pile-ups in it would be lowered if the pile-ups were replaced by an equivalent square network of edge dislocations with lines and Burgers vectors in the AB interface. This leads one to ask why inclined pile-ups are made. An answer to this question is given below.

3.2. Motion of dislocations through multilayers

Relief of stresses generated in a multilayer by its substrate requires that dislocations be transported towards or into the interface between multilayer and substrate. This can take place by dislocation motion through the multilayer, through the substrate, or along the multilayer–substrate interface. Glide along the interface seems unimportant except, possibly, near the specimen edge. The presence of slip lines on the surface of the multilayer, but not on the surface of the substrate, indicates that the dominant process, away from the specimen edge, is motion through the multilayer. That this should be so is not surprising. The thickness of the multilayer was always much less than the thickness of the substrate. This means that the elastic (misfit) stresses in the multilayer were always much larger than those in the substrate.

An isolated dislocation with lines parallel to the multilayer plane finds it difficult to move through the multilayer for the two reasons given in I. In GaAs/Ga(As,P) multilayers the coherency strains in individual layers are large ($\sim 1\%$) but the difference between the elastic constants of the film materials are small. Thus, in GaAs/Ga(As,P) one would expect the difficulty of moving a dislocation to result largely from the coherency strain.

For a dislocation to move through a GaAs/Ga(As,P) multilayer under the influence of the overall tensile stress present there we need some mechanism by which the dislocation can be pushed through the compressed GaAs (or C) layers. One method for doing this, and perhaps the only one, seems to be to generate a pile-up. The large elastic stresses exerted on the leading dislocation in a pile-up by those that follow seem able to push a short length of the leading dislocation two layers nearer the multilayer–substrate (or AB) interface. At the ends of this length are two superkinks of opposite sign. These kinks move apart under the influence of the overall stress in the multilayer and transport additional lengths of dislocation towards AB. Thus, superkinks play an important role in the movement of dislocations through multilayers. This role is the same as that played by ordinary kinks in the motion of dislocations through crystals[5]).

A consideration of the glide force on superkinks provides a simple argument in support of our interpretation of the role of pile-ups. If a multilayer is not stressed by its substrate then the ratio of the elastic strains in the B and C layers is

$$\varepsilon_B/\varepsilon_C = G_C h_C / G_B h_B. \qquad (2)$$

$G_{B,C}$ are the shear moduli of B and C. The Poisson ratios of B and C are assumed equal.

When eq. (2) is satisfied the glide force on the portion of a superkink that threads the B layer is equal and opposite to the force on the portion that threads the C layer. The net force on the superkink is zero.

If the multilayer is strained by $\Delta\varepsilon$ to accommodate part of the misfit between the multilayer and substrate then the elastic strains in the B and C layers are $\varepsilon_C - \Delta\varepsilon$ and $\varepsilon_B + \Delta\varepsilon$. The force on the portion of the superkink that threads the B layer is now larger than that on the portion that lies in C. This causes a superkink like the one in fig. 3 to move to the right and brings the dislocation nearer to the interface between the multilayer and the substrate. The conclusion suggested by this result is that there is no driving force for the motion of members of a pile-up through a multilayer unless the multilayer is stressed by its substrate.

3.3. Number of dislocations in pile-ups

An approximate value for the maximum number of dislocations expected in a pile-up can be found very simply. The dislocations in a pile-up relax the misfit strain over a distance roughly equal to the thickness of the multilayer. The number of dislocations needed to relax the strain over this distance is approximately

$$N_{\max} = [2h(n_B+n_C)f_m]/b, \tag{3}$$

where n_B and n_C are the number of B and C layers, and f_m is the misfit between the multilayer and its substrate. In our samples f_m is approximately

$$(a_C-a_B)/(a_C+a_B) \approx 0.01.$$

The maximum number of dislocations in a pile-up in a multilayer with $h = 200$ Å, $n_B+n_C = 60$, $f_m = 0.01$ and $b = 4$ Å is 60. If the thickness of the layers is increased to 700 Å then $N_{\max} = 210$. Thus, our interpretation of pile-ups is consistent with the height of the slip lines we have found.

3.4. Nature of obstacles to glide

The observations described in section 2.2 suggest that the most important obstacles to migrating threading dislocations are arrays of dislocations on intersecting slip planes. That this should be so is not surprising. To illustrate this we consider a train of threading dislocations that meets an array of dislocations on an intersecting {111} plane, and assume that the obstructing array contains one dislocation for each interface in the multilayer (see I).

When the leading glide dislocations meet the obstruction, the portions of them that lie in the Ga(As,P) or B layers penetrate the array. This is because penetration is aided by the following glide dislocations and by the misfit strain in the B layers. However, penetration will cease after portions of a small number of glide dislocations have moved through the array. This is because each penetrating dislocation causes a localized reduction in the strains present to accommodate misfit between B and C layers. The elastic strains in B and C layers reduced to zero in the vicinity of the penetrating dislocations when the number of penetrating dislocations reaches $\sim 2fh/b$. In GaAs/Ga(As,P) multilayers with $h = 200$ Å this number is about 2.

After penetration has eliminated the misfit strain in B and C layers the behavior of the impacted pile-up is expected to resemble the motion of glide dislocations through a dislocation forest[9]. If the threading dislocations bow between the members of the array but do not cut them[9]), then the total force needed to push a threading dislocation through the array is

$$2Q\,(n_B+n_C), \tag{5}$$

where Q is the dislocation line tension.

The force exerted on the leading member of a pile-up containing N dislocations is approximately

$$\frac{2G(1+\nu)}{(1-\nu)}\,h(n_B+n_C)\,N\left[f_m-\frac{Nb}{2(n_B+n_C)h}\right]b\cos\lambda. \tag{6}$$

λ is an angle defined in I. The term in square brackets is the misfit strain in the multilayer. It decreases as N increases because the pile-up accommodates part of the misfit between the multilayer and substrate. If

$$Q = \frac{Gb^2}{4\pi(1-\nu)}\left(\ln\frac{h}{b}+1\right), \tag{7}$$

and $n_B+n_C = 60$, $h = 200$ Å, $b = 4$ Å and $\nu = \frac{1}{3}$ there is no value of N that enables the entire lead dislocation to pass through the array.

If an obstructing array contains fewer dislocations than the one considered above, or if h is much larger than 200 Å, then (5) and (6) predict that the array will transmit the leading members of a pile-up and block the remainder. However, it is worth emphasizing that our calculation overestimates the stress driving the pile-up and thus probably underestimates the blocking power of an array. Observations of the AB interface show that about half the misfit between a multilayer and its substrate is accommodated not by pile-ups but by misfit dislocations that lie in AB. If allowance is made for this (by halving f_m) one finds that arrays on intersecting slip planes block pile-ups more effectively than (5) and (6) suggest.

3.5. Spacing of dislocations in pile-ups

The dislocations in the pile-ups discussed in 2.1 were separated by even numbers of layers. In conventional pile-ups the dislocation spacing increases in a complicated but gradual way as one moves back from the front of the pile-up[5]). This difference between multilayer pile-ups and conventional ones results from the coherency strain in individual layers. If one of the dislocations in fig. 1 were moved upwards a little the tensile stress in the Ga(As,P) (or B) layer would push it back down. On the other hand, if one of the dislocations were moved downwards the compressive stress in the GaAs (or C) layer would push it back up.

3.6. Role of cracks

There is little doubt that the cracks in fig. 9 were made to relieve stresses generated as a result of misfit between the multilayer taken as a whole and its substrate. The misfit between multilayer and substrate gives rise to tensile stresses parallel to the multilayer plane. These stresses are relieved by cracks on {110} planes approximately perpendicular to the multilayer plane. The roles ascribed to cracks and pile-ups here and in 3.1 are consistent with the emergence of a pile-up from the tip of one of the cracks in fig. 9[8]).

4. Final remarks

The results in I and in this paper suggest methods for improving the perfection of multilayers. Misfit dislocations between individual layers could be avoided by keeping layer thickness below h_{crit}. If the ideas discussed in section 3 are correct then pile-ups, threading dislocations, slip lines and cracks could be avoided by matching the lattice parameter of the substrate to the average lattice parameter of the multilayer.

GaAs/Ga(As,P) multilayers have been prepared to test these suggestions. These new multilayers were all much more perfect than those described here and in I. They contained no misfit dislocations, no cracks, and so few threading dislocations that we have not found one by transmission electron microscopy. Slip lines formed as a result of misfit between the multilayers and their substrate were also not present. These multilayers will be described in Part III.

Acknowledgments

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