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Formation of misfit dislocations in strained-layer GaAs/In_xGa_{1-x}As/GaAs heterostructures during postfabrication thermal processing

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It is demonstrated that relaxation of GaAs/In_xGa_{1-x}As/GaAs strained-layer heterostructures can be brought about by postfabrication thermal processing. Misfit dislocations are introduced into the structure during thermal processing, even though the thickness of the strained layer is well below the critical value predicted by the Matthews–Blakeslee model. The misfit dislocations are observed to be of both 60° mixed type and 90° pure edge type. As no relaxation occurs at the lower temperatures encountered during fabrication by molecular-beam epitaxy, it can be inferred that the critical condition for the formation of misfit dislocations is not only a function of strained-layer thickness and composition, but also of temperature. This observation cannot be accounted for by differential thermal expansion or diffusion across the strained-layer interfaces, but the temperature-dependent Peierls force may offer an explanation. The high temperature required to produce relaxation of these structures suggests that they are sufficiently thermally stable for most practical applications. © 2003 American Institute of Physics. [DOI: 10.1063/1.1627463]

I. INTRODUCTION

Strained-layer semiconductor lasers have huge commercial significance for the telecommunications industry. The reliability of these devices is dependent on the stability of the strained layer, and hence on the propensity to form misfit dislocations at the strained-layer interfaces. The ability to reliably predict the level of dislocation introduction and consequent strain relaxation is vital.

It is well known that if the misfit between an epilayer and its substrate is sufficiently small and a critical thickness of the epilayer is not exceeded, the deposited atomic layers will be strained to match the substrate and a coherent structure is thus formed. Strained-layer structures have been thought to be stable provided the strained-layer thickness is below a critical value, which is dependent on the difference between lattice parameters of the epilayer and substrate.^{1,2} For the majority of systems, the critical thickness determined experimentally by detecting bulk relaxation of the structure is found to be much greater, by a factor of up to 10, than that predicted by the generally accepted equilibrium models.³ This has been presumed to be because, at finite crystal

growth temperatures, energy barriers can prevent thermodynamic equilibrium being reached, so the kinetic development of the misfit dislocation array lags behind the equilibrium misfit dislocation density.^{2,4} So, the thermodynamic models of critical thickness divide strained-layer structures into stable ones, where the layer thickness is below critical, and metastable ones where it is above critical.¹ There is also a practical explanation for the discrepancy, arising from the fact that bulk relaxation measurements, e.g., using high-resolution x-ray diffraction, are insensitive to the formation of the first few isolated misfit dislocations. In contrast, electron microscope imaging of GaAs/In_xGa_{1-x}As/GaAs has shown the onset of the dislocation distortions that underpin the Matthews–Blakeslee model⁵ of relaxation at thicknesses close to, and even below, the critical value.⁶ The same article has proposed the existence of a different, but unspecified, mechanism for the bulk relaxation that occurs at layer thicknesses well above the Matthews–Blakeslee criterion.

The stability of strained-layer semiconductor structures has been investigated by post-growth thermal processing. Zhou and Cockayne⁷ found that the equilibrium critical thickness of In_{0.2}Ga_{0.8}As/GaAs was reduced from 10–12 nm for 800 K postgrowth annealing to 6–8 nm for 870 K annealing. Lourenco *et al.*² investigated the effects of thermal processing at temperatures up to 1273 K and found that

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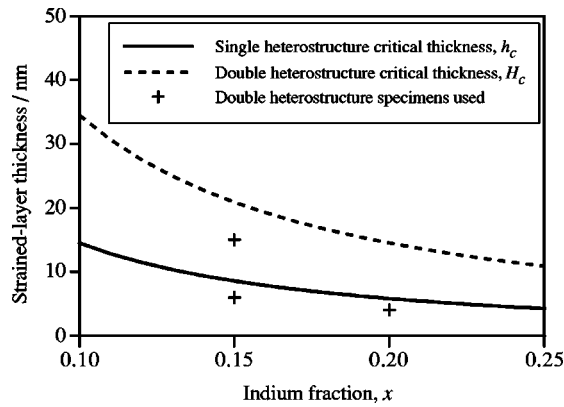


FIG. 1. Comparison of specimens used and critical thickness predicted by the Matthews–Blakeslee model for $\text{In}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$ heterostructures.

single 140 nm layers of $\text{In}_{0.1}\text{Ga}_{0.9}\text{As}$ were metastable. Beanland *et al.*⁸ found that additional misfit relief was produced in a 140 nm layer of $\text{In}_{0.1}\text{Ga}_{0.9}\text{As}$ on GaAs by rotation of 60° misfit dislocations into edge orientation during postgrowth thermal processing.

In this article, we present the results of an investigation into the effects of thermal processing on strained-layer GaAs/ $\text{In}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$ heterostructures. In each case, the thickness of the $\text{In}_x\text{Ga}_{1-x}\text{As}$ strained layer was below the critical value predicted by Matthews and Blakeslee⁵ for a double heterostructure.

II. EXPERIMENTAL DETAILS

All of the structures considered here comprise a single strained layer of $\text{In}_x\text{Ga}_{1-x}\text{As}$ of thickness h between surrounding layers of GaAs. The specimens were grown by molecular-beam epitaxy (MBE). A 50 nm AlAs layer was first grown epitaxially on a GaAs (001) substrate, followed by a 200 nm layer of GaAs, a layer of $\text{In}_x\text{Ga}_{1-x}\text{As}$ of thickness h , and a second 200 nm layer of GaAs. The growth temperature was 800 K for the $\text{In}_x\text{Ga}_{1-x}\text{As}$ layer and 870 K for other layers. The growth rate was $1 \mu\text{m}/\text{h}$. The composition and layer thickness were calibrated by x-ray diffraction. The surface morphology was monitored during growth by reflection high-energy electron diffraction.

Three types of specimen were considered: $x=0.15$ with $h=6$ nm, $x=0.15$ with $h=15$ nm, and $x=0.20$ with $h=4$ nm. In each case, h was below the critical value, H_c , predicted by the Matthews–Blakeslee model⁵ for misfit dislocation formation in a double heterostructure (Fig. 1). In the cases of $x=0.15$ with $h=6$ nm and $x=0.20$ with $h=4$ nm, the strained-layer thickness was also below the critical thickness, h_c , predicted for a single layer of $\text{In}_x\text{Ga}_{1-x}\text{As}$ on GaAs. In these two cases, no misfit dislocations would have been expected. In the sample with h falling between h_c and H_c , some misfit dislocations might have been expected since, during growth, the specimen would have been a single heterostructure before the second GaAs layer was added. In fact, no misfit dislocations, and hence no relaxation, were observed in any of the structures prior to thermal processing.

The bulk specimens (approximately $5 \text{ mm} \times 5 \text{ mm}$) were annealed at differing temperatures up to 1350 K in a nitrogen

atmosphere within a Carbolite furnace. The specimens were plunged into the furnace and, once the target temperature had been attained, were held there for 300 s before being withdrawn from the furnace to cool. Specimens were annealed in pairs, placed face-to-face in order to reduce the possibility of arsenic loss from the surfaces. Subsequent imaging by cathodoluminescence (CL) and transmission electron microscopy (TEM) showed that no surface damage had occurred.

The use of CL imaging on a JEOL JSM-820 scanning electron microscope allowed dislocations to be examined at low magnification in a bulk specimen, thereby avoiding the risk of the dislocation configuration being altered by specimen preparation. Film specimens for TEM observation were prepared using the epitaxial lift-off technique.⁶ This technique involves etching away the sacrificial AlAs layer in order to release the heterostructures from the substrate. TEM observation was on a JEOL 2000FX operated at 200 kV. Cross-sectional TEM inspection allowed the thickness of the strained layer to be confirmed.⁶

III. RESULTS

A. Cathodoluminescence imaging of 60° dislocations

In Fig. 2, CL imaging shows the introduction of misfit dislocations into GaAs/ $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}/\text{GaAs}$ strained-layer structures by thermal processing. Figures 2(a) and 2(b) show specimens prior to any thermal processing, where the strained-layer thicknesses, h , are 6 and 15 nm respectively. Although misfit segments on threading dislocations have been observed in some subcritical structures,⁶ these figures show that no identifiable dislocations can be found by CL imaging at lower magnification. It can therefore be assumed that these structures are globally coherent. Figure 2(c) shows a CL image of a specimen with $x=0.15$ and $h=25$ nm. This specimen is for comparison only; it is not part of the present investigation and was not thermally processed. The misfit dislocations formed during MBE growth are clearly revealed, indicating the global relaxation of the structure.

No misfit dislocations were introduced into the $x=0.15$ specimens by thermal processing at 1170 K, as shown in Fig. 2(d). However, at a temperature of 1220 K, many misfit dislocations were introduced for both $h=15$ nm [Fig. 2(e)] and $h=6$ nm [Fig. 2(f)].

Similar findings were made for the specimen with $x=0.2$ and $h=4$ nm, as shown in Fig. 3. No dislocations were introduced into this kind of structure until the temperature was raised to 1350 K, as shown in Fig. 3(b). The misfit dislocations formed during thermal processing, shown in Figs. 2(e), 2(f), and 3(b), do not show any significant difference in their geometries from those seen in as-grown structures [Fig. 2(c)], although they have a higher density.

The black spots in Figs. 2(f) and 3(b) indicate that surface damage occurs during thermal processing at temperatures of 1220 K or above. Because the background of the TEM images from these specimens (Figs. 4 and 5) is unchanged from that from the as-grown specimens,⁶ this damage appears to be limited and is not expected to influence the dislocation structures.

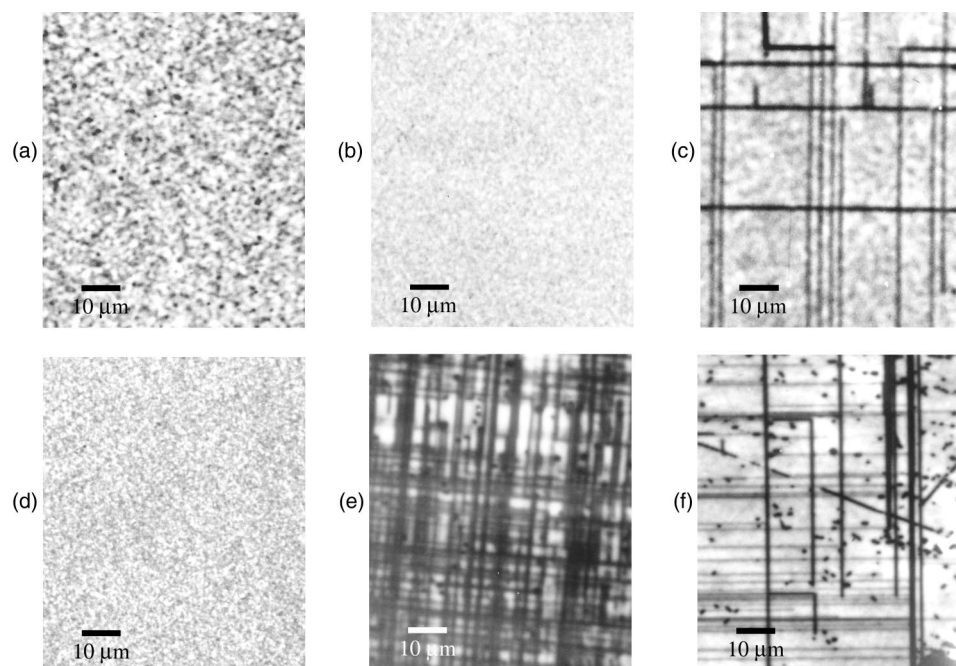


FIG. 2. CL images of GaAs/ $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}/\text{GaAs}$ heterostructures: (a) As-grown, $h = 6$ nm, (b) as-grown, $h = 15$ nm, (c) as-grown, $h = 25$ nm, (d) after 300 s at 1170 K, $h = 15$ nm, (e) after 300 s at 1220 K, $h = 15$ nm, and (f) after 300 s at 1220 K, $h = 6$ nm.

B. Transmission electron microscopy of 60° dislocations

Figure 4 is the TEM counterpart of the CL image in Fig. 2(e) and shows bright- and dark-field images of misfit dislocations formed in GaAs/ $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}/\text{GaAs}$ with $h = 15$ nm by thermal processing at 1220 K. All of these misfit dislocations are 60° type.⁵ Figure 5 is a bright-field image showing similar dislocation structures formed in GaAs/ $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}/\text{GaAs}$ with $h = 4$ nm during thermal processing at 1350 K. The apparent fuzziness of the dislocations in the bright-field image of Fig. 5 arises because some dislocations are present in pairs, as revealed by the dark-field image shown in Fig. 4(b).

The question now arises as to whether both dislocation segments of a pair are located in the same interface or at separate interfaces. In Fig. 4(b), two different configurations can be seen when a dislocation pair crosses another dislocation. Segments B and C cross segment E without interaction, while segments A and D interact with E to produce a bright spot in the image,⁹ arrowed. It can therefore be inferred that the components of each pair are located in different inter-

faces. Dislocation segments A and D are in same interface as E, but B and C are not. The dislocation pair A and B share the same (111) slip plane, as do the pair C and D.

Since there is a single point of interaction between the dislocation pairs and dislocation E, it can be inferred that E is not a couple, but rather a single misfit dislocation located at the lower interface. The observation that some misfit dislocations formed during thermal processing are present in pairs, while the others such as E occur singly, suggests that different mechanisms were involved in their formation. As there were no misfit dislocations present before thermal processing, the formation of dislocation E cannot be explained by the operation of a Matthews—Blakeslee mechanism during growth when the thickness of the $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}$ layer exceeded h_c . Although the paired dislocations are consistent with the Matthews—Blakeslee model, the single dislocations appear to have resulted from a second mechanism. The existence of a second mechanism, thought to be responsible for bulk relaxation at layer thicknesses above H_c , has been previously proposed.⁶

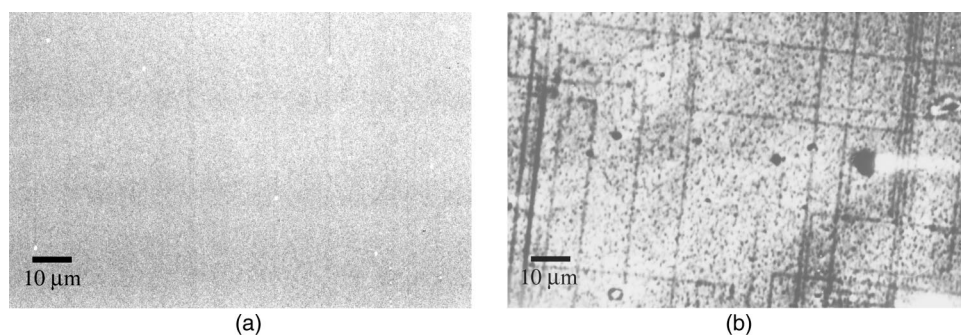


FIG. 3. CL images of GaAs/ $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}/\text{GaAs}$ heterostructures, $h = 4$ nm (a) as-grown and (b) after 300 s at 1350 K.

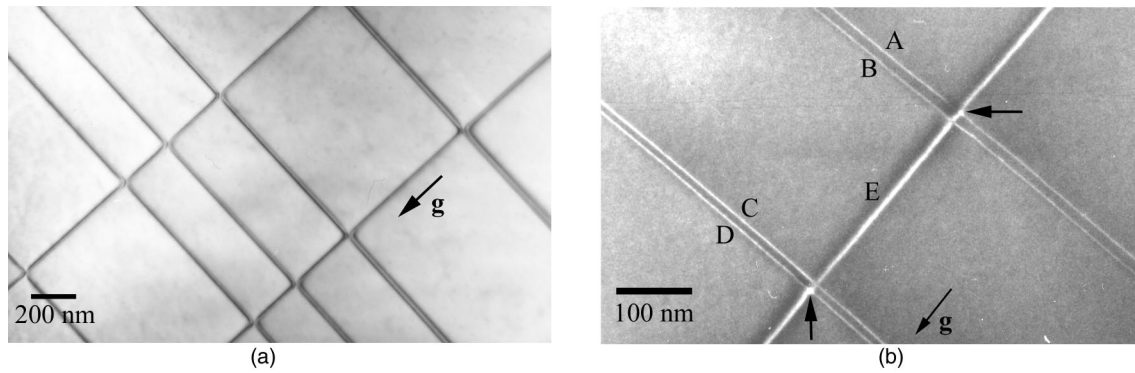


FIG. 4. TEM images of misfit dislocations in GaAs/In_{0.15}Ga_{0.85}As/GaAs ($h=15$ nm) after thermal processing at 1220 K for 300 s: (a) bright field, $\mathbf{g}=[220]$ and (b) dark field, $\mathbf{g}=[220]$. Segments B and C cross segment E without interaction, while segments A and D interact with E to produce the arrowed bright spots.

C. Transmission electron microscopy of edge-type misfit dislocations

A second type of misfit dislocation was found in GaAs/In_{0.15}Ga_{0.85}As/GaAs ($h=15$ nm) after thermal processing at 1220 K, as shown in Fig. 6. This type occurs in pairs, oriented parallel to [010], and formed on threading dislocations. The spacing of the dislocation pairs is wider when viewed by [400] reflection [Fig. 6(b)] than when viewed by [220] reflection [Fig. 6(a)]. This suggests that the dislocation pair is located in a (001) plane. The dislocation pair is invisible when viewed using $\mathbf{g}_1=[040]$ and $\mathbf{g}_2=[131]$, shown in Figs. 6(c) and 6(d), respectively. Given the invisibility criteria that $\mathbf{b}=\mathbf{g}_1 \times \mathbf{g}_2$ where $\mathbf{g}_1 \cdot \mathbf{b}=0$ and $\mathbf{g}_2 \cdot \mathbf{b}=0$, it can be determined that the Burgers vector of the dislocation pair is in the (010) plane and in the $[40\bar{4}]$ direction. Therefore, the dislocation pair shown in Fig. 6 is pure edge type. Since this type of dislocation is located at the interfaces,^{10,11} they are misfit dislocations. As it originates from a 60° threading dislocation, the combined Burgers vector of the pair must cancel, leaving a net 60° Burgers vector arising from the short dislocation segment that joins the pair.

The edge misfit dislocations produced by thermal processing have a variety of geometries, but they are all rather short and are distributed throughout the specimen, as shown in Fig. 7. Dislocation A was generated on the 60° dislocation B. Dislocation B would have been generated first during thermal processing, and then dislocation A generated from

it.^{10,11} Dislocation C was generated on the threading dislocation D. Dislocation E appears more complex and is possibly a dislocation group formed by movement of several dislocations. Dislocations F and G are particularly interesting, as they have formed a closed loop. They are presumed to have resulted from the directional expansion of dislocation loops under the effects of misfit stress.^{10,11}

IV. DISCUSSION

A. Temperature dependence of relaxation

Postgrowth thermal processing has been shown to introduce misfit dislocations into strained-layer structures. In two of the samples $h < h_c$ and in the other $h_c < h < H_c$. Although some misfit dislocations might have been expected in the third sample, none were observed in any of the samples prior to processing.

The misfit strain, f , in a strained-layer structure is given by

$$f = \frac{a_o - a_s}{a_s}, \quad (1)$$

where a_s and a_o are the lattice parameters of the substrate and overlayer respectively.¹² When the coherence between a strained layer and its substrate is lost, the misfit strain is replaced by a plastic strain component, δ , due to the misfit dislocations and a residual elastic strain component, ϵ :¹³

$$f = \epsilon + \delta \quad (2)$$

The value of δ can be estimated from the misfit dislocation spacing, S :

$$\delta = \frac{b_e}{S}, \quad (3)$$

where b_e is the interface-plane component of the Burgers vector in the direction of the misfit dislocation spacing, S .

The relaxation achieved by the formation at 1220 K of misfit dislocations in GaAs/In_{0.15}Ga_{0.85}As/GaAs heterostructures is approximately 5%–8% of the misfit strain for both $h=6$ nm and $h=15$ nm. These experimental results suggest that there is a critical temperature at which the strained-layer structures become unstable and the formation of misfit dis-

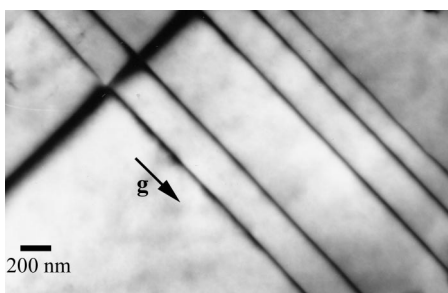


FIG. 5. Bright-field TEM images of misfit dislocations in GaAs/In_{0.2}Ga_{0.8}As/GaAs ($h=4$ nm) after thermal processing at 1350 K for 300 s, $\mathbf{g}=[220]$.

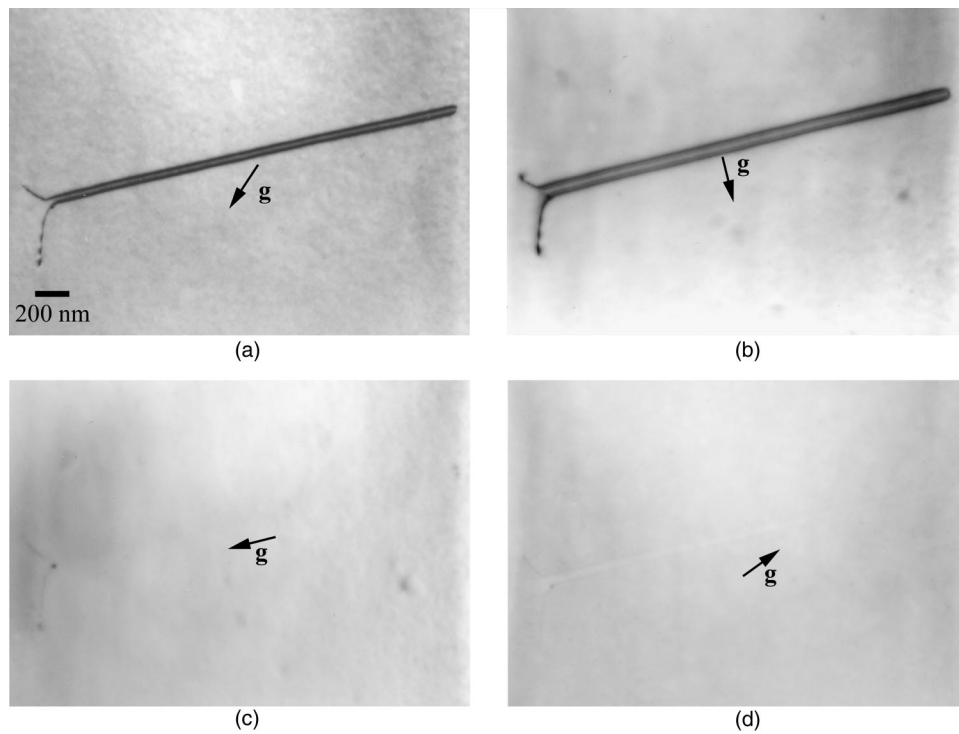


FIG. 6. Bright-field TEM images of a misfit dislocation in GaAs/In_{0.15}Ga_{0.85}As/GaAs ($h=15$ nm) after thermal processing at 1220 K for 300 s, viewed with different Bragg reflections: (a) $\mathbf{g}=[2\bar{2}0]$, (b) $\mathbf{g}=[400]$, (c) $\mathbf{g}=[040]$, (d) $\mathbf{g}=[131]$.

locations becomes thermodynamically favorable. For GaAs/In_{0.15}Ga_{0.85}As/GaAs heterostructures with $h=25$ nm, widespread formation of misfit dislocations occurred during fabrication at 870 K. For similar structures with smaller strained-layer thicknesses, h , of 6 nm and 15 nm, a higher temperature of up to 1220 K was required for the formation

of misfit dislocations. Therefore, the critical condition for the formation of misfit dislocations is not only a function of strained-layer thickness and composition, but also of temperature.

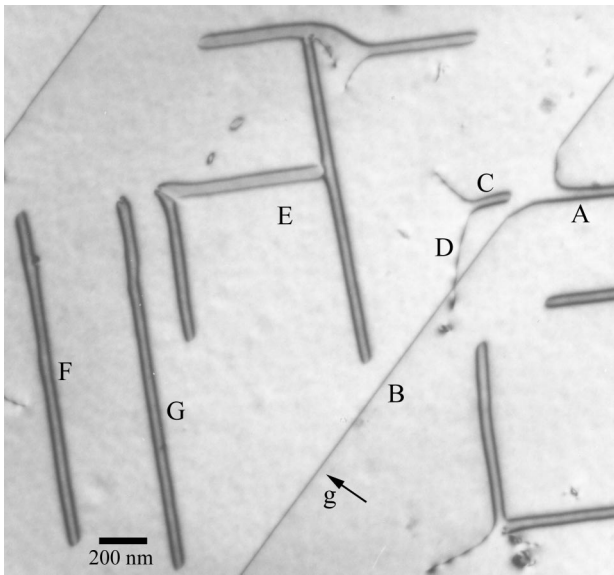


FIG. 7. Bright-field TEM image of 60° and edge misfit dislocations formed in GaAs/In_{0.15}Ga_{0.85}As/GaAs ($h=15$ nm) during thermal processing at 1220 K for 300 s; $\mathbf{g}=[220]$. A has formed on the 60° dislocation B, which formed before it. C was generated on the threading dislocation D. E has probably resulted from the movement of several dislocations. F and G are closed loops.

B. Differential thermal expansion

Here, we assess whether the difference in thermal expansion coefficients between the substrate and strained layer could account for the temperature dependence of the formation of misfit dislocations.

The thermal expansion coefficient of GaAs is $6.89 \times 10^{-6} \text{ K}^{-1}$ at 800 K, which is the temperature of fabrication of the heterostructures, rising to $7.64 \times 10^{-6} \text{ K}^{-1}$ at 1300 K,^{14,15} which is of the order of the thermal processing temperature. The thermal expansion coefficient $\alpha(x)$ for In _{x} Ga _{$1-x$} As can be calculated as:¹⁶

$$\alpha(x) = 5.20 + 0.83(1-x) \times 10^{-6} \text{ K}^{-1}, \quad (4)$$

yielding values of $\alpha(0.15) = 5.906 \times 10^{-6} \text{ K}^{-1}$ and $\alpha(0.2) = 5.864 \times 10^{-6} \text{ K}^{-1}$. Although no temperature or temperature dependence is specified here, the thermal expansion coefficient of GaAs is clearly greater than that for In _{x} Ga _{$1-x$} As. Therefore, as the temperature rises, the misfit between In _{x} Ga _{$1-x$} As and GaAs would be expected to decrease. Differential thermal expansion cannot, therefore, provide an explanation for the formation of misfit dislocations at high temperatures.

C. Diffusion across the interfaces

Interdiffusion in semiconductor heterostructures can occur during both growth and subsequent processing,¹⁷ thereby changing the compositions of the layers adjacent to interfaces and ultimately removing the interface altogether. The melting point, T_m , of $\text{In}_x\text{Ga}_{1-x}\text{As}$ can be calculated by linear interpolation between T_m for GaAs (1513 K) and T_m for InAs (1215 K),¹⁸ yielding 1468 K for $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}$ and 1453 K for $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$. Thus, the thermal processing temperature is $0.83 T_m$ at 1220 K and $0.93 T_m$ at 1350 K, i.e., high enough for interdiffusion to occur. If significant interdiffusion had occurred, the thickness of the strained layer would be effectively increased while its indium concentration, x , would be reduced. The overall effect would be to increase the stability of the structure, owing to the nonlinearity of the Matthews–Blakeslee criterion. Therefore, like differential thermal expansion, interdiffusion cannot provide an explanation for the formation of misfit dislocations at high temperatures.

D. Activation barrier for dislocation movement

Modifications to the Matthews–Blakeslee model have previously been proposed which predict an increase in critical thickness with decreasing temperature.^{19,20} These models are based on the notion that dislocation movement is opposed by the Peierls force, which creates an activation barrier. The effect of this is to raise the critical thickness at low growth temperatures below approximately 800 K. By extrapolation of this argument, it is possible that high temperatures could reduce the Peierls force and therefore reduce the critical thickness below the Matthews–Blakeslee criterion.

V. CONCLUSIONS

The critical condition for the relaxation of strained-layer structures through the formation of misfit dislocations has been shown to be a function of temperature as well as strained-layer thickness and composition. Higher temperatures enable the formation of misfit dislocations in structures with thinner strained layers. Consequently, a coherent strained-layer structure produced by MBE growth can be relaxed by a postgrowth treatment at a higher temperature than the fabrication temperature. Burgers vector analysis has

shown that the misfit dislocations produced are of both 60° mixed type and 90° pure edge type. It has been shown that the temperature dependence cannot be accounted for by differential thermal expansion or diffusion across the strained-layer interfaces. The temperature-dependent Peierls force may provide an explanation, although previous investigations have concentrated on its effect at temperatures below, rather than above, normal growth temperatures. Although the underlying mechanism is not yet clear, the high temperature required to produce relaxation suggests that these structures are sufficiently stable for most practical applications.

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