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High-resolution electron microscopy study of ALMBE InAs grown on (001) GaAs substrates

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High-resolution electron microscopy has been used to study the distribution of defects in a heteroepitaxial system with high lattice mismatch (InAs/GaAs, $(a_{InAs} - a_{GaAs})/a_{GaAs} = 7.2\%$), grown by atomic layer molecular beam epitaxy. The study has been carried out on cross-section samples. A high density of dislocations, mainly of the Lomer type, has been found in a region of the epilayer near the interface. Banding contrast is observed in the interfacial region; possible explanations for this contrast are given.

1. Introduction

Highly mismatched heteroepitaxial systems are of increasing importance for the realization of advanced semiconductor devices. The interest in the successful growth and characterization of lattice-mismatched III–V semiconductor systems stems from the considerable flexibilities in the design of integrated optoelectronic devices and light waveguides which are available when compound semiconductors are used [1]. If lattice-mismatched heterostructures are to be used in device applications, the role of dislocation generation and propagation in these materials must be understood. The difference in lattice constants in these structures leads to strain in the epilayer, and it is important to measure this strain for several reasons: (a) the strain is related to the formation of misfit dislocations, (b) strained layers are inherently interesting materials, and (c) the electrical and optical properties are strongly dependent on the amount of strain. In fact, one can utilize the strain to "fine-tune" the device properties [2].

In this study we have characterized the structure of defects in the interfacial region of InAs layers grown on (001) GaAs substrates (7.2% lattice mismatch) by atomic layer molecular beam epitaxy (ALMBE). Observations are reported based upon high-resolution electron microscopy (HREM). We have observed lattice relaxation in the ⟨001⟩ growth direction existing over a wide region at the InAs/GaAs interface, visible in the HREM images from the periodic bands of image contrast which it causes. Until recently, the consequences of elastic relaxation in HREM studies of thinned materials have usually been ignored. This work has taken into account the fact that when relaxation due to sample preparation dominates, specimen properties measured from HREM images will not reflect the properties of either the stressed bulk or the fully relaxed materials [3]. It is important, however, to realize that this type of strain contrast is not necessarily an impediment to the correct interpretation of HREM micrographs.

2. Experimental details

Highly mismatched InAs layers with thicknesses of 250 nm have been grown directly on

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(001) GaAs substrates by ALMBE at the Spanısh Natıonal Center for Mıcroelectronıcs at Madrıd [1] The lattıce mısmatch ıs 7 2%

The cross-sectıonal specımens for electron mıcroscopy were prepared ın the standard fashıon Two cross-sectıons were glued face-to-face wıth M-bond 600 glue, prethınned usıng mechanıcal thınnıng and polıshıng, dımpled to 40 μm and then fınally thınned to perforatıon usıng low-angle (17°), 4 kV, Ar^+ ıon-mıllıng at lıquıd N_2 temperature The thın TEM specımens were then examıned usıng eıther a JEOL 2000EX (at Cádız Unıversıty) or a JEOL 4000EX (at Oxford Unıversıty) Movement or formatıon of dıslocatıons dıd not occur durıng observatıon ın the mıcroscopes

3. Results and discussion

3 1 Interface dıslocatıon structure

Careful analysıs of a number of HREM ımages of ⟨110⟩ cross-sectıonal specımens of the InAs/GaAs ınterface showed that Lomer dıslocatıons domınated (75% were of the Lomer type) In addıtıon, undıssocıated 60 ° dıslocatıons were observed (25%) Screw dıslocatıons dıssocıated ınto two 30° partıals were rarely observed Fıg 1 shows a typıcal ⟨110⟩ cross-sectıon mıcrograph The ımage clearly shows that the Lomer dıslocatıons are evenly dıstrıbuted over a regıon of the InAs epılayer up to 30–40 nm from the ınterface, and that most of the dıslocatıons are out of the plane of the ınterface Karasev et al [4] reached a sımılar conclusıon for InAs layers wıth thıcknesses of $t \geq 10$ nm that were formed ın multılayered heterosystems of alternatıng InAs and GaAs layers

It has been suggested that each Lomer dıslocatıon ıs formed as the result of the ınteractıon of two 60° dıslocatıons wıth Burgers vectors ın the $(1\bar{1}1)$ and $(\bar{1}11)$ glıde planes ıntersectıng at the ınterface [5], as follows

$$a[10\bar{1}]/2 + a[0\bar{1}1]/2 = a[1\bar{1}0]/2 \quad (1)$$

Thıs would be a reasonable descrıptıon for the materıal observed ın thıs study, sınce ıt ıs ob-

Fıg 1 HREM ımage of [110] cross-sectıon showıng InAs/GaAs ınterface Note the dıstrıbutıon of dıslocatıons ın the regıon close to the ınterface

served that the Lomer dislocations are near the interface plane, having remained in this location following interaction (fig 1) The 60° dislocations, however, are farther away, which could be due to the fact that they have not interacted and so have moved

This material was studied during growth by RHEED [1] and it was found that total relaxation occurred after the growth of eight monolayers This result means that the misfit dislocations were initially concentrated in the region close to the interface, and must therefore have moved during growth

Fig 2 shows an image obtained with only the (000) beam contributing, diffraction contrast is observed due to a 60° dislocation which has its core in the interfacial region ($InAs_I$ in the figure) This interpretation was confirmed by diffraction contrast measurements Analysis of larger regions of the sample revealed very few of these dislocations

As shown by Merwe and Ball [6], and by Matthews [7], the dislocations serve to relax the mismatch-induced strain between a heteroepitaxial film and its substrate [8], although several processes can impede dislocation formation It is interesting to note that, by assuming the mismatch to be completely relaxed by misfit dislocations, the mean distance d_m between those dislocations is given by

$$d_m = b_p / f', \qquad (2)$$

where

$$f' = \text{mismatch} = (a_{InAs} - a_{GaAs}) / a_{InAs} = 0\ 068$$

b_p = edge component of the Burgers vector of the dislocation projected over the interface

In the present case, we have estimated a value for b_p taking into account the number of observed dislocations (75% Lomer type and 25% undissociated 60° type) The resulting value for b_p would then be

$$b_p = b_{p60°disl}/4 + 3b_{Lomerdisl}/4 = 0\ 3498 \text{ nm} \qquad (3)$$

d_m, calculated from eq (2), has a value of 5 1441 nm The mean value measured from the images

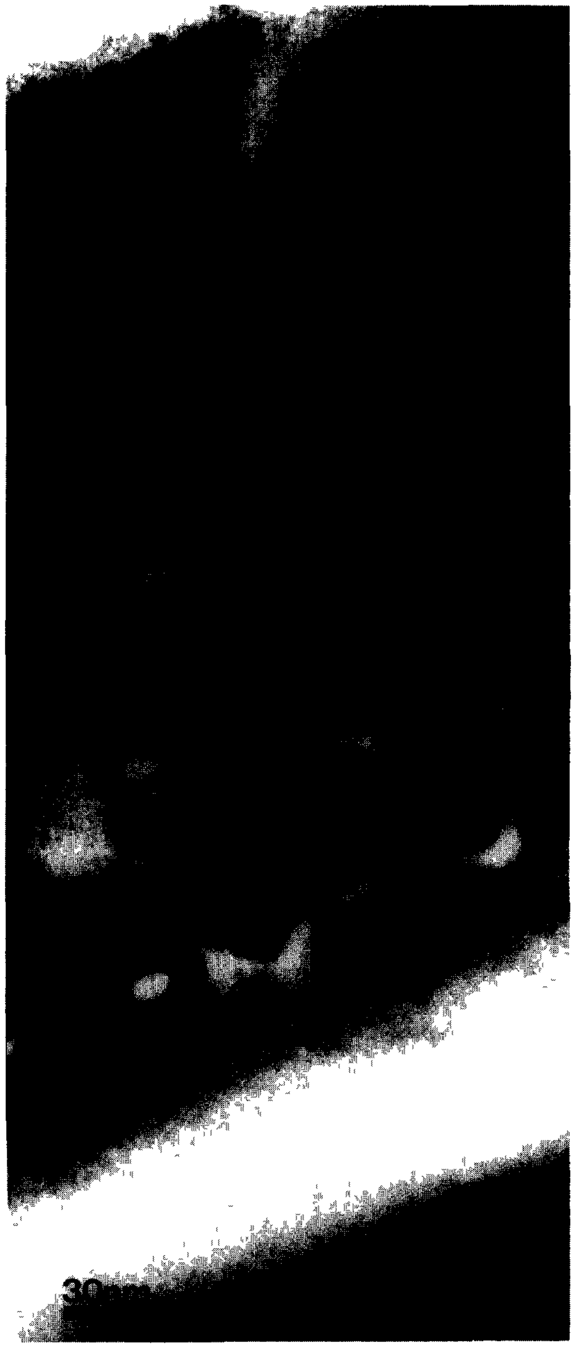

Fig 2 Bright-field [110] TEM image obtained using only (000) beam Contrast due to one 60° dislocation which has its core in the $InAs_I$ region can be seen

for the separation distance between dislocations is equal to 5 41 nm This implies that the relaxation of strain at the InAs/GaAs interface occurs mainly by generation of the observed dislocations This measurement was made in a region less than 40 nm from the interface plane, and we can thus deduce that relaxation of the mismatch occurs in a zone close to the interface

3 2 Banding in the epitaxial layer

Periodic bands of image contrast perpendicular to the ⟨001⟩ growth direction are visible in fig 1, extending 30–40 nm into the InAs layer from the InAs/GaAs interface It is difficult to analyze the lattice constant component (a_{pll}) parallel to ⟨011⟩, but no fluctuations in a_{pll} larger than 2 5% were observed in any specimen in the InAs layer

An image recorded with the electron beam parallel to the ⟨110⟩ direction is shown in fig 3b A detailed analysis is very fruitful for determining variations in the lattice constant component perpendicular to the interface (a_{per}) from a region of the InAs layer extending about 40 nm from the interface The measured a_{per} lattice constant for InAs fluctuates over the 40 nm of the InAs layer closest to the InAs/GaAs interface (the region referred to as $InAs_I$) The measurements of the lattice constant components a_{per} parallel to ⟨001⟩ are tabulated in fig 3a It is of interest that this plot shows that most of the values of a_{per} for $InAs_I$ were

$$(a_{0GaAs} = 0\ 565 \text{ nm}) < a_{per} \text{ for } InAs_I < (a_{0InAs} = 0\ 606 \text{ nm})$$

The plot shows several minima (imaged as white banding contrast on fig 3b), at 2, 20, 42, 62, 76 monolayers (ML) and also maxima (imaged as dark banding contrast), suggesting that both tetragonal dilatations and contractions are present

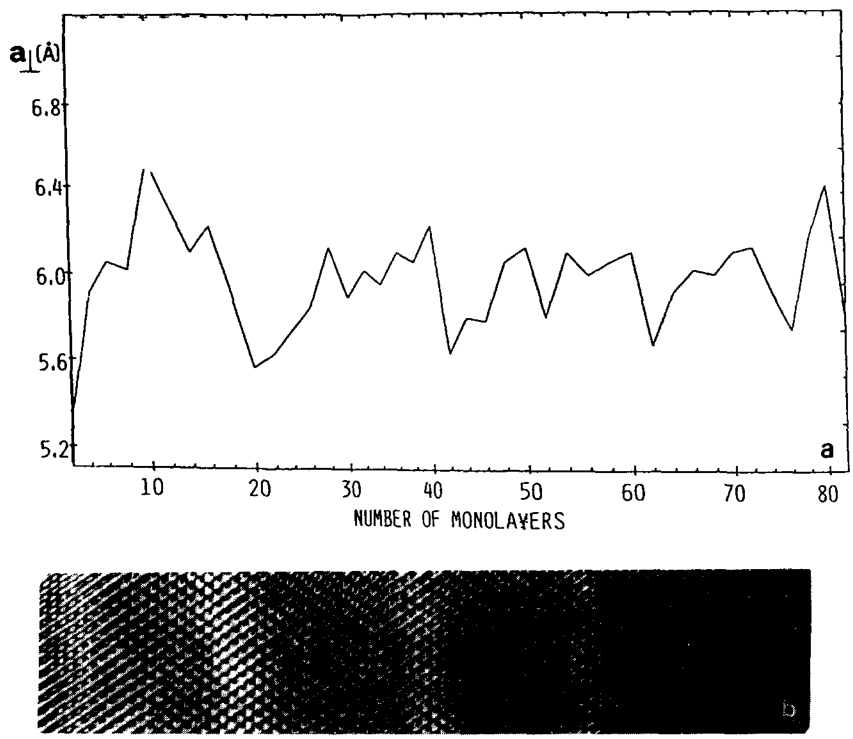

Fig 3 (a) Variation in lattice parameter in growth direction, a_{per}, measured from HREM micrograph shown in (b), versus the number of monolayers in the [001] direction (b) HREM micrograph of the region $InAs_I$ Note correlation between banding contrast in HREM image and change in lattice constant in the $InAs_I$ layer The tetragonal distortions described above provide an adequate description for the black and white banding contrast observed

In addition to tetragonal distortion, changes in lattice plane orientation were also seen in the $InAs_I$ region Other workers [4,9,10] have reported similar observations of this kind for InAs The change in orientation can be seen by comparing the {111} planes in the dark contrast regions of fig 3b with those in the white contrast regions In the present study, the relative plane inclination caused by these deformations ranged from 1° to 3°, which corresponds to a tetragonal contraction with variations for a_{per} of the $InAs_I$ layers between 4 and 10% (calculated by neglecting the deformation in a_{pll})

It was originally suggested that the origin of the banding contrast lay in the presence of $Ga_xIn_{1-x}As$ alloys located in the interfacial region This suggestion has, however, been rejected because the temperature (400 ° C) of the GaAs substrate during epitaxial growth is not high enough for Ga diffusion to occur We therefore suggest the possibility that the banding is caused by elastic relaxation The "strain supression" method of Timoshenko and Goodier [11] has been used, applying the approach of Gibson and Treacy [3] to calculate the effect of surface relaxation in thinned superlattice specimens in the presence of a single interface (thickness/superlattice period $\rightarrow \infty$) These calculations give values for ϵ perpendicular to the interface plane that are three orders of magnitude lower than those obtained experimentally The observed strain can therefore only be explained by considering additional factors such as the presence of steps in the interface [12] (present in this material), and the interaction of the strain fields of the high density of dislocations at the interface

4. Conclusions

The mismatch in InAs/GaAs heterostructures has been analyzed The presence of Lomer and 60° undissociated dislocations, which appear to relax a large part of the mismatch, has been observed The stresses are high in the InAs layer up to a distance of about 30–40 nm from the GaAs/InAs interface, displaying a complex interplay between strains and dislocations in this region The fact that not all of the specimen strain fields are necessarily inherent to the original material has been taken into account

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References

[1] A Ruiz, L González, A Mazuelas and F Briones, Appl Phys A 49 (1989) 543

[2] S N G Chu, A T Macrander, K E Strege and W D Johnston, Jr , J Appl Phys 57 (1985) 249

[3] M M J Treacy and J M Gibson, J Vac Sci Technol B 4 (1986) 1458

[4] V Yu Karasev, N A Kiselev, E V Orlova, A K Gutakovski, S M Pintus and S V Rubanov, Inst Phys Conf Ser 100 (1989) 33

[5] N Otsuka, C Choi, L A Kolodziedski, R L Gurshar, R Fisher, C K Dengi, H Morkoc, J Tafto and J C H Spence, J Vac Sci Technol B 4 (1986) 896

[6] J H van der Merwe and C A B Ball, in Epitaxial Growth, Ed J W Matthews (Academic Press, New York, 1975) part B, ch 7

[7] J W Matthews, in Epitaxial Growth, Ed J W Matthews (Academic Press, New York, 1975) part B, ch 8

[8] J W Matthews, ın Dıslocatıons ın Solıds, Ed F R N Nabarro (North-Holland, Amsterdam, 1979)
[9] C D'Anterroches, J Y Marzın, G Le Roux and L Goldsteın, J Crystal Growth 81 (1987) 121
[10] C D'Anterroches, J M Gerard and J Y Marzın, NATO ASI Ser B Phys 203 (1989) 47
[11] S P Tımoshenko and J N Goodıer, Theory of Elastıcıty (McGraw-Hıll, New York, 1970)
[12] M M J Treacy, NATO ASI Ser B Phys 203 (1989) 255