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# Structural characterization of highly strained InAs $N$ monolayer lasers and quantum well structures by X-ray diffraction and transmission electron microscopy

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X-ray interference effect and transmission electron microscopy are used to study the relaxation process in a series of laser structures as a function of InAs content in the quantum well. It is shown that the X-ray interfrence effect is a powerful, fast and non-destructive method to assess the strain status in samples of this kind. A set of strained layer laser structures containing $N$ monolayers of InAs ($N\times(\mathrm{InAs})_1(\mathrm{GaAs})_3$ with $N = 1, 3, 5, 7$) in an 8 nm quantum well active region and a set of strained layer quantum wells consisting of P monolayers of InAs ($P\times(\mathrm{InAs})_1(\mathrm{GaAs})_Q$ with $P = 2$, 4 and $Q = 2$, 4) were grown [Dotor et al., J. Crystal Growth 127 (1993) 46] by atomic layer molecular beam epitaxy. X-ray interference effect and cross-section transmission electron microscopy analysis of the samples show that in the series of lasers with $N$ monolayers of InAs the whole laser structure is coherent with the substrate (and consequently dislocation free) for 1 and 3 monolayers of InAs, while a sample with 5 monolayers of InAs is in a certain stage of relaxation (dislocation density $n_d \simeq 10^7$ cm$^{-2}$) and a sample with 7 monolayers of InAs is almost completely relaxed ($n_d \simeq 10^8$ cm$^{-2}$). In strained layer quantum well samples, the influence of the InAs/GaAs thickness ratio ($P/Q$) on the critical thickness has also been studied. These results are compared with those predicted by theoretical critical thickness models. Optical characterization as well as threshold current measurements of the lasers are correlated with X-ray diffraction and transmission electron microscopy relaxation status results.

## 1. Introduction

Strained layer semiconductor structures have received considerable attention in the last few years. Although use of strain can, in principle, improve device performances (e.g. lower threshold current in quantum well (QW) lasers), it is well known that design of strained structures is limited by the thickness at which the formation of dislocations becomes energetically favourable (critical thickness) and degradation of crystalline quality takes place [1]. Recently, single ultrathin strained layers of InAs as well as short period InAs/GaAs superlattices (with a 7.16% misfit) have been tested as an alternative to $\mathrm{Ga}_{1-x}\mathrm{In}_x\mathrm{As}$ alloys in order to obtain higher quality material. Several groups have reported results about devices using structures of this kind [2]. We have used X-ray diffraction (XRD) analysis combined with simulation using dynamical theory of diffraction as a non-destructive, post-growth means of evaluating the indium content, a key point for the design of these devices [3] and the relaxation process due to increasing total content of In in the QW. Transmission electron microscopy (TEM) is used to confirm the relaxation status obtained by XRD and to study the defect structure in samples of this kind.

## 2. Experimental procedure

Two types of samples were studied: laser structures and quantum well structures. The structure of the laser samples is as follows: (001) GaAs:Si substrate, 0.5 μm $\mathrm{n^+}$-GaAs buffer layer, 1 μm $\mathrm{n^+}$-$\mathrm{Al_{0.6}Ga_{0.4}As}$ ($n \sim 5\times10^{17}$ cm$^{-3}$) cladding layer, 1000 Å undoped $\mathrm{Al_{0.2}Ga_{0.8}As}$ waveguide,

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80 Å undoped quantum well that differs from sample to sample, 1000 Å of undoped $Al_{0.2}Ga_{0.8}As$, 1 μm of $p^+$-$Al_{0.6}Ga_{0.4}As$ ($p \sim 5 \times 10^{17}$ $cm^{-3}$) and 1500 Å $p^{++}$-GaAs ($p \sim 5 \times 10^{18}$ $cm^{-3}$) contact layer. The five different strained wells in the laser structures consist of 100 Å of $Ga_{0.8}In_{0.2}As$ (sample A) and $N$ ML of InAs ($N = 1, 3, 5, 7$) (samples B, C, D and E) separated from each other by either 2 or 3 monolayers (ML) of GaAs. The samples that consist only of strained QWs are nominally $P$ ML of InAs ($P \times (InAs)_1(GaAs)_Q$ with $P = 2$, 4 and $Q = 2$, 4).

The samples were grown at low temperature (350°C) by a development of the conventional molecular beam epitaxy (MBE) technique named atomic layer MBE (ALMBE) [4]. The main feature of ALMBE is a periodic perturbation of the growing surface by means of the cyclic modulation of the molecular beams which enhances two-dmensional growth kinetics.

High resolution X-ray diffraction measurements were performed by means of a double-crystal diffractometer around the (004) GaAs reflection. Experimental scans were compared with calculated diffraction patterns [5] using the dynamical theory of X-ray diffraction. In this way we determined the structural parameters of the different layers such as thickness, chemical composition and strain status.

Cross-section specimens were prepared for TEM study using the conventional sandwich technique. Two transmission electron microscopes, a JEOL 2000 EX and a JEOL 1200 EX, were used for carrying out this study. High resolution electron microscopy (HREM) and difraction contrast were used to examine the quality of the QWs and the defects which are present in the studied specimens. The material was studied in regions for which contrast due to superficial relaxation associated to thinning [6] does not appear.

## 3. Results and discussion

In structures of this kind, a characteristic modulation of the intensity (Pendellösung) due to the interference of X-ray waves inside the crystal is predicted by the simulation. Among the samples with the laser structure we observe that this modulation exists in the samples containing 10 nm of $Ga_{0.8}In_{0.2}As$ (sample A), 1 and 3 ML of InAs (samples B and C, fig. 1a) in the laser active region. For the sample with 5 ML of InAs (sample D) this modulation is not observed and the background intensity is normal. In the sample with 7 ML of InAs (sample E, fig. 1b) no Pendellösung is observed and the background intensity is much higher than normal. Chemical composition and thickness of the $Al_xGa_{1-x}As$ alloy layers and the InAs content found were very

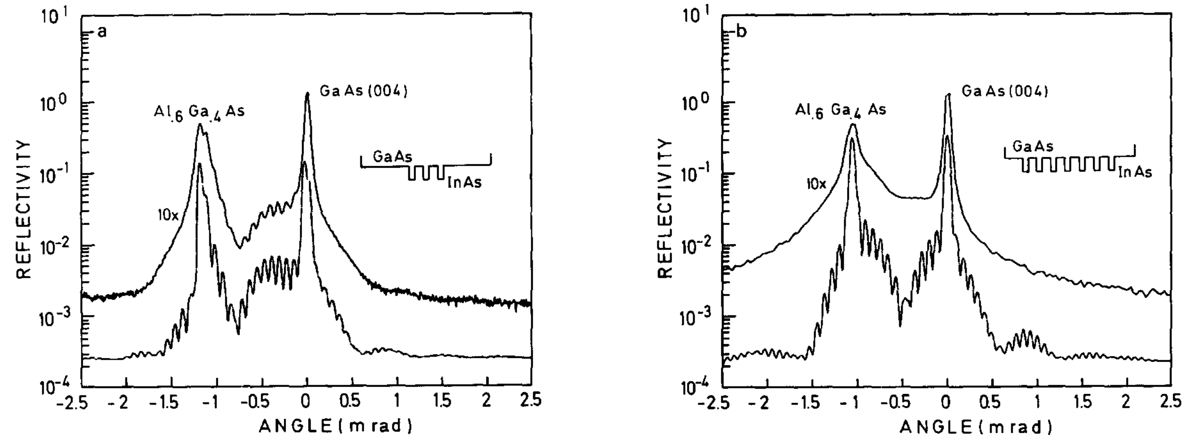

Fig. 1. Experimental (———) and simulated (– · –) diffraction pattern of a laser structure containing 3 ML (a) and 7 ML (b) of InAs in the quantum well. Experimental diffraction patterns have been shifted for clarity.

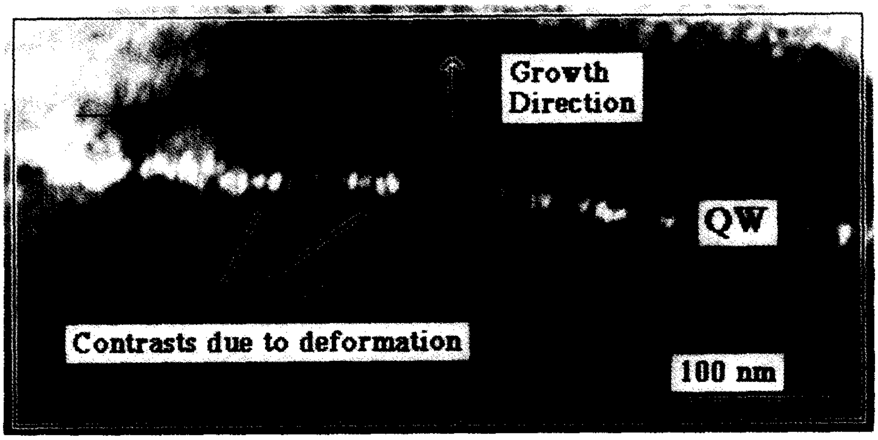

Fig. 2. Dark-field (220) image taken in the QW region of laser with 7 ML of InAs. Contrast due to deformation in the QW appears.

close to the nominal values. In all the samples that consist only of the strained QWs, interference fringes were always observed. In this case the relaxation status can be assessed by measuring the angular distance between interference peaks at both sides of the substrate peak [7].

The relaxation status of the laser samples (A–E) found by TEM is in agreement with the XRD results: samples A, B and C were found to be defect free, while defects appeared for sample D ($N = 5$ ML InAs) with a dislocation density $n_d \approx 10^7$ cm$^{-2}$ and in sample E ($N = 7$ ML InAs) with

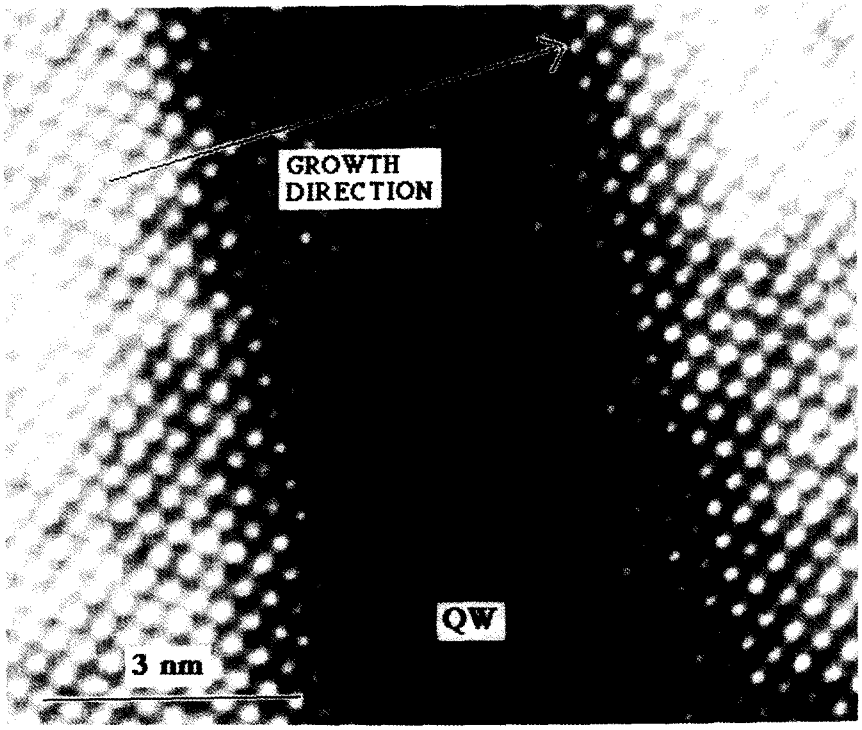

Fig. 3. HREM image showing that QW possesses flat and well-defined interfaces.

$n_d \approx 10^8$ cm$^{-2}$. The sample with 7 ML InAs shows a particular defect distribution. Groups of defects with triangular shape appear to be disposed with a certain periodcity (period ≈ 3 μm). Dark field (220) images obtained from this sample show much contrast probably due to deformation in the region of the interface (fig. 2). Some vicinality has been observed by HREM for the substrate surface in strained layer laser structures; the inclination of the substrate surface in relation to the (001) plane becomes greater than 2° in some regions. The other set of samples, strained layer QWs, showed completely flat and well-oriented interfaces.

The values of the strain status obtained by XRD in this study have been plotted in a thickness versus misfit diagram (fig. 4). Matthews and Blakesle's critical thickness theory [1] for a single heterojunction has also been plotted for comparison. The agreement of experimental XRD data with the Mathews–Blakesle model is good when the misfit is larger than 1.5%. The dislocation density found by TEM in sample J (fig. 4) is much lower than the other relaxed samples. The dislocation density estimated is lower than $10^7$ cm$^{-2}$.

The chemical composition and relaxation status of the different structures obtained by XRD are shown in fig. 5. The $X$ axis corresponds to the total InAs content in the structure. The $Y$ axis corresponds to the GaAs thickness between two InAs layers. Structures found relaxed have been plotted by stars and structures found strained have been plotted by circles. Data marked by arrows have been taken from previous work [8]. A clear relationship between the GaAs/InAs thickness ratio and the relaxation status is found, as expected. For the separation between the region of relaxed and strained samples, the relation $Q = 1.26(P - 2.3)$ holds, where $Q$ represents the GaAs barrier thickness and $P$ the total InAs content. A study of similar samples to find out whether this relation holds for a wider range of GaAs/InAs thicknesses is under way.

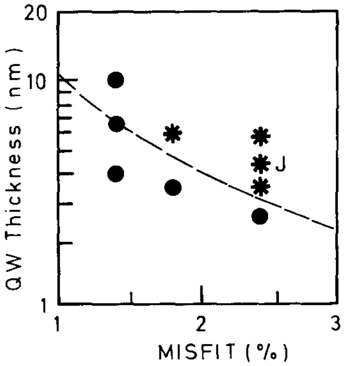

Fig. 4. Plot of the values of QW thickness versus misfit. Dashed line corresponds to theory of Matthews and Blakeslee [1]. Sample J has been marked (see text).

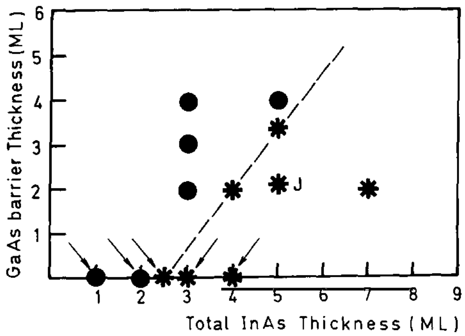

Fig. 5. Plot of the GaAs barrier thickness versus total InAs content. The straight line corresponding to $Q = 1.26(P - 2.3)$ separates the relaxed and the strained sample regions. Sample J has been marked (see text).

Characterization of the samples as laser devices [9] shows that samples with $Ga_{0.8}In_{0.2}As$ (sample A), 1 ML InAs (sample B) and 3 ML InAs (sample C) have good laser characteristics (e.g., $J_{th} = 1.97$ kA cm$^{-2}$ and $\lambda = 884$ nm for sample B), while the sample with 5 ML InAs shows poorer performance and the sample with 7 ML InAs does not lase, as expected by the high density of dislocations in the active region. In addition, the agreement between the transitions observed in the optical measurements and the results of an envelope wavefunction calculation is very good for coherently strained lasers (samples A, B and C). Transition for the relaxed laser samples cannot be fitted with the nominal structural parameters (thicknesses and compositions) considering an in-plane parameter equal to that of the substrate. The fitting improves qualitatively, considering the relaxation status found by XRD.

## 4. Conclusions

The relaxation process in lasers and QW with InAs layers has been studied by XRD and TEM. Strain status is correlated with optical properties and characterization of the laser devices. Good agreement with the critical thickness theory of Matthews and Blakeslee is found for misfits larger than 1.5%.

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