Growth and x-ray characterization of strain compensated GaAs/AlAs distributed Bragg reflectors

A. Mazuelas,[a] H. Nörenberg,[b] R. Hey, and H. T. Grahn[c]

Paul-Drude-Institut für Festkörperelektronik, Hausvogteiplatz 5-7, D-10117 Berlin, Germany

We have grown strain compensated GaAs/AlAs distributed Bragg reflectors by solid source molecular beam epitaxy using carbon doping densities up to about 2×10^{20} cm^{-3}. The residual strain with respect to the GaAs substrate can be as low as 1×10^{-4}. This results in a large increase of their critical thickness with regard to the undoped case. We demonstrate that simulations of the x-ray diffraction patterns are essential in order to determine the chemical profile as well as the structural parameters of the GaAs:C and AlAs:C layers with high accuracy. The effective incorporation of carbon on lattice sites is in AlAs:C twice as large as in GaAs:C using the same incident carbon flux. © *1996 American Institute of Physics.* [S0003-6951(96)01106-7]

Carbon is increasingly substituting Be as a *p*-type dopant of Al$_x$Ga$_{1-x}$As layers due to its extremely low diffusivity.[1,2] Several investigations deal with the incorporation of C in GaAs,[1–8] while other reports have focused on the device application of Al$_x$Ga$_{1-x}$As:C.[5,9–11] The C doping is achieved with a wide range of growth techniques, among them: solid source molecular beam epitaxy (MBE),[1,4,12–16] metalorganic MBE,[3,17,18] chemical beam epitaxy,[19] and gas phase techniques.[2,7,9,10] In addition to its large hole concentration, carbon doping of GaAs and AlAs produces also a strong contraction of the crystal lattice. This is due to the significantly smaller covalent radius of carbon compared to the covalent radii of As, Ga, and Al. As an important application of carbon doping of GaAs and AlAs, we propose thus the synthesis of *p*-type GaAs/AlAs strain compensated distributed Bragg reflectors (DBR) on GaAs substrates, which represent the *p*-type mirror in vertical cavity surface emitting laser (VCSEL) structures. The strain compensation should result in higher quality DBRs, since above a certain total thickness and/or Al composition the lattice mismatch in Be doped and undoped DBRs produces a degradation by relaxation of the structure.[20]

X-ray diffraction (XRD) is widely used to assess the chemical composition, the strain status and the structural quality of epitaxial materials. Its use as a standard tool to study dopants is not so widespread since the concentration of dopants is always much smaller than the concentration of the host material. Furthermore, the strain which is produced by doping is generally very small. Nevertheless, as shown previously,[3] XRD can be used in Al$_x$Ga$_{1-x}$As:C to determine the concentration of carbon on lattice sites (C_{As}).[21]

In this letter, we demonstrate that carbon doped DBR with full strain compensation with respect to the substrate can be grown by MBE. We also show that simulations of the XRD patterns constitute an essential tool to determine the chemical profile including the dopant in complex heterostructures such as DBRs.

We used a resistively heated graphite filament as the carbon source. It was operated near its maximum power at around 570 W. GaAs/AlAs layers were grown by solid source MBE on (001) GaAs substrates at a substrate temperature of 505 °C and beam equivalent pressure (BEP) ratio of 15 for GaAs. The growth rate was 1 μm/h for GaAs and 0.2 μm/h for AlAs. The XRD measurements were performed in a double crystal diffractometer using the Cu $K\alpha_1$ line around the (004) reflection. The simulations of the XRD patterns were performed using a fully dynamical model.[22] The total carbon concentration C_T was determined by secondary ion mass spectrometry (SIMS).

Previously, we optimized the C incorporation in GaAs and AlAs singles layers making extensive use of XRD as a characterization tool.[16] The ratio between the on lattice carbon concentration C_{As} and the total carbon concentration C_T yields the effective incorporation of carbon on lattice sites ($\beta = C_{As}/C_T$), while the ratio between the hole concentration and the carbon on lattice sites yields the electrical activation of the dopant. Both parameters are essential to understand the behavior of the dopant.

In this work we focus our attention on the XRD characterization of doped DBRs. In order to act as an efficient mirror, the DBR must fulfill extreme demands in terms of the thicknesses of the individual layers and the periodicity of the superlattice (SL). For device applications such as VCSELs with doped DBRs, a nondestructive characterization technique capable of assessing these structural properties including the strain tuning by doping with great accuracy is essential.

We show in Fig. 1 the XRD pattern of an undoped 10 period DBR with 70.7 nm GaAs and 80.6 nm AlAs for an operating wavelength of about 980 nm. In the case that the DBR is composed of two binary semiconductors, XRD determines uniquely the average strain, the period of the SL, and the thicknesses of the individual layers. The period of the angular positions of the SL peaks allows the determination of the SL period (Table I). In determining the chemical composition, a certain ambiguity remains in the assignment of the zeroth order peak, which corresponds to the effective lattice constant of the crystal. This ambiguity can be removed by comparing the XRD determination with other techniques, e.g., the calibration of the effusion cell fluxes as

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[a] Electronic mail: mazuelas@pdi.wias-berlin.de
[b] Present address: University of Oxford, Department of Materials, Parks Road, Oxford OX1 3PH, United Kingdom.
[c] On leave at: Tokyo Institute of Technology, Research Center for Quantum Effect Electronics, 2-12-1 O-okayama, Meguro-ku, Tokyo 152, Japan.
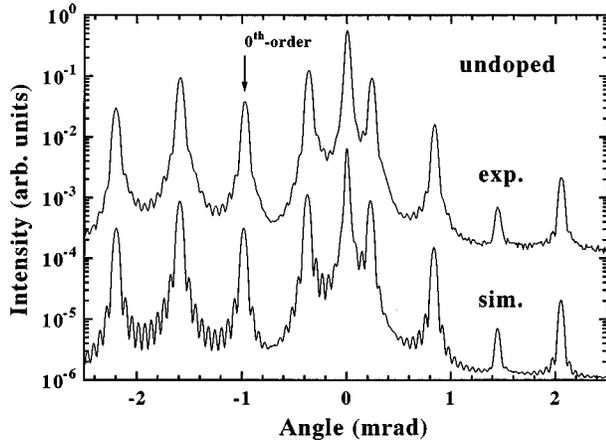
FIG. 1. Experimental (exp.) and simulated (sim.) XRD patterns of an undoped 10-pair GaAs/AlAs DBR (sample A) measured around the GaAs (004) reflection. The parameters are listed in Table I. The position of the 0th-order peak refers to the effective lattice parameter of the SL.
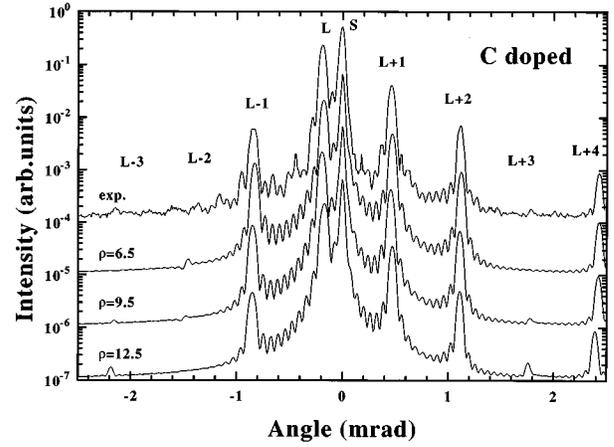

FIG. 2. Experimental and simulated XRD patterns of a doped 10-pair GaAs:C/AlAs:C DBR (sample B), which is nearly strain compensated. The simulations were made by varying the spatial distribution of carbon within one period $\rho = C_{As}(AlAs)/C_{As}(GaAs)$. The parameters for the best fit are listed in Table I.

determined by the oscillation period of the specular beam by means of reflection high-energy electron diffraction. However, a more powerful tool to determine these parameters is the simulation of the XRD experimental patterns. In this case, the assignment of the SL zeroth order peak (and consequently the determination of the chemical profile) becomes unambiguous. Furthermore, it is possible to assess the crystalline perfection. The comparison between the experimental and simulated pattern in Fig. 1 demonstrates that the crystalline quality of the undoped DBR is very high. The sharp peaks also indicate a perfect periodicity along the growth direction. The determined value of the period was only 1% larger than the intended one. Several measurements were carried out along the wafer radius and good homogeneity was observed.

The comparison of experimental and simulated XRD curves becomes essential for the full characterization of doped AlAs:C/GaAs:C DBRs. The average compound is the quaternary AlGaAsC, for which all the structural parameters such as thicknesses and composition cannot be determined from the position of the peaks alone. In the following, we will show that the simulation of the XRD patterns leads to an exact determination of the composition profile including the dopant with a concentration between 10^{19} cm^{-3} and 10^{20} cm^{-3} (between 0.1% and 1% compared to the concentration of As).

The angular spacing between the SL satellites determines the period of the DBR, while the position of the zeroth order of the SL is linked to the Al/Ga and As/C ratios through the net strain they produce. There is another parameter, which only influences the intensity distribution within the XRD pattern, the spatial distribution of carbon within one period. This can be defined through the parameter $\rho = C_{As}(AlAs)/C_{As}(GaAs)$. Figure 2 shows the results of the simulations for different values of ρ keeping the Al concentration X_{Al} fixed at 0.58. The best agreement between the experimental and simulated curves occurs for $\rho = 9.5$. Note that the satellites $L-2$, $L-3$ and $L+3$ are simultaneously low in intensity. Their strength is very sensitive with respect to the actual value of ρ. When the simulations are performed varying ρ over a wide range ($4 \leqslant \rho \leqslant 22$) for $X_{Al} = 0.54$, no satisfactory agreement with the experimental curve could be obtained. Varying X_{Al} for different values of ρ leads to similar results. The parameters of the best simulation are listed in Table I. As the growth rate for GaAs was five times larger than for AlAs, one would expect $C_{As}(AlAs)$ to be five times larger than $C_{As}(GaAs)$. However, we find a ratio of 9.5. We attribute this difference ($\rho = 9.5$ instead of 5) to a different on lattice C incorporation in AlAs and GaAs for a common incident carbon flux [$\beta(AlAs) = 1$ and $\beta(GaAs) = 0.5$].

The net reduction of the average strain due to the carbon doping is obvious from the shift of the zeroth order peak, which is only 0.183 mrad apart from the substrate peak. This value corresponds to a strain ϵ_{zz} of 2.82×10^{-4} [$\epsilon_{zz} = (a_s - a_e)/a_s$], where a_s and a_e refer to the lattice parameters in the growth direction of the substrate and epitaxial layer, respectively). The critical thickness corresponding to this strain is around 6 μm according to the Matthews and Blakeslee model.[23] This is a large increase in comparison to the critical thickness ($h_c \approx 1 \mu$m) for an average Al composition of 54% in the corresponding undoped DBR.

Proper adjustment of the carbon concentration leads to DBRs with negligible strain. We have grown several DBRs with strain below $\epsilon_{zz} = 1 \times 10^{-4}$. In Fig. 3 the XRD pattern of one of the fully strain compensated DBR is shown. The structural quality is again very high. In the case of negligible strain, the corresponding critical thickness diverges as the misfit approaches zero. This means that it is possible to grow

TABLE I. Structural parameters of several DBRs obtained from the simulations of the XRD patterns. The values listed are the thickness (d), the on lattice site carbon concentration in GaAs and AlAs, and the perpendicular strain (ϵ_{zz}).

Sample	d_{GaAs} (nm)	d_{AlAs} (nm)	$C_{As}(GaAs)$ (cm^{-3})	$C_{As}(AlAs)$ (cm^{-3})	ϵ_{zz}
A	70.7	80.6	—	—	1.50×10^{-3}
B	59.0	81.5	1.6×10^{19}	1.5×10^{20}	2.82×10^{-4}
C	69.3	81.4	2.0×10^{19}	1.8×10^{20}	8.30×10^{-5}

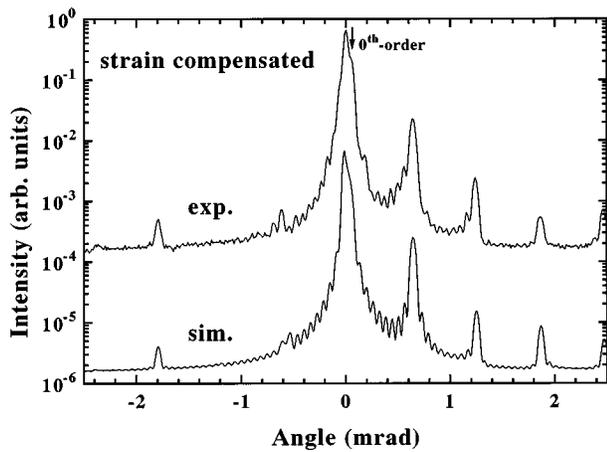Appl. Phys. Lett., Vol. 68, No. 6, 5 February 1996

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FIG. 3. Experimental and simulated XRD patterns of a 10-pair doped GaAs:C/AlAs:C DBR (sample C), which is strain compensated with respect to the substrate. The parameters are listed in Table I.

unrelaxed DBRs with much larger thickness compared to the undoped or Be doped cases. For the DBR shown in Fig. 3, the calculated critical thickness is around 20–30 times larger than for the undoped DBR. We have experimentally verified this increase in the critical thickness for DBRs with 20 periods.

The simulation shown in Fig. 3 for the perfectly strain compensated DBR leads to the same conclusion with regard to the lower incorporation of C on lattice site in GaAs. The very good agreement between the experimental curve and the simulated patterns for both DBRs (Figs. 2 and 3) proves that the assumption that the carbon doping is perfectly periodic along the growth direction is realistic. This means that C_{As}(AlAs) and C_{As}(GaAs) remain constant throughout the DBR which is also confirmed by the SIMS measurements.

In order to support the results of a lower incorporation of carbon on lattice sites in GaAs as determined from the XRD measurements, we performed SIMS measurements on doped DBRs and on thick AlAs:C and GaAs:C single layers. For example, in sample C, SIMS gives a total carbon concentration in GaAs of C_T(GaAs)$=3.7\times10^{19}$ cm^{-3} and in AlAs of C_T(AlAs)$=1.8\times10^{20}$ cm^{-3}. While in AlAs the value of C_T agrees well with C_{As} as determined by XRD, the total carbon concentration in GaAs is about a factor of two larger than the carbon concentration on lattice sites.

In summary, GaAs:C/AlAs:C DBRs were grown by solid source MBE using a graphite filament with on lattice site carbon concentrations of 2×10^{19} cm^{-3} in the GaAs:C and up to 1.8×10^{20} cm^{-3} in the AlAs:C layers. For these carbon concentrations strain compensated DBRs ($\epsilon_{zz}\leq1\times10^{-4}$) have been realized thereby increasing their critical thickness. We demonstrate that simulations of XRD patterns are essential to determine the chemical profile of highly doped DBRs nondestructively and with high accuracy. For the same incident carbon flux, the effective incorporation of carbon on lattice sites is larger by a factor of two in AlAs:C than in GaAs:C.

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