

Proc. of the XXVII Intern. School on Physics of Semiconducting Compounds, Jaszowiec 1998

HIGH RESISTIVITY AlGaAs GROWN BY LOW TEMPERATURE MBE

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Al_{0.3}Ga_{0.7}As layers were grown by molecular beam epitaxy using substrate temperature 200–300°C, tetrameric As and two values of As/Ga+Al flux ratio i.e. 3 or 8. The post-growth annealing was performed *in situ* at 600°C for 20 min under As-overpressure. The samples were characterised by reflection high-energy electron diffraction, transmission electron microscope and room-temperature *I*–*V* measurements of *n*⁺/LT grown layer /*n*⁺ resistors. The resistivity and trap-filled limited voltage have been determined. The best layers exhibited ρ of the order of 10⁹ Ω cm, were monocrystalline, uniformly precipitated and without dislocations.

PACS numbers: 73.61.Ey

1. Introduction

It is commonly known that molecular beam epitaxy (MBE) of GaAs and AlGaAs at significantly reduced substrate temperature (200–300°C) results in non-stoichiometric material with 1–2% excess As atoms [1, 2]. An incorporation of As into the crystal lattice is strongly dependent on the growth conditions. Upon a proper thermal treatment, excess arsenic precipitates and low temperature (LT) grown layers become semi-insulating. Compared to SiN_x and SiO₂ they present much better matching of the thermal expansion coefficient and thermal conductivity to GaAs and are suitable for overgrowth. These make LT GaAs and AlGaAs potentially very useful for current-blocking layers in electronic and optoelectronic devices. In addition, LT AlGaAs as a material with low refractive index would be able to provide in combination with GaAs both electrical isolation and waveguiding effect.

This study was undertaken in order to establish MBE growth conditions for obtaining AlGaAs layers having good insulating properties, without extended defects and well oriented surface. This is not a trivial task because of the critical thickness for dislocations formation and the lack of a precise method to measure substrate temperature in the temperature range below 450°C. We have continued our earlier works on low temperature grown GaAs [3, 4].

2. Experiment

The layers used in this study were grown in MBE Riber 32P system on (100) oriented n^+ -GaAs substrates with a tetrameric arsenic source. The typical growth rate of $1 \mu\text{m/h}$ was used, calibrated at 590°C from reflection high-energy electron diffraction (RHEED) oscillations. The substrates were mounted to molybdenum blocks using indium solder. Prior to the growth the wafers were outgassed at 430°C without arsenic for 30 min and the oxide was desorbed by heating under As overpressure up to 630°C . The $0.5 \mu\text{m}$ thick GaAs:Si ($2 \times 10^{18} \text{ cm}^{-3}$) buffer was grown followed by the 10 nm GaAs:Si ($2 \times 10^{16} \text{ cm}^{-3}$) spacer. Then the growth was terminated and substrate temperature decreased to the low temperature range $200\text{--}300^\circ\text{C}$. The undoped $0.5 \mu\text{m}$ $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ layer was deposited. We chose two values of the group V to group III flux ratio 3 or 8. The growth was interrupted once again and samples were heated while temperature reached 600°C . LT layer was annealed for 10 min under As overpressure to ensure (2×4) As-stabilised surface and then capped with the 10 nm thick GaAs:Si ($2 \times 10^{16} \text{ cm}^{-3}$) spacer and 200 nm of GaAs:Si ($2 \times 10^{18} \text{ cm}^{-3}$) contact layer. Thus, the total time of post-growth annealing treatment was 22.5 min.

To test the role of interfaces GaAs/AlGaAs in complex structures, a certain modification of the geometry of LT region was introduced in the sample # 261 (Fig. 1).



Fig. 1. Transmission electron microscope (TEM) cross-section of the sample #261. The LT region consists of three LT $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ layers separated by LT GaAs. The critical thickness is exceeded. Apart from precipitates, the dislocations are seen.

Resistors were made by forming top and bottom Au/Ge/Ni alloyed ohmic contacts and defining by standard photolithography and dry etching circular mesas of diameter of $200 \mu\text{m}$.

The big problem in MBE system for III-V compounds is precise substrate temperature control for the low temperature range. Below 450°C an infrared pyrometer does not work and reading of the temperature is possible only by means of the thermocouple which is placed at the bottom of molyblock. We have calibrated our thermocouple against indium melting point (157°C) and by an infrared Ircon-V type pyrometer in the range $450\text{--}630^\circ\text{C}$. The calibrations were done for each of used molybdenum blocks. An influence of the opened Ga and Al effusion cells has been taken into account. We have found that at 200°C a substrate temperature rise due to the radiation from Ga and Al cells is $55\text{--}75^\circ\text{C}$ depending on the state of molyblock.

During the growth, the surface of the samples has been probed during using RHEED. We observed the following evolution of a diffraction pattern. The layers grown at standard temperatures, 585 and 600°C, exhibited the standard (2×4) reconstruction. On cooling down, the reconstruction changed into (2×2) at about 480°C. As soon as the group III beams were switched on, the surface lost immediately the reconstruction and showed the (1×1) pattern. After closing the Al and Ga shutters the twofold reconstruction returned. Annealing caused a recovery of the clear (2×4) pattern suggesting an improvement in the crystal quality. The layers grown at the highest of chosen temperatures i.e., 300°C, demonstrated the (2×4) pattern before annealing.

3. Results and discussion

The current blocking properties were determined by resistivity and trap-filled limited voltage (an intersection point of the linear and "trap-filled" regions of the log-log $I-V$ curve) extracted from room-temperature current-voltage characteristics. These parameters and the growth conditions are compared in Table. Generally, LT AlGaAs shows three orders of magnitude higher resistivity than that obtained for LT GaAs.

TABLE

Comparison of crystal and electrical properties of the resistor structures including the LT $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ layer grown at different substrate temperatures and III/V flux ratios.

Sample	Growth temp. [°C]	V/III flux ratio	Thickness of LT layer [nm]	ρ at 2.5 V [Ω cm]	V_{tf} [V]
#254	200	3	500	4.2×10^7	11
#261	220	8	308	1.1×10^8	6*
#255	250	3	500	3.0×10^7	17
#256	250	8	500	5.9×10^7	17
#265	300	3	500	8.5×10^8	10
#266	300	8	500	3.7×10^9	10

*Note that the LT layer is thinner than in other structures.

Some contradiction with respect to ρ and V_{tf} is seen. The 250°C grown layers exhibit much higher V_{tf} than the 300°C grown ones, but lower resistivity. The same behaviour have been observed by Verma et al. [5] and was explained by residual hopping conduction in the material grown at 250°C.

In the TEM pictures (Fig. 2) As precipitates in the buffer layer are visible. This is the evidence that the arsenic diffusion occurred during the annealing. To prevent this, AlAs confining layers or AlAs/GaAs superlattices should be applied to inhibit this phenomenon [6].



Fig. 2. Cross-sectioned TEM picture of the sample #265. The interfaces are planar and the layer uniformly precipitated. Extended defects are not present. Note the precipitates in the buffer layer.

We have not done detailed investigations of the critical thickness for dislocation formation. However, we have observed that when the growth temperature exceeded about 230–240°C an abrupt increase in the critical thickness occurred (from about 30 nm at 220°C to above 500 nm at 250°C for V/III flux ratio of 3). This is in agreement with earlier findings reported by Eaglesham et al. [7].

The values of ρ and V_{th} from Table and I – V characteristics (not presented here) point out that all LT AlGaAs layers obtained in our experiments are effective in providing the current blocking. However, only those grown at 250°C and 300°C have structural quality suitable for device applications i.e. they are monocrystalline, uniformly precipitated, without dislocations and with the smooth surface.

4. Summary

$\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ layers were grown by molecular beam epitaxy at substrate temperatures 200–300°C, using tetrameric As and two values of As/Ga+Al flux ratio i.e. 3 or 8. Very high resistivities of the order of 10^8 – 10^9 Ω cm and high breakdown voltages: 10–18 V for 500 nm thick layers, have been obtained. This is the substantial improvement of insulating properties comparing to LT GaAs for which 10^6 Ω cm resistivity are typical. The best results in terms of electrical parameters have been obtained for the growth temperature of 250°C and the highest V/III flux ratio. The crystal quality of the layers was monitored *in situ* by RHEED. Transmission electron microscope studies showed that LT AlGaAs is monocrystalline, uniformly precipitated and free of extended defects and dislocations.

Acknowledgment

This work was supported by grant no. 8T11B00712 and PBZ28.11/P7 of the Committee for Scientific Research.

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