SELECTIVELY ETCHED SERIES OF p-n JUNCTIONS IN GaAs

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Received 7 April 1995

UDC 538.971

PACS 68.65.+q

Simultaneous S and Zn doping was applied to obtain multi-layered vapour phase epitaxial structures of GaAs. SIMS studies have shown that the simultaneous presence of S and Zn in the gas phase does not influence the incorporation of S or of Zn during the growth process. Epitaxial structures with n-p-n-p-n-p-n series of layers have been obtained by variation of S doping at constant Zn partial pressure. The layer thicknesses were 0.3 and 3 μ m. SIMS profiles show good abruptness of the junctions, so n-type GaAs lamellae have been obtained by selective electrochemical etching from such layered structures. Results show a possible way for fabrication of micro–machined devices from GaAs.

1. Introduction

Micro–machining, fabrication of micro–sensors, micro–actuators and similar semiconductor devices, and micro–vacuum–devices, require very thin sheets and lamellae. The dimensions are usually smaller than what can be achieved by mechanical treatment. Therefore, mainly chemical and electrochemical methods are

used. These methods are based on selective etching of some layers, some parts of the "bulk" of a semiconductor structure, for example, exploiting the etching rate dependence on crystalline orientation or doping differences. These chemical (mainly wet chemical) methods are very well developed and wide-spread in silicon practice, in Si based micro-machining [1].

Much less work has been devoted to micro-machining in the practice of the compound semiconductors. Sensors and actuators developed on the basis of III-V semiconductors are processed also using some selective wet etching, but the selectivity is based on the composition differences. In one wafer ternary (multinary) compounds are combined with binary compositions (e.g. GaAlAs or GaInAs with GaAs, etc.) [2]. Presence of Al and, in general, ternary (multinary) compounds require sophisticated, expensive technologies (like MBE, MOCVD), and the method limits the possibilities of using simple alloys for such purposes. It should be stressed that GaAs is one of the most promising materials for micro-sensor applications.

In this work, GaAs and combination of GaAs layers of different conductivity types have been used for preparation of very thin sheets, lamellae and needles using the most conventional chloride type vapour phase epitaxial method.

2. Experimental

In the experiments an Effer-Nozaki type $AsCl_3/Ga/H_2$ open tube system was used to grow epitaxial layers and structures of GaAs [3]. Our reactor uses one doping line. S doping can be performed from SF_6 +He mixture in a wide doping concentration range $(1 \times 10^{15} - 1 \times 10^{19} \text{ cm}^{-3})$. Temperature of the source was kept in the range of 800–820 °C, the deposition temperature of 730–740 °C. (100) GaAs wafers were used as substrates misoriented to 3–4°.



Fig. 1. (a) Scheme of the VPE reactor with a divided deposition zone and an additional solid source and (b) the temperature profile in the reactor tube.

For simultaneous doping, additional GaAs solid sources were used above the Ga melt source. They were placed in the source region. Figure 1 shows the scheme of the arrangement of the reactor tube. The etching/transport rate of this additional

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solid source can be controlled both by its position (temperature + HCl partial pressure) and by the second (bypass) line of $AsCl_3$. Dilution can be varied by the main H₂ line. This arrangement allowed to dope the growing layer simultaneously both by S and by the dopant of the additional GaAs source. In these experiments, highly doped GaAs:Zn was used as secondary source to obtain Zn doped layers. The two-level deposition zone allowed the use of different substrate materials (e.g. n⁺⁺ and semi-insulating GaAs) in the same run, avoiding some cross-diffusion processes [4]. The doping level assured by the additional solid source is constant during the run.

Mono– and multilayered GaAs epitaxial structures have been grown. The level of S doping was varied. Structures with a series of p-n junctions have been grown. Samples cut out from such structures underwent selective electrochemical etching by tiron. The samples have been attacked by the etchant on their cut front end. Electric contacts have been formed to each layer of the epitaxy on the other end of the sample. An appropriate voltage for anodic current, between zero and 3 V, was chosen for each sample. p type layers in the structure have been etched out due to their hole conductivity [5].

3. Results and discussion

Distribution of the impurities in depth of the epitaxy has been evaluated by SIMS measurements. Figure 2 shows a typical S and Zn distribution through the layers. (SIMS measurements have been performed with a CAMECA ims3f apparatus using Cs^+ and Cs^- sputtering ions for Zn and S, respectively. These two separate profiles are combined into one in Fig. 2). The depth profile shows clearly



Fig. 2. Distribution of the dopants through the epitaxial layers of the sample 3338i-1 doped with Zn on a constant level and S of varying concentration, measured by SIMS.

that the Zn concentration remains constant, while the S concentration varies in steps "on and off". Differences of the S signal levels in the "valleys" are mainly due to the diffusion processes during the growth process [4]. Perhaps ion mixing during the sputtering plays also role, though perpendicular ion incidence was used, but with high energy and on great surfaces of $150\mu m \times 150\mu m$. The abruptness and the "depth" of the S concentration steps are sufficiently good for obtaining thin layered p-n-p-n structures.



Fig. 3. (a) The scheme of the epitaxial structures and the etching direction before and (b) after the etching.



Fig. 4. (a) A scanning electron microscopic view of the end face of the sample 3409n-3. Two p-layers have been etched way. The two parallel thin black lines show the grooves between the n-layers on the general view (\times 75, 0°) and (b) the character of the grooves is seen on its detail (\times 500, 0°).

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Different structures have been grown with p-n regions and cut into "bricks". The ends of such pieces have been treated in proper electrochemical etchant, as shown in Fig. 3a. The etching removed the p-type layers from amidst the n-type regions (Fig. 3b). The width and the length of the lamellae were up to several mm. The thicknesses were varied.

Figure 4 shows a bird's eye view of the sample 3409-n-3 in scanning electron microscope (SEM, JEOL). The epitaxial structure of this sample consists of three n-type layers and two p-type layers in-between. The thickness of the etched out p layers was about 3 μ m. Fig. 4a shows a "total" view of the end face of the sample, while Fig. 4b shows a detail. Figure 5 shows the same sample, but a close view of the edge of the lamellae. Figure 5a is the view of the edges and the walls of the n sheets and Fig. 5b shows the corner of the sample.



Fig. 5. (a) Scanning electron micrographs of the etched 3409-n-3 sample, the edge $(\times 500, 15^{\circ})$ and (b) the corner $(\times 2000, 10^{\circ})$.

Figure 6 shows the grooves in the sample 3427-n-3. Four p-layers were grown of 0.27 μ m thickness. There is also a thicker ($\approx 2.5 \ \mu$ m) p-layer between the substrate and the n-type buffer ($\approx 3.5 \ \mu$ m) layer. Figure 6a shows a more general view, Fig. 6b shows the structure of the edges of the lamellae. The roughness of the edges of the thin (0.3 μ m) n-type sheets could be caused by the electrolyte and probably can be improved by technological conditions.

A needle/tip was cut out from one of the 3 μ m n-type lamella. The general view and the point of the needle are shown in Fig. 7. The radius of curvature at the point is smaller than 15 nm, although the length of the needle is about 1 mm.

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Fig. 6. (a) Scanning electron micrograph of the sample 3427-n-3, a general view $(\times 1500)$ and (b) the details of the submicron lamellae $(\times 10000)$.



Fig. 7. Scanning electron micrographs: (a) of a needle cut out from a 3 μ m thick GaAs sheet (x350) and (b) of the point of the tip (x5000). The background of the picture is the conducting silver paste used for fixing of the needle in the microscope.

4. Conclusions

The possibility of p-doping of VPE GaAs epitaxial layers have been demonstrated using GaAs:Zn wafers as solid sources, without modification of the conventional open tube system. The possibility of preparation of abrupt doping junctions (and, so, also p-n junctions) is assured by the absence of synergetic effects between the two dopants, S and Zn. No interaction between the impurities in the gas phase and in the growth process was observed, according to the SIMS measurements.

It was shown that a selective electrochemical etching can be carried out on GaAs structures with p-n junctions. As a result, n-type GaAs sheets/lamellae can be prepared. Successful etching experiments have been done down to submicron thicknesses (0.27 μ m). The surface area of such lamellae can reach several mm².

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Also, it was shown that tips with very small radius of curvature at the point of the tip can be fabricated from such thin GaAs sheets.

These results are very promising for the development of micro-machined devices based solely on GaAs. Possibility of fabrication of micro-sensors, manipulators, actuators and small radii for micro-vacuum electronics was demonstrated.

Acknowledgements

The authors are very indebted to their staff, to Mrs. E. Jakocska, Mrs. Gy. Kiss and Mrs. K. Szedlacsek. This work was supported partly by the Hungarian National Research Foundation (OTKA, grants No. T4178, F4135, E12012 and T15619) and from the loan of the National Committee for Technological Development (OMFB, contract No. 91-97-07-0316).

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SELEKTIVNO JETKANI NIZOVI p-n SPOJEVA U GaAs

Primijen
jeno je istovremeno dodavanje S i Zn tijekom rasta višeslojnih epitak
sijskih struktura GaAs iz plinovite faze. SIMS mjerenja ukazuju da prisutnost S i Zn tijekom rasta sloja ne utječe na njihovo ugrađivanje. Epitaksijske strukture sa n
–p–n–p–n–p–n nizom slojeva postignute su mijenjanjem dodavanja S pri stalnom parcijalnom tlaku Zn para. Debljine slojeva bile su 0.3 i 3 μ m. SIMS profili pokazuju oštre granice spojeva. Selektivnim elektrokemijskim jetkanjem postignute su lamele GaAs n–tipa.