54. IWK
Internationales Wissenschaftliches Kolloquium
International Scientific Colloquium

Information Technology and Electrical Engineering - Devices and Systems, Materials and Technologies for the Future

Faculty of Electrical Engineering and Information Technology

Startseite / Index:
http://www.db-thueringen.de/servlets/DocumentServlet?id=14089
ABSTRACT

Motivation for the presented work was to eliminate the necessity to use Pt counter electrode by adding an oxidizing agent of sufficient strength to the aqueous KOH solution. We studied photo-assisted electrodeless etching (ELPEC) of GaN in a K$_2$S$_2$O$_8$/KOH solution irradiated continuously with UV light. In our work we investigated the impact of mask material on GaN patterning. As mask material thin layers of Ti, Pt and Au were used. Details are given in experimental part. The ratio of mask covered surface area to uncovered one was 7:1, 1:2 and 1:5. The K$_2$S$_2$O$_8$ oxidizing agent concentration was kept in the range from 0.006 to 0.1 M; the KOH electrolyte concentration was kept in the range from 0.004 to 0.04 M.

1. INTRODUCTION

Group III-nitride semiconductors such as gallium nitride (GaN), aluminum nitride (AIN) and indium nitride (InN) offer many advantages for fabrication of electronic devices, such as Schottky diodes and high-electron mobility transistors which possess high breakdown voltages, high operating frequencies and high operation temperatures. They have opened a new era in the field of semiconductor materials and devices. They are all direct band gap materials with GaN to 6.2 eV (AlN) and form a complete series of ternary alloys which cover the whole visible spectrum (GaN) to 6.2 eV (AlN) and form a complete series of ternary alloys which cover the whole visible spectrum. They are all direct band gap materials with electron mobilities which enhance the formation of Ga$_2$O$_3$. As soon as the holes are consumed, there is an excess of electrons in the semiconductor. With time, any new holes created by UV light quickly recombine with the oversupply of electrons and further etching is significantly reduced.
However, presence of $K_2S_2O_8$ enables consuming the excess of electrons [4]:

$$S_2O_8^{2-} + 2e^- \rightarrow 2SO_4^{2-}$$

Authors [6] obtained a smooth surface by using a chopped UV source in electrodeless photo-electrochemical (ELPEC) etching of GaN.

2. EXPERIMENTAL

Two GaN epitaxial layers grown on a (0001)-oriented sapphire substrate by metal-organic chemical vapor deposition (MOCVD) were used in this study. One of them (A) consisted of 2.5 $\mu$m thick Si-doped ($n\sim4\times10^{17}$ cm$^{-3}$) GaN layer, the second one consisted of an undoped-GaN layer 930 – 990 nm thick covered by 20 nm thick AlN layer (B).

Non-annealed thick layer of Ti (70 nm and 100 nm), Au (110 nm) and Pt (115 nm) were used as etch masks. A 100 W filtered mercury-xenon lamp (EXFO Acticure® 40 000) with a light intensity of up to 160 mW/cm$^2$ was used. The light intensity was measured by wideband detector of UV radiation EXFO Radiometer R 5000. Etch depths were measured by Dektak 150 (Veeco), while the surface morphology after etching was characterised by scanning electron microscope (SEM) LEO 1550 with a resolution of 2 nm and also by optical microscope. The ELPEC etching was carried out in a standard electrochemical cell (Fig. 2). The samples were immersed in non-stirred solution incorporating $K_2S_2O_8$ and KOH. Solutions were made up freshly and all experiments were carried out at room temperature without electrical contact to the sample.

In the work we have used different ratios of covered and uncovered surface areas, namely 7:1 for mask a), 1:2 for mask c) and 1:5 for mask b), Fig. 1.

3. RESULTS AND DISCUSSION

To determine GaN optimal etching conditions we have used sample (A). As mask Ti (100 nm thick) was used. $K_2S_2O_8$ oxidizing agent concentration was kept in the range from 0.006 M to 0.1 M, KOH electrolyte concentration was kept constant at 0.004 M. At the source output a constant intensity of 6 mW/cm$^2$ was kept. We have found out that for the oxidizing agent concentrations from 0.03 M to 0.1 M homogeneous sample etching occurs. No trenches were observed in the vicinity of the mask (Fig. 3). For the oxidizing agent concentrations from 0.006 M to 0.03 M was the etched surface covered with whiskers.

3.1. Samples with other etched/unetched area ratios

Conditions achieved in previous experiments we have applied also for the sample (B). The aim was to get surface evenly etched over the whole area. Ti mask was deposited only at sample edges, therefore the covered/uncovered ratio was 1 : 4. At $K_2S_2O_8$ concentration of 0.1 M we were changing the KOH concentration in the range from 0.004 to 0.04 M. We have found out that at electrolyte concentration of 0.004 M whiskers were observed in the surface of the
sample (Fig. 4). Whiskers could be observed over the whole etched area. At electrolyte concentration of 0.04 M preferred etching of GaN could be observed near the mask. Surface at a distance of more than 100 µm was untouched.

If the mask/unmasked ratio is changed to 1:5, similar results as for the previous situation of 1:4 are achieved (Fig. 5).

Figure 4 SEM view of sample (B) surface with Ti mask etched with 0.004 M KOH and 0.1 M K$_2$S$_2$O$_8$. The ratio masked/unmasked is 1:4

Figure 5 SEM view of sample (B) surface with Ti mask etched with 0.025 M KOH and 0.05 M K$_2$S$_2$O$_8$. The ratio masked/unmasked is 1:5

3.2. The influence of mask material on etching rate and homogeneity

For etching conditions of 0.004 M of KOH and 0.05 M and 0.1 M of K$_2$S$_2$O$_8$ without stirring, sample (B) was etched with masks Ti (70 nm), Pt and Au. Mask covered to uncovered surface ratio was 1:2 (Fig. 1c). Source — sample distance was 8.5 and 4 cm with intensity at the source output of 6 mW/cm$^2$. Etching time was 30 min.

The lowest mask influence was observed for Ti mask. Pronounced etching took place for uncovered surface up to distance about 25 µm away from the mask edge (Fig. 6). In the area at mask edge the etching depth was about 85 nm. Uncovered surface far from the mask was etched less. Light intensity increase as the source — sample distance was decreased did cause faster etching but the 25 µm limit did not change. The K$_2$S$_2$O$_8$ concentration increase did not influence the etching rate significantly. Sample surface underneath the mask did exhibit some outgrowthed objects (Fig. 6).

A different situation occurs when Pt and Au are used as masks. If Au as mask is used with K$_2$S$_2$O$_8$ concentration of 0.05 M and KOH concentration of 0.004 M at sample/source distance of 8.5 cm, at close vicinity to metal edge, at about 2 - 3 µm distance GaN is completely removed up to the substrate. In the range from 5 to 60 µm from the mask edge whiskers are formed, their height is comparable with GaN thickness. For larger distances the etch rate is further decreased. At a distance larger than 150 µm the surface stays unetched. Light intensity increase achieved by bringing the sample closer to radiation source causes pronounced GaN etch rate increase over the whole unmasked surface (Fig. 7).

Figure 6 SEM view of sample (B) surface with Ti mask etched with 0.004 M KOH and 0.05 M K$_2$S$_2$O$_8$. The ratio masked:unmasked is 1:2

Figure 7 SEM view of sample (B) surface with Ti/Au mask etched with 0.004 M KOH and 0.1 M K$_2$S$_2$O$_8$. The ratio masked:unmasked is 1:2
Using Pt as mask the etch rate is high in the mask vicinity and sample is etched down to substrate. By distance increase (50 µm) the etching is homogenous. Further distance increase initiates whisker formation (Fig. 8).

Figure 8 SEM view of sample (B) surface with Pt mask etched with 0.004 M KOH and 0.1 M K$_2$S$_2$O$_8$.

The oxidizing agent concentration increase from 0.05 M to 0.1 M does not influence etching. Whereas the radiation intensity increase does increase the etching rate.

4. CONCLUSION

In this work we have studied n-GaN and i-GaN photoenhanced chemical etching with oxidizing agent. We have found out that:

Optimal etching conditions were set with sample (A). For masked/unmasked ratio of 7 : 1 the GaN surface is etched homogeneously over the whole area. In the concentration range of the oxidizing agent from 0.03 to 0.1 M and electrolyte concentration of 0.004 M neither trenches nor whiskers are formed, defects are not highlighted. For smaller oxidizing agent concentrations, whiskers are formed where defects occur. Changing the covered/uncovered ratio to 1 : 5 or even to 1 : 2 the etched surface quality is changed. We have found out that the layer is predominantly etched in the very vicinity of the mask edge up to a distance of 50 µm. Oxidizing agent concentration for given KOH concentration does not have influence on the quality of the etched surface.

From used masks Ti, Pt a Au the less suitable was the Ti mask 70 nm thick.

Very similar results were achieved for Pt and Au masks. Mask influence was detectable for distances of up to 40 - 50 µm. Etching was faster than for Ti mask. The whole sample surface was etched. Outside the area with mask influence the etching process was slow and even, only thin layers could be removed. In the mask vicinity the etching was pronounced.

5 ACKNOWLEDGEMENTS

This work was done in Center of Excellence CENAMOST (Slovak Research and Development Agency Contract No. VVCE-0049-07) with support of project APVV-0548-07, grant VEGA 01/0689/09 and 01/0220/09

6. REFERENCES