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Progress in Semiconductor Materials V—Novel Materials and Electronic and
Optoelectronic Applications

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Infrared Materials and Devices

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High aspect ratio etching of GaSb/AlGaAsSb for photonic crystals

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ABSTRACT

Photonic crystal structures defined by interferometric lithography were etched into GaSb and AlGaAsSb with 90% Al content using Inductively Coupled Plasma (ICP) Reactive Ion Etching (RIE) with BCl₃ and BCl₃/Ar gas mixture. Effects of DC bias, hole diameter, etch time and gas composition, on the etch rate of GaSb were investigated. Hardened photoresist (PR) was used as an etch mask for the experiments.

INTRODUCTION

Emitters in the 1.7-2.4 μm (mid-IR) spectral range are useful for a broad range of applications such as tunable diode laser absorption spectroscopy, free space optical communications and medical surgery. GaSb-based emitters are well suited for emission in the mid-IR range and we are interested in using photonic crystals (PC) in these materials. PCs are useful for enhancing light extraction from LEDs, PC defect lasers and PC distributed feedback lasers. As the periodicity of any given PC structure is directly proportional to the wavelength the PC is designed for, working in the mid-IR would also be beneficial to the study of photonic crystals themselves as the critical dimensions would be larger than at near IR or visible and thus the PCs will be easier to fabricate.

The structure of a typical GaSb based light emitter consists of high Al content (80-90%) AlGaAsSb upper and lower cladding layers with a core consisting of low Al (around 25%) AlGaAsSb spacer and high Ga content GaInAsSb quantum wells (QW). To have a uniform etch when the PC pattern is etched into or through the core, there should be no selectivity between the high and low Al content AlGaAsSb. It is assumed that the selectivity between GaSb and high AlGaAsSb forms the upper bound of the selectivity between the high and low Al content AlGaAsSb. The cladding layers of such a structure are usually 1-2 μm and the core between 0.4 and 1 μm . We wish to examine the feasibility of manufacturing a photonic crystal with an in-plane TE band gap in this structure. As a first step we wish to characterize the etch of photonic crystal features in these structures in order to determine what structures are possible to manufacture. There has been some previous work done on dry etching of these materials [1,2,3], but there is little on the etching of the features required for PCs [4]. PCs with different lattice constants and hole diameters are experimented with in order to give us flexibility in the future design of PCs. Sidewall profiles are paid close attention to as non-vertical sidewalls can increase out-of-plane losses and makes realistic simulations harder.

We begin this paper by examining the effect of changing DC bias on the GaSb etch rate, the PR sputter rate, and the etch profiles. Then the effects of hole diameter on the etch rate of GaSb, and the evolution of the average etch rate of GaSb and AlGaAsSb with etch time, are reported.

We end this paper by examining the effects of adding Ar to the gas mixture on etch rates and etch profiles. Preliminary tests with metal and SiN masks are also presented.

EXPERIMENTAL DETAILS

Interferometric lithography

Interferometric lithography (IFL) was used to create the photonic crystal patterns. The IFL setup consisted of a 355 nm frequency tripled Nd:YAG laser and a sample holder with an attached perpendicular mirror that can be rotated to face an expanded beam from the laser at different angles.

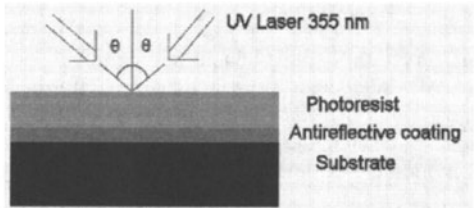


Figure 1. Principle of interferometric lithography.

Figure 1 shows the principle of the interferometric exposure. Two coherent beams with an angle of incidence of θ degrees interfere to create a sinusoidal intensity pattern in the PR. The antireflective coating (ARC) is present to suppress reflections from the substrate which could cause vertical standing waves in the PR. The period of the sinusoidal wave and thus the pitch of the grating is given by Eq. 1:

$$d = \frac{\lambda}{2 \sin(\theta)} \quad (1)$$

In order to create a 2D photonic crystal pattern in the PR, the sample is exposed once and then rotated and exposed again so that the second interference pattern is at an angle to the first one. This angle will then be the angle between the two lattice vectors. Negative PR is used to form circular holes in the PR by developing away the unexposed PR in the spots where both exposures had intensity minimums. Columns in the PR can be formed in the same spots by using a positive PR.

PR etch mask

GaSb and AlGaAsSb with a 90% Al content were patterned using the interferometric lithography described above and a negative tone PR (Futurex NR7-500P) with a thickness of 900 nm. After lithography the samples were UV-hardened using a MJB3 mask aligner and hardbaked. The ARC was then etched in an O₂ plasma using a Plasmalab μ P RIE. ICP etching with a PR mask, now approximately 700 nm thick, was carried out with a Plasma-Therm SLR Series ICP and GaSb and AlGaAsSb samples were placed side by side and etched simultaneously to obtain an accurate measure of the selectivity. SEM micrographs showing etch profiles on cleaved samples were taken with a JEOL 6400F SEM. Etch rates and etch depths were extracted from these micrographs. All etch rates were obtained by dividing the etch depth by the etch time,

unless otherwise indicated. Error bars used in this paper represent estimated errors in the extraction of data from SEM micrographs without any basis in statistics.

SiN etch mask and metal mask

For the SiN mask a SAMCO Model PD10 PECVD was used to deposit around 300 nm of SiN on a GaSb sample and IFL was carried out with a negative tone resist, followed by UV hardening, hardbake and ARC etch described in the previous section. After the O₂ etch of the ARC, the SiN was etched in SF₆ using RIE. The metal mask was formed using IFL on a GaSb sample with a positive tone photoresist (SPR 505a) followed by the deposition of 10 nm of titanium and 60 nm of nickel by e-beam evaporation. A metal pattern with holes was then formed using the lift-off technique. After which the ARC was either sputter-etched in the ICP at the initial stage of the GaSb etch recipe using BCl₃ or separately etched in O₂.

RESULTS

Selectivity between etch rate of GaSb and PR vs. DC bias

Initial experimentation showed that a good set of starting parameters for the ICP etch on GaSb was a pressure of 2 mTorr, a BCl₃ flow of 30 sccm, an ICP power of 300 W, a DC bias of 240 V, and no Ar flow. The resulting etch profile with these parameters can be seen in Figure 2(a). The sidewalls of the etch profile is seen to have an angle around 80°.

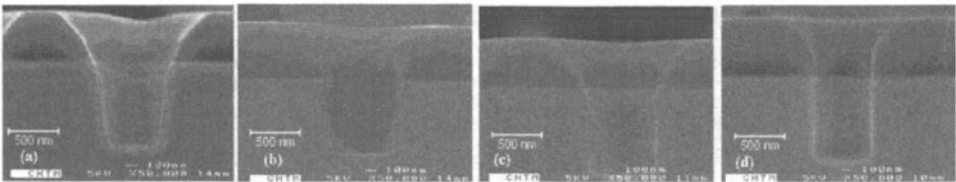


Figure 2. Etch profiles in GaSb for different DC biases using a PR mask: (a) 240 V, (b) 180 V, (c) 140 V, and (d) 100 V.

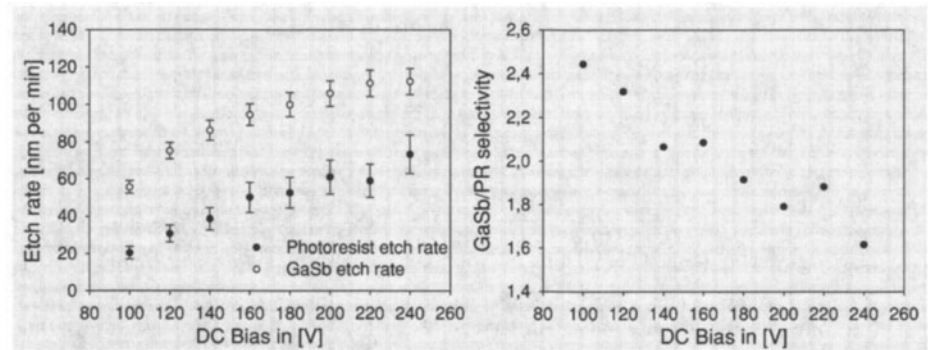


Figure 3. Etch rate of GaSb and PR and GaSb/PR selectivity vs. DC bias. Parameters: 300 W ICP power, 2 mTorr pressure, 30 sccm BCl₃. Etch depths vary from 660 nm to 840 nm.

The selectivity between the GaSb and PR was, however, found to be poor with these parameters. In order to obtain better GaSb/PR selectivity the DC bias was lowered to reduce PR sputtering. The effect of the DC bias on the etch rate of GaSb and PR is shown in Figure 2 and Figure 3.

As can be seen from Figures 2 and 3, lowering the DC bias to 100 V increases the GaSb/PR selectivity to 2.5 while at the same time straightening the sidewalls of the etch profile. We believe this is due to lessened erosion of the mask while at the same time maintaining enough sputtering to ensure an anisotropic etch. Lowering the DC bias below 100V leads to undercut of the etch mask. Having found the best DC bias for the baseline parameters, the etch time was increased to attain deeper etches. A 30 min etch resulted in a etch depth of 1.7 μm for GaSb and 1.56 μm for AlGaAsSb with a hole diameter of 680 nm, corresponding to an aspect ratio of 2.5 for GaSb and 2.3 for AlGaAsSb. Etch rates were 57 nm/min and 52 nm/min for GaSb and AlGaAsSb, respectively, yielding a selectivity of 1.1. Looking at Figure 4 we believe this is close to the maximum attainable etch depth with these parameters and this particular PR mask, as the PR mask is starting to erode laterally.

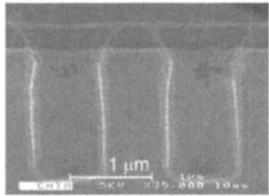


Figure 4. Etch profile at 300 W ICP power, 2 mTorr pressure, 30 sccm BCl_3 flow, 100 V DC bias, and 30 min etch time.

GaSb/AlGaAsSb etch rate dependence on hole diameter and etch time

It was noticed during this experiment that both hole size and etch depth had an effect on the etch rate and experiments were undertaken to examine these effects. The results are presented in figure 5.

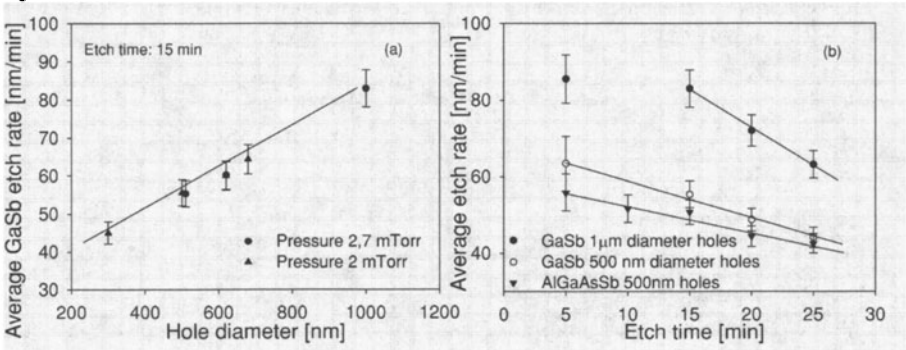


Figure 5. Average etch rate vs. hole diameter and etch time, 300 W ICP power, 2.7 mTorr pressure unless otherwise indicated, 30 sccm BCl_3 flow, and 100 V DC bias. Straight lines are guides for the eye.

Looking at Figure 5(a) the average etch rate for the first 15 minutes seems to have a near linear dependence on the hole diameter. This indicates that the aspect ratio attainable is not heavily dependent on the feature size, it also seems to indicate that the etch is either limited by supply of reactants or removal of etch products. As seen in figure 5(b), the average etch rate decreases as the hole gets deeper. Assuming a constant PR sputter rate, this means a decreasing selectivity between GaSb and PR thus making a thicker mask a requirement for aspect ratios beyond 2.5.

GaSb/AlGaAsSb etch rate dependence on gas composition

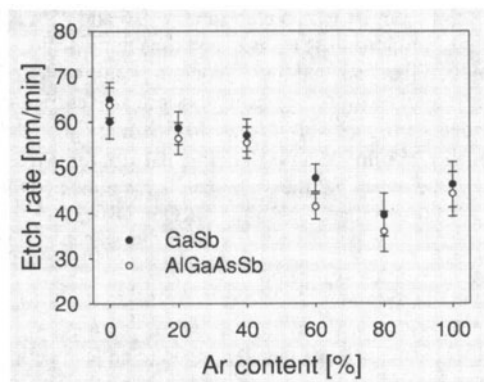


Figure 6. Average etch rate vs. Ar content for a 15 min etch. Process parameters: 300 W ICP power, 2 mTorr, 30 sccm total gas flow, and 100 V DC bias.

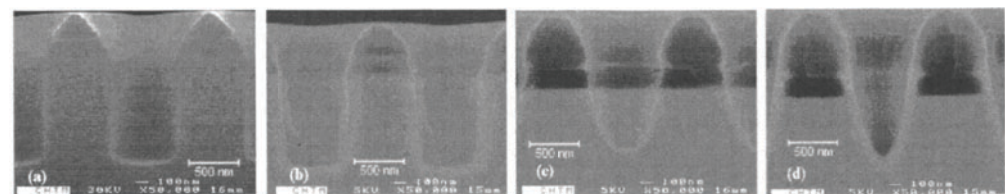


Figure 7. Etch profiles for GaSb at different Ar content, (a) 0%, (b) 40 %, (c), 80%, and (d) 100 %. Etch parameters same as Figure 6.

From Figure 6 it seems that the etch rate for both GaSb and AlGaAsSb falls with increasing argon content. More interesting, however, is the fact that the selectivity between GaSb and AlGaAsSb does not change appreciably. According to [1], for more freestanding structures GaSb should etch faster than AlGaAsSb in plasmas with high argon content. Figure 7 shows the the etch profile changes with higher Ar content, from slight undercut to V shaped. This seems to indicate that chemical etching is responsible for the lateral undercut of the mask. In addition, the amount of PR left on the samples seems to increase, while the sidewall passivation and possibly redeposited reaction products become more visible, as the argon content increases. This seems to indicate that Ar sputters both the PR and the passivation layer more slowly. We believe that the V shape of the etch profile indicates that the etch rate at these conditions is limited by the sputter rate of the passivation layer. If we assume that the bombarding ions have a distribution around

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normal incidence and the passivation needs to be removed at a certain rate in order for the underlying material to be etched, then the etch profile can be explained by the fact that as the hole gets deeper the edges of the hole are hit by a decreasing flux of ions. If the etch rate is limited by the sputtering of the passivation, then this would explain the lack of selectivity between GaSb and AlGaAsSb.

Results with alternative etch masks

Preliminary experiments with a SiN mask were carried out at 300 W ICP power, 2 mTorr pressure, 100 V DC bias, 30 sccm BCl₃, and etch times of 15 and 25 minutes. The etch depth attainable was found to be similar to the PR mask and thus not worth the complication of another etching step. In addition the SiN mask had more problems with mask undercut, although it seemed more resistant to lateral erosion. Experiments were also carried out with a titanium/nickel mask under the same conditions as the SiN mask with the exception of a DC bias of 140 V and an etch time of 15 min. At an etch depth of 1 μm for a 1.15 μm diameter hole there was no visible undercut of the mask, but the metal layer was gone and the ARC was the remaining mask. It was also found that etching the ARC in O₂ was a better solution than sputter etching in the ICP.

CONCLUSIONS

GaSb etch rate vs. DC bias was investigated and led to an etch depth of 1.7 μm for GaSb and 1.56 μm for AlGaAsSb with aspect ratios of 2.5 and 2.3, respectively. Selectivity between GaSb and AlGaAsSb was close to 1.1, and selectivity between GaSb and PR 2.9. Average etch rate for both GaSb and AlGaAsSb decreases as hole diameter decreases and also as etch depth increases. Adding Argon to the BCl₃ decreases the etch rate slightly, but greatly decreases lateral etching. Selectivity between GaSb and AlGaAsSb remained close to 1.1 for all plasma conditions explored.

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3.3 μm high brightness LEDs

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ABSTRACT

Deep mesa etching and surface roughening have been implemented to InAs flip-chip LEDs emitting at 3.3 μm (300 K). Near field and power measurements confirmed the output power enhancement of about 2 and brightness increase with an equivalent to a black body temperature of about 1250 K.

INTRODUCTION

Recent years have seen extensive research on the mid-IR (2-5 μm) diodes and one can find publications on resonant cavity [1] and optically pumped [2] as well as on conventional (with point top contact and flat surface) LEDs that have already broken the 1 mW output power barrier that is necessary for most practical applications. Several applications, e.g. spectroscopic measurement with grating, call for sources having high radiance (mW/cm^2) or apparent temperature values. The episcide-down bonded InAs LEDs with broad mirror anode and flat out-coupling surface emitting at 3.3 μm at room temperature have already shown the ability to simulate the black body heated up to 593 K (positive contrast $\Delta T_a = 300$ K in the 3-5 μm range) [3]. Electrically pumped GaSb/InAs multilayer devices grown onto GaSb also reached the $\Delta T_a = 300$ K value at 50 mA pump current [4]. Both above examples considered structures with relatively high losses due to total internal reflection at the air/semiconductor interface and it is thus clear that there is still room for device performance improvements.

Deep mesa LED construction narrows the internal radiation diagram due to the reflections from the inclined mesa sidewalls and thus contribute to the out-coupling enhancement. The effect of the above geometrical factor is well known for the InSb negative luminescent devices emitting at 6 μm [5] and efficient NIR and visible LEDs [6], however, to the best of our knowledge there have been no attempts so far to investigate the impact of the mesa dimensions on output power of the mid-IR LEDs with wavelengths in the 3- 5 μm range.

Microtexturing the surface offers enhanced out-coupling efficiency due to suppression of the total internal reflection [2, 6], however, this technology has been not implemented on electrically pumped mid-IR LEDs with wavelengths in the 3 - 6 μm range.

We report on apparent temperature and output power measurements in InAs LEDs with deep mesa and textured out-coupling surface operating at room temperature and grown onto transparent heavily doped n^+ -InAs substrates.

EXPERIMENTAL DETAILS

Heterostructures were grown onto heavily doped n^+ -InAs (Sn) (111) ($n^+ > 10^{18} \text{ cm}^{-3}$) substrates transparent to radiation at 3.3 μm due to Moss-Burstein effect and consisted of 2-7 μm thick n-InAs active layers and p-InAsSbP claddings or contact layers. Fig. 1 presents typical composition distribution together with the 77 K band gap scheme; the zero point ($L=0$) corresponds to the n^+ -InAs/n-InAs interface while $L=6 \mu\text{m}$ indicates the p-InAsSbP/air

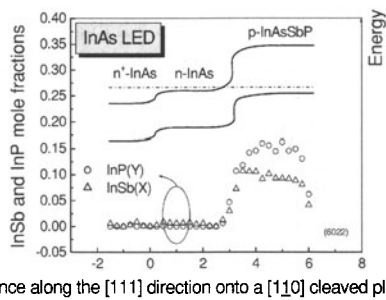


Fig. 1 Distribution of Sb and P atoms in InAs LED structure (composition profile) together with band diagram.

interface. $\text{InAs}_{1-x-y}\text{Sb}_x\text{P}_y$ cladding was reasonably lattice matched with InAs substrate ($y \sim 2.2 \times$) and had an energy gap as big as 460 meV (77 K). Due to the step at the $n^+ \text{-InAs}/n\text{-InAs}$ interface we could expect hole confinement in $n\text{-InAs}$ that is beneficial for the LED operation.

Heterostructure wafers were treated by a multistage wet photolithography process that includes two-stage etching of the mesa and that enables us to prepare flip-chip devices with $\sim 240 \mu\text{m}$ wide mesa as shown schematically in Fig. 2. A U-like cathode as well as a circular anode ($D_a = 210 \mu\text{m}$) were formed by thermal evaporation of gold films followed by an electrochemical deposition of $\sim 2 \mu\text{m}$ thick gold layer.

For the sake of a reference some chips lacked one of the etching processes and thus were considered as “shallow” mesa LEDs. The wafers were lapped down to $\sim 100 \mu\text{m}$ by chemical etching before chip separation.

Fig. 2 (on the right) demonstrates $0.9 \times 1 \text{ mm}^2$ chip contact surface with $H_m \sim 40 \mu\text{m}$ deep mesa with $h_m = 10 \mu\text{m}$ deep cathode contact areas called later as “deep mesa devices”. The chips were further soldered onto silicon submounts with Pb-Sn bonding areas and mounted onto massive copper heatsink.

The devices were biased with pulse currents of $\tau = 5 - 10 \mu\text{s}$ duration with a repetition rate of $f = 2 \text{ kHz}$. A phase sensitive technique together with 77 K cooled CdHgTe or InSb photodiodes were used in electroluminescence (EL) measurements. Common features of the fabricated LEDs were superluminescence and blue shift of the emission spectrum at 77 K due to the dynamic Moss-Burstein effect and superiority of the negative luminescence power conversion efficiency



Fig. 2 Schematic of the InAs deep mesa “flip-chip” LED grown onto $n^+ \text{-InAs}$ and mounted onto Si header (cross section in the N-M direction) (at the left) and photo of the unmounted chip with contacts (at the right). D_m - is the mesa diameter, h_m - is the distance from the $p\text{-InAsSbP}$ surface to the deepest cathode areas, (“depth of the cathode”), H_m - is the total depth/height of the internal reflector, t - is the total $p\text{-InAsSbP}/n\text{-InAs}/n^+ \text{-InAs}$ structure thickness, A- is the anode, C- is the cathode.