# 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 BCl<sub>3</sub> and BCl<sub>3</sub>/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  $\mu$ m (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. 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  $\mu$ m and the core between 0.4 and 1  $\mu$ m. We wish to examine the feasibility of manufacturing a photonic crystal with an inplane TE band gap in this structure. As a first step we whish 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.

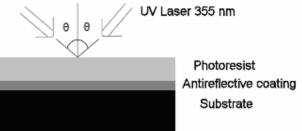


Figure 1. Principle of interferometric lithography.

Figure 1 shows the principle of the interferometric exposure. Two coherent beams with an angle of incidence of  $\theta$  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:

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

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 O<sub>2</sub> plasma using a Plasmalab  $\mu$ 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.

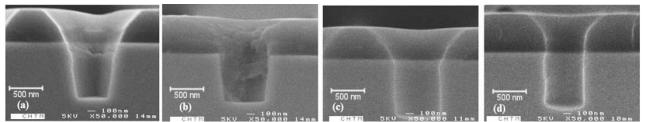
## SiN etch mask and metal mask

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  $O_2$  etch of the ARC, the SiN was etched in SF<sub>6</sub> 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 BCl<sub>3</sub> or separately etched in  $O_2$ .

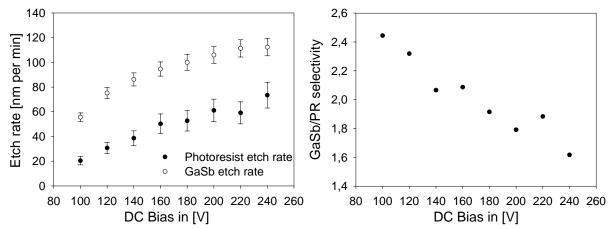
# RESULTS

## 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 BCl<sub>3</sub> 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  $80^{\circ}$ .



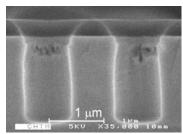
**Figure 2.** Etch profiles in GaSb for different DC biases using a PR mask: (a) 240 V, (b) 180 V, (c) 140 V, and (d) 100 V.



**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 BCl<sub>3</sub>. 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.

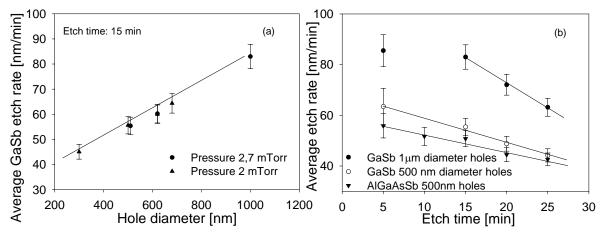
As can be seen from Figures 2 and 3, lowering the DC bias to 100 V increases the GaSb/PR selectivity to 2.5 while at the same time straightening the sidewalls of the etch profile. We believe this is due to lessened erosion of the mask while at the same time maintaining enough sputtering to ensure an 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  $\mu$ m for GaSb and 1.56  $\mu$ m 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<sub>3</sub> 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<sub>3</sub> 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

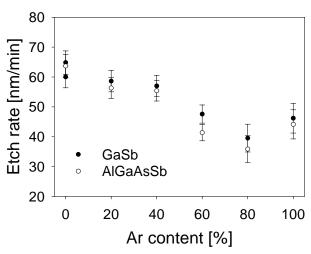


Figure 6. Average etch rate vs. Ar content for a 15 min etch. Process parameters: 300 W ICP power, 2 mTorr, 30 sccm total gas flow, and 100 V DC bias.

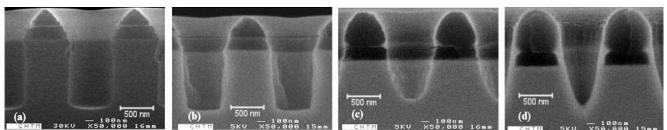


Figure 7. Etch profiles for GaSb at different Ar content, (a) 0%, (b) 40 %, (c), 80%, and (d) 100 %. Etch parameters same as Figure 6.

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

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 BCl<sub>3</sub>, 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  $\mu$ m for a 1.15  $\mu$ m 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 O<sub>2</sub> 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  $\mu$ m for GaSb and 1.56  $\mu$ m 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 BCl<sub>3</sub> 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.

### ACKNOWLEDGMENTS

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