Cl₂-Etching and MBE-Regrowth for GaAs/AlOₓ Photonic Crystals

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ABSTRACT

We propose a new method for fabricating high index contrast gratings made of gallium arsenide (GaAs) and aluminum oxide (AlOₓ) buried in GaAs based waveguide structures. A central part of this method is a UHV-compatible thermal chlorine etch and MBE-regrowth on etched GaAs and AlₓGa₁₋ₓAs. We describe results on surface roughness evolution during the etch obtained by in-situ elastic light scattering. These measurements allow us to determine the process parameters that lead to a smooth etched surface on which we demonstrate successful regrowth.

INTRODUCTION

Thin optical gratings buried in semiconductor waveguides form the basis of distributed feedback (DFB) lasers, one of the most important components in optical communication systems. These gratings are typically comprised of two different semiconductor alloys with a difference in refractive indices of approximately 0.2. This small index contrast leads to a relatively small coupling constant for the modes of the waveguide, resulting in a high sensitivity of the laser performance to optical feedback, temperature fluctuations, and the random phase of the grating at the cleaved edge of the structure (facet phase). These properties can be improved by increasing the mode coupling constant by using high index contrast gratings comprised of GaAs (n=3.4) and aluminum oxide AlOₓ (n=1.6). A logical extension of this idea are two-dimensional buried gratings that can be fabricated in the same way. They could be used in novel optoelectronic devices such as saddle point lasers [1] or in integrated vertical output couplers for surface-emitting lasers [2]. Here, the high index contrast is beneficial because it reduces the size of the grating needed for efficient coupling into the radiation mode. We note that recent progress in crystal growth technology has made the fabrication of long wavelength lasers on GaAs-substrates possible, either through the use of InAs quantum dots [3] or the nitrogen containing alloy InGaNAs [4] as the active layer. Our choice of materials for the grating layer is compatible with these technologies.

The fabrication of the gratings will proceed in several steps. First a structure is grown by molecular beam epitaxy (MBE) that contains a 30-50 nm thick layer of Al₀.₉₈Ga₀.₀₂As buried under a GaAs cap layer. This cap layer is then patterned with a one- or two-dimensional grating by standard lithographic and etching techniques without etching through the GaAs cap and exposing the Al₀.₉₈Ga₀.₀₂As-layer to air. This is important because, upon exposure to atmosphere, the aluminum forms a stable oxide on the surface that cannot be removed and makes successive regrowth impossible. The sample is next moved into an ultra-high vacuum (UHV) etch chamber that is attached to the MBE-system. Here, a final etch step is carried out by heating the sample to temperatures between 200°C and 500°C in a low pressure (10⁻⁶-10⁻⁴ Torr) chlorine atmosphere. The fact that the etch rates for GaAs and Al₀.₉₈Ga₀.₀₂As are comparable ensures that the pattern propagates into the buried layer with minimal distortion. After completion of the etch, the sample...
can be transferred in UHV into the growth chamber of the MBE where regrowth of GaAs can proceed. The sample now contains a buried grating made of GaAs and Al_{0.98}Ga_{0.02}As. To produce the high index contrast grating, we make use of the well-known oxidation properties of AlGaAs with a high aluminum content. Trenches are etched \textit{ex-situ} into the sample that expose the grating layer. The Al_{0.98}Ga_{0.02}As can then be oxidized by exposure to hot water vapor at 400°C forming a grating of AlO_x in a GaAs-matrix. Note that for the case of a two-dimensional grating, for oxidation to proceed from the etched trenches through the entire structure, the Al_{0.98}Ga_{0.02}As must be continuous, i.e. the resulting grating will consist of GaAs pillars surrounded by AlO_x. In this study, we concentrate on properties of the \textit{in-situ} Cl_2-etch and subsequent regrowth, which are an integral part of the proposed fabrication process, namely the creation of smooth surfaces.

**EXPERIMENTAL DETAILS**

The especially designed UHV-chamber in which the Cl_2-etch takes place is pumped by a corrosion resistant turbomolecular pump and an ion pump which is turned off during the etch. The base pressure is approximately 5 x 10^{-9} Torr. It contains an all refractory metal sample heater with a pyrolytic boron nitride heating element. Temperatures are measured with a thermocouple inside the heater that was initially calibrated by a diffuse reflectance spectroscopic measurement of the actual sample temperature [5] with an accuracy of approximately 10°C. The Cl_2-gas is fed into the chamber through a leak valve and the pressure is monitored with a cold cathode gauge which was found to be much more resistant to Cl_2-exposure than a hot filament ion gauge. The etch chamber is attached to a V80H MBE-system through a gate valve. Four viewports on the chamber having line of sight to the sample surface allow optical measurements to be carried out \textit{in-situ} during the etch.

To monitor surface roughness as it evolves during the etch, we use elastic light scattering (ELS) which is an extremely sensitive yet easily implemented technique. In our setup, the beam of a HeNe laser (\lambda=632 nm) is incident on the sample at an angle of 65° to the surface normal and the diffusely scattered light from the sample surface is detected by a photomultiplier tube at an angle of 45°. In the limit in which the roughness is much smaller than the wavelength of the light, the scattered intensity is proportional to the power spectral density of the surface [6] at a length scale of approximately 420 nm.

In order to avoid artifacts in these measurements that could result from the native oxide layer on the surface, the samples were prepared in the same way as samples for MBE growth. The epi-ready wafers are exposed to light from a UV lamp for ten minutes, which results in growth of an oxide layer on the surface. They are then moved into the growth chamber of the MBE system where they are heated to about 600°C under an arsenic overpressure to desorb the oxide. We note that while this treatment removes the oxide efficiently, it does not clean the surface of SiO_2 contamination. It also results in a surface with a characteristic morphology and this initial roughness can be seen in the light scattering signal.

For regrowth experiments, the samples were moved from the etch chamber into the growth chamber without breaking the vacuum. After completion of the etch, we waited approximately 15 minutes before transferring the samples to allow the Cl_2-gas to be pumped out of the system. The samples were then brought to their growth temperature of 600°C under an arsenic overpressure. Regrowth was started by opening the shutter of the gallium cell without any further
sample preparations. During growth, the ELS signal from a UV mercury lamp was recorded. This setup has been described in detail elsewhere [6].

RESULTS AND DISCUSSION

To map out the conditions that allow etching without surface roughening, a series of etches was performed on plane GaAs substrates at different Cl₂ pressures. During these etches, the ELS signal was recorded while the temperature was slowly increased. In figure 1 a), we show the results for a pressure of 5 x 10⁻⁶ Torr. The etch is started at a sample temperature of 200°C and then the temperature is ramped up to 500°C over a time period of 40 minutes. The surface has some initial roughness due to the sample preparation as described above. This roughness decreases during the early phase of the etch. This can be more clearly seen in figure 1 b) where we show the initial decrease (during a different etch) on an expanded time scale. We believe that this smoothing of the surface is due to the fact that the etch propagates along the local surface normal. In this case pointy structures on the surface are effectively removed by a sideways propagating etch, leading to a decreasing surface roughness. For a more quantitative explanation, continuum surface evolution equations analogous to the ones that describe MBE growth could be used [7].

As the substrate temperature is slowly ramped up a smooth surface is maintained until the temperature reaches approximately 420°C. At this point, there is a very sharp onset of surface roughening that leads to a sudden increase in the intensity of the light scattered from the surface. This roughness can easily be seen by looking at the sample with a scanning electron microscope after the etch. X-ray photoelectron spectroscopy measurements taken on a rough sample after the etch showed that the surface is depleted of arsenic. The formation of a surface layer that is depleted of one of the constituents of the GaAs crystal has been described for this etch in the literature [8]. The reason for its formation is that the rate constants for the two surface reactions

![Figure 1: a) ELS signal during etch at a Cl₂-pressure of 5 x 10⁻⁶ Torr as a function of time while the substrate temperature is slowly increased. b) Enlarged view of the initial surface smoothing during an etch at 1 x 10⁻⁴ Torr.](image-url)
(2 \text{Ga} + 3 \text{Cl}_2 \rightarrow 2 \text{GaCl}_3 \text{ and } 2 \text{As} + 3 \text{Cl}_2 \rightarrow 2 \text{AsCl}_3) \text{ are different, yet for etching to occur, the number of molecules of the two etch products GaCl}_3 \text{ and AsCl}_3 \text{ that are removed from the surface has to be equal. This imbalance results in a non-stoichiometric surface layer, which has previously been assumed to be of a constant thickness. From our data, we conclude that its thickness is a function of the substrate temperature and the pressure, since the temperature at which the onset of roughening occurs is dependent on the Cl}_2-pressure. This is shown in figure 2, which depicts a phase diagram for rough and smooth etching. Generally, we find that at a lower pressure a smooth etch can be maintained up to a higher temperature. The rate of the increase of roughness during the etch in the rough regime is found to be limited by the supply of reactants, consistent with a linear dependence of the etch rate on pressure that has been reported in the literature [9].

The next important step of the described method for fabricating high index contrast gratings is regrowth on the \textit{in-situ} etched surfaces. Following the procedure described above, we were able to obtain epitaxial growth of GaAs on both etched planar surfaces of GaAs and Al\textsubscript{0.65}Ga\textsubscript{0.35}As. In figure 3, we compare the ELS signals during regrowth on GaAs and on Al\textsubscript{0.65}Ga\textsubscript{0.35}As. The ELS signal for regrowth on GaAs did not show any significant deviations from standard growth on epi-ready substrates. The small increase in the signal shortly after the start of the growth is believed to be caused by surface contaminants which are rapidly overgrown. Atomic force microscopy images after growth confirmed that the regrown film had a rms-roughness (0.5 nm for a 2 \mu m x 2 \mu m image) and surface morphology comparable to a typical MBE-grown GaAs buffer layer. During regrowth on Al\textsubscript{0.65}Ga\textsubscript{0.35}As, we observe an initial increase in the ELS signal by a factor of 3, indicating increased surface roughness, which then disappears after growth of approximately 100 nm of GaAs. We believe that this behavior is due to partial oxidation of the aluminum that occurred between the etch and the regrowth and could

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\includegraphics[width=\textwidth]{figure2.png}
\caption{Phase diagram for smooth and rough etching obtained by ELS data.}
\end{figure}
be avoided by improving the base pressure in the system. Further studies concerning the interface quality are in progress.

CONCLUSIONS

In conclusion, we have determined a temperature and pressure regime for smooth thermal chlorine etching of GaAs and AlGaAs by the use of in-situ light scattering measurements. Successful regrowth of GaAs was demonstrated on both etched GaAs and Al$_{0.65}$Ga$_{0.35}$As. We have proposed that these techniques can be used to fabricate buried high index contrast gratings, which could be the basis for improved DFB-lasers and novel optoelectronic devices.

ACKNOWLEDGEMENTS

We thank NSERC, Rogers Canadian Cable Labs, NRC, DAAD (J. S.), and the BC Science Council (M. A.) for research support.

REFERENCES