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Single-Step RIE Fabrication Process of Low Loss InP Waveguide Using CH₄/H₂ Chemistry

M. Lysevych,^z H. H. Tan, F. Karouta, and C. Jagadish*

Department of Electronic Materials Engineering, Research School of Physics and Engineering,
The Australian National University, Canberra, ACT 0200, Australia

Single-step CH₄/H₂-based reactive ion etching (RIE) process, without alternating O₂ plasma treatment, has been developed with virtually no polymer buildup on the etched surface. InP ridge waveguides up to 1.9 μm high were etched without any mid- or post-etch O₂ plasma treatment. Smooth and polymer-free sidewalls led to a slight undercut resulting in sidewall slope of ~85°. Very low waveguide propagation losses (0.28 dB/cm) of “shallow” etched waveguides were obtained. The etched surface has a root-mean-square roughness of 0.66 nm.

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To fully utilize the advantages of optical communication and signal processing systems, photonic integrated circuits with multiple functionalities need to be developed. In order to achieve full integration of different photonic devices, such as lasers, waveguides, modulators, and detectors (especially devices based on photonic crystals), a range of different processing technologies is required, including etching. Dry etching is an indispensable tool in photonics due to depth control uniformity, selectivity and anisotropy especially in photonic crystal fabrication. Reactive ion etching (RIE) is the most commonly used dry etching technique because of its lower cost, simple, and robust design.

Methane–hydrogen (CH₄/H₂) chemistry used for etching InP-based structures results in good post-etch surface quality.^{1–6} This process is advantageous over the Cl₂-based chemistry etching due to its nontoxic, noncorrosive properties and the ability to carry out the etching at room temperature. However excessive polymer formation^{1,7} is the main drawback which could negate all these advantages. Generally polymer buildup is removed by O₂ plasma treatment.^{3,8–11} In that case a typical etching process consists of alternating the etching (CH₄/H₂) with short cleaning (O₂) steps. This significantly increases the process time and prohibits the use of resists and other organic materials as the etching mask. In this work we demonstrate a process using CH₄/H₂ chemistry that is capable of suppressing polymer formation. A 1.9 μm deep etch is demonstrated in a waveguide structure.

Experimental

To study the etching parameters, S-doped InP (100) wafers ($n = 2 \times 10^{18} \text{ cm}^{-3}$) were used. For waveguide loss measurements, metallorganic chemical vapor deposition (MOCVD)-grown sample consisting of 0.3 μm of InP buffer layer, followed by 1 μm thick InGaAsP ($\lambda = 1.1 \text{ μm}$) waveguiding layer, and capped with a 2 μm InP layer was used. The sample was grown on S-doped InP (100) substrate ($n = 2 \times 10^{18} \text{ cm}^{-3}$). All layers were undoped.

Etching was carried out in an Oxford Instruments Plasmalab 80+ RIE system with a 13.56 MHz RF source, using a carbon plate as the sample holder. Prior to etching the sample, the chamber was cleaned with O₂ plasma. The samples were initially cleaned with O₂ plasma before being deposited with 375 nm of SiN_x by plasma-enhanced chemical vapor deposition (PECVD) at 300°C. The SiN_x layer was used as the hard mask during the etching process. Standard photolithography was then carried out, followed by RIE etching, to transfer the pattern of 3 μm wide stripes with a pitch of 300 μm onto the SiN_x layer using CHF₃/O₂ chemistry. Later, the photoresist was removed in acetone.

Prior to etching the samples, they were treated with O₂ plasma again to remove any remaining photoresist residue which could lead to micromasking. This step is very important for smoothness of the etched surface.⁷ Samples were exposed to the plasma discharge of CH₄ (20 sccm) and H₂ (70 sccm) at an RF power of 250 W and pressure of 25 mTorr. The self-generated dc-bias was about 200 V.

The etched samples were evaluated using a high resolution scanning electron microscope (SEM) and surface roughness was measured with an optical profilometer. Fabry–Perot method¹² was employed to determine the waveguide losses.

Results and Discussion

Parameters that have significant influence on CH₄/H₂ RIE etching of InP are total flow rate of the gases, their ratio, process pressure, and RF power.

During etching the methane radicals (methyl) may contribute to two processes, etching of InP and polymer formation.^{1,7,13} Methane radicals react with In on the sample surface to form highly volatile metallorganic compounds. At the same time hydrogen removes phosphorus from the surface of the sample by forming phosphine. However, if phosphorus removal is not fast enough, the surface of the sample can become indium depleted. In the absence of indium (on the sample surface depleted of In and the mask surface), methane radicals react with each other to form polymer chains.¹³ By reducing methane concentration, the etch process can be made diffusion-limited so that there would be no indium depletion on the surface and, hence, polymer formation could be suppressed on the etched surface. However, reducing methane concentration can significantly reduce the etch rate. Thus to achieve diffusion-limited etching regime without significantly reducing the etch rate, methane concentration has to be controlled carefully. There are reports in the literature^{3,5,11,14} that the CH₄/H₂ ratio should be within 1/2 and 1/4. High methane concentration leads to extensive polymerization and spontaneous micromasking,^{1,13,14} while low methane concentration leads to reduced etch rate.^{13,14}

As a starting point the standard Oxford Instrument's recipe was employed (CH₄: 20 sccm; H₂: 70 sccm, 35 mTorr, 150 W). Several papers^{6–9,15} have reported a complete or a significant reduction of polymer formation with the addition of Ar during etching. Therefore, a set of four samples was etched as summarized in Table I. Initially all the samples were supposed to be etched at the same pressure of 35 mTorr. However, the small turbo pump of the system could not sustain such pressure for recipes with Ar due to increased total gas flow. Hence, the process pressure had to be increased for the recipes with Ar.

As seen from the SEM images in Fig. 1a–1d, the introduction of Ar does not reduce the amount of polymer deposited on the sample. In fact the recipe without Ar (Fig. 1a) appears to result in the least amount of polymer formation, as well as the highest etch rate. The

* Electrochemical Society Fellow.

^z E-mail: lmb109@physics.anu.edu.au

Table I. Process conditions used to determine the influence of the Ar flow on the etching and polymerization processes.

CH ₄ /H ₂ /Ar (sccm)	Pressure (mTorr)	RF power (W)	Time (min)	Figure 1
20/70/0	35	150	30	a
20/70/10	55	150	30	b
20/70/50	55	150	30	c
20/70/90	75	150	30	d

sample etched with CH₄/H₂/Ar (20/70/90) had a significant undercut and noticeable sidewall roughness. Hence, the idea of Ar addition was disregarded in further experiments.

Another set of samples was etched to determine the influence of RF power on etching and polymerization. As expected, the etch rate increased with the increase of RF power but no correlation between RF power and polymerization, sidewall slope or surface roughness

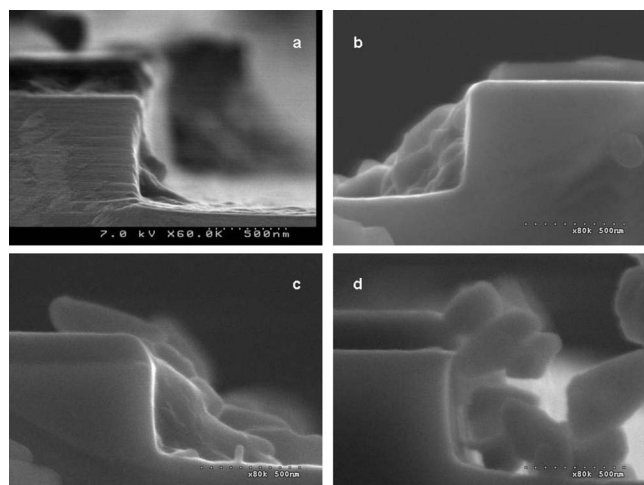


Figure 1. Cross-sectional SEM images of the samples etched with (a) CH₄/H₂/Ar (20/70/0) at 35 mTorr; (b) CH₄/H₂/Ar (20/70/10) at 55 mTorr; (c) CH₄/H₂/Ar (20/70/50) at 55 mTorr; and (d) CH₄/H₂/Ar (20/70/90) at 75 mTorr.

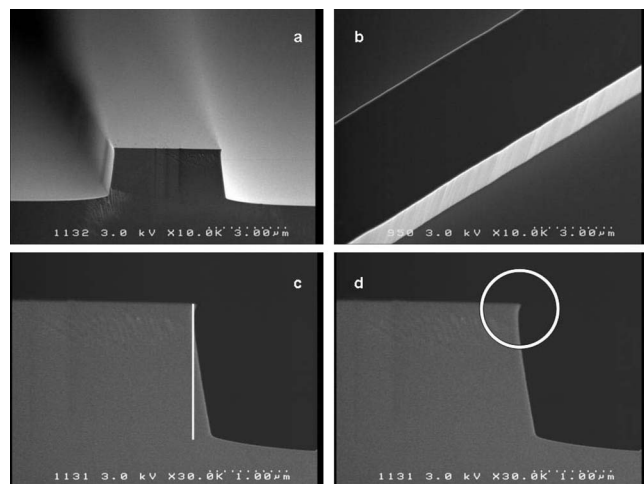


Figure 2. Cross-sectional views (a,c,d) and sidewall view (b) of the waveguide ridge, after the mask has been removed by 5% HF solution.

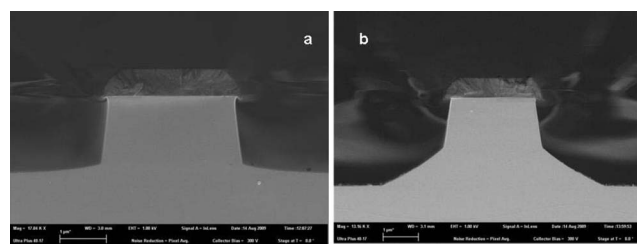


Figure 3. Cross-sectional SEM images of the sample prior to wet etching (a) and after a short wet etching in HCl:H₃PO₄ (1:3) solution (b).

was observed (not shown). Because 300 W was the maximum output power of the RF generator, the value of 250 W was selected for experiments in the next section.

A survey of the literature revealed a very big spread (7.5–90 mTorr) in the recommended process pressure.^{1,5,7,8,11,13,16} Unfortunately, most of the studies were done with the focus on increasing the etch rate and not polymerization reduction. Process pressure strongly affects polymer formation rate, because it is related to the residence time of methane in the process chamber. Therefore, lowering the process pressure reduces the time methane stays in the chamber (without changing the ratio of the gases), and as a result decreases the possibility of polymerization. Hence, we reduced the process pressure to the lowest possible value for this gas flow (CH₄: 20 sccm, H₂: 70 sccm) that can be sustained by the turbo pump in the system (25 mTorr). This resulted in a remarkable reduction in polymerization. In fact no polymer was observed anywhere on the sample apart from the top of the mask which can be easily removed by removing the SiN_x mask in 5% HF solution.

Figure 2a shows the cross-sectional view of the etched ridge with the mask removed. The ridge height is about 1.9 μm. The ridge sidewalls and the etched surface are flat and polymer-free, as seen in Fig. 2b. The sidewalls are almost vertical (~85° slope; Fig. 2c), with a small undercut directly under the mask (Fig. 2d). These are due to polymer buildup on the mask. Polymer buildup on the mask cannot be completely eliminated because on the surface of the mask, methane radicals have no indium to react with and can only polymerize. However, by adding a small amount of O₂ (less than 1%) to the etching gas mixture, polymer buildup can be suppressed resulting in perfectly vertical ridge sidewalls and no undercut, as reported in Ref. 11.

The use of low pressure in this recipe eliminates the need of mid- or post-etch O₂ plasma treatment. Another added advantage is that because the polymerization occurs on the mask, it protects the mask

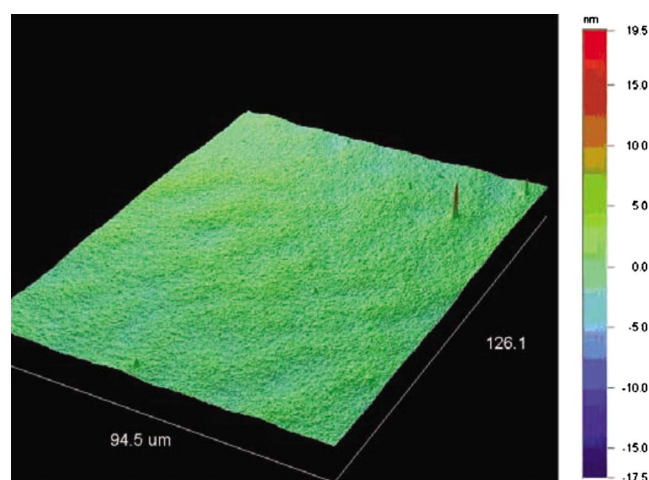


Figure 4. (Color online) 3D topography of the etched surface.

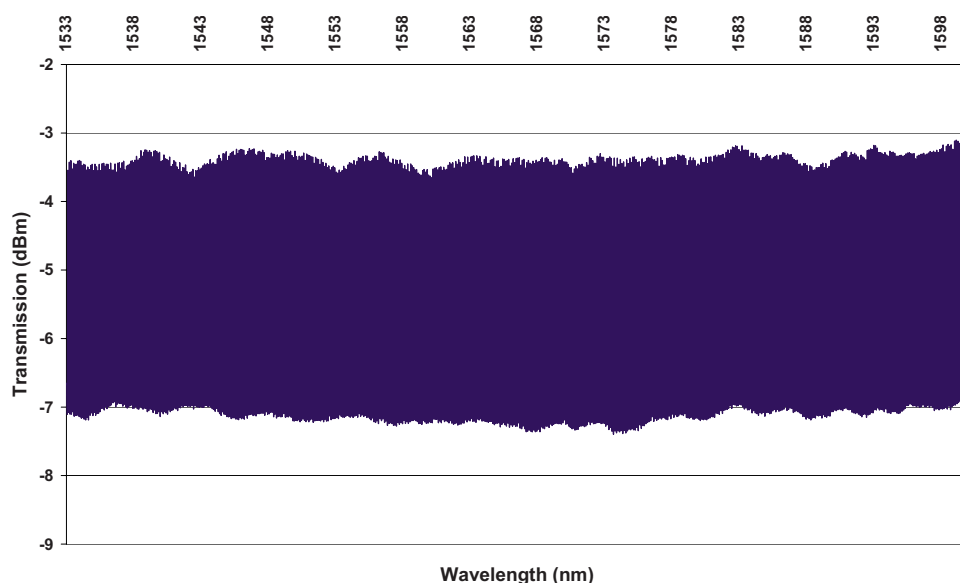


Figure 5. (Color online) Fabry-Perot fringes of a 3 mm long, 3 μm wide waveguide.

from erosion. As a result, a photoresist mask can be employed instead of a hard mask such as SiN_x , which would significantly reduce the number of processing steps.

To confirm the absence of polymer on the surface or the sidewalls, the sample was wet etched in $\text{HCl}:\text{H}_3\text{PO}_4$ (1:3) solution (standard solution for wet etching of InP). The cross-sectional SEM view of the sample pre- and post-wet etching processes are shown in Fig. 3. Comparing the two images, the sample on the right has been etched in both vertical and horizontal directions conclusively proving the complete absence of a protective polymer after the RIE step.

To evaluate the quality of the etching recipe “shallow” etched waveguides were fabricated. The MOCVD-grown waveguide structure as described earlier was patterned with 3 μm SiN_x stripes and then etched to about 150 nm above the waveguiding (InGaAsP) layer to create lateral confinement. The mask was then removed and surface roughness of the sample measured with an optical profilometer. The three dimensional (3D) image of the etched surface scanned by the profilometer is shown in Fig. 4. The root-mean-square surface roughness was determined to be 0.66 nm, which is close to the lowest reported value in the literature achieved by RIE.⁶

Waveguides with lengths varying from 3 to 10 mm were characterized for their losses using the Fabry-Perot method. Only zero order mode was observed for the field launched at the center of the waveguide. Figure 5 shows the measured Fabry-Perot fringes of a 3 mm long waveguide (ridge width 3 μm). High contrast between the peaks and troughs of the fringes indicates low propagation losses in the waveguide. In addition, the contrast is quite uniform across the wavelength range measured. The average propagation losses extracted from these measurements are very low, with the values of 0.28 dB/cm for the transverse magnetic (TM) mode and 0.39 dB/cm for the transverse electric (TE) mode. These values of losses are adequate for fabricating state of the art photonic devices.¹⁷ The high quality of these waveguides confirms the excellent surface smoothness of the sample after the etching process.

Because the problem of polymerization is eliminated on the etched surface and does not require O_2 plasma treatment, the use of a hard mask becomes redundant. Standard photoresist mask would greatly simplify the waveguide fabrication process. To test the feasibility of photoresist being employed as the mask for RIE etching, samples with only photoresist mask were etched. Because polymer is being deposited only on top of the mask, this is quite beneficiary for the photoresist mask as the polymer protects the photoresist mask from erosion as a result of ion sputtering.

Waveguides fabricated with the “soft” (photoresist) mask (etch-

ing depth 1.9 μm) had losses identical to those fabricated using the “hard” mask and no erosion of the mask was observed.

Conclusion

A simple, single-step CH_4/H_2 -based RIE process has been developed. By reducing the process pressure we were able to suppress polymer buildup on the etched InP surface. The absence of any polymer on the surface or the sidewalls was confirmed by SEM observation and wet etching. This process was then employed to fabricate 1.9 μm high ridge waveguides, resulting in polymer-free sidewalls and smooth surface with rms roughness of 0.66 nm. Almost vertical ($\sim 85^\circ$) sidewall etching was achieved.

Waveguide losses measured using the Fabry-Perot method were 0.28 and 0.39 dB/cm for the TM and TE modes, respectively. These results confirm the very good quality of the fabricated waveguides and consequently the effectiveness of the developed RIE process.

Simple photoresist masks were successfully employed to fabricate ridge waveguides and their losses were identical to those fabricated using conventional SiN_x masks.

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