Low temperature ICP etching of InP/InGaAsP heterostructure in Cl₂-based plasma

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Abstract. The paper presents the process development results of low temperature ICP etching of InP/InGaAsP heterostructure in a Cl₂/Ar/N₂ plasma using multi-stage process. It is shown that the introduction of additional polishing etch steps effectively removes defect layer formed after etching of the heterostructure. The angle of inclination of the side walls of the elements formed by etching reached 87°, while the thickness of the defect layer did not exceed 80 nm.

1. Introduction

At present, optoelectronics is a dynamically developing field. Various technological platforms are used to create integrated optoelectronic devices. One of these technological platforms are devices based on InP [1]. The creation of optoelectronic devices often requires the formation of waveguide structures with a high aspect ratio and a smooth surface morphology, and the width of the elements can be less than 1 µm [2–4]. The manufacturing process of such devices often requires performing several sequential etching operations. Methods of plasma etching are used for such waveguide structures. One of them, the most common is plasma chemical etching in inductively coupled plasma (ICP). The advantage of this method is the possibility of independent control of the density and energy of plasma ions, which provides flexible control of the etching process.

For plasma etching of InP, processes with the use of gas mixtures based on CH₄/H₂ and Cl₂ are most common [2–14]. Etching of InP-based heterostructures in a chlorine-containing plasma allows to obtain a smooth surface morphology after etching with a high aspect ratio of formed structures. To obtain a profile of structures with a high aspect ratio, passivating additives N₂, O₂, etc. are introduced into the composition of the gas mixture [2–7, 12–14]. However, the processes of etching InP in chlorine-containing plasma, have its own disadvantages. InClₓ compounds formed during the etching process have low volatility at room temperature. The redeposition of InClₓ on the substrate can lead to a high level of surface roughness of the formed structures. To solve this problem, the substrate is heated to 150°C and above [2–5, 7, 8, 10] before etching, which increases the requirements for an etching equipment. In [6, 13, 14] it was shown that it is possible to effectively control the desorption of InClₓ compounds from the substrate surface by increasing the fraction of the physical component of the etching process. Thus, InP etching can be performed without preheating the substrate.
The paper presents the optimization of low-temperature ICP etching process of an InP/InGaAsP heterostructure in a Cl$_2$/Ar/N$_2$ plasma using multi-stage etching.

2. Experimental conditions
In the experiments, semi-insulating InP substrates were used, with an InP/InGaAsP p-i-n heterostructure formed on their surface, and an InGaAs cap layer was formed on top of heterostructure layers.

Process optimization of plasma etching of heterostructure layers was performed using a single-layer dielectric mask. For this purpose, silicon nitride film with a thickness of 300–400 nm was deposited on the surface of substrates, using inductively coupled plasma chemical vapor deposition method. The formation of topology elements in the dielectric was performed by the ICP etching in SF$_6$-based plasma using a single-layer photoresist mask. The photoresist mask was formed by contact lithography.

Substrates with the prepared dielectric mask were divided into samples with an area of about 1 cm$^2$. Further, ICP etching of the heterostructure was performed using a gas mixture of Cl$_2$/Ar/N$_2$ in various modes. The composition of the gas mixture Cl$_2$/Ar/N$_2$ was 10/20/20 sccm, respectively. The value of the inductively discharge power $P_{ICP}$ was fixed at 700 W, the etching pressure was 5–15 mTorr.

After etching, samples were cleaved across formed relief elements. Control of samples was performed using scanning electron microscopy (SEM) related method with the Raith 150 two apparatus. Etching profile, etching depth at the surface level ($h_{etch}$), the thickness of the defect layer ($h_{grass}$) and the total etching depth ($h_{total} = h_{etch} + h_{grass}$) were controlled on samples.

3. Experimental results
Figure 1 shows the dependences of the etching rate of the heterostructure and the thickness of the damaged layer, and figure 2 shows SEM images of the etched structure profiles in a Cl$_2$/Ar/N$_2$ plasma at different values of the $P_{RF}$ power.

![Figure 1](image)

**Figure 1.** Dependencies of the etching rate of the heterostructure in Cl$_2$/Ar/N$_2$ plasma and the thickness of the defect layer after etching on the power $P_{RF}$, with a process pressure of 5 mTorr.

As can be seen from the figures, with an increase in the power $P_{RF}$ and fixed other process parameters, the etching rate of the InP-based heterostructure linearly increased, reaching 1 µm/min with a power of 200 W. But even sharper growth was observed in the thickness of the defect layer. If, at a power $P_{RF}$ of 25 W, a smooth morphology of the substrate surface was observed after the etching process. Then, with an increase in power up to 50 W, the thickness of the defect layer after etching was
about 0.13 µm, and a further increase it up to 75 W led to a sharp increase in the layer thickness to 0.55 µm, reaching 1.5 µm at a discharge power of 200 W.

![Figure 2. SEM images of the cross section of structures formed after etching in a Cl₂/Ar/N₂ plasma with different PRF power: (a) 25, (b) 50, (c) 75 and (d) 200 W, with a process pressure of 5 mTorr.](image)

Also with an increase in power, the anisotropy of the etching process increased. There is a contradiction. On the one hand, it is necessary to increase the power $P_{RF}$ value above 75 W in order to form a vertical profile of the elements after etching. However, it is necessary, on the contrary, to reduce the $P_{RF}$ value of power to 25 W and less in order to form a smooth defect-free surface.

In an attempt to resolve this contradiction, the etching of the heterostructure was performed in several stages. At the first stage, the heterostructure was etched to a total depth of 1.7–2.5 μm. Then one or several cycles of short-term polish-etching were performed with preliminary cooling of samples to room temperature. The etching pressure was increased to 15 mTorr. The etching results are shown in table 1.

| Process # | Main etching | $P_{RF}$ (W) | $h_{total}$ (μm) | $h_{etch}$ (μm) | $h_{grass}$ (μm) |
|-----------|--------------|--------------|------------------|-----------------|------------------|
| 1         | Main etching | $P_{RF} = 75$ W | 1.76             | 1.49            | 0.27             |
| Polishing | $P_{RF} = 65$ W, $t_{etch} = 30$ s | 1.77 | 1.66 | 0.11 |
| Main etching | $P_{RF} = 65$ W, $t_{etch} = 3×30$ s | 1.82 | 1.72 | 0.1 |
| 2         | Polishing | $P_{RF} = 65$ W, $t_{etch} = 30$ s | 2.47 | 2 | 0.47 |
| Main etching | $P_{RF} = 100$ W | 2.42 | 2.2 | 0.22 |
| 3         | Polishing | $P_{RF} = 65$ W, $t_{etch} = 3×30$ s | 2.47 | 2 | 0.47 |
| Polishing | $P_{RF} = 100$ W, $t_{etch} = 30$ s | 2.45 | 2.22 | 0.23 |
|          | $P_{RF} = 100$ W, $t_{etch} = 3×30$ s | 2.44 | 2.36 | 0.08 |

As can be seen from table 1, an increase in the process pressure to 15 mTorr resulted in a decrease in the initial thickness of the defect layer in comparison with the data obtained by etching at a process pressure of 5 mTorr. This is apparently due to the decrease in the energy of ions entering the substrate. The introduction of polish-etching cycles resulted in a consistent decrease in the thickness of the defect layer. The process proceeded most efficiently at equal $P_{RF}$ power values in the main process and polish-etching cycles (process #3). As the gap in $P_{RF}$ power between the main process and polish-
etching cycles increased, the removal efficiency of the defect layer decreased (process #2). At the same time, the total etching depth $h_{\text{total}}$ in all three etching modes remained almost unchanged. This indicates that at a substrate temperature close to room temperature, bulk etching is practically absent, and only the defect layer is etched.

Figure 3 shows SEM images of the cross-section of etched heterostructures using multi-stage process #3. Structures had a close to rectangular profile (angle of side walls slope up to 87°). The defect layer was practically absent. At the same time, its appearance has changed, instead of a continuous grass-like layer; individual peaks up to 80 nm high were observed.

Figure 3. SEM images of the cross section of structures formed after etching in a Cl$_2$/Ar/N$_2$ plasma using a multi-stage process #3: (a), (b) waveguides; (c) etched surface.

4. Conclusion

The paper presents the process development results of ICP etching of InP-based heterostructures in the Cl$_2$/Ar/N$_2$ plasma using multi-stage process. The developed etching process has a high anisotropy (angle of side walls slope up to 87°). It is shown that the introduction of additional polish-etching cycles performed in the same mode, but at a substrate temperature close to room temperature, can effectively remove the defect layer without increasing the total etching depth of the heterostructure. The depth of the defect layer reached 80 nm and can be improved by introducing additional polish-etching cycles. An advantage of the process is also no need for preheating the substrate.

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