Study and optimization of room temperature inductively coupled plasma etching of InP using Cl₂/CH₄/H₂ and CH₄/H₂

Chee-Wei Lee*, D. Nie, T. Mei, M.K. Chin

Photonics Research Centre, School of Electrical and Electronic Engineering, Nanyang Technological University, Singapore 639798, Singapore

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Abstract

We report an optimized room-temperature etching recipe for Indium Phosphide (InP) based on the inductively coupled plasma (ICP) reactive-ion etch using Cl₂/CH₄/H₂ gasses. The process was optimized using design of experiment (DOE) (Taguchi method). The results, in terms of etch rate, surface roughness and etched profile, are compared with the more conventional CH₄/H₂ without chlorine. The Cl₂-based recipe does not require substrate heating and thus can be more cost effective and widely applied. The Cl₂/CH₄/H₂ process generally gives a reasonable higher etch rate (as high as 848 nm/min) and cleaner surface with no polymer formation, but it requires a high ICP power. The CH₄/H₂ process produces lower etch rate (with possibly polymer contamination), but smoother surface and better structural verticality at a lower ICP power. Both processes give very good selectivity against the silicon dioxide (SiO₂) mask. The selectivity of InP against oxide mask (up to 35:1) for the Cl₂/CH₄/H₂ process is one of the highest reported so far. The etched structure possesses good verticality and good surface quality comparable to that obtained under elevated temperature condition (>200 °C).

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Indium Phosphide (InP) is a highly important base material used to fabricate many optoelectronic devices for optical communication applications. For the fabrication of many photonic devices [1–5], dry etching is often essential to obtain the precise dimensions and highly anisotropic structural profile [6,7]. Different chemistries, such as CH₄/H₂ [8–13], Cl₂ (with different additives) [14–18], SiCl₄ [19], BCl₃ [20] and N₂/H₂ [21] have been demonstrated for etching InP. The most widely used recipes are based on CH₄ and Cl₂. For CH₄/H₂ chemistry, the reaction products are highly volatile as compared to the chlorine-based chemistry for substrate temperature lower than 200 °C. This is advantageous in achieving good surface and sidewall qualities. However, the polymer formation and low etch rate could be problematic.

For chlorine-based recipes, the polymer formation is avoided and the etch rate is much improved, but the need to raise the substrate temperature to around 200 °C or more to improve the volatility of etched products would incur additional cost and process time, and limit the etch mask to dielectric materials or metals (photoresists cannot be used at such high temperatures). Thus, it would be desirable to optimize the Cl₂-based processes to achieve reasonable etch rate and surface quality at room temperature (i.e., without intentional heating). Cl₂/CH₄/H₂ [22] and BCl₃ [20] recipes have been reported for InP etching using electron cyclotron resonance (ECR) at slightly lower temperature (~150 °C), and the etch rates obtained were 250 and 800 nm/min, respectively. Cl₂/Ar and Cl₂/CH₄/Ar etching of InP using ECR [23] and inductively coupled plasma (ICP) [24], respectively, at room temperature have been reported, but the physical bombardment by the heavy Argon ions (Z = 40) could cause significant surface damage. In this letter, we report for the first time the optimization of ICP etching of InP in Cl₂/CH₄/H₂ gas mixture under room temperature condition. The results, in terms of etch rate, surface roughness and etched profile, are compared with those using CH₄/H₂ gas mixture. ICP is advantageous over reactive-ion etching (RIE) because it...
provides independent control of the ion energy and plasma density, and also over ECR because it is relatively simple and more economical for production [25].

In the optimization, the Taguchi method [26] was used. Taguchi method provides a more systematic and efficient way of determining near-optimum etching parameters. It greatly reduces the number of experimental configurations required to quantify the influence of each parameter, and to obtain the near optimum process parameters. An Oxford Plasmalab 100 ICP machine (RF source of 13.56 MHz) was used in the dry etching of semi-insulating InP. The SiO2 etch mask is deposited using plasma-enhanced chemical vapor deposition (PECVD), and patterned using photoresist AZ-5214E and RIE. The chamber is pumped down to $5 \times 10^{-6}$ Torr prior to the process. The etch depth is determined by a surface profiler, and the etching profile is observed by scanning electron microscopy (SEM) and atomic force microscopy (AFM).

For the etching of InP with Cl2, due to the involatility of the InClx product at room temperature, the surface tends to be rough because of the formation of InClx clusters. Hence, CH4 gas is added as the product formed between CH4 and indium is volatile even at low temperature. Also, H2 is added to improve the phosphorus removal and to smoothen the etching surface. However, as complementary gases to Cl2, their flow rates will be much smaller compared to the optimized CH4:H2 process. It is found that the etch rate improves with higher concentration of chlorine gas and lower CH4 and H2 flow rates. As CH4 and H2 flow rates are increased, the chlorine gas is more diluted and the etch rate is reduced. With pure chlorine and high ICP power, very high etch rates can be obtained but the surface is rough due to the formation of InClx clusters. Therefore, CH4 and H2 are still needed to reduce the surface roughness. Generally as observed by SEM, the etched material surface is much cleaner than pure CH4/H2 process due to the lack of polymer formation caused by chlorine cleaning [27].

Based on our design of experiment (DOE) results, as a compromise between high etch rate and smooth surface, we selected the RF power, Cl2, CH4 and H2 flow rates to be 100 W, 10, 8 and 4 sccm, respectively. For the Cl2 gas, 10 sccm is chosen as a compromise between etch rate and surface quality, as reactive gases like chlorine tend to cause more damage to the surface [27]. Higher CH4 flow rate is chosen to avoid the formation of indium-rich surface. A moderate H2 flow rate is chosen to smoothen the etching surface without reducing the etch rate seriously. With these parameters set, the ICP power is varied at two different process pressure levels to investigate its effect on the etch rate, surface quality and structural verticality. As seen in Fig. 1, the etch rate increases with the ICP power at the low-pressure level (4 mTorr). At the higher pressure level (15 mTorr), the etch rate is initially small and increases linearly only when the ICP power is higher than 800 W. This probably because, under the higher process pressure, it is harder for the reacted product to escape from the material surface. More polymers could be formed on the etching surface as well under the higher process pressure. Hence, until the ICP power (i.e., ion density) exceeds a certain threshold value, then, the product will be cleared away by the ion-assisted desorption [23]. The low process pressure not only gives a higher etch rate, but also a more vertical etch profile as the ions are less deflected off axis at low pressure. These results suggest that a wide range of etch rate and profiles can be obtained by selecting the appropriate ICP power.

The optimized etched profile is shown in Fig. 2(a). The ICP power used is 1200 W, and the process pressure is chosen to be 4 mTorr. The DC bias is about −240 V. The etch rate obtained is about 488 nm/min, with selectivity against oxide of 35:1. The rms surface roughness is about 3.7 nm, as shown in Fig. 2(b). The relatively high etch rate and anisotropic etching obtained at high ICP power, even without intentional substrate heating, suggests that the InClx etch products are being efficiently removed by ion-assisted desorption, which prevents the formation of thick InClx selvedge layer [23].

\[ \text{CH}_4/\text{H}_2 \] recipe has been widely used for the etching of InP. It is demonstrated that the recipe is able to produce better surface and sidewall roughness than the Cl2-based process [27] due to the high volatility of the etched products, i.e., In(CH3)3 and PH3. The gases used are also non-corrosive and nontoxic, and no substrate heating is required. For a typical waveguide application, the optimum recipe that gives reasonably high etch rate and smooth surface is found to be the combination of 110 W for RF power, 200 W for ICP power, 18 mTorr for process pressure, and 30:10 sccm for the CH4:H2 gas flow ratio. The DC bias is about −465 V. To prevent polymer formation on the SiO2 surface that may cause overcutting of the sidewall [12,28], and also to maintain a cleaner material surface over a long etching duration, we have added 1 sccm of O2 gas (2.5% of total flow rate), which has

**Fig. 1.** Etch rate as a function of ICP power at two different process pressure levels, for the Cl2/CH4/H2 process. The RF power is 100 W, Cl2 flow rate is 10 sccm, CH4 flow rate is 8 sccm and H2 flow rate is 4 sccm.
no significant effect on the etch rate. This recipe gives a very smooth surface and a nearly vertical etch profile, as shown in Fig. 3(a). The rms surface roughness is found to be about 1.69 nm, as shown in Fig. 3(b). The etch rate for InP is about 114 nm/min, with a selectivity of 58:1 against oxide. Although an etch rate as high as 600 nm/min was reported before with Argon addition in ICP [29], the rms surface roughness (> 70 nm) is inferior. The relatively high selectivity is due to the low RF power, and the non-reactive nature of the gases used.

In conclusion, we have optimized the CH$_4$/H$_2$ and Cl$_2$/CH$_4$/H$_2$ process for ICP etching of InP by utilizing the Taguchi method. The etch rates produced are reasonable, and good surface quality is obtained. The Cl$_2$-based recipe does not require substrate heating and thus can be more cost effective and widely applied. The two processes have different properties and are suitable for different applications. The Cl$_2$/CH$_4$/H$_2$ process generally gives a significantly higher etch rate and smoother surface with no polymer formation, but requires a high ICP power. The CH$_4$/H$_2$ process produces lower etch rate (with possibly polymer contamination), but smoother surface and better structural verticality at a lower ICP power. A small amount of oxygen is added to eliminate polymer formation. The recipe without chlorine is also safer to use. Lower ICP power is preferable to reduce surface and sidewall damage. Both processes give very good selectivity against the oxide mask. The selectivity of InP against oxide mask for Cl$_2$/CH$_4$/H$_2$ process is one of the highest reported so far.

References