# Optimization of inductively coupled plasma etching for low nanometer scale air-hole arrays in two-dimensional GaAs-based photonic crystals\*

Peng Yinsheng(彭银生)<sup>1,†</sup>, Ye Xiaoling(叶小玲)<sup>1</sup>, Xu Bo(徐波)<sup>1</sup>, Jin Peng(金鹏)<sup>1</sup>, Niu Jiebin(牛洁斌)<sup>2</sup>, Jia Rui(贾锐)<sup>2</sup>, and Wang Zhanguo(王占国)<sup>1</sup>

(1 Key Laboratory of Semiconductor Materials Science, Institute of Semiconductors, Chinese Academy of Sciences, Beijing 100083, China) (2 Institute of Microelectronics, Chinese Academy of Sciences, Beijing 100029, China)

**Abstract:** This paper mainly describes fabrication of two-dimensional GaAs-based photonic crystals with low nanometer scale air-hole arrays using an inductively coupled plasma (ICP) etching system. The sidewall profile and surface characteristics of the photonic crystals are systematically investigated as a function of process parameters including ICP power, RF power and pressure. Various ICP powers have no significant effect on the verticality of air-hole sidewall and surface smoothness. In contrast, RF power and chamber pressure play a remarkable role in improving sidewall verticality and surface characteristics of photonic crystals indicating different etching mechanisms for low nanometer scale photonic crystals. The desired photonic crystals have been achieved with hole diameters as low as 130 nm with smooth and vertical profiles by developing a suitable ICP processes. The influence of the ICP parameters on this device system are analyzed mainly by scanning electron microscopy. This fabrication approach is not limited to GaAs material, and may be efficiently applied to the development of most two-dimensional photonic crystal slabs.

Key words: photonic crystal; GaAs; inductively coupled plasma etching; scanning electron microscopy DOI: 10.1088/1674-4926/31/1/012003 PACC: 5240H; 5275; 4270Q

## 1. Introduction

Photonic crystals  $(PC)^{[1,2]}$  are optical materials with periodic changes in the dielectric constant on the wave-length scale, analogous to the crystal structure of a semiconductor, in which photonic band gaps can be created for certain ranges of photon energies. Various applications have been predicted and are expected to be realized by photonic crystals, including ultra small optical circuits, photonic integrated circuits and quantum dot single photonic sources<sup>[3, 4]</sup>. Ideally, such optical electronic devices should be realized using a three-dimensional system, but an alternative system using a dielectric slab with a two-dimensional (2D) photonic crystal, called a 2D PC slab<sup>[5, 6]</sup>, is also very attractive for its relatively easy fabrication. It uses the effect of the 2D PCs to confine light in the in-plane direction, and the refractive index contrast to confine light in the vertical direction<sup>[7, 8]</sup>.

The fabrication of PC necessitates pattern transfer techniques with good control of both structure size and shape. Recently, the requirements have become more important with the resurgence of structures requiring vertical profiles and very smooth surfaces in dimensions on the order of hundreds of nanometers, e.g., photonic band gap devices<sup>[9–11]</sup>, which are particularly demanding in this respect. Among the different dry etching methods available, inductively coupled plasma (ICP) seems to be the most appropriate candidate with its large flexibility for plasma control and uniformity. It is possible, unlike in a reactive ion etch (RIE) system, to control independently the density of the plasma and the energy of the ions to achieve large etch rates without significant crystallographic damage for a wide range of materials<sup>[12]</sup>.

There have been some reports of ICP etching of GaAsbased photonic band gap devices<sup>[13, 14]</sup>. One of the main challenges, however, remains the fabrication of photonic band gap structures where the main difficulty lies in the realization of PC with air-hole diameter < 150 nm features with vertical and smooth sidewalls. In this paper, we mainly describe the fabrication of 2D PC micro-cavities with low nanometer scale air-hole arrays using an ICP etching system. The sidewall profile and surface characteristics of PC are systematically investigated as a function of process parameters including ICP power, RF power and chamber pressure, and the mechanisms are also discussed. PC structures with air hole diameters as low as 130 nm and etch depths of around 400 nm with uniform size, vertical sidewalls and smooth profiles have been achieved by optimizing various process parameters in a chlorine-containing plasma etching system.

## 2. Experiment

The sample structure reported in this article has been designed for quantum dot (QD) single photon resources for long wavelength applications in two-dimensional GaAs-based PC micro-cavities. The materials used here consist of a 200-nmthick undoped GaAs slab waveguide grown by molecular beam epitaxy (MBE) and clad by air on the upper surface and a 1000nm-thick sacrificial Al<sub>0.7</sub>Ga<sub>0.3</sub>As layer on the n-type GaAs substrate side. A single layer of InGaAs QDs is embedded 100 nm below the surface in the center of the GaAs waveguide.

\* Project supported by the State Key Development Program for Basic Research of China (Nos. 2006CB604904, 2006CB604908) and the National Natural Science Foundation of China (Nos. 60676029, 60990315, 60625402).

<sup>†</sup> Corresponding author. Email: cynthia@semi.ac.cn

Received 15 July 2009, revised manuscript received 18 August 2009

Due to the typical relatively poor dry-etching resistance for the electron beam resist<sup>[15]</sup>, a hard mask has been used instead. The hard mask used in this article is SiO<sub>2</sub>. Although the SiO<sub>2</sub> mask is prone to erosion under high temperature processes, it also has the advantage of easy patterning and RIE etching with well defined profiles, which is crucial to ensure good sidewall quality and verticality. 150 nm of SiO<sub>2</sub> is first deposited by plasma enhanced chemical vapor deposition. The thickness of the SiO<sub>2</sub> layer is measured using an ellipsometer. Electron beam lithography is then used to write the test patterns, typically 300 nm, on the SiO<sub>2</sub> mask layer. RIE with a CHF<sub>3</sub>/Ar plasma is used to etch the SiO<sub>2</sub> layer for pattern transfer.

The ICP tool used in this study is a load-locked plasma etching system based on a cylindrical coil configuration. Power is coupled to the plasma by exciting the coil with a 2 MHz RF potential. The resulting magnetic field induces a circumferentially oriented electric field that largely fills the chamber, creating high density plasma. Ion energies are controlled by superimposing a RF bias of 13.56 MHz on the sample. The samples are loaded into the reactor by mounting them on a sapphire carrier wafer and the wafer is then clamped to the cathode. The ICP chamber is conditioned by running a BCl<sub>3</sub>/Cl<sub>2</sub> plasma for 30 min prior to conducting the designed experimental runs. Based on previous experience with this material system and similar devices, a mixture of  $BCl_3/Cl_2/Ar$ , in the ratio of 1/1/3, is deemed suitable for this particular process chemistry. For all the tests described in this article, the same sample size is used and the etch time is controlled using an end-point detection system. With such conditions, the three main parameters under investigation are ICP power, RF power and chamber pressure. Sidewall and surface characteristics are inspected under a scanning electron microscope. Additionally, in our experiment, the three primary requirements on the etching are firstly, the etch depth is required to reach the requisite thickness, i.e. the etched holes need to extend through the GaAs slab waveguide into the sacrificial layer (> 200 nm), in order to form a suspending photonic crystal slab structure by undercut chemical wet etching the Al<sub>0.7</sub>Ga<sub>0.3</sub>As sacrificial layer. Secondly, the etched holes need to be vertical to reduce loss. Finally, the etched sidewalls should be as smooth as possible to reduce scattering losses.

#### 3. Results and discussion

## 3.1. Influence of the RFpower

Theoretically, the RF power level sets a DC bias, which is basically a potential difference between the plasma coils and the lower electrode upon which the sample sits. This DC bias drives the ions into the sample. A large DC bias will impart significant kinetic energy to the ions, making physical sputtering a dominant process. A large ion sputtering component will significantly affect the etch mask as well. In addition, the large DC bias can heat the sample.

The influence of the RF power is here studied by keeping the chamber pressure, ICP power and other parameters constant. The process is performed at a pressure of 2 mTorr with ICP power arbitrarily fixed at 300 W as a starting point. RF power is then varied over a range of 50–110 W. Figures 1 shows typical SEM photographs of a photonic crystal with the RF power between 50 and 100 W. The significant characteristics

observed are that high RF power (100 W) causes the enlargement of air holes for photonic crystal structures: the diameter of an air hole is around 220 nm, which is noticeably larger than that of the air hole on the  $SiO_2$  patterns (the hole diameter of the SiO<sub>2</sub> mask layer patterns is around 110 nm.). In addition, significant roughness is introduced into the upper edges of air hole sidewalls, though the bottom part of the holes and the surface of GaAs are very smooth. However, relatively lower RF power (50 W) results in a bowing profile on the upper part of holes, while holes exhibit good circularity and uniformity on the surface of the GaAs slab, and the diameter of holes is around 130 nm, which is nearly equal to the diameter of holes on the SiO<sub>2</sub> mask layer. This can be explained by the fact that increasing the RF power will be translated into an increase in the acceleration of the ions, and the etching process is largely dominated by physical sputtering, as shown in Fig. 2(a), which will decrease the etching selectivity between the GaAs and SiO<sub>2</sub> materials. The enlargement and sidewall roughness of air holes probably results from erosion of the SiO<sub>2</sub> mask, which, as it recedes, leads to degradation of the intended etch pattern, as shown in Fig. 2(c). When the mask recedes enough to reach the underlying substrate, this will result in enlargement of the etch pattern and the introduction of significant roughness on the top edge of etch sidewalls. However, under low RF power, the etching process is largely dominated by chemical etching, as shown in Fig. 2(b). In addition, mask erosion can be delayed by decreasing the RF power, leading to the improvement of selectivity between the GaAs material and SiO<sub>2</sub> mask, thus forming the holes with almost equal dimensions of SiO<sub>2</sub> patterns. On the other hand, the etch depth of holes is around 400 nm at high RF power and around 300 nm at low RF power, i.e. the corresponding aspect ratio is around 1.8 : 1 and 2.3 : 1, respectively, indicating the higher etch ratio at high RF power. However, the etch depths all extend through the GaAs slab waveguide region completely for the two different RF powers and reach the requisite etch depth.

#### 3.2. Influence of the chamber pressure

The chamber pressure is another important process parameter. Typically, the chamber is held at a baseline pressure of  $\sim 10^{-7}$  Torr when no process is being run; typical process pressures are below 10 mTorr. Based on our previous experiments, the effect of the chamber pressure on the etch behavior can be somewhat difficult to gauge. In principle, if the etch is one in which chemical etching is the dominant mechanism, an increased chamber pressure will increase the concentration of the reactive elements and can speed up the etch rate. If the etch is primarily a physical sputtering process, an increased pressure will also initially increase the etch rate, but may eventually cause it to slow, as the increased pressure may cause collisions between ions that will reduce the kinetic energy with which they bombard the surface.

In our experiment, the influence of chamber pressure on the verticality of photonic crystals with nanometer scale airhole diameters is now discussed, keeping other parameters constant. When the RF power and ICP power are fixed at 70 W and 300 W, respectively, the pressure is varied between 1.0 and 1.8 mTorr in an attempt to find a suitable value for obtaining the desired verticality. Figure 3 shows SEM photographs for the etched photonic crystals with the chamber pressure varying



Fig. 1. (a), (b) SEM photographs of photonic crystals when RF power is 100 W, exhibiting remarkable enlargement of air holes and roughness on the upper part of holes. (c) SEM photograph of a cleaved device at 70 W power after GaAs etching but before the removal of the SiO<sub>2</sub> mask layer and sacrificial layer, showing the bowing shape of sidewalls. (d) SEM photograph from the top view after chemical wet etching the SiO<sub>2</sub> mask layer and sacrificial layer, exhibiting relatively uniform and rounded air holes on the GaAs slab surface.



Fig. 2. Schematic pictures showing a variety of profiles of etched substrates and masks with the verticality degraded by physical sputtering effects represented by a radical effect. (a) Physical sputtering. (b) Chemical etching. (c) Mask retardation.

over 1.3 and 1.6 mTorr. It can be observed that at 1.6 mTorr, the sidewall exhibits a bowing shape in the middle of the holes (see Figs. 3(a) and 3(b)). The hole diameter on the surface of GaAs is around 130 nm and the depth of holes is around 300 nm, which is similar to the profile of holes at the low RF power above, but they have two remarkably different characteristics. On one hand, the bowing profiles are formed in the middle of the holes, not on the upper part of the holes as in those formed at low RF power; on the other hand, the profile here is more rounded than that of the holes mentioned above. This probably results from different etching mechanisms.

When the chamber pressure decreases, the bowing shape profile is gradually eliminated, and the sidewalls exhibit trapezoid profiles when the pressure is 1.3 mTorr, as seen in Figs. 3(c) and 3(d); the diameter and etch depth of holes is around 130 nm and 350 nm, respectively, which is nearly equal to the diameter of holes at high chamber pressure. A possible reason for this is that at higher chamber pressures the energetic ions undergo an increased frequency of collisions before they strike the sample, which begins to randomize the ion trajectories and degrade the etch anisotropy, so the etching curvature of the sidewalls is rather evident. Moreover, increasing the chamber pressure also implies that the chemical-etching component is enhanced. When the chamber pressure decreases, the balance between physical sputtering and chemical etching is improved, and thus the bowing shape is gradually eliminated. On the other



Fig. 3. Magnified SEM photographs of profiles with different chamber pressures. (a), (b) At high chamber pressures, a bowing profile which is similar to that at low RF power is seen. (c), (d) At low chamber pressures, the bowing profile is gradually eliminated and a trapezoid profile is formed.

hand, the aspect ratio is 2.3 : 1 and 2.7 : 1 for high chamber pressure and low chamber pressure, respectively. This can be explained by the fact that ions obliquely incident into holes have difficulty reaching the bottom of the hole, as the size of the hole is narrower because the mean-free-path of ions at higher pressures is short; in other words, obliquely-incident ions are increased. However, at a lower pressure, the mean-free-path is much longer than that at a higher pressure, or in other words, normally-incident ions are dominant, so that a relatively high aspect ratio is created at low pressures, but the etch depths all extend through the GaAs active region completely into the sacrificial layer.

## 3.3. Influence of ICP power

In principle, ICP power controls the density of ionized atoms. In addition, it can have an effect on the sample temperature because dense plasmas generated by high ICP powers can cause heating of the sample, which can influence the etch rate, sidewall profiles, and hence are expected to have an impact on the chemical aspect of the etching. This effect has been exploited in our etching of photonic crystals with wide dimensions. However, here it is operated as a function of ICP power between 200 and 300 W, keeping the pressure (1.3 mTorr) and RF power (70 W) fixed; we notice little variation in etching quality as a function of ICP power over the range of ICP powers explored, but the etch depth is deeper than the GaAs active layer. One possible reason for this is that photonic crystals with low nanometer scale dimensions are less dependent on temperature variation over the range of ICP power explored.

#### 3.4. Optimum conditions for photonic crystals fabrication

The conditions that lead to the above effects are affected to a possibly larger extent by the confined etching geometry. For photonic crystals with large size patterns as in the previous experiment, it can be confirmed that the optimum conditions are obtained for RF and ICP power fixed, respectively, at 70 W and 300 W with a pressure of 2 mTorr at room temperature. This process is nonetheless applied to those of small sizes with the same epitaxial wafer structure. The design used in this article is based mainly on photonic crystal micro-cavities for QD single photon sources with hole diameters < 150 nm.

Taking the factors mentioned above into account, it is found that RF power and chamber pressure have the maximum influence on surface quality and, especially, on the verticality of sidewalls, while ICP power has less influence on etching quality. The primary modification here in the process is an optimization in RF and chamber pressure. Based on the above experimental results, the final photonic crystal etching conditions are settled on: the chamber pressure is 1.0 mTorr, RF power is 90 W, ICP power is 200 W, and the etching time is 160 s.



Fig. 4. SEM photographs of a photonic crystal after optimizing the various process parameters. (a) The holes of a photonic crystal exhibit significantly vertical and smooth sidewalls. (b) Top view close-up SEM photograph of a complete photonic crystal micro-cavity.

The resulting devices exhibit significantly smooth and vertical sidewalls, as shown in the SEM photograph of Fig. 4(a). No bowing profile is observed in the sidewall, indicating suitable process parameters among RF power, chamber pressure and ICP power. In addition, the holes exhibit uniform and circular size, with the diameter of holes being as low as around 130 nm and the etch depth around 400 nm.

#### 4. Conclusions

In conclusion, we have studied the plasma etching of twodimensional GaAs-based photonic crystals using an ICP etching system. The influence of the RF power, chamber pressure and ICP power on the surface smoothness and verticality of hole profiles has been systematically investigated and an optimum process has been developed for the achievement of the desired photonic crystal structures with low nanometer scale holes. RF power and chamber pressure have the maximum influence on the verticality and play a significant role in improving the sidewall verticality and etching surface quality of air holes. In contrast, ICP power has no significant effect on etching characteristics for the tool conditions used here. Its mechanisms have been also discussed and are thought to be due to the balance effect between physical sputtering and chemical etching. Vertical and smooth air holes with hole diameters as low as 130 nm have been achieved by careful optimization of the various etching parameters.

#### Acknowledgements

The authors are sincerely grateful to Dr. Fan Zhongchao at the Engineering Research Center of Semiconductor Integrated Technology, Institute of Semiconductors, Chinese Academy of Sciences, and Dr. Li Junjie at the Laboratory of Microfabrication, Institute of Physics, Chinese Academy of Sciences, for valuable discussions, and to Mrs. Hu Ying and Mrs. Liang Ping for their help and encouragement throughout the course of this work.

#### References

- Yablonovitch E. Inhibited spontaneous emission in solid-state physics and electronics. Phys Rev Lett, 1987, 58: 2059
- [2] John S. Strong localization of photons in certain disordered dielectric superlattices. Phys Rev Lett, 1987, 58: 2486
- [3] Chang W H, Chen W Y, Chang H S, et al. Efficient single-photon sources based on low-density quantum dots in photonic-crystal nanocavities. Phys Rev Lett, 2006, 96: 117401
- [4] Englund D, Fattal D, Waks E, et al. Controlling the spontaneous emission rate of single quantum dots in a two-dimensional photonic crystal. Phys Rev Lett, 2005, 95: 013904
- [5] Johnson S G, Fan S H, Villeneuve P R. Guided modes in photonic crystal slabs. Phys Rev B, 1999, 60: 5757
- [6] Chutinan A, Noda S. Waveguides and waveguide bends in twodimensional photonic crystal slabs. Phys Rev B, 2000, 62: 4488
- [7] Noda S, Chutinan A, Imada M. Trapping and emission of photons by a single defect in a photonic bandgap structure. Nature, 2000, 407: 608
- [8] Song B S, Noda S, Asano T. Photonic devices based on in-plane hetero photonic crystals. Science, 2003, 300: 1537
- [9] Yablonovitch E. Applied physic—how to be truly photonic. Science, 2000, 289: 557
- [10] Noda S, Tomoda K, Yamamoto N, et al. Full three-dimensional photonic bandgap crystals at near-infrared wavelengths. Science, 2000, 289: 604
- [11] Ferrini R, Houdre R, Benisty H, et al. Radiation losses in planar photonic crystals: two-dimensional representation of hole depth and shape by an imaginary dielectric constant. J Opt Soc Am B, 2003, 20: 469
- [12] Etrillard J, Bresse J F, Daguet C, et al. New self-aligned processes for III–V electronic high speed devices. J Vac Sci Technol A, 1999, 17: 1174
- [13] Combrié S, Bansropun S, Lecomte M, et al. Optimization of an inductively coupled plasma etching process of GaInP/GaAs based material for photonic band gap applications. J Vac Sci Technol B, 2005, 23: 1521
- [14] Reese C, Gayral B, Gerardot B D, et al. High-Q photonic crystal microcavities fabricated in a thin GaAs membrane. J Vac Sci Technol B, 2001, 19: 2749
- [15] Youtsey C, Adesida I. In: Shul R J, ed. Handbook of advance plasma processing techniques. Berlin: Springer, 2000: 466