

Plasma etching characteristics of Ge-B-P doped SiO₂ film for waveguide fabrication

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Ge-B-P doped silica films were fabricated by Flame Hydrolysis Deposition and their plasma etching characteristics were investigated for the fabrication of waveguide devices. Ge-B-P doped silica films were used for the Planar Lightwave Circuit device employed in the next generation optical communication networks. Since the deep etching of silica film is required for the successful fabrication of these devices, Inductively Coupled Plasma etching was employed in this work. To find the optimum etching conditions, the etching characteristics in terms of various etching conditions, such as reactive gas flow rate, the source power, and the bias power, were studied. The formation of fluorocarbon polymer deposition was investigated and the correlation to the overall etching characteristics, such as the etching speed, the roughness of the etched surface and sidewall, were analyzed.

Key words: Optical waveguide, PLC, ICP, Plasma etching.

Introduction

Silica based Planar Lightwave Circuit (PLC) becomes a key component in modern WDM communication systems. Especially, in the Fiber To The Home (FTTH) scheme where the access network connecting an optical fiber network directly to a small office or home user is emphasized, the need for cheap and reliable Wavelength Division Multiplexing (WDM) passive components is driving the research on various types of PLC devices such as splitter, coupler, and WDM Multiplexer (MUX)/Demultiplexer (DEMUX). Materials for PLC devices are normally SiO₂-based amorphous glasses doped with P, B, and Ge. These dopants are used to control the refractive index and viscous sintering temperature of the core and clad layers which are essential units of a waveguide structure. SiO₂ has long been known as the most reliable passive dielectric medium with extremely stable chemical durability and low propagation loss. Without this material, modern optical communication would not be realized. Most of the commercialized PLC devices employ SiO₂-based materials to build up the waveguide structure and the reason is clear if one consider the stability and excellent passive optical properties of this material.

Similarly to Vapor-phase Axial Deposition (VAD) or Outside Vapor Deposition (OVD) method used in the manufacture of silica optical fibers, the clad and core layer of SiO₂ PLC devices are deposited by a hydro-

lysis reaction of chloride sources in a high temperature oxyhydrogen flame, which process is called Flame Hydrolysis Deposition (FHD) [1, 2]. A silicon wafer is normally used as a substrate in this process. Films deposited by FHD are normally in the form of porous soot and subsequently these porous films are sintered into dense and transparent amorphous SiO₂ glass films. After deposition, the core layer is etched into a square waveguide by lithography and a plasma dry etching process which are common processes semiconductor. By employing these process technologies proven in the semiconductor industry, the integrated optical waveguide devices have been mass produced cheaply.

The plasma dry etching process used to fabricate ridge or channel type waveguides is basically identical to that in the conventional semiconductor industry except for the depth to be etched and the aspect ratio to be produced. In single mode PLC waveguides operating at a wavelength of 1.55 μm (the wavelength used in the modern optical communication networks), a square waveguide with an edge dimension of approximately 8 μm is needed under $\Delta=0.7\%$ (the index difference between core and clad). Therefore, the plasma etching process to fabricate these waveguides should etch this depth easily and quickly while maintaining a low sidewall roughness of waveguides. In the viewpoint of a semiconductor process engineer, this work is challenging since the etching depth and the etching rate to be achieved are quite large. Furthermore, in the waveguide, the shape of the waveguide should be square which requires highly selective etching, and the roughness of the waveguide sidewalls must be maintained low which requires a precise control of the reactivity of the etching gases. The lateral dimensions also must be precisely

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preserved during the etching process since the propagation modes in the branching section of waveguide devices are very sensitive to lateral dimensions. Therefore, the branching sections even in simple waveguide devices such as splitters and couplers require an etching profile with an extremely high aspect ratio, typically $\sim 80:1$.

Considering the requirements in waveguide the fabrication process, the most suitable plasma etching process might be an Inductively Coupled Plasma (ICP) etching technique, in which the high density plasma formed from the reactive gases is used for etching, since the bombardment effect and the reaction effect can be controlled independently by controlling the bias voltage and plasma source power. The superiority of this technique over the Reactive Ion Etching (RIE) technique has been proven for various materials and this is the case for SiO_2 [3].

In the research described here, the plasma etching characteristics of amorphous SiO_2 films doped with Ge, B, and P were studied using an ICP etching technique as functions of various etching parameters such as source power, bias voltage, and reactive gas flow rate. The suitability of this technique for the etching of SiO_2 films and optimum etching conditions were sought to fabricate silica PLC devices.

Experiments

The film with a core composition (Ge-B-P doped SiO_2) was deposited by FHD. The source chemicals were SiCl_4 , GeCl_4 , BCl_3 , and POCl_3 and the carrier gas (Ar); the flow rate of these source chemicals were 44.5, 25, 20, and 10 sccm, respectively. The temperature of source chemicals was about 39°C . The temperature of the oxyhydrogen flame was about 1600°C and that of the substrate was 200°C . The porous soot films deposited on silicon wafers were sintered at 1350°C into transparent amorphous films. The final thicknesses of the films were between 6 to 8 μm . The compositions of sintered dense films were typically $\text{SiO}_2:\text{GeO}_2:\text{B}_2\text{O}_3:\text{P}_2\text{O}_5 = 79.8:15.5:0.2$ in wt %. The refractive indices of films were measured by a Prism Coupler on several spots to confirm the uniformity of the film quality and composition, which were typically 1.4545 ± 0.0005 at $\lambda = 1.55 \mu\text{m}$.

A Cr etching mask was selected since it adheres to silica film well and is strong enough to resist the bombardment of the reactive gas and Ar^+ ions in the plasma during an etching process of long duration, typically 40 minutes [4]. The selectivity, the etching ratio between the silica film and the mask film, against the reactive gas used in this study, CF_4 , is also high enough to achieve an etching depth of more than 6 μm . The Cr film was deposited on the silica film by DC Magnetron Sputtering up to 700 nm. To pattern the Cr etching mask, photoresist film was coated onto the Cr mask by

a spin coater and exposed to 365 nm UV light for 1 minute under a silica photomask after soft baking at 135°C . Hard baked photoresist film was then wet-etched by a photoresist stripper. The Cr film was also wet-etched by a Cr etchant solution.

Plasma etching was carried out in the ICP etcher. The source power (RF2, 13.56 MHz) generating a helical motion of ions was supplied to the coil antenna attached to the top plate of the chamber. A bias voltage (RF1, 13.56 MHz) was applied to the electrodes located at the top and bottom of chamber and was designed to be controlled independently of the source power. The source power generated the high density plasma through the helical motion of ions and the bias voltage generated a vertical motion or bombardment motion of the ions. The gases used for etching were Ar and CF_4 . The total pressure in the chamber, the CF_4 flow rate, and the powers of RF1 and RF2 were controlled as etching process variables to optimize the etching behavior.

The etched profile and surface morphology were observed by Scanning Electron Microscopy (SEM) and the surface roughness was measured by an optical interferometric surface profiler (OISP) or Atomic Force Microscopy (AFM). X-ray Photoelectron Spectroscopy (XPS) was used to analyze the etching residue on the surface.

Results and Discussion

The reactive gas CF_4 has been known to exhibit no pure chemical etching effect on SiO_2 -based materials and is unlikely on Si [5]. CF_4 does not adsorb dissociatively on SiO_2 , which means that an F radical, the crucial chemical etching agent in the case of Si etching, does not adsorb on SiO_2 . The unsaturated radicals such as CF_2 and CF_3 , which are formed from CF_4 feedstock gas by the plasma, are also known not to spontaneously etch SiO_2 chemically. Therefore, the plasma etching process of SiO_2 by CF_4/Ar is not "pure chemical" etching even though a reactive gas is used. However, the chemical effect of etching is achieved by the aid of ion bombardment or the sputtering effect generated by the externally supplied bias voltage. Accelerated ions collide on the surface of the film or reaction products and make these materials more reactive by supplying extra energy. Ion bombardment is known to have two effects on the etching process. It generally increases the F/CF_y (unsaturated) ratio, which may adversely affect the etching of SiO_2 due to a lower concentration of unsaturated gases. It also accelerates desorption of the CF_y related polymer deposited on the SiO_2 surface, which enhances the etching of SiO_2 .

As mentioned above, unsaturated radicals such as CF_2 and CF_3 are known to form a $\text{SiC}_x\text{O}_y\text{F}_z$ "polymer" film by the reaction with SiO_2 under the assistance of plasma ion bombardment and this polymer layer plays a key role in the etching process [5, 6]. The polymer

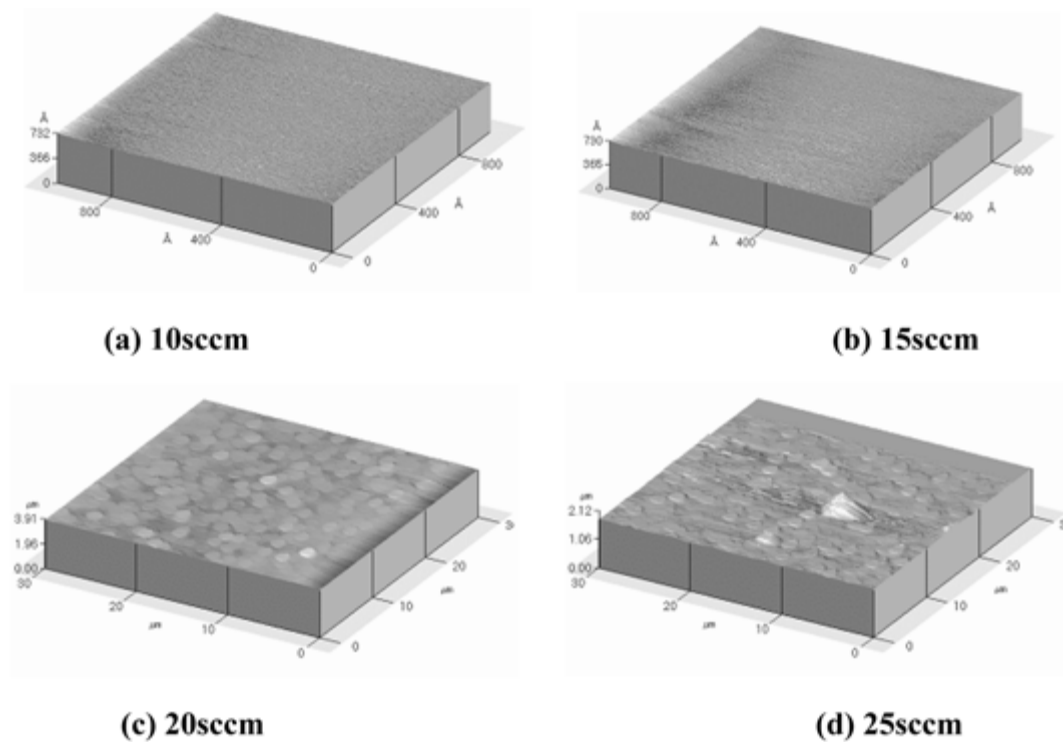


Fig. 1. AFM images showing the variation of surface roughness with gas flow rate. Source power 1000W, bias voltage 150V, working pressure 5 mTorr (6.7×10^{-5} Pa).

formed produces the volatile etching products containing Si and O, such as SiF₄, SiF₂, SiOF, CO, CO₂, and COF₂. These volatile etching products are easily flushed out by the gas flow supplied from outside the chamber. Therefore, a balance of the thickness of the residual polymer layer is critical in the etching process. Too thick a polymer layer may prohibit further etching by weakening the plasma bombardment effect on the SiO₂ underneath the polymer layer, while too thin a polymer layer may slow down the etching process due to insufficient formation of volatile species.

In this experiment, the polymer layer was observed to form under a high flow rate of CF₄ source gas. Figure 1 shows the surface roughness of etched surfaces as a function of CF₄ gas flow rate. As the flow rate of the source gas increased, the etched surface became rougher which means that there were unetched residual polymer layers left even after the etching process. The surface roughness increased severely at a flow rate of more than 15 sccm and this result suggests that the polymer layer formed was too thick to be etched out under the applied etching conditions. The presence of the polymer layer was also checked by XPS as shown in Figure 2. The C 1s peaks were detected from this polymer layer and this result confirms that the polymer layer is formed on the surface of SiO₂ and remained after the etching process. The peak positions of C 1s in this study were between 290–292 eV, which are close to that of fluorinated carbon peaks measured on a flat open etched surface [7, 8]. The C 1s peaks are known

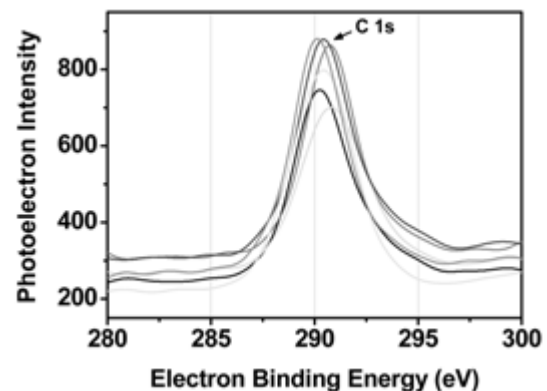


Fig. 2. XPS C 1s peak of the etching residue on the Ge-B-P doped SiO₂ films etched by ICP. XPS was scanned several times at various spots on the same specimen.

to exhibit a peak shift depending on the bonding state of the carbon. The bonding of carbon to oxygen leads to a peak shift of +1.5 eV per single bond, whereas the fluorination of carbon produces an even larger peak energy shift [8]. Compared to that reported in reference [8], the peak position of C 1s in this study showed a large peak shift which is almost more than 5 eV. This suggests that the fluorocarbon films formed on the surface of SiO₂ in this study contain large amounts of F and O and the F/C ratio is relatively high, probably bigger than 1.

The etching rate also reached a saturated value of ~220 nm/minute at a flow rate above 15 sccm as shown

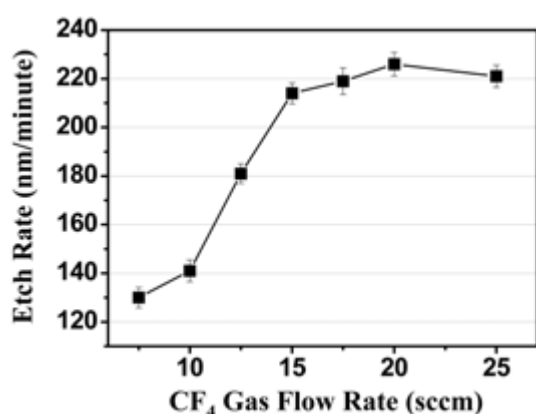


Fig. 3. Etching rate as a function of gas flow rate. Source power 1000W, bias voltage 150V, working pressure 5 mTorr (6.7×10^5 Pa).

in Figure 3, which means that the polymer layer formed became thicker than the optimum thickness for the given etching conditions. At a flow rate lower than 15 sccm, the etching rate exhibited the sharp increase as a function of the flow rate of the source gas. This result indicates that at a low flow rate of the source gas the formation of the polymer layer is actively assisting the etching process and the etching process is accelerated as the amount of the polymer increases. Therefore, under this experimental condition the etching proceeds in a “chemically-enhanced sputtering regime.” However, once the thickness of the polymer layer becomes thicker than the optimum, the polymer layer becomes an inhibitor of the etching process and the etching rate becomes slower. In this condition, the etching process can be assigned to a “fluorocarbon polymer suppression regime.” Similar results were reported by other authors. In particular, Jung et al. [6] reported that the etching rate showed a mild increase as a function of the CF₄ source gas flow rate. It is believed that their flow rate was from 10 to 30 sccm where the polymer layer starts to accumulate and the etching process becomes inefficient due to the thick polymer layer. They also observed the formation of a polymer layer by Scanning Electron Microscopy and this polymer layer looked like “grass.”

The etching process can be controlled by other etching process parameters, such as source power and bias voltage. Figure 4 shows the effect of the source power on the etching rate. As the source power increases, the etching rate shows a mild increase. Since in this experiment a saturated gas, CF₄, was used for as the reactive source gas, it had to be decompose to unsaturated gas species in order to produce reactive gaseous species such as CF₂ and CF₃. This decomposition process is accomplished by plasma energy and the decomposed species would become abundant as the source power increases. Therefore, the etching rate is expected to increase as the source power increases and the unsaturated gaseous species become more abundant. Jung et

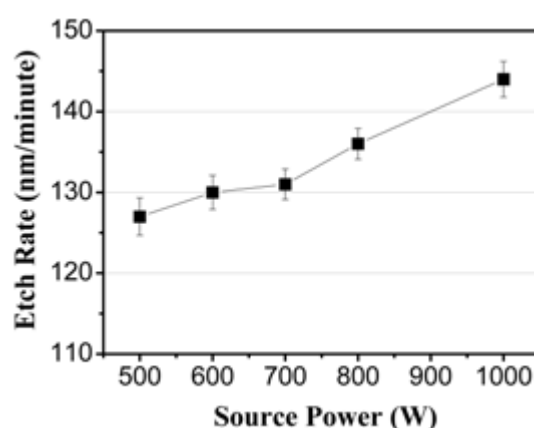


Fig. 4. Etching rate as a function of source power (RF2). Bias voltage 100V, CF₄ gas flow rate 10sccm, working pressure 5mTorr (6.7×10^5 Pa).

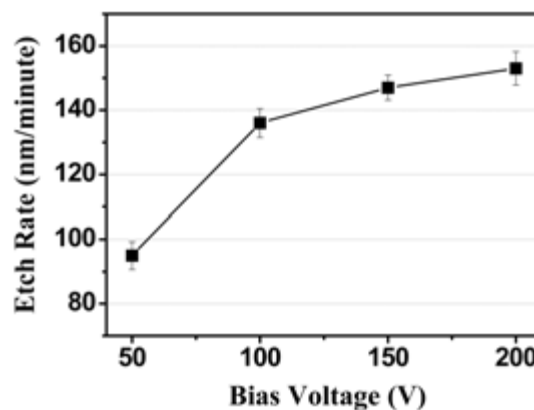


Fig. 5. Etching rate as a function of bias voltage (RF1). Source power 800W, CF₄ gas flow rate 10sccm, working pressure 5mTorr (6.7×10^5 Pa).

al. also reported similar results [6].

Bias voltage also has a profound impact on the etching behavior. Bias voltage accelerates the gaseous species charged by the source plasma power and generates the ion bombardment effect which is the energy source of the surface reaction producing the volatile etching products. Therefore, it is expected that as the bias voltage increases the etching rate increases, while the selectivity between etching mask material and etched material decreases. As expected, the etching rate measured in these experiments increases as the bias voltage increases as shown in Figure 5. It was also experimentally observed that the deposition of the fluorocarbon polymer layer was reduced as the bias voltage was raised. As the fluorocarbon polymer reacts with the oxygen originating from SiO₂ to form volatile etching products and this reaction is enhanced by ion bombardment, the increased bias voltage would induce a faster removal of the polymer layer leading to a high etching rate. The dependency of removal of the fluorocarbon polymer layer on the ion flux or ion energy has been studied extensively [9] and it was reported that in holes with high aspect ratio the ion energy loss effect leads to a

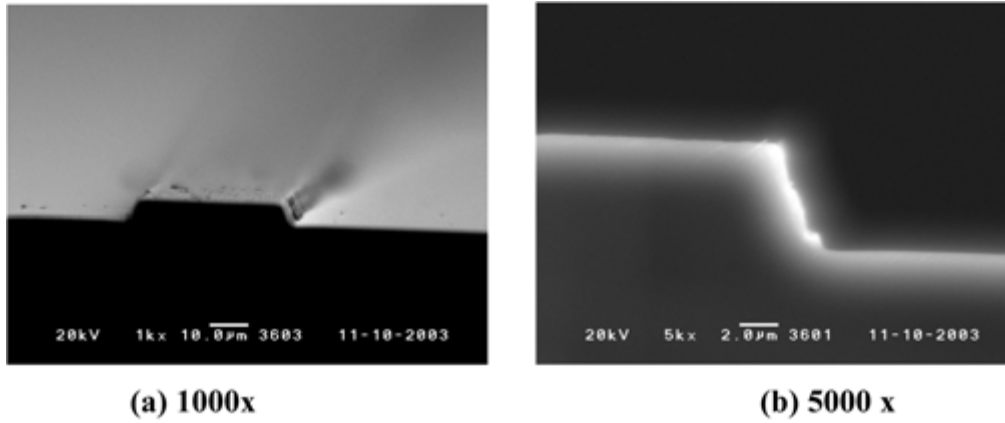


Fig. 6. Examples of SEM micrographs of non-vertical etched sidewall. Source power 1000W, bias voltage 150V, CF₄ gas flow rate 15sccm, working pressure 5mTorr, etching time 30 minutes.

high deposition rate of the polymer in the bottom of holes. This experimental observation and explanation can be applied successfully to the results in this work.

The Cr etching mask used in this work was deposited up to ~500 nm thick, which was almost etched out after the etching process of 6 mm thick silica waveguide films. This result indicates that the selectivity between the Cr mask and silica film was roughly 1:12. In conjunction with mask etching, the dimensional loss at the top of waveguide pattern has to be checked since mask erosion results in dimensional loss and a sloped profile. In some etching conditions, the sidewall angle was not vertical and the waveguide profile sloped as shown in Figure 6. A waveguide with a sloped profile, however, did not show any lateral dimensional loss at the top of the waveguide, which means that mask erosion is not the cause of the sloped profile. The formation of a sloped profile was analyzed precisely by Bazylenko and Gross [10]. By assuming isotropic polymer deposition, a steady state sidewall angle ϕ_0 is determined to be the value at which the etching and deposition rate of the polymer layer establishes a balance, $ER_{polymer}(\phi) = DR_{polymer}$. Here, $ER_{polymer}$ and $DR_{polymer}$ are the etching and deposition rate of the polymer layer. The polymer etching rate is comprised of the ion flux to the sidewall and reactive sputtering yield, and the angular dependence of the etching rate comes from the angular dependence of the reactive sputtering yield as shown in the following Eq. (1);

$$ER_{polymer} \propto c_1 + c_2 I_a \cos(\phi) Y(I_a, E_i, \phi), \quad \alpha d \leq 1. \quad (1)$$

The deposition rate of the polymer layer is assumed not to exhibit an angular dependence and is then given in Eq. (2);

$$DR_{polymer} \propto \gamma(T) I_p. \quad (2)$$

Here, c_1 and c_2 are the proportional coefficients, I_a is the flux of active etching species at the polymer surface,

α is the probability of polymer etching by diffusing active species per unit of film thickness, d is the thickness of the polymer film, I_i is the ion flux, ϕ is the sidewall angle, Y is the reactive sputtering yield, E_i is the ion energy, γ is the sticking probability of the polymer-forming species, and I_p is the flux of polymer-forming species. Therefore, to increase the steady state sidewall angle ϕ_0 to achieve a more vertical sidewall, the polymer deposition rate should be kept low while the polymer etching rate should be high enough. A steady state sidewall angle ϕ_0 lower than 90°, like the case shown in Figure 6, means that the polymer deposition rate is too high. Therefore, the polymer deposition rate should be lowered by reducing the flow rate of source gas or the polymer etching rate should be increased by increasing the bias voltage. Since increasing the bias voltage, however, deteriorates the selectivity between the mask and the etched material, it is better to reduce the flow rate of the source gas to produce a vertical deep etching profile even though the total etching rate of the film is reduced.

Conclusions

The plasma etching of Ge-doped silica glass waveguide film was performed using high density plasma of a reactive gas, CF₄, in an inductively coupled plasma etcher. As theoretically predicted, the overall etching characteristics were strongly dependent on the flow rate of the reactive gas, which determines the formation of the polymer layer on the etched film. It has been shown that the etching rate saturates quickly as the flow rate of the CF₄ gas increases. The saturation occurs in the range of the transition between the polymer “enhancing” and “suppression” regime. It is adequate to consider the effects of other factors affecting the etching process such as source power and bias voltage by interpreting the polymer formation since the reactive etching is mediated by the polymer formed during the reaction.

As shown by the experimental results in this study and theoretical analysis, it is clear that the most crucial factor which should be carefully controlled during the reactive etching is the flow rate of the reactive gas and the resulting polymer formation. In waveguide etching, the importance of an accurate control of the polymer formation can never be overemphasized since the etching depth and the aspect ratio to achieve are relatively high and the verticalness and smoothness of sidewall are critical in the waveguide performance.

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