

Optimization of reactive-ion etching (RIE) parameters for fabrication of tantalum pentoxide (Ta_2O_5) waveguide using Taguchi method

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Abstract. In this paper, we demonstrate the optimization of reactive-ion etching (RIE) parameters for the fabrication of tantalum pentoxide (Ta_2O_5) waveguide with chromium (Cr) hard mask in a commercial OIPT Plasmalab 80 RIE etcher. A design of experiment (DOE) using Taguchi method was implemented to find optimum RF power, mixture of CHF_3 and Ar gas ratio, and chamber pressure for a high etch rate, good selectivity, and smooth waveguide sidewall. It was found that the optimized etch condition obtained in this work were RF power = 200 W, gas ratio = 80 %, and chamber pressure = 30 mTorr with an etch rate of 21.6 nm/min, $\text{Ta}_2\text{O}_5/\text{Cr}$ selectivity ratio of 28, and smooth waveguide sidewall.

1 Introduction

Recently there has been an increasing interest in fabrication and characterization of waveguide layer and photonics crystal (PC) slab structure for investigating various optical nonlinear effects such as optical parametric amplification process [1], supercontinuum generation [2] and harmonic generations [3]. The planar waveguide and the PC slab structure are a desirable structure to realize a compact optical components device (e.g., tunable laser source). Among the available materials, the tantalum pentoxide (Ta_2O_5) has received much attention due to its high refractive index ($n > 2$), low absorption over a wide wavelength region 0.3 to 10 μm [4], high nonlinear refractive index $n_2 = (7.23 \pm 0.36) \times 10^{-19} \text{ m}^2/\text{W}$ [5], large value of χ^3 [6] and high optical damage threshold [7]. Recently, researchers have shown optical non-linear behaviour and demonstrated supercontinuum generation [8] and OPO [9] in Ta_2O_5 waveguides, suggesting a good stability and high damage threshold limits.

In order to make waveguide geometry and PC slab structure, Ta_2O_5 material has to be etched. However, it has been found that Ta_2O_5 material is quite challenging to be etched thus smooth vertical waveguide sidewall is difficult to be realized. The smooth vertical waveguide sidewall of waveguide is essential for low propagation loss transmission. A dry etching technique using a reactive-ion etching (RIE) etcher can be used to etch Ta_2O_5 as it is able to produce finer features with higher anisotropic etch profile. The RIE etcher has several variables factors (i.e., RF power, gas ratio and process pressure) that correspond to output parameters (i.e., etch rate, selectivity, surface roughness and sidewall angle). It requires many experiments run to find optimum setting

thus increases development time and cost. Thus design of experiment (DOE) of Taguchi method can be employed for the optimization process. This method is more systematic and efficient approach for determining near optimum settings, with small number of experiments configurations due to the use of orthogonal array.

In this work, design of experiment (DOE) of Taguchi method was employed to optimize the RIE process parameters for fabrication of Ta_2O_5 waveguide for a high etch rate, good selectivity, and smooth waveguide sidewall using RIE etcher in Southampton Nanofabrication Centre. A mixture of trifluoromethane (CHF_3) and argon (Ar) gas ratio was used with chromium (Cr) hard mask in this experiment. Optimum conditions setting of Ta_2O_5 etching recipe have been determined for smooth vertical sidewall, high etch rate and selectivity.

2 Experiment

2.1. Samples preparation

6-inches silicon wafers with $\langle 100 \rangle$ orientation, coated with 2 μm silicon dioxide (SiO_2) was used as an initial wafer. A first layer of 400 nm Ta_2O_5 and second layer of 1 μm positive resist (Shipley S1813) were deposited on the wafer. Waveguide pattern from a mask was exposed by an EVG 620T mask aligner. The exposed resist was then dissolved by a developer (Shipley MF319), and hard baked at 120 $^\circ\text{C}$ for 30 minutes. Wet etching (Cr etchant) was used to transfer the pattern into Cr metal layer and the residual resist was then removed by Acetone. The sample was diced into 2 x 2 cm small chip

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size. Samples were then inspected by optical microscope to reject defective chips prior to the etching experiments.

2.2. RIE system configuration

The etching of Ta₂O₅ experiments were carried out in OIPT Plasmalab 80 RIE etcher. This system has equipped with fluorine based reactive gas. It consists of parallel plate electrode powered with 13.56 MHz RF system. The substrate is placed on top of lower electrode, connected with RF generator. The opposite electrode is located on the roof of the chamber connected to ground. Plasma contains ions and neutral species is generated by exposing a neutral gas (Ar) to the strong RF potential power at low pressure. Ions generated from plasma are accelerated in the electric field, bombarding with surface of the material. The neutral species reacts chemically with samples and the material can be etched away. The RIE takes advantage of both the physical and the chemical etching mechanism and etch rates that are faster with an anisotropic etching profile are achieved. This RIE machine has a range limitation of the RF power and the chamber pressure which its optimum operating power is at 200 W and minimum pressure is at 15 mTorr respectively. A quartz cover plate was used in this experiment.

2.3. Experimental parameters

In our experiment, three factors such as RF power (W), gas ratio (%) and chamber pressure (mTorr) and levels were selected to optimize high etch rate, good selectivity and the smooth vertical waveguide sidewalls. Based on preliminary experiment data and machine operational limitation, range and levels of the variable were selected as shown in Table 1.

Table 1. Range and level of parameters selected in this experiment.

Factor	RF power (W)	Gas ratio (%) CHF ₃ /(CHF ₃ +Ar)	Chamber pressure (mTorr)
Level 1	100	20	15
Level 2	150	50	30
Level 3	200	80	45

A combination of CHF₃ and Ar gas ratio was used to etch Ta₂O₅ material. Gas ratio of 20, 50, and 80 % were a mixture of CHF₃ = 10 sccm and Ar = 40 sccm, CHF₃ = 25 sccm and Ar = 25 sccm, and CHF₃ = 40 sccm and Ar = 10 sccm respectively. In this case L9 orthogonal array was selected; nine different runs were executed as shown in Table 2. The temperature of substrate plate and the etching time were kept constant at 20 °C and 10 minutes respectively.

Table 2. A L9 orthogonal array

Trial No.	RF power (W)	Gas ratio (%) CHF ₃ /(CHF ₃ +Ar)	Chamber pressure (mTorr)
1	100	20	15
2	100	50	30
3	100	80	45
4	150	20	15
5	150	50	30
6	150	80	45
7	200	20	15
8	200	50	30
9	200	80	45

2.3. Experimental procedure

The experiment was executed according to experimental configuration as listed in Table 2. In this experiment, initial thickness of Ta₂O₅ and Cr layer of the pre etching sample is measured by ellipsometer and Dektak profiler respectively. The process chamber is cleaned by striking oxygen plasma for 5 minutes to remove any residual polymers deposited on a wall inside chamber process from previous etching run. The process chamber cleanliness is essential to obtain a repeatability of etching process. After venting procedure, a chamber lid is opened and the sample is then placed at a centre of the quartz cover plate. The chamber lid is closed and pumped down. Once a 0 mTorr pressure is achieved, a selected recipe is then executed. During the etching process, a reflected power and a DC bias value at process window is closely monitored to check if any unstable process occurs. When the etching process is finished, the process chamber is vented and the sample is unloaded. The thickness of Ta₂O₅ and the etch depth of the post etching sample is then measured by the same method that is mentioned above. An etching rate and a selectivity of the each experiment runs are calculated as described by Figure 1 and equations 1, 2, and 3. Figure 2 shows the SEM image of pre etching process of the waveguide samples. The commercial software Minitab® has been used to analyse experimental results based on Taguchi method to determine optimum level of RF power, gas ratio and chamber pressure for smooth vertical waveguide sidewall and high etch rate and selectivity. Finally, a separate verification test was performed to fabricate ridge Ta₂O₅ waveguide with the obtained optimum parameters in this work. The sample etch profile and waveguide sidewall roughness was observed with a scanning electron microscope (SEM).

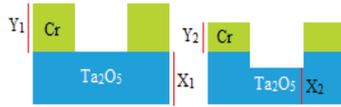


Fig. 1. Schematic of pre and post etching of waveguide sample for etch and selectivity calculation.

$$Ta_2O_5 \text{ etch rate} = (X_1 - X_2) / \text{Etching time} \quad (1)$$

$$Cr \text{ etch rate} = Y_1 - (Y_2 - (X_1 - X_2)) / \text{Etching time} \quad (2)$$

$$\text{Selectivity} = Ta_2O_5 \text{ etch rate} / Cr \text{ etch rate} \quad (3)$$

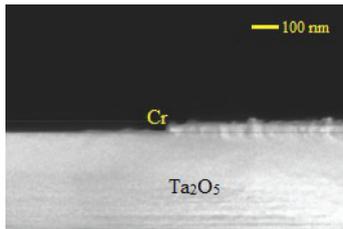


Fig. 2. SEM image of pre-etching process waveguide sample with 50 nm Cr hard mask and 400 nm Ta₂O₅ layer.

3 Results and Discussion

The experimental results of etching rate and selectivity of different experiment runs as listed in Table 2 are summarized in Table 3.

Table 3. Experimental results

Trial No.	Ta ₂ O ₅ etch rate (nm / min)	Cr etch rate (nm / min)	Selectivity (Ta ₂ O ₅ /Cr)
1	7.5	0.7	11.1
2	8.8	1.2	7.4
3	6.9	1.2	5.8
4	13.2	0.8	17.6
5	13.4	1.6	8.5
6	15.3	1.4	11.2
7	17.1	2.1	8.2
8	15.4	0.5	30.4
9	21.6	0.8	28

An advantage of Taguchi method is its design experiment is orthogonal which mean of each level can easily be calculated thus influential rank of three factors can be estimated as shown in Table 4.

Table 4. Mean of each level of three factors

Parameter	Level	RF power (W)	Gas ratio (%)	Chamber pressure (mTorr)
Ta ₂ O ₅ etch rate	1	7.7	12.6	12.7
	2	14.0	12.5	14.5
	3	18.0	14.6	12.5
	Delta	10.3	2.1	2.0
	Rank	1	2	3
Cr etch rate	1	1.0	1.2	0.8
	2	1.2	1.1	0.9
	3	1.1	1.1	1.6
	Delta	0.2	0.1	0.8
	Rank	2	3	1
Ta ₂ O ₅ / Cr selectivity	1	8.1	12.3	17.6
	2	12.4	15.4	17.7
	3	22.2	15.0	7.5
	Delta	14.1	3.2	10.2
	Rank	1	3	2

In Table 4, delta is the value of maximum mean minus minimum mean. This delta value indicates an influential factor for each parameter response. For Ta₂O₅ etch rate the rank of three factors is RF power > Gas ratio > Pressure. For Cr etch rate case the rank is Pressure > RF power > Gas ratio and for the selectivity the rank is RF power > Pressure > Gas ratio.

Ta₂O₅ etch rate and selectivity were chosen for detailed study. As can be seen, the rank of each influential factor for each parameter response was different. The aim of this work is to obtain vertical sidewall of Ta₂O₅, thus etch rate and selectivity were focused for detailed study. It can be observed from Figure 3(a) that RF power has significant influence on Ta₂O₅ etch rate. It is found that increasing RF power increases Ta₂O₅ etch rate. This trend is as expected which higher RF power provides large voltage difference (higher DC bias) that accelerates positive ions and neutral species to bombard the material to be etched with high energy. Besides, higher concentrations of reactive species will be formed with increasing RF power.

The Gas ratio and chamber pressure factors are less significant compared to RF power on Ta₂O₅ etch rate as indicated in Figure 3(a). As can be observed from the graph, etch rate of Ta₂O₅ increased with increasing gas ratio. High gas ratio means that high flow rate of CHF₃ reactive gas is injected into chamber compared to Ar gas. It could be explained that the increase of CHF₃ flow rate

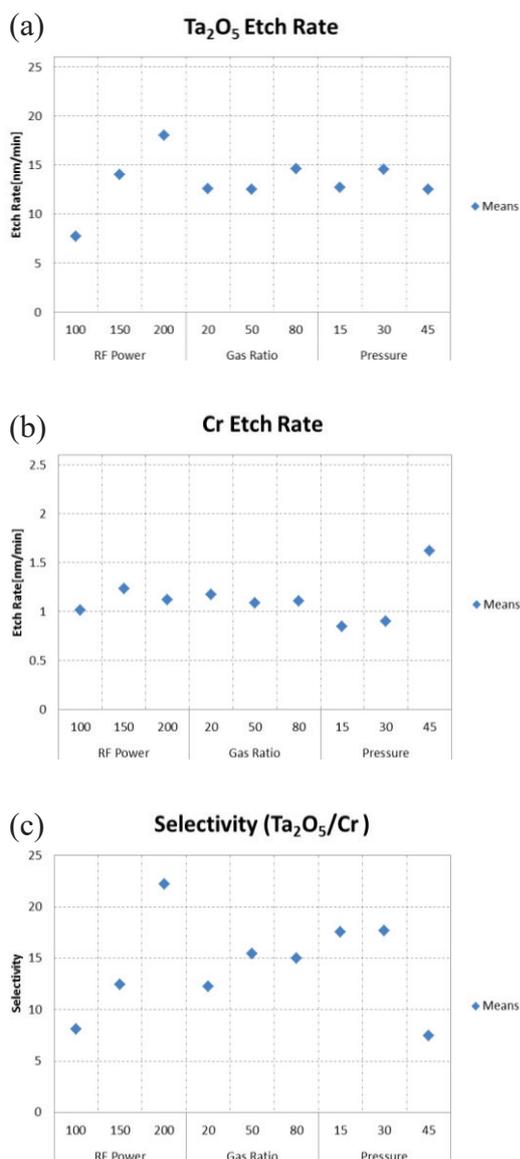


Fig. 3. Trend charts of levels of factors versus parameters. (a) Ta₂O₅ etch rate (b) Cr etch rate and (c) Selectivity (Ta₂O₅ / Cr).

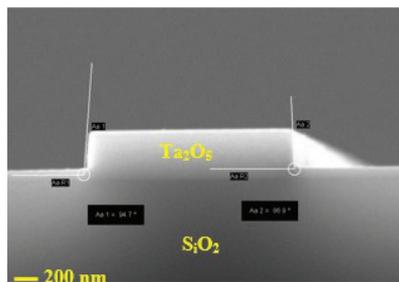


Fig. 4. SEM image of post-etching sample of optimized parameters (Trial No. 9).

increase the density of F atoms, hence increase the Ta₂O₅ etch rate. From the graph we found that Ta₂O₅ etch rate increases as the chamber pressure increases. This phenomenon could be due to the increase in gas density (positive ions and neutral species) thus enhance the etch rate. However, the etch rate was eventually dropped at chamber pressure of 45 mTorr. Beyond a certain limit, gas density in the process chamber result in more

collisions of ions and species which can reduce mean free path of bombarding ions, thus reduce etch rate. Furthermore, more collisions of ions may affect the number of ions attacking vertically towards the sample (loss of direction) thus anisotropic profile could be degraded. Based on the experimental results, optimum values for three variable factors for high etch rate and selectivity are RF power of 200 W, gas ratio of 80 % (CHF₃ = 40 sccm, Ar = 10 sccm) and chamber pressure of 30 mTorr. As can be observed in Table 2, the combination of optimum values were considered in L9 orthogonal array (Trial No. 9). The etching profile of this sample was observed by SEM as displayed in Figure 4. Based on the figure, about 87~89 ° of smooth vertical sidewall is obtained. The etch rate of these parameters is 21.6 nm/min. We carried out the verification experiment using the same process parameters with etching time of 20 minutes with the aim to fabricate Ta₂O₅ ridge waveguide (100 % of 400 nm Ta₂O₅ layer to be fully etched).

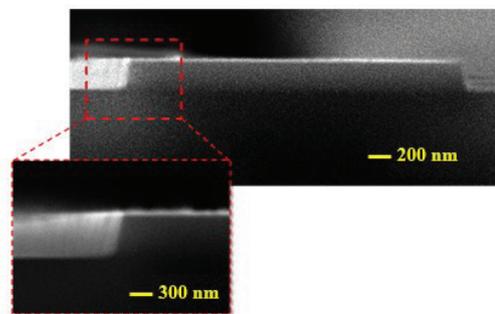


Fig. 5. SEM image of verification run samples with 20 minutes of etching time.

Figure 5 shows a smooth sidewall of the etching profile for this verification run. However, it should be noted that the obtained sidewall angle is about 83~85 ° less vertical from the previous run with identical process parameters and shorter etching time. The less vertical sidewall must have been due to longer etching time. These optimized parameters can be used for fabrication of Ta₂O₅ waveguide in this work, but are not suitable for PC slab structure formation. This is because the PC slab requires highly smooth vertical sidewall of PC structure to minimize low loss transmission [10]. A superior inductively coupled plasma (ICP) etching can be utilized to obtain higher etch rate and smooth vertical sidewall angle [11].

4 Conclusions

In this paper, DOE of Taguchi method was employed to investigate the optimum etching recipe for fabrication of Ta₂O₅ by using OIPT Plasmalab 80 RIE etcher. The variable factors studied were RF power, gas ratio and chamber pressure. Based on the experimental data, it was found that the optimum conditions for RF power, gas ratio and process pressure were 200 W, 80 % and 30 mTorr respectively. With the obtained optimum setting, smooth sidewall angle of 87~89 ° with 21.6 nm/min etch

rate and selectivity of 28 were demonstrated. However, waveguide sidewall angle was slightly decreased to 83~85 ° when the etching time was increased to 20 minutes maybe due to loading effect.

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