Int. J. Nanoelectronics and Materials **4** (2011) 37-48

Improvement in processing of micro and nano structure fabrication using O_2 plasma

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Received 23 October 2009; received in revised form 17 February 2010; accepted 9 April 2010

Abstract

Plasma has frequently been used by the industry as a last step surface preparation technique in an otherwise predominant wet-etched process. Recent research of the chemistry of plasma led to a great understanding of plasma processes. It is by controlling the plasma conditions and gas mixtures, ultra-fast plasma cleaning and etching is possible. With enhanced organic removal rates, plasma processes become more desirable as an environmentally sound alternative to traditional solvent or acid dominated process, not only as a cleaning tool, but also as a patterning and machining tool. In this paper, improvement in the processing of nanogap fabrication using O_2 plasma is discussed including the parameters for PR patterning with two times limited in the O_2 plasma process. For applications that have not been possible with limited usefulness, plasma processes are now approaching the realm of possibility.

We introduce this work to fabricate and characterize the nanogap device fabrication using O_2 plasma technique for biosensor fabrication. In this review, two masks designs are proposed. The first mask is for the lateral nanogap and the second mask is for a gold pad electrode pattern, and the lateral nanogap is introduced in the fabrication process using silicon, and gold as an electrode. Conventional photolithography technique is used to fabricate this nanogap (NG) based on the plasma etching technique. The increase in etching time when we apply the O_2 plasma means an increase in the amount of etching, while in the case of reducing the time of etching means reducing the amount of etching, as shown in the results.

Keywords: Plasma Etching; Photolithography; Nanogap; Electrodes.
PACS: 52.7.Bn; 85.40.Hp; 62.23.-c; 82.45.Fk.

1. Introduction

Advanced semiconductor integrated circuit device chips are typically manufactured through processes which provide multiple conductive layers and multiple insulating layers upon a plurality of active semiconductor substrate regions. These layers and regions are typically defined and formed through etching and masking processes which involve

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photolithographic exposure and developing of blanket photo resist layers. Commonly, a photo lithographically exposed blanket photo resist layer will be developed into a patterned photo resist layer through which features, to be formed within or upon a semiconductor substrate, are defined.

Subsequent to photolithographic exposure and development, patterned photo resist layers which remain upon a semiconductor substrate may be exposed to semiconductor manufacturing processes which significantly alter the chemical or physical structure of those patterned photo resist layers. Such manufacturing processes may include: (1) implanting of high doses of dopant species into those patterned photo resist layers, (2) exposure of those patterned photo resist layers to substantial quantities of radiative energy, or (3) exposure of those patterned photo resist layers to elevated temperatures. Each of these manufacturing processes may produce changes in patterned photo resist layers which yield substantial concentrations of oxidized species within those patterned photo resist layers.

Typically, patterned photo resist layers are removed prior to forming over-lying layers of the semiconductor structure within which those patterned photo resist layers were formed. Commonly, patterned photo resist layers are removed from the semiconductor substrates upon which they were formed through exposure of the patterned photo resist layers to the oxygen Reactive Ion Etched (RIE) plasma. However, patterned photo resist layers which contain substantial quantities of oxidized species may be difficult to remove from semiconductor substrates through the oxygen Reactive Ion Etched (RIE) plasma processes since oxidized species within patterned photo resist layers are often unaffected by the chemical and physical effects of the oxygen Reactive Ion Etched (RIE) plasma. Thus, it is often necessary to include into such oxygen Reactive Ion Etched (RIE) plasma a reducing material which may react effectively with the oxidized species contained within the oxidized patterned photo resist layer. The most common reducing material which may be incorporated into the oxygen Reactive Ion Etched (RIE) plasma to assist in the removal of oxidized species within the patterned photo resist layers is hydrogen gas. Hydrogen gas is typically incorporated into the oxygen Reactive Ion Etched (RIE) plasma in the form of gas mixtures such as nitrogen/hydrogen and helium/hydrogen mixtures.

While oxygen Reactive Ion Etched (RIE) processes which contain reducing materials such as hydrogen gas are usually quite effective in removing oxidized organic residues such as oxidized photo resist residues from the semiconductor substrates, such Reactive Ion Etched (RIE) plasma is not free from the problems. In particular the hydrogen gas which contains reducing materials when used within the oxygen Reactive Ion Etched (RIE) plasma is typically expensive in comparison with other materials used within the Reactive Ion Etched (RIE) plasma. In addition, hydrogen gas mixtures when supplied to the oxygen Reactive Ion Etched (RIE) plasma also possess an inherent safety concern which is related to the explosive characteristics of the hydrogen gas in ambient atmospheres containing oxygen.

Therefore, although it is desirable to provide the oxygen Reactive Ion Etched (RIE) plasma containing a sufficient quantity of reducing material to effectively remove oxidized organic residues such as oxidized photo resist residues from the semiconductor substrates, it is also desirable that the reducing material incorporated into that oxygen Reactive Ion Etched (RIE) plasma, supplies the plasma which is both economical and safe to use in a manufacturing environment. It is towards these two goals that the present invention is directed [1].

The main steps in this review is to fabricate the micro and nanogap structure using O_2 plasma technique, SOI wafer is used to fabricate the lateral micro and nanogap structure and gold for electrode. Actually the parameters for the photo resist etching is different

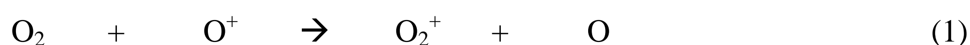
from that which used for cleaning, thus we use pure oxygen in this process so we call O_2 plasma technique that to take care the dry etching process apply only in the photo resist layer without damage the down layer (Si material). The thickness for the photo resist is about 200nm that we use and the spin parameters is change from one sample to another just to focused on the effect of the thickness on the O_2 plasma technique. Formation of the micro and nano gap is aided by O_2 plasma technology, with the use of different times to take a closer look at the impact of this technology. The Al material are used as a hard mask for the photo resist layer through the etching process before applied wet etching to remove the Al layer.

In our limited studies of the effects of photo resist (PR) parameters on O_2 plasma conditions, we have observed that the plasma system is a complex process. By varying a few parameters, we have been able to enhance organic removal rates by several orders of magnitude. We have observed in our laboratory that very small changes in photo resist (PR) parameters can cause large changes in plasma performance.

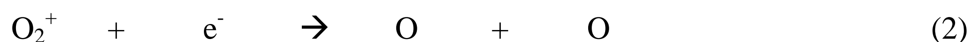
The chemistry of the plasma is what determines what can be etched. For example, anything carbonaceous, including diamond, and many other polymers such as PTFE can be readily etched with pure oxygen. Most metal oxides can be etched with argon. Passive layers such as SiO_2 or Si_3N_4 can be removed only with CF_4 or 90% CF_4/10% O_2. Silicon can be etched with SiF_4. Aluminum can be etched with BCl_3.

Plasma etching is one of several cleaning mechanisms achieved by plasma processing. Physical sputter etching simply bombards surfaces with energetic particles that knock off surface particles. Reactive ion etching (RIE) employs highly reactive gases that react with surface atoms and molecules to form volatile species. Plasma etching is similar to RIE but employs neutral radicals and can operate at relatively high pressures (0.2 to 1.2 Torr).

The oxygen plasma in the plasma etcher is established by sending an RF signal at 13.56 MHz to an antenna in the top of the chamber filled with oxygen under vacuum. A matching impedance circuit is used to minimize the power reflected from the antenna back to the RF power source. The oscillating electric field of the RF signal provides the ionizing energy necessary for reactions (1) and (2) described below. In plasma etching, neutral oxygen radicals are formed as the product of two reactions:



$$O_2 + O^+ \rightarrow O_2^+ + O \quad (1)$$



$$O_2^+ + e^- \rightarrow O + O \quad (2)$$

These radicals are relatively long-lived species and can exist outside of the plasma region.

The oxygen plasma can be used to clean surfaces by reacting with hydrocarbon residues. The resulting volatile products of the reaction such as CO, CO_2 and H_2O are swept away by the pumping action of the vacuum and the convective flow of the process gas. The same mechanism can be used to etch polymers provided there is not significant cross linking.

The oxygen plasma can also be used to bond poly (dimethylsiloxane) [-O-$Si(CH_3)_2$-], also known as PDMS, to a variety of substrates including glass, silicon and PDMS itself. It is believed that exposure to an oxygen plasma results in hydroxyl groups [-OH] in place of methyl groups [-CH_3] on the PDMS surface. Upon conformal contact, the hydroxyl groups of the two surfaces bond in a condensation reaction to form a covalent bond [-Si-O-Si-] and release a water molecule. The surfaces are then irreversibly bonded.

Table 1: Suggested Recipes.

Process	Pressure (Torr)	Power (W)	Time (s)
Cleaning	0.40	12	varies
PDMS Bonding	0.1	20	35

The mechanistic and parametric complexity of a plasma etching environment often causes confusion and delays in the development of a suitable plasma etching process. Plasma pressure may be one of the most important process parameters. In systems where the primary mechanism of plasma etching is the chemical etching component, increased pressure in the chamber results in more reactive species present. In a system in which ion bombardment is the primary action, the increase in pressure does not have such a pronounced effect although the system pressure can effect ion density and energy. For example in etching the Si in a CF_4/O_2 plasma, in a low pressure regime, ion bombardment is the dominant player and the result is anisotropic etching. At higher pressures, neutrals play an important role and chemical etching enhances etching rate resulting in a more isotropic etching [2].

Pressure of the system also impacts the dc self-bias. In a reactive ion etching system, increased pressure will reduce dc self-bias. At lower pressures, electron temperature will increase and result in an increase in dc bias. Therefore, the etching rate of ion-enhanced processes could be reduced with increasing pressure [3]. In the higher pressure systems, lower peak to peak voltages are required which result in lower average ion energies [4]. Lower pressures result in higher energy and less off-axis ion bombardment. In other words, as pressure increases, ion flux increases but ion energy decreases. As pressure decreases, ion fluxes decrease and ion energy increases [5].

Figure 1a [6] shows SEM micrographs of a SiC mesa etched at an optimum condition for 5 min, 800 W (ICP)-125 W (2480 V bias)-8 mTorr-100% NF3-9 cm, which yielded an etch rate of about 400 nm/min. Nearly vertical sidewalls (,86°) and a smooth surface (RMS roughness , 1 nm) are clearly observed. An effort to increase the etch rate by applying higher ICP coil power resulted in a re-entrant mesa with damaged mask, as shown in Fig. 1b, which typically represents an etching process dominated by the chemical action of etching reactants [7, 8]. Enhanced etching at the base of the sidewall (the “trenching” effect) and severe sputtering of the Ni mask were observed when higher bias power was applied (Fig. 1c). It has been reported that the trenching effect results from the enhanced ion bombardment at the sidewall base, due to the deflection of energetic ions impinging onto the sidewalls at glancing angle, and occurs during the dry etching at high sample bias conditions [8].

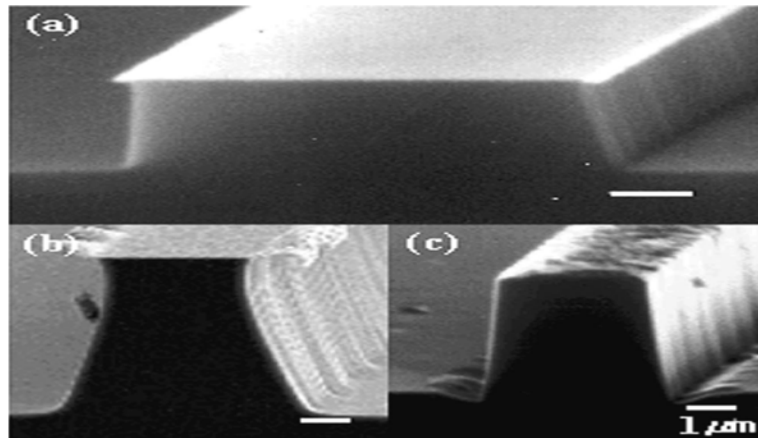


Fig. 1: SEM micrographs of SiC mesas, ICP-RIE etched at (a) 800– 125 W (2480 V)-8 mTorr-9 cm, (b) 800–150 W (2560 V)-8 mTorr-9 cm, and (c) 1000–125 W (2430 V)-8 mTorr-9 cm.[6].

2. Mask Design

In this work, a SOI wafer is used as the starting material to fabricate a nanogap biosensor. The first step is to fabricate the two designs as two masks, as had been proposed and the silicon nanogap with the gold electrode process flow is designed. This research mainly focuses on the issue related to the fabrication of the biosensor and the development of a new technology. The sidewall etching using the O_2 plasma (RIE) to form thin nanogap metal cantilevers which configure the 3-D nanogap electrode grid array structure. The anisotropy of the RIE is modeled and the etching profiles are simulated. This method is proved to be applicable by analysis and experiments [9].The starting material use in this work is the SOI wafer, 100mm in diameter (a 4 inch wafer) as shown in Figure 2.

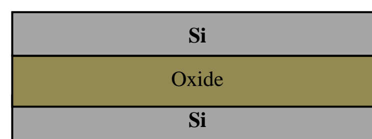


Fig. 2: SOI wafer.

The first process is to check the wafer type from its specification, measure the wafer thickness (SOI thickness), and sheet resistance then lightly scribe the backside of the wafer and protect the top surface using the scribing tool provided. Making a gentle but visible mark and then place the scribed wafer in a container, the wafer is cleaned before each of the processes.As for the lithography process, two photomasks are employed to fabricate the nanogap using conventional photolithography and O_2 plasma techniques.

A commerical chrome mask is used in this research for better photomasking process. This mask is used to develop the gold electrode with the silicon nanogap. The photomasks are designed via AutoCAD software and then printed onto a chrome glass surface. Figure 3 is the first mask for the nanogap electrode formation with the length and width of 5000μm and 2500μm respectivily.

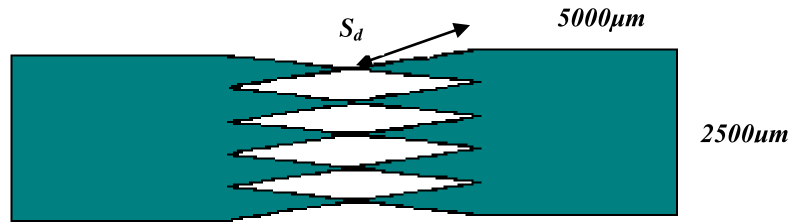


Fig. 3: Design Specification of the Mask1.

Table 2 shows the length of the end electrode. This is simply to check the best angle for the best nanogap formation after the etching process.

Table 2: Difference dimensions for S_d.

S_d	µm
1	1100
2	1000
3	900
4	800
5	700
6	600

The symbol S_d refers to the dimension for the Side angle in the design for nanogap formation. It is shown that when S_d is large the nanogap becomes very sharp and the sharpness decreases with the lesser dimension of S_d.

Figure 4 shows the actual arrangement of device design on a chrome mask. It is consists of 160 dies with 6 different designs.

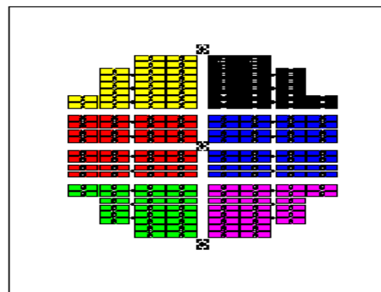


Fig. 4: Schematic design of the actual mask on chrome glass.

Figure 5 is a schematic device design of Mask 2 with 5000μm long and 2500μm wide. The distance between the two rectangles is indicated as S_a bearing the same dimension with S_d according to the theorem of Pythagoras, and the dimension of S_a can be defined mathematically
as shown in Figure 6.

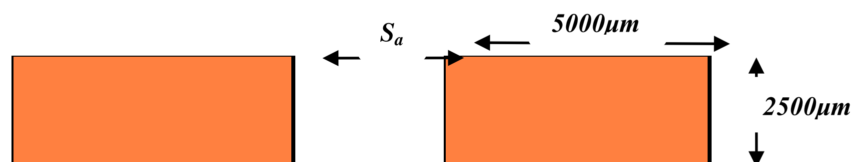


Fig 5: Design Specification for Mask 2.

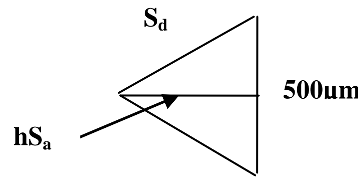


Fig 6: Schematic representation S_a, where $S_a=2hS_a$

Table 3: Variance Dimensions for S_a verses the dimension for S_d.

S_d (µm)	$hS_a=((S_d)^2-(250)^2)^{½}$ (µm)	$S_a=2\ hS_a$ (µm)
1100	1071	2139
1000	968	1936
900	864	1729
800	759	1519
700	653	1307
600	545	1090

From Table 3 the dimension for S_a depends on the dimension of S_d ,. The calculated S_a is based on $S_a=2hS_a$. Figure 7 is a schematic mask on a chrome glass.

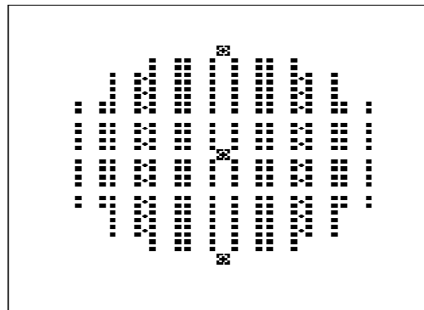


Fig. 7: Schematic Mask on chrome glass.

3. Methodology

We start the process steps silicon micro and nanogap fabrication with gold electrode, by cleaning the SOI wafer before depositing 135nm of Al as a hard mask to avoid damaging the Si layer during etching, using the RIE. In order to study the optimization of the photoresist (PR) patterning using O_2 plasma stripping in nano structures, the SOI wafer must be cut after Al deposit process to four pieces where the applied photoresist (PR) different as shown in the Table 4.

Table 4: various parameters for photoresist (PR) process.

No. Sample	Step 1 for PR	Step 2 for PR
1	Rpm=600	Rpm=4000
2	Rpm=600	Rpm=3000
3	Rpm=800	Rpm=4000
4	Rpm=800	Rpm=3000

After the photoresist (PR) coating, we use the same group of samples from Mask1 for all the samples (SOI/Al/PR wafer), and develop the resist before hard mask process to apply the wet etching for Al layer.

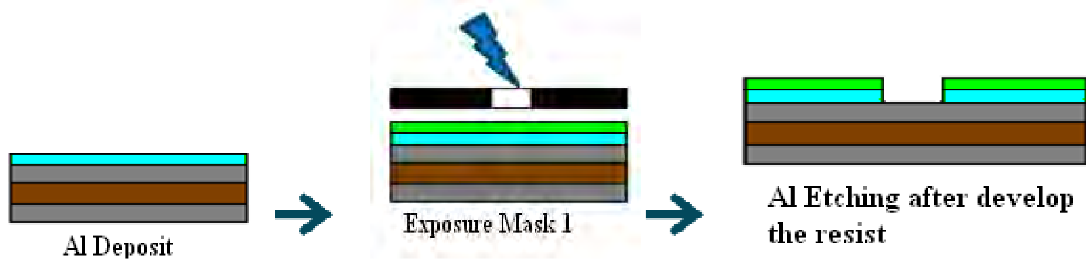


Fig. 8: The first steps for nano structure fabrication.

Formation of the nanogap is aided by O_2 plasma technology, with the use of different times to take a closer look at the impact of this technology. Therefore, after the wet- etching of the Al layer, it is best to fabricate the gap using the RIE /O2 plasma and the following parameters as in Table 5.

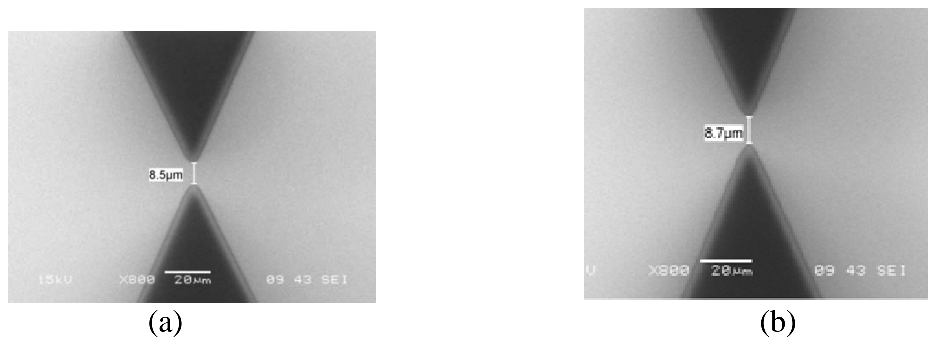
Table 5: Explain the parameters for O_2 plasma process

CF_4	CHF_3	SF_6	O_2	Ar	Bias	Power ICP Power	APC/Control (Pa)
0	0	0	20	0	30	200	1.00

4. Results and Discussions

Using the parameters of Table 5 for the RIE with a time value of 10 sec. Sample1 and sample2 have produced the following results as shown in Figure 9, where sample1 is different from sample2, as shown in Table 4.

The initial gap size for sample1 is 8.5µm whereas after O_2 plasma etching the final gap size is 8.7µm and the removal of the resist via PR strip increases the size of the gap by 0.2µm as shown in the Figure 9.



(a) (b)

Fig. 9: SEM photo show the image gap as 15 kv for sample 1: (a) Before O_2 plasma the size of the gap is equal to 8.5µm, (b) After O_2 plasma the size of the gap is equal to 8.7µm.

The initial gap size for sample2 is 8.5μm whereas after O_2 plasma etching the final of the gap size is 9.4μm and the removal of the resist increases the size of the gap by 0.9μm as shown in the Figure 10.

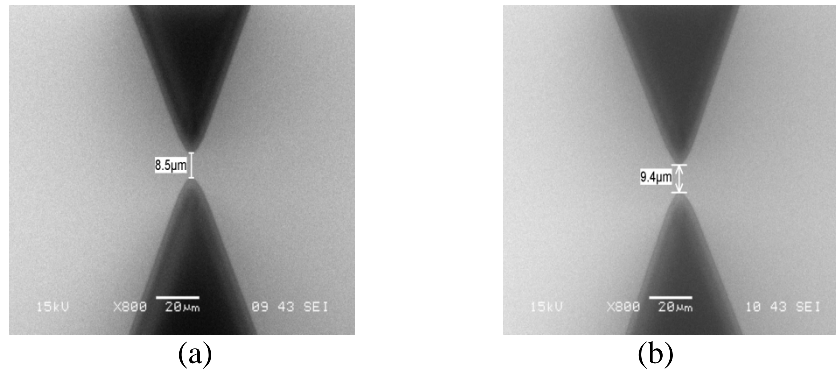


Fig. 10: SEM photo shows the image gap as 15 kv for sample2: (a) Before O_2 plasma the size of the gap is 8.5μm, (b) After O_2 plasma the size of the gap is 9.4μm.

It appeared clear is that the parameters used in the process of photoresist (PR) in Figure 9 the size of the resist removal is by farless than 0.2μm which is better than the one shown in Figure 10, where the size of the resist removal is close to 0.9μm.

In the case of reduction in the time (t=5 sec) in the process of O_2 plasma and the same parameters, we apply the process to sample 3 and sample 4 as shown in Figure 11 and Figure12, where the initial gap size for the Sample 3 is 14.5μm whereas after O_2 plasma etching the final gap size is 14.7μm and the removal of the resist increases the size of the gap by 0.2μm as shown in the Figure 11.

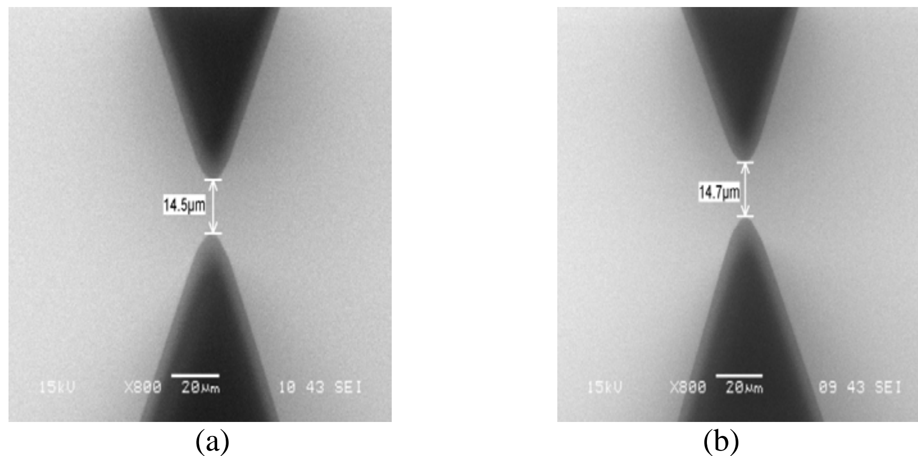
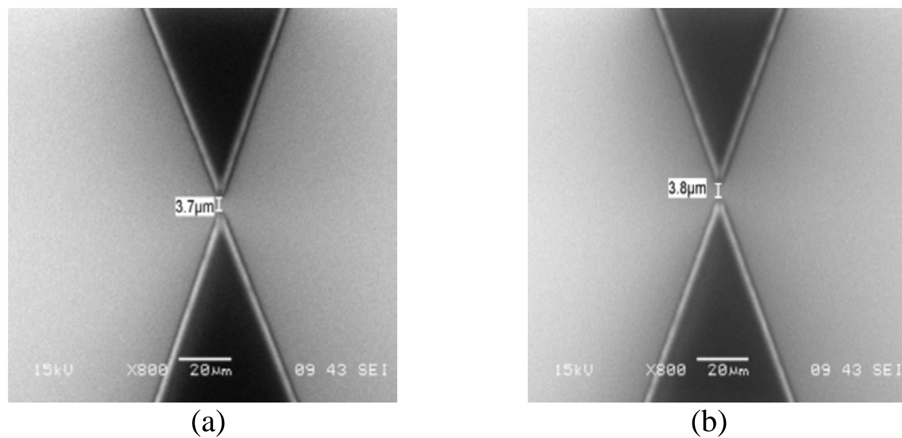


Fig. 11: SEM photo show the image gap as 15 kv for sample3: (a) Before O_2 plasma the size of the gap is 14.5μm, (b) After O_2 plasma the size of the gap is 14.7μm.

While in Figure 12 the initial size of the gap for sample 4 is 3.7μm after O_2 and plasma etching the final gap size is 3.8μm and the resist removal increases the size of the gap by 0.1μm.



(a) (b)

Fig. 12: SEM photo show the image gap as 15 kv for sample4: (a) Before O_2 plasma the size of the gap is 3.7μm, (b) After O_2 plasma the size of the gap is 3.8μm

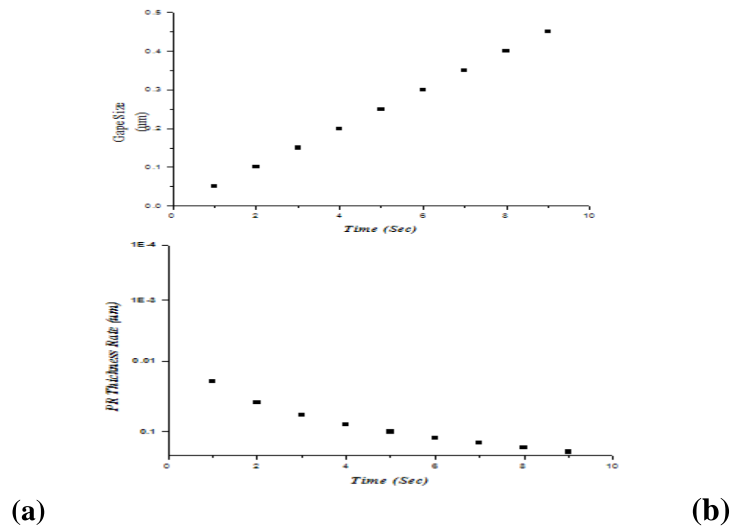
The amount of etching during the recording of the initial value of the fourth samples before using the O_2 plasma and then the final value after using the O_2 plasma, and that the time of etching for sample1 and sample2 is 10 seconds, while for sample 3 and sample 4, the etching time is 5sec. Note again that Table 6 shows that the lowest value recorded by the etching results are 0.1μm to a sample 4 at the time of etching is 5 sec, but the highest rate of etching was recorded for the sample 2 when the time of etching is 10 sec, it is thus clear that the increase in etching time when we apply the O_2 plasma leads to an increase in the amount of etching, while in the case of reducing the time of etching means reducing the amount of etching, as shown in Table 6.

Table 6 : Explain the size gap befor and after apply O_2 plasma process.

Sample No.	**The size gap before O_2 plasma (μm)**	**The size gap after O_2 plasma (μm)**	**Time for O_2 Plasma process**
1	8.5	8.7	10 sec
2	8.5	9.4	
3	14.5	14.7	5 sec
4	3.7	3.8	

The first object is to provide the oxygen Reactive Ion Etch (RIE) plasma which contains a sufficient quantity of reducing material to effectively yet safely remove the oxidized organic residues such as oxidized photo resist residues from the semiconductor substrate surfaces.

In our limited experimental studies for the effects of photo resist (PR) parameters on O_2 plasma conditions, we have observed that the plasma system is a complex process. By varying a few parameters, we have been able to enhance organic removal rates by several orders of magnitude. We have observed in our laboratory that very small changes in photo resist (PR) parameters can cause large changes in plasma performance.



(a) **(b)**

Fig 14: Chart shows the impact of increased the time in the process of O_2 plasma on both of: The gap size, (b) the photo resist thickness rate.

Theoretically, To understand the impact of some parameter such as a time for the O_2 plasma technique that is clear in Figure 14, where an increase in the time are established parameters in Table 4 and Table 5 lead to an increase in the rate of drilling are moving to introduce the pattern to the addition time inside the RIE equipment and thus lead to an increase in the size of the gap as shown in Figure 14(a) [1]. Either the impact of the time on the thickness of the photo resist layer in the same process of O_2 plasma leads to an decrease in the rate of the stripping amount and a decrease in the layer thickness, as shown in the Figure 14(b) [10].

Discussed in this research are the different parameters used in the process of photo resist coating (PR) and the effect of O_2 plasma process which proved that the final task is very important in the nanoeletronic fabrication process. The photo resist (PR) here helps as preliminary work on identifying the gap size of the addition to the chrome mask and the specific distinction of the parameters where the results are useful for the O_2 plasma to determine more accurately the breadth of the gap which is less than 0.2µm, and thus presuming the area and prospects for future studies developing the nanotechnology using O_2 plasma technique.

The use of technical O_2 plasma is very useful here in this study and to make the practical nanogap in the field of nano structure fabrication, where after the final use, we need to remove the resist layer to be after the wet etching process to the Al layer, including the use of dry etching by RIE to the silicon layer and then removing the Al layer to be the thin-film deposition of the oxide layer prior to the deposition of Ti/Au, in order to increase connectivity and to facilitate the detection of a plant's future. After that, the photo resist layer is applied to expose Mask2. Then we use the wet etching for Ti/Au layer, after removing the resist, it is the final nano structure fabrication in this study as shown in the Figure 15.

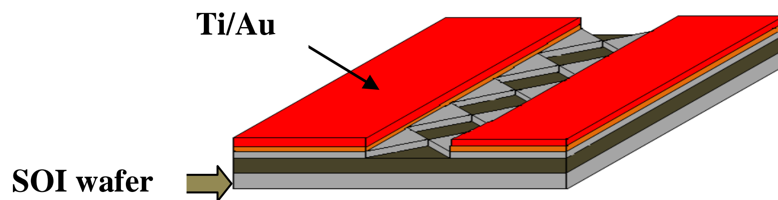


Fig. 15: Gold/Ti –Silicon/Oxide-Silicon device fabrication.

5. Conclusion

These are only a few applications in which an enhanced plasma system may be ideal. Much is known about the optimization of the plasma process, but much more remains to be understood. With a continuing effort to understand the mechanism involve in plasma etching, the usefulness of these systems will expand. Plasma is a viable alternative to other large waste producing techniques; however the slow process time has been the limiting factor in its usefulness. By understanding the mechanism involved in the process of questioning and designing the plasma system to best suit the need, plasma cleaning, etching and machining will become the first choice industrial process.

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