A MODIFIED INDUCTIVELY COUPLED PLASMA FOR HIGH-SPEED, ULTRA-SMOOTH REACTIVE PHASE ETCHING OF SILICA GLASS

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ABSTRACT

We report on the etching of borosilicate glass substrates in a conventional and modified inductively coupled plasma – reactive ion etch (ICP-RIE) tool. We present the etch rates and surface roughness of borosilicate glass in various fluorine based plasmas using C_4F_8, SF_6, Ar, NF_3, and H_2O gases. In the conventional ICP-RIE etching mode an etch rate of 0.55 μm/min at a rms surface roughness of 25 nm was obtained at C_4F_8, SF_6 flow rates of 5 sccm, O_2 flow rate of 50 sccm, 2000 W of ICP power, 475 W of substrate power. A maximum etch rate of 0.67μm/min was obtained at a high rms surface roughness of 450 nm by increasing flow rate of C_4F_8 to 50 sccm. Using the modified ICP-RIE system consisting of a gas diffuser ring clamped to the substrate holder, the physical component of the etching was considerably reduced and we have been able to achieve etch rates ~0.72 μm/min with surface smoothness of ~1 nm for borosilicate glass and fused silica respectively after 5 minutes etches.

KEYWORDS

Modified ICP-RIE, fused silica, borosilicate glass, plasma etching, inductively coupled plasma, glass etching mechanism

INTRODUCTION

Most of the early efforts in silicon dioxide (glass) etching were directed towards realizing features for microelectronics application such as waveguides [1], phase shift masks [2], etc. Hence, these plasma processes for patterning glass focused on controlling the selectivity between silicon oxide and silicon [3], reducing the gate oxide damage [4], and decreasing sidewall roughness [5]. Konuma et al reported the study on reactive ion etching of SiO_2 in NF_3 + H_2O for cleaning native oxide from silicon surfaces and examined the chemical impact of etchant on Si/SiO_2 selectivity [6]. With the growth of microelectromechanical system (MEMS) and microsystems in the last two decades, the direction of glass etching has shifted to bulk glass microfabrication. Microfluidic lab-on-chip devices [7], micro-optical devices [8], and chip-scale packaging of MEMS require high aspect ratio, high etch rate and low surface roughness etching characteristics.

Inductively Coupled Plasma – Reactive Ion Etching (ICP-RIE) process is widely used in etching because it allows independent control of the plasma density and energy of etchant ions. It provides stable high density plasma in a relatively low pressure environment. Glass etching with SF_6 and Ar/Xe gases in ICP-RIE system have achieved etch rates ranging from 0.5 μm/min to 1 μm/min [9] – [12]. These etches were developed premised upon the use of Ar/Xe ions to increase ion bombardment (physical component) and fluorine based gases are used to provide the reactive ions (chemical component) to increase the overall etch rate [9][12]. Inert gases are believed to help reduce the re-deposition of micro-mask particles and more effectively removes any non-volatile residues. In general it results in smoother etched surfaces with an average surface roughness ~2 nm [11]. In 2006, Yamakawa et al. have reported an etch rate of 14 μm/min on a 4 μm thick boro-phospho-silicate glass using a microwave-excited non-equilibrium atmospheric pressure plasma source with NF_3, He, and H_2O gases [13]. It is an exciting accomplishment in glass etching field, but their work does not report how the process varies with other types of glasses such as fused silica or borosilicate, especially in the context of deep etching. Table 1 summarizes the reported glass etching results in terms of etch rate and roughness since 1999.

Table 1: Summary of the reported glass etching results from literature.

Reference	Etch rate (μm/min)	Roughness (nm)	Glass type
Abe et al [15], 1999	0.5	2	Quartz
Li et al [16], 2000	0.6		Borosilicate
	0.5		Fused silica
Ichiki et al [10], 2003	1.2	~ 1000	Borosilicate
Park et al [12], 2005	0.75		Borosilicate
Akashi et al [17], 2006	0.55		Borosilicate
Lu et al [18], 2006	0.65		Borosilicate
Goyal et al [11], 2006	0.54	2	Borosilicate
Kolari [19], 2007	0.6		Borosilicate
Queste et al [20], 2010	1	30	Borosilicate
Zhang et al [14], 2014	1	0.5	Fused silica

Recently, our group reported high-speed, ultra-smooth etching of fused silica substrate with SF_6, NF_3 and H_2O gases in a modified ICP-RIE system [14]. Using the new configuration, we were able to achieve an unprecedented surface roughness of ~5 Å at an etch rate of ~1 μm/min in fused silica substrates. In conventional ICP-RIE mode, etching is believed to be accomplished by high energy ions inducing a damaged silica layer by physical bombardment on which fluorine mediated surface reactions accomplish silica removal. On the other hand, the modified diffuser ring etching system showed that with NF_3 and H_2O gases, the etch rate was higher and the etched fused silica surface became smoother than the results of conventional etches under identical pressure conditions. Analyzing the RGA data with corresponding etch rates and roughness, showed that a) fast etch rates are dominated by high partial pressure of atomic fluorine and SF_x ions; b) HF seems to play a less important role in glass etch rate than atomic fluorine and SF_x ions, but processes with large peaks of HF could be associated with smoothly etched surfaces. It is believed that HF helps undercut the micro-grassing features and results in smooth etch surfaces.

In this paper, we report the etching performances of borosilicate glass in fluorine plasma using the modified etcher and illustrate how the etching conditions influence the physicochemical reaction environment.

EXPERIMENT SETUP

Sample preparation

500 µm thick, double-side polished 4 inch borosilicate (80.6% SiO_2, 12.6% B_2O_3, 4.2% Na_2O, 2.2% Al_2O_3, 0.1% CaO) wafers were cleaned in Nanostrip® for 30 minutes and then deposited with 15 nm chromium and 150nm gold seed metal layers using e-beam evaporator. Wafer were patterned using Shipley™ 1827 photoresist and developed in 25% Microposit™ 351 developer to form 3.5 µm thick photoresist pattern. 2-3 µm nickel was electroplated as hard mask with feature widths ranging from 5 µm – 200 µm. SPR 220-7 was coated to form 10 µm photoresist for thicker electroplated nickel and deep etches. Thereafter, the photoresist and the underlying Cr/Au seed layers are removed using photoresist stripper and Cr/Au wet etchants respectively in order to obtain clean glass regions to be etched.

Chamber modification

Alcatel AMS 100 ICP-RIE etch tool was modified in this work. Instead of conventionally feeding gases through the ICP source, a stainless steel gas diffuser ring was attached to the metal plate of the mechanical clamping plate of the etcher and is used to locally introduce NF_3 and H_2O vapor gases in the vicinity of the wafer as shown in Figure 1. H_2O vapor gas was generated in a 15 cm high stainless steel container, placed on a hot plate at 50 °C and MKS® mass flow controller was used to introduce the metered amount of water. An *in-situ* residual gas analyzer (RGA) is connected to the reactor chamber in order to analyze the plasma and reaction product species, stylus profilometer (P-16, Tencor®) was used to characterize the smoothness of the obtained etch topography. Ring diffused NF_3 and H_2O vapor gases are controlled by separate mass flow controllers. Available ranges of power and gas flow rates of individual gases for the modified etch system are listed in Table 2.

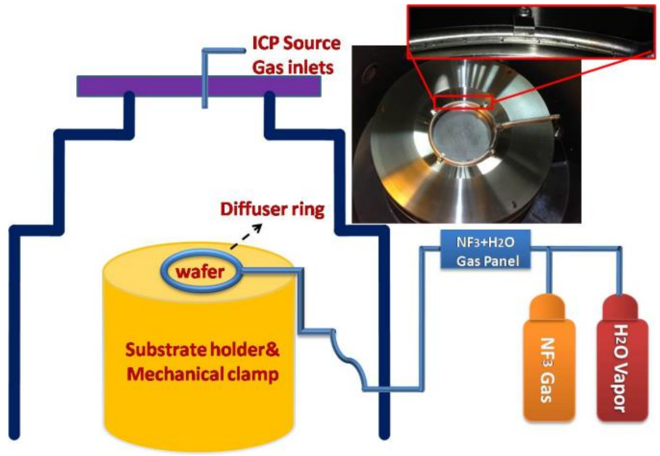

Figure 1: Schematic illustration of the modified ICP-RIE systems with ring-diffuser system. Inset shows the photos of the actual stainless steel showerhead nozzles.

Table 2. Summary of the process parameter space available on the modified ICP RIE system.

	Process parameters	Range
ICP Source Gases	SF_6	0 – 200 sccm
	Ar	
	C_4F_8	
	O_2	
Ring Diffuser Gases	NF_3	0 – 300 sccm
	H_2O	0 – 250 sccm
Physical Parameters	Source Power	0 – 3000 W
	Substrate Power	0 – 600 W
	Pressure	0 – 75 mTorr

RESULTS AND DISCUSSIONS

Conventional Etching method on Borosilicate

In our previous work, borosilicate glass etching had been investigated with SF_6 and Ar gases [11]. Here we will examine the etching characteristics by adding C_4F_8 and O_2 for the etching of borosilicate glass. From preliminary DOE [11], the optimum processing parameters for accomplishing borosilicate glass are shown in Table 3.

Table 3: Optimum process conditions for the etching process.

Process Design Parameter	Units	Optimum value
ICP Power	Watts	2000
Substrate Power	Watts	475
O_2 Flow Rate	sccm	50
SF_6 Flow Rate	sccm	5
C_4F_8 Flow Rate	sccm	5
Ar Flow Rate	sccm	50
Operating Pressure	mTorr	< 5
Temperature of Substrate	°C	20
ICP – Substrate Distance	mm	120

In $SF_6/C_4F_8/Ar/O_2$ based etching, when the flow rates of C_4F_8 or SF_6 were increased while keeping all other factors constant as in Table 3, the etch rate increased along with deterioration in surface roughness of the etched features. Figure 2(a) shows the etch rate and surface roughness obtained as a function of C_4F_8 flow rate. The etch rate increases by 30% from 0.55 μm/min to 0.675 μm/min when the flow rate of C_4F_8 is increased 10 times from 5 sccm to 50 sccm. However, with such an increase there is a 20 times deterioration in the surface roughness from ~25 nm to ~450 nm. Similar trends are observed for increase in flow rate of SF_6 wherein 30% improvement in etch rate and 40 times deterioration in surface roughness is obtained with 10 times increase in flow rate (Figure 2(b)). Hence increase in flow rate of these two gases does not help much in terms of increase in etch rate but causes undesirably large increase in surface roughness. This is because the fluorine ions and radicals as provided by 5 sccm flow rate of the two gases are sufficient for providing the required chemistry. Any flow rate higher than that causes formation of carbon polymer compounds on the surface and reduces the effectiveness of sputtering by charged ions thereby causing an increase in the surface roughness.

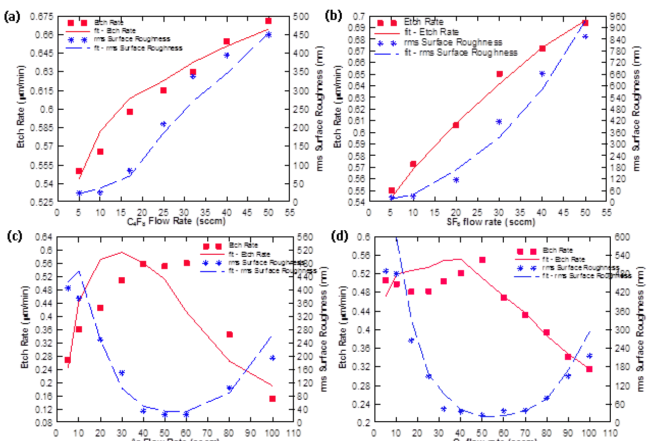

Figure 2: Variation of surface roughness (blue) and etch rate (red) as a function of variation in flow rates of (a) C_4F_8, (b) SF_6 (c) Ar and (d) O_2 gases during etching for $SF_6/C_4F_8/Ar/O_2$ based chemistry. All other parameters are kept at their optimum value as in Table 3.

When we increase the flow rate of Ar for $SF_6/C_4F_8/Ar/O_2$ based chemistry, there is an increased uniform stream of Ar ions incident on the surface of the wafer. This causes not only increase in etch rate but also improvement in the surface roughness due to rapid and efficient removal of non-volatile reaction products and stray particles from the surface of the wafer (Figure 2(c)). However, when the flow rate of Ar is increased to 70 sccm, the etch rate drops and the surface roughness increases. This effect can also be attributed to the increased operating pressure inside the etching chamber due to enhanced flow rates of Ar gas. It is known that there is significant reduction in the etch rate and increase in the surface roughness with an increase in the operating pressure inside the etching chamber [11]. Additionally, with an increase in operating pressure the randomizing collisions experienced by the Ar and other charged species increases causing them to loose energy thereby making the sputtering process at the surface of the sample less efficient.

Similar trends are obtained when flow rate of oxygen is varied from 5 sccm to 100 sccm (Figure 2(d)). However, the etch rate does not increase when flow rate of oxygen is increased from 5 sccm to approximately 50 sccm. This is because oxygen does not participate in the actual etching process but provides oxygen ions and radicals to scavenge carbon polymers formed on the surface of the wafer. Figure 2(d) shows the surface roughness reduces significantly from 480 nm to ~ 30 nm when the flow rate of oxygen is increased from 5 sccm to 30 sccm. From 30 sccm to 70 sccm the improvement in surface roughness is not significant and it is almost constant. This is because at very low oxygen flow rates, there is insufficient removal of polymers formed on the surface thereby resulting in formation of micro-asperities on the sample surface due to micromasking resulting in high surface roughness. With increase in oxygen flow rate the removal becomes efficient thereby yielding smooth surface finish. With further increase in the flow rate of oxygen above 70 sccm, the effect of high operating pressure kicks in thereby causing reduction in the etch rate and also increase in the surface roughness.

Borosilicate Glass Etching Characteristics with Ring Diffuser System

Here, SF_6 is still introduced from the ICP source. Meanwhile, NF_3 and H_2O vapor gases flow from the additional inlet through the diffuser ring into the chamber. Diffuser ring is supposed to locally feed NF_3 and H_2O vapor gases in the vicinity of the wafer. Once source power and substrate power are turned on, SF_6 gas is ionized by the source power into SF_x^+ ions. The substrate power drives these positively charged ions towards the NF_3 and H_2O vapor gas cloud in the vicinity of the wafer and dissociates the highly unstable NF_3 gas into NF_x radicals.

A maximum etch rate of 0.72 μm/min was achieved for SF_6:NF_3:H_2O :: 20:20:25 combination by using diffuser ring system. This etch rate is higher by 15% as compared to the conventional only-SF_6 etching under identical pressure and powers. A surface roughness of 1.1 nm was obtained which is an improvement of two orders. A comparison of the RGA data of conventional SF_6 etching and the modified diffuser ring etching configurations are plotted in Figure 3. It is clear that for the same pressure, increased atomic fluorine, HF, NF_X peaks are observed in the diffuser ring system. These active species are believed to tune the etching mechanism from physical bombardment dominated mechanism to chemically induced process. The observation of a large SiF_3, a product species of glass etching, is also detected in the RGA data of diffuser ring system. 8% larger ion flux is also observed in the case of etching borosilicate glass in comparison to fused silica, clearly indicating the need for enhanced ion bombardment to remove the non-volatile dopant atoms such as Al, Na, and Ca etc.

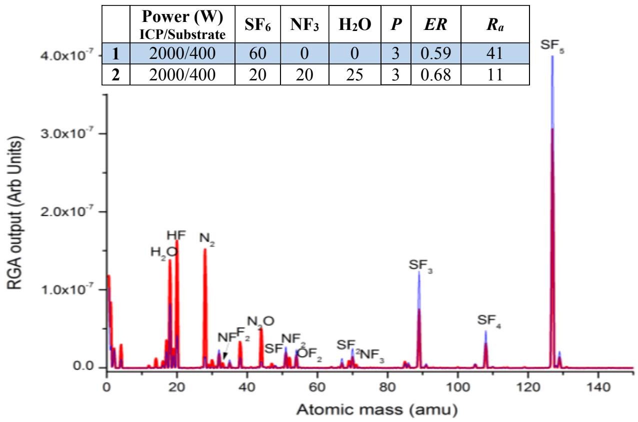

Figure 3: RGA data comparison between processes #1(Blue) and #2(Red). Increased F, HF, NF_X and SiF_3 peaks are observed in the diffuser ring system.

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