



The behaviour of continuous flow xenon difluoride etching of silicon

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Abstract

The characterisation of silicon etching by xenon difluoride is discussed along with the presentation of process behaviours of etch parameters. Selectivity towards key semiconductor and MEMS materials are also discussed. We discuss a continuous flow process whereby xenon difluoride flows throughout the etch process. Unlike pulse processing regimes, which show slower overall etch rates and can be physically limited in process capabilities, the continuous process shows high etch rates on blanket and test designs, which highlight undercut etching rates. Undercut etch rates of up to 18 microns per minute are reported.

1 Introduction

Vapour phase etching of silicon by xenon difluoride has been discussed by a number of authors: from its initial generation and discussion of its manufacture [Weeks *et al.* 1962], the benefits of etching silicon in a dry environment have been described. The avoidance of the stiction effects observed in wet etching processes such as KOH and the observed high selectivity towards many common CMOS and micro and nano electromechanical structures (MEMS/NEMS). Selectivity towards these key materials allow complex devices to be realised. These devices typically use either single crystal silicon or polysilicon as the sacrificial layer, which have many subsequent layers deposited on their surface. Some, like photoresists, can be applied as a simple mask, which does not react or interfere with the reaction of silicon [Chu *et al.* 1997]. Others, such as silicon nitride and silicon dioxide have been reported with low measurable etching rates, which provide high selectivity values sufficient for design constraints that may be imposed at early stages [Weeks *et al.* 1962; Chu *et al.* 1997; Williams *et al.* 2003; Winters *et al.* 1979; Ibbotson *et al.* 1984].

The move to systems that incorporated hardware configurations using an expansion chamber in which systems generated a high pressure of xenon difluoride (approximately 3 Torr) became prevalent as research groups aimed to increase etching rates using the XeF₂ etch process [Williams *et al.* 2003]. This etch process is carried out in cycles or pulses, hence being described as pulse-processed. The number of cycles is increased to meet the etch profile requirements of the user. This can in some cases, require etching processes with very high etch times.

While there is much information about pulse processing [Williams *et al.* 2003; Winters *et al.* 1979; Ibbotson *et al.* 1984], information relating to continuous flow processes and their behaviours have not been widely discussed in literature [O'Hara *et al.* 2005; Zhu *et al.* 2007]. In this paper, the process behaviours of basic blanket film etching of polysilicon as well as the behaviours of undercut etching conditions of both polysilicon and single crystal silicon are described. Additionally, the behaviour of common structural materials is discussed including selected metals under etching conditions, the relative etch behaviours of which are described in this paper.

2 Experimental set-up

Etching of all samples took place in a memsstar Alpha silicon etch system. This system uses a continuous flow configuration for the xenon difluoride where an N_2 carrier gas flows through a solid source bubbler bringing the xenon difluoride vapour into the chamber as described in Figure 1. All etches were conducted between a pressure of 1 Torr and 9 Torr at ambient temperature. The N_2 carrier gas flows were varied between 25 and 175 standard cubic cm per minute (sccm). The improvement in process control in the continuous flow configuration is due, as the name suggests to the continuous flow of N_2 gas through the XeF_2 source material. This allows for consistent control of the XeF_2 concentration within the etch chamber. This control is less precise under pulse processing conditions.

Blanket film etch rate analysis samples consisted of $\langle 100 \rangle$ 150 mm silicon wafers and 1-micron thick polysilicon layers deposited by LPCVD deposition in a Tempress tube furnace at 550 mTorr in temperatures of 650°C on 150 mm wafers. These polysilicon films were deposited onto thermally oxidised $\langle 100 \rangle$ silicon wafers with a thickness of 100 nm. This was for ease of measurement of the polysilicon.

Undercut etch samples consisted of silicon wafers with $\langle 100 \rangle$ orientation and LPCVD polysilicon films deposited on thermally grown oxide films. With the addition of a masking layer deposited using standard photolithography methods. The undercut structure was patterned in a basic mask using SPR350 photoresist deposited to a thickness of 1.5 micrometres and exposed with a Karl Suss MA6/8 system for 15 seconds in the soft contact mode. The mask

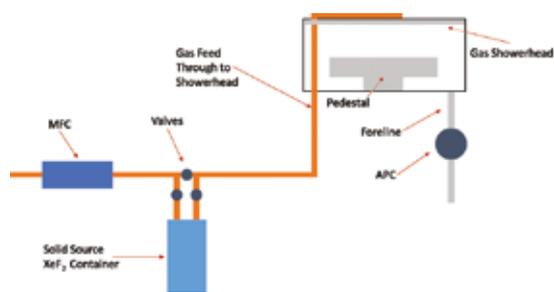


Figure 1. Memstar Alpha silicon etch system hardware diagram.

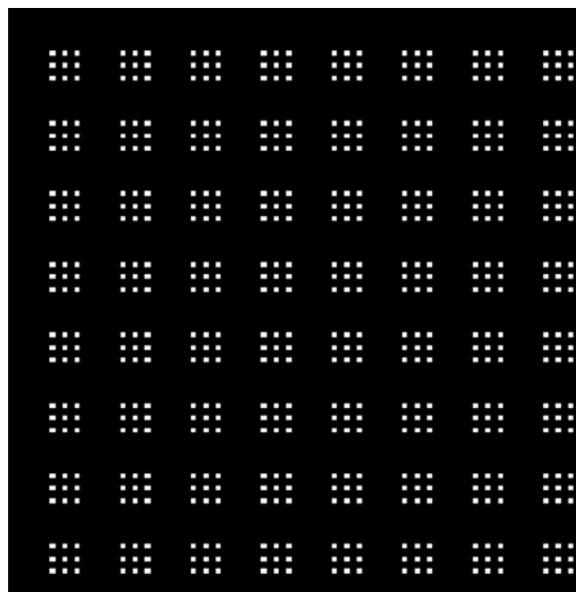


Figure 2. Section of mask used for undercut structures.

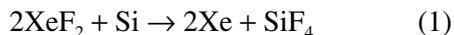
design was a nine square array design in a 3 by 3 arrangement. Each square was 100×100 micrometres with 200 micrometres between each square and 1 mm spacing between each array as shown in Figure 2. Undercut measurements were taken using a Zeiss Axiotron optical microscope system, which is able to measure the undercut distance of the silicon. Film thickness measurements are confirmed with the use of a Nanospec 3000 optical microscope. Selection of common metals, Al, Al-Si (1%), Cr and Cu, were deposited to a thickness of 300 nm. An Oxford Plasmalab sputter system deposited these selected metal samples onto $\langle 100 \rangle$ 100 mm silicon wafers. The deposition conditions are given in Table 1. A Dektak surface profilometer is used to measure the thickness of both deposited metal layers and photoresist films.

Table 1. Metal deposition conditions for selected metals in the Oxford Plasmalab system.

Material	Power (watts)	Pressure (mTorr)	Deposition time (minutes)
Aluminium	1000	3	60
Aluminium-Silicon (1%)	1000	3	60
Cr	500	3	94
Cu	1000	3	30

3 Results & discussion

The reaction of XeF_2 and Si is described in equation (1):



This reaction follows a standard reaction route of surface chemisorption/physisorption of XeF_2 on the surface of the silicon sacrificial surface as defined by early investigations into XeF_2 [Chu *et al.* 1997]. The xenon and fluorine dissociate from each other within the silicon and the reactants, Xe and SiF_4 , are formed and desorb from the silicon surface. The mask used for these tests is thin enough to be optically clear for measuring undercut and uniformity of the etch process. This work will show how uniformity is affected by varying the process conditions.

Figure 3 shows the initial flows for the Alpha etch system. N_2 carrier gas flows from 25 sccm to 175 sccm. The flow of XeF_2 is controlled by a nitrogen carrier gas through a solid source bubbler and increases with higher carrier gas flow. The relationship between XeF_2 and N_2 flow is not linear, resulting in lower concentrations of XeF_2 vapour at higher N_2 carrier gas flows as shown in Figure 4. The concentration of XeF_2 in this figure is calculated using the flow calibration function of the etch system. It calculates the amount of N_2 and XeF_2 under the same process conditions. From this, a relative concentration of XeF_2 to N_2 can be determined.

The reaction of xenon difluoride with silicon requires a physisorbed layer, which subsequently dissociates and forms the process reactants. The speed of the etch reaction is determined by the level of interaction of the XeF_2 with silicon surfaces. For undercut and blanket etch rate

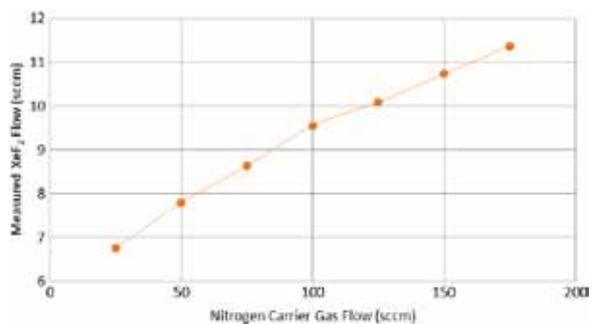


Figure 3. Effect on XeF_2 with increasing N_2 carrier gas flow.

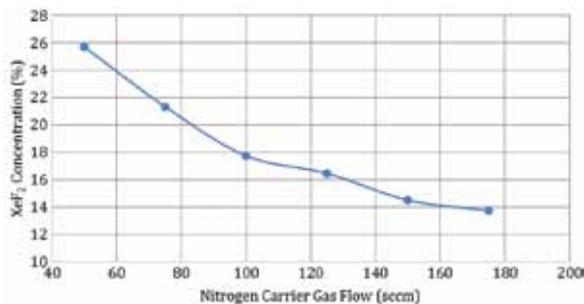


Figure 4. Concentration change of XeF_2 with relation to increasing carrier gas flow.

processes, differing behaviours are shown in the following figures. Observations of the etching depth of blanket polysilicon films in Figures 5 and 6, which show increased etch depth with increasing pressures and increasing N_2 carrier gas flows. The primary change in etching depth is related to the increase in carrier gas flow, which introduces more XeF_2 into the reaction chamber. Uniformity is calculated using a 9-point measurement across the wafer with results within 1-sigma of variation. The standard deviation and mean of the 9 data points are calculated, and uniformity is calculated by equation (2):

$$\text{Uniformity (\%)} = \left(\frac{\text{Standard Deviation}}{\text{Mean}} \right) \times 100 \quad (2)$$

Uniformity is improved with increasing pressure from 4% to 2.5% can be attributed to the XeF_2 being more uniformly distributed across the process chamber. With increasing gas flow, there is a slight fluctuation from 3.85% to 4.1%. This variation is not considered significant.

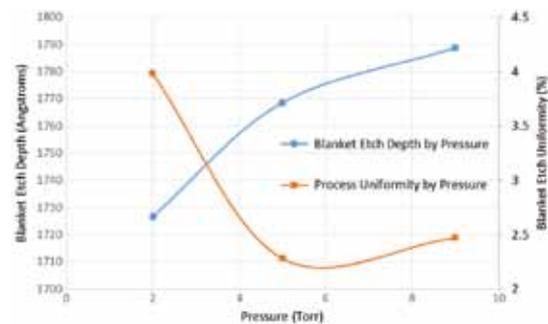


Figure 5. Blanket polysilicon etch depths and uniformity by pressure at 50 sccm carrier gas.

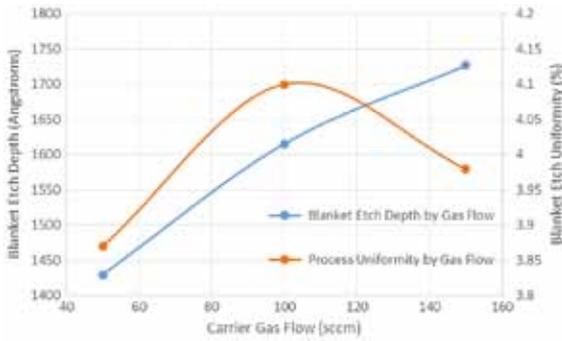


Figure 6. Blanket polysilicon etch depths and uniformity with varying gas flows at 2 Torr.

Undercut etching of polysilicon films, described in Figures 7 and 8, and single crystal silicon, Figures 9 and 10, are shown. The results now define etch rates, rather than etch depths, since the undercut etch rates are greater than the etch depths observed on a blanket polysilicon film. Polysilicon films have been observed with undercut etching rates greater than 18 $\mu\text{m}/\text{min}$ with high pressure etching conditions. The increase in pressure increases the residence time of the reactant gasses allowing for increased interaction between the XeF_2 and silicon surface, where higher etch rates are observed when utilising lower N_2 carrier gas flows. The increased undercut etch rate with lower N_2 carrier gas flows for polysilicon are explained in conjunction with Figure 4, where XeF_2 concentrations are observed to increase as the N_2 carrier gas flow is decreased. Uniformity results are substantially improved to the region of 0.5% across a 150 mm wafer with reduced pressure and N_2 carrier gas flows. In this low-pressure configuration, the gas distribution and hence, the XeF_2 concentration, across the wafer is more evenly established, improving the uniformity of

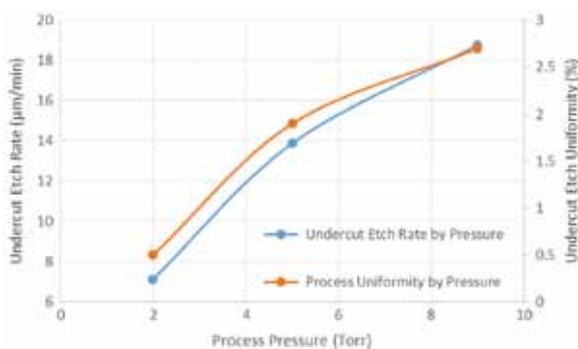


Figure 7. Polysilicon undercut etch rate and uniformity with varying process pressure and 50 sccm carrier gas.

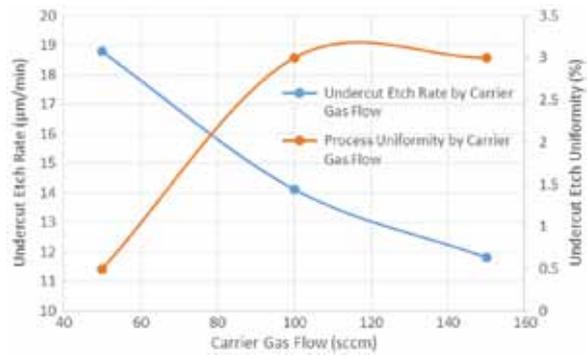


Figure 8. Polysilicon undercut etch rate and uniformity with varying carrier gas flows at 9 Torr.

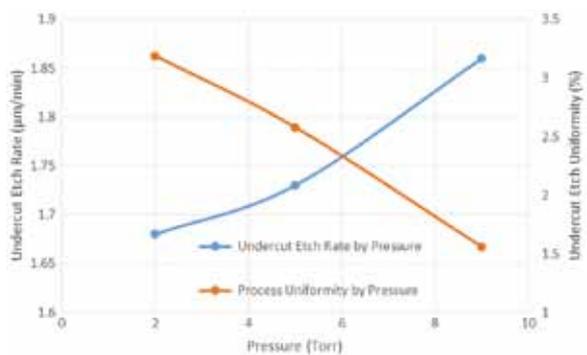


Figure 9. Single crystal Silicon Undercut Etch and Uniformity Rates with varying pressure and 50 sccm carrier.

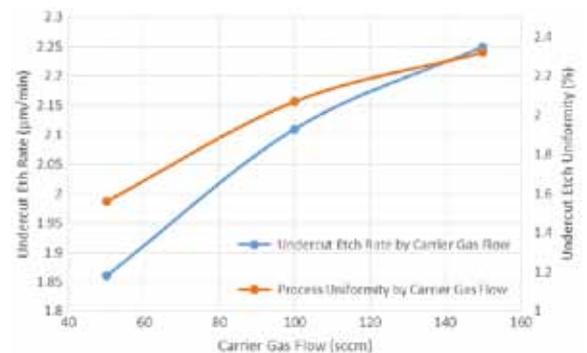


Figure 10. Single crystal silicon undercut etch rate and uniformity with varying XeF_2 carrier gas flow at 9 Torr.

the etch profile. The role of the carrier gas flow suggests that a greater level of XeF_2 interaction with the silicon surface subsequently breaks the silicon bonds in a more efficient manner. Increasing pressure further allows the relative partial pressure of XeF_2 to increase in tandem with the effects of the N_2 carrier gas flow. This increase in pressure allows the partial pressure of the XeF_2

vapour to increase in line with the overall chamber pressures.

In single crystal silicon undercut processing, a difference in etch rate is observed in contrast to polysilicon films. The etch rate increases with increasing pressure, however in contrast to polysilicon films, increasing carrier gas flow leads to an increase in the etch rate of single crystal silicon undercut etching rates.

This difference in behaviour may be accounted for the difference in the geometry of the etch profile as shown in Figure 11. In both examples, the etching front increases as the time increases. However, the single crystal etch will continue not only in a lateral direction as observed in polysilicon, but downwards into the silicon substrate. The added XeF_2 introduced, while diluted by the greater increase in relative N_2 concentration is still able to enhance the undercut etch in a way not found while etching polysilicon films.

The main observation within this work has shown that maintaining a high partial pressure of XeF_2 in the process chamber, by either N_2 carrier gas flow control or over process pressure control, will result in an increased silicon etch rate for polysilicon. Supplying high levels of XeF_2 , with an increased N_2 carrier gas flow will result in higher undercut etching rates in single crystal silicon. When etching single crystal films, the silicon is consumed by XeF_2 immediately. It is believed therefore, that the partial pressure, and hence concentration of XeF_2 when etching single crystal silicon films is continually being depleted in a way not observed during the polysilicon undercut processes. With increasing

Table 2. Observed etch behaviours of selected metals after 30 minute etch in XeF_2 .

Material	Step change (nm)	Etch rate (nm/min)
Aluminium	No change	N/A
Aluminium-Silicon (1%)	No change	N/A
Chromium	No change	N/A
Copper	10	0.3

pressure, the etch uniformity appears to decrease due to an enhancement in a loading effect during the undercut etching of polysilicon. This can be improved by lowering the process pressure and slowing the etch rate across the wafer, thus regaining a high etch uniformity. Slowing the etch process by decreasing carrier gas flows also reduces the non-uniformity. As described, with $\langle 100 \rangle$ silicon, the silicon is consuming the XeF_2 as quickly as possible and restricting this ensures a more even etch. Thus with single crystal films, the uniformity is improved when utilising increased pressure, leading to a more uniform etch profile and even utilisation of the XeF_2 molecule across the 150 mm wafer.

Etching of functional metal layers was conducted in a 9 Torr process with 50 sccm of N_2 carrier gas in a single continuous etch step of 30 minutes. As has been shown in previous results for polysilicon etching, this provides a fast etching rate when etching both polysilicon and single crystal silicon. Table 2 describes the etching results for these deposited metals. These results appear to agree with similar work reported in literature with a pulse-processing configuration [Williams *et al.* 2003].

4 Summary

Etching behaviours of both single crystal silicon and polysilicon, films have been described in both blanket and undercut etch structure designs. We have shown the effect of process parameters such as material carrier gas flows and process pressure in affecting etch rates and uniformity. No change was observed with Al, Al-Si (1%), Cr and Cu films. Undercut etching rates of $18 \mu\text{m}/\text{min}$ have been observed on 150 mm on polysilicon films with uniformities below 5%, while etching rates of $2.25 \mu\text{m}/\text{min}$ are reported for single crystal silicon.

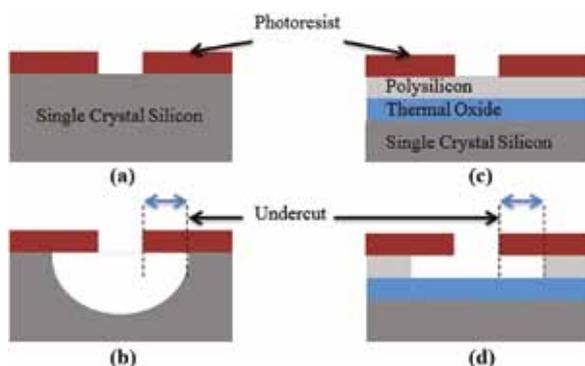


Figure 11. Profile examples of single crystal silicon before (a) and after (b) etching, polysilicon before (c) and after (d) etching.

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Tony O'Hara received a BSc degree in Physics from Heriot-Watt University and a PhD from Heriot-Watt University in 1985 and 1990 respectively. He worked at Edinburgh University researching optical MEMS devices for 5 years before joining Applied Materials as a process engineer for 6 years. He is currently a Senior Technologist at memsstar developing processes involving vapour HF and XeF₂ etching and SAM coatings for MEMS and Semiconductor products worldwide.



Changhai Wang received a BSc degree in semiconductor physics and devices from Jilin University, China, in 1985, and an MSc Degree in optoelectronic and laser devices and a Ph.D. degree in low-power all-optical switching devices from Heriot-Watt University, Edinburgh, UK, in 1988 and 1991, respectively. He is currently a lecturer in electrical and electronic engineering in the School of Engineering and Physical Sciences, Heriot-Watt University. His current research interests include fabrication and assembly of microstructures, MEMS devices and sensors, MEMS and 3D packaging, and laser-assisted methods for MEMS and electronics manufacturing.