THE EFFECT OF TEMPERATURE ON THE ETCH RATE AND ROUGHNESS OF SURFACES ETCHED WITH XEF₂

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Abstract

In this work we present results from a pulsed etching system with XeF₂ for an expanded temperature range while at the same time determining the roughness of the substrate left behind. The experimental apparatus used for the work presented in this paper is capable of temperature ranges from approximately 100 to 800 K. Data was taken at a constant etching pressure of 1.2 Torr so the effect of temperature on roughness and etch rate could be studied. Etch rates and surface roughnesses were characterized using a vertical scanning and phase shifting interferometer, respectively.

Introduction

One of the greatest challenges in the fabrication of MEMS is preventing damage to devices during the etching process. Plasma etching causes accelerated degradation of masking materials and can cause heat damage. Liquid etchants have higher selectivities but may induce stiction failure in released devices [1-4]. An attractive alternative are gas phase etchants.

XeF₂ is a gas phase etchant that spontaneously etches silicon at room temperature and offers a solution to these problems [5]. It is a white, crystalline substance that has a sublimation pressure of 3.8 Torr [6]. Sublimated XeF₂ experiences a thermodynamically favorable reaction with silicon [7] that causes free radical F to be absorbed into the top molecular layers of the Si. Multiple chemical events such as this lead to a volatile species being released from the surface. The overall reaction is given below:

\[ 2\text{XeF}_2 + \text{Si} \rightarrow 2\text{Xe} + \text{SiF}_4 \]  

(1)

XeF₂ offers very high selectivity during etching. Etch rates for metals, photoresists and SiO₂ are typically more than 100 times slower than that of Si [5, 8-11]. Because XeF₂ etches at room temperature with such high selectivity it is an ideal etchant when damage to features is a concern. As an isotropic etchant, XeF₂ is also well suited for undercutting and releasing features. As a line-of-sight etchant, it induces undercut profiles and induces roughness. Some researchers have noted that roughness occurs because etching begins at etch sites which expand out to meet each other [12]. Faster etch rates and longer etch times make these non-uniformities become larger, and the surface becomes rougher [13]. Optimization of the XeF₂ etching process requires achieving an intelligent balance between cost, etch rate, and surface roughness.

The following details our preliminary efforts in understanding the effect of pulse durations and elevated temperatures in XeF₂ etching. Pulse duration is studied for durations between 15 seconds and 30 minutes. Roughness is studied between 300 and 800 K at a constant pulse pressure of 1.2 Torr.

Experimental Setup

Experimental apparatus

Many XeF₂ etching apparatuses consist of two main vacuum chambers, an etching chamber and an expansion chamber. In this work a similar set of chambers is utilized. Both chambers are equipped with Baratron pressure sensors to monitor pressure while etching. Additionally, a third small source chamber is connected to the expansion chamber to supply XeF₂ into the system. The chambers are isolated from each other and the vacuum pump by pneumatic valves. A layout of
the etching system is shown in Figure 1. There is a raised stage in the center of the etching chamber that both holds samples in the center of the chamber and heats and cools them. The stage can be heated and cooled to allow etching at a wide temperature range (~100 K – 800 K).

![Figure 1: Basic layout of XeF₂ etching apparatus](image)

**Etching procedure**

A pulsed etching procedure is utilized in this work. Etching is accomplished by opening the expansion chamber to the source chamber and allowing XeF₂ to sublimate. After a target pressure is reached in the expansion chamber the valve between the source and expansion chamber is closed. The etching chamber is then opened to the expansion chamber and a pulse of XeF₂ is released into the etching chamber. The pressure is allowed to come to equilibrium between the two chambers. At this point the XeF₂ has come into contact with the silicon substrate and etching has begun. The Si is exposed to the XeF₂ for some duration of time which will be referred to hereon as the pulse duration.

**Sample Preparation**

Silicon wafers with a layer of thermal oxide were used in this work. The SiO₂ layer was patterned using photoresist. SiO₂ in the areas without photoresist were removed by HF leaving a nearly atomically flat Si surface. The remaining photoresist was removed to leave samples of Si masked only with SiO₂. The exposed areas of Si were 80 mm² [14]. This prevents any etch rate limitations due to diffusion in small geometries and leaves a less curved surface on which to measure etch depth and surface roughness.

**Results and Discussion**

Etch rates are determined by rate limiting steps in a chemical process. In the case of pulsed XeF₂ etching there are 2 different etch rate limiting regions, a reaction rate limited region and a diffusion limited region. At the beginning of a pulse the silicon surface is saturated with XeF₂, and etching proceeds as quickly as the reaction between XeF₂ and Si can occur – reaction limited regime. After some period of time a boundary layer of gaseous SiF₄ builds up over the Si surface, see Equation 1, preventing impinging XeF₂ from encountering the Si surface. When this occurs, the diffusion of XeF₂ through the SiF₄ boundary layer is the limiting factor and as such this regime is termed the diffusion limited regime. The 2 distinct reaction regions can be seen in the Figure 2.

Figure 2 displays etch rate data as a function of pulse duration. Each sample is placed in the reactor for a total of 10 pulses at a given pulse duration. Then etch depth is determined via a vertical scanning interferometer. This etch depth is divided by pulse duration and an etch rate is attained.

![Figure 2: Reaction vs. diffusion limited regions at room temperature and 0.2 Torr.](image)

The results in Figure 2 imply that, for a given pressure, there is exists an optimum pulse time. What is optimal is dependent on the user’s concerns. Short pulses may waste large amounts of XeF₂, but have relatively higher etch rates. Conversely, longer pulses may use up all the XeF₂, but etching may take excessive amounts of time.

The final surface roughness and etch rates achieved when etching with XeF₂ is directly related to the temperature of the silicon substrate. Adding heat increases the energy of the silicon and changes its reaction probability with XeF₂. In an article by Ibbotson et al. [15], the authors surmised that at temperatures below approximately 450 K physisorption was responsible for etching of Si, and above this temperature XeF₂ immediately dissociates on the surface. These assertions were based on etch rate data for a continuous flow setup, not for a pulsed etching system. However, this data is valuable in understanding the fundamental etching characteristics of XeF₂ on Si.

For lower temperatures where physisorption and surface migration can happen, molecules of XeF₂ and/or F⁻ would find kinks and edge sites on which to come to
rest and etch. This would effectively lower the surface roughness. Additionally, it would be anticipated that surfaces would be smoother and etch rates higher at elevated temperatures in this range and rougher with lower etch rates at lower temperatures in this range as it is assumed that reaction rates follow an Arrhenius type relationship.

In the case where immediate dissociation occurs, at temperatures a bit higher than for physisorption, the roughness should again increase and the etch rate may increase, but at a lower rate with temperature. Molecules of XeF₂ randomly fall on the surface and thus a roughness would be induced because where the molecule lands is roughly where the etching would occur. Molecules and radicals do not have chance to move to edges sites that would aid in decreasing roughnesses. Etch rates at higher temperatures in this range would increase because the number of impinging atoms will increase with temperature.

Figures 3 and 4 show data that support these assertions. The data presented was collected using five 50 second pulses at 1.2 Torr for each sample. Figure 3 shows how the surface roughness changes with temperature. This preliminary data demonstrates that for temperatures below 700 K the surface roughness generally decreases with increasing temperature. For temperatures above 700 K the roughness increases again. This again is attributed to the different etching mechanisms involved [15].

![Figure 3: Roughness versus temperature at 1.2 Torr of XeF₂](image)

Figure 4 demonstrates how the etch rate changes with increasing temperatures. In this case the etch rate increases linearly until 500 K and then the rate continues to increase until 600 K, but at a slower rate. For 700 K and above the etch rates are lower than any other temperatures. This again is generally consistent with previous literature [15] for temperatures above 500 K. The differences between the exact numbers in Ibbotson et al. are attributed to the differences in etching setups, continuous (Ibbotson et al. [15]) and pulsed (this work).

![Figure 4: Etch rate versus temperature at 1.2 Torr of XeF₂](image)

**Conclusions**

It can be seen that temperature has a direct effect on the etch rate and the resulting surface roughness when etching silicon with XeF₂. To achieve smoother surfaces, silicon must be heated above room temperature. However, too much heat will result in increased surface roughness. Etch rates also increase with temperature, but only to a point. This implies that an optimum temperature exists that will result in the best overall etching results for creating MEMS devices. In the work presented, it can be seen that the 500 - 600K range is close to the optimal etching temperature because this range gives fast etch rates with smooth surfaces.

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**References**


