

EFFECT OF ULTRASOUND ON THE ORIENTATION-DEPENDENT ETCHING OF SINGLE CRYSTAL SILICON

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ABSTRACT

We have assembled a reactor to perform KOH etching which, besides fine control of etching temperature, stirring, and solution concentration, allows etching with and without 25kHz ultrasound. To study the etch rate and morphology of the etched surfaces we use plain and lithographically masked <100> silicon wafers.

The roughness of the plain wafers after etching was quantified by the ratio of the diffuse and specular reflectances in the 800-240 nm range. The mask windows are parallelograms oriented at different angles and were designed to allow a 2-step etch. The first step reveals slowly etching planes and, after selective removal of predetermined regions of the mask, the second step of etching reveals fast etching planes. For the geometrical measurements of the resulting facets we use plane and cross-section image capture and quantitative image processing techniques, and stylus profilometry. We have obtained a relative improvement of the etched surface by the use of ultrasound.

INTRODUCTION

Anisotropic etching of silicon in strong bases is a crucial process in the fabrication of micromechanical structures like pits, bridges, beams, channels and mirrors. That is why there is a great interest in controlling the etch rate and morphology of the crystallographic planes.

The influence of the etching chemistry (composition, concentration, temperature), the silicon wafer characteristics (surface orientation, doping) and the mask geometry on etching rate have been comprehensively studied and reviewed [1,2]. Ultrasound has been reported to improve the morphology of high aspect ratio cavities [3] and even to give a surprisingly high enhancement of the (100) etch rate [4]. The importance of both results for silicon micromechanics led us to start this study about the influence of ultrasound on KOH etching not only of the (100) surface but of many different planes as well.

EXPERIMENTAL

Anisotropic etching was performed in a reactor (Figure 1) which consists of a pyrex vessel containing the KOH solution, and its lid to which are attached the following devices: (a) a water-cooled polyethylene condenser, (b) a teflon coated temperature sensor (PT-100) connected to a digital thermometer (Omega DP41-TC), (c) a mechanical teflon stirrer, and (d) a teflon substrate holder with teflon clamps. The container is immersed in an ultrasonic water bath (Thornton, 240 watts, 25 KHz) in which the water is circulated continuously and temperature is controlled within 0.2 °C, using a thermostatic circulating bath (Fisher Scientific).

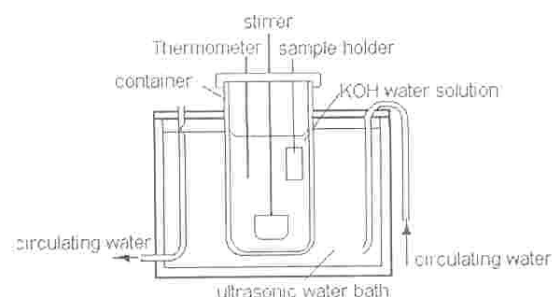


Figure 1 Reactor for KOH etching

Bare silicon samples and patterned oxidized (100) silicon samples (p-type, 1-10 Ωcm , 10 mm X 14 mm) were used. The pattern (Figure 2a) consists of 58 1mm X 2 mm cells, each containing a parallelogram shaped window within a rectangular one. After the lithography, and before KOH etching, each cell on the silicon sample contains a parallelogram region of exposed silicon, within a rectangular region covered with oxide, about 0.5 μm thick. Between the rectangles there is a frame covered with thicker (about 1 μm) oxide. The parallelograms are arranged in a series of increasing azimuthal angle ϕ , with 1 degree increment.

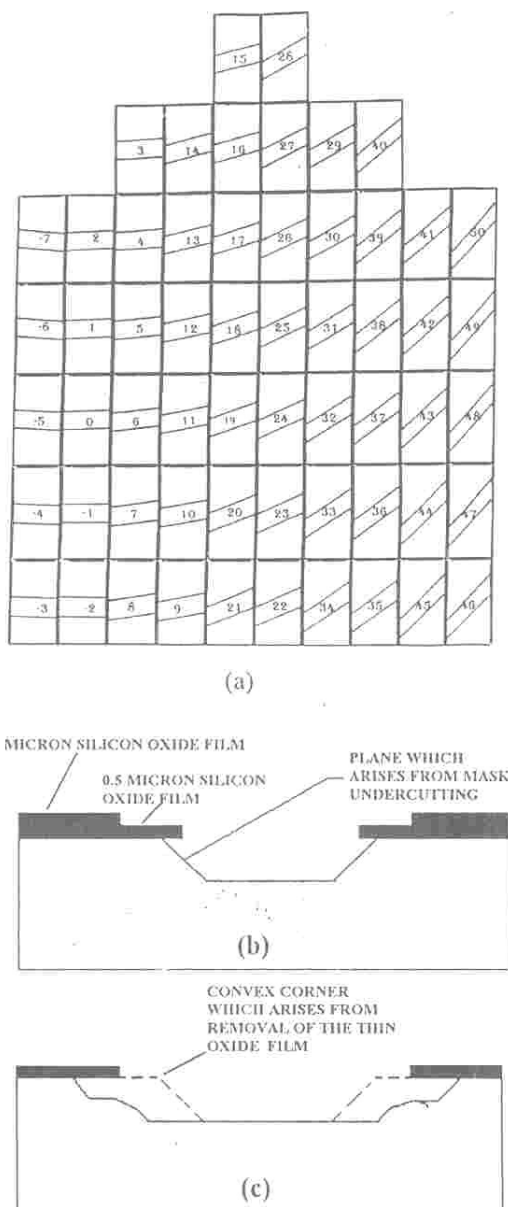


Figure 2 (a) Two-level pattern with parallelogram shaped windows, (b) cross section of a cell, (c) exposure of convex corners by removal of the thin oxide film

Etching through the parallelograms causes the formation of cavities with long walls made of the slowest etching planes among all those that intersect the (100) sample surface with azimuthal angle ϕ . We call these "slow planes" (Figure 2b). If the thin oxide is removed, the silicon convex edges in the intersection of these walls with the (100) plane are exposed. A second KOH etching step causes the appearance of the fastest etching planes among those of same azimuthal angle ϕ (Figure 2c). We call these "fast planes". The present report is limited to the first step of the experiments, and therefore to the effect of ultrasound on the etch rate of the "slow planes". For a (100) silicon wafer, as used here, the parallelogram whose sides are

precisely parallel to the [110] direction gives rise to a cavity with the long walls made of (111) planes. For these, the undercut is minimum, and there is no terracing of the walls. This cavity was identified after the KOH etching, by visual inspection with an optical microscope, using the criteria both of no terracing and of minimal undercut. The thin oxide film which appears as a thin stripe at the border of the smaller sides of the parallelogram provides a reference to determine the cavity with minimal undercut. This allowed the assignment of the azimuthal angles ϕ of each of the "slow" planes. Measurement of the zenithal angle θ between these planes and the crystal surface completely determines this orientation of these crystal planes. The corresponding etch rate is calculated from θ , undercut length and etching time.

Before KOH etching the silicon sample was clamped to the holder and etched for 15 seconds in a 5% solution of HF in water for removal of any native silicon oxide, and then thoroughly rinsed in 17 MOhm-cm DI water. The masked silicon samples were etched in a 30% KOH water solution at 40 (570 min.), 60 (246 min.) and 80 (76 min.) °C with and without ultrasound assistance, but always with mechanical stirring.

Images of the windows, and of the cavities corresponding to them, were obtained with a CCD camera attached to a microscope trinocular head and captured by a video board installed in a PC computer. Distance measurements were performed on these images (Figure 3) using image processing software (Global Lab Image). These distance values were used in equations 1 and 2 to calculate the zenithal angle θ and the etch rate for the wall planes.

$$r = \frac{\sin \theta \cdot [(T - M)/2]}{t} \quad (1)$$

$$\theta = \arctg \left[\frac{P}{(T - B)/2} \right] \quad (2)$$

where: T = width of the top region of the cavity

M = width of the mask window

P = cavity depth

B = bottom width

t = etching time

θ = angle formed between the cavity wall and the silicon surface

The cavity depths were obtained both by the microscope focus adjustment and by stylus profilometry.

In a separate experiment, the bare samples, consisting of 2" wafer quarters, were etched in a 20% KOH water solution, also with and without ultrasound assistance, at 40 (17 min.), 60 (4.5 min.) and 80 (1.5 min.) °C. The roughness of their surfaces was quantified by the R_d / R_{total} ratio [5] where:

R_d = diffuse reflectance

$R_{total} = R_s + R_d$ (R_s = specular reflectance)

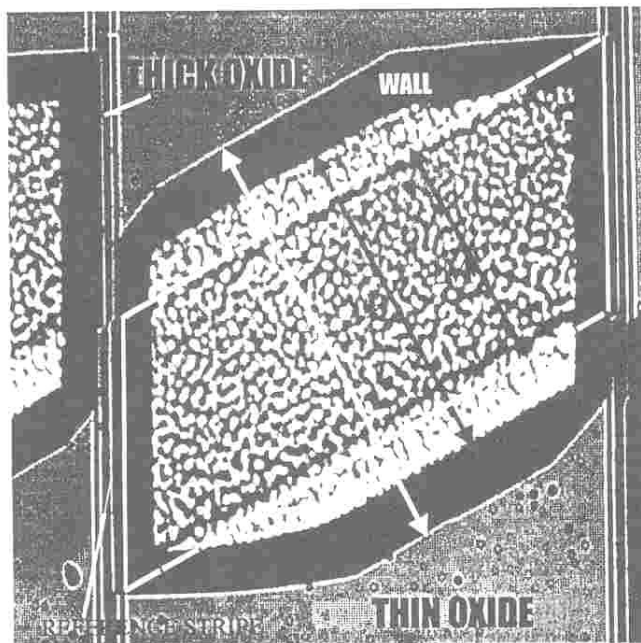


Figure 3 Distances which were measured from image process software. The window (white parallelogram) is projected on its corresponding cavity.

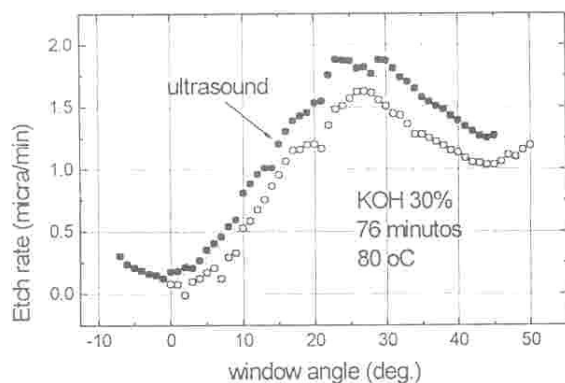


Figure 4 - Ultrasound effect on the etch rate of masked silicon

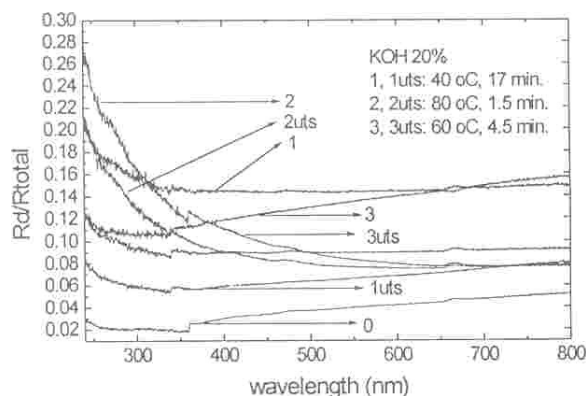


Figure 5 - Ultrasound effect (uts) on R_d / R_{total} ratio at different etching conditions.

The reflectance spectra (200-800 nm) for R_d and R_s determination were acquired by using an integrating sphere attached to a UV/VIS U3501 Hitachi spectrophotometer

DISCUSSION

The source of error in the etching rate calculations is the resolution for the edges situated at the tip of the arrows shown in figure 3. The edges areas are well defined in the no-etched samples from which the M value has been obtained. A pixel side, of approximately 2 micra, was estimated as the error for the M values. Poorer measurements were possible at the T and B arrow tips as result of the roughness in the cavity edges caused by the etching. This was estimated to be equivalent to a two pixel error in the T and B measurements. The results are plotted in Figure 4. It is important to notice that although the absolute etch rate difference obtained looks small on the scale of microns/minute, a differences in etch rate of 0.25 $\mu\text{m} / \text{min}$ during a 76 minute etch corresponds an plane advancing an extra 19 μm , well above the resolution of the length measurements. Also, the increase in etch rate due to ultrasound occurs for all planes measured.

Our results confirm the literature claims of increased etch rate with ultrasound assisted etching, and extend this result to a whole class of planes. The effect is far smaller than reported in Ref. 4 for the (100) etch rate only. Ultrasound in our case (30% KOH, 80 °C) raises the etching rate of the (100) ($\phi=45^\circ$) by 25 %. We find that the absolute increase in the etch rate is weakly dependent on ϕ , even though there is no a priori reason for it being so. This implies a higher effect of ultrasound for the slower etching planes, at least among the "slow" planes measured so far. If the ultrasound increase the etch rate through transport effects only, this points out to a bigger importance of transport effects on the anisotropy of the etch rate than it is usually assumed [1]. This will be tested with the measurement of the "fast" planes appearing in the etching of exposed silicon convex corners, which we still have not done.

The bottom of the cavities obtained at lower temperatures (40 and 60 °C, 30% KOH) have shown such poor morphology that a separation line between the sidewalls and the bottom was difficult to locate. Measurements based on cross sections of the cavities are under way and will provide more information.

The morphology has improved by using ultrasound-assisted etching on both plain and masked silicon. Plain wafer samples etched at same KOH concentration, time and temperature show smaller R_d/R_{total} ratio, corresponding to smaller rms roughness under ultrasound influence (Figure 5). We have also observed in the masked silicon wafers that the width of the windows, which becomes narrower as the window angle increases, affects the morphology. The cavity bottom becomes apparently smoother for greater ϕ angles. This is still to be confirmed quantitatively, but it could be attribute to the limited transport in narrow cavities, which has been called "crevice effect" by others [6].

Morphology improvement has been attributed by other authors [3] to the role ultrasound would play in reducing the residence time of the hydrogen bubbles on the silicon surface. According to that interpretation, the bubbles would work as pseudo masks which would avoid local etching and cause hillocks. The longer the bubbles remained on the surface the rougher the surface would be. That can be tested, in further work, by direct measurement of the effect of ultrasound on bubble residence time.

CONCLUSION

Silicon exposed by opening parallelogram shaped windows in masking thermal SiO_2 , has been etched with a 30 % weight KOH water solution at 80 °C for 76 minutes. The etch rate of a whole class of planes intersecting the (100) plane at every angle ϕ , with a resolution of 1 degree, with and without the presence of 25 kHz ultrasound has been determined. The overall shapes the $r \times \phi$ curves, with and without ultrasound, are similar, but ultrasound increases the etch rate of all the planes by roughly the same amount, implying a larger relative increase in slow-etching planes.

In other experiment, larger area bare silicon samples were etched in KOH and their roughness quantified by the ratio of diffuse to total reflection. Plain wafer samples etched at same KOH concentration, time and temperature show smaller R_d/R_{total} ratio, corresponding to smaller rms roughness under ultrasound influence.

The mechanism through which this happens will be clarified by the continuation of these experiments, and experimental results on the etching rates of the "fast" planes, and on the temperature and solution concentration dependence of the ultrasound effect.

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