

REAL-TIME, IN-SITU MICROSCOPIC OBSERVATION OF SILICON ETCHING IN KOH

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ABSTRACT

We have built a reactor that allows real-time, in-situ observation of the silicon surface during etching in liquids. We discuss the issues in etching which it can help to elucidate, and exemplify them by showing a sequence of images of hydrogen bubble formation, growth and motion on the surface of silicon being etched in a KOH solution.

We have observed tracks left on the surface by the bubbles. Subsequent measurement with a profilometer shows that these tracks are made of craters, which implies that pinned bubbles initiate craters under themselves. The steps in growth of the cavities are discussed.

INTRODUCTION

Deep orientation-dependent etching of Silicon in KOH solutions is widely used for the fabrication of micromechanical devices. Recently there has been an increase in interest in the roughness of the etched surfaces, and in control of this roughness, due to the development of micro-opto-mechanical devices, in which the surfaces are often used as optical reflectors. Roughness includes also structures that appear not as consequence of lithographic patterning, but nevertheless do have some regular features. Well-known examples of these spontaneous structures are

pyramids in (100) surface, steps in planes vicinal to (111) and corrugations on (110) surfaces. All these patterns have sizes within the resolution of optical microscopy.

Our initial motivation for building the reactor described below was to enable the real-time and in-situ observation of the genesis and evolution of these patterns. Another application is the study of effects related to the flow patterns over the solid, and the study of the gas bubbles produced in the etching and their influence on the etched surface finish.

DESCRIPTION OF THE REACTOR

A reaction cell is mounted under the objectives of a long working distance microscope (Leica Microzoom II). The cell contains the silicon sample, and the KOH solution is continuously circulated through it and a large reservoir inserted in a temperature-controlled bath. The circulation is achieved by a Teflon centrifugal pump immersed in the KOH solution. A video camera is attached to the microscope trinocular head, and the composite video signal is shown on a monitor and stored in a video cassette. Selected video sequences are captured by a video board in a PC computer for analysis and geometrical measurements, using image processing software. The apparatus is shown schematically in Fig 1.

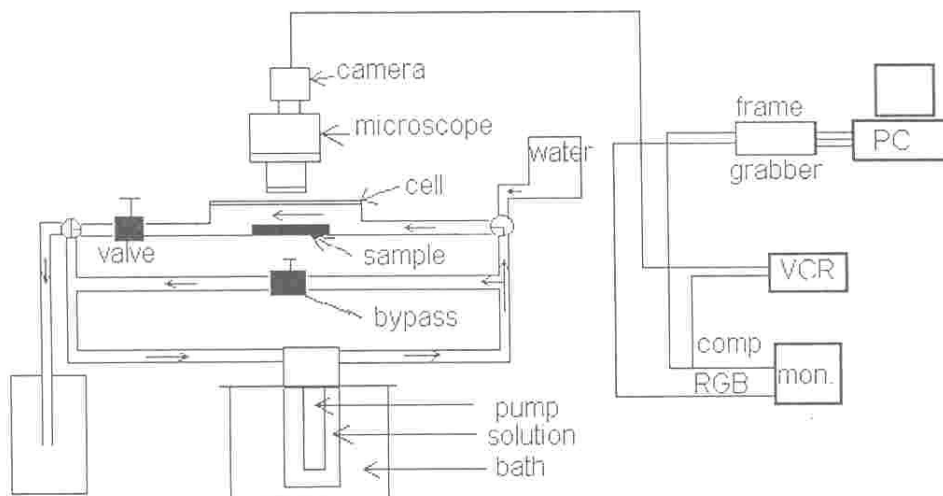


Figure 1 : schematics of the reactor

With this apparatus we can study the etching surface in-situ and in real time, under controlled temperature and flow conditions. With some modifications it can be used for membrane thickness monitoring by near-infrared transmission and *in-situ*, real-time spectroscopy of the solid-liquid interface.

CRATERS AND BUBBLES

In an interesting study, Palik *et al* [1] have argued that the primary cause of roughness of silicon surfaces etched in KOH is random masking of the surface, primarily by bubbles, and possibly also by silicates (both products of the chemical reaction).

A specific form of roughness, namely shallow round craters, is often observed on the etched surfaces. Kendall *et al* [2] have measured these spontaneously occurring craters and have figured out a way of intentionally producing and using them as approximately spherical mirrors.

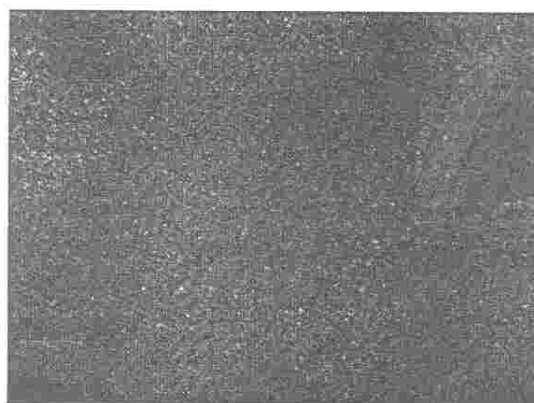
As an example of the use of the reactor, we present experimental evidence that:

1. The occurrence of craters is correlated with the previous permanence of a H_2 bubble on the same spot.
2. Crater tracks correlate with excursions of the bubble on the silicon surface.

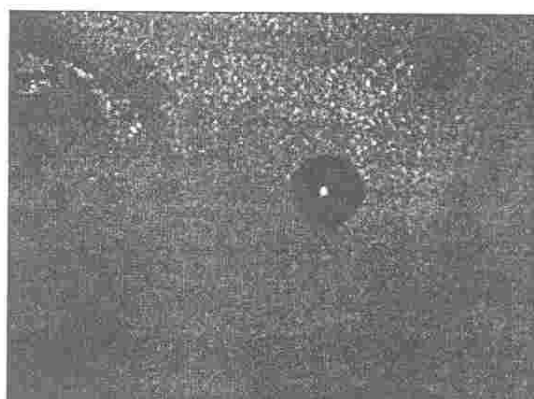
RESULTS AND DISCUSSION

We show in Figure 2 a time-sequence of images of the same spot on a Si sample etched in 10% KOH in water at 70 °C. The images were obtained with vertical, reflected illumination, and the sample was resting horizontally, with the KOH solution passing on top of it.

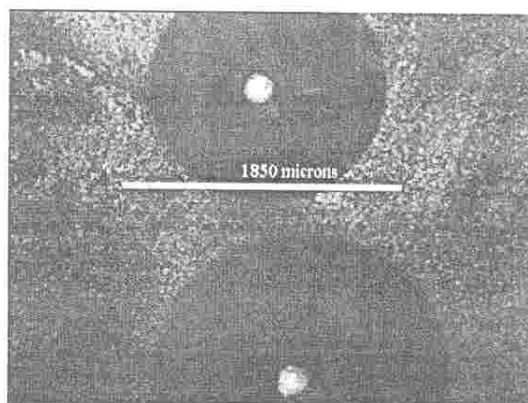
We see the following sequence: a bubble appears, growing to a diameter of 460 microns in 4 seconds. After 1 minute, the bubble has moved a little to the side, is still growing (at a much lower rate) and another large bubble has approached it from the lower side of the image area. Both bubbles continue to grow, while wandering a little on top of the silicon. The image in Fig 2(c) already shows clearly a "track" left by both bubbles. The final image shows the tracks left by both bubbles as they moved out of the field of view. Note that the conditions of the etch (low KOH concentration, 70°C, and stagnant regions of fluid flow), lead to a very rough surface finish. Under the vertical incident illumination, the tracks left behind by the wandering bubbles appear brighter than the region around them. With this illumination conditions, dark areas should correspond to low specular reflection (i.e. rough surfaces). The tracks left behind by the wandering bubbles are more reflective and therefore should be less rough. We have also observed that as etching proceeds, the tracks became progressively darker, even though this is not shown in the frames presented. It can also be noticed that the radius of the round spots that form the track is much smaller than the bubble radius, which is consistent with a spot radius approximately equal to the contact radius and a small contact angle of the bubble with the surface.



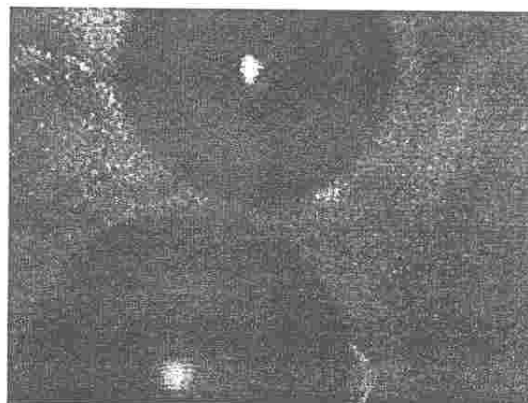
(a) - image at $t=0$



(b) - image at $t=4$ s



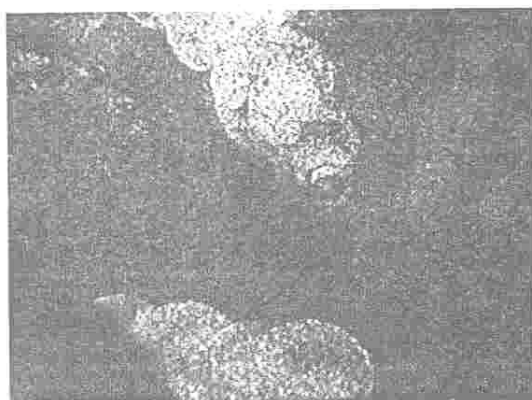
(c) - image at $t=1$ min.



(d) - image at $t=1$ min, 23 s



(e) - image at $t=2$ min.



(f) - image at $t=3$ min.

Figure 2 - Sequence of frames taken from a video cassette recording of a fixed region of the silicon surface during etching.

Careful examination of the tracks after the etch is stopped and the sample is removed gives additional information. An experimental profile of one of the round spots in a track is shown in Fig 3, verifying that it is indeed a crater.

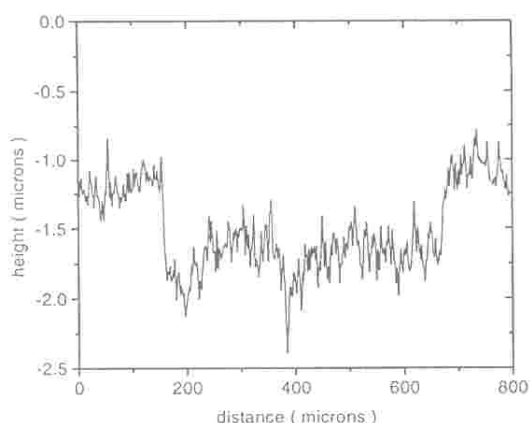


Figure 3 - Profile of a crater

An image of a track made of only two round spots is shown in Figure 4, and the profiles along the lines drawn on that image are shown in Figure 5. The spot marked "a" was left behind by the bubble as it wandered to the position

of the one marked "b", which was occupied by the bubble at the time the etch was interrupted. The former can be described as a cavity, but the latter one consists of a circular trench, or a cavity with a strongly convex bottom. Or it can be described as a concave cavity with a mesa inside. It is well known [3] that such mesas with (100) tops made of exposed silicon will tend to be etched away, due to the attack of fast etching planes at convex corners. So, taking the profiles as representing two stages in the evolution of craters, we speculate that a crater like the one in Figure 3 comes about in two steps:

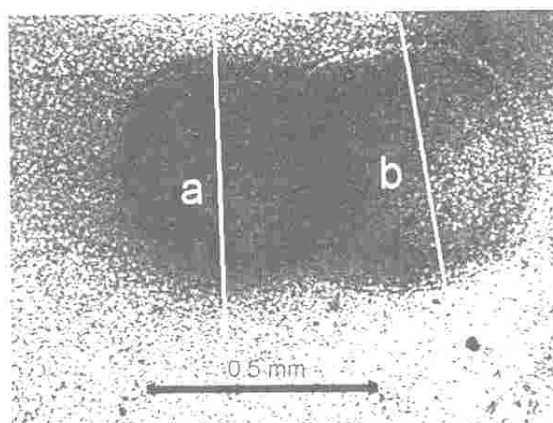


Figure 4: A 2-spot track. The white lines mark the direction of the profilometer traces shown in Figures 5a and 5b

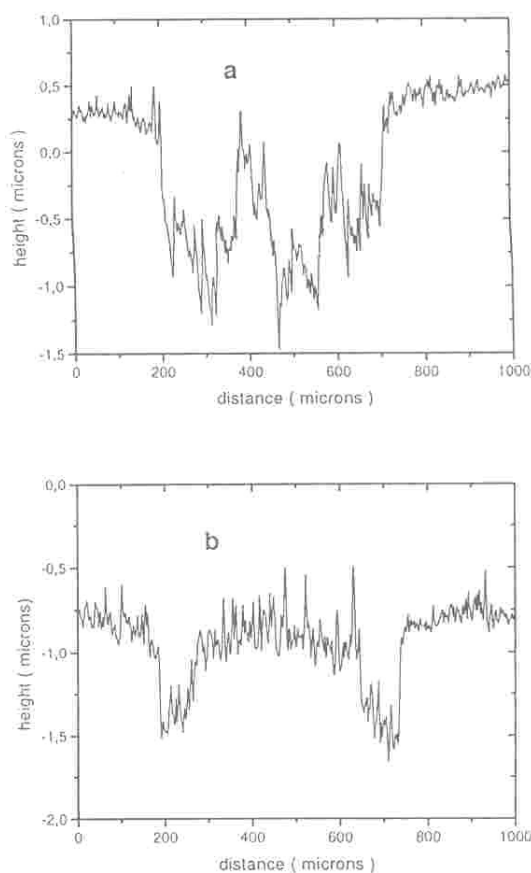


Figure 5: Profiles along the lines a and b marked in Fig 4.

(1) A ring-shaped trench, or cavity with a convex bottom is etched under an attached bubble as it grows in place. This requires the etch rate of the silicon surface to be enhanced in some way around the contact perimeter of the bubble. Determining whether this enhancement is maximal exactly at the contact perimeter requires further measurements.

(2) After the bubble moves away from that spot, the convexity decreases. Or, thinking about the initial cavity as a ring-shaped trench, the trench broadens both outwards and inwards, becoming an almost flat-bottomed cavity.

We have seen isolated ring-shaped trenches on the silicon surface after etching. According to the aforementioned mechanism, they should correspond to bubbles that grew in a fixed place and did not move around before the etch was interrupted. The trenched profile can be explained by the contact radius rate of growth decreasing as a function of time. We measured the growth rate of the bubble radius for several bubbles, and it decreases with time. For a fixed contact angle this would imply a decreasing rate of growth for the contact radius. With a time-independent surface etch rate enhancement at the contact perimeter, the depth profile could become trenched. More measurements are required to verify this mechanism step by step. In particular we have made no direct measurements comparing the contact radii to the crater radii, and need to investigate the dependence of these on the solution properties (molarity and temperature).

We refrain from speculating on the mechanism by which etching could be accelerated around the perimeter of an attached bubble, but only note that there are other semiconductor etch-enhancement effects that could be related to this. One is the trenching seen at the bottom of cavities obtained by etching through a mask, as in the case of the fabrication of membranes [4]. In this case the etching is enhanced near the walls of the cavity. The present effect has some analogy with that, with the bubble surface playing the same role of the walls. Another one is the so-called contact etching of semiconductors, discussed by Rindner and Lavine [5], who have shown how under certain circumstances, touching the etch surface with a probe (irrespective of the probe being a metal or an insulator) also causes an increase of the etch rate under the probe. We have also observed the same effect in KOH etching of silicon, and plan to study it with our apparatus. We also plan to reproduce the shallow spontaneous and induced concave cavities observed by Kendall *et al* [2] at higher molarities with real-time, in-situ observation, in order to establish whether they are correlated or not with attached bubbles.

CONCLUSION

We have built an apparatus that allows real-time, in-situ microscopic observation of the silicon-solution interface during KOH etching, and pointed out its applications. As a preliminary result, we have shown images of bubbles attached to the silicon surface during etching, and wandering on it. We observed tracks left by these bubbles during etching, which resulted in crater tracks on the etched surface of silicon and made some observations about the time evolution of these bubble-induced craters.

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