Investigating Anisotropic Etching Patterns of Silicon Using Scanning Electron Microscopy (SEM) and Light Microscopy

H. Alex Hsain, Sarah Klug, Felix Kaess

ABSTRACT

Anisotropically KOH etched Silicon (100) is etched for pressure sensing applications and is studied using optical microscopy and scanning electron microscopy. Images of 5x, 10x, 20x, 50x and 100x magnification gathered from the Olympus light microscope are analyzed using v0.70 color clustering imaging software and a density distribution of pyramids is acquired for varying magnification. These images show that greater magnification indicates a sparser distribution of pyramidal arrays. SEM images are taken at 5,000x, 6,000x, and 20,000x magnification and are processed using KLONKS software to assess average vertex angle and pyramidal base area to develop a comparative baseline for future investigation of anisotropic etching using Si (100).

I. Introduction

Anisotropic silicon etching is a key technique for the fabrication of micromechanical devices. Their first application included the etching of V-grooves on <100> silicon or U-grooves on <110> silicon in order to fabricate MOSFET transistors for high power and high current densities. Silicon structures can be fabricated in highly controllable and reproducible manners due to the strong dependence of the etch rate and dopant concentration needed for certain crystalline structures. Typical structures include thin membranes, deep and narrow grooves, and cantilevers with single or double sided suspension. Important fields of application include the fabrication of passive mechanical elements, sensors, actuators, as well as micro-optical components.

Here we investigate anisotropic wet etching on silicon for the purpose of creating a thin film pressure sensor. We aim to create an electronic sensor that emulates the properties of natural skin, where large arrays of pressure-sensitive pixels on flexible material are required. A thin film dielectric, namely polydimethylsiloxane (PDMS), is placed in between two electrodes. The rubber film stores electrical charge, like a battery. Pressure exerted on the film causes it to compress and changes the amount of electrical charge the film can store. If using a uniform thin film, the molecules of the film are compressed and become entangled. When the pressure is released, the film cannot assume its original state, and so the sensor loses its ability to read measurements accurately. However, if a micro-structure is used such as a pyramid, the thin film will behave more like an ideal spring, enabling the film to compress and rebound after experiencing pressures. Mannsfeld et al. describe fabricating a mold for PDMS using photolithography on (100) wafers followed by a potassium hydroxide etch. Here, we aim to replicate the anisotropic etching characteristics and utilize the phenomenon of reverse electrowetting as our source of electrical charge.

In traditional electrowetting, a voltage applied across a water droplet causes the water droplet to "wet" the surface and spread. The potential difference applied to the water droplet causes a buildup of charges which enables the drop to overcome its surface tension and wet the surface. Krupenkin et al. developed a novel mechanical-to-electrical energy conversion technique utilizing reverse electrowetting in which a mechanical displacement will generate electrical energy. The pressing of an electrode onto the water droplet increases the droplet's surface area and its contact with the dielectric, analogous to increasing the surface area of a conventional capacitor. This increased capacitance results in an instantaneous current. After releasing the electrode plate, surface tension drives the drop back into its original shape, similar to decreasing the capacitor surface area, allowing the excess charge to be released and flows back into the circuit in the reverse direction. Because small mechanical actuation will cause instantaneous current, reverse electrowetting can be used to detect small changes in pressure by relating the change in current to the force experienced by the electrode plate. Reverse electrowetting's sensitivity to small changes in electrical charge (in the range of nA) renders it ideal in detecting small changes in pressure for applications in thin films.

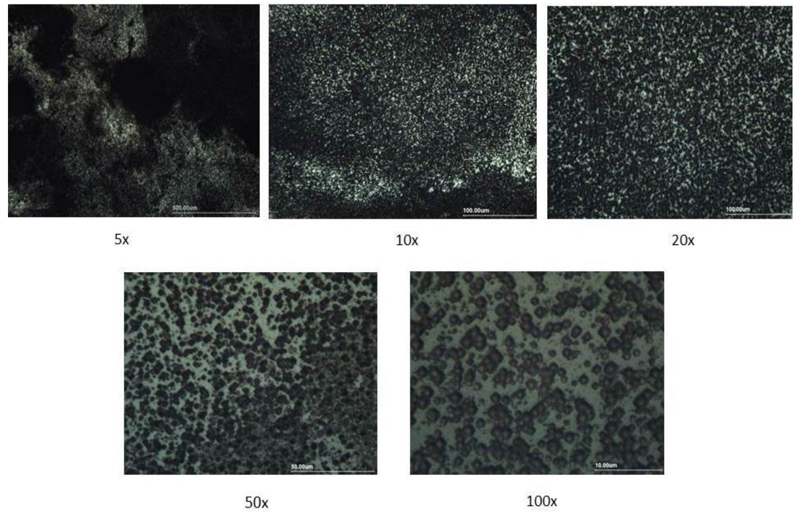
We aim to develop a thin film of PDMS that will act as a dielectric layer in our reverse electrowetting set-up. Ideally, we would like to fabricate uniformly etched pyramidal arrays to obtain accurate and precise current measurements. The etched pyramidal arrays increase the surface area of the dielectric, allowing more contact with the water droplet, which we hypothesize will lead to more sensitive readings. This measure of instantaneous current will then be related to the pressure experienced by the electrode plate. In the following discourse, we investigate the outcome of a potassium hydroxide etch on (100) Si wafers to be used as an inverse mold for our PDMS dielectric thin film.

II. Methods

A. Light Microscopy

Light microscopy utilizes light to view certain properties that cannot normally be seen with the naked eye by using the properties of lenses. Optical microscopes help to obtain a visual representation of the material's surface topography at various magnifications and enable certain optical properties to be studied such as color, brightness, and contrast.

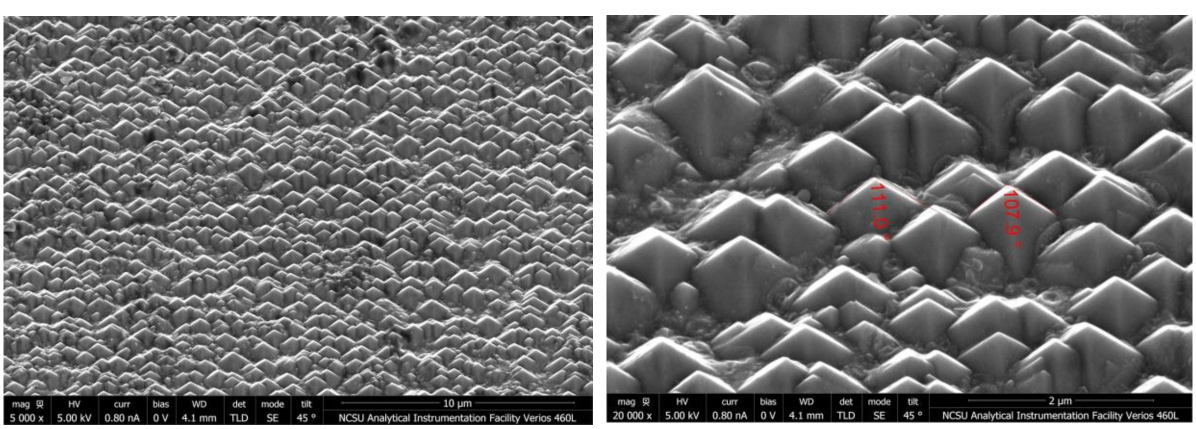
Images were taken of the pyramidal arrays etched onto silicon using the Olympus optical microscope at magnifications of 5x, 10x, 20x, 50x and 100x. With each increase in magnification, more details of the pyramids can be seen. The images also show that the pyramids were not etched in a uniform linear pattern. In the images of the lower magnifications, the color change is the only observable way to discern the differences between pyramids and empty spacing. As magnification increases, it becomes easier to differentiate which areas are covered by the etched pyramidal arrays and which locations remain vacant. Since color is an optical property, we can utilize the fact that the Olympus captures the pyramidal arrays as a darker color and the empty spacings absent of them as lighter in contrast. Images at 5x, 10x, 20x, 50x, and 100x were taken with the Olympus microscope and processed using v0.70 color clustering imaging software. Pyramidal arrays were quantified by #141416 hexadecimal, a dark charcoal, while the empty spacing was detected to be #3A3E38, comparable to slate or ash. The ratio of #141416 to #3A3E38 was plotted against magnification to discern how our observation of pyramidal density changed with magnification.



A) Olympus images at 5x, 10x, 20x, 50x, and 100x magnification

B. *Scanning Electron Microscopy*

The traditional light microscope has been used to magnify specimens and observe micro-features, however, optical microscopy is limited in our investigation because it does not closely reveal the mirco-formation of the pyramids, the angle of protrusion, or the dispersion of the arrays. In circumstances where wish to observe smaller features in greater detail, we use scanning electron microscopy (SEM). SEM utilizes a focused beam of electrons to observe micro-features. The electrons interact with the atoms in the sample and produce various signals that can be detected and analyzed to determine the sample's surface topography and composition. The beam is scanned in a raster scan pattern, and its position is combined with the detected signal to produce an image. The most common SEM mode of detection – and the one we use for our current investigation – is secondary electrons emitted by atoms excited by the electron beam. Here, we utilize SEM to study the patterning of pyramidal arrays on silicon.

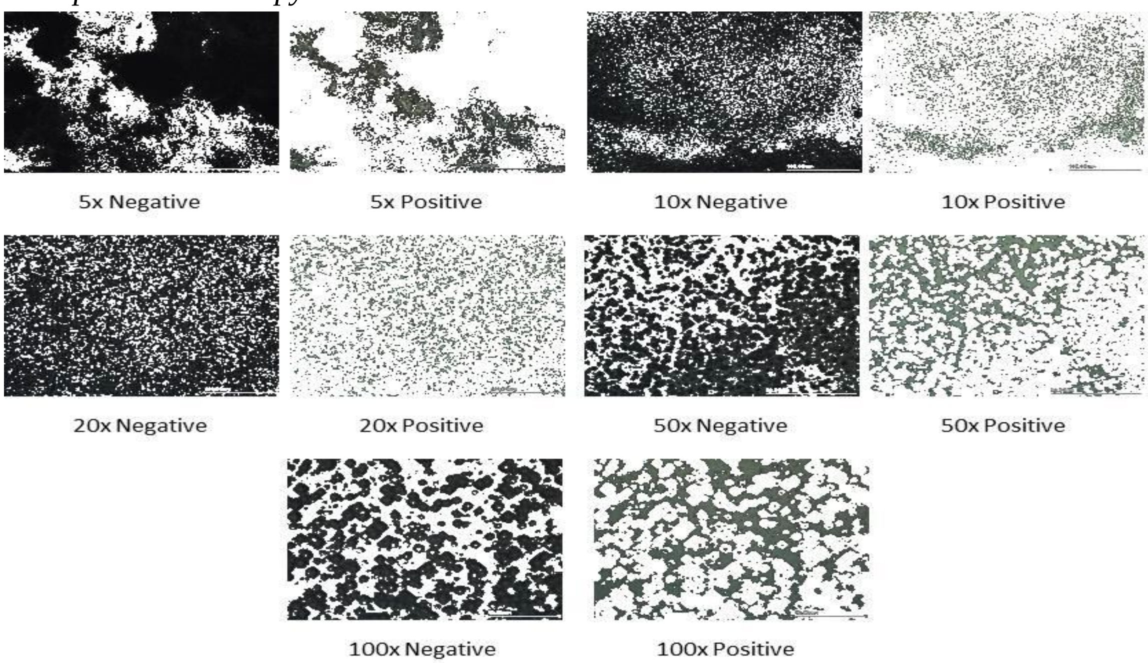


B), C) (100) Si KOH Anisotropic Etched at 45 degrees tilt angle

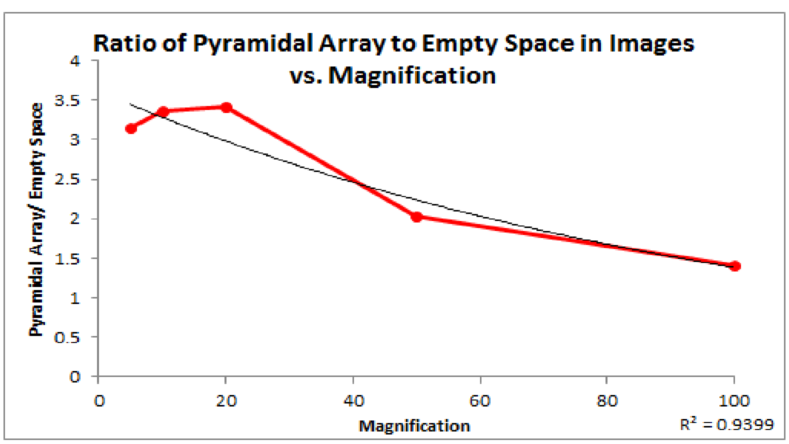
Figures B and C indicate a high density of pyramidal arrays at the chosen observation location. At 5,000x magnification, we are able to observe a varying distribution of pyramidal size and spread. We notice that the pyramids are closely packed together with no observable linear formation. Figure B and C were taken at 5,000x and 20,000x magnification and a tilt angle of 45 degrees, respectively. We observe the sharp corners of the pyramidal arrays generated by the tip of the pyramid. In these images, the pyramids also vary in size, with conglomerates of smaller pyramids clumped together.

III. Results

A. Optical Microscopy



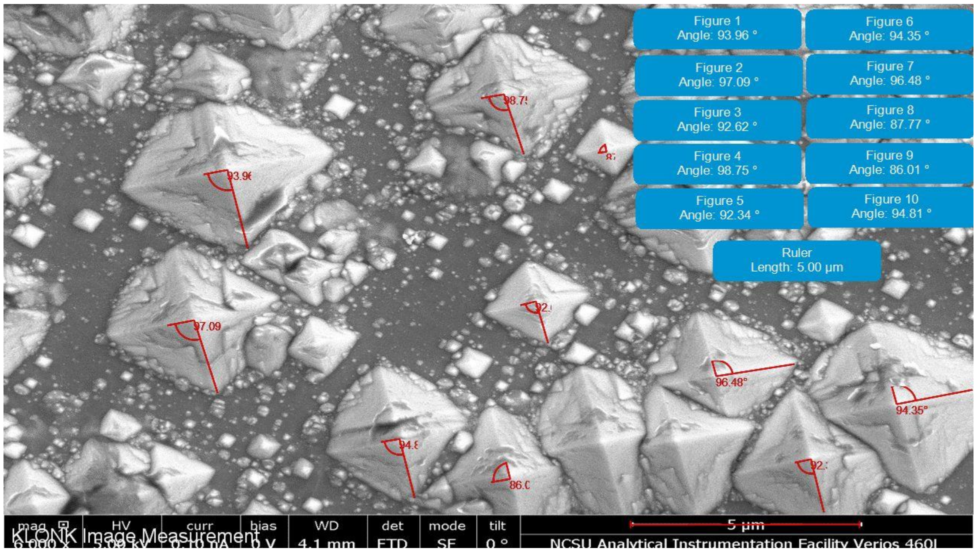
D) Positive and negative images used for calculating the ratio of pyramidal arrays to empty spacing



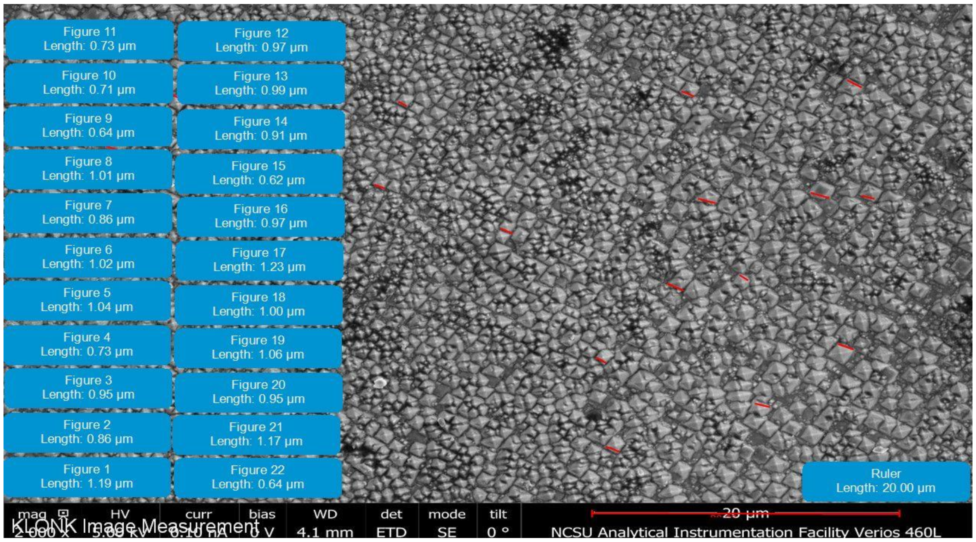
E) The change in ratio of pyramidal arrays to empty spacing as a function of magnification

The graph above displays the ratio of the areas covered by the pyramidal arrays (#141416) to the empty spaces (#3A3E38) in the images taken with the light microscope. The ratio generally decreases as the magnification increases. Images 1-5 show less density in the pyramidal arrays as the magnification increases, which accounts for the decrease in the darker colored regions. The image taken at the lowest magnification most likely did not account for all of the pyramidal arrays, whereas the image taken at 100x shows greater detail of the distribution of the pyramidal arrays. The dark spots, denoted by the hexadecimal color #141416, appear in the first image but also include many empty spaces that could not been seen at such small magnification.

B. Scanning Electron *Microscopy*



F) Ten randomized data points were located for determining mean vertex angle



G) Twenty randomized data points were located for determining mean base area

Base Surface Area (μm^2)	**Angle of Vertex**
1.84$\pm$0.16	93.4 ± 2.85

SEM images at 2,000x and 6,000x magnification were processed using KLONK Image Measurement software. Ten data points were taken from image F and it was found using a 95% confidence interval that the true mean angle that the triangular face makes with the vertex is 93.4$\pm$2.85. Similarly, 20 randomized observations in image G indicate that the average base surface area is 1.84 $\pm$0.16 μm^2 .

IV. Discussion

Light microscopy and scanning electron microscopy was utilized to study the quality of anisotropically etched Si (100) for pressure sensing applications. Both forms of microscopy revealed properties of the material that were unable to be observed with the naked eye. The optical nature of light microscopy enabled the use of color properties in determining the density distribution of the pyramidal arrays of the selected specimen. Lightly colored spots were quantified as "empty spacing" and the darker areas were quantified as pyramidal arrays. As magnification increases the density of the pyramidal arrays decreased, indicating that the distribution of pyramidal arrays was more sparse than first observed. While optical microscopy enabled color properties to be studied, optical microscopy was limited in viewing the micro-formation of the pyramidal arrays. SEM was utilized to characterize the vertex angle of the pyramids as well as the surface area of the pyramidal base. Generally, a greater surface area and vertex angle will be more favorable in pressure sensing applications because more contact area will allow for more conductive regions with the water droplet, increasing the sensitivity of the measurements. These values acquired from the SEM will aid in future optimization of the KOH etching technique. Future comparative investigations will be performed on various etching rates and KOH concentration to determine how to best optimize a dielectric film for the reverse electrowetting pressure sensing device.

References

Highly sensitive flexible pressure sensors with microstructured rubber dielectric layers Mannsfeld, S., Tee, B., Stoltenberg, R., Chen, Christopher, Barman, S., Muir, B., Sokolov, A., Reese, C., Bao, Z. (2010, September 12). Nature Materials 9, 859-864. Doi: 10.1038/nmat2834

Reverse electrowetting as a new approach to high-power energy harvesting. Krupenkin, T., Taylor, A. (2011, August 23). Nature Communications. Doi: 10.1038/ncomms1454