Microtensiometers with Patterned Porous Silicon

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Abstract:

The mission of this project was to decrease significantly the porous membrane in our MEMS-fabricated tensiometer [1] to be used as a probe with high spatial-resolution. Following the protocol of Ohmukai [2], we used photolithography with image reversal to pattern the membrane. We modified the membrane's etching recipe since the area coverage was less than original devices. We reported on the characterization of the patterned membranes by optical and electron microscopy and the formation of test devices based on anodic bonding with glass. We concluded with our measurements of permeability of the patterned membranes and perspectives for future experiments and applications.

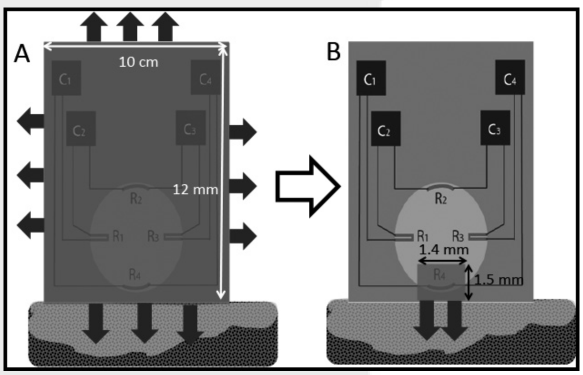

Figure 1: Mission. Two tensiometers measuring a soil sample with varying water potentials. Figure A represents a device with a full membrane receiving information from all its surroundings, while (B), with a smaller membrane, receives information from a specific area (higher resolution).

Introduction:

Our tensiometer quantifies the water potential in plants and soils directly as pressure. It measures the pressure in an internal water-filled cavity that equilibrates with the measured media through a porous silicon (PoSi) membrane, a layer of nanopores electrochemically etched on a silicon wafer. Previously the PoSi connected the cavity with the perimeter of the whole sensor, thus the sensor equilibrated with all its surroundings. Our goal (Figure 1) was to decrease the PoSi so the sensor will have a higher spatial resolution. We faced two main challenges. First, we had to use a technique to decrease the patterned area that did not compromise the bonding process or the ability to sustain large tensions. Second, there was a need to increase the permeability of the PoSi to compensate for the decrease in cross-sectional area relative to the original design.

In a broader view, by characterizing these devices we could improve water management in agriculture by providing *in situ* values of water availability in soils and plants.

Experimental Procedure:

Porous Membrane Patterning. We designed a mask using L-edit with the new porous membrane dimensions, 1.4 mm wide and 1.5 mm tall. The patterning process consisted of basic photolithography steps using S1827 photoresist. We did an image reversal and a hard bake. After the wafer was successfully patterned, we proceeded with creating the pores in the areas not covered by the photoresist.

PoSi Etching. Using the previously-used electrochemical etching setup [1], and a 1:1:2 HF:water:ethanol solution (mixed in a separate container) we were able to create the PoSi. We used the same current density as in the previous versions of the tensiometer's porous membrane of 20 mA/cm^2, so given the change in the total area coverage from the whole wafer to 1.3%, the current had to change from 900 mA to 12 mA. However, the time the wafer was exposed to the current remained at five minutes. In order to increase permeability, we created bigger holes in some devices by lowering the HF concentration to 21%. After the wafer was successfully etched, it was anodically bonded to glass, diced using the dicing saw, and studied under an optical and electronic microscope.

Permeability Testing. We compared previous devices (full membrane) with new patterned devices with membrane containing small and big pores. The purpose of the tests was to determine how effective these new devices were. Test devices had a 16 μm-deep cavity ranging from 1.4 to 2 mm in diameter, and either a full or patterned membrane. First, each device was filled with water at high pressure (~ 950 psi) using a pressure bomb [1]. Second, three to four water-filled tensiometers were placed in a sealed Petri® dish that contained a saturated NaCl salt solution setting a relative humidity of 76%. Finally, the devices where placed under a stereoscope to record the time each of them took to empty. The experimental setup can be found in Figure 2.

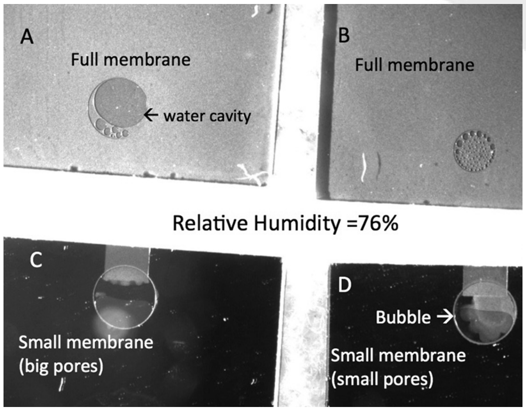

Figure 2: Permeability test. Devices (A) and (B) contain porous membrane covering them completely, while (C) and (D) have the new patterned porous membrane. All four of them (A, B, C and D) have bubbles in the water cavity, which means they have started to empty.

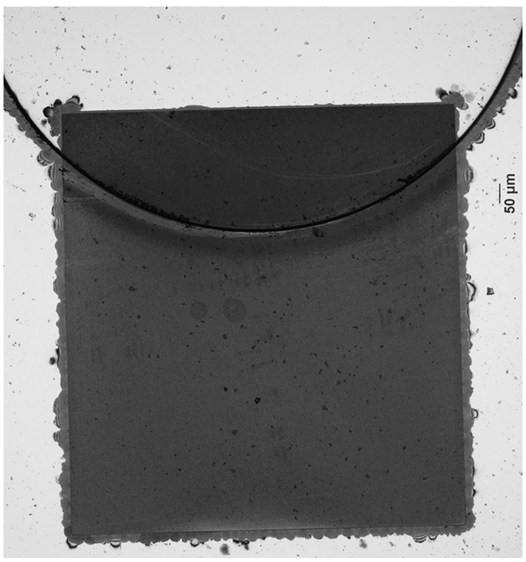

Figure 3: Top view of tensiometer with the new patterned membrane.

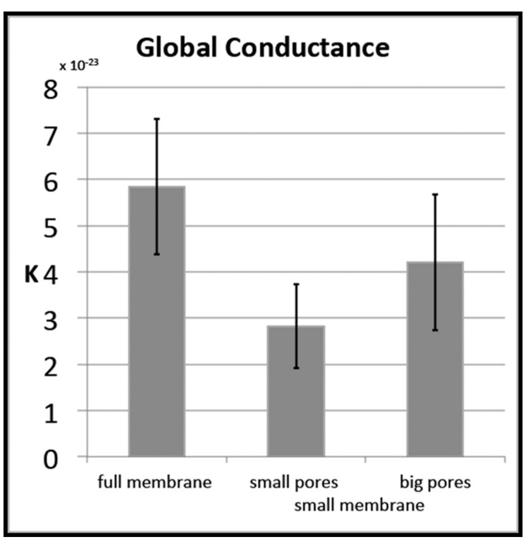

Figure 4: Global water conductance graph.

Results and Conclusions:

We successfully decreased the patterned area with photolithography patterning (Figure 3). Regarding its thickness, the membrane was expected to be ~ 5 μm thick, however it measured ~ 2 μm. We tried to correct this issue—altering the etching by increasing the current density and the exposure time—but the wafer deformed quickly. One reason for the decrease in thickness was that the larger ratio of edge to bulk etch areas led to a larger portion of the current passing through the edges, decreasing etching of the central area. Also, the devices were bondable.

With the emptying times, the membrane total area coverage, and the Darcy's law, we were able to compare each type's global conductance and permeability. Darcy's law states that the global conductance is directly proportionate to the area of PoSi, so it was expected that the full membrane devices had a higher conductance (Figure 4). The small membrane/small pores devices were two times less conductive than full membrane devices. Bigger pores were created in the patterned devices to compensate for the membrane area loss, however these were not as conductive as wanted. The permeability stayed approximately the same; values ranged from 1.25 to 2.25. We concluded that there is a need to increase conductivity for faster response time.

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References:

[1] Pagay, V., et al. Lab Chip 14, 2806-17 (2014).

[2] Ohmukai, M., et al. J. Mater. Sci. 16, 119-121 (2005).