Fabrication of a Selective Ion Pump: Anodization of an Aluminum Oxide Membrane

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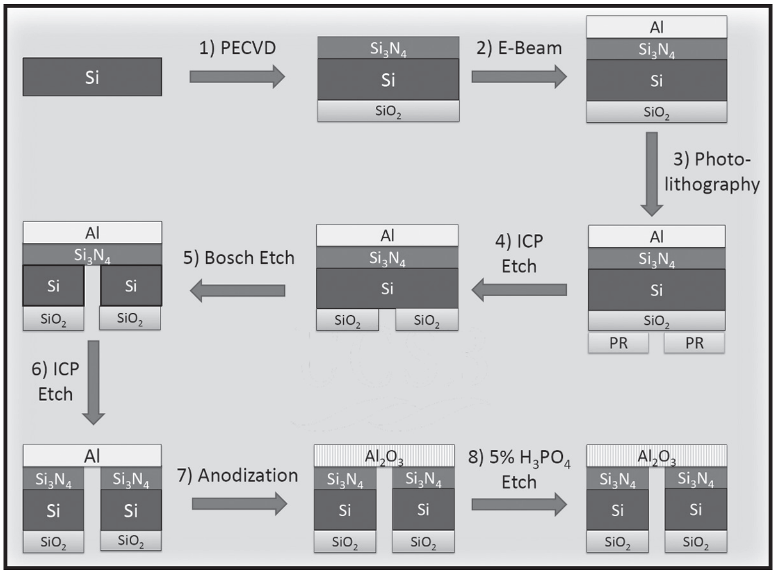

Figure 1: An outline of the fabrication process.

Abstract:

Anodized aluminum oxide nanoporous membranes show great potential for biological interfaces due to their controllable pore size, pore regularity, and biological stability. In this study, anodized aluminum oxide was investigated for use in a through silicon selective neural ion pump. A thin layer of aluminum and silicon nitride was suspended over a 120 μm by 120 μm window in a silicon wafer. The anodization of the aluminum layer was achieved using sulfuric or oxalic acid at voltages between 12.5 and 40 V at 1°C, to form pores between 12.5 and 27.5 nm. The silicon nitride and alumina barrier layer formed were then etched using reactive ion etching and phosphoric acid etching to form a suspended aluminum oxide nanopore membrane.

Introduction:

Electronically interfacing with nervous systems holds immense potential for restoring function to damaged sensory organs [1]. Neural network interfacing, however, has thus far been limited by dangers and inefficiencies in electrode stimulation of neurons [2]. Implementing a selective neural ion pump to stimulate a neural response instead can stimulate neurons more safely and efficiently, but for this method to be practical, pumps would have to be small enough to reference individual or small groups of neurons and quickly deliver sufficient amounts of K^+ [3]. This can be achieved by decreasing the path length the K^+ would have to travel by fabricating a three dimensional stack device with a thin rigid membrane, in contrast to planar ion pumps demonstrated by Richter-Dahlforsm et al. [4].

In this study, nanoporous anodized aluminum oxide (AAO) was tested as a membrane material, because it demonstrates controllable pore size, pore regularity, and biocompatibility [5]. Anodization is also a scalable process making the move from single pumps to an array of pumps relatively easy. For these reasons, AAO membranes were fabricated and suspended over through silicon (Si) windows in preparation for use in a selective neural ion pump.

Experimental Procedure:

Using plasma enhanced chemical vapor deposition, 100 nm of silicon nitride (Si_xN_y) and 1.5 μm of silicon oxide (SiO_2) were deposited on opposite sides of a 0.5 mm thick double-side polished Si wafer. Then 100 to 1000 nm of aluminum (Al) was deposited onto the Si_xN_y layer using electron beam evaporation. Using photolithography, a hard mask was etched into the SiO_2 layer for Bosch etching. Four hours and thirty-seven minutes of Bosch

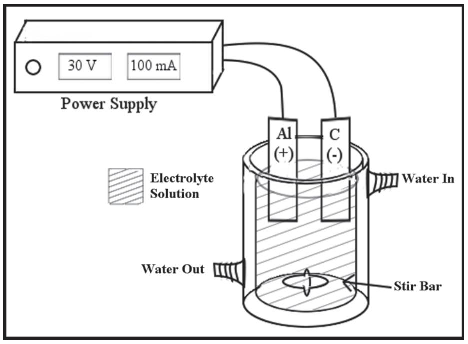

Figure 2: A diagram of the anodization setup.

etching separated the wafer into 15 by 15 mm squares with 120 by 120 μm windows through the Si in the center. Across the windows a thin layer of Si_xN_y and Al was left suspended. The Si_xN_y layer was then removed using ICP etching leaving the Al layer. This was then anodized using the setup shown in Figure 2. 3M insulating tape was used to protect the SiO_2 side so that pore formation occurred only on the side exposed to solution.

Anodizations took place at 1°C in 0.3 M oxalic acid or 5% (w) sulfuric acid using a graphite counter electrode. A constant voltage set between 12.5 and 40 V with a maximum current of 100 mA was applied for 2 to 60 minutes producing AAO nanopores with a thin barrier layer of alumina at the bottom of each pore. The 3M tape was removed and SPR220-3.0 photoresist was applied to the opposite side protecting the AAO except where exposed by the Si window. This allowed the barrier layer to then be etched using 5% (w) o-phosphoric acid, keeping pores intact. The nanoporous membranes were then characterized by field emission scanning electron microscopy (FESEM) to determine pore diameter, uniformity, and depth.

Results:

FESEM images of the resulting pores showed a linear relationship between average pore diameter and applied anodization voltage. Pores formed at a ratio of 0.59 nm/V and 0.68 nm/V for oxalic acid and sulfuric acid respectively, as shown in Figure 3. Irregularities in the Al surface during pore nucleation and an insufficient anodization length resulted in pores with lower regularity than those formed in other two-step processes as described by Wood and O'Sullivan [6]. The pores did, however, form completely through the Al as seen in the cross sectional image in Figure 4.

Backlit optical microscopy was used to evaluate the completed suspended membrane and the successful anodization of the Al. Unanodized Al or unremoved Si and Si_xN_y would show up as shadows on the optical microscope image. The absence of these shadows confirmed the suspension of a thin AAO membrane.

Future Work:

The next step in this project is to use these membranes in fabricating a selective ion pump similar to the pumps fabricated by Richter-Dahlfors, et al., and test the ion pump's ability to deliver K^+.

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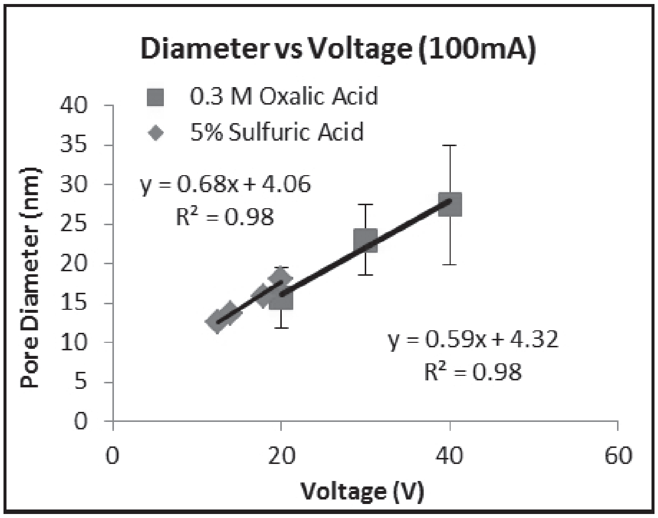

Figure 3: A plot of the average pore diameter against anodization voltage base on FESEM images.

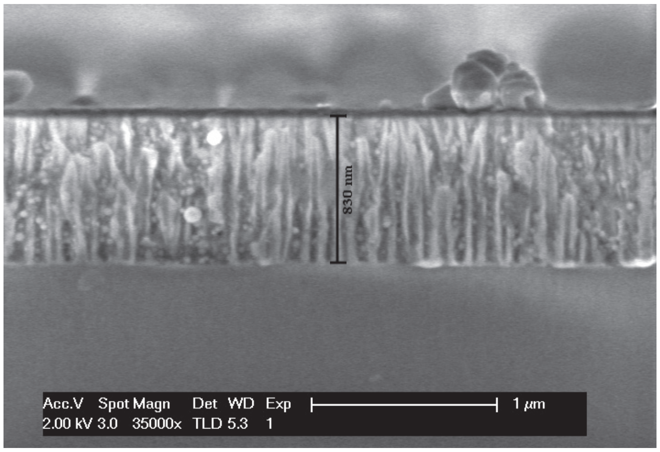

Figure 4: FESEM images of a cross section of AAO nanopores.