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SAND 2001-3398
Unlimited Release
Printed November 2001

Investigation of Nanoscience Technologies: Final Report

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Prepared by
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ABSTRACT

The intention of this project was to collaborate with Harvard University in the general area of nanoscale structures, biomolecular materials and their application in support of Sandia's MEMS technology. The expertise at Harvard was crucial in fostering these fundamentally interdisciplinary developments. Areas that were of interest included: (1) nanofabrication that exploits traditional methods (from Si technology) and developing new methods; (2) self-assembly of organic and inorganic systems; (3) assembly and dynamics of membranes and microfluidics; (4) study of the hierarchy of scales in assembly; (5) innovative imaging methods; and (6) hard (engineering)/soft (biological) interfaces.

Specifically, we decided to work with Harvard to design and construct an experimental test station to measure molecular transport through single nanopores. The pore may be of natural origin, such as a self-assembled bacterial protein in a lipid bilayer, or an artificial structure in silicon or silicon nitride.

Summary of Accomplishments

Derek Stein, a Harvard University graduate student under Prof. Gene Golovchenko, came to Sandia and helped us design a probe station based that will accommodate both natural and artificial pore platforms. We also visited the molecular biology laboratory of Prof. Dan Branton, a pioneer in the area of protein pores in membranes. He gave us designs for an experimental work station that will accommodate Derek's ideas. It was constructed at Sandia and is discussed below.

Single-channel ion pore apparatus

With designs from our collaborators at Harvard University (Dan Branton and Derek Stein), we have set up an apparatus for measurement of ion currents through single pore membranes. It will also be used to measure currents through solid state pores. The manufacture of the latter will discussed below. A variable-temperature patch clamp apparatus has been built, and is shown schematically below in Fig. 1. It is comprised of two reservoirs between which a small Teflon aperture can be positioned. We developed a technique to form ~25 μm apertures in a Teflon tube interconnecting the two reservoirs and are poised to insert bilayer lipid membranes (*e.g.*, diphytanoylphosphatidylcholine, "DPPC") over the aperture. A single protein pore will be created by the addition to one reservoir of α–haemolysin protein which is known to self-assemble into a heptameric channel 100 Å long and 29 Å in diameter. Other protein pores will be examined as well (*e.g.*, gramicidin). Electronics developed for patch clamps (Axon Instruments) have been acquired and will be used for picoampere measurements across the membrane before and after pore formation. To insure that we have single-channel measurements, rapid flow pumps and electronic valve switching have been installed to flush out residual proteins in solution as soon as ionic current through a single pore is detected (~100 pA).

We will be able to accurately characterize lipid bilayers and protein pores relative to previous studies and to assess modifications to either membranes or pores. Of particular interest is the testing of supported bilayers, required for scanning probes.

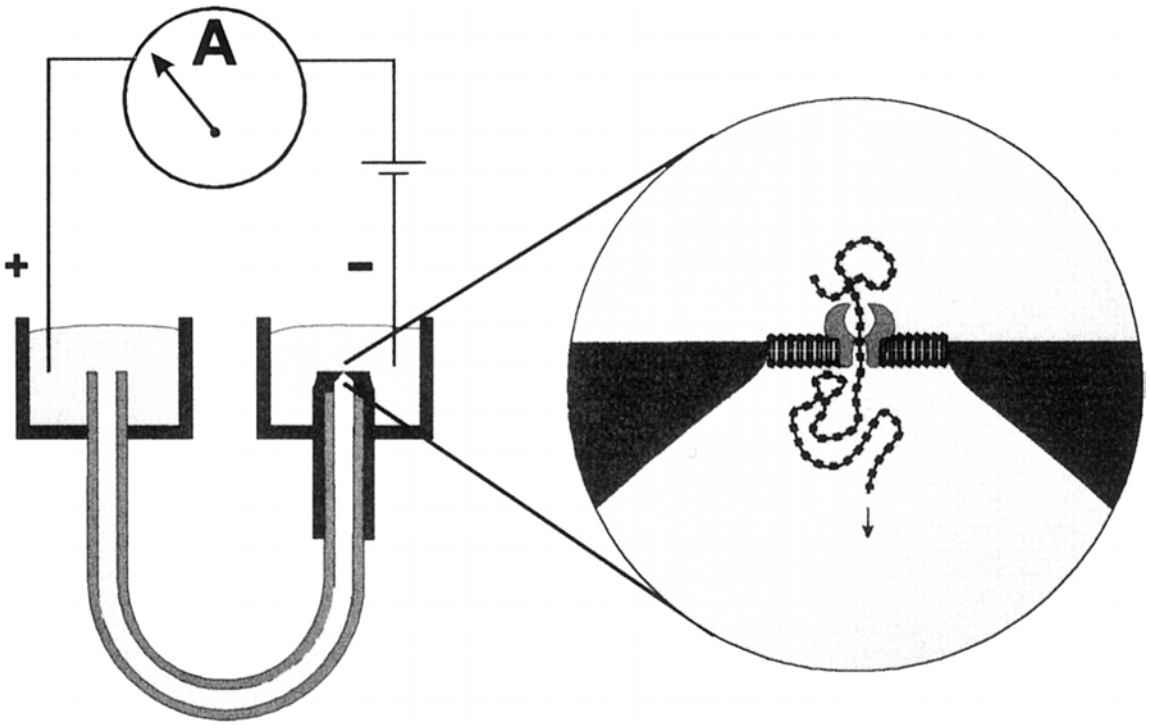

Fig. 1 Single channel ion pore apparatus

Fabrication of solid-state nanopores

Lipid bilayers are very delicate and have a short lifetime. More robust single-channel ion pores can be made from silicon substrates. However, standard lithographic or e-beam systems are unable to operate on the nm scale. We have a novel approach to this problem that relies on the highly reproducible thickness of deposited or grown films. We anticipate the use of these solid state pores at Sandia and at Harvard. They are created by the intersection of two orthogonal trench structures, one on top of the other (see A in Fig. 2 below). The "diameter" of the nanopore is thus equal to the width of the trenches (B). The effective "length" of the nanopore is equal to the interfacial layer thickness between the two trenches.

The schematic shows the critical steps in the formation of the trench. In C, the face of a layer of silicon is exposed and oxidized to form the sacrificial portion of the trench. This oxidation process is highly controllable. In D, the hole formed by the trench is filled in with silicon. In E the wafer is then planarized by chemical mechanical polishing and the top of the oxide filled trench is exposed. This process is then repeated after the deposition of a thin protection layer. The second oxide filled trench is oriented orthogonal to the first. At the end of the process, the oxide filling the trench is selectively removed using HF. Another advantage of the approach is that the membrane thickness can be considerably thicker than the ~2 nm hole diameter, thus increasing robustness. However, the membrane is still only ~200 nm thick and to improve yield the thinned membrane is supported on a thicker 800 nm silicon nitride membrane with a ~800 nm hole centered at the crossing point of the trenches. The parts are released in a combination of KOH etching to remove the bulk of the substrate, hot phosphoric acid to etch a protective SiN layer, and HF to remove the trench oxide. The entire process consists of 4-5 mask levels. Parts have been fabricated and structures verified with TEM. One problem encountered to date is obtaining accurate metrology on these fine structures, since TEM micrographs are prone to distortion.

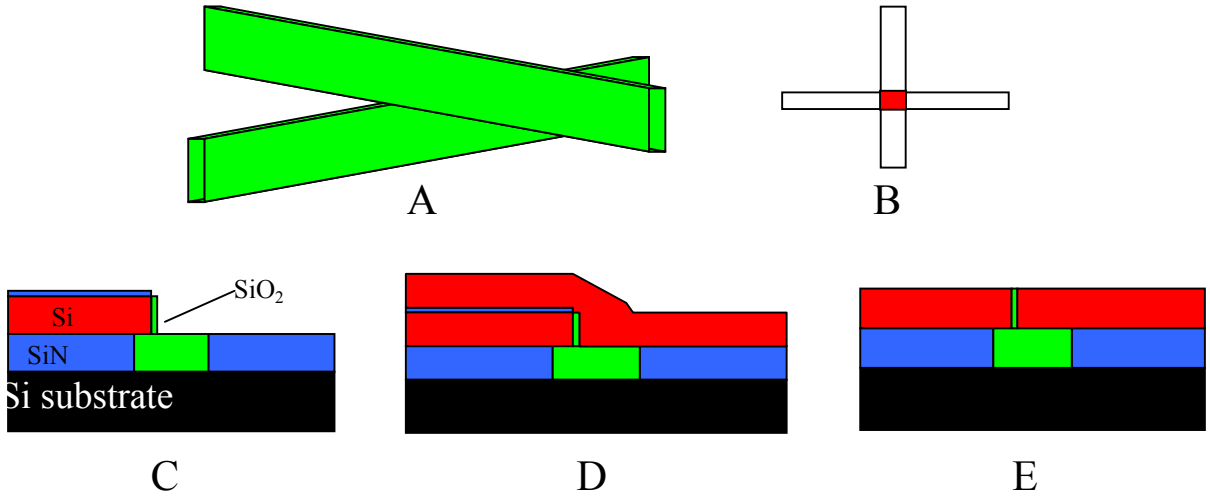

Fig. 2 Manufacture of solid-state nanopores

Summary

In summary, we have built a single channel ion pore apparatus and created procedure to create solid-state nanopores to use here and at Harvard (in Prof. Golovchenko's and Prof. Branton's laboratories). This project has been very fruitful in our ability to acquire technology from those groups at Harvard and to pass on technology to them. We anticipate future interactions.

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