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Focused Ion Beam Fabrication of Isolated Nanopores in Membranes: Overcoming Effects of Channeling

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Solid-state membranes having isolated, nanometer-scale pores are currently of interest for a variety of research activities including studies of near-field optics[1] and investigations of biomolecule-surface interactions[2]. For example, with regard to studies of DNA translocation through membranes, an isolated nanopore offers the ability to evaluate the behaviors and properties of a single molecule. This is of interest for sequencing DNA – central to genomics. To date, research involving solid-state membranes has largely relied upon fabricating pores into commercially-available, freestanding silicon nitride or silicon oxide membranes that are bonded at their edges to much thicker silicon substrates[3,4]. Recently, several studies have demanded a more diverse set of membrane materials and pore geometries.

In this study, we demonstrate a method for fabricating nanopores in different membrane materials including polycrystalline metals. First, low stress films of the desired material are coated by vapor deposition onto commercially-available silicon nitride membranes (attached to silicon substrates for ease of handling). Focused ion beam methods are then used to locally remove an approximate 25 x 25 μm window of silicon nitride providing access to the deposited material. Once exposed, this new membrane is FIB sputtered at a single pixel to define a pore.

For sputtering nanopores, we employ a dual lens, ion-pumped Magnum FIB column (FEI, Co.) mounted on a custom vacuum system. We also utilize a backside channelplate detector for endpointing in order to minimize pore diameter and overcome the challenges of point-to-point variation in sputter yield (a common challenge to working with polycrystalline metal membranes). The endpointing instrument includes an APD 3040MA 12/10/8 D backside microchannelplate detector (Burle Industries, Inc.) and external electronics (preamplifier, computer with software). With real time monitoring, we are able to identify the time of perforation and terminate sputtering soon thereafter. For these experiments, the detector front plate is biased to –1500 V and the back plate is grounded. See Fig. 1.

Representative detector responses associated with nanopore fabrication are shown in Fig. 2. This includes data taken from FIB single-pixel sputtering of amorphous silicon nitride, silicon nitride with a capping layer of W, nickel and aluminum. In each set of experiments, we use a focused, 30 pA gallium beam. Membrane thicknesses are ~ 200 nm. Previous analysis of the silicon nitride 'drilling' experiments demonstrates that a through hole (pore) is completed at the time that corresponds to the onset of a maximum detector signal[4]. We refer to this as the time to perforation. These prior experiments also showed that hole diameter increases if the ion beam is kept 'on' for longer time. The benefit of the endpointing technique shown in Fig. 1 is clear. If a minimum pore diameter is desired, FIB sputtering can be terminated soon after the onset of a maximum detector signal. For metals this endpointing technique is essential to compensate for point-to-point variations in sputter rate. The two metals (Ni and Al) are affected by ion channeling as x-ray diffraction shows the membranes to be polycrystalline with a moderate in-plane texture. As such, holes are not completed in metals at a single time (as for amorphous silicon nitride). For Al, the time to perforation varies greatly due to channeling effects and surface roughness. A nanopore fabricated in a nickel membrane is shown in Fig. 3.

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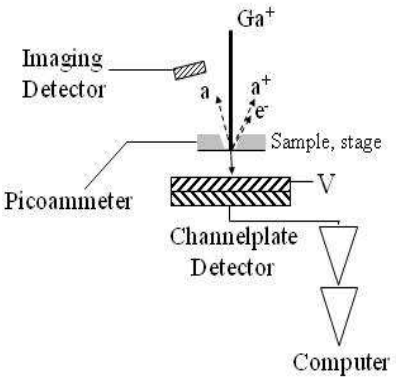

FIG. 1 Experimental setup for detecting the time of perforation when FIB 'drilling' foils and creating small-diameter nanopores.

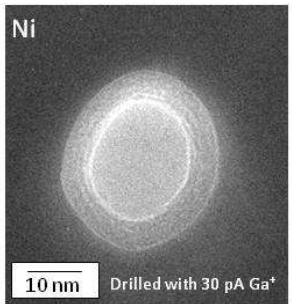

FIG. 3. Transmission electron image of a nanopore sputter-fabricated by a 30 keV focused gallium ion beam in a polycrystalline nickel membrane.

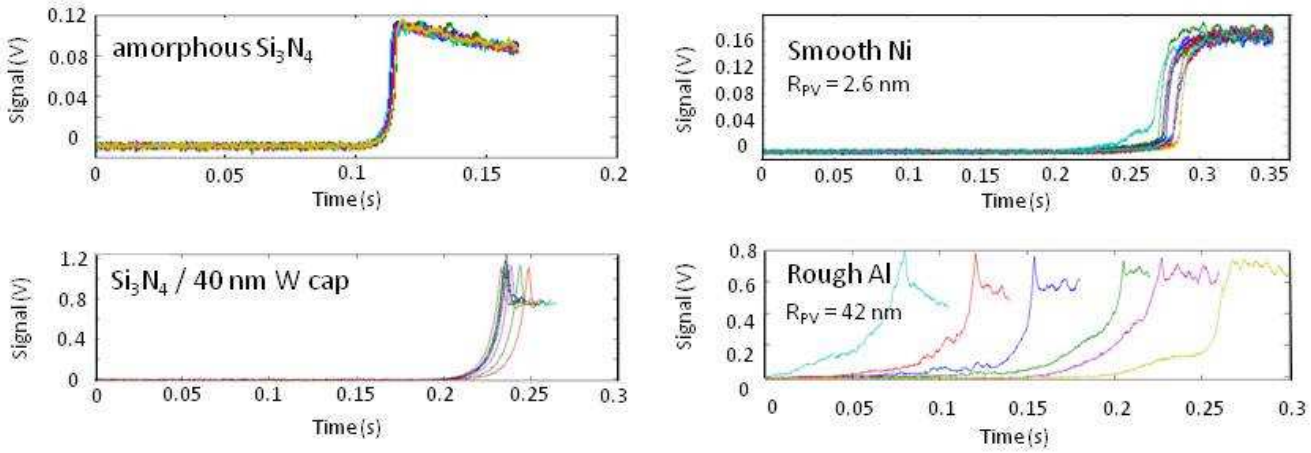

FIG. 2 Detector responses when FIB sputtering through various membranes at random points. Note, the onset of a maximum signal has been correlated previously with the time of perforation. Therefore, the data sets show a reproducible time for perforating amorphous membranes and a highly-variable time for perforating polycrystalline metals, particularly rough Al.

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