

# Patterning of porous silicon nitride membrane by CsCl self assembly

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Thin silicon nitride membrane has been used for transmission electron microscopy (TEM) “windows” due to its transparency to high energy electrons [1]. However, to completely eliminate the background scattering, porous membrane is desirable. Micro-scale porous membrane TEM windows fabricated by photolithography and etching is commercially available. For nano-scale pores, though it has been fabricated using focused ion beam (FIB) milling (for the application of extraordinary optical transmission devices), FIB is too slow for practical applications. On the other hand, the high throughput nanoimprint lithography cannot be used to pattern the membrane because it will break the fragile membrane easily. Previously porous nano-crystalline silicon membrane has been fabricated by self-assembly of a thin amorphous silicon film during a rapid thermal annealing process [2]. In this work we report the fabrication of porous silicon nitride membrane by CsCl self assembly and pattern transfer. Such a porous membrane would also find applications in separation or filtration of macromolecules or nano-particles [3-4].

The fabrication process is shown in Figure 1. First, silicon nitride membrane was fabricated using the standard photolithography and anisotropic KOH etching process. Then a liftoff layer of PMGI (poly(dimethyl glutarimide)) was spun-coated, onto which CsCl was thermally evaporated and allowed to self-assemble in air to form hemispheres. Next, PMGI is etched using oxygen RIE with over-etch to obtain an undercut profile for easy liftoff. Subsequently, Cr was evaporated and lifted off by dissolving PMGI using in AZ 300 MIF developer. Finally, holes were etched in the nitride membrane and Cr removed by Cr etchant. As can be seen, the current process is of low cost and high throughput.

The hole diameter and inter-hole spacing depend mainly on the CsCl film thickness, the air humidity and exposure time to air. For instance, when a very thin CsCl of 3 nm was exposed to air for 24 hours, the island size is around 100 nm, and inter-hole spacing roughly 600 nm (Figure 2a). The completed porous membrane is shown in Figure 2b. With 15 nm CsCl and exposure to air for 60 min, the hole size in the membrane is about 150 nm and is rather uniform, though some holes were missing due to incomplete liftoff of Cr.

<sup>1</sup> A. W. Grant, Q. H. Hu and B. Kasemo, *Nanotechnol.*, 15, 1175 (2004).

<sup>2</sup> C. C. Striemer, T. R. Gaborski, J. L. McGrath and P. M. Fauchet, *Nature*, 445, 749 (2007).

<sup>3</sup> J. L. Snyder, et al. *J. Membrane Sci.*, 369, 119 (2011).

<sup>4</sup> T. R. Gaborski, et al. *ACS Nano*, 4, 6973 (2010).

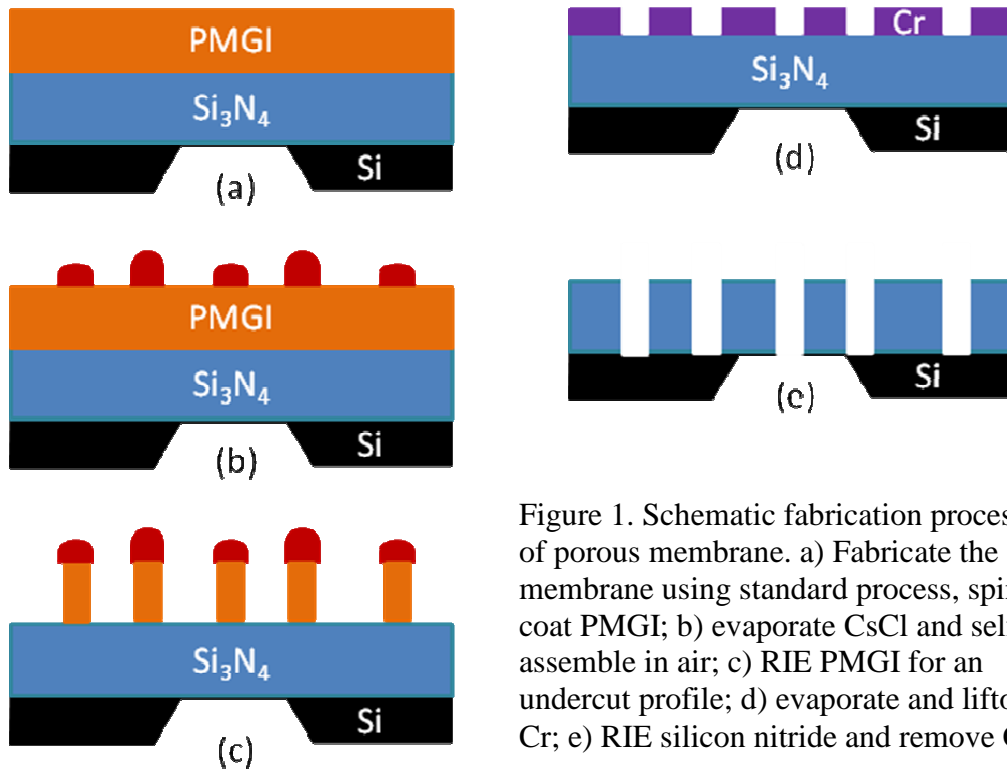


Figure 1. Schematic fabrication process of porous membrane. a) Fabricate the membrane using standard process, spin coat PMGI; b) evaporate CsCl and self-assemble in air; c) RIE PMGI for an undercut profile; d) evaporate and liftoff Cr; e) RIE silicon nitride and remove Cr.

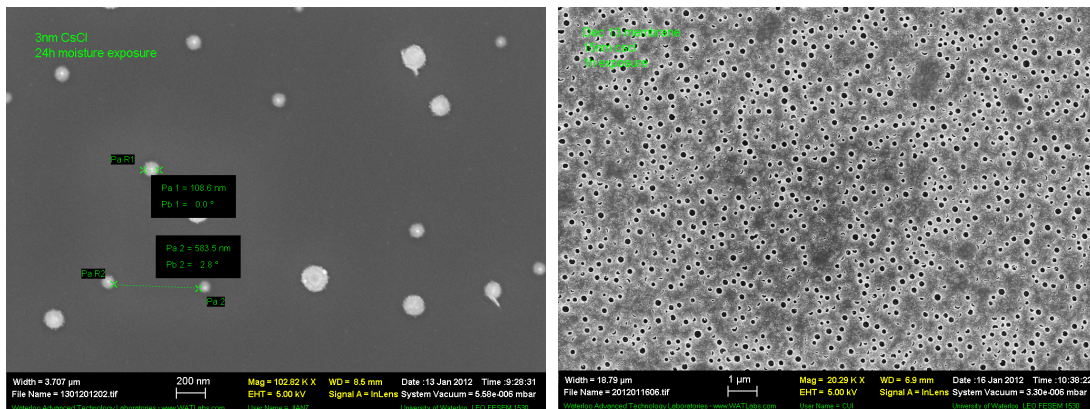


Figure 2. (a) SEM image of self-assembled CsCl, with film thickness of only 3 nm and exposure to air for 24 hours. (b) SEM image of completed porous silicon nitride membrane with pore diameter of  $\sim 150$  nm. Here 15 nm CsCl was exposed to air for 60 min.