

# Nanofluidic Liquid Cell with Integrated Electrokinetic Pump for *In Situ* TEM

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Enclosed cells for the measurement of liquid environments by the transmission electron microscope (TEM) are enabling breakthroughs in material growth and dissolution, electrochemistry, nanofluidics, biomineralization, and soft materials.<sup>1</sup> There are a number of different designs for TEM liquid cells, determining the functionality and fluidic interface of these devices. Some, with liquid thicknesses  $\sim 1 \mu\text{m}$ , permit liquid flow.<sup>2</sup> Others, with liquid thicknesses  $\sim 100 \text{ nm}$ , allow for high-resolution imaging, but, if flow is possible, have limited flow control. Recently, we developed a monolithic liquid cell that maintains a constant thickness of liquid of  $\approx 100 \text{ nm}$  across a viewing area of  $200 \mu\text{m} \times 200 \mu\text{m}$ .<sup>3</sup> The ability to control fluid flow through our nanofluidic cell would greatly enhance its utility by allowing the addition of reactive species at predetermined times during observation and the removal of reactive fluid radiolysis products. However, current approaches to pump fluids through liquid cells typically involve the use of macroscopic equipment such as syringe pumps and capillaries external to the TEM. Nanofluidic liquid cells would require prohibitively high pressure to pump in this way, and, because of the concomitant low flow rates, would suffer from very slow exchange of fluids through macroscopic capillaries.

Here, we present an integrated electrokinetic pump to solve these problems and enable future integration of lab-on-a-chip analysis with the TEM. Figure 1 illustrates the critical steps in the fabrication process that enable the wafer-scale integration of the nanofluidic viewing area, fluid reservoirs, fluidic channels, and pump electrodes. The most important step is the use of a sacrificial layer of Cr, which can survive deposition of the top layer of SiN. The Cr sacrificial layer can then be etched out without compromising the properties of the photopatternable adhesive (PA). Figure 2 shows optical and scanning electron micrographs (SEM) of the fabricated device. The availability of such devices will enable the analysis of a wide range of processes *in situ*.

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<sup>1</sup> Ross FM Opportunities and challenges in liquid cell electron microscopy. *Science*, **350**, aaa9886-1, (2015)

<sup>2</sup> Ring, EA, de Jonge N. Microfluidic System for Transmission Electron Microscopy. *Microsc. Microanal.* **16**, 622–629, (2010)

<sup>3</sup> Tanase M, Winterstein J, Sharma R, Aksyuk V, Holland G, Liddle JA, High-Resolution Imaging and Spectroscopy at High Pressure: A Novel Liquid Cell for the Transmission Electron Microscope. *Microsc. Microanal.* **21**, 1629–1638, (2015)

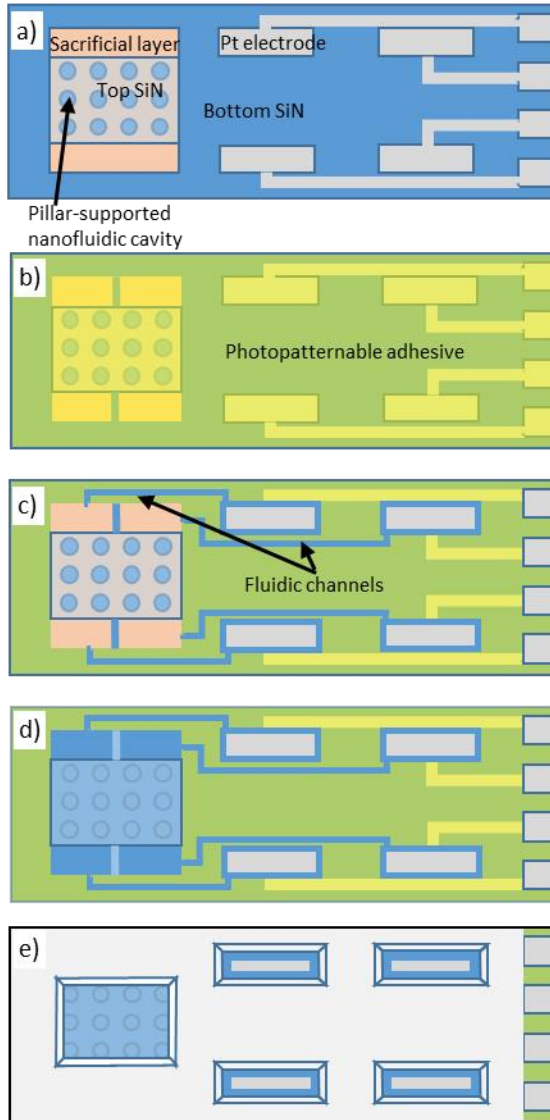


Figure 1. Schematic illustrating the process for fabricating a nanofluidic liquid cell with integrated electrokinetic pump. **a)** Deposition of bottom SiN layer, deposition and patterning of Pt electrodes, sacrificial layer and top SiN layer. **b)** Spin-coating of photopatternable adhesive (PA). **c)** Patterning of PA. **d)** Removal of sacrificial layer. **e)** Bonding of fluid reservoirs.

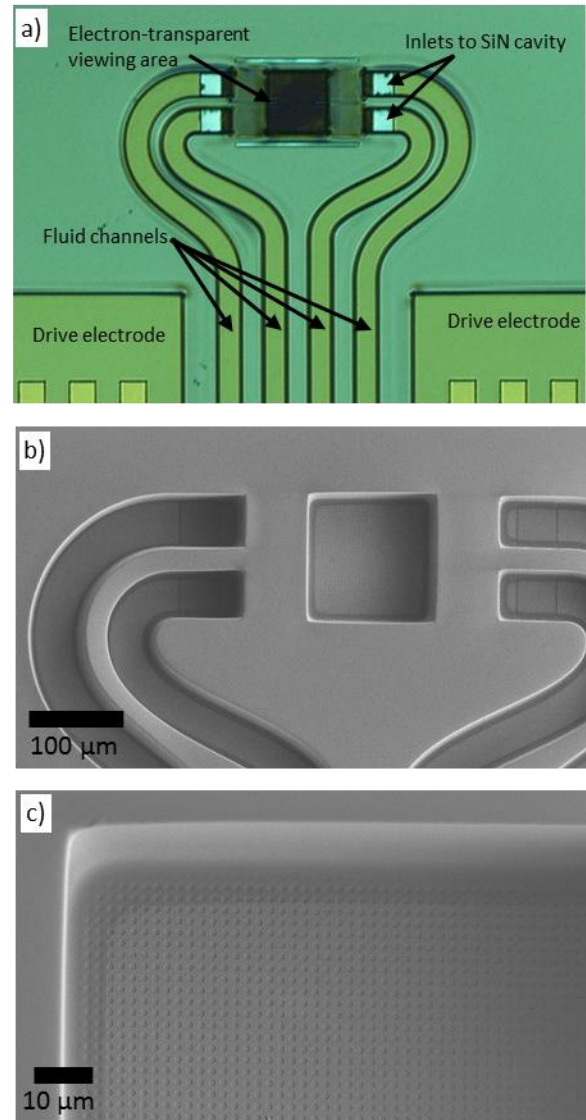


Figure 2. Micrographs showing liquid cell **a)** Optical brightfield micrograph of fluidic cell showing fluidic channels in PA adhesive, electron transparent viewing area and drive electrodes for electrokinetic flow control. **b)** Scanning electron micrograph showing interfaces of fluidic channels with SiN membrane viewing area. **c)** SEM micrograph of pillar-supported nanofluidic, electron-transparent viewing area.