A Liquid Cell for *In Situ* TEM: Design and Fabrication Challenges, and Solutions

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The transmission electron microscope (TEM) is a powerful characterization platform, capable of providing atomic-scale information on the structure, composition and morphology of materials. The restrictions on sample types imposed by the typical vacuum requirements for the TEM have led to the development of environmental TEMs (ETEMs) and enclosed sample cells to allow for the *in situ* and *operando* observation of materials undergoing dynamic processes. [1] The latter have the advantage of enabling access to higher pressures and more diverse chemistries than can be tolerated by ETEMs, and have been the subject of considerable research efforts.

Recently,[2] we introduced a design and fabrication process flow for a monolithic cell that maintains a thin fluid layer at a uniform thickness and can withstand high pressures. The cell can be integrated with electrokinetic flow control to enable dynamic modification of liquid chemistries inside the cell (Fig. 1). Here, we discuss an optimized fabrication process that satisfies the disparate constraints imposed by the desired functionality, and detail the steps taken to overcome process and material compatibility problems encountered in previous versions of the device. We now have a device design and process flow, both of which are robust and modular (Fig. 2), and allow for rapid modification of the functionality of the device to include options for temperature control, electrical and electrochemical measurement, and electrokinetic flow control. In addition, the thickness of the fluid layer may be varied from the nanometer to the micrometer scale. Finally, as a consequence of the unique encapsulant properties of the Cr_2O_3 sacrificial layer material that we employ, it is possible to pre-populate the active area of the device with lithographically-patterned nanoscale structures made from a wide range of materials. These may, for example, take the form of a precisely-defined array of model catalyst nanoparticles. This additional degree of control opens up new opportunities to conduct *in situ* and *operando* measurements.

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[1] Ross FM Opportunities and challenges in liquid cell electron microscopy. *Science*, **350**, aaa9886-1, (2015)

[2] Ray *et al*., EIPBN 2016, Nanofluidic Liquid Cell with Integrated Electrokinetic Pump for *In Situ* TEM

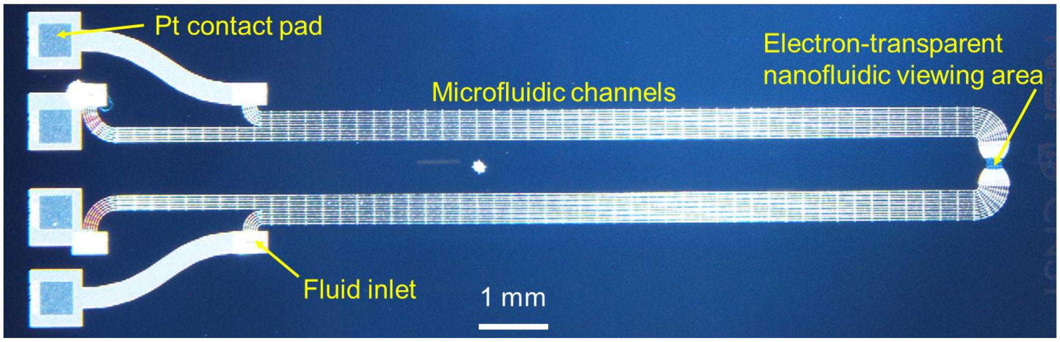

Figure 1. Optical micrograph of the nanofluidic cell with integrated electrokinetic liquid pumping scheme. Pt electrodes make direct contact to the liquid to establish the fields necessary to drive liquid flow.

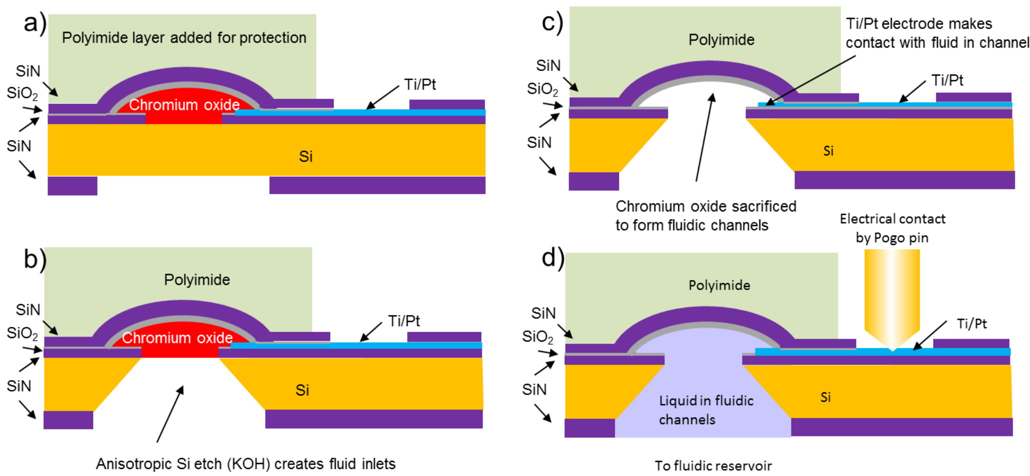

Figure 2. Final stages of the device fabrication process flow, illustrating the use of a Cr_2O_3 sacrificial layer to define the micro- and nanofluidic channels. The Cr_2O_3 also serves to protect the channel interiors and Ti/Pt electrodes from attach during the backside KOH etch. A thick (10 µm to 20 µm) photopatternable polyimide layer is used to protect the fluidic channels from mechanical damage that may occur during mounting of the cell into the TEM holder.