Fabrication of Nanoimprint-based Liquid Cells for Electron Microscopy incorporating e$^-$-transparent Al_2O_3 windows grown conformally by “flow-through” Gas Phase Deposition

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Abstract

Liquid cells are valuable tools that allow imaging of samples *in situ* at room temperature using transmission electron microscopes (TEMs). TEMs operate in a transmission mode under high vacuum. This environment brings unique challenges. On the one hand resolution depends critically on the thickness of the liquid and material layers the electrons pass through. On the other hand, the cell must be mechanically stable to withstand TEM vacuum conditions.

Previous studies have shown that good resolution can be achieved for liquid cells with window thicknesses not exceeding 100 nm. Most currently available liquid cells are fabricated from either silicon/silicon nitride or graphene sheets that are subsequently sealed. These liquid cells typically have simple geometries, often consisting of a single large rectangular chamber where the sample is placed and imaged [1].

Robust fabrication methods for more complex geometries without intrinsic weak points form sealing are still lacking.

Our goal is to develop liquid cells with complex microfluidic designs while maintaining high structural integrity. This will be achieved by combining established cleanroom fabrication techniques to fabricate channel systems with features down to 350 x 700 nm and flow-through gas phase deposition (GPD) to conformally coat channel walls and form e$^-$-transparent membranes (thickness <100 nm).

For the final applications, the sample will be located in nanoslits, which are connected to the inlet and outlet holes of the chip via microchannels. In the nanoslits the liquid is only confined by the thin, suspended Al_2O_3 membrane, which forms the windows and is transparent to the e$^-$-beam. The liquid cell design includes pillars inside the nanoslits to stabilize this membrane (Fig.1a).

In the first step of fabrication the liquid cell layout is structured on a silicon wafer using e$^-$-beam lithography (Voyager) as well as photolithography (Picomaster, MaskAligner), followed by reactive ion etching (RIE) to define the channel depth. This master structure is then transferred to a polymer substrate, via double stamping, using the method of direct nanoimprinting and UV-curable OrmoStamp® (EVG). For the final liquid cells we use a polycarbonate (PC) chip with pre-drilled inlet holes as a substrate. Samples are sealed with a multilayer coverslip (PC foil / PVA / 3Dresyn R190D90) to form closed channel systems (Fig. 1b). The Al_2O_3 membrane confining the liquid is fabricated by GPD. Conformal coating of the inner walls of the high-aspect-ratio microchannels and nanoslits is achieved using a custom-built GPD system that is operated in flow-through mode. Trimethylaluminium (TMA) and gaseous water are alternately introduced and carried through the structure by a nitrogen pressure gradient, forming Al_2O_3. The desired layer thickness is achieved by adjusting the number of deposition cycles [2,3].

After the GPD process, the coverslip can be selectively or fully removed by RIE to expose the underlying Al_2O_3 membrane. Complete removal of the coverslip shows a conformally grown Al_2O_3 membrane in the microchannels and nanoslits that is supported by the introduced pillar system. Different pillar designs yield varying levels of membrane suspension (Fig. 1c). To evaluate membrane suspension and stability under flow, test samples were prepared. Here, the coverslip is only partially removed in 28 × 28 µm window areas. This increases the stability of the Al_2O_3 membrane in all areas except for the window areas, through which the electrons must be transmitted (Fig. 1d). The flow tests revealed a high stability of the entire liquid cell as well as a good suspension of membrane in approximately 70 % of all exposed nanoslits.

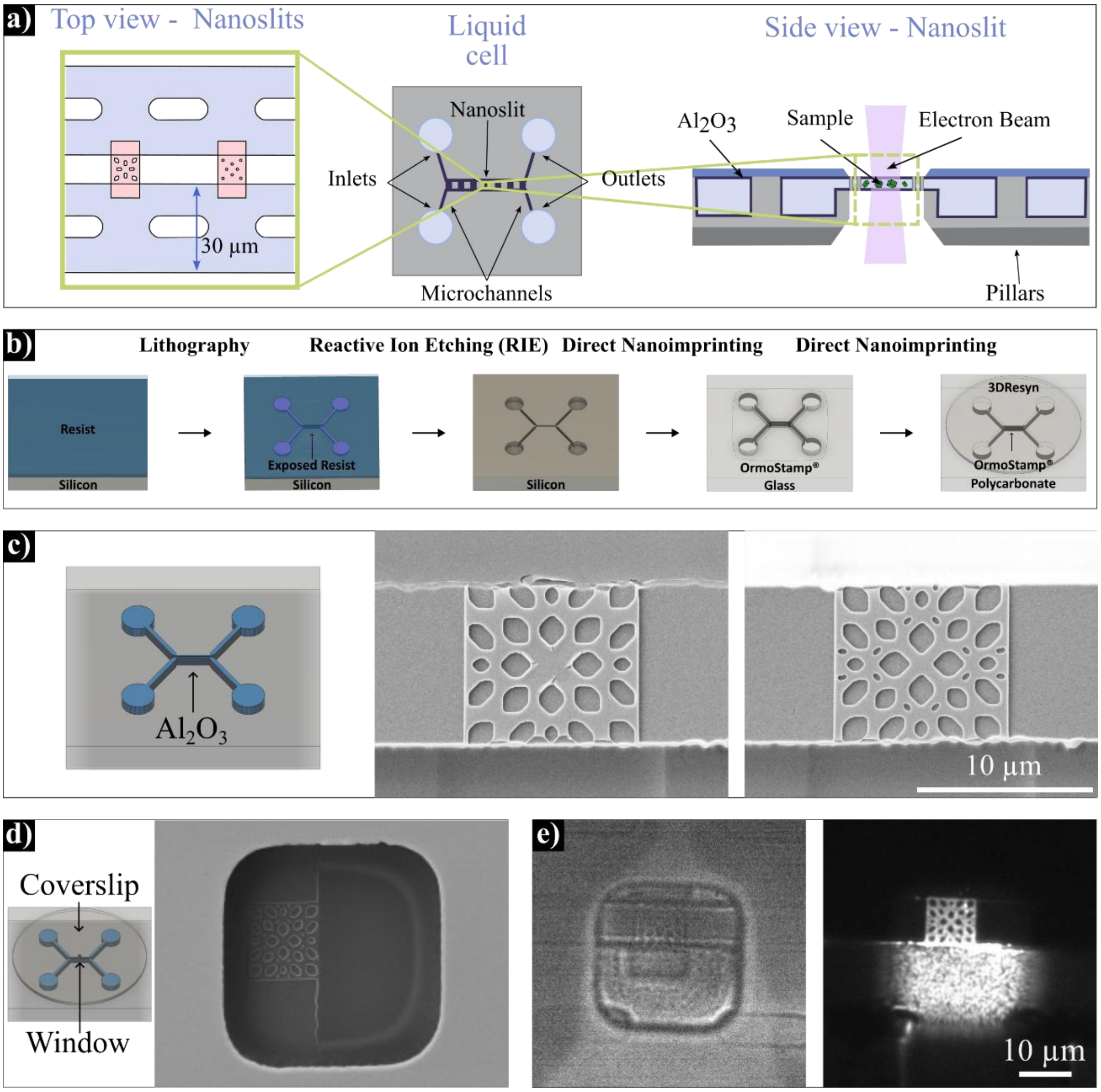

Figure 1: a) Overview of liquid cell concept, highlighting the nanoslits as the most important feature. b) Overview of the first fabrication steps using established cleanroom techniques. c) SEM images of a completely exposed Al_2O_3 membrane that was grown inside on the channel system and is supported by pillars in the nanoslit. d) Liquid cell after structuring of the coverslip to only expose membrane in windows of 28 x 28 µm. e) Brightfield and fluorescence image of 100 nm fluorescent latex beads stuck to the liquid cell walls after flow tests, indicating a homogenous flow through the entire area of the nanoslit.

References

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