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Fabrication of electron transparent membranes and nanostructures in fluidic devices by NIL and "Flow-Through"-gas-phase deposition

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Background incl. aims

Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are invaluable tools for visualizing and analyzing samples with nanometer resolution. Integration of liquid cells with these microscopy techniques has expanded their capabilities, allowing dynamic imaging and real-time analysis under controlled liquid conditions [1,2]. However, existing liquid cells still face limitations such as high production cost, restricted geometries, and reliance on slit-like chambers formed by connecting two membranes with spacers.

Methods

We present a novel fabrication method for producing micro-, and nano channels and 3D structures, self-sealed with electron transparent thin Al2O3 membranes, as sketched in Fig. 1a), 1b) and 1c). For this, we make polymeric foils (<10 µm) containing micro-, and nanofluidic structures. Fabrication of the fluidic devices involves direct UV nanoimprint lithography (NIL) using a customizable and versatile stamp design adaptable to specific geometries [3]. Then, the channels are coated selectively from the inside with Al2O3 by using our self-developed gas-phase deposition [4]. These inorganic electron-transparent structures can be suspended by removing the polymer material around them, enabling their use as liquid cells for investigating dynamic molecular behavior within confined spaces, such as in TEM.

Results

Our method allows fabrication of liquid cells with a variety of lateral dimensions, even down to nanometric channels, and complex 3D structures with graded depths and widths. The fluidic device contains two microchannels which connect the inlet and outlet holes. These microchannels are interconnected by several nanochannels. For fluidic devices fabrication, a stamp is placed and aligned onto a polycarbonate plate which has been covered with a UV curable polymer containing pre-patterned holes. The assembly is then cured with UV-light, and the substrate and stamp are separated manually. Next, a polymer coverslip is used to seal the channel system of the fluidic device. The Al2O3 coating is achieved through a specialized gas phase deposition (GPD) reactor operating in a "flow-through" mode (ftGPD), providing conformal coating of the various structures [4]. The reactor's gas and vacuum ports align with the holes in the polycarbonate plate, facilitating connection to the imprinted fluidic system. By precisely controlling precursor gas flow across the microchannel, a controlled pressure gradient is established, enabling conformal deposition of Al2O3 onto various structures, including slits, chambers, and micro- and nanochannels. This method enables to tune the Al2O3 thickness, ranging from a few nanometers to hundreds of nanometers, allowing for adjustment of mechanical stability and electron transparency as needed. This strategy effectively circumvents existing constraints associated with liquid cell geometries, particularly those limited to slit-like chambers. In subsequent fabrication steps, the micro-, nanochannels or membranes can be selectively suspended by masking and reactive ion etching (RIE). Fig 1a) shows hollow Al2O3 microchannel which are used for flow tests. Fig. 1b) shows an example of a suspended, hollow Al2O3 nanochannel with a cross section of 500 x 500 nm and a suspended length of 20 µm.

To validate the electron transparency of the Al2O3 membrane, we created a TEM grid sample coated with an Al2O3 membrane fabricated in our GPD reactor. Subsequently, polystyrene beads were deposited both above and below the Al2O3 membrane, and imaged using SEM and TEM techniques (Fig. 1 d) e) and f), respectively). SEM images (Fig. 1 (d)) captured the same area with two different detectors, where polystyrene beads beneath the membrane were detectable solely by the secondary electron secondary ion (SESI) detector. In contrast, TEM imaging (Fig. 1 (e)) of the sample in transmission mode at a separate location demonstrated the feasibility of imaging beads through the Al2O3 membrane. We also made a sandwich, by placing another Al2O3 membrane on top, and imaged the beads in TEM at 200 keV, as shown in Fig 1 f). Furthermore, we also imaged gold nanoparticles placed below the Al2O3 membranes deposited and suspended from the top in one of our microchannels. The particles were visible in the SEM at an acceleration voltage of 8kV (Fig. 1 c)). All these results confirming the electron transparency of the membranes deposited using our method. To deploy this system as liquid cells in a TEM specimen holder, detachment of the structured and coated polymer foil from the substrate and subsequent cutting to a 3 mm total diameter are necessary.

Conclusion

This fabrication method offers a versatile approach for creating liquid cells with complex geometries, overcoming limitations of existing slit-like chamber designs. The electron-transparent Al2O3 membranes enable dynamic imaging of samples within confined spaces using SEM and TEM, with potential future applications in nanoscale research and analysis. Further investigations using these liquid cells inside SEM and TEM will be presented at the conference.

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Graphic:

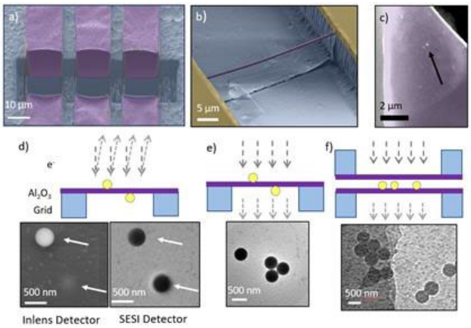

Figure 1: a) shows hollowed Al_2O_3 coated microchannel. b) displays a suspended nanochannel with a cross section of 500 x 500 nm and a suspended length of 20 µm. c) shows an SEM image of gold nanoparticles underneath the Al_2O_3 membrane. d) Sketch of the experiment to demonstrate the electron transparency of the aluminum oxide coating. A TEM Grid is coated with an Al2O3 layer and polystyrene beads are placed above and underneath. With an SEM, images of the beads could be taken at 5 kV e) This TEM image reveals the beads through the Al_2O_3 layer. The image was captured at 200 kV. f) Two TEM grids, coated with Al_2O_3, were stacked on top of each other with polystyrene beads placed in between. In order to image the beads using a TEM, the layer must be thin enough to be electron-transparent.

Keywords:

electron-transparent membrane, liquid flow cell

Reference:

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