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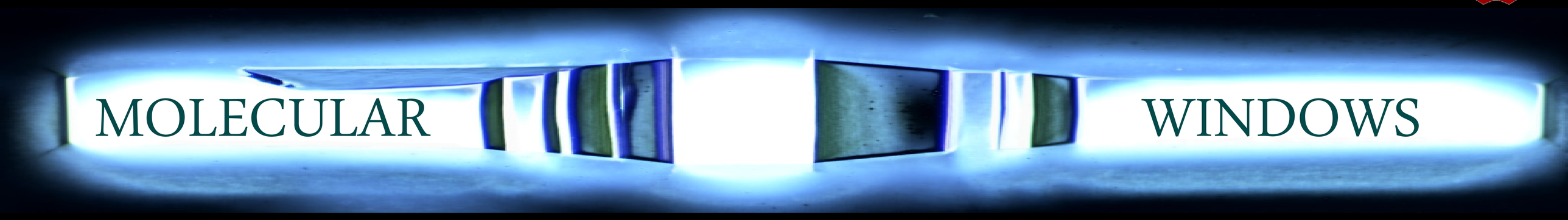
Publication date:
2013

Document Version
Publisher's PDF, also known as Version of record

DTU Nanotech
Department of Micro- and Nanotechnology



Novel Micro-fabricated Chip With Micro-channels for In-situ Observation of Liquid Samples and Processes in TEM

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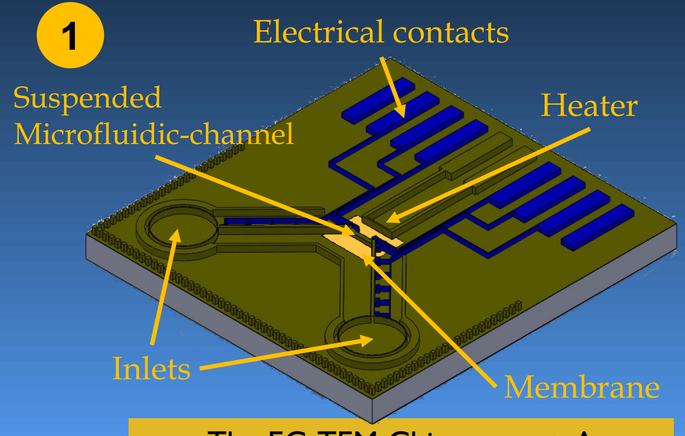
Developing a new in-situ chip

With the resolution of TEM reaching atomic scale increasing emphasis is being placed on ways to image samples live and under realistic conditions, it is therefore necessary to broaden the scope of electron microscopy to non-traditional samples such as liquids. State of the art systems use a sandwich approach of two identical chips with electron transparent windows[1]. In this work we present a monolithic design which avoids the alignment and bulging issues of such systems.

1



The EC-TEM Chip concept. A microfluidic channel suspended on a supporting membrane, all made in electrontransparent SiN_x

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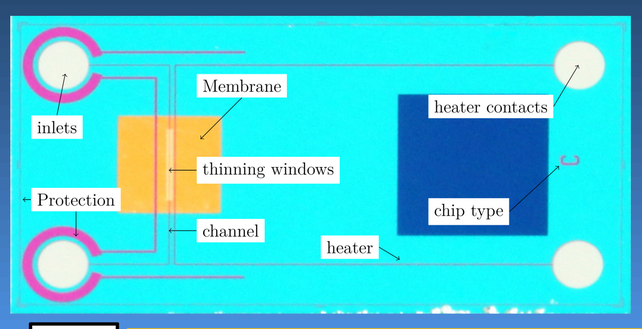
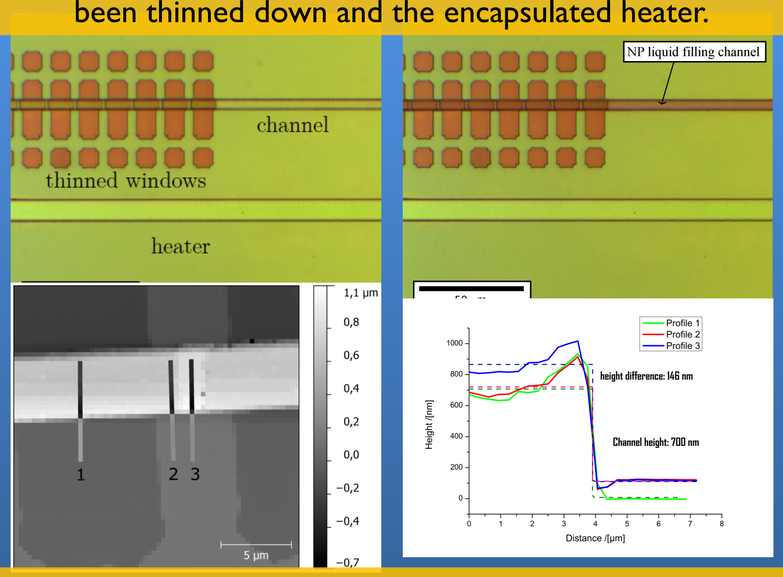


Optical image of a finished TEM chip prototype. The chip is 6.6 mm long, 4.8 mm wide and 351 µm thick.

3

Optical images of filling the SiN_x channels with Au NP liquid. Also visible in the images are the window areas where the SiN_x has been thinned down and the encapsulated heater.



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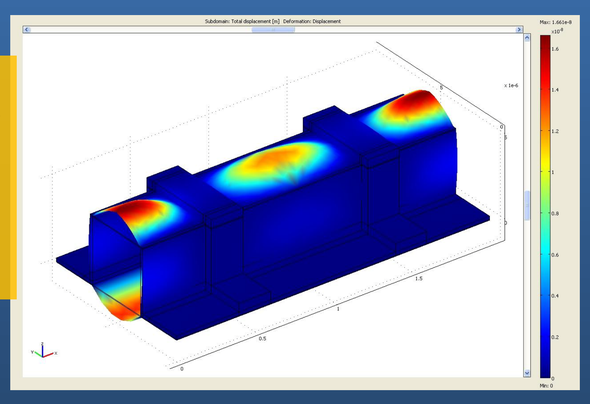
AFM scan of a channel on the TEM chip prototype. The channel and two thinned windows are visible. Three line-scan-profiles are visible and shown in the graph verifying designed dimensions

Fabrication and Characterization

The chip was fabricated using standard cleanroom fabrication techniques. The channel was defined in a sacrificial Si layer, covered with SiN_x, and then etched out. To ensure mechanical stability the top layer of SiNx was 175 nm thick and selected areas of the channel on the membrane were thinned down with an anisotropic etch to 25 nm to create thinner window regions. In this first prototype, the channel was 700 nm thick with 25 nm SiNx above and below it. AFM scans, optical images, and COMSOL simulations were used to characterize the prototype.

5

FEM simulations performed in COMSOL. These simulations show the window regions will bulge at total of 26 nm versus the ~2-3 µm often quoted for sandwich-chip systems[2-3].



TEM imaging and Conclusion

The TEM chip prototype was filled with a liquid containing 30 nm diameter Au nanoparticles (NP) and placed in a standard TEM holder. Images of the TEM chip were taken in a Technai T20 G2. The inlets of the channel, through which the liquid was introduced, were sealed with an acrylic varnish which dried and removed some of the liquid in the channel, resulting in bubbles.
This prototype has proven to significantly reduce sample preparation time for liquids[2] and allows for simply imaging of liquids in the TEM. Future versions will have reduced the channel height to improve the achievable resolution and include various active components such as heaters and electrodes. This work is being submitted to a special issue of the Journal of Microscopy and Microanalysis. In addition to this TEM system we are also developing an in-situ SEM electrochemicla setup[4].

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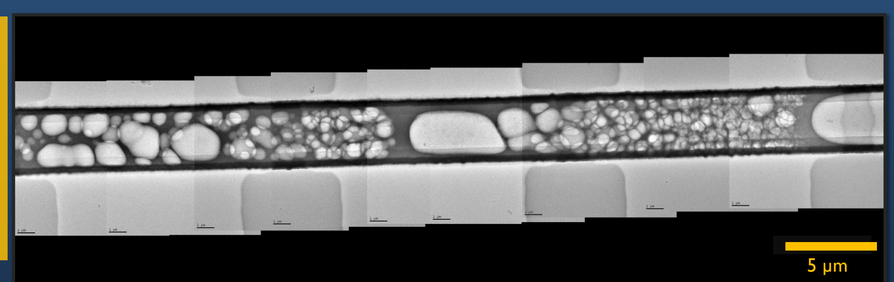
TEM image composite of liquid with bubbles in the micro-channel.



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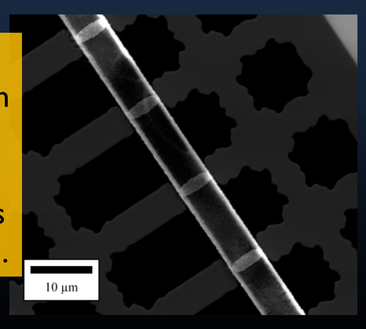
SEM image of an empty channel on the membrane. The periodic thinned windows are clearly visible.



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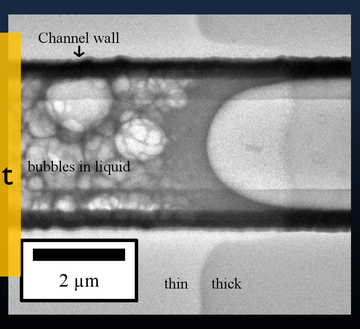
TEM image of channel filled with NP liquid. Note the shape of the liquid which is consistent with the hydrophilic SiN_x channel.



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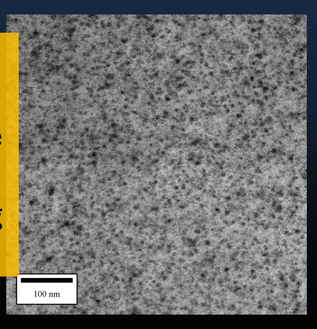
TEM image of Au nanoparticle containing liquid in the channel. The NP are out of focus because they are moving around in the liquid.



[1] de Jonge, N. and Ross, F. M., *"Electron microscopy of specimens in liquid"*, **6**, Nature Nanotechnology, 2011
[2] Peckys, D. B., Veith, G. M., Joy, D. C. and de Jonge, N., *"Nanoscale imaging of whole cells using a liquid enclosure and a scanning transmission electron microscope"*, 4, PLoS ONE, 2009
[3] Yang, J. and Paul, O., *"Fracture properties of LPCVD silicon nitride thin films from the load-deflection of long membranes"*, **97-98**, Sensors and Actuators A: Physical, 2002
[4] Jensen, E., Købler, C., Jensen, P. S. and Mølhave, K., *"In-situ SEM microchip setup for electrochemical experiments with water based solutions"*, **129**, Ultramicroscopy, 2013