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In-situ WetSTEM™ Analysis of FIB Fabricated Fluidic Structures

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Advanced techniques for analysis and fabrication can be used to further develop the field of microfluidics. Microfluidic devices for sensing and biomedical applications have been under rapid development over the past decade, with a variety of methods being used to produce tools for the study of confined fluid flow [1]. The challenge has been not only in device fabrication but also in the experimental observation and understanding of fluidic behaviour at the sub-micron and nanoscale. While some analysis has been successful using techniques such as fluorescent microscopy [2], the resolution has limited detailed examination of microchannel wall-liquid interactions. The Environmental SEM (ESEM™) has been a particularly helpful system of choice for experimental studies [3], but these experiments have been limited to nanostructures placed on a substrate and do not exclude the contribution of substrate-nanostructure interactions. Therefore, the investigation of fluidic behaviour in sub-micron and nano-sized channels has been vastly explored theoretically, but supportive experimental analysis is still lacking. In this work, a Focused Ion Beam (FIB) was used to create controlled structures of varying geometry and materials. This was combined with ESEM to perform wetting experiments on the fabricated channels. Specifically, by using STEM in a wet environment (WetSTEM), we provide *in-situ* analysis on the behaviour of confined fluids.

To fabricate various fluidic structures, the samples are lifted out of a substrate *in-situ* and mounted on a TEM half grid using an FEI Versa 3D™ DualBeam™. The samples can then be thinned in order to planarize the sides using the FIB and milled in order to fabricate the structures. Using this method, it is possible to generate structures on the same sample that exhibit different geometries and sizing in different materials, allowing for multiple analyses in a single session (Fig. 1).

Wetting experiments were then performed in the ESEM. A Peltier cooling stage for temperature control was maintained at a constant temperature, and vapor pressure in the chamber was increased or decreased to induce condensation or evaporation of water, respectively. Analysis was performed utilizing a WetSTEM sub-stage, which incorporates a STEM detector directly into the Peltier cooling stage.

As shown in Figure 2, the flow of water into the capillaries can be monitored *in-situ* during a condensation experiment. Since the samples are not placed directly onto a substrate, it is possible to decouple the effect of substrate-sample interactions, which is not possible during the top-down approach in traditional wetting experiments. Also of note is the ability to differentiate the water from the wall of the channel at high resolution. This allows for detailed observations of the interaction between the capillary wall surface and the liquid, as well as the direct observation and measurement of factors such as contact angles and contact lines. This becomes particularly important as the size of the capillary decreases and wall-liquid interactions begin to govern the behavior of the fluid.

By using a DualBeam, it is possible to fabricate precise structures of varying geometry and surface chemistry with different materials. Combined with WetSTEM in an ESEM, the wetting behavior inside sub-micron and nano-scale capillaries can be investigated in real-time. This study provides supportive experimental analysis and discussion of established theories in wetting and fluidic behavior in capillary structures.

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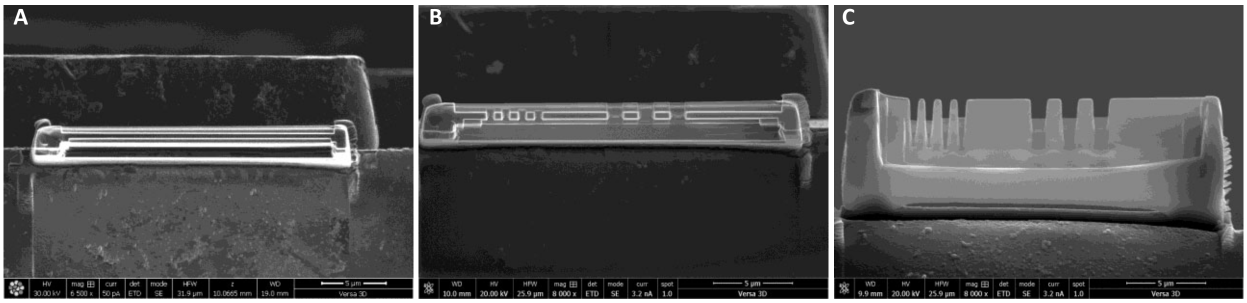

Figure 1. (A) Top-down view of sample after *in-situ* lift-out from Si, mounted and thinned, on the TEM half-grid. (B) Top-down and (C) Cross-sectional view of sample after fabrication.

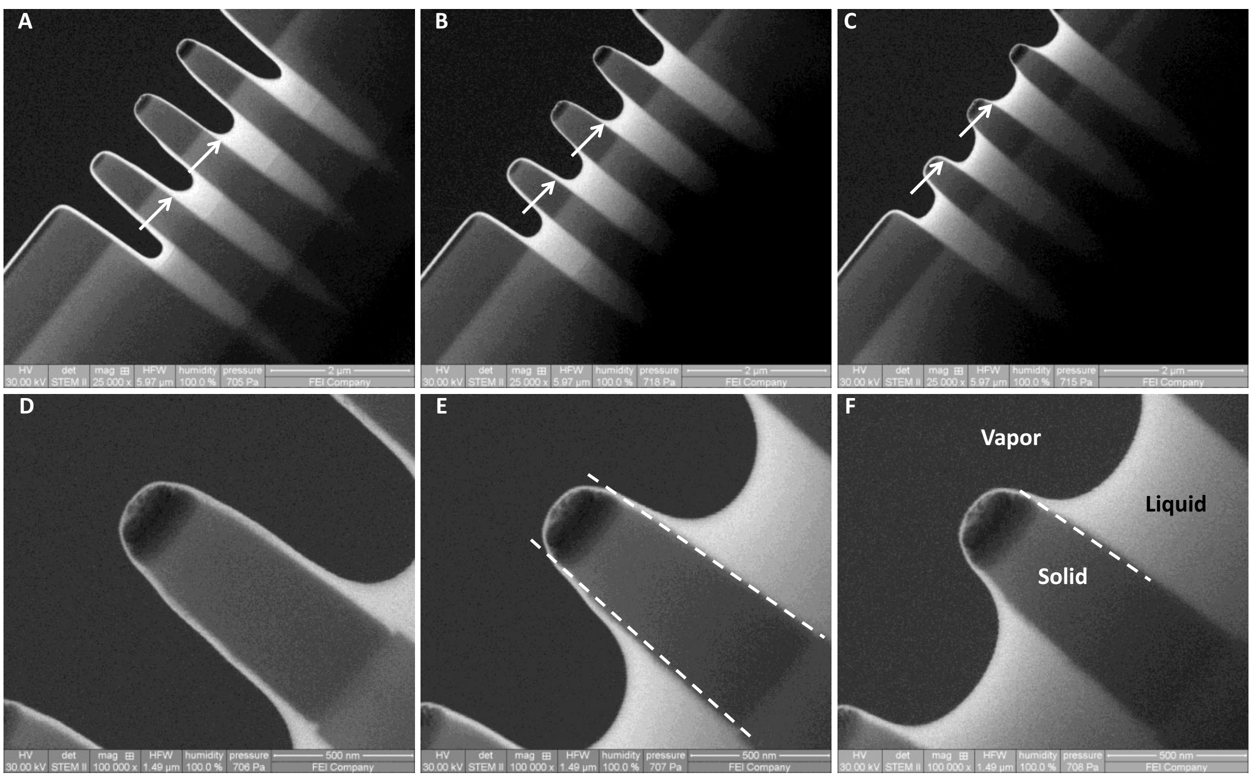

Figure 2. A-C: Dark Field WetSTEM images showing the flow of water through fabricated structures during a condensation experiment (arrows show movement of contact line). D-F: Dark Field WetSTEM images showing the ability to differentiate between the water and the material edge (shown in E with dashed lines) as well as the interphase between the solid, liquid and vapor phases (labeled in F).

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