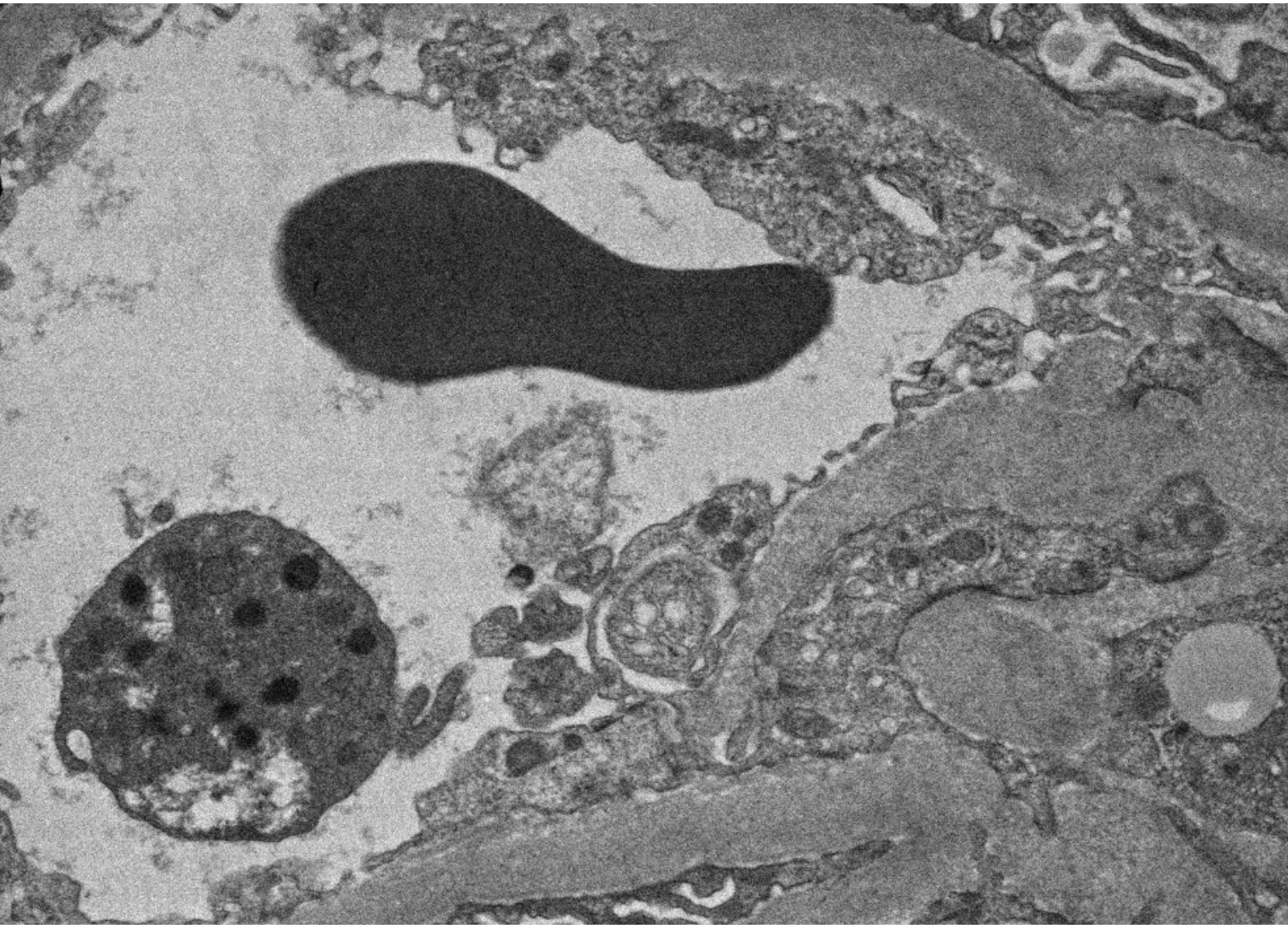
White Paper



Scanning Transmission Ion Microscopy for Imaging and Nanofabrication in the Helium Ion Microscope

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Scanning Transmission Ion Microscopy for Imaging and Nanofabrication in the Helium Ion Microscope

Author: Dr. Adam Hall
Joint School of Nanoscience and Nanoengineering, University of North Carolina at Greensboro, Greensboro, NC 27401,

Dr. Jijin Yang
Carl Zeiss Microscopy, LLC, Thornwood, NY 10594

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Thin (<100 nm) samples play a central role in a number of important research directions. For example, freely-suspended thin films like graphene and silicon nitride are being used increasingly for device purposes. Meanwhile, narrow cross-sections of biological specimens can reveal fine detail of cellular structure or even localize materials used for drug delivery.

Challenge

High quality imaging of thin samples can be performed routinely using transmission electron microscopy (TEM). However, such analysis has been challenging with scanning charged-particle microscopes like the scanning electron microscope (SEM) or the helium ion microscope (HIM). Largely, this is due to the fact that such instruments form images through the collection of secondary electrons (SE) produced by the interaction of the incident beam with the sample. In very thin samples, the interaction volume does not release a significant amount of SE, causing the contrast of resulting images to lie beneath the noise floor.
One solution that has been demonstrated with SEM mimics the operation of a TEM by instead forming an image from the beam that passes through the thin sample[1]. In this case, dense areas result in a reduction in transmission and thus in local image brightness and vice-versa. This method has been greatly successful, but generally requires the inclusion of a scanning transmission electron microscopy (STEM) detector positioned below the sample stage. Such an endeavor is costly and difficult in some instruments where many detectors and features may be present already.

ORION NanoFab Solution

Here, we explore a HIM alternative for performing scanning transmission ion microscopy (STIM). Transmission imaging with the HIM can be accomplished through the simple use of a STIM holder as shown in Fig. 1. This holder contains a through-hole that leads to an angled, polished metal surface below. The sample plate itself is designed hold typical 3 mm TEM grids and chips, but is interchangeable to accommodate other sample sizes as well. In this setup, the He beam that transmits through the sample continues on to interact with the metal surface, producing electron emission that is directed towards the SE (E-T) detector used for conventional imaging. In this way, no additional detectors need to be installed for transmission image formation.

Optimal imaging conditions are achieved through a few simple steps. After a sample is loaded and the holder is introduced into the imaging chamber, the stage position is adjusted. First, the stage rotation is set for 180 degree and the vertical (Z) position is changed to 13 mm. This blocking orients the polished metal surface towards the SE detector and positions the sample platform high enough to prevent it from obstructing emission produced below it. Next, the beam is optimized using either an unimportant area of the sample support or the top plate of the holder. A small feature is identified to fix the focus, stigmators, and wobble. Once this has been adjusted adequately, magnification is decreased and the sample is moved in order to position the portion of the sample that is above the through-hole in the imaging area. The correct area can be identified as a bright region in the low magnification image. Some focus correction may be necessary, but stigmation should not need to be adjusted further. At this point, a voltage is applied to the sample stage in order to limit SE emission from the top of the sample holder. This will cause nearly the entire detector signal to come from the transmitted beam. The sample voltage option can be found under the ET-detector tab and a value of 40 V is used typically.

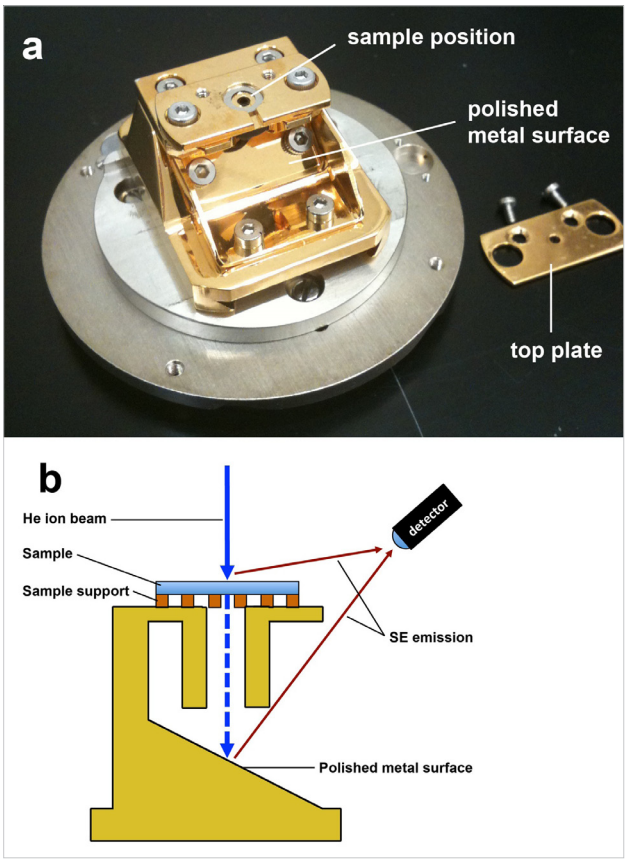

Figure 1 *The transmission holder (a) photograph of the holder with the top plate removed. (b) Diagram of the holder, showing the path of the transmitted He+ beam through a thin sample and onto the angled surface below. Secondary electron emission can be detected from either the top surface or the polished metal surface by the SE detector.*

Examples

The first example of transmission imaging with the HIM discussed here is a biological specimen. For these images, kidney cells were stained and fixed using preparation techniques common to TEM imaging. A thin (<100 nm) sample was produced using an ultramicrotome and affixed to a copper TEM grid, which was subsequently loaded onto the HIM transmission stage. A typical image is shown in Fig. 2. Cell organelles and objects can be identified clearly, including red blood cells (gray region near image center) and nuclear material (feature in lower left of image). These images are of a quality comparable to recent published work with TEM[2].

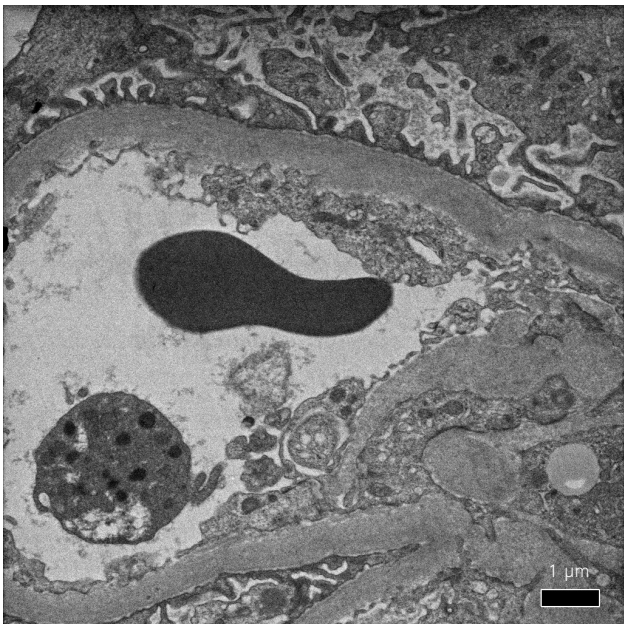

Figure 2 *Kidney cells HIM transmission image of fixed and stained kidney cells.*

Further examples of transmission imaging utilize thin suspended membranes of silicon nitride. In the examples shown here, we use membranes with initial thickness of ~25 nm and demonstrate the ability to mill samples in situ. Fig. 3 shows a transmission image of an array of 100 nm x 100 nm squares milled in the suspended membrane[3]. The squares were formed by rastering the beam through a square pattern for set amounts of beam dwell time that increase from left to right and from top to bottom in the image. As the membrane is made progressively thinner, a larger amount of the incident He+ beam is able to transmit and thus the image appears brighter.

Finally, with sufficient beam dosage at a single point, nanopores can be formed in the suspended membrane[4]. Fig. 4 shows a transmission image of a 5x5 array of nanopores in which the total beam dwell time is increased from left to right and from top to bottom, resulting in through-holes that range in diameter from ~5 nm to ~20 nm. Transmission imaging offers the ability to directly visualize HIM milled structures immediately following their fabrication. Resolution approaching that of TEM is not possible, as the long exposure time required would itself mill the material being imaged, but a size determination can be made to within about 3 nm even with a fast capture. We have demonstrated similar imaging capabilities using silicon nitride membranes as thin as 10 nm and as thick as 100 nm and can achieve similar image contrast and resolution.

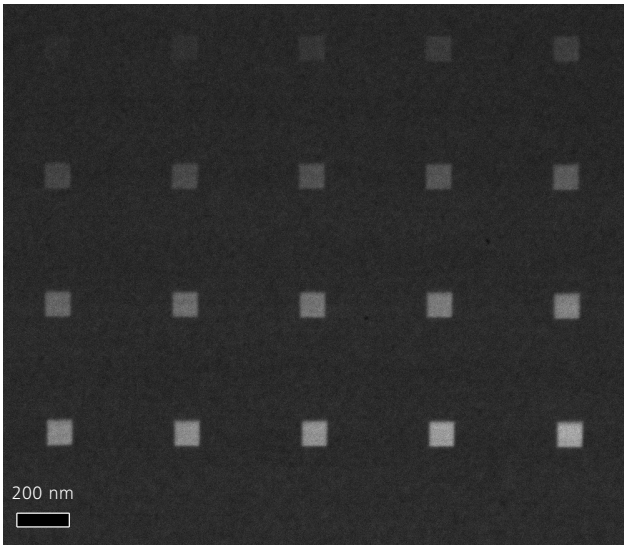

Figure 3 *Square HIM-milled patterns A 4x4 array of milled squares (100 nm) with increasing He+ dose from left to right and from top to bottom. Membrane thickness within the squares ranges from 24 nm (upper left)*

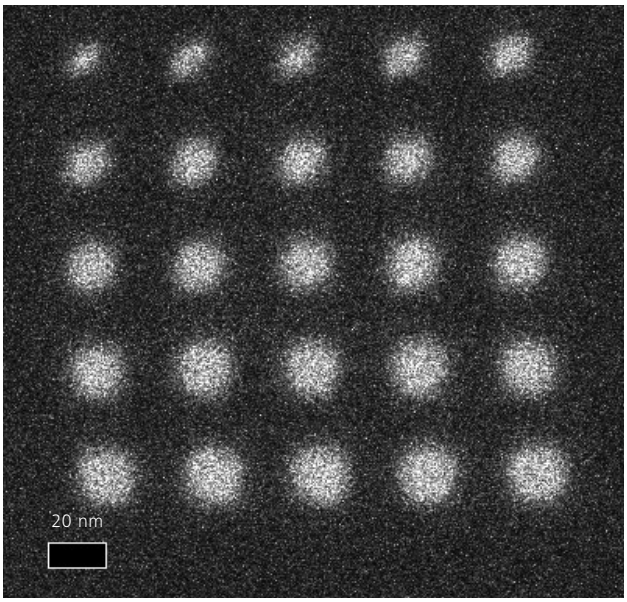

Figure 4 *HIM-milled nanopores An array of nanopores formed by single-point He+ beam exposures for total dwell times that increase from left to right and top to bottom. Scale bar is 20 nm.*

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1. *R. J. Woolf, D. W. Tansley and D. C. Joy, Journal of Physics E-Scientific Instruments 5 (3), 230-& (1972).*
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Carl Zeiss Microscopy GmbH
07745 Jena, Germany
Materials bzw. BioSciences
microscopy@zeiss.com
www.zeiss.com/orion-nanofab

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