Nanopores in Silicon Nitride Membranes, Graphene and CNM: Milling and Imaging Techniques at the Helium Ion Microscope

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The helium ion microscope (HIM) is a charged particle microscope that utilizes helium ions for probing of a sample.[1] In the low dose regime, the HIM operates as microscope, high doses enable material modification and sputtering. Compared to conventional focused ion beams (FIB) using metal ions like gallium, the HIM offers a very small focal spot size down to 0.35 nm and a strongly localized sputter interaction with the material. We employ the HIM for both milling nanopores in free standing membranes as well as for the inspection of nanopores. The helium ion beam with its unique properties overcomes the resolution limit of conventional FIB tools[2] as we show in a comparison with a high resolution gallium FIB. We investigated three different materials: 30 nm thick silicon nitride, graphene and 1 nm thick carbon nanomembranes (CNM) made from aromatic self-assembled monolayers by electron-induced cross-linking[3]. By HIM milling and imaging we can detect smallest nanopores at 3 nm diameter in all membranes. Further studies on CNM with an atomic resolution scanning transmission electron microscope (STEM) revealed even nanopores with diameters of less than 2 nm made by ion beam exposure.

Different strategies for milling and characterizing pores with HIM will be discussed. In particular, we demonstrate two approaches of creating nanopores into membranes of different materials. One approach allows to control the aspect ratio of the pores while the other approach yields smaller diameters. A comparison about the feasibility of different characterization methods includes the ion generated secondary electron signal of HIM, the He^+ transmission signal as well as STEM. Sputter yield values for the investigated membrane types and the He^+ beam profile were obtained from dose-dependent nanopore diameters.

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[1] G. Hlawacek, V. Veligura, R. van Gastel, and B. Poelsema, J. Vac. Sci. Technol. B **32**, 020801 (2014).
[2] H. Kollmann *et al.*, Nano Lett **14**, 4778 (2014).
[3] P. Angelova *et al.*, ACS Nano **7**, 6489 (2013).

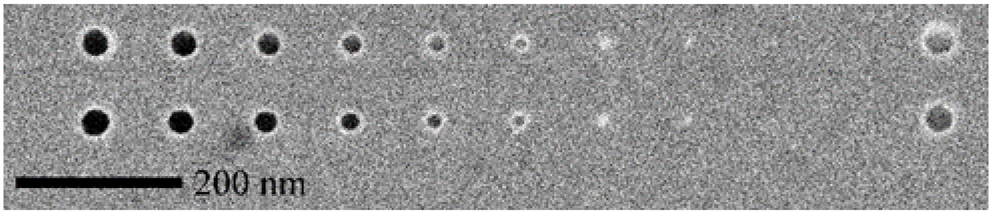

Figure 1: Scanning transmission HIM image of nanopores in a silicon nitride membrane. The pattern consists of circular areas with decreasing diameters from 40 nm (left) down to 2 nm (right) and a final 40 nm circle on the far right as a marker. The ion dose of $2.4 \cdot 10^{18}$ ions/cm² is too low to mill the complete area, so that the 40 nm diameter in the exposure results in a 30 nm pore on the sample.

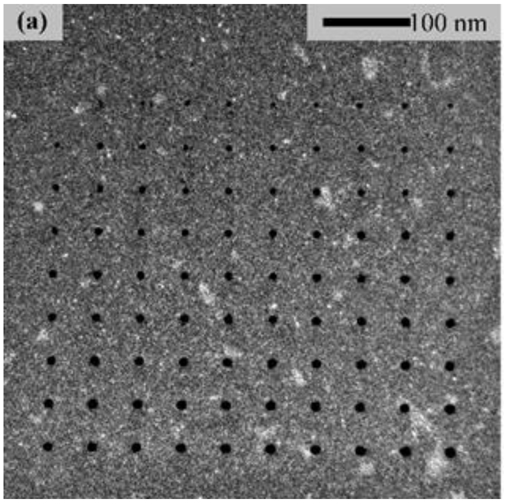

Figure 2: STEM images of nanopores in a CNM. The 10x10 dose array was milled by spot exposure. The shortest exposure time was applied in the upper left corner with increasing dose from left to right and by each line from top to bottom.

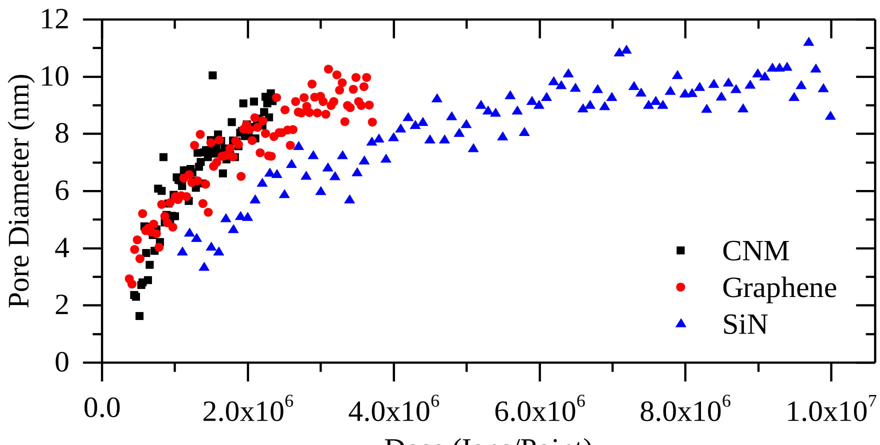

Figure 3: Diameters of nanopores milled with helium ions into different membranes extracted from microscope images. In the case of graphene and silicon nitride the sizes were determined on HIM images, for CNM STEM data were used.