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Atomically Thin Graphene Membrane Technology

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Membranes are used in fluid separation processes such as water desalination, natural gas purification, and carbon capture to filter unwanted components from a mixture. An ideal membrane can provide high purity filtration at high flow rates.[1] Unfortunately, conventional polymer membranes suffer from an inherent tradeoff between flow rate and purity which limits their overall performance. Graphene has been proposed as an alternative membrane material that could overcome current membrane limits.

Being a single atom thick, porous monolayer graphene poses minimal flow resistance and can support holes similar in size to single molecules.[1,2] Molecules smaller than the holes can pass through the membrane while larger molecules cannot, resulting in selective filtration. Graphene is inherently impermeable to gases but can be perforated by methods such as oxygen plasma etching, UV-ozone exposure, and electron beam drilling to allow gas molecules to pass through.[1,2] Measurements on micron-scale areas of graphene have demonstrated high purity filtration while measurements on centimetre-scale areas have shown moderate purity filtration at high flow rates.[1]

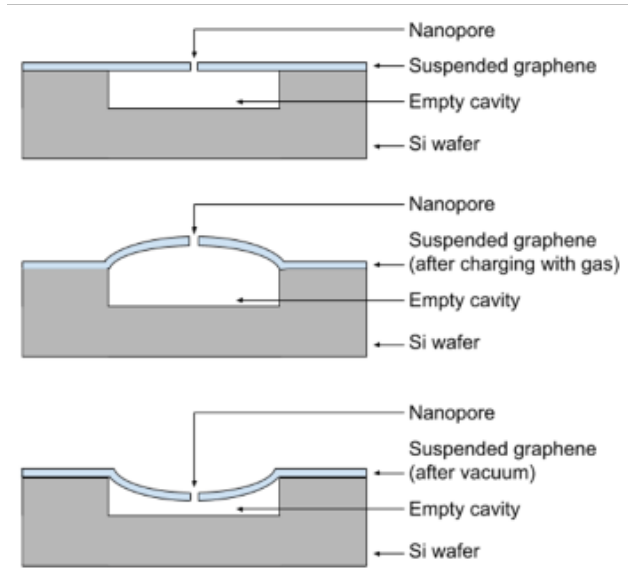

Figure 1: a schematic of suspended graphene over the Si wafer cavity under standard pressure (top), 1 atm pressure (middle), and vacuum (bottom) conditions.

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In this project, we seek to determine the relationship between gas flow rate and sub-nanometer graphene pore geometry. The minuscule flow rates through these pores cannot be determined using conventional fluid measurement tools. Instead, graphene is suspended over a small cavity that is subsequently charged with gas[3] (Figure 1). This causes the graphene to bulge out like a balloon. As gas leaks out of the sub-nanometer pore, the graphene nano-balloon gradually deflates. Flow rates are indirectly measured by monitoring the graphene deflection over time using an atomic force microscope.

At the Western Nanofabrication Facility, we produced a platform to measure flow rates through pores in graphene and other two-dimensional materials. Arrays of circular micro-cavities were patterned on silicon wafers through photolithography. Multiple layers of graphene were individually stacked on the wafer using a polymer-free wet transfer technique. A focused ion beam was then used to drill a 10-30 nm hole through the suspended graphene layers (Figure 2). A final layer of graphene will be transferred onto the wafer and perforated using one of the methods listed previously. This configuration provides a sufficiently large graphene balloon to measure deflection changes as gas escapes. Furthermore, this arrangement limits the available flow area to improve measurement resolution while ensuring that the sub-nanometer pore can be located for additional imaging.

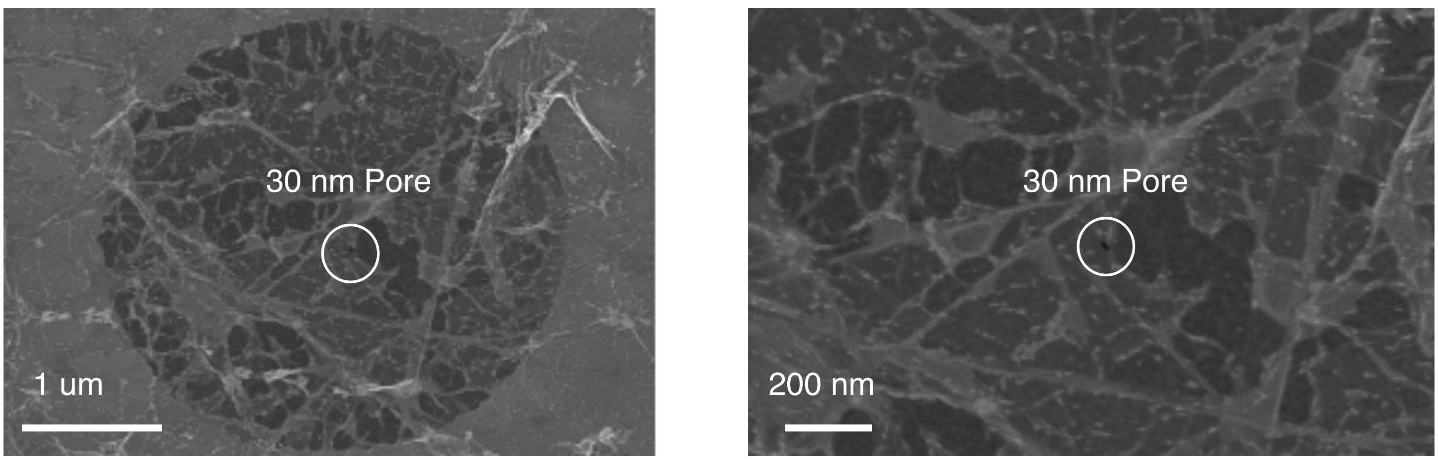

Figure 2: a scanning electron microscope image of suspended graphene over a 5 µm diameter cavity. Circled is a 30 nm hole created with a focused ion beam.

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2. Rollings, R. C., Kuan, A. T., and Golovchenko, J. A. Ion selectivity of graphene nanopores. *Nature Communications* **7**, 11408 (2016).
3. Bunch, J. S. *et al.* Impermeable atomic membranes from graphene sheets. *Nano Letters* **8**, 2458-2463 (2008).

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Creation of New Porous Material via Nanoparticle-Assisted Chemical Etching

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The demand for a more efficient method of air filtration has been growing in recent years due to the expansion of industrial production and increasing world population. One of the possible solutions is the usage of porous and reusable membrane filters with the porous size small enough to filter out undesirable particles. The creation of pores can be achieved by chemical etching of the desired nanoparticles in the media.

In our work, we used polypropylene-graphene (PPG) media with nanoparticles embedded in this composite. The advantage of this system is the enhanced mechanical strength of polypropylene due to the presence of graphene and that by controlling the size and quantity of our nanoparticles we are able to control the porosity of our filter. At first, the commercial SiO_2 nanoparticles (99.99%, Sigma-Aldrich Co.) were used, for the low-cost purposes, in our PPG composite. These nanoparticles were further etched with hydrofluoric acid. The samples before and after etching were characterized by scanning electron microscopy (SEM) at the Western Nanofabrication Facility (WNF) in Figure 1.

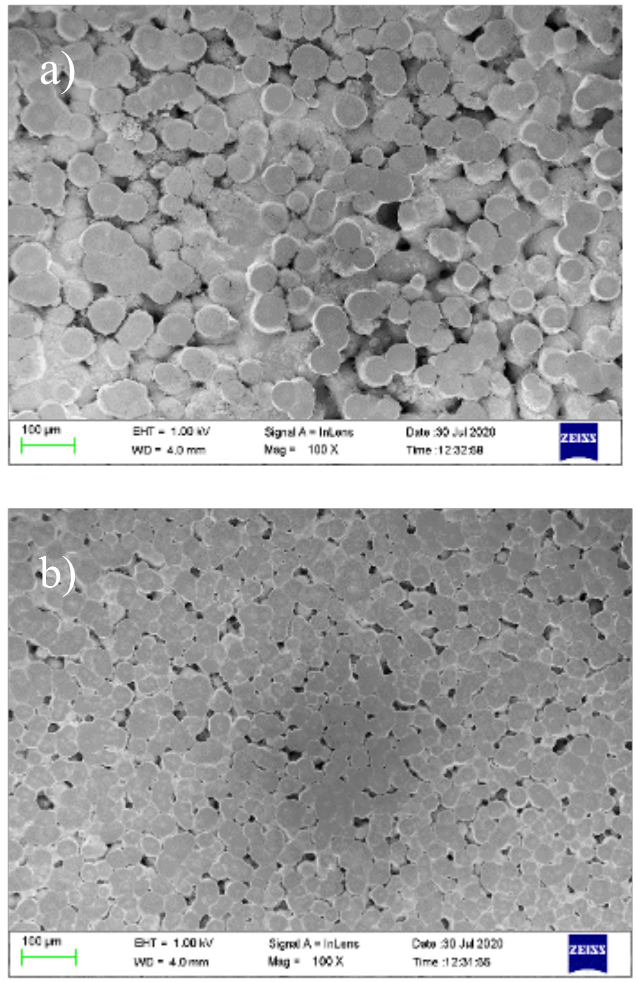

Figure 1: a) PPG with SiO2 nanoparticles
b) PPG after etching SiO2 nanoparticles

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For an ecologically and commercially better way of pore creation, metallic tin nanoparticles were proposed for further usage. To synthesize these nanoparticles tin (ii) acetate (Sigma-Aldrich Co.) was used as a precursor for the synthesis via chemical reduction method. [1] The achieved nanoparticles were further characterized with SEM and energy dispersive x-ray microscopy (EDX) at the WNF, results are shown in Figure 2. Our future work includes the creation of tin nanoparticles in an inert atmosphere to reduce their oxidation during the synthesis process. They will be implemented into the polypropylene-graphene composite to replace commercial SiO_2-nanoparticles, as a much safer and cost-effective way of creating pores.

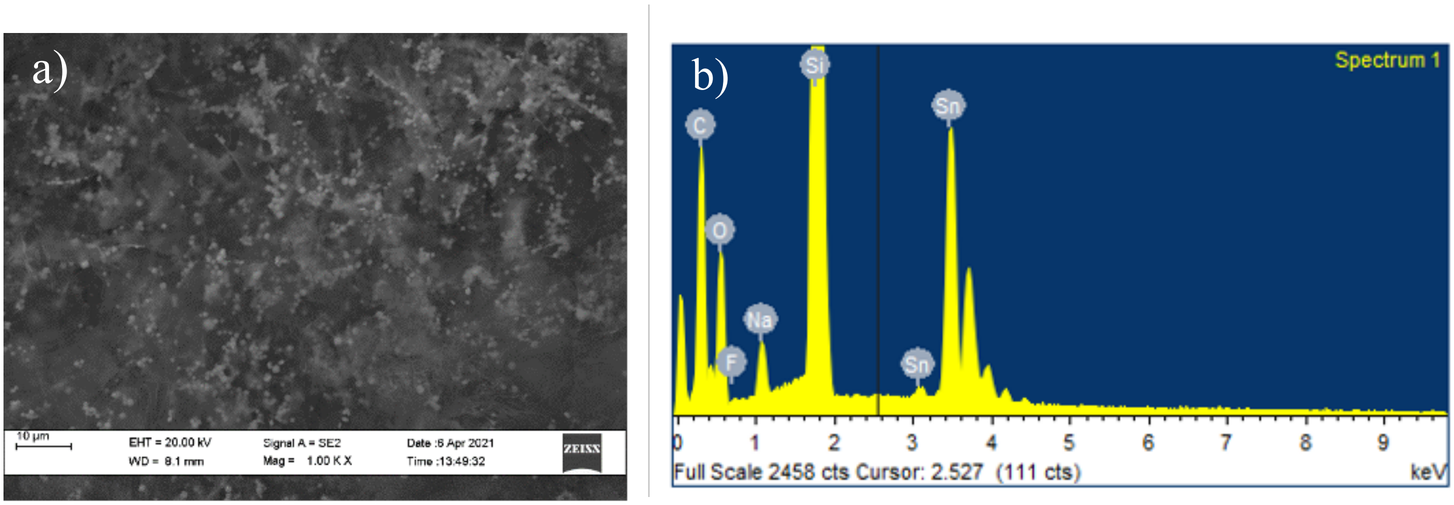

Figure 2: a) Synthesized tin nanoparticles surrounded by dodecylamine; b) EDX spectrum of the tin nanoparticle.

References:

1. Chee, S.-S., Lee, J.-H. (2012). Reduction synthesis of tin nanoparticles using various precursors and melting behavior. Electronic Materials Letters, 8(6), 587-593. doi:10.1007/s13391-012-2086-y

The Western Nanofabrication Facility is a professionally staffed cleanroom designed to support education, research and industrial collaboration in the fabrication and characterization of structures and devices of nano and sub-micron scale. The Nanofab is a user-fee supported facility. It is open to academic, government and industrial users. The Nanofab is a "hands-on" facility where users are trained and supervised on the use of equipment and processes. Analytical and processing services are also available.

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