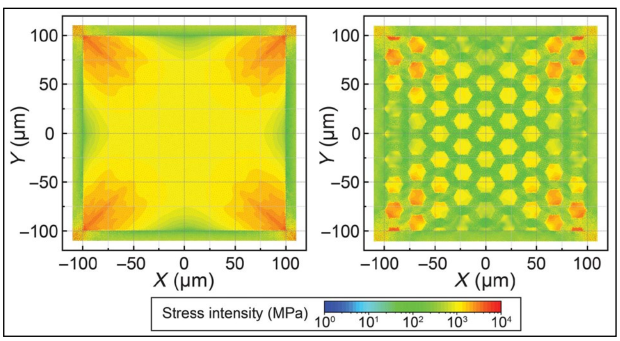
Closed-cell ultrathin microchip design elevates *in situ/operando* electron microscopy

In situ transmission electron microscopy (TEM) is a powerful tool that allows researchers to examine materials at nanoscale resolution, in real-world conditions, in real time. In the most common form of closed-cell *in situ* TEM, a sample is sandwiched between a pair of membranes in the specimen rod in a gas or liquid medium that keeps the sample isolated at the selected pressure. However, these membranes also tend to overzealously scatter incoming electrons, which is a detriment to the achievable resolution and limits the ability to monitor nanoparticles in action. Now, Kunmo Koo and Vinayak Dravid of Northwestern University and colleagues have crafted a thinner membrane backed by a honeycomb-shaped support structure, allowing it to retain the sturdiness of a thicker, more traditional membrane.

As reported in a recent issue of *Science Advances* (https://doi.org/10.1126/sciadv.adj6417), the research team worked with silicon nitride, a material already used to construct such membranes. Typically, *in situ* TEM relies on silicon nitride membranes that are about 50-nm thick. Researchers want a thinner membrane, but making a flat layer of silicon nitride much thinner than 50 nm comes at grave risk to the membrane's structural stability. If the membrane perforates, the experiment is ruined.



Finite element analysis of stress intensity at a 1-atm pressure gradient for the conventional unsupported SiN_x membrane with a thickness of 50 nm (left) and an ultrathin 10-nm SiN_x membrane with a 1-μm Si supporting structure (right). The window size is 200 μm × 200 μm. Credit: *Science Advances.*

Instead of a single, flat membrane, Koo and colleagues created a membrane with two major layers. Their key addition was a honeycomb-shaped support structure, crafted from boron-doped silicon, that was about a micrometer thick. They could then glaze the structure's hexagonal holes by depositing a layer of silicon nitride. This process resulted in an array of silicon nitride "windows," each as thin as 10 nm.

When the researchers put their new membrane to a gauntlet of deflection tests, it held up to pressure better than its thicker, traditional counterpart. The cells yielded significantly improved figures of merit in resolution, diffraction, and spectral visibility, coming close to the primary microscope specifications.

"The new design introduced in the paper significantly reduces this thickness while maintaining the mechanical integrity, thus offering potentially noticeably improved data quality," says Christian Kübel, professor of *in situ* electron microscopy at the Karlsruhe Institute of Technology, Germany, who was not involved in the research. "Thus, this holds a lot of promise to improve *in situ* TEM studies."

The same sort of structure could also be fitted into other types of microstructures, in applications outside *in situ* TEM, the researchers say.

Rahul Rao