Sensing the Invisible: Ultrathin (UT) Membrane Chip for In-Situ Microscopy

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Background incl. aims

Operando or in-situ S/TEM methods utilizing amorphous silicon nitride (SiNx) membrane encapsulated chips to confine fluids for electron microscopy have become popular in recent decade, A great number of prior innovators have shown this to be an effective approach for probing fluid-surface/nanostructure interactions and related phenomena. Such “closed-cell” platform has many practical and technological advantages over the differential pumping environmental TEM (ETEM).

Unfortunately, however, conventional fluid-cells suffer from additional and significant electron scattering from the top and the bottom membranes, which are typically 30-50nm thick to maintain integrity/stability during the operation. Thus, the total thickness of >60-100 nm of the encapsulating membranes imposes many adverse effects on the post electron optics, such as increased chromatic aberrations. This naturally results in significant degeneration of signal quality and loss of spatiotemporal resolution, diffuse interference in the electron diffraction, and plasmon-dominated electron energy loss spectra (EELS).

Methods

We have recently reported development of a robust, functional and scalable backing support strategy to enable the thinnest possible (<10 nm) SiNx gas encapsulation material [1, 2]. Inspired by the natural honeycomb geometry, our novel design provides for hexagonal backbone that can neatly anchor ultrathin (~<10 nm) SiNx membrane with excellent stability and consistent performance. Unlike graphene-based encapsulations, stability under the electron beam is comparable to a 50 nm SiNx membrane, which is sufficient for most high-resolution S/TEM applications on non-electron sensitive materials.

Results

We show that our UT chip increases contrast of typical nanoparticles at 1 atm Ar gas by ~70 % and the accessible information limit is enhanced by >130 % compared to the conventional encapsulation. More importantly, the t/λ_i is reduced from nominally ~1.0 to 0.3 using a 1 Atm gas cell. This greatly enhances spectral visibility and significantly improved S/N for EELS excitations. Thus, spatiotemporal detection of gas species, down to ~nanometer scale is now being routinely achieved.

Conclusions

The presentation will cover the design and implementation of UT membrane fluid-cell for in-situ gas-solid interactions. It will also argue that combining monochromatic source with UT membrane may open new opportunities for molecular-scale understanding of dynamic fluid-surface phenomena. [3]

Keywords:

Ultrathin (UT) membrane, in-situ microscopy

Reference:

References: [1] K Koo, SM Ribet, C Zhang, PJM Smeets, Rd Reis, X Hu, and VP Dravid, Nano Lett 22 (2022), p. 4137. doi: 10.1021/acs.nanolett.2c00893; [2] VP Dravid, X Hu, and K Koo, US Provisional Patent, No. 63413097 (2022); Science Advances, 2024: DOI: 10.1126/sciadv.adj6417

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