Free-standing ultrathin metal-oxide membranes as electron transparent windows for studying gas-solid and liquid-solid interfaces at high pressures

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Transitioning to a clean, renewable energy supply has become increasingly urgent to secure humankind's future. In recent decades, great efforts have been devoted to developing highly efficient and low-cost catalysts to produce green energy carriers as substitutes for fossil fuels, such as H_2 or carbon-based synthetic fuels. In this race, tailoring a particular process's efficiency and selectivity requires using and optimizing a specific catalyst. For this, phase, morphology, stability, and fundamental interactions with the reactants and products at operation conditions, particularly at the gas/solid or liquid/solid interfaces, must be known. However, this rational, bottom-up approach has become challenging, and historical development has relied heavily on trial-and-error-based research. Particularly, constraints in the experimental methods available to characterize gas/solid and liquid/solid interfaces limit most studies to relatively low pressures, far from operating conditions in industrial processes.

Thus, significant efforts have been dedicated to developing new methods to study catalysts under realistic conditions. In the last decade, electron-transparent graphene windows have been used in ambient pressure X-ray photoelectron spectroscopy (AP-XPS) to separate liquids and gases at ambient pressure from a high vacuum [1]. Few more experimental works have followed this design to substitute 2D materials with ultrathin metal oxides [2] and SiN_x [3] free-standing layers. Following these pioneering works, we present free-standing, ultrathin Al_2O_3 (up to 10 nm) membranes fabricated by atomic layer deposition (ALD) on low-cost supports with varying aperture dimensions: (i) TEM SiN perforated membranes (⌀ = 1 µm), (ii) single-hole stainless steal apertures ((⌀ = 20 µm), and (iii) TEM Cu-grids ((⌀ = 80 µm). Their continuity has been examined by scanning electron microscopy (SEM) and atomic force microscopy (AFM), whereas their chemical composition and homogeneity by energy dispersive X-ray (EDX) mapping. Additionally, confocal µ-Raman microscopy complements the chemical and structural characterization. These 10 nm thick Al_2O_3 membranes supported on 20 µm apertures can withstand 1 bar of pressure difference for more than 24 hours.

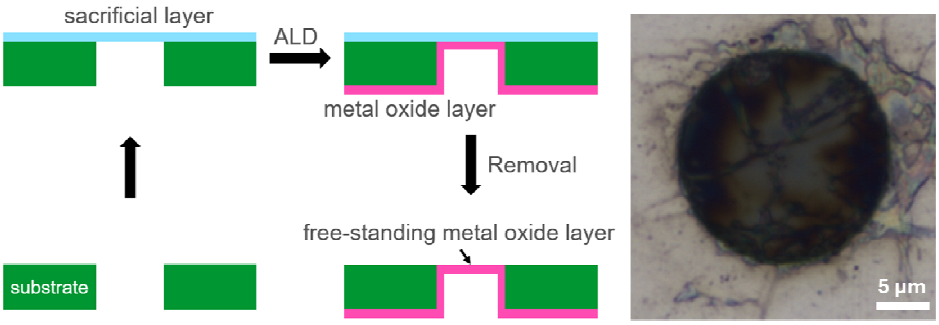

Figure. Left: frabitation steps of free-standing, ultrathin ALD membranes. Right: optical image of a ⌀=20 µm aperture with 10 nm thick Al_2O_3 free-standing film.

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