Tuning Nanopores in Tubular Ceramic Nanofiltration Membranes with Atmospheric-Pressure Atomic Layer Deposition: Prospects for Pressure-Based In-Line Monitoring of Pore Narrowing

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Abstract

Atomic layer deposition (ALD) is known for its unparalleled control over layer thickness and 3D conformality and could be the future technique of choice to tailor the pore size of ceramic nanofiltration membranes [1]. However, a major challenge in tuning and functionalizing a multichannel ceramic membrane is posed by its large internal pore volume, which needs to be evacuated during ALD cycling. This may require significant energy and processing time. This study presents a new reactor design, operating at atmospheric pressure, that is able to deposit thin layers in the pores of ceramic membranes. The flow diagram is shown in Figure 1.

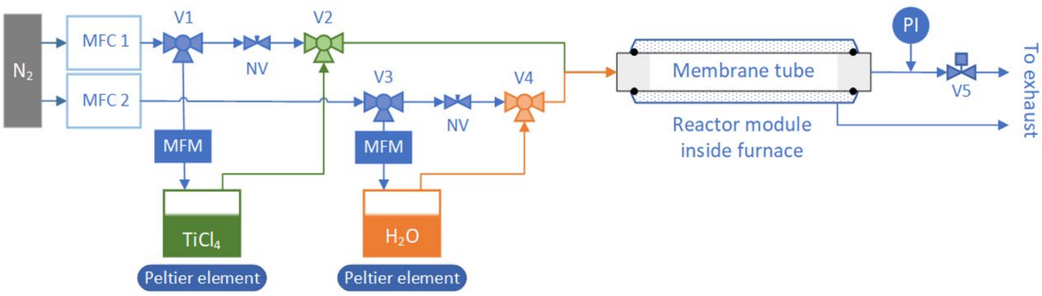

Figure 1. Schematic drawing of gas lines in the atmospheric-pressure ALD reactor. The grey areas on the membrane tube indicate the approximate length of the glass seals on the inside and outside of the membrane tube. The black dots indicate the positioning of the O-rings providing the seal between the feed/permeate side of the membrane. The dotted area around the membrane tube represents the annular volume in the stainless-steel module, where a nitrogen flow takes away eventually permeated precursors and reaction products. The lengths of the membrane tube and reactor module are adaptable between 5 and 50 cm. In the lines behind the reactor, a pressure indicator (PI) and a valve (V5) are installed for the in-line permeance tests.

In this design, the reactor wall is formed by the industrial tubular ceramic membrane itself, and carrier gas flows are employed to transport the precursor and co-reactant vapours to the reactive surface groups present on the membrane surface. The layer growth for atmospheric-pressure ALD in this case proceeds similarly to that for state-of-the-art vacuum-based ALD. Moreover, for membrane preparation, this new reactor design has three advantages: (i) monolayers are deposited only at the outer pore mouths rather than in the entire bulk of the porous membrane substrate, resulting in reduced flow resistances for liquid permeation; (ii) an in-line gas permeation method was developed to follow the layer growth in the pores during the deposition process, allowing more precise control over the finished membrane; and (iii) expensive vacuum components and cleanroom environment are eliminated. This opens up a new avenue for ceramic membrane development with nano-scale precision using ALD at atmospheric pressure.

The work shown in this abstract has been published recently in the Separations special issue “Research Progress in Nanomaterials and Processes for Separation Membranes”. https://doi.org/10.3390/separations11010024

1. Chen, M.; Nijboer, M.P.; Kovalgin, A.Y.; Nijmeijer, A.; Roozeboom, F.; Luiten-Olieman, M.W.J. Atmospheric-Pressure Atomic Layer Deposition: Recent Applications and New Emerging Applications in High-Porosity/3D Materials. *Dalton Transactions* **2023**, doi:10.1039/d3dt01204b.