"Novel Nano-Engineered Thin Film Composite Membranes for Seawater and Brackish Water Desalination"

Subcontract from DE-EE0005771
University of Colorado / GE Global Research
1/24/18-12/31/18

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Overview

Timeline

- Continuation Award issued Jan. 2018
- Projected End date Dec. 2018
- Project 50% complete

Project Focus

- Continuation award focused on solving difficulties when molecular layer deposition (MLD) films are deposited on porous supports.

Budget

		Base Period (8.25 months)	Option Period (2.75 months)	Total Planned Funding
DOE Funded		$75,000	$25,000	$100,000
Project Cost Share		$16,721	$8,080	$24,667

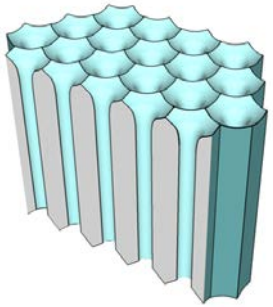
History & Goal

- Original award to GE was a collaboration between GE, the University of Colorado and NIST.
- Continuation award to the University of Colorado now managed by GE.
- University of Colorado goal was to develop spatial MLD methods to deposit & test ultrathin polyamide films for desalination membranes.

Objectives

4 Goals for Enabling Polyamide MLD Films for Desalination Membranes

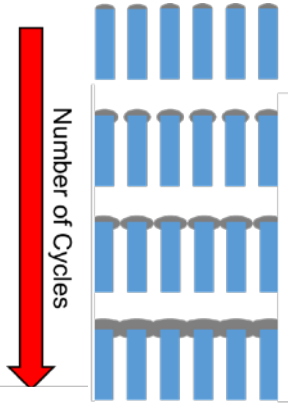
1. **Preventing Pore Collapse with Heating in Vacuum**



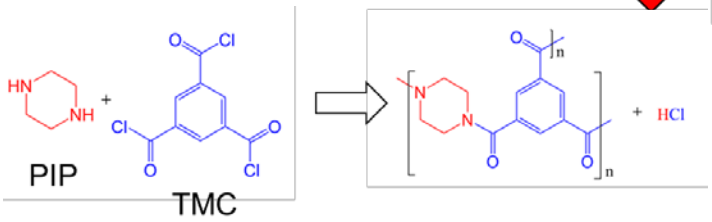
Use anodic aluminum oxide (AAO) membranes to test fabrication concepts

2. **Pore Sealing to Prevent MLD in Pores of Porous Support**

Use plasma atomic layer deposition (ALD) to cap pores

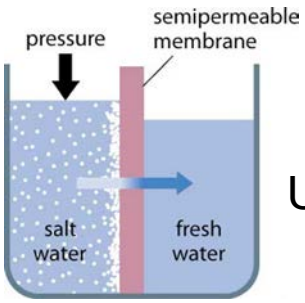


3. **Controlling Cross-Linking of the MLD Film**



Use different amine precursors to tune cross-linking

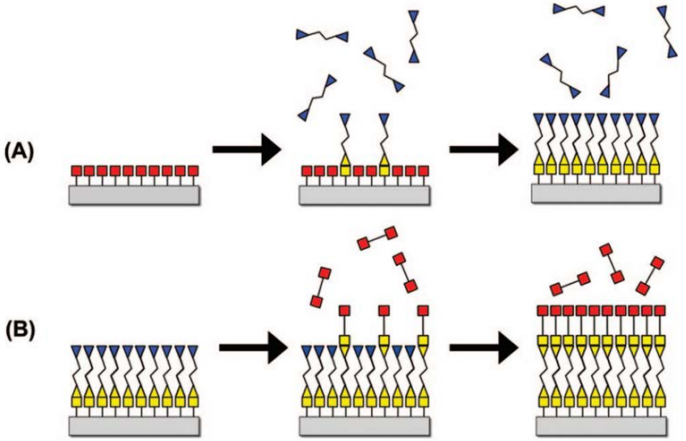
4. **Testing & Optimization of Membranes**



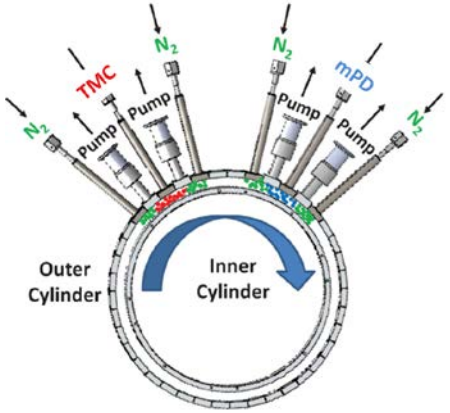
Use dead-end cell for desalination testing

Technical Innovation

- Current polyamide desalination membranes are formed using interfacial polymerization methods. Poor control over film thickness
- Our approach is based on molecular layer deposition (MLD). MLD is gas phase process based on self-limiting surface reactions that yields Ångstom-level thickness control

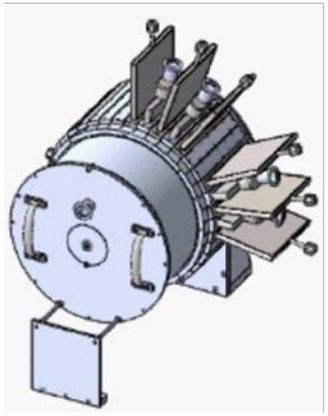


- MLD is implemented using spatial MLD reactor where substrate moves through spatially separated continuously flowing reactants

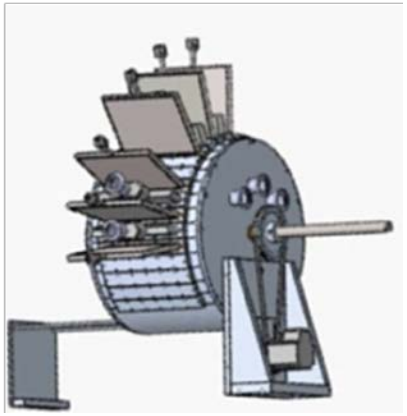


- Spatial MLD in rotating cylinder reactor is 10-100 times faster than temporal MLD

Front View

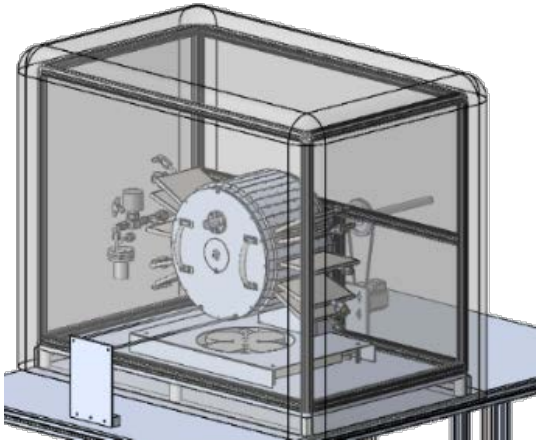


Back View

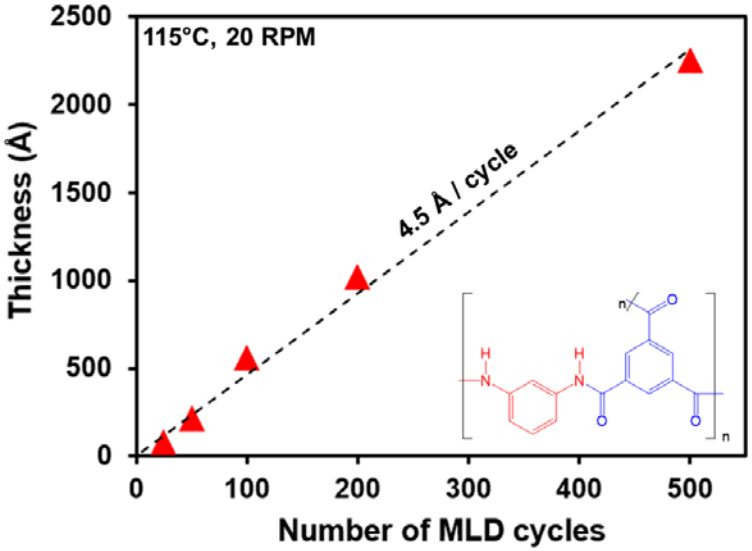


- Have developed new spatial MLD reactor with adjustable placement of precursor, purge and pumping modules

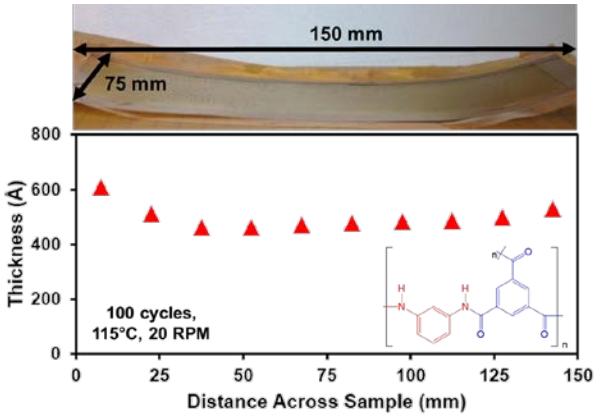
Technical Innovation



- Have built spatial MLD reactor and reactants in isothermal enclosure to avoid cold spots that lead to chemical vapor deposition (CVD)

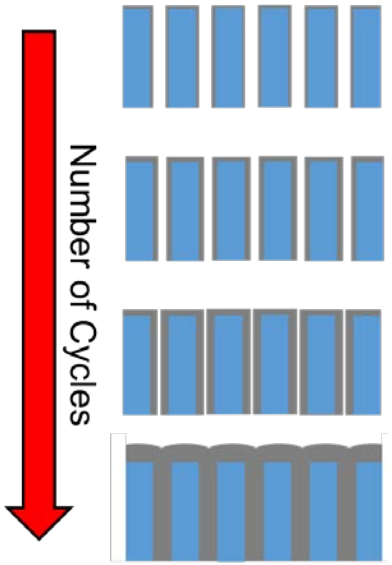


- Can control linear growth of polyamide MLD film with growth rate of 4.5 Å per MLD cycle using mPD & TMC



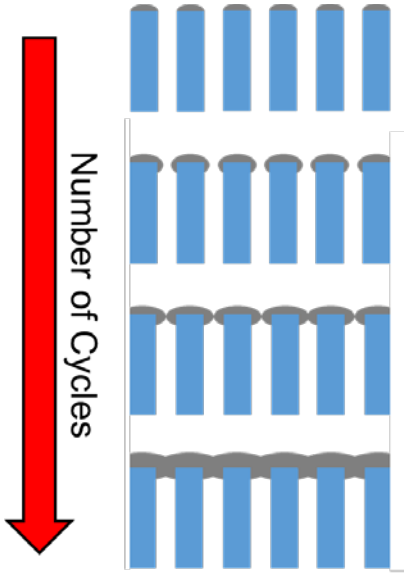
- Can obtain excellent uniformity in MLD film thickness over samples that are 150 mm x 1 m

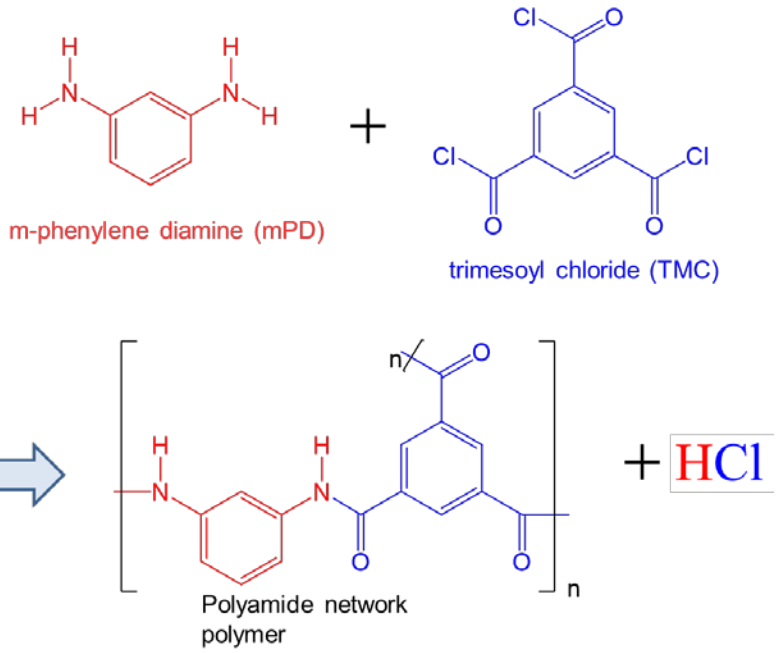
Technical Approach



- *Problem encountered in early work:* MLD film on porous support blocks pores

- *Proposed solution:* Cap surface of porous support before MLD using Al_2O_3 plasma atomic layer deposition (P-ALD)





- Deposit polyamide MLD film using m-phenylene diamine (mPD) and trimesoyl chloride (TMC). This polyamide film is proven desalination membrane

Technical Approach

Fabrication Strategy

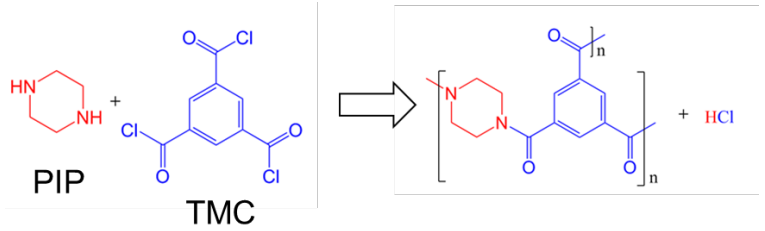
Starting porous support (anodic aluminum oxide (AAO))

Al_2O_3 P-ALD on AAO for pore capping

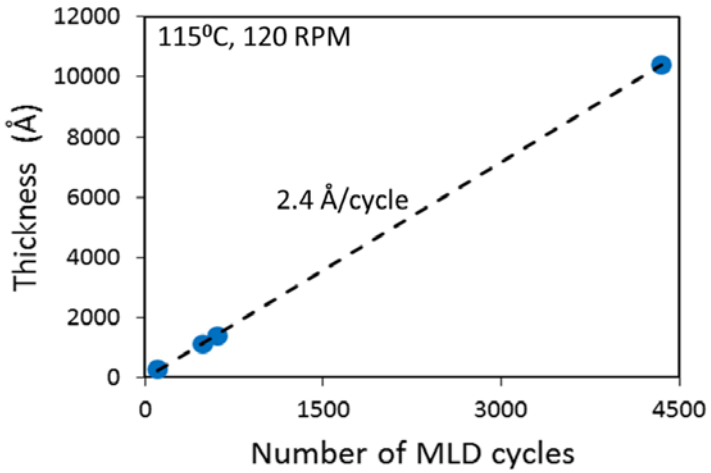
Polyamide MLD on Al_2O_3 P-ALD capping layer

Back-etching of Al_2O_3 P-ALD capping layer for permeable support

Control polyamide cross-linking to tune permeance



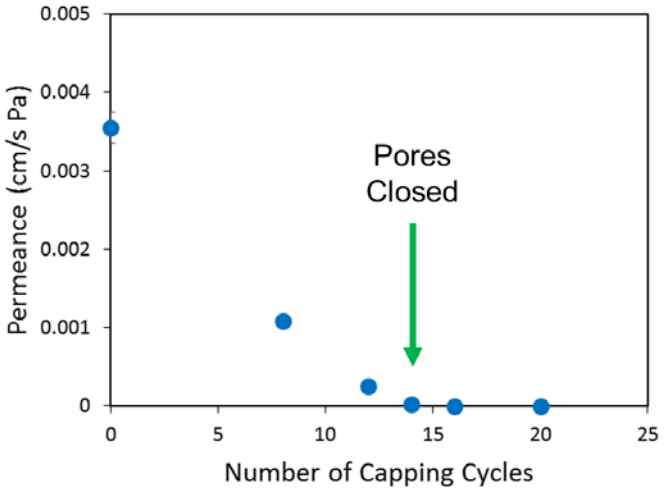
Use piperazine (PIP) instead of TMC for polyamide MLD



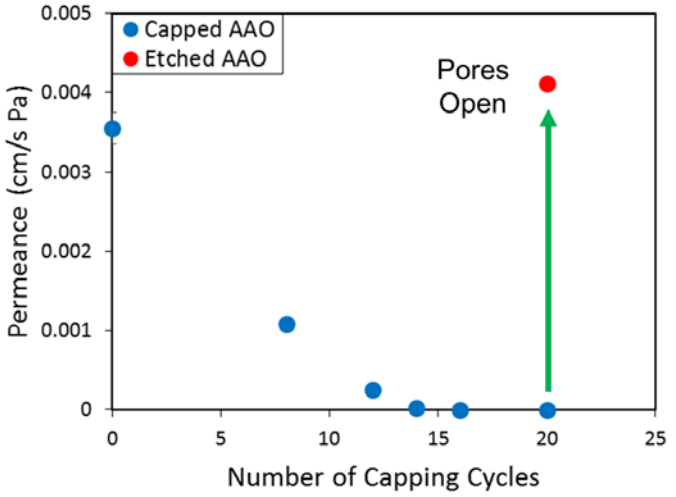
Linear growth of polyamide MLD film using PIP with growth rate of 2.4 Å/cycle

Results and Accomplishments

- Can prevent pore collapse by using AAO membranes. Once strategy is confirmed using AAO membranes can transition to polysulfone membranes.
- Have demonstrated pore sealing of AAO membranes using Al_2O_3 plasma ALD capping layer
- Can back-etch the Al_2O_3 plasma ALD capping layer to regain membrane permeance
- Have demonstrated polyamide MLD film growth using both mPD & PIP. Still need to show that mixtures of mPD & PIP can control film cross-linking
- Before the end of the project, need to test the polyamide MLD membrane for desalination performance



Pores closed after 14 P-ALD capping cycles at Al_2O_3 thickness of 32 Å



Pores open after back-etching using NaOH solution

Transition

- For future technology development and transition to commercial marketplace, need to demonstrate improved performance of polyamide MLD membranes compared with commercial MLD membranes
- Hopefully, improved performance (excellent desalination with high H_2O permeance) will attract the interest of companies
- Commercialization will require large format polyamide MLD using roll-to-roll reactors at reasonable web speeds
- Roll-to-roll ALD has been recently demonstrated using a spatial ALD rotating cylinder reactor similar to rotating cylinder reactor used for polyamide MLD

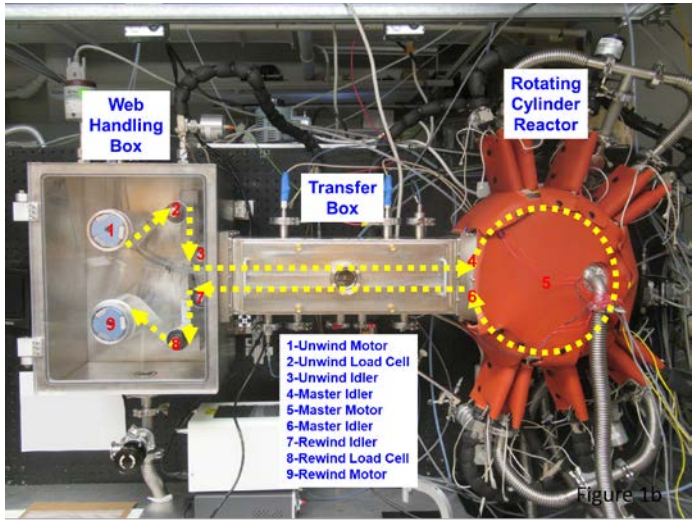


Figure 1b

- Roll-to-roll ALD reactor composed of rotating cylinder reactor, transfer box and web handling box

Questions?