Nano-scale coating on nanofibers using Spatial Atomic Layer Deposition

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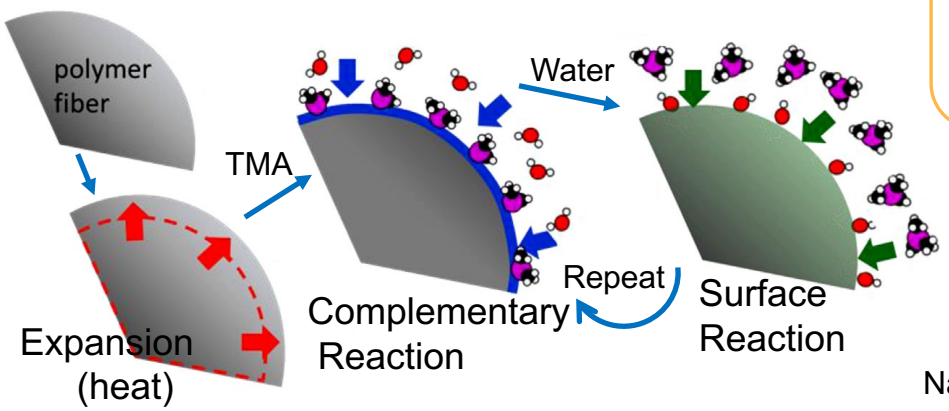
Outline

1. Introduction – Atomic Layer Deposition
2. Objective
3. Sample preparation – Electrospinning and ALD
4. Results – XPS, SEM, and solubility
5. Summary

Atomic Layer Deposition (ALD)

■ The schematic process of ALD



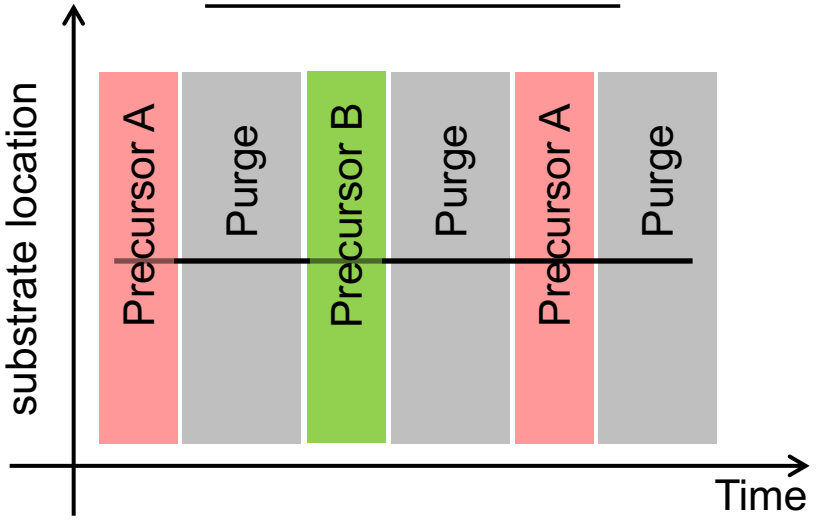
Surface coating or modification process to be uniform with metal oxides

for polymer

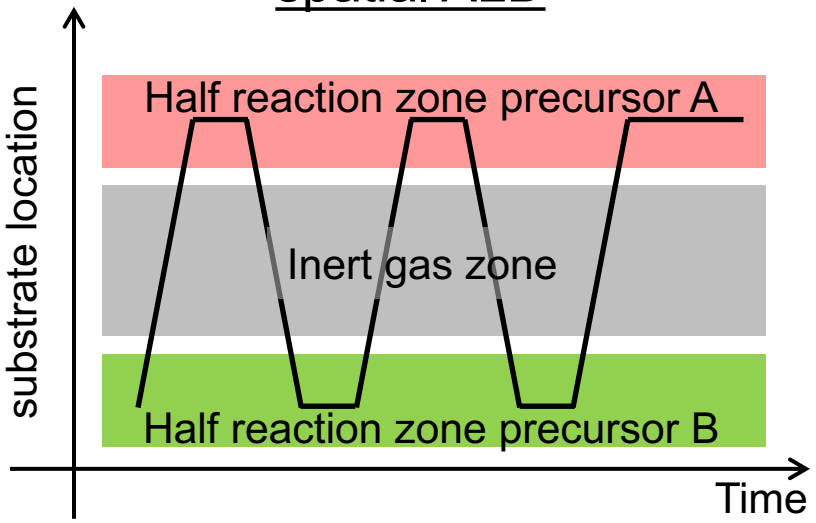
- to improve barrier properties
- to modify surface wetting…

Nancy A. Vogel et al., *Adv. Mater. Interfaces*, **2**, 1500229 (2015).

conventional ALD



spatial ALD



Difference in how to deliver the precursors to the substrate

P. Poodt et. al., *J. Sci. Technol. A: Vacuum, Surfaces, and Films* **30,** 010802 (2012).

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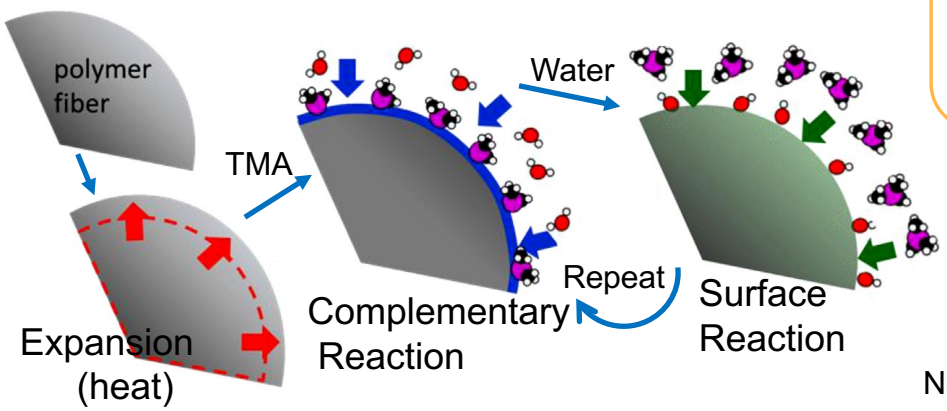

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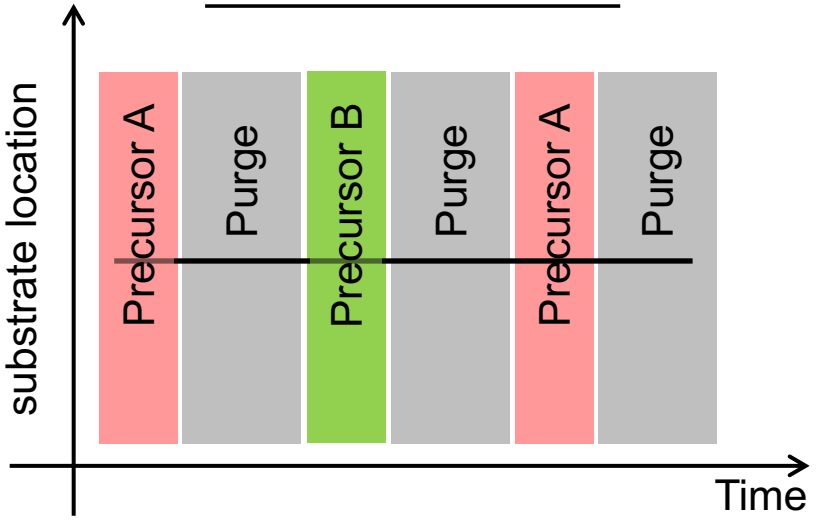

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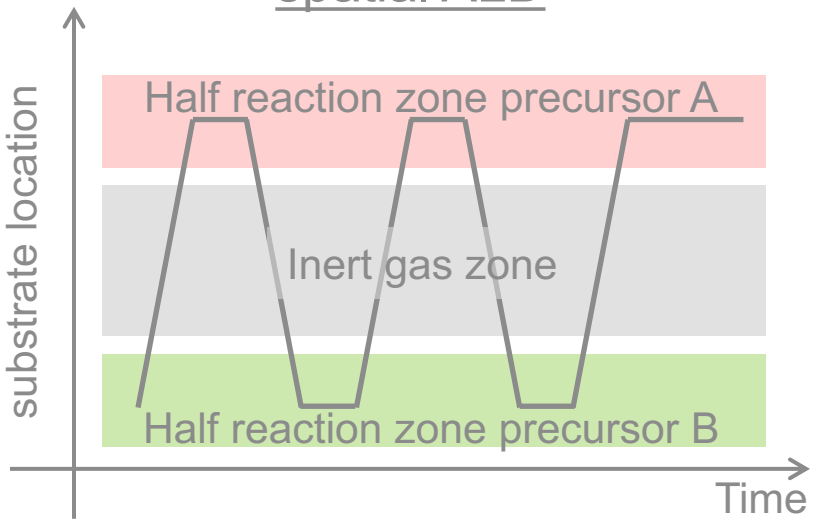

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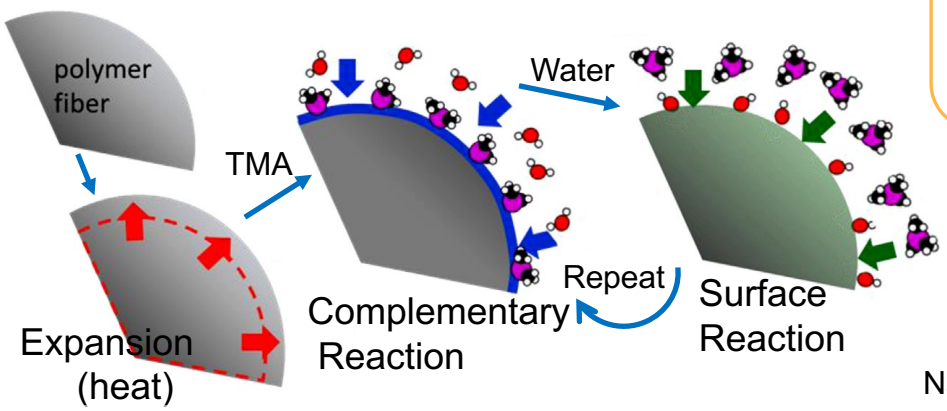

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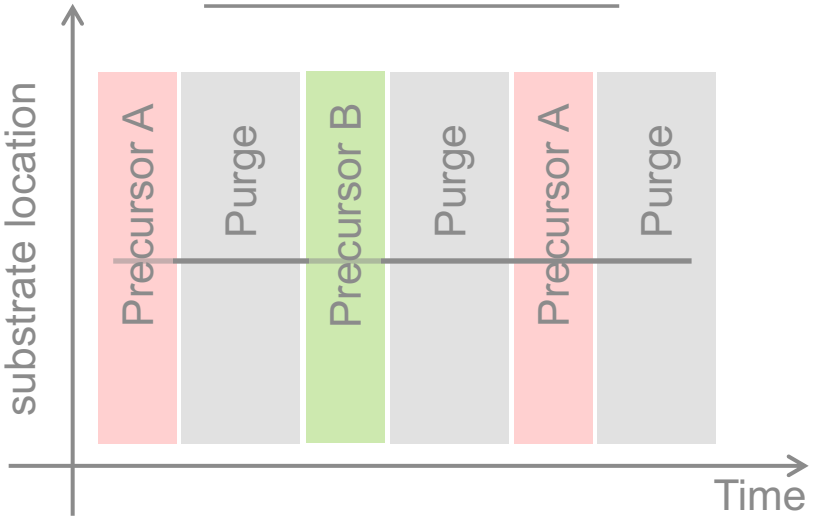
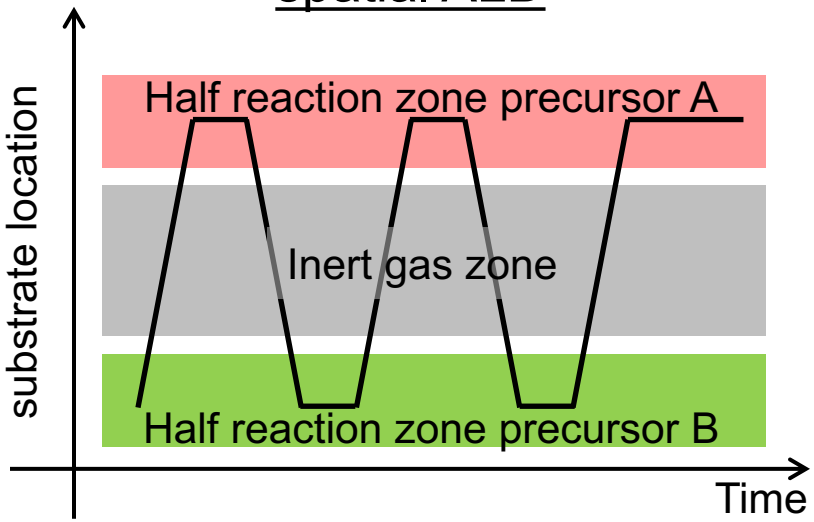


spatial ALD



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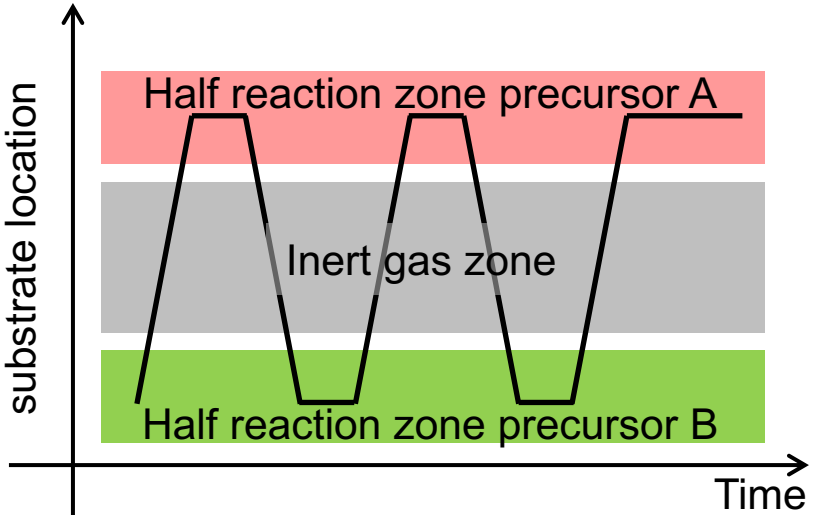
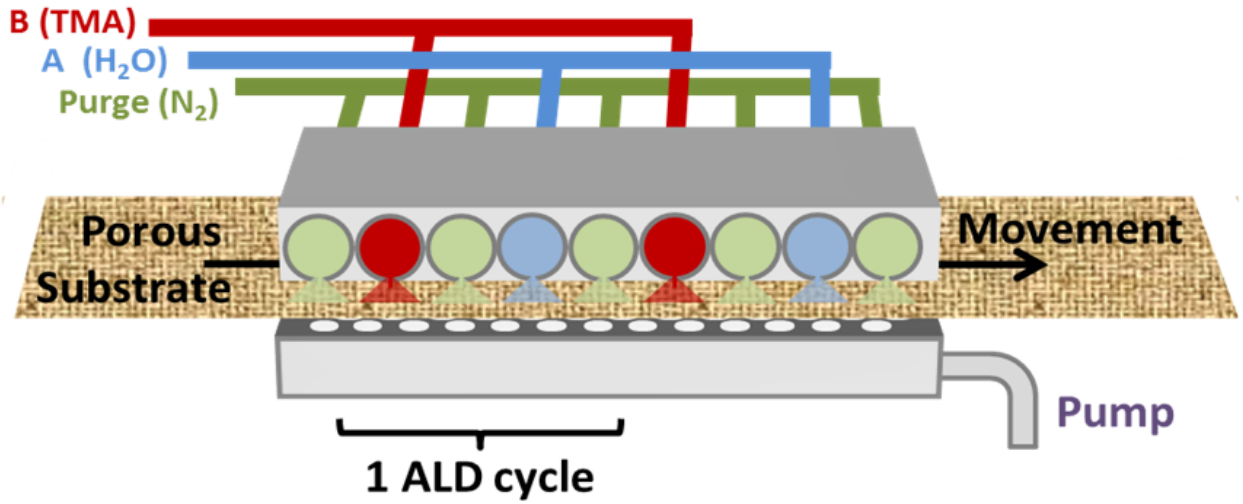
Advantage of Spatial ALD



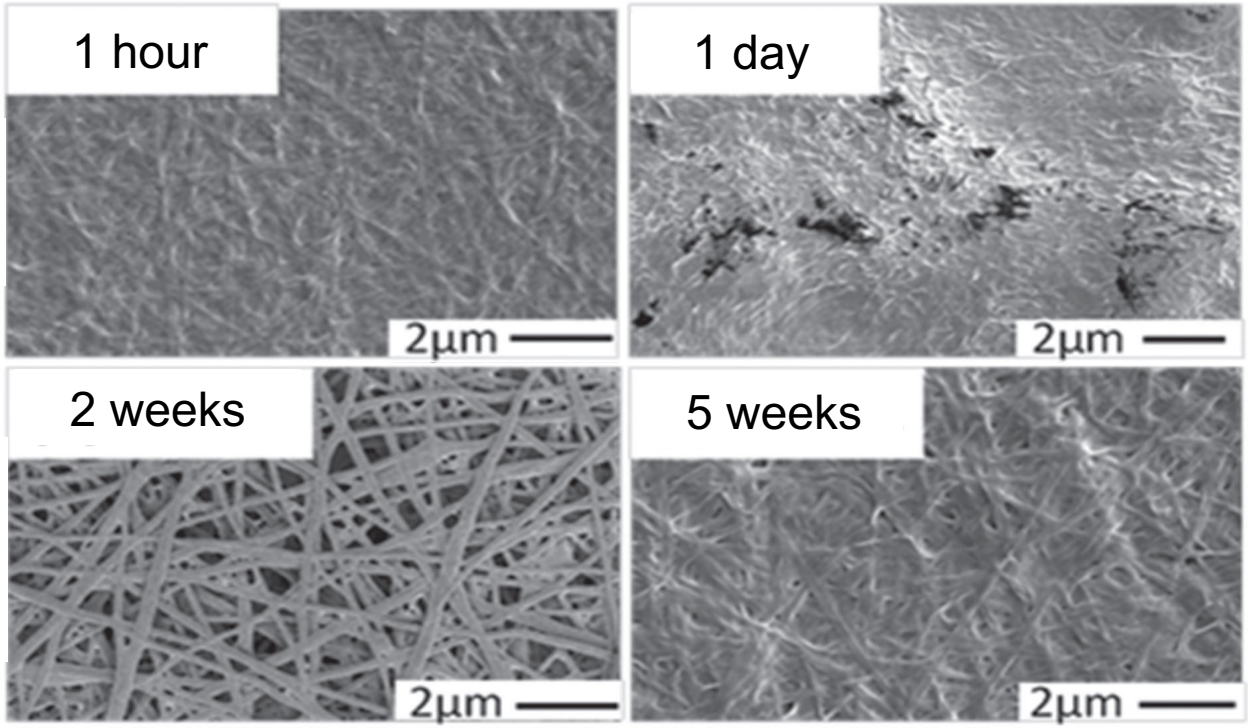
- ✓ Roll-to-roll process
- ✓ No vacuum needed
- ✓ Treat larger sample

Schematic illustration of the reactor



Advantage of ALD modification for polymer

- Scanning electron micrographs of electrospun poly (vinyl alcohol) mats treated with conventional ALD and soaked in water



With increasing
the number of ALD cycles

Extending mat dissolution

(from minutes to weeks)

Al_2O_3 coatings on
the nanofiber surface

↓

an effective
surface modification

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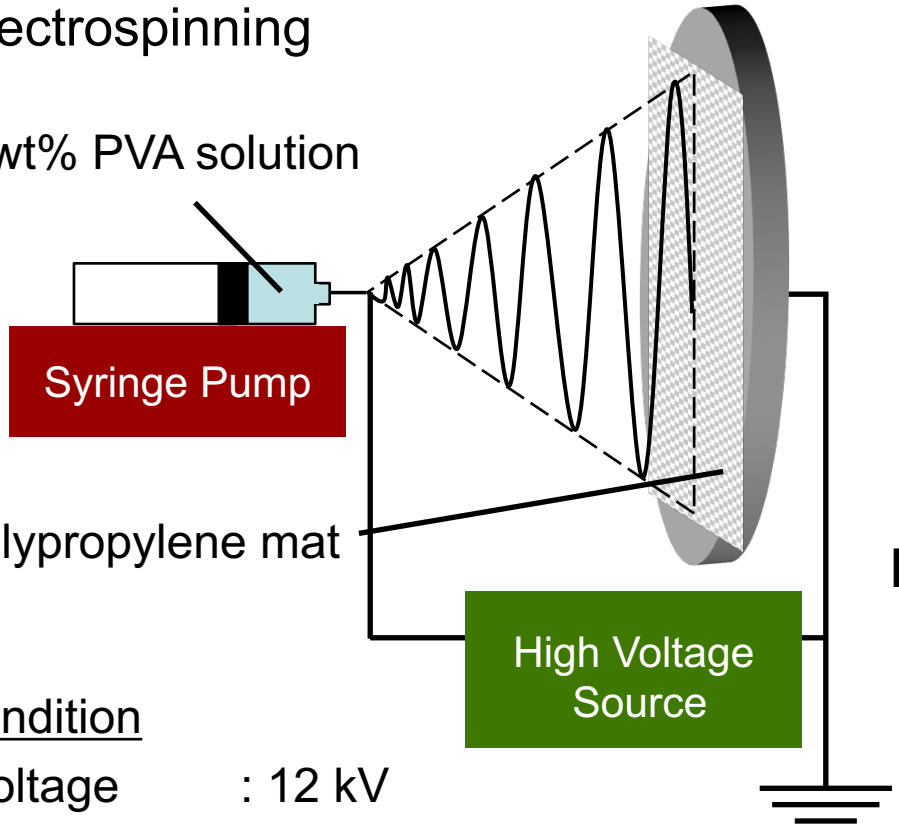
Objective

To characterize an effect of the number of spatial ALD cycles on water stability for electrospun PVA nanofiber

Growth of nanofiber on polypropylene mat

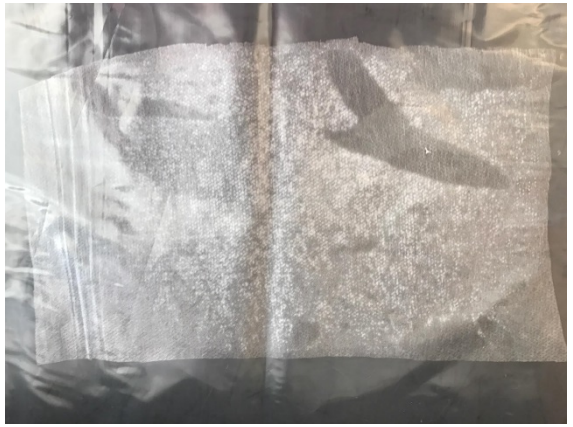
■ Schematic illustration of electrospinning



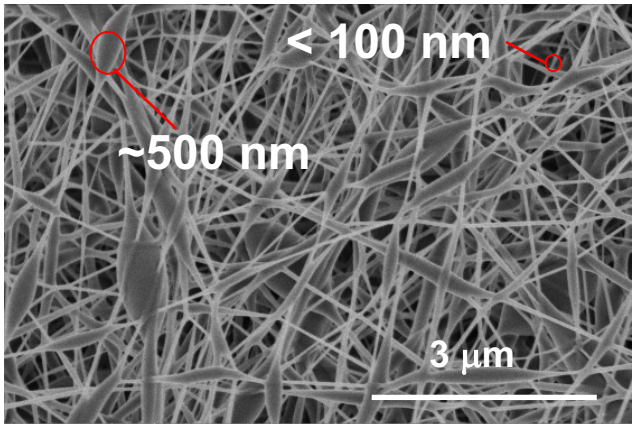
Condition

Voltage	: 12 kV
Flow rate	: 0.5 mL h^{-1}
Time	: 1 h
Temperature	: 22 °C
Humidity	: 30 %

□ Photograph of obtained mat

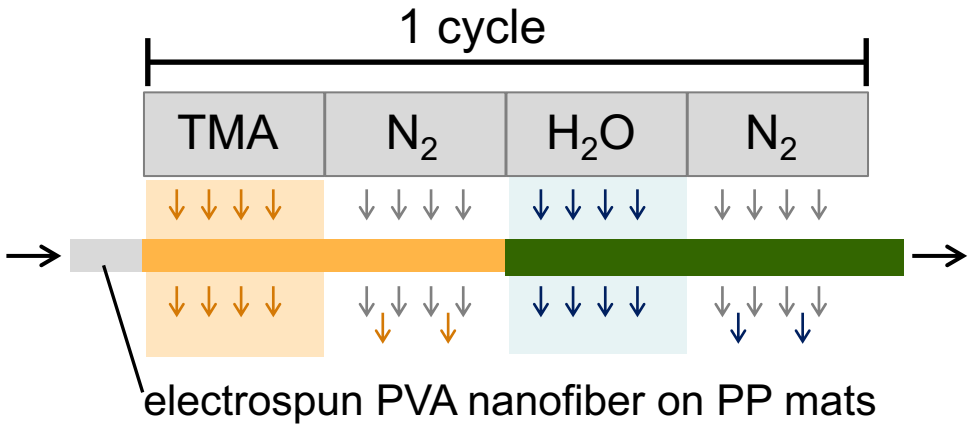


□ SEM image of PVA nanofiber



Materials and method

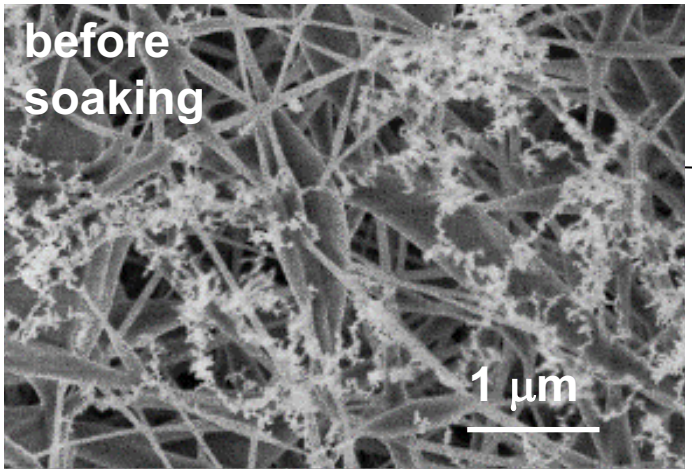
- Coating fibers with Aluminum oxide by Spatial ALD

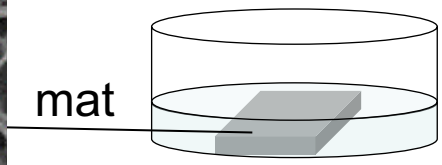


Temperature : 70 °C
Flow rate : TMA 50-400 sccm
H_2O 50-400 sccm
N_2 2 SLM
Number of cycles : 3, 33, 99, 198

- SEM
- X-ray photoelectron spectroscopy (XPS)

- Investigation of water stability



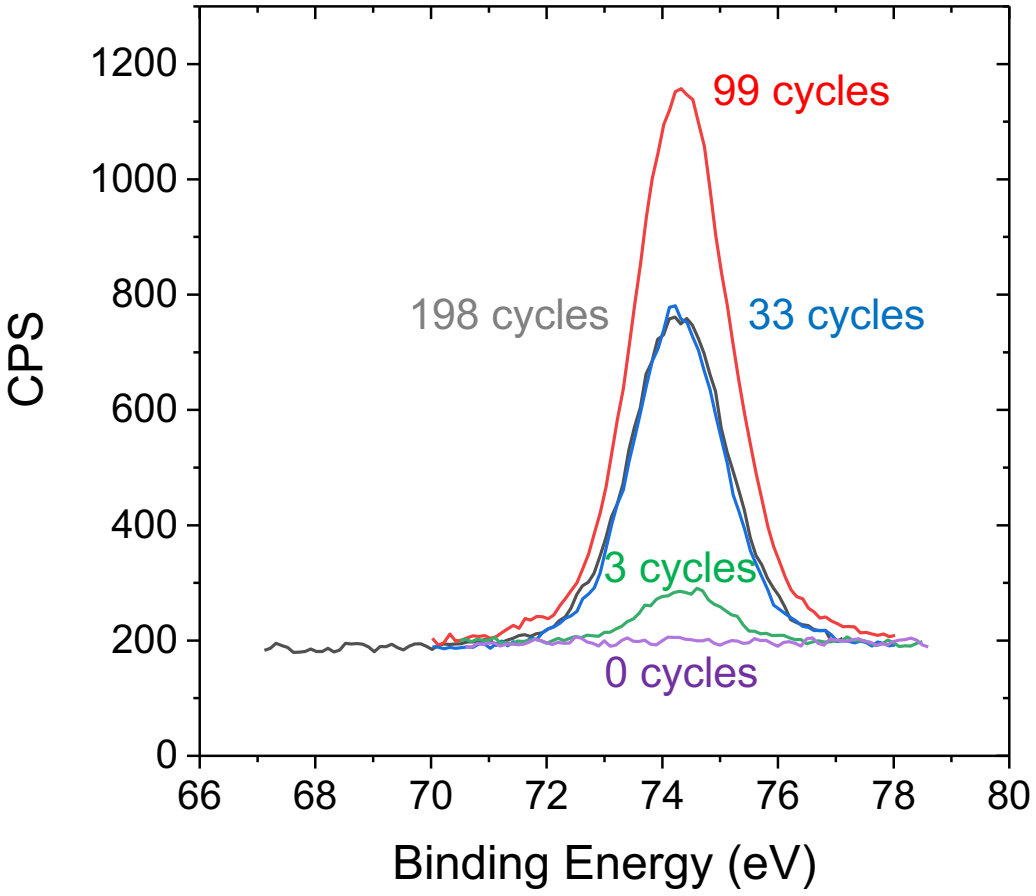


- SEM

AIF analytical instrumentation facility

Amount change of Al_2O_3 for ALD cycles

- XPS spectra of PVA electrospun mats for Al_2O_3



ALD condition

TMA = 200 sccm, H_2O = 200 sccm

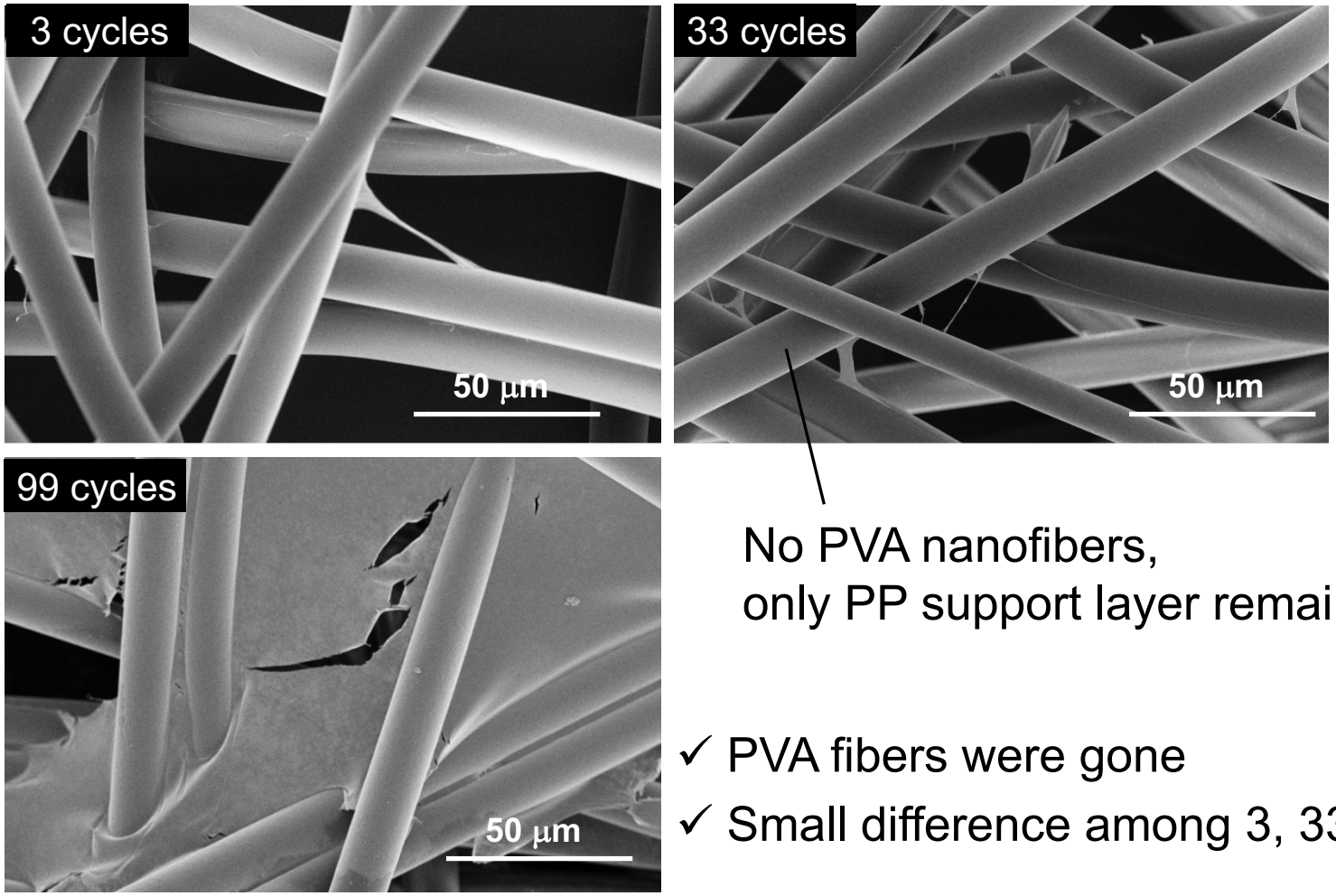
- ✓ Intensity increased with increasing ALD cycles among 3, 33, 99 cycles
- ✓ 198 cycle doesn't fit trend of increasing Al

  → Could be due to non-uniformities in mats

Water stability

- SEM images showing absence of PVA nanofibers after soaking

ALD Condition TMA = 50 sccm, H_2O = 100 sccm



No PVA nanofibers,
only PP support layer remained

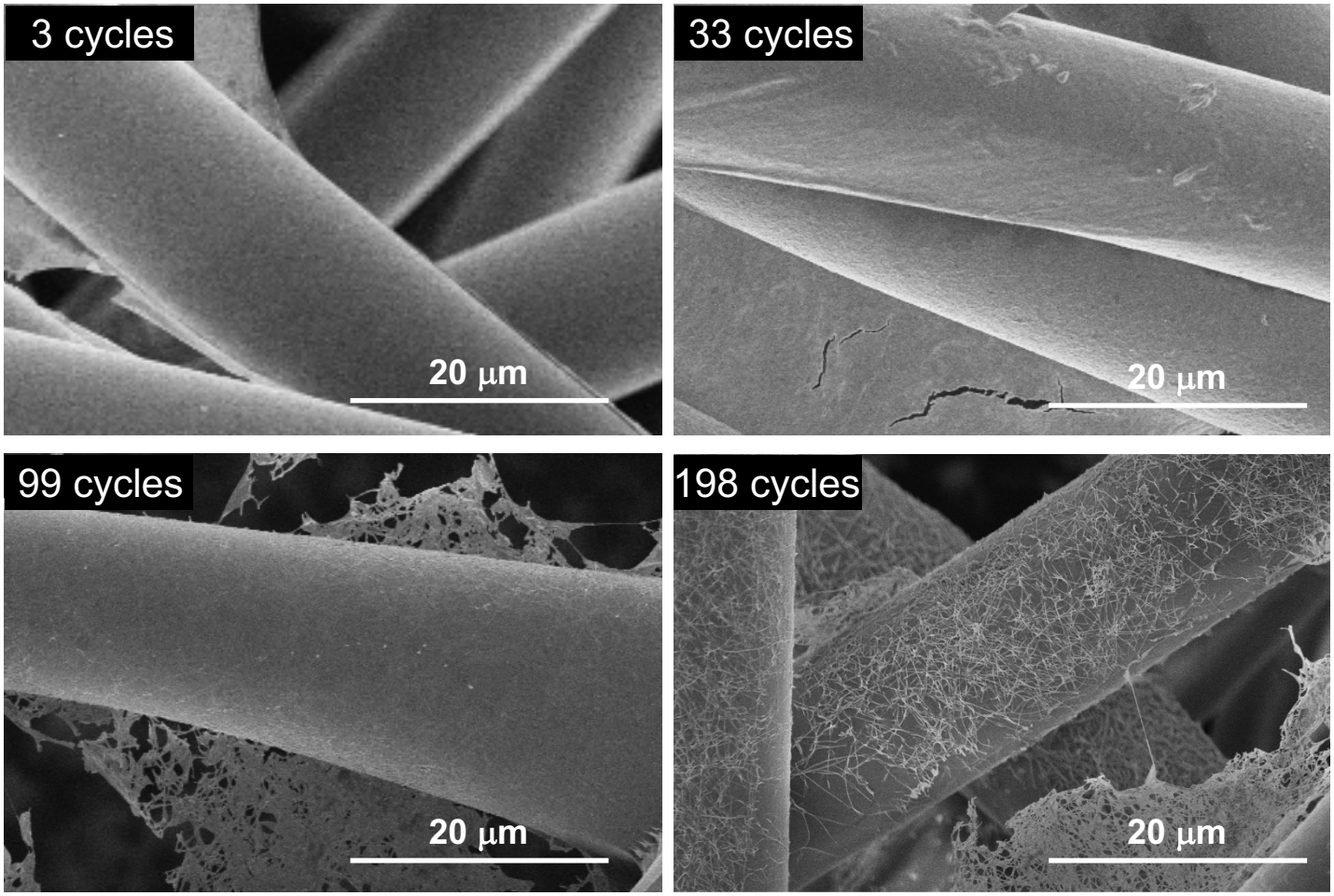
✓ PVA fibers were gone

✓ Small difference among 3, 33, 99 cycles

Water stability

- SEM images of PVA nanofibers treated with ALD and soaked in water for 1 hour

ALD Condition TMA = 200 sccm, H_2O = 200 sccm

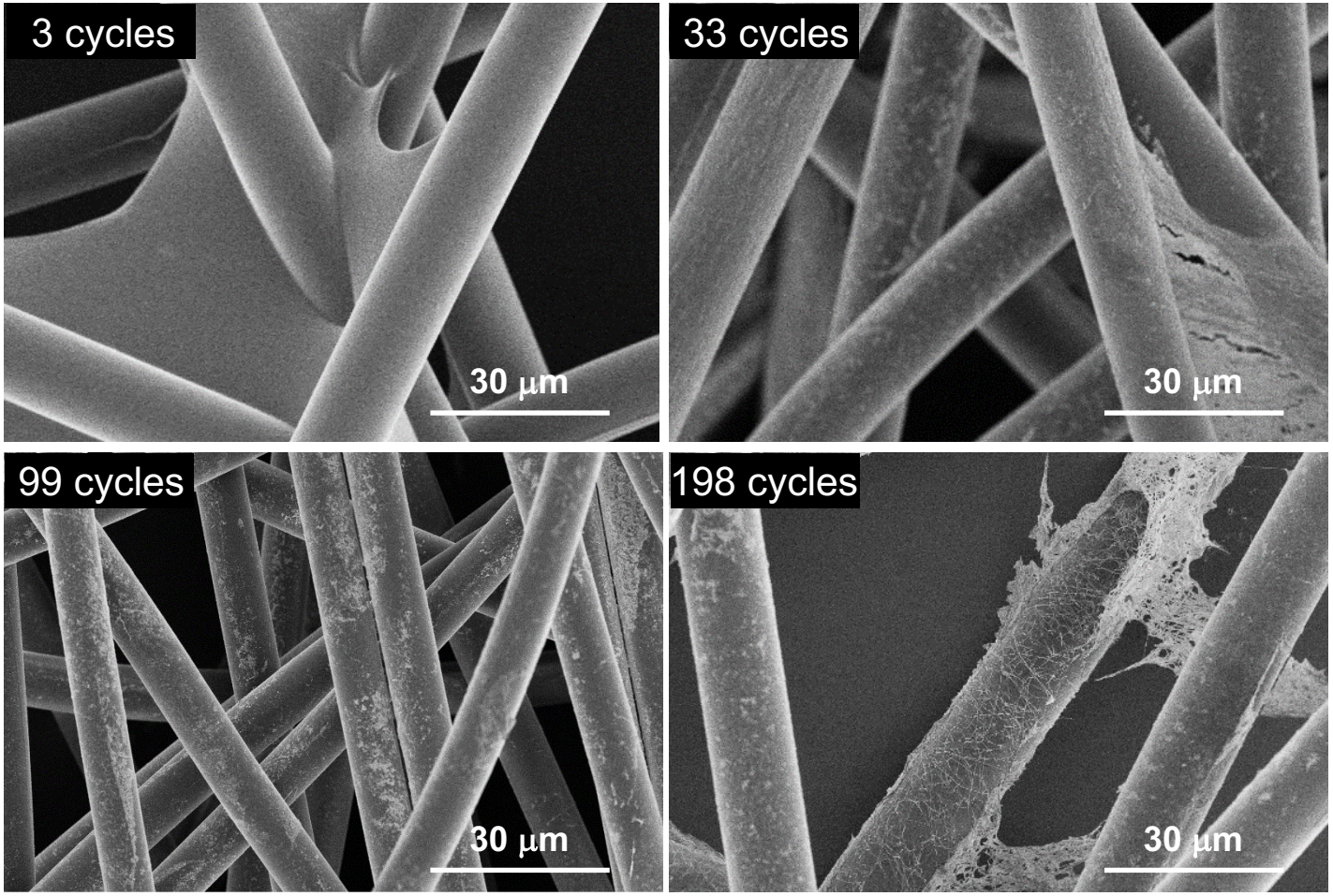


✓ In the condition of 99, 198 cycles, some of fibers remain.

Water stability

- SEM images of PVA nanofibers treated with ALD and soaked in water for 1 hour

ALD Condition TMA = 400 sccm, H_2O = 400 sccm

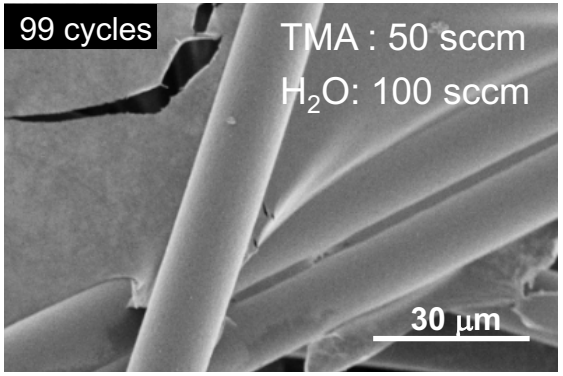


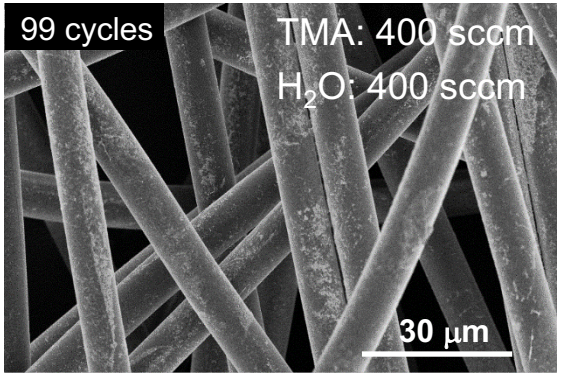
✓ In all conditions, PVA were partially or fully dissolved.

Summary

To characterize an effect of the number of spatial ALD cycles on water stability for electrospun PVA nanofiber

- ✓ From the results of XPS, the amount of Al_2O_3 on the surface of PVA nanofibers increased between 3 – 99 cycles.
- ✓ Solubility of PVA nanofiber for water decreased with increasing TMA and water flow rate.
- ✓ It would be needed to explore ideal condition of electrospinning for forming PVA nanofibers and spatial ALD for coating with Al_2O_3.





Future work

- Exploring ideal condition to coat the electrospun mat

  - investigate the condition of making PVA mat uniformly by changing the time of electrospininng

  - compare the gas permeability depending on thickness of the mat and flow rate in the spatial ALD

Thank you so much for your kind attention.