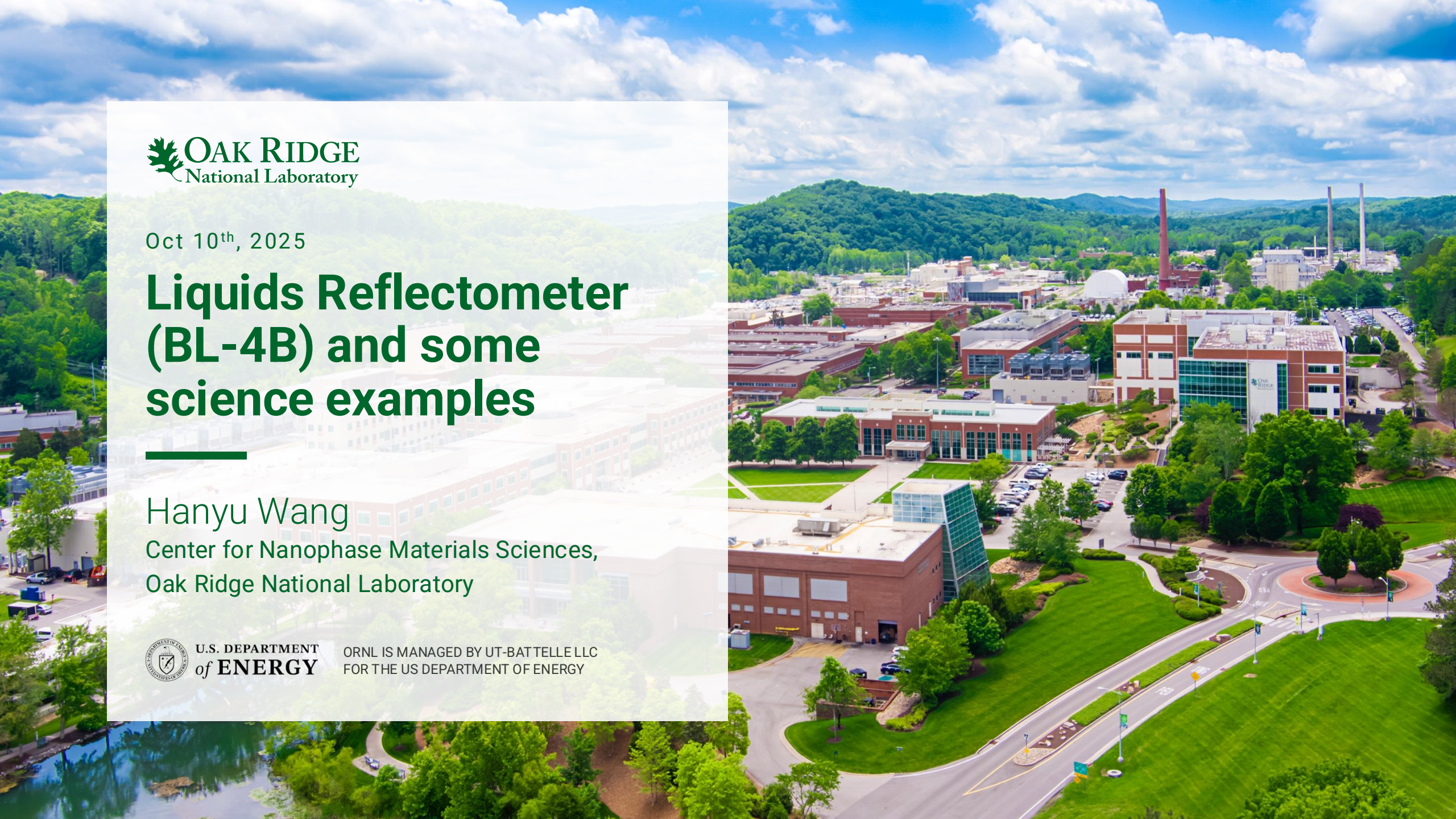


OAK RIDGE National Laboratory

Oct 10th, 2025

Liquids Reflectometer (BL-4B) and some science examples

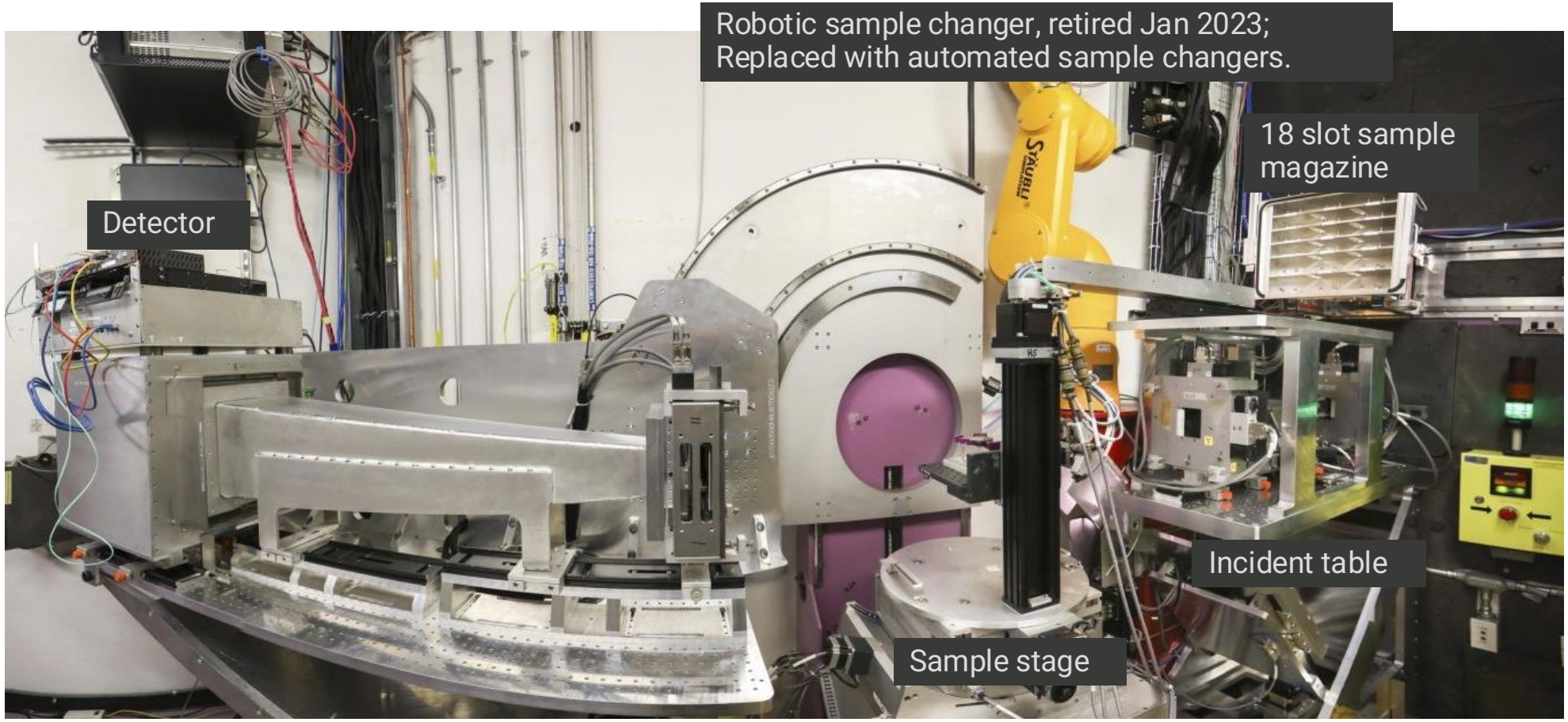
Hanyu Wang

Center for Nanophase Materials Sciences,
Oak Ridge National Laboratory

U.S. DEPARTMENT of ENERGY

ORNL IS MANAGED BY UT-BATTELLE LLC
FOR THE US DEPARTMENT OF ENERGY

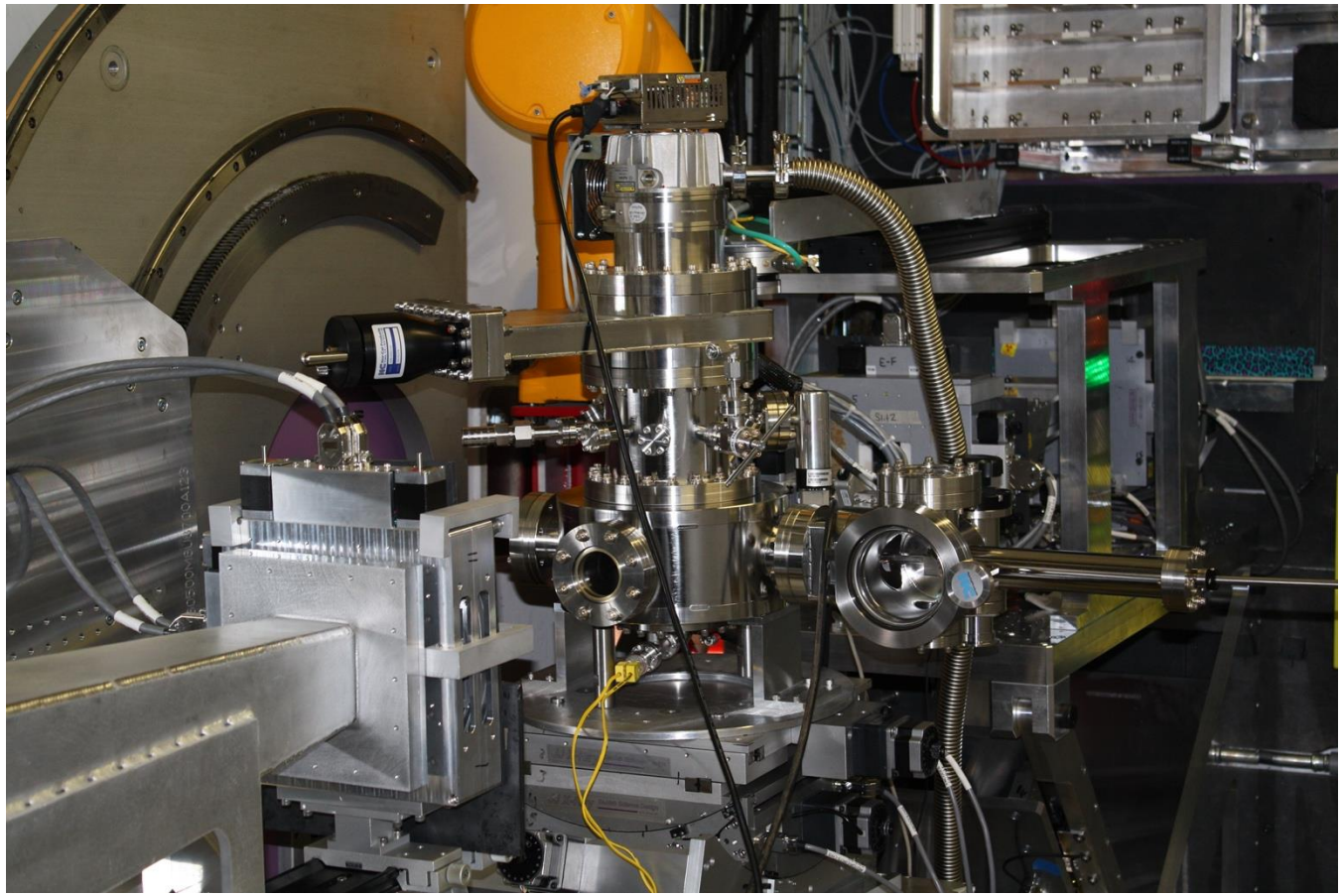
Liquids Reflectometer, BL-4B at SNS, ORNL



LR probes surface and interfacial structures of thin films on length scales of 0.5 nm to 350 nm.

- Change in thickness, scattering length density and roughness
- Air/solid, liquid/solid, air/liquid
- Operates at 60 Hz (3.4 Å) and 30 Hz (6.8 Å), tentatively 20 Hz

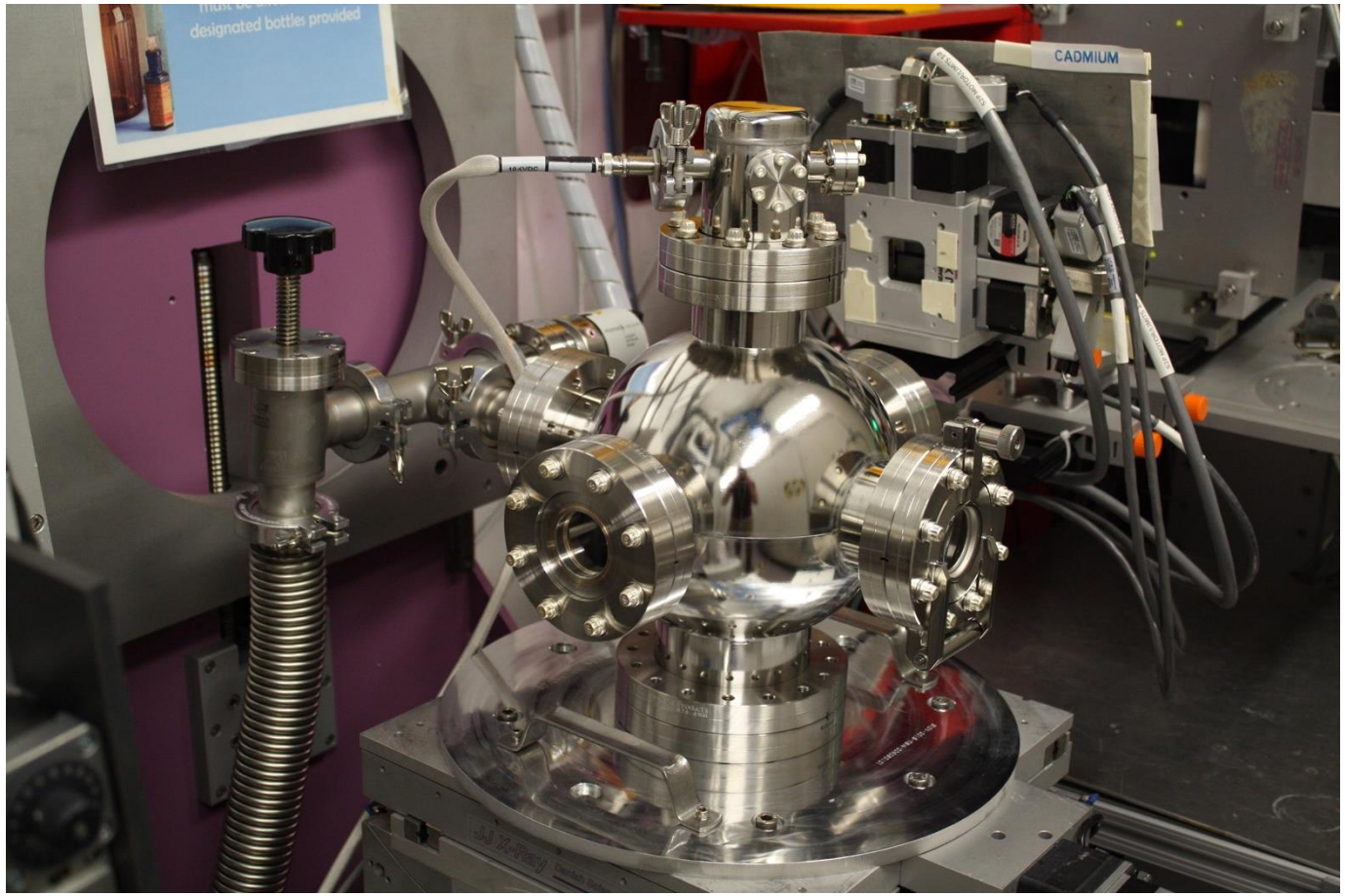
Multi-environment UHV chamber



Gas absorption studies

- Base pressures < 10^{-9} Torr
- Temperature control from RT to 600 ^{o}C
- Load lock system from sample loading
- Precision gas doping
- Can be upgraded to incorporate UV-Vis or IR spectroscopy

High-field chamber

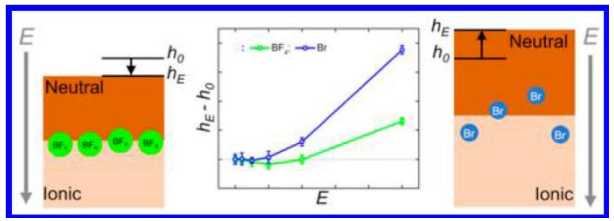


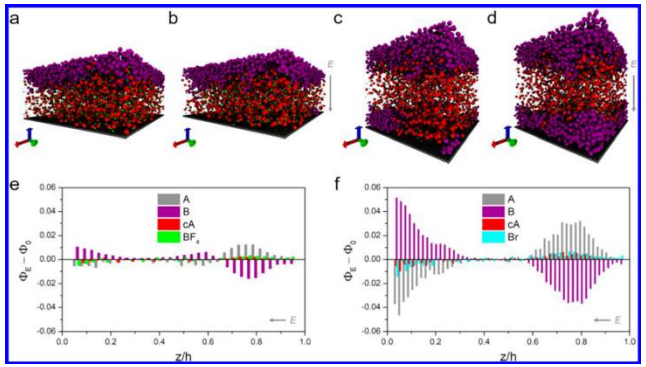
- Applied electric fields to 7 kV over 7mm gap
- High Vacuum ~ 10^{-7} Torr
- Temperature - RT – 200 °C

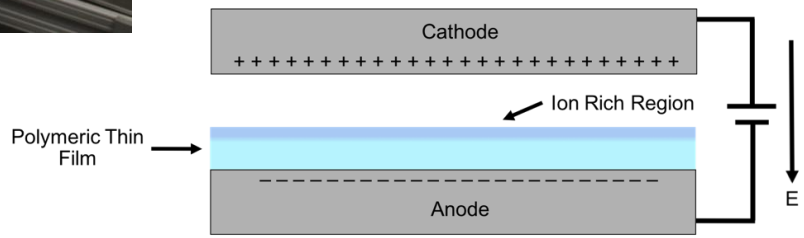
ACS APPLIED MATERIALS & INTERFACES
Research Article
Cite This: ACS Appl. Mater. Interfaces 2018, 10, 32678–32687
www.acsami.org

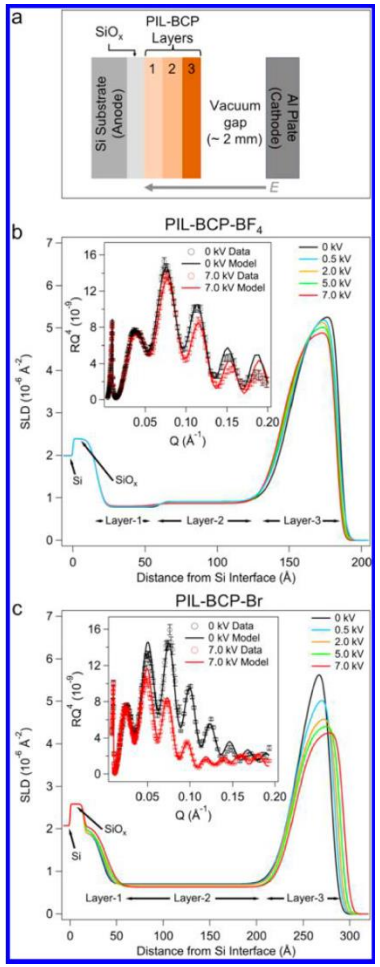
Nanoscale Resolution of Electric-field Induced Motion in Ionic Diblock Copolymer Thin Films

Jason W. Dugger,[†,#] Wei Li,[†,#] Mingtao Chen,[‡] Timothy E. Long,[‡] Rebecca J. L. Welbourn,[§] Maximilian W.A. Skoda,[§] James F. Browning,[⊥,∥] Rajeev Kumar,[⊥,†] and Bradley S. Lokitz*[,†]



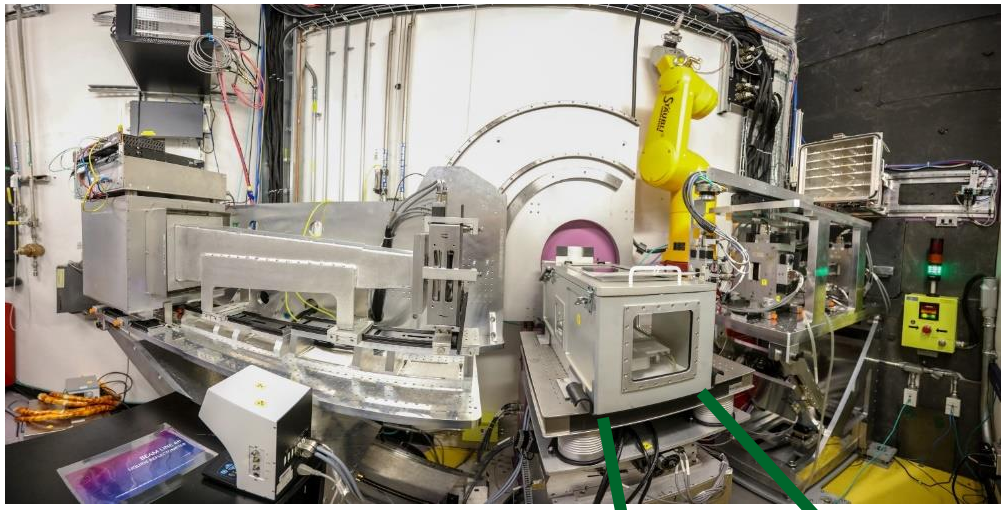




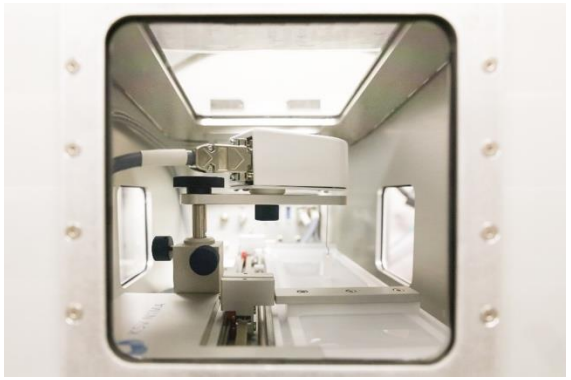


OAK RIDGE National Laboratory | SPALLATION NEUTRON SOURCE

Langmuir Film Studies



- Materials Science
- Polymer Science
- Biomembrane Science

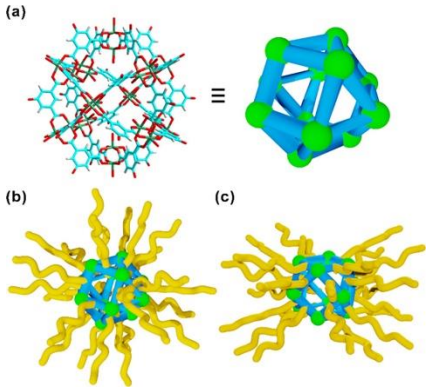


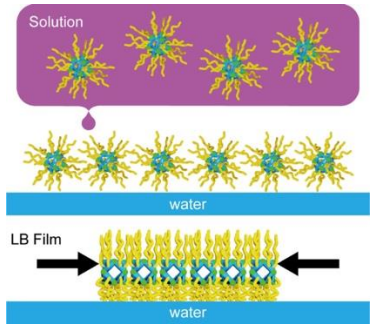
Solvated and Deformed Hairy Metal–Organic Polyhedron

Mu Li, Mingxin Zhang, Yuyan Lai, Yuan Liu, Candice Halbert, James F. Browning,* Dong Liu,* and Panchao Yin*

Cite This: *J. Phys. Chem. C* 2020, 124, 15656–15662

Read Online



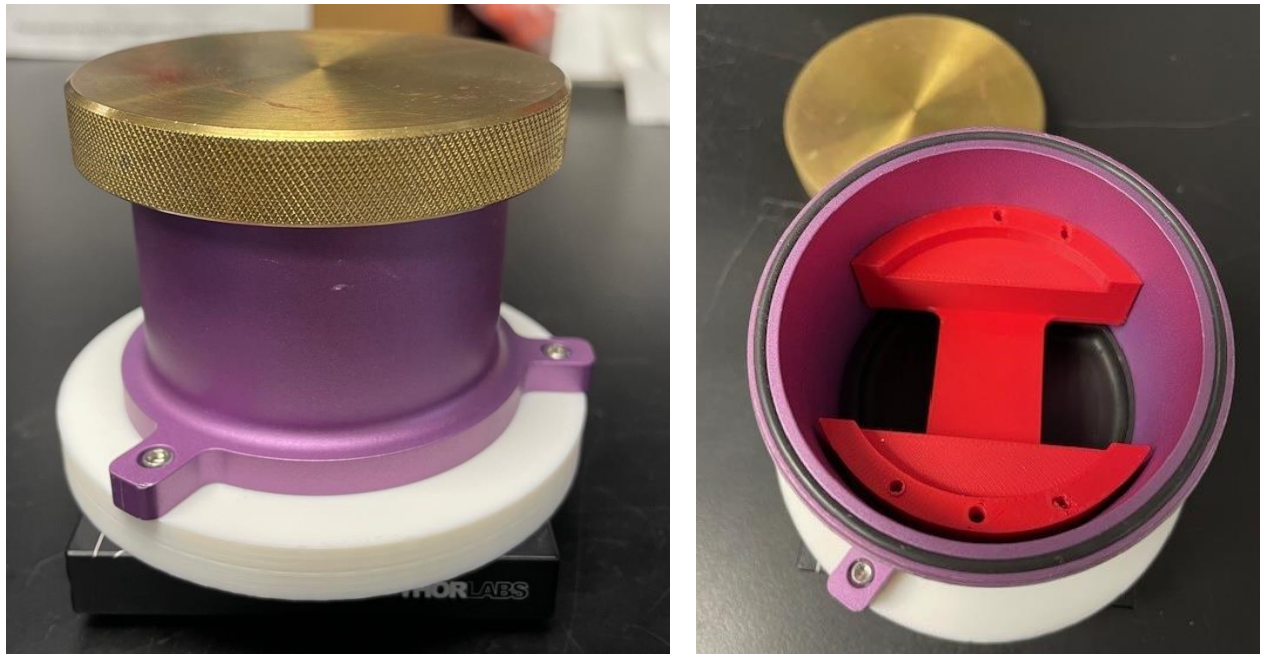


Probe COVID-19 virus infection dynamics in real time in 2020

Critical questions:

- To protect the host:
  - What factors affect the insertion of fusion peptide (FP) into the host membrane?
    - Membrane compositions, e.g., charge density, mechanical properties
    - Effective concentration of FP
  - Can we impede membrane fusion?
    - Block the insertion of FP
    - Block the folding of HR1 and HR2

Humidity can (humidity chamber)

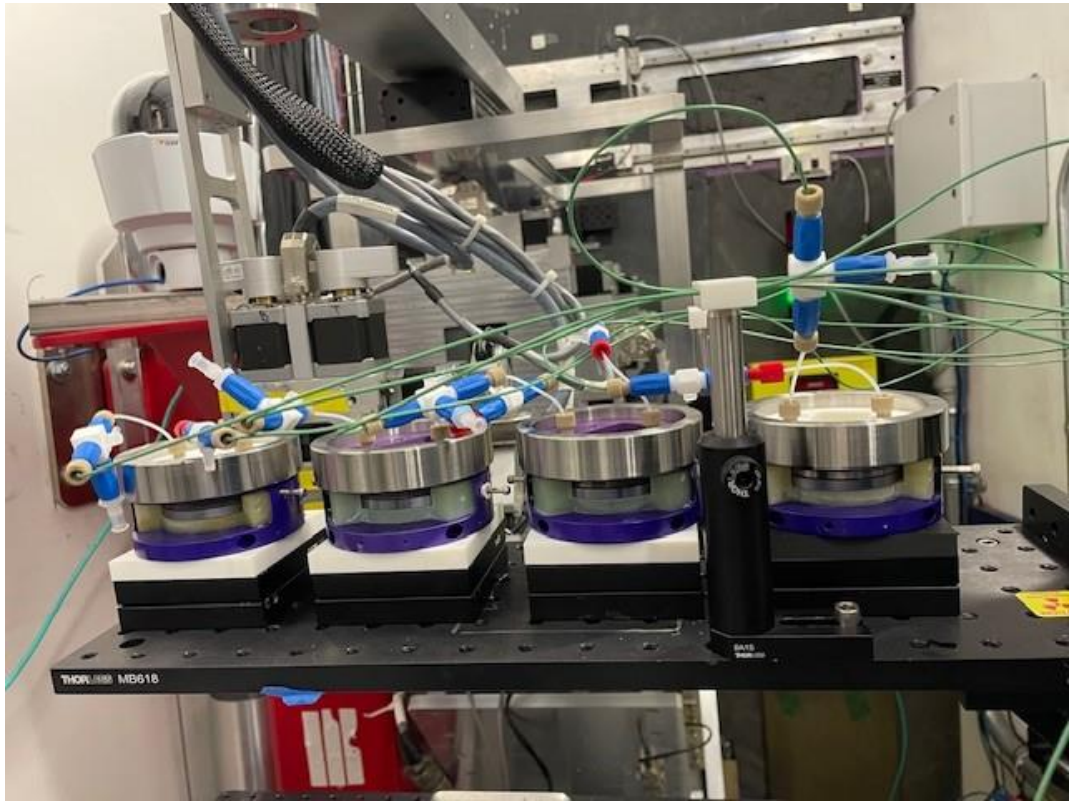


Humidity can

Humidity study:

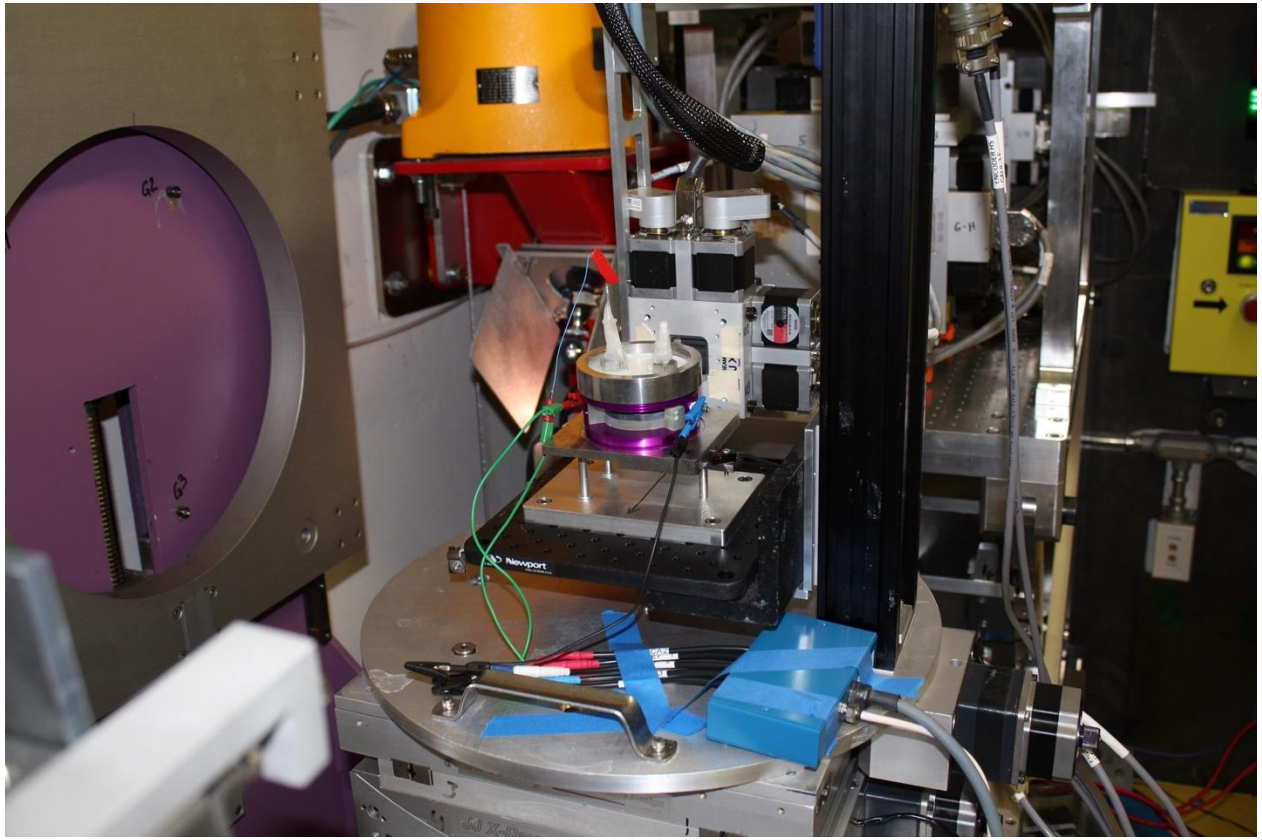
- Humidity can
  - Fixed single point relative humidity study
  - 3D printed sample holder can accommodate both reflect-up and reflect-down measurement
- Humidity chamber
  - Precise and controlled humidity for top and bottom separately

Liquid cell



- Solid-liquid interface
- Mainly use Si substrates (2-inch diameter × 5 mm thickness)
- Automatic control for up to 4 cells (liquid exchange via HPLC pump)

Electrochemical Cell



In situ electrochemistry

- Ion transport and interphase formation in energy storage materials
- Corrosion phenomena
- Redox in chemistry and biology

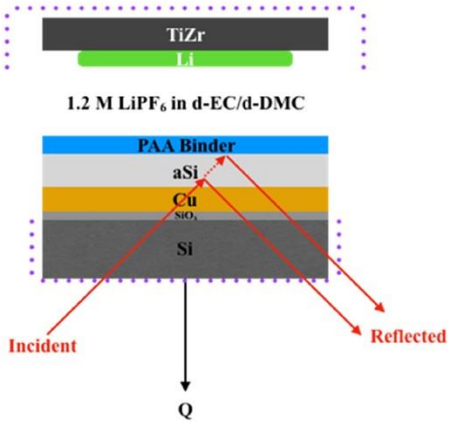
ACS APPLIED MATERIALS & INTERFACES

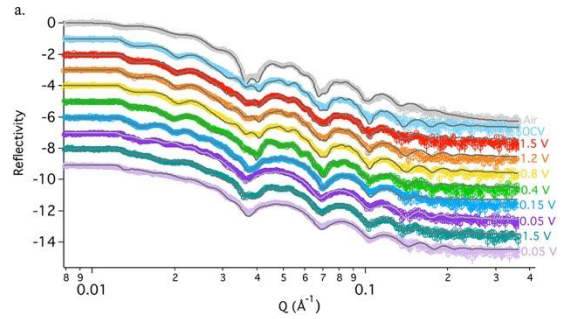
www.acsami.org Research Article

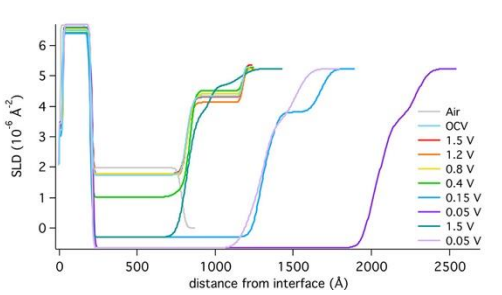
The Study of the Binder Poly(acrylic acid) and Its Role in Concomitant Solid–Electrolyte Interphase Formation on Si Anodes

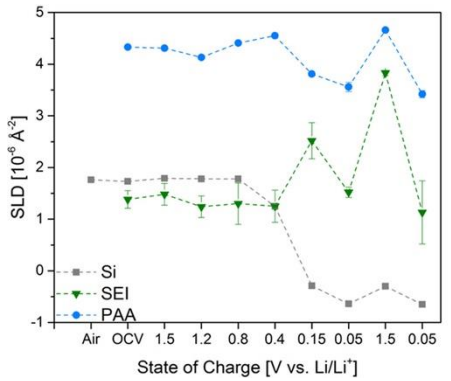
Katie L. Browning,* Robert L. Sacci,* Mathieu Doucet, James F. Browning, Joshua R. Kim, and Gabriel M. Veith*

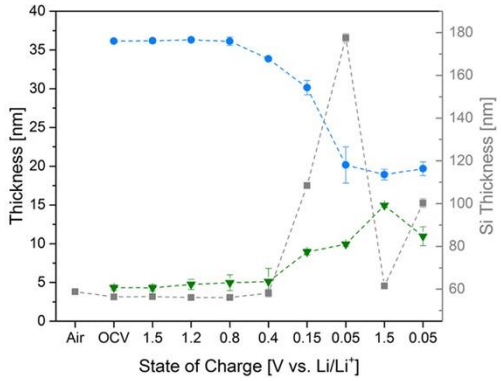
Cite This: *ACS Appl. Mater. Interfaces* 2020, 12, 10018–10030 Read Online







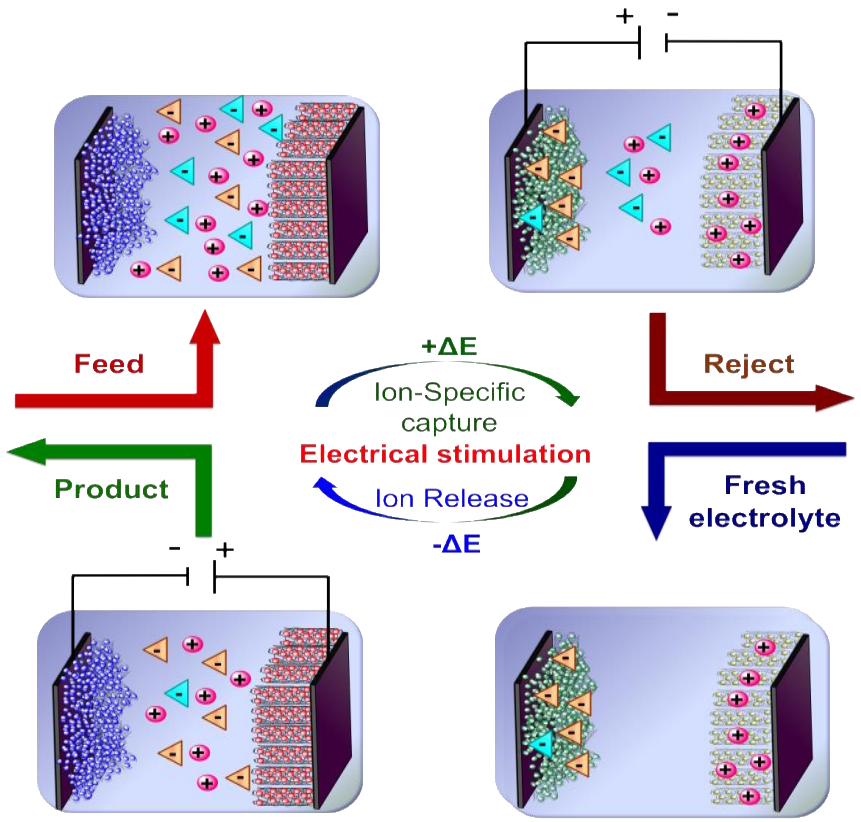




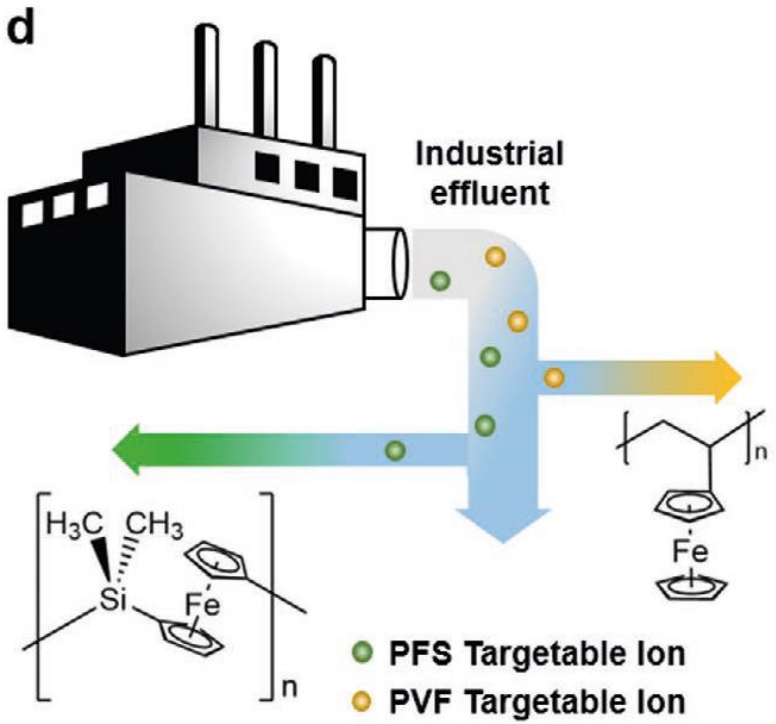
OAK RIDGE National Laboratory | SPALLATION NEUTRON SOURCE

Science case 2: Understanding ion selectivity with redox-polymers

- Fundamental understanding of contributing mechanisms, and control of interactions to achieve ion-selectivity.
- An important platform by tailoring advanced redox-polymer for targeting heavy metal recovery and remediation.

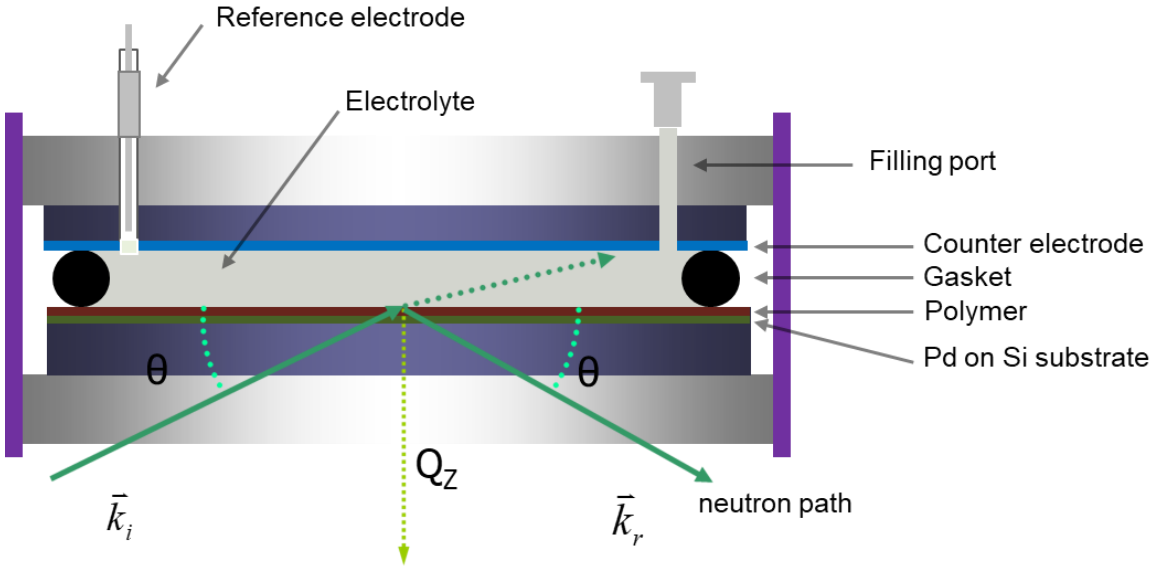


Development of redox-selective materials

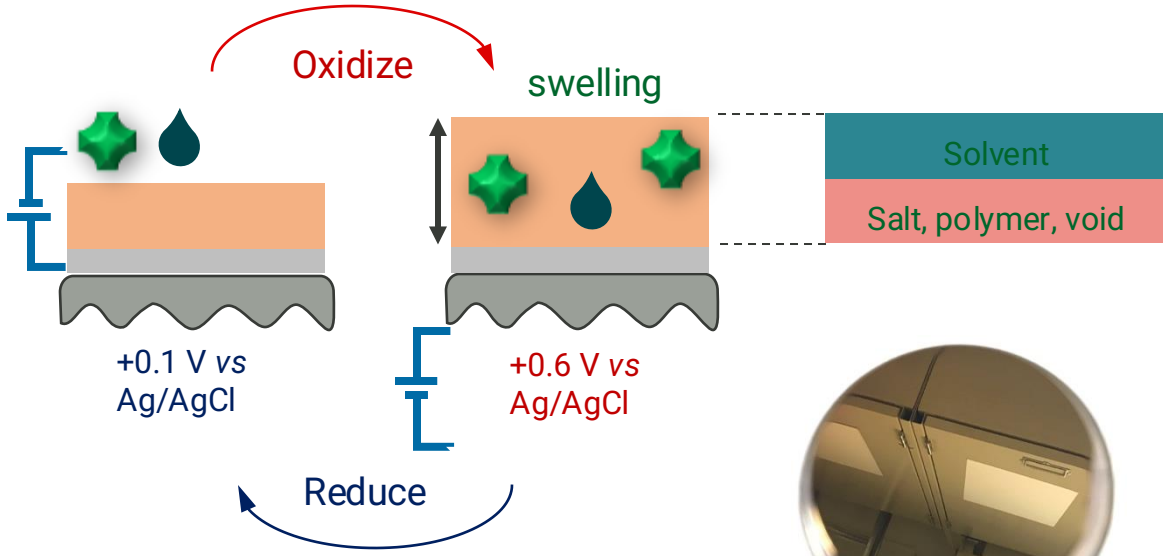


Chen et al, Adv. Funct. Mater. 2021, 31, 2009307

Experiment preparation



Schematic diagram of the EC cell and NR geometry

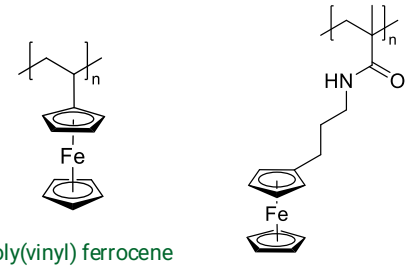


Sample on a 2-inch diameter Si wafer

Working electrode: redox-active polymer
Counter electrode: TiZr
Reference electrode: Ag/AgCl
Electrolyte: Target ions in H_2O or D_2O or mix of H_2O and D_2O

NR: To probe the spatial distribution, ion migration and film swelling as a function of applied potentials on polymer thin film.

Salt: Cr, Mo, Re, V, etc.



PVF: poly(vinyl) ferrocene
PFPMAm: 3-ferrocenylpropyl acrylamide

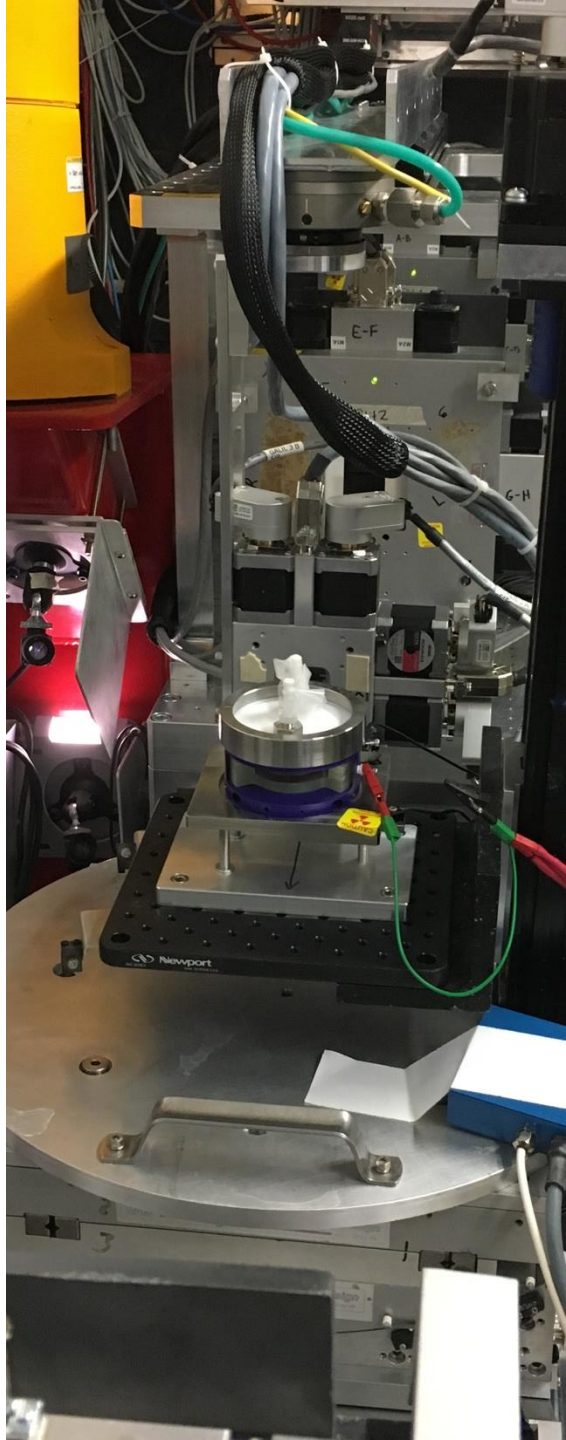
Pendant group polymer

Experiment plan

- Measure ion selectivity and hydration by comparing combinations of polymer, target ion, and solvent (H_2O/D_2O).
- Apply a series of potential for each system.
- Because of the way we wanted to measure, no automated control between EC and NR possible… very labor intensive.

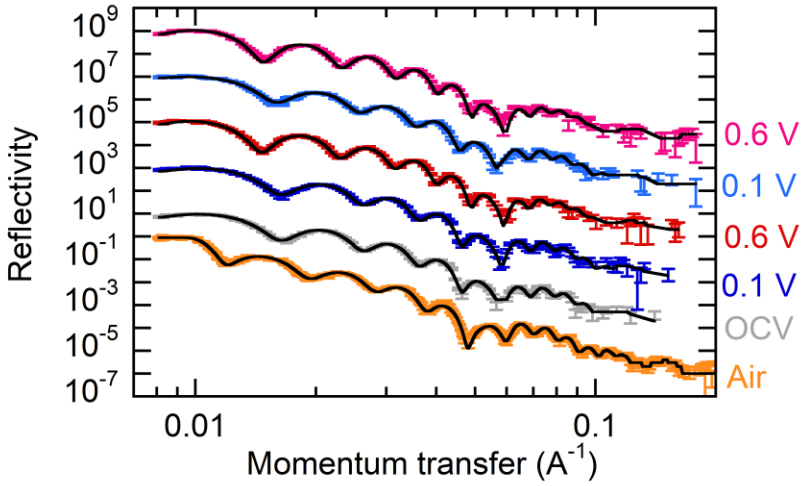
Suggested time-resolved measurements. This was done during the relaxation period between each applied potential…

Dry	equilibrium measurement
Open Circuit	equilibrium measurement
+0.1 V vs Ag/AgCl	equilibrium measurement
	dynamic measurement (~30 min)
+0.6 V	equilibrium measurement
	dynamic measurement (~30 min)
+0.1 V	equilibrium measurement
	dynamic measurement (~30 min)
+0.6 V	equilibrium measurement

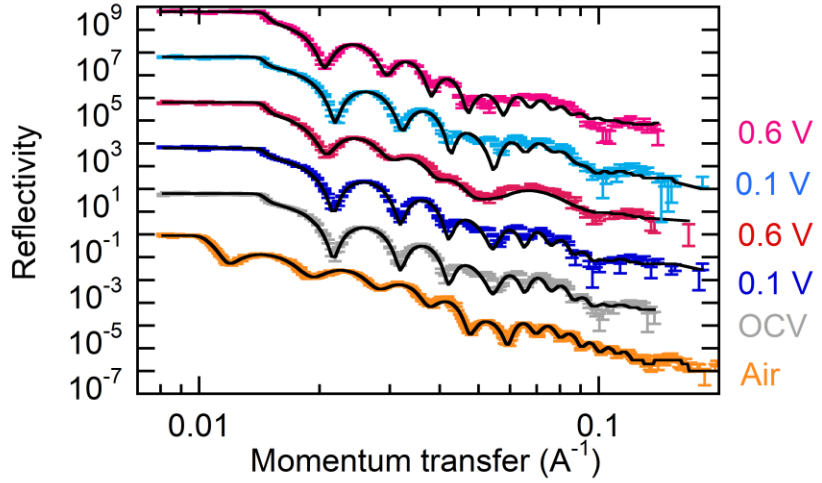


NR and SLD profiles (a very small part of data…)

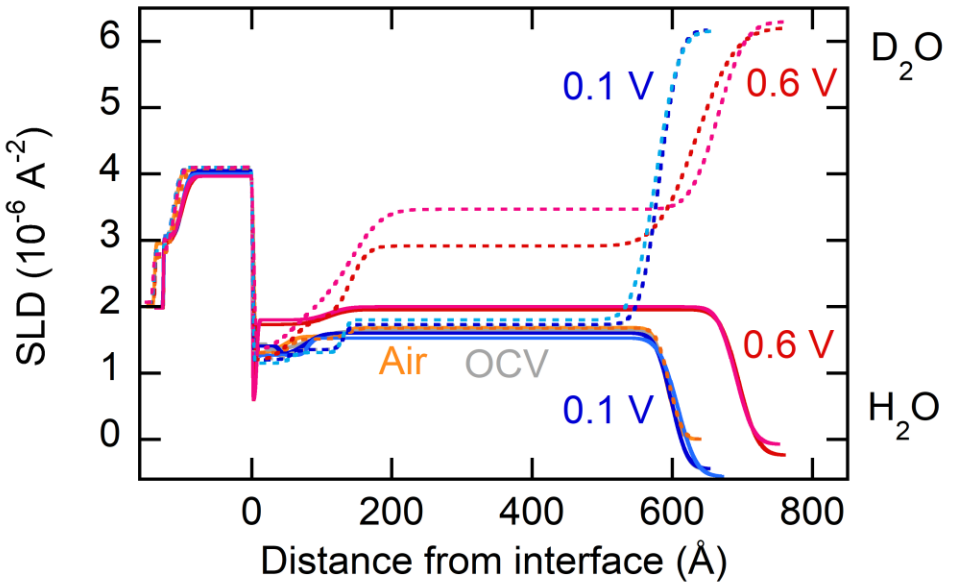
H_2O - **S**



D_2O - **T**

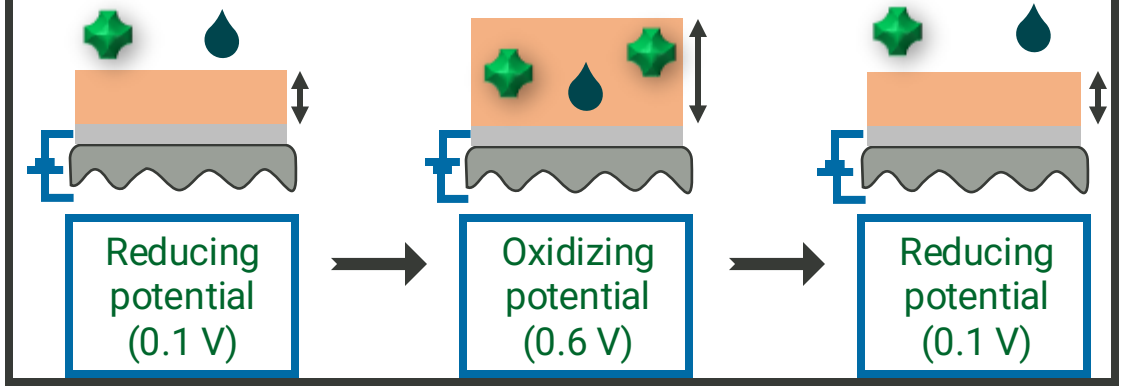


100% ReO_4^-



H_2O - **S**
D_2O - **T**

In situ neutron reflectometry: study solvation and swelling

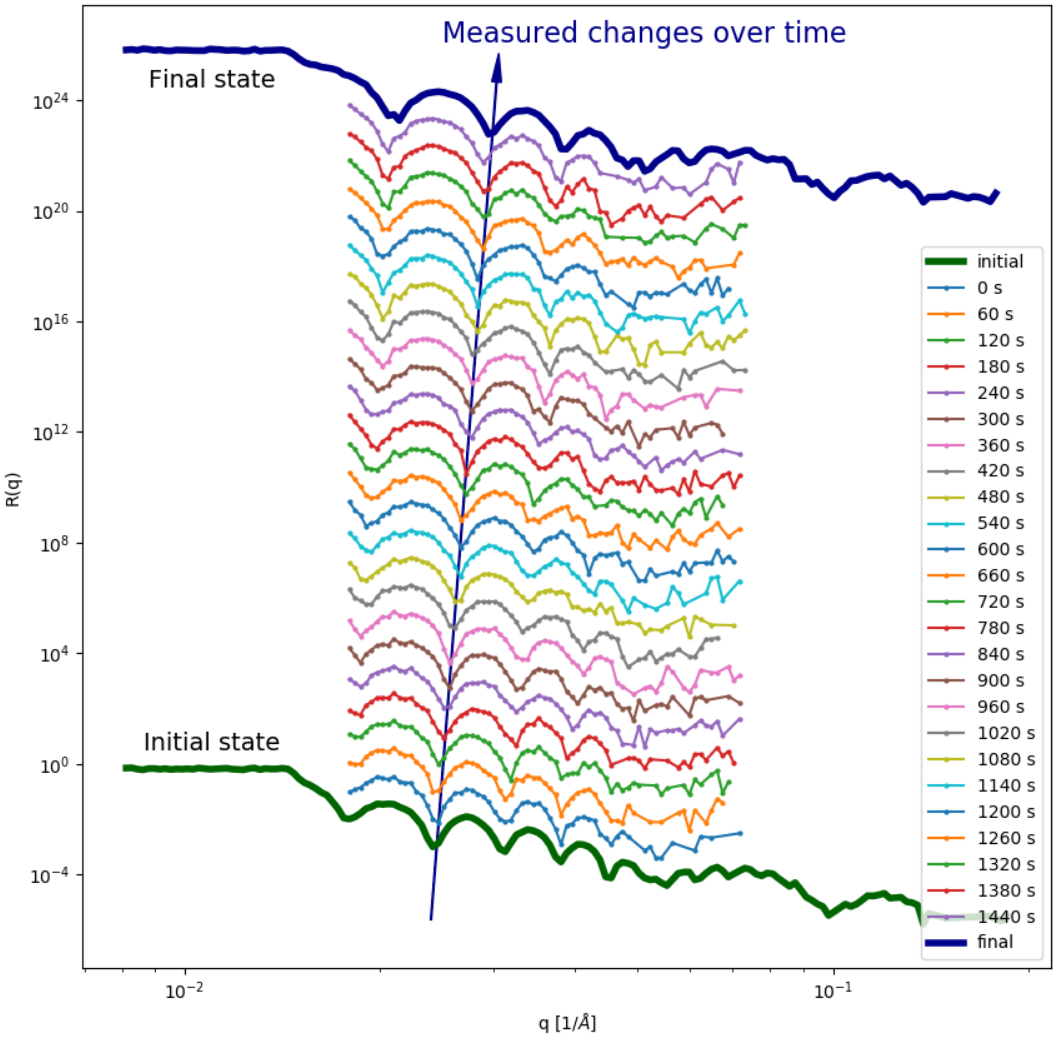


NR and SLD profile monovalent behavior for PFPMAm.

R. Candeago, et al. *JACS Au* 2024, 4, 3, 919–929

New type of measurements: time-resolved reflectometry

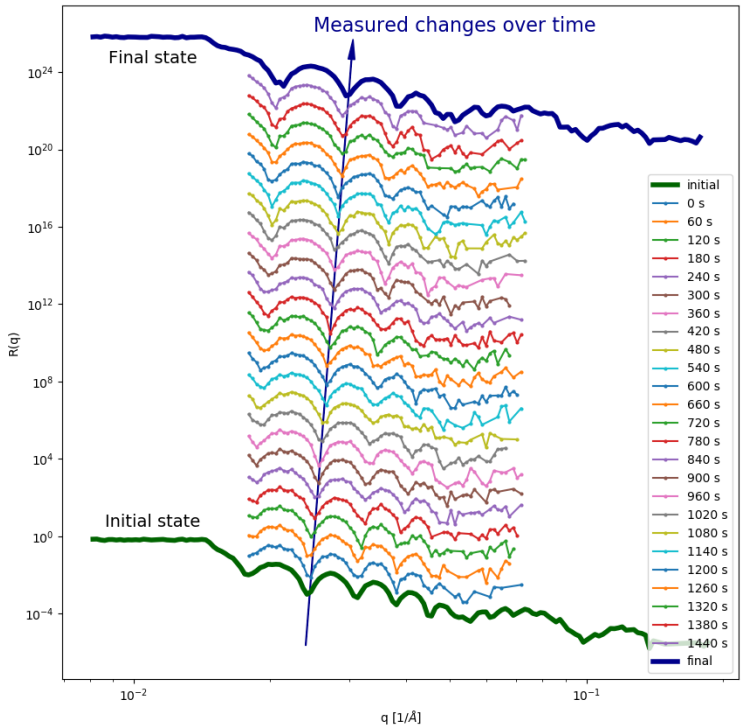
- It complements our standard measurements.
- Time scale available at 4B is well matched to EC processes.
- Our standard measurement is made of multiple runs, e.g., 7 runs at 60 Hz, or 3 runs at 30 Hz.
- Operating at 30 Hz and judiciously picking **θ** and **λ** band, could measure a range of 0.017 < Q < 0.05 1/Å.
- Initial and final states measured for complete Q range (~45 minutes).
- Data sliced in 1-minute intervals.



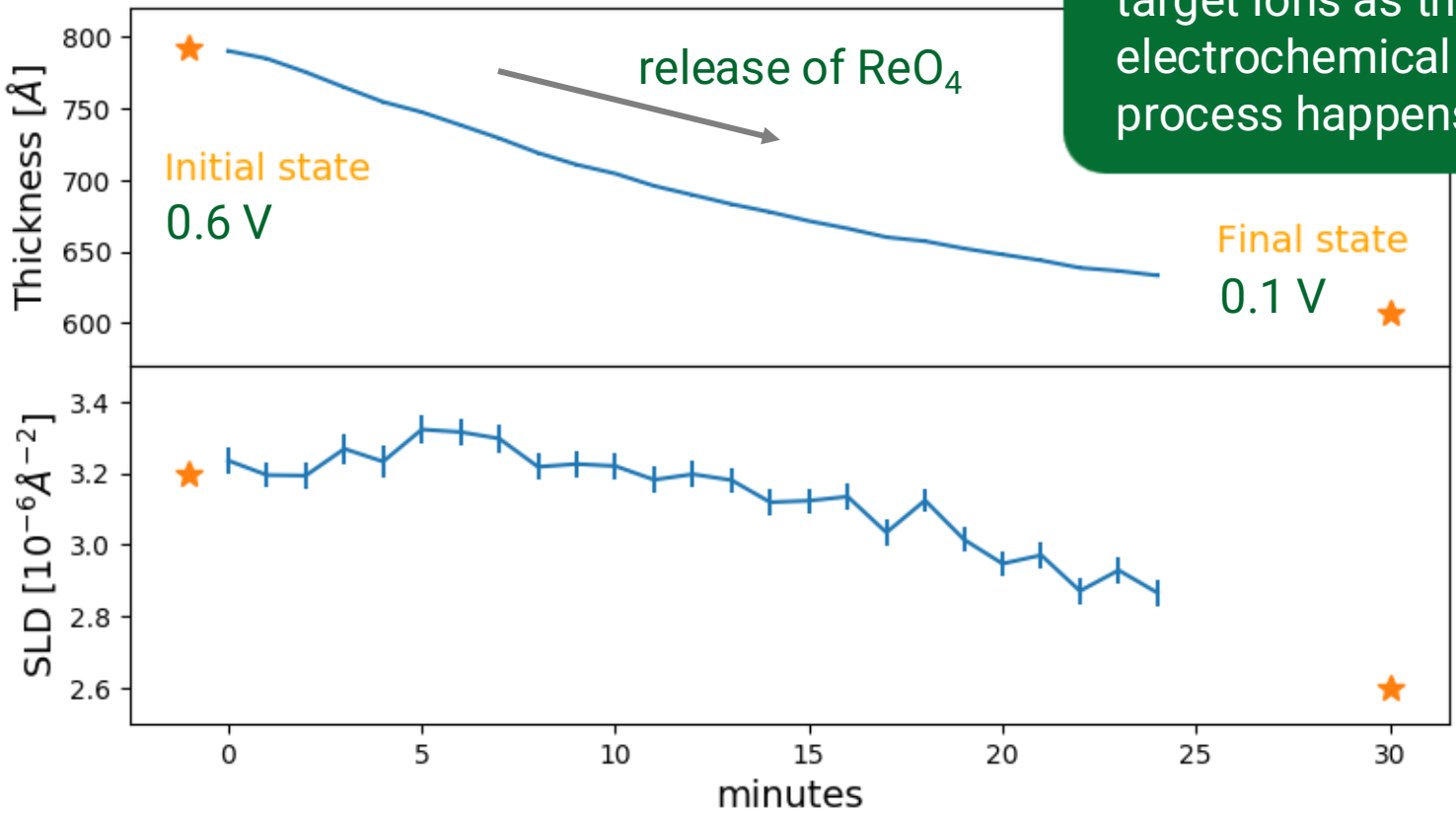
R. Candeago, et al. *JACS Au* 2024, 4, 3, 919–929

Need for new analysis approach: Bayesian fitting loop

- That's a lot of data to fit: **2,600 time-resolved reflectivity curves**…
- We developed a way to use the initial state as prior information and perform iterative fits, with each fit result becomes the prior for the next.



PFPMAm with ReO_4 in D_2O

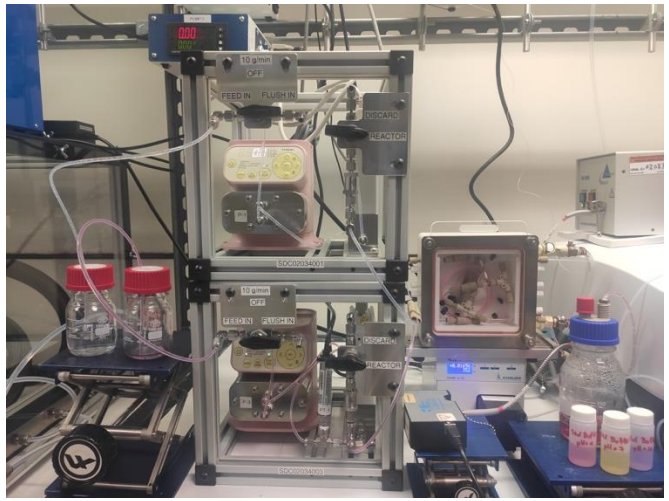


We can see the capture/release of target ions as the electrochemical process happens!

R. Candeago, et al. *JACS Au* 2024, 4, 3, 919–929

Capabilities at Macro group CNMS

Autonomous Continuous Flow Reactor Synthesis for Atom Precision with Scalability (AutoFlowS)



Snapdragon

Large Scale synthesis

Multiple reactors in one flow

Can connect to a large amount of instrumentation

Phoenix by ThalesNano

- High pressure, high temperature
- Two compartments for temperature control
- Gas module

Vapourtec

- Small scale synthesis
- Organic synthesis
- Deuteration

Various analytical techniques

Raman
UV-Vis
QCM
GPC/HPLC
SPR
ESR
RI

3D printing lab

Fused deposition modeling (FDM)

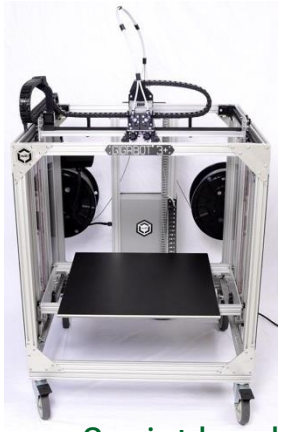
Ultimaker 2

Single print nozzle

Ultimaker 3

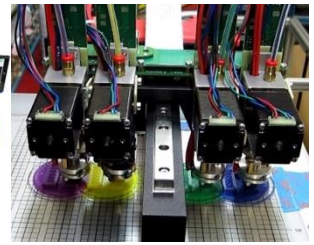
double print nozzle

Gigabot



2 print heads. Big size print.

Hyrel 3D



Up to 5 print heads

Suitable materials: Filament. Constant diameter during extrusion.

Common materials: ABS, PLA, PC

Stereolithography (SLA)

Formlab 3B

Formlab 3

Form-cure

Post curing of SLA printed object
- UV light chamber,
- Heated chamber (up to 70 C)
- Selected time

Visible light source
Photocentric

Suitable material: Photo-polymerization at a fast rate. Good flowability.

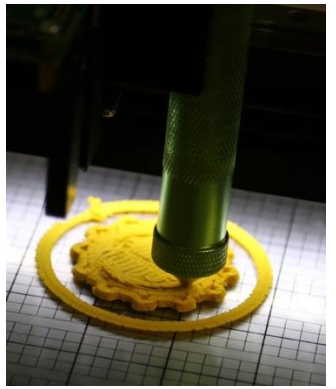
Typical example: Acrylate based materials

Direct Ink Writing (DIW)

Hyrel 3D

Syringe head:
DIW printing

Heated “syringe”
Up to 200 C



Suitable materials:
- Fluids with thixotropic rheology

Common materials:
Metal, ceramic, thermoset, thermoplastic, solution, emulsion, hydrogel…

Selective Laser Sintering

Sinterit Lisa

Suitable material:
Powder based material

Common materials:
Nylon 12

Injection molding machine

Model 150A injection machine

APX-PIM injection machine

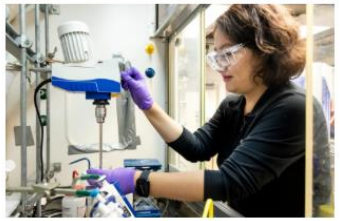
User Facilities

Center for Nanophase Materials Sciences

Home | About Our Team | About CNMS | Research | Research Sections and Groups | User Information | Media and Events

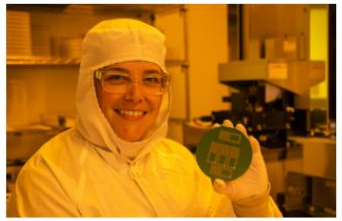
The Center for Nanophase Materials Sciences (CNMS) at Oak Ridge National Laboratory (ORNL) offers the national and international user community access to staff expertise and state-of-the-art equipment for a broad range of nanoscience research, including nanomaterials synthesis, nanofabrication, imaging/microscopy/characterization, and theory/modeling/simulation.

Quick Resource Links

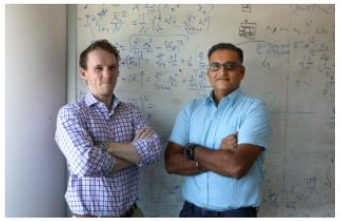


Staff Expertise

Capabilities and Instruments



Research Impact



Become a CNMS User

Spallation Neutron Source (SNS) and Center for Nanophase Materials Sciences (CNMS) at Oak Ridge National Laboratory (ORNL)

Welcome to submit user proposals to CNMS!

https://www.ornl.gov/facility/cnms
https://neutrons.ornl.gov/