

DTU

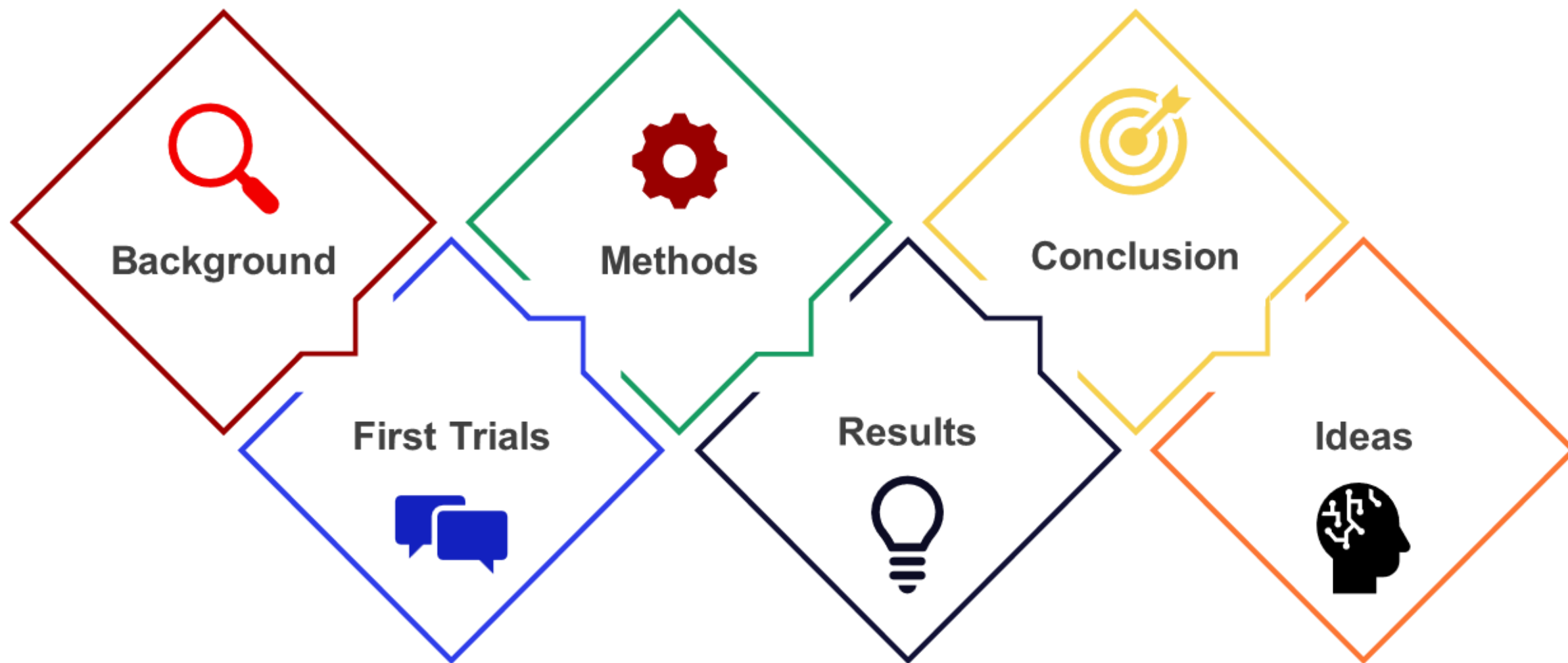


Quantitative Characterization of Hydrogel Layers on Antifouling Coatings

Presenter: **Shawn Lindner**

Research Group: CoaST, Chemical & Biochemical Engineering, DTU (Denmark)

Supervisors: Kim Dam-Johansen, Markus Schackmann, and Narayanan Rajagopalan







- **What is a hydrogel?**

- Microporous hydrophilic three-dimensional polymer network
- Containing large amount of water (typically 60-99 wt%)

- **Hydrogels as antifouling strategy?**

Hydrated, soft surfaces resist fouling by mimicking biological tissues (e.g., fish and seaweed)

- Protein/Polysaccharide adhesion is weak [1]
- Organisms cannot detect the surface [2]
- Softness of hydrogels can make barnacles peel off with balanced gel strength [3]
- Synergy with Cu_2O (hydrogel can act as reservoir) [4]

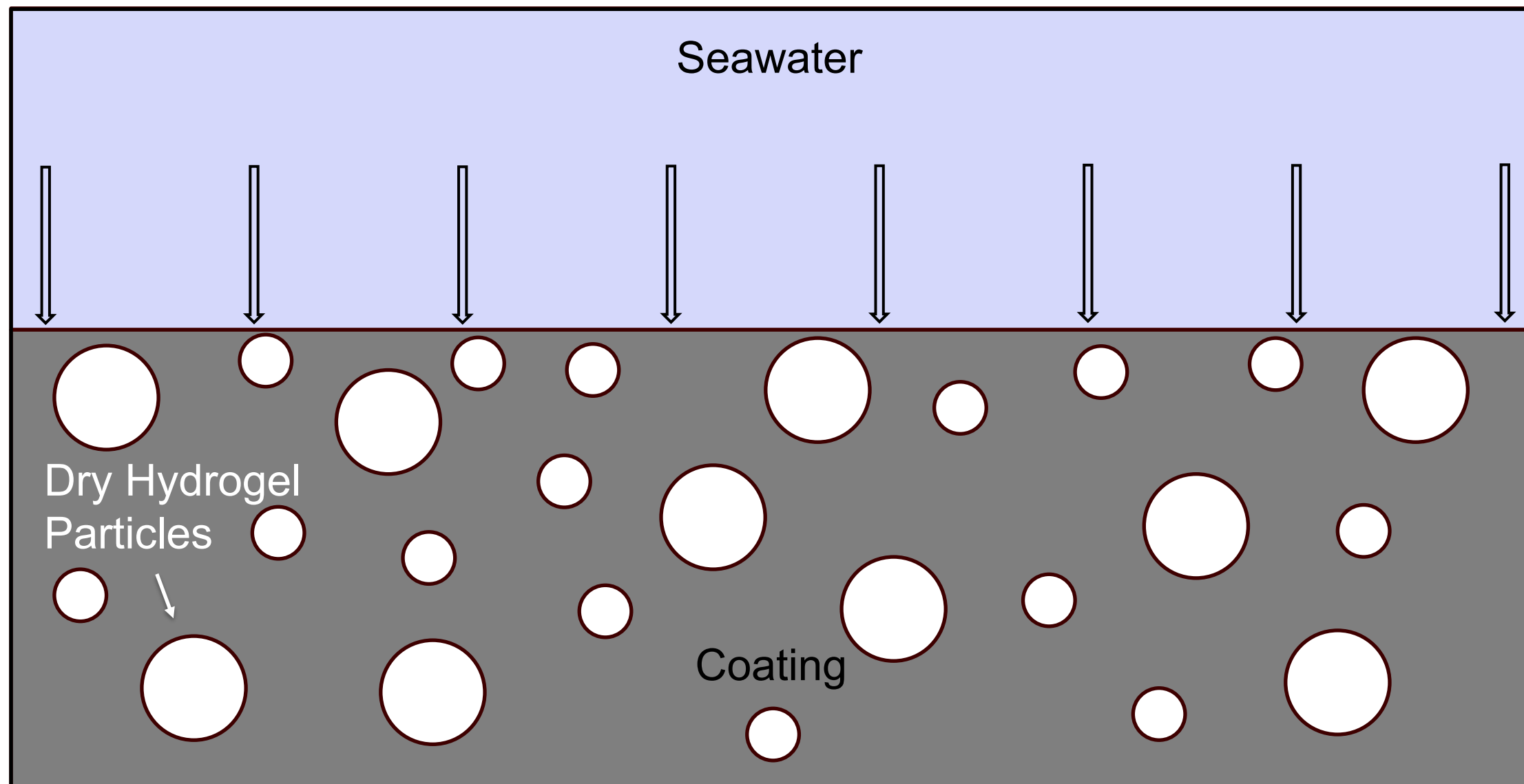
→ **Potential to reduce biocides**



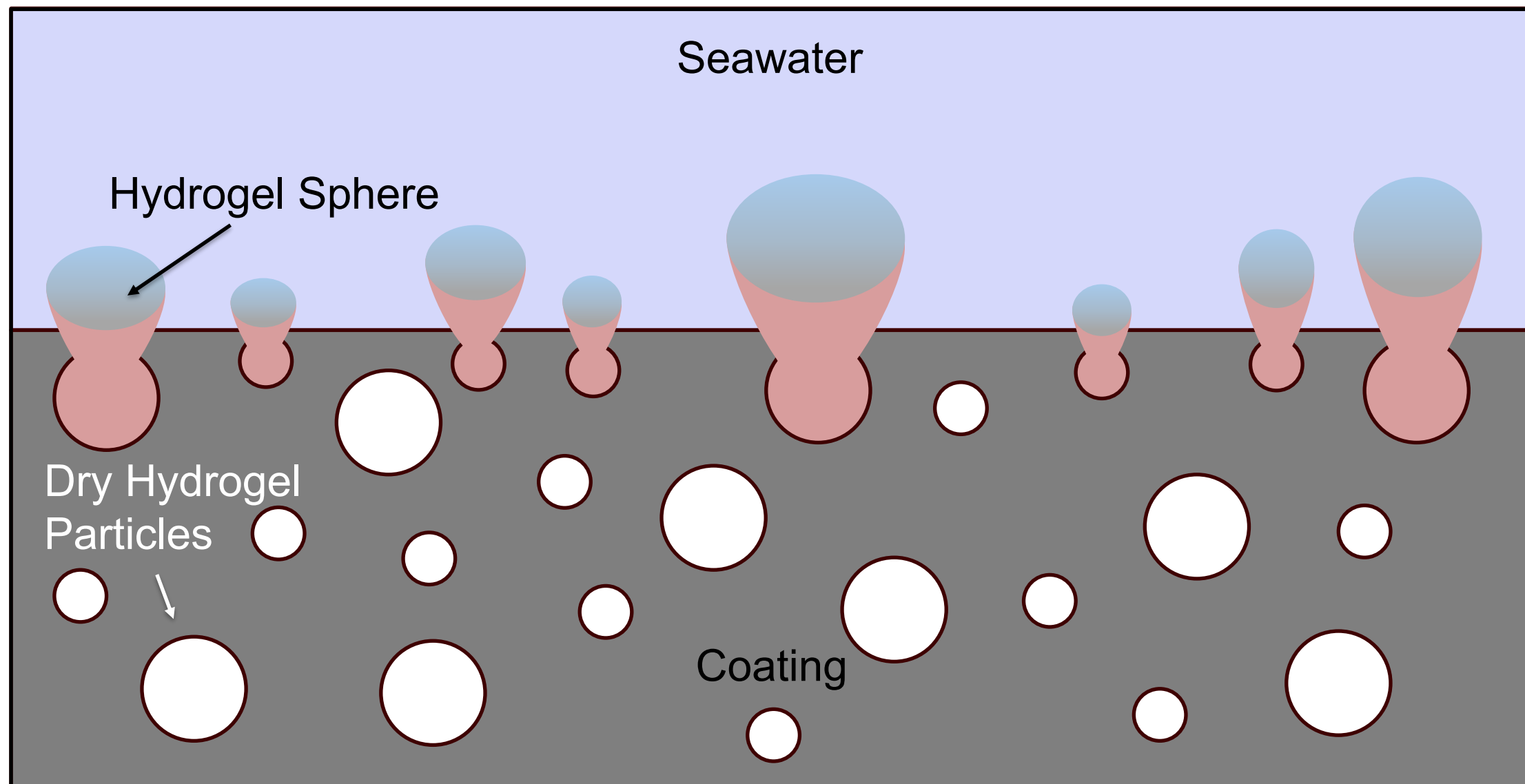
Our Approach

- Hydrogel is built in-situ upon seawater contact atop the coating
- Non-toxic hydrogel precursor particles (Xanthan Gum)
- Solid/gel transition instead of sol/gel
- Cuprous oxide is stored in the hydrogel, making its use more efficient

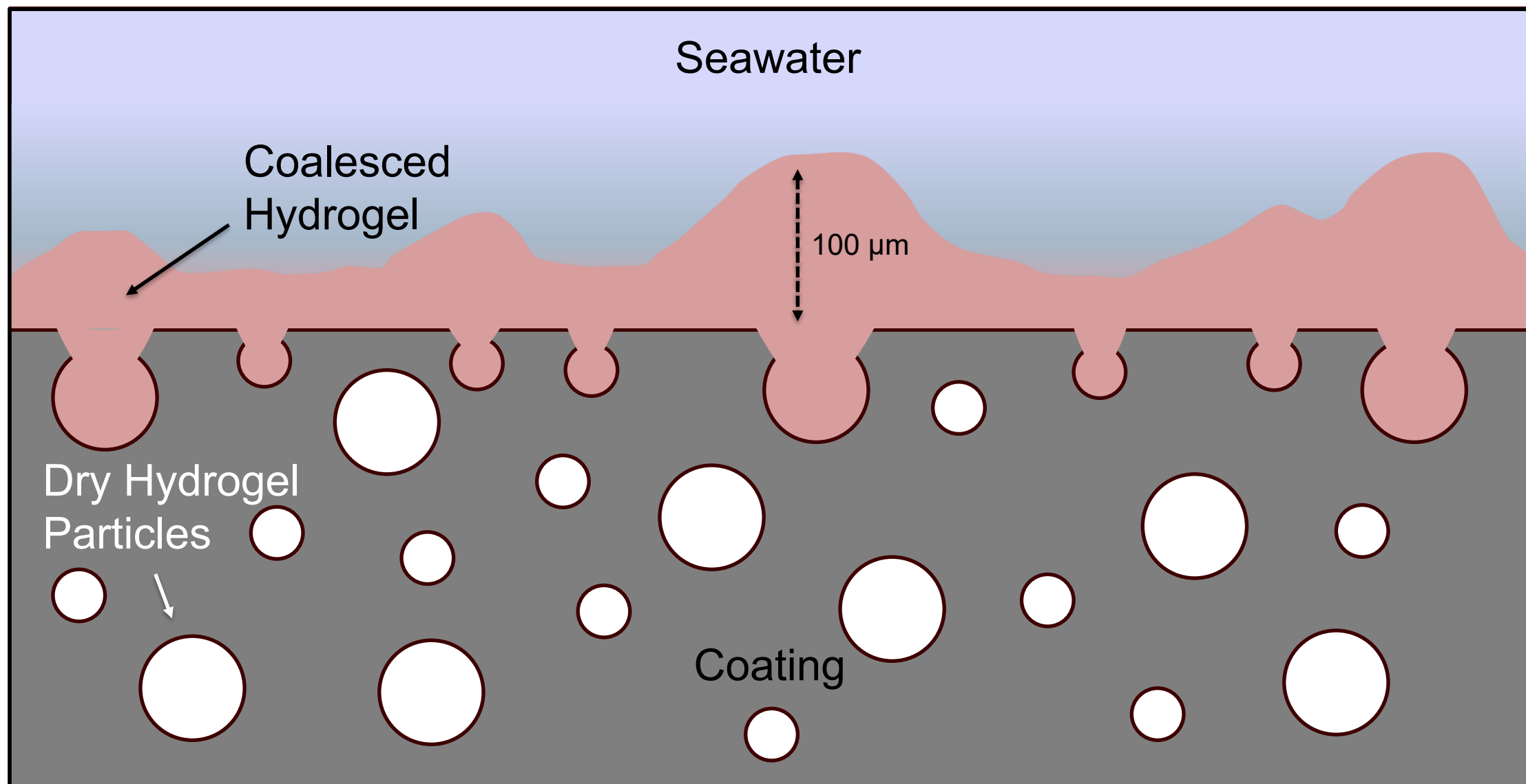
Background – Gel formation



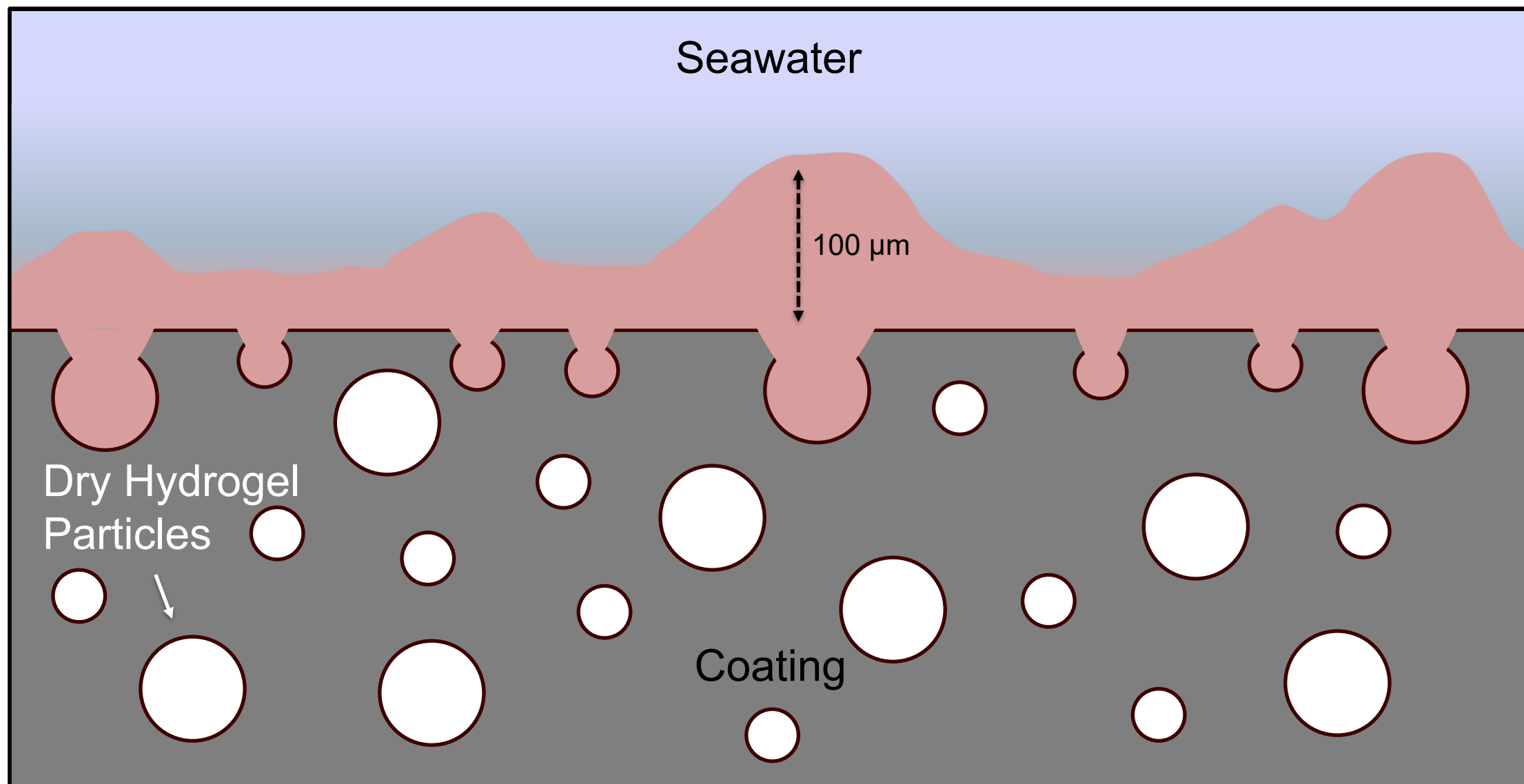
Background – Gel formation



Background – Gel formation



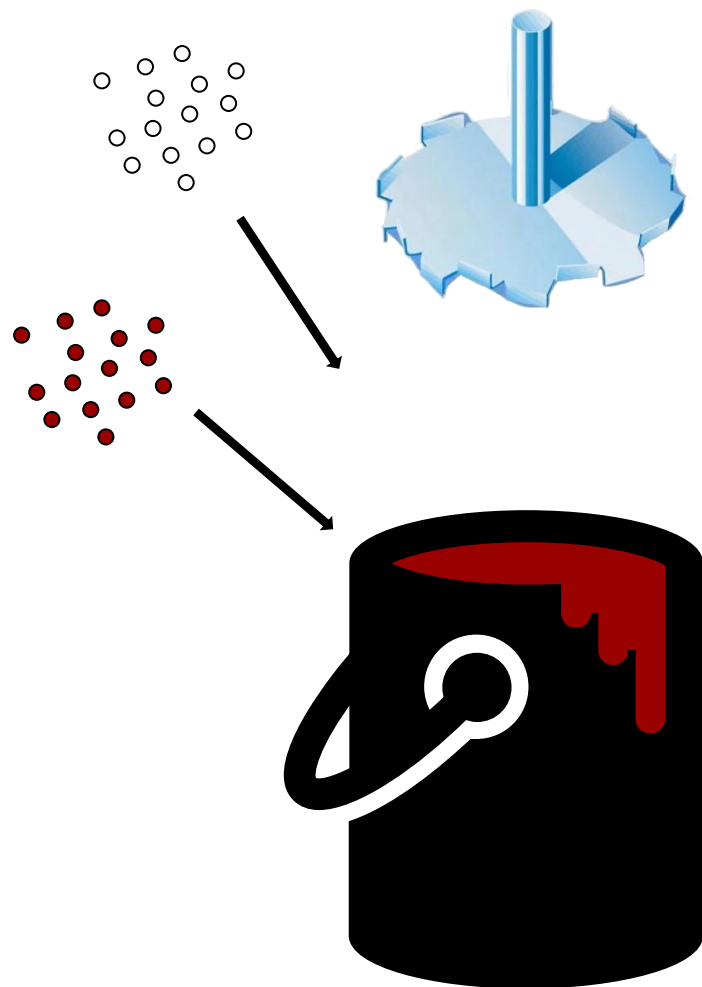
Background – Gel formation



Background – Coating Production and Immersion



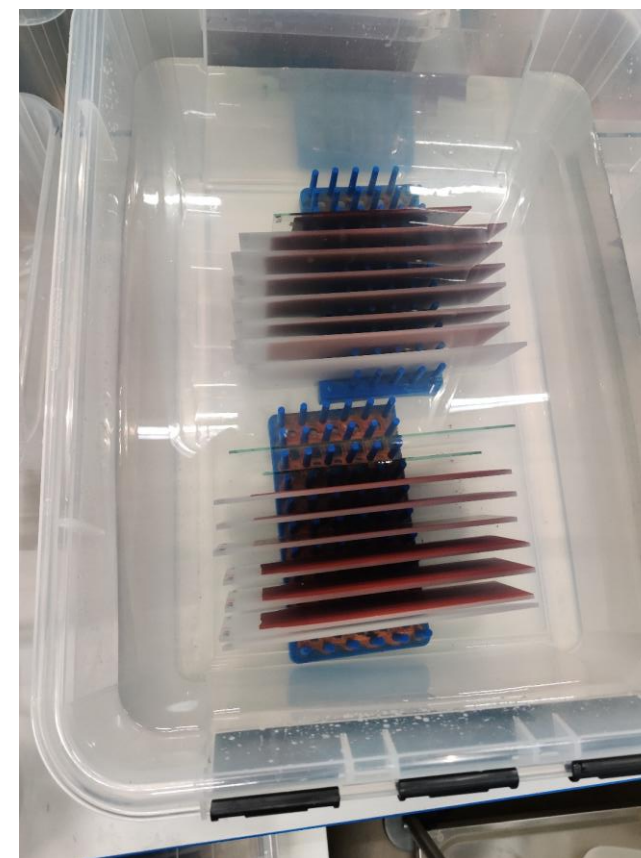
1. **Xanthan Gum** (powder) is dispersed together with binder and other pigment with high speed dissolver



2. **Application** on acrylic panel



3. **Immersion** in artificial seawater

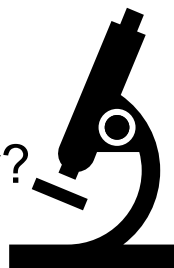




- **Goal:** Hydrogel visualization and thickness distribution measurement
- **Challenges:**

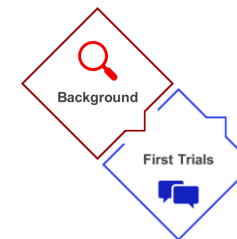
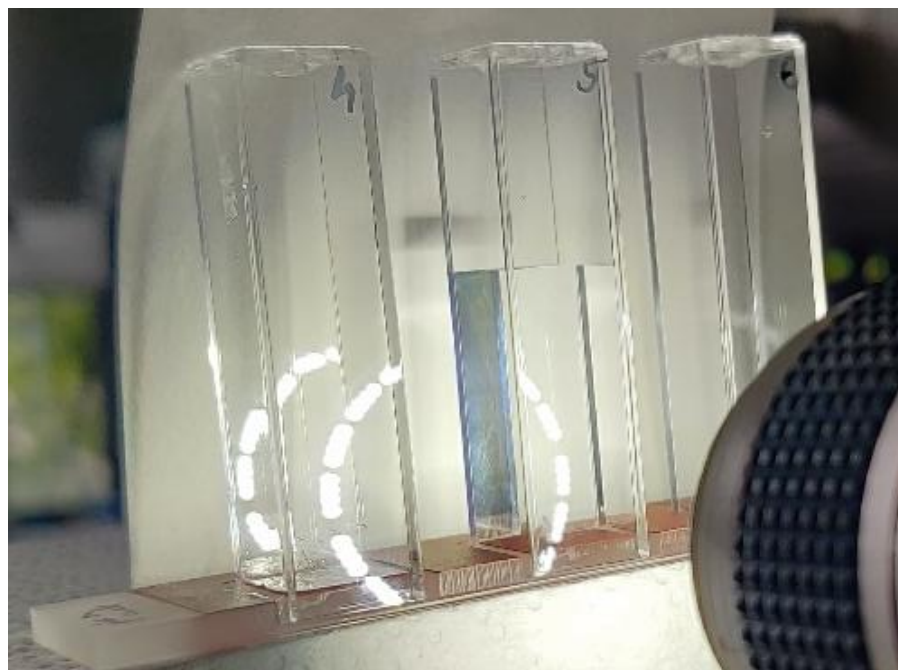
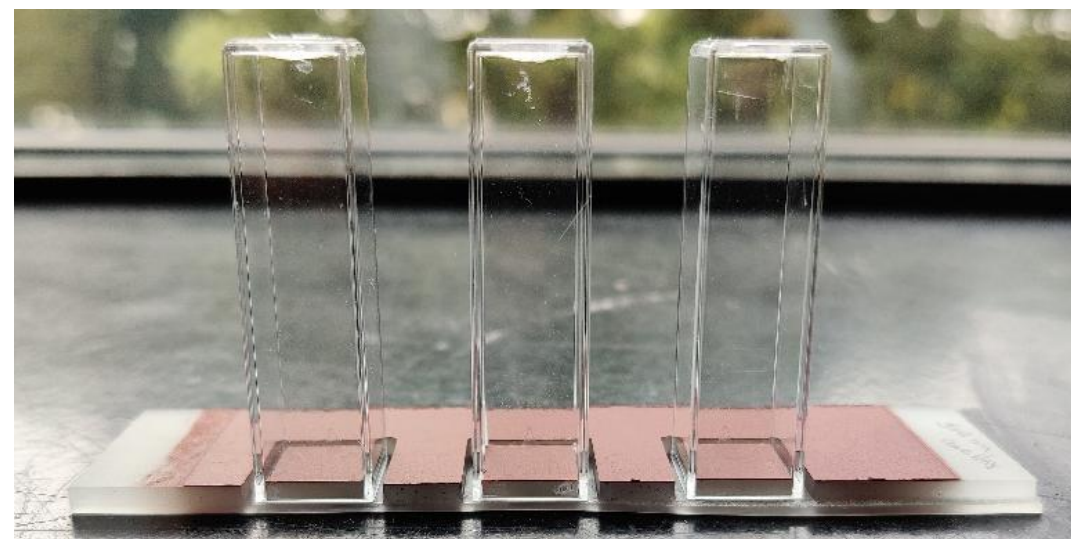
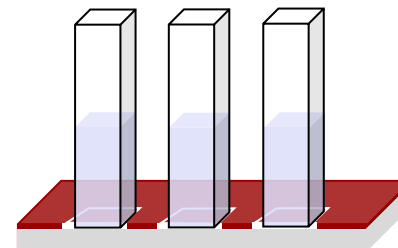
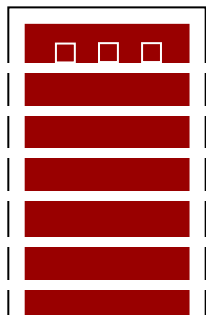
Hydrogel Property	Characterization Requirements
Thin (around 100 μm)	High resolution
Fragile (gel strength around 300 Pa)	Non-destructively
Transparent	High contrast
Fast drying	Measurement while being immersed in sea water

→ **First Trial Approach:** Light Microscopy?

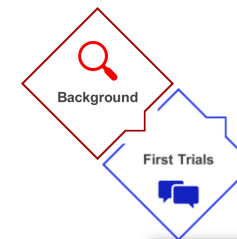




(Crosscut) Microscopy



First Trial: Light Microscopy



30x mag in artificial seawater

500 μm

Hydrogel

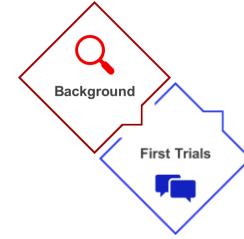
Coating

00 min 00 s

Speed Factor: 10x

30x mag in DI water

500 μm



Low contrast to
environment

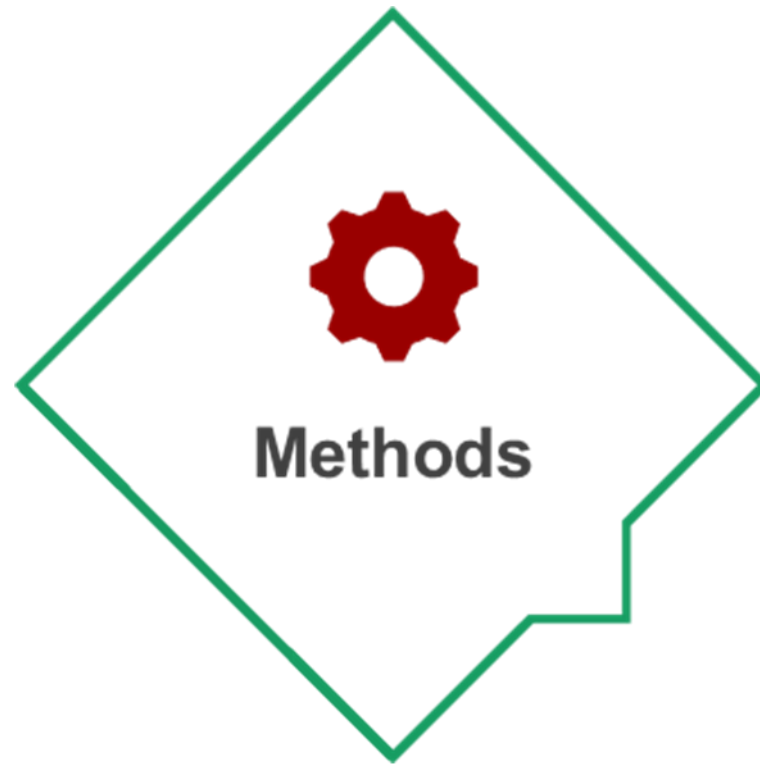
Conclusion...

Rough edge
due to milling

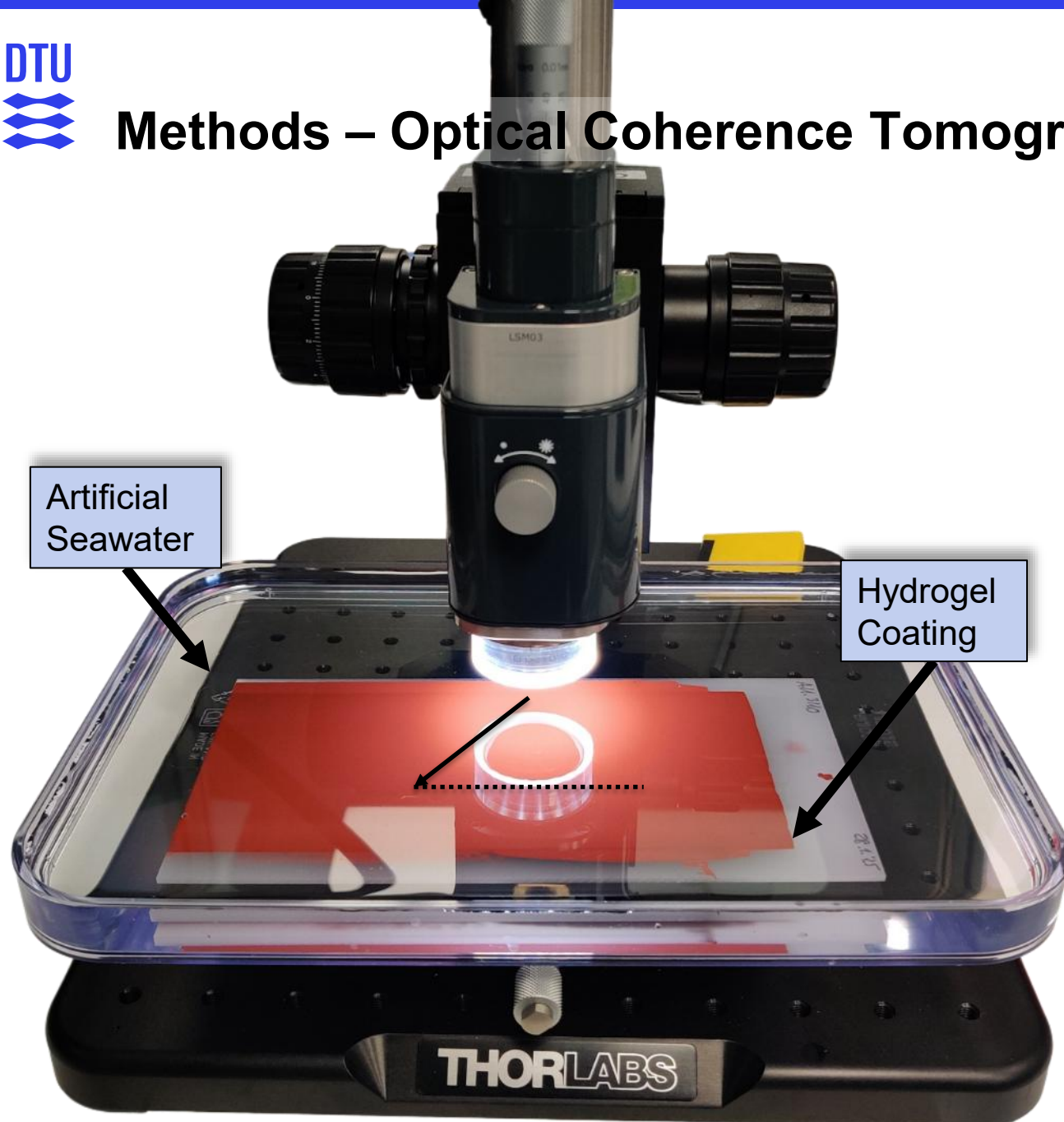
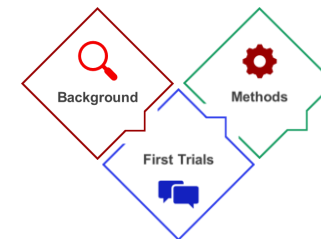
Seeing through the
hydrogel landscape

→ Tough to detect and quantify the hydrogel

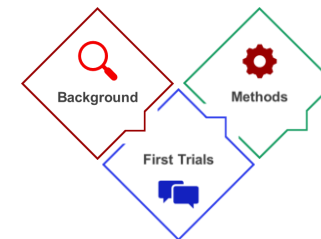




Methods – Optical Coherence Tomography (OCT)



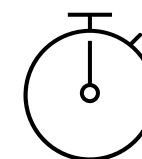
Methods – Optical Coherence Tomography (OCT)



Scans the coating with a laser under water



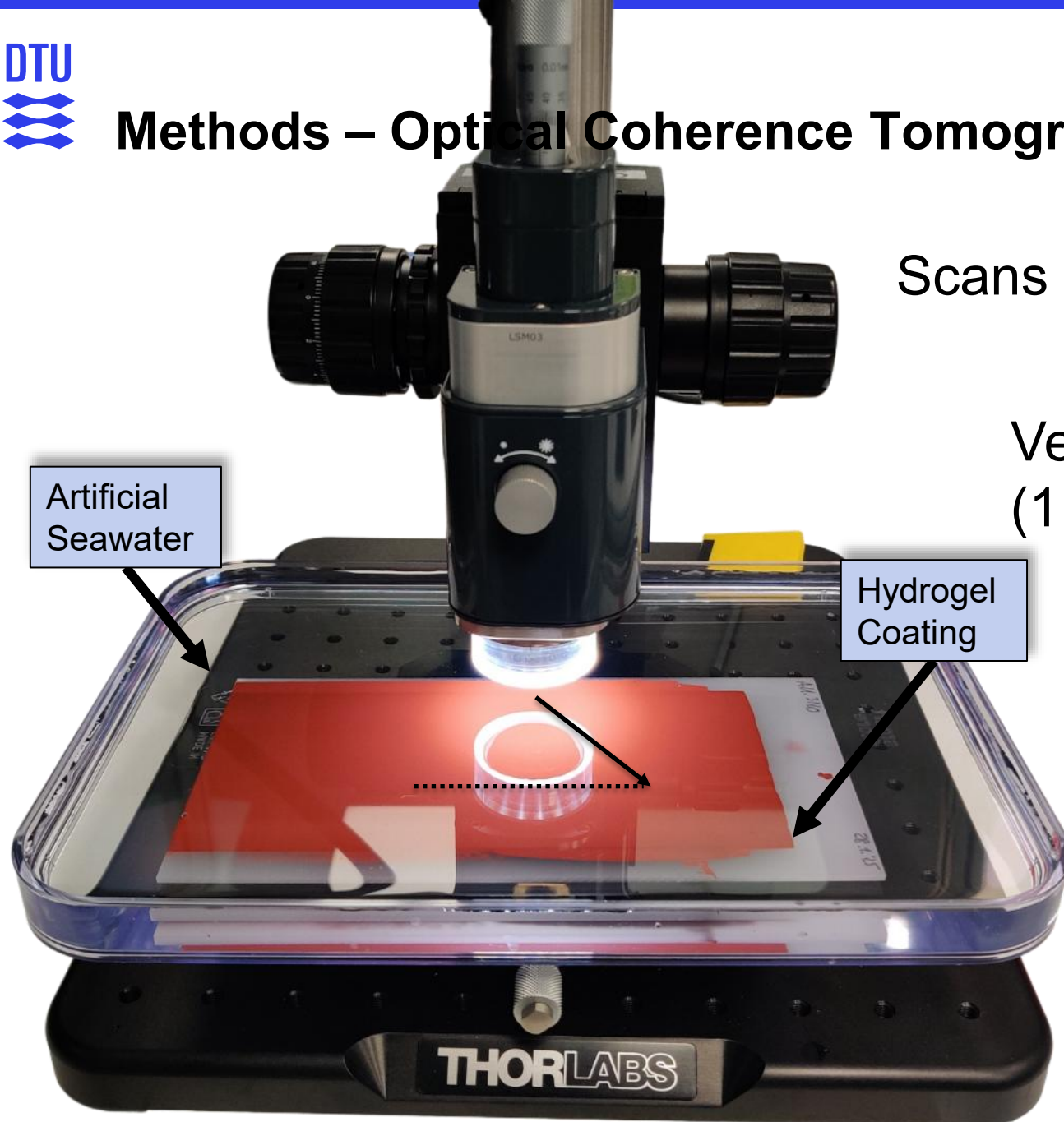
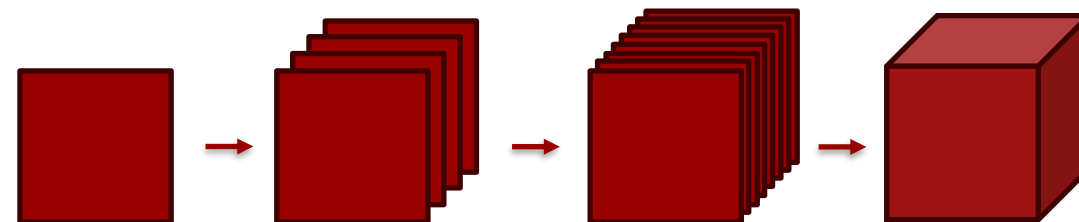
Very fast measurement
(1 cm in under 1 second)



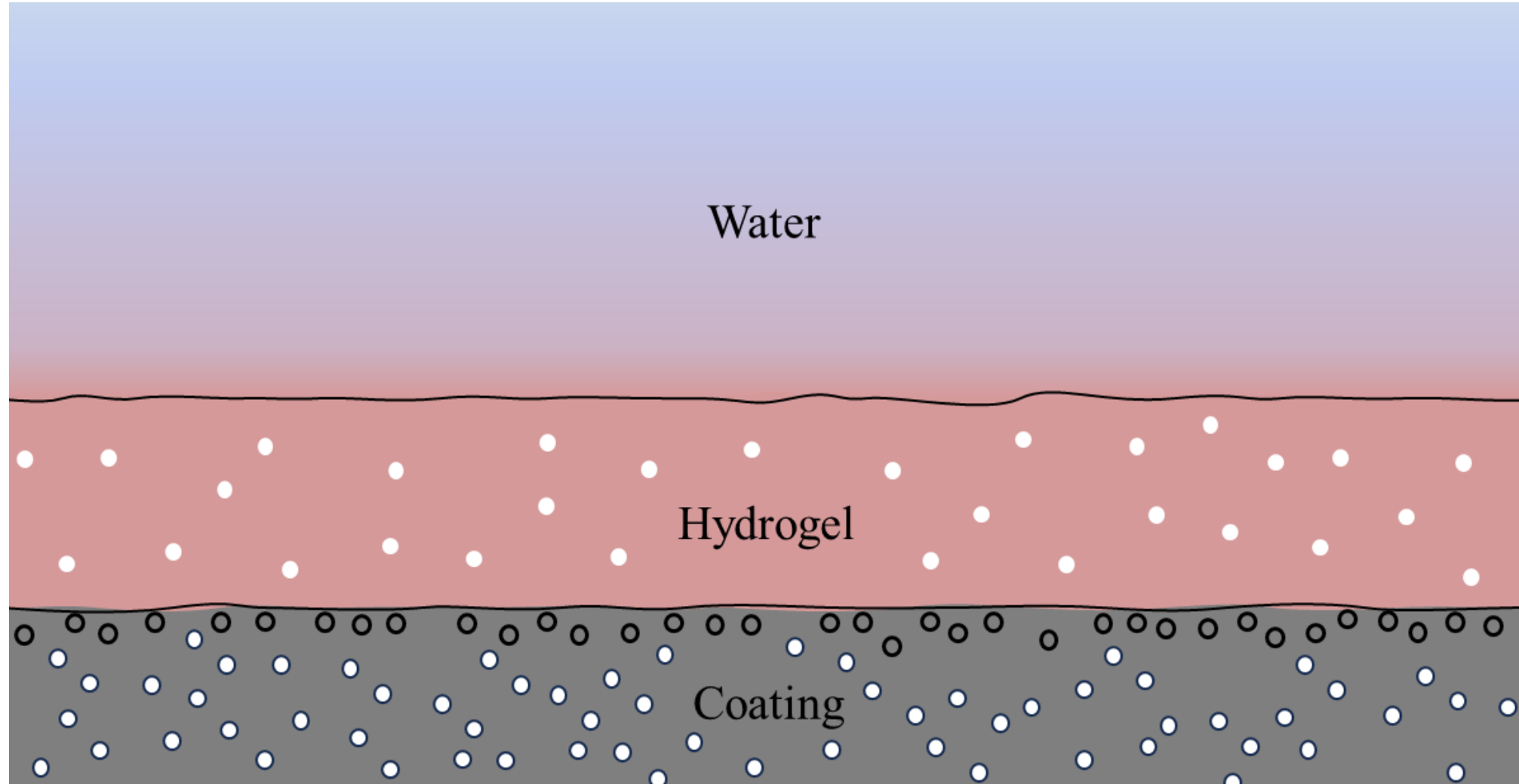
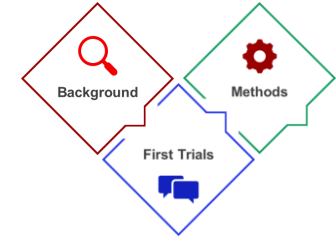
Micrometer-scale depth imaging



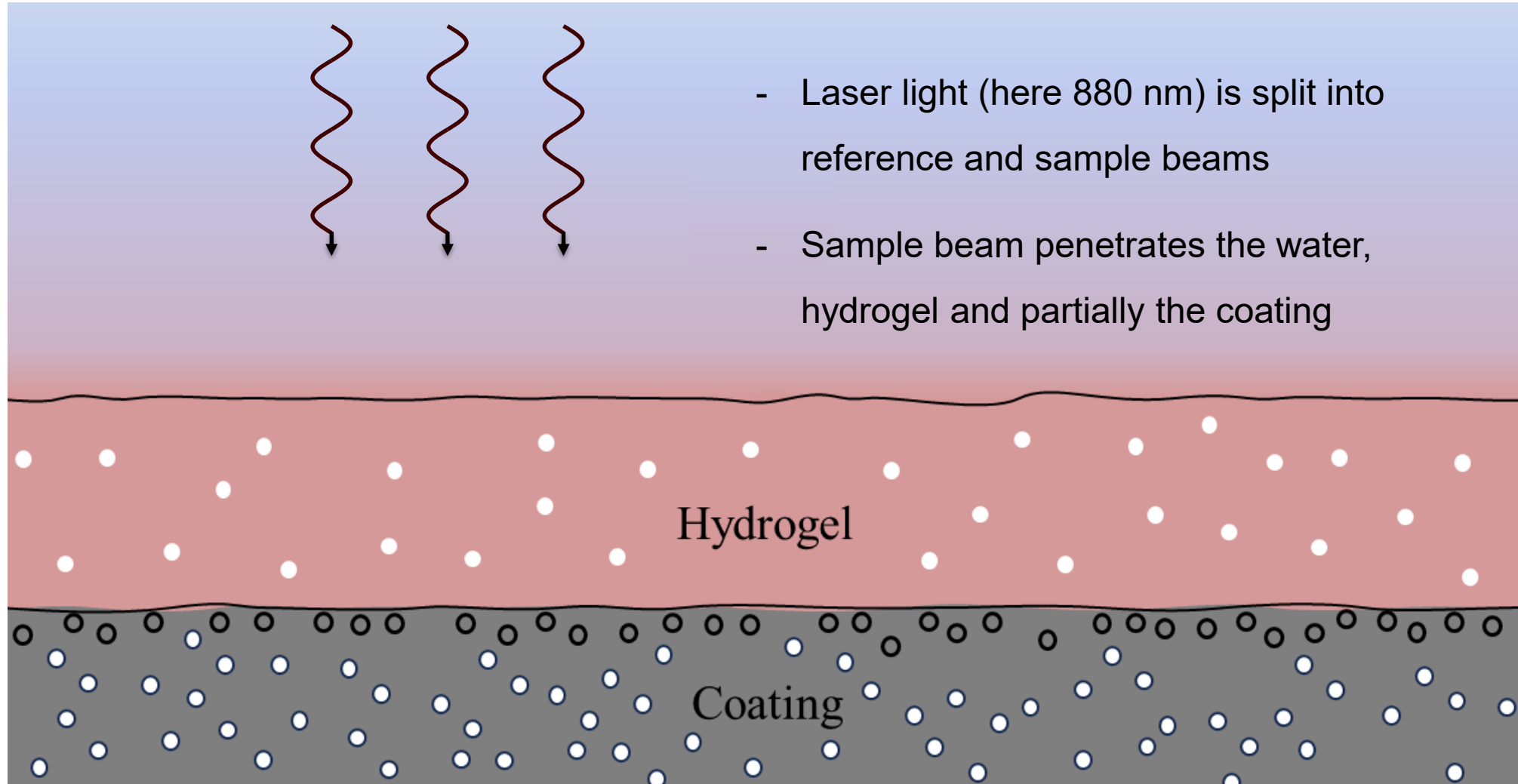
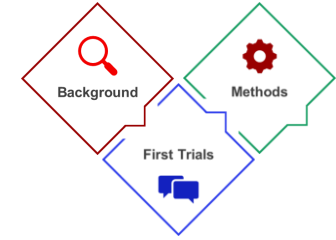
Creates 2D (crosscut-like, B-Scan)
or 3D images (C-Scan)

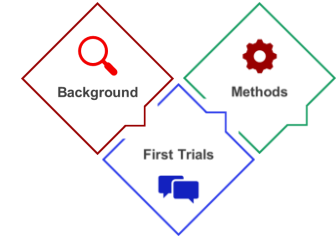


Methods – Optical Coherence Tomography (OCT)



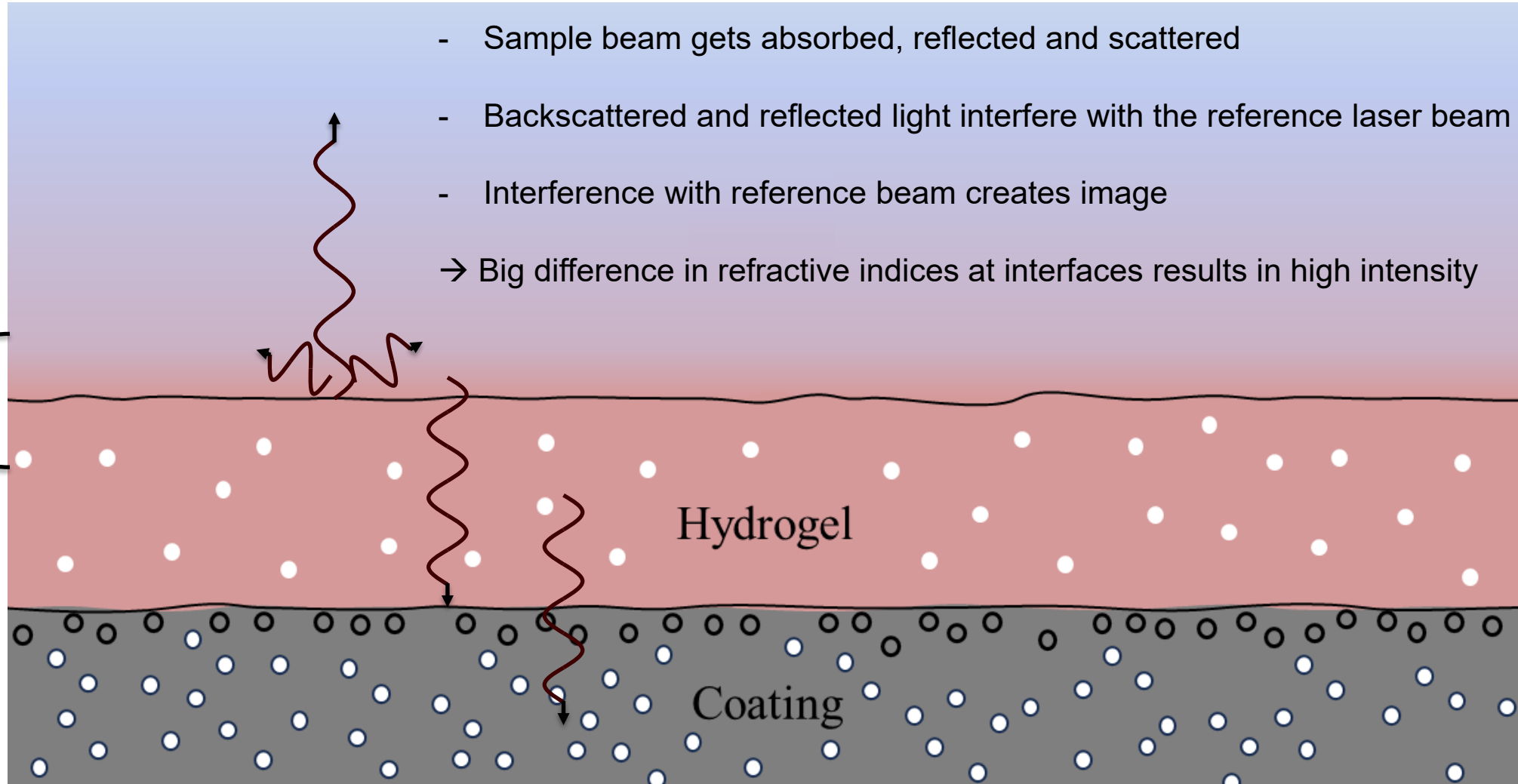
Methods – Optical Coherence Tomography (OCT)



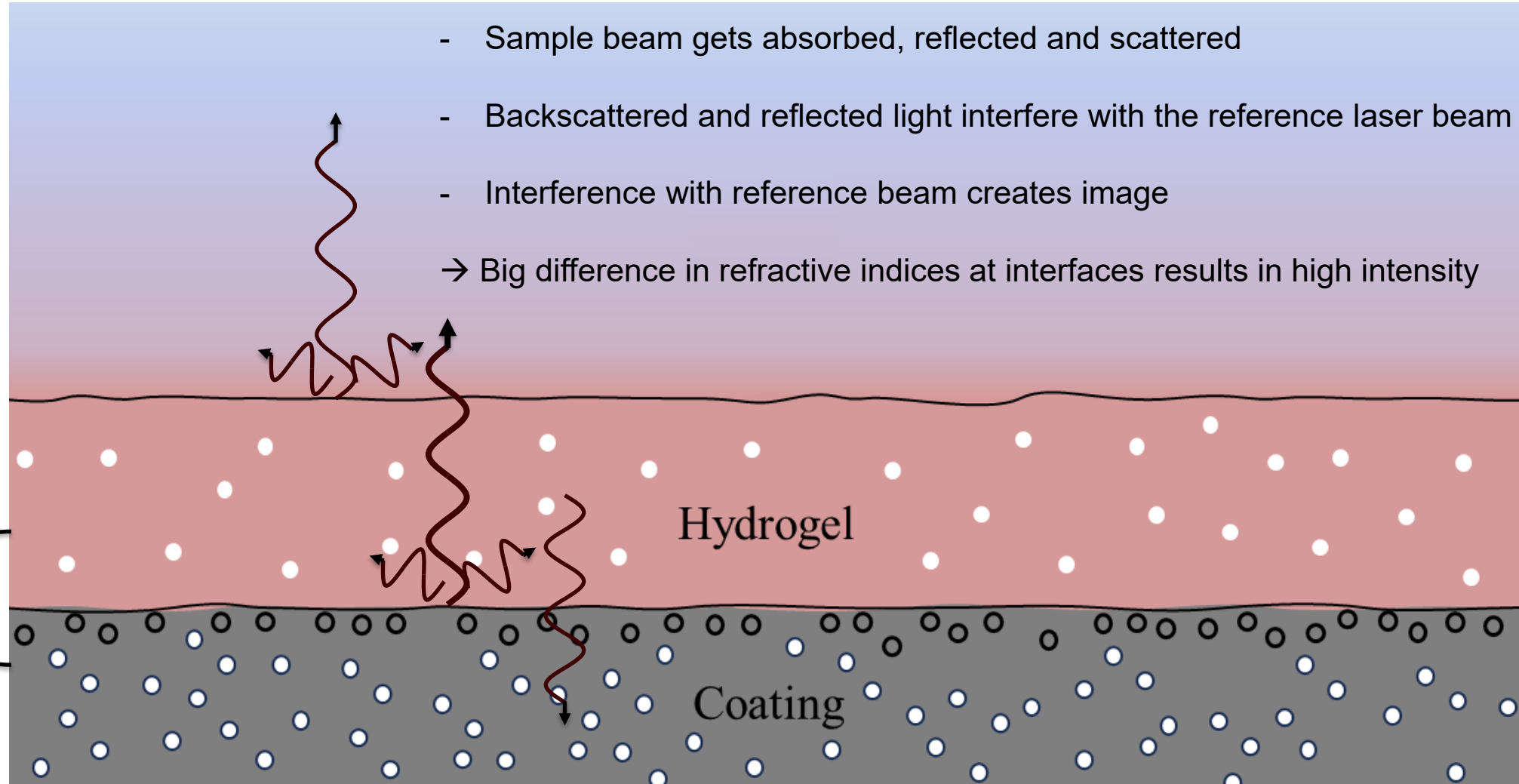


- Sample beam gets absorbed, reflected and scattered
 - Backscattered and reflected light interfere with the reference laser beam
 - Interference with reference beam creates image
- Big difference in refractive indices at interfaces results in high intensity

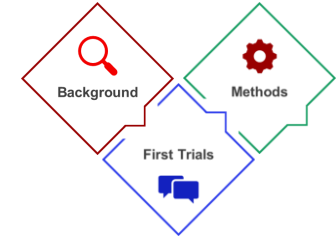
Δn is small = weak signal



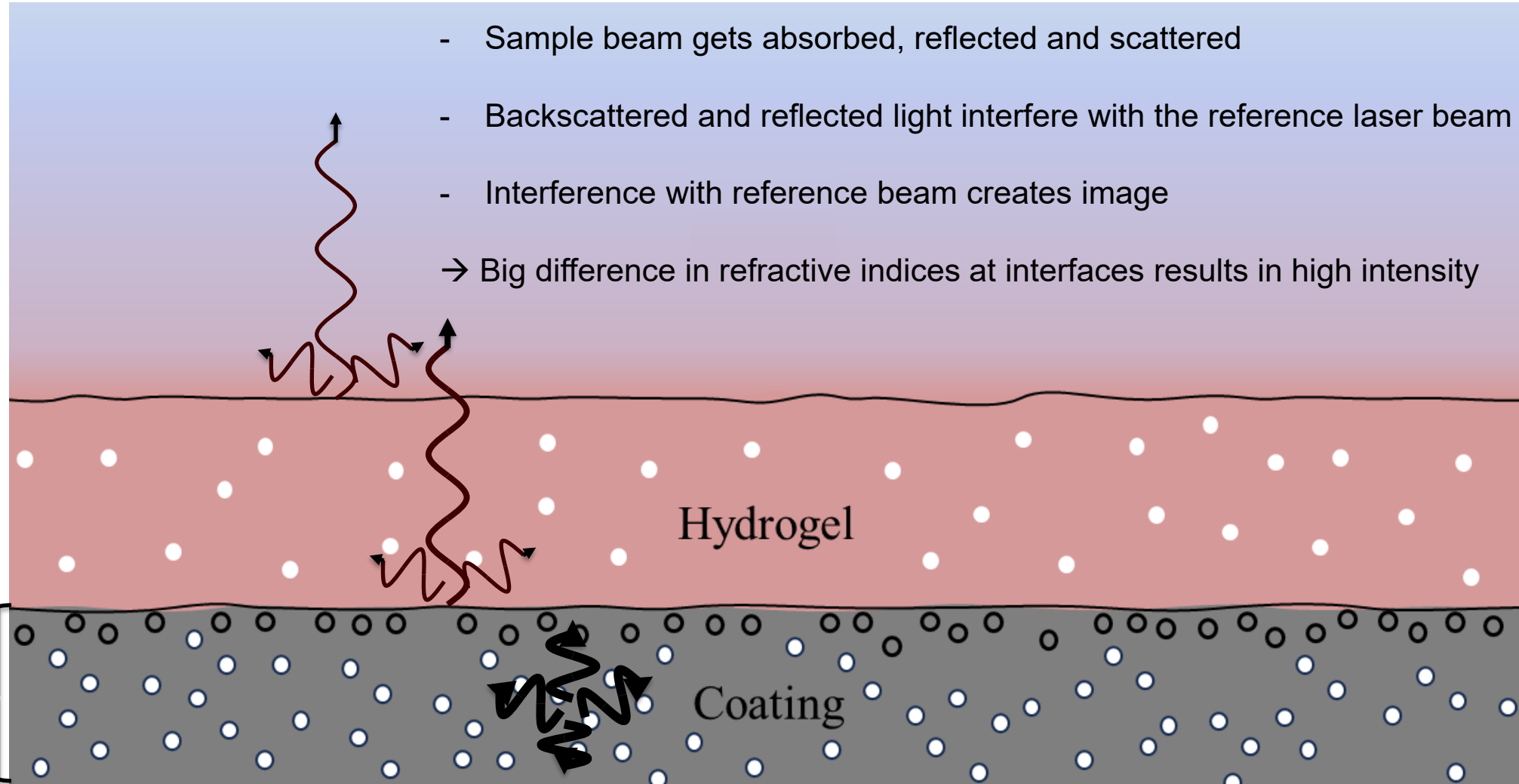
- Sample beam gets absorbed, reflected and scattered
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Δn is big
= strong
signal

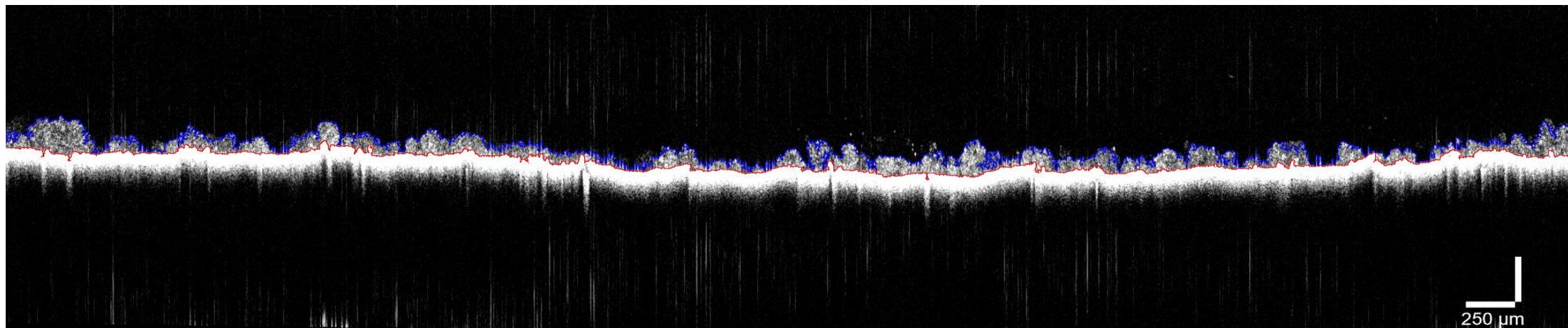
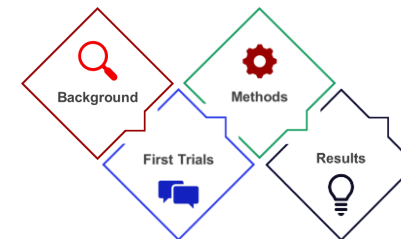


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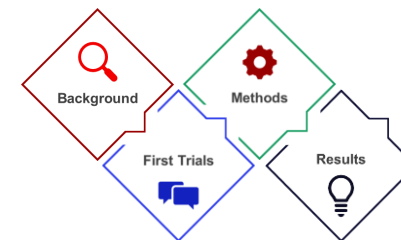
Strongly
scattering
pigments
= no depth
resolution





Quantification of hydrogel parameter with
B-Scans on different spots on panel

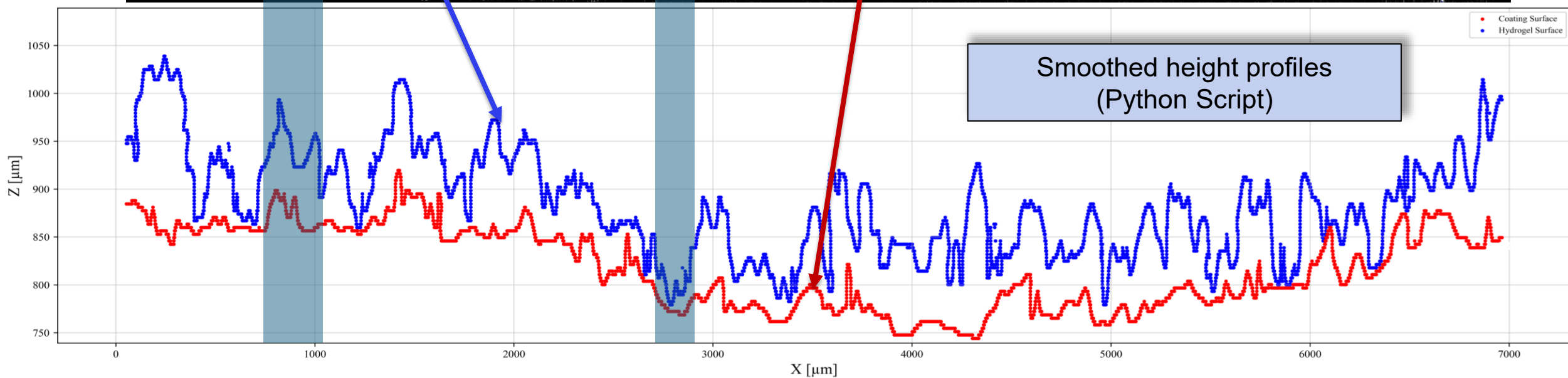
Detection of **interfaces**, short wavelength **smoothing**
and **XZ coordinate extraction** with ImageJ

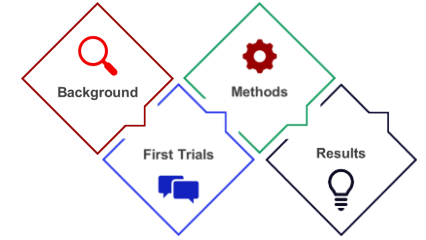


Hydrogel

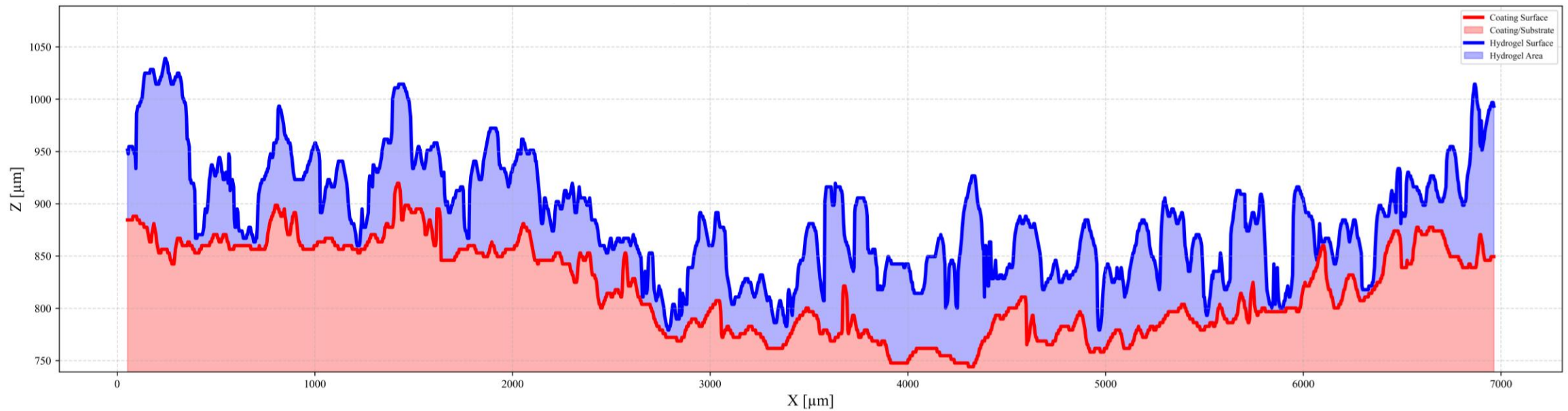
No gel on
coating

Coating

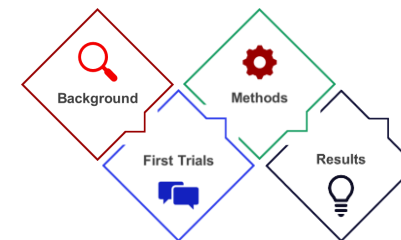
Coating damage
where hydrogel
particles were250 μm 



Height profiles with filled overhangs (more than one Z-coordinates)

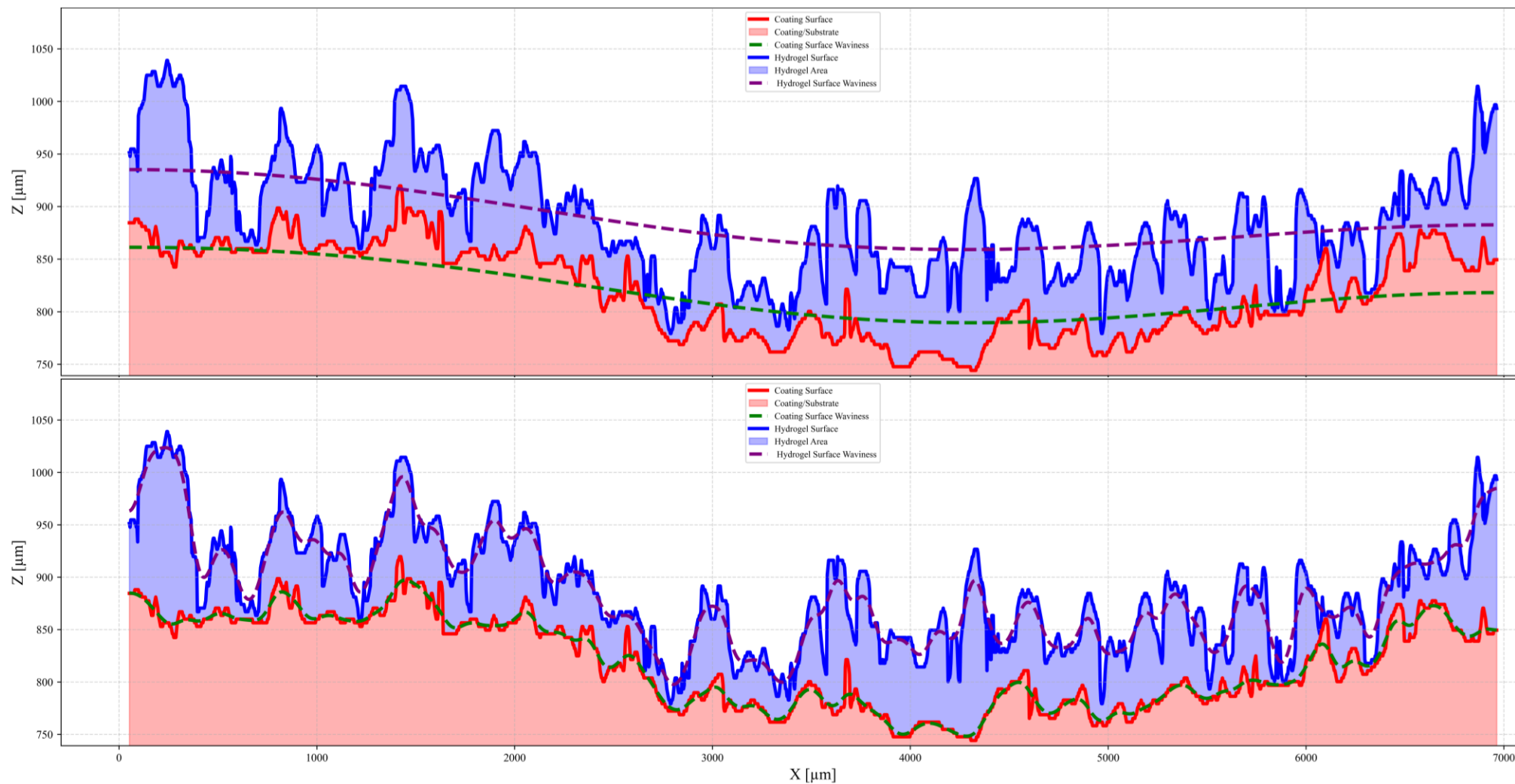


Results - Waviness correction

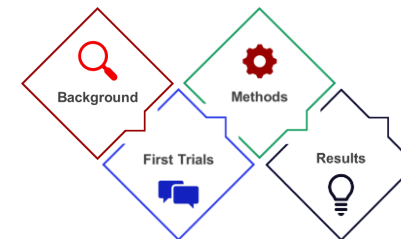


Waviness cut-off wavelength:

8.0 mm



0.25 mm

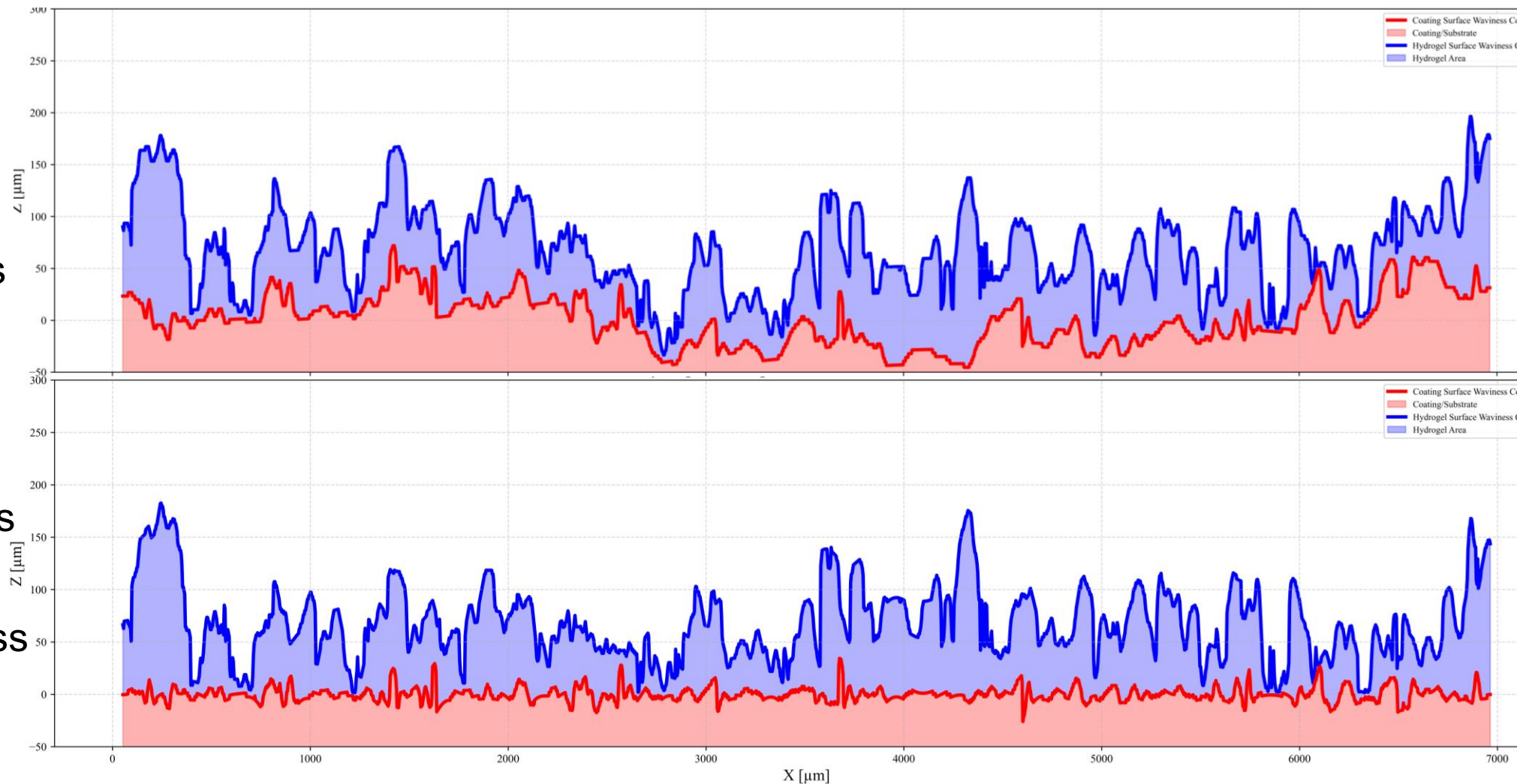


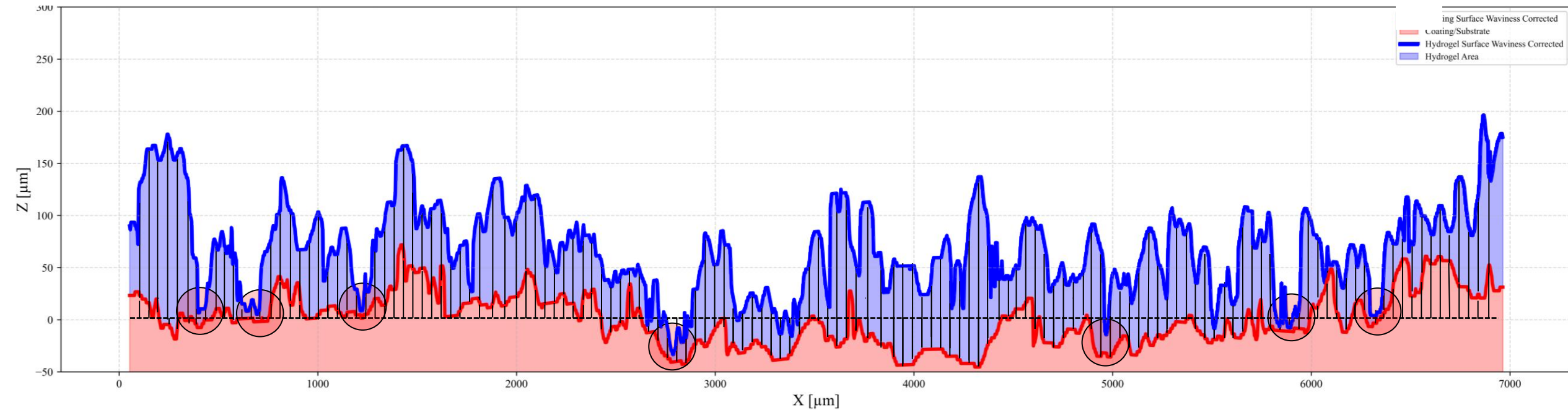
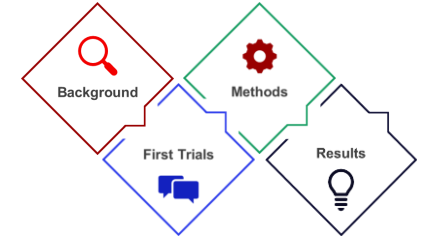
8.0 mm

- Coating Waviness
 W_q : 24.0 μm
- Coating roughness
 R_q : 24.8 μm

0.25 mm

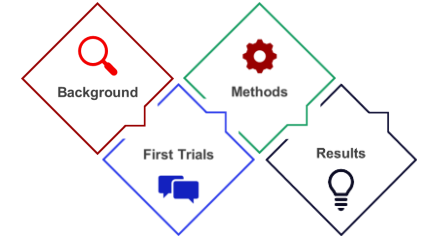
- Coating Waviness
 W_q : 41.5 μm
- Coating roughness
 R_q : 6.9 μm





Analysis of parameter

- **Coating Roughness R_q** (Root mean square roughness) → Distance between height and base line squared for each pixel
- **Hydrogel Height Distribution & Area** → Distance between coating and hydrogel interface for each pixel
- **Hydrogel Coverage** → Coating length without hydrogel



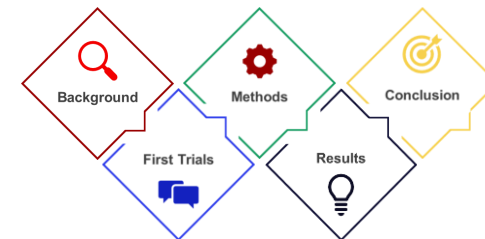
Limitations

- Hydrogel thickness **under 15 μm increases uncertainty** for quantification
(hydrogel/coating ImageJ workflows are conducted separately hence border detection errors add up)
- **Resolution limit** of the OCT with the given setup is around **4 μm**
- Low contrast images due growing **out of focus** with unclear interface are difficult to handle
→ E.g., hydrogel system in DI water

DI water

500 μm



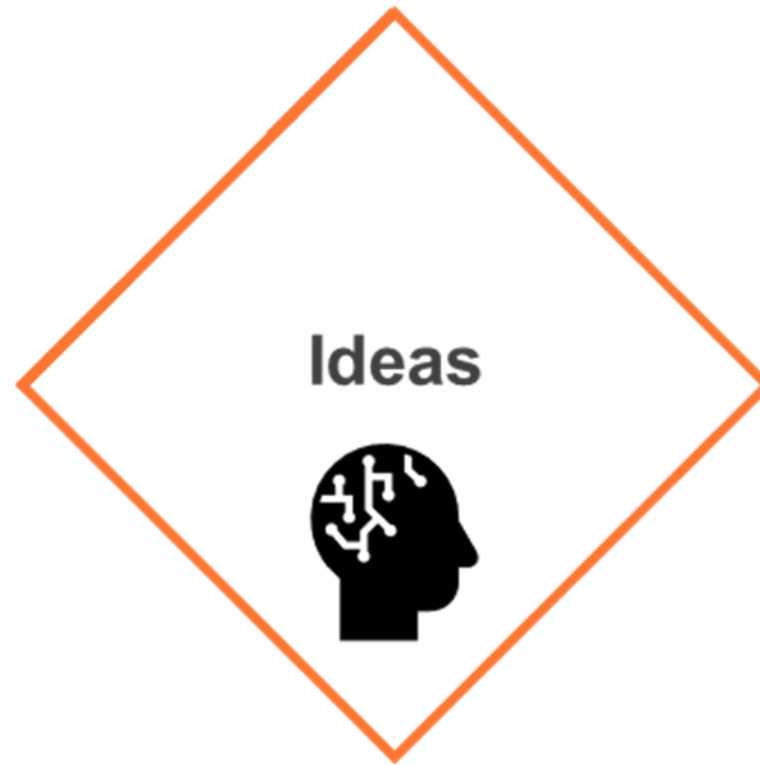


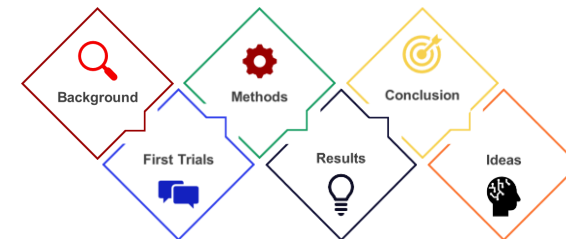
OCT visualizes gel formation process which is needed to understand and improve the hydrogel coating system

- Real-time monitoring of hydrogel systems
- Hydrogel thickness and volume
- Hydrogel coverage
- Roughness of the coating

Rheological hydrogel analysis adds value by revealing mechanical properties

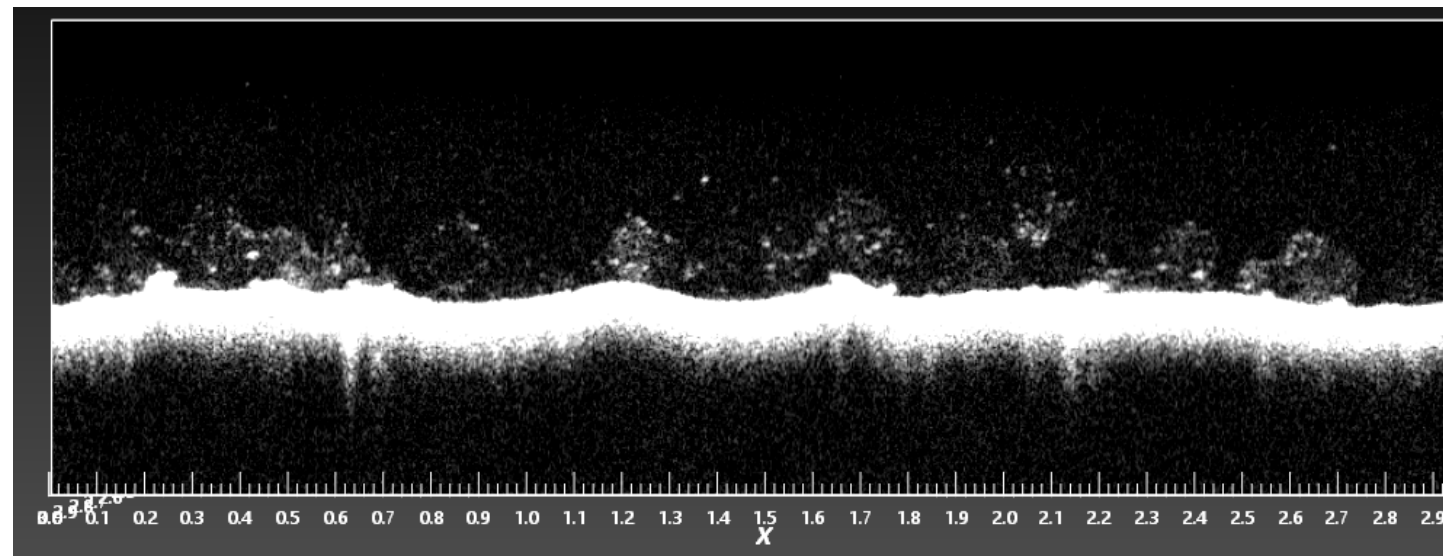
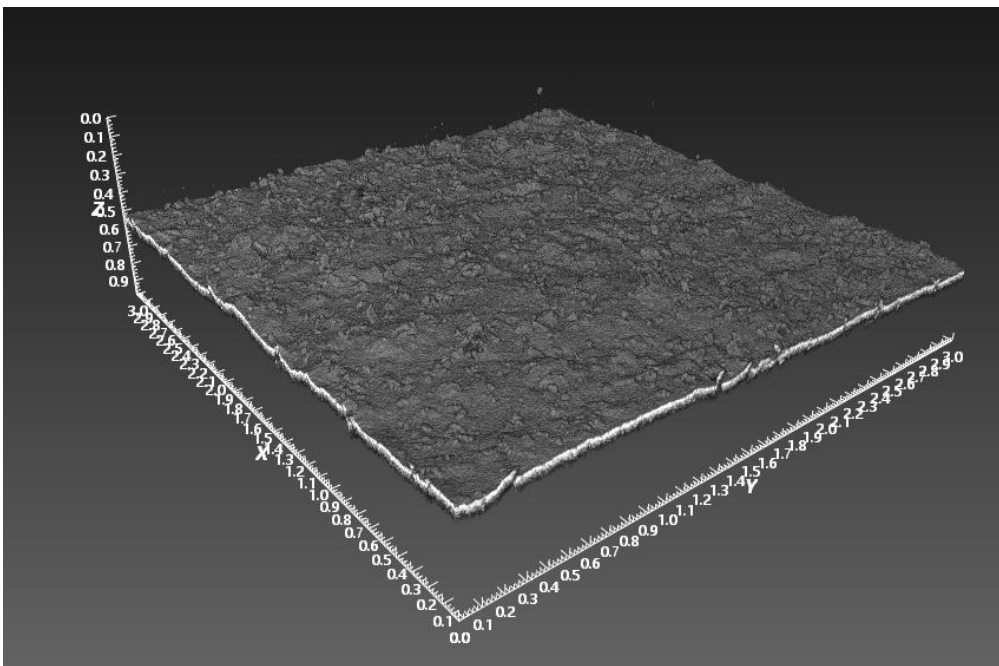
- Together, these methods provide robust characterization of hydrogels and can be applied to other systems





Process Optimization

- Quantify overhang structures
- Refining coating/hydrogel interface detection and full automation for live quantification
- Applying C-Scan analysis to be independent of homogeneity assumption across the sample



Thank You!

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