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# Development of Antibacterial Metal Oxide Thin Films for Neurostimulation Applications Using Atomic Layer Deposition

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Metal oxide thin films with antibacterial properties can be deposited via atomic layer deposition (ALD) on the surface of neurostimulation and cardiac rhythm management electrodes to prevent risks of post implantation infections and bacteria colonization. In this work, we report on the development of antibacterial platinum-iridium electrodes using a two-step process. Electrodes are first hierarchically restructured using femtosecond-laser hierarchical surface restructuring technology and then ALD is used to deposit ultrathin metal oxides of ZnO on hierarchically restructured electrodes. Structural, chemical, and mechanical properties of ZnO films were studied using X-ray diffraction, X-ray photoelectron spectroscopy, energy dispersive X-ray spectroscopy, scanning electron microscopy and nanoindentation. The antibacterial properties of the ALD-coated electrodes were also studied, particularly, the killing effect on the two common types of bacteria (*E. coli* and *S. aureus*) responsible for implantation infections.

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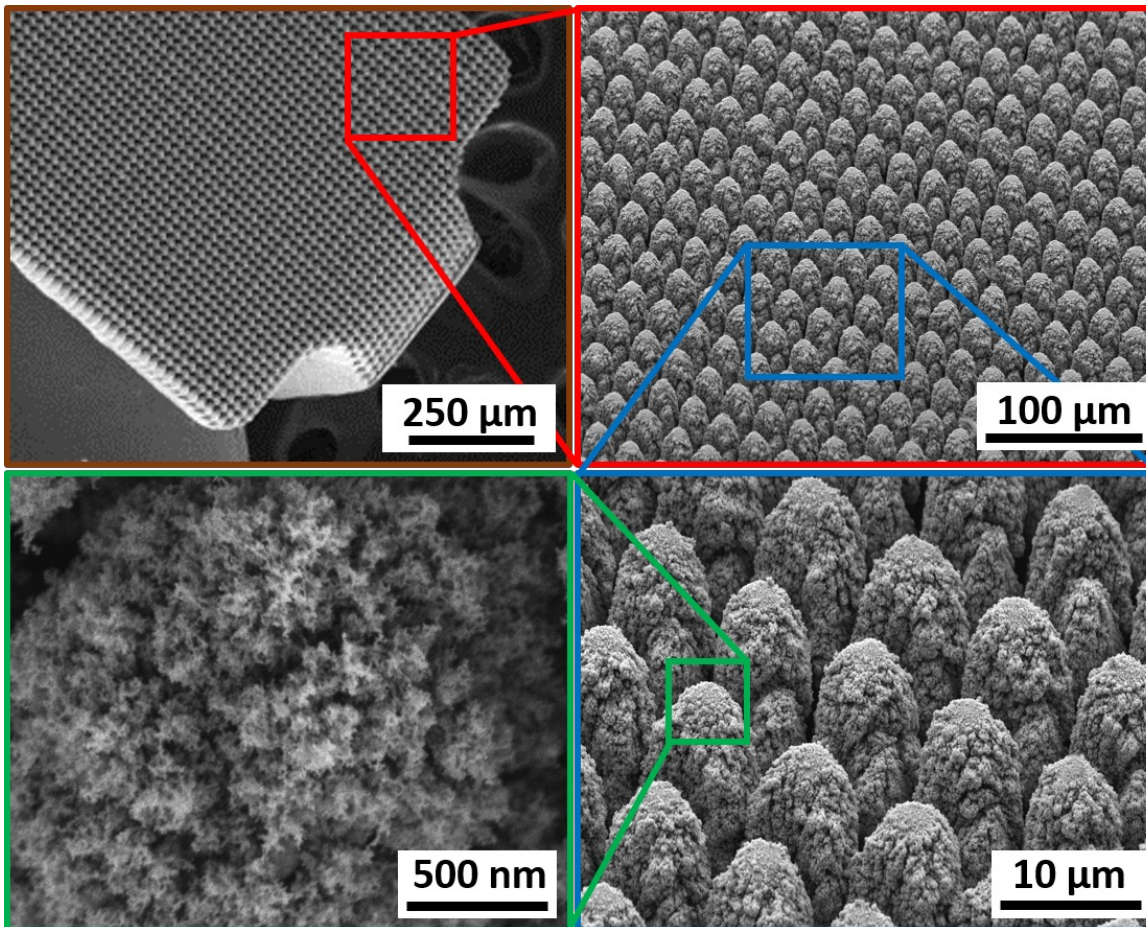
**Daniel Ammerman, Gregory Caputo, Jefferey Hettinger, Rowan University**

**Sahar Elyahoodayan, University of Southern California**

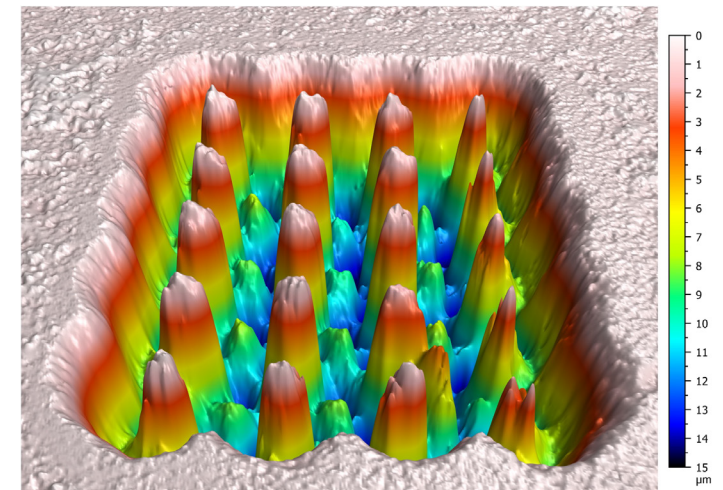
## Outline

- Hierarchical Surface Restructuring (HSR™)
- The need for antibacterial electrodes
- Motivation - Why ALD?
- ALD of CuO and Cu<sub>x</sub>O
- Electrode surface characterization: SEM, AFM, EDS, XRD and XPS
- Electrochemical measurements
- Antibacterial measurements
- Concluding remarks

# Introduction- Hierarchical Surface Restructuring (HSR™)

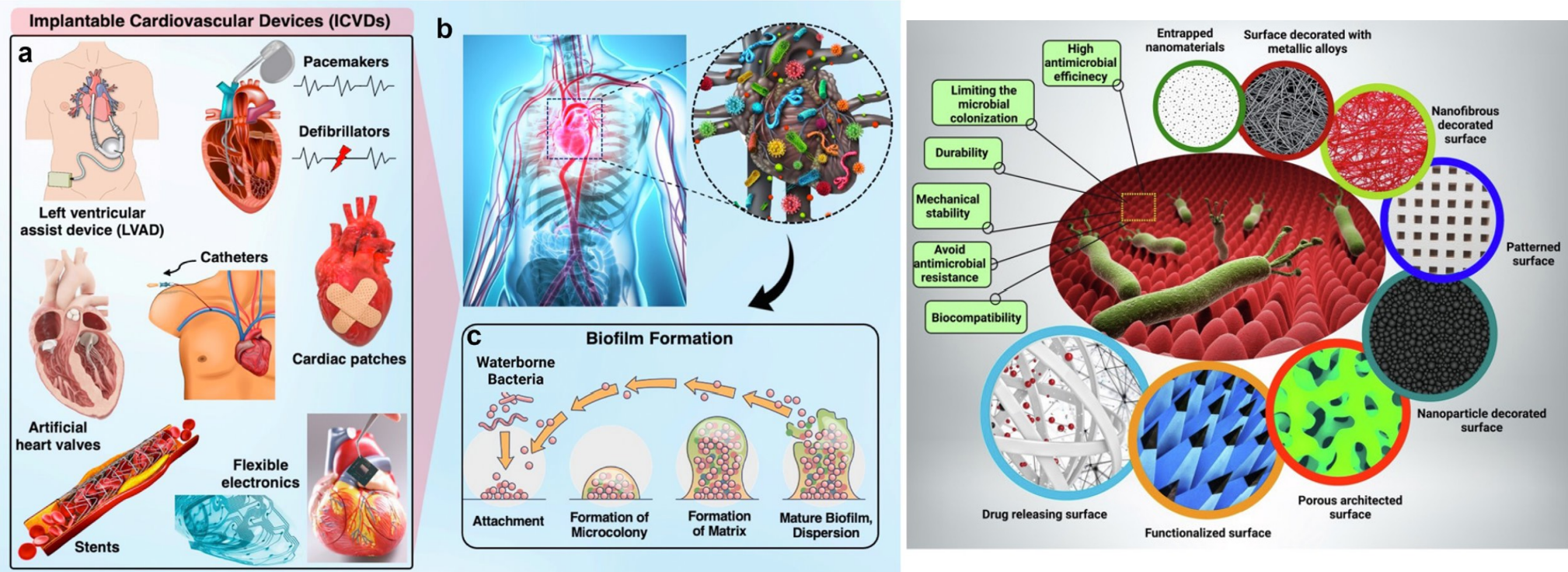


Hierarchical surface structure induced on the surface of a Pt-10Ir alloy electrode used for a paddle-lead spinal cord stimulation electrode array



<https://www.nature.com/articles/s41598-022-18161-4>

# The need for antibacterial electrodes

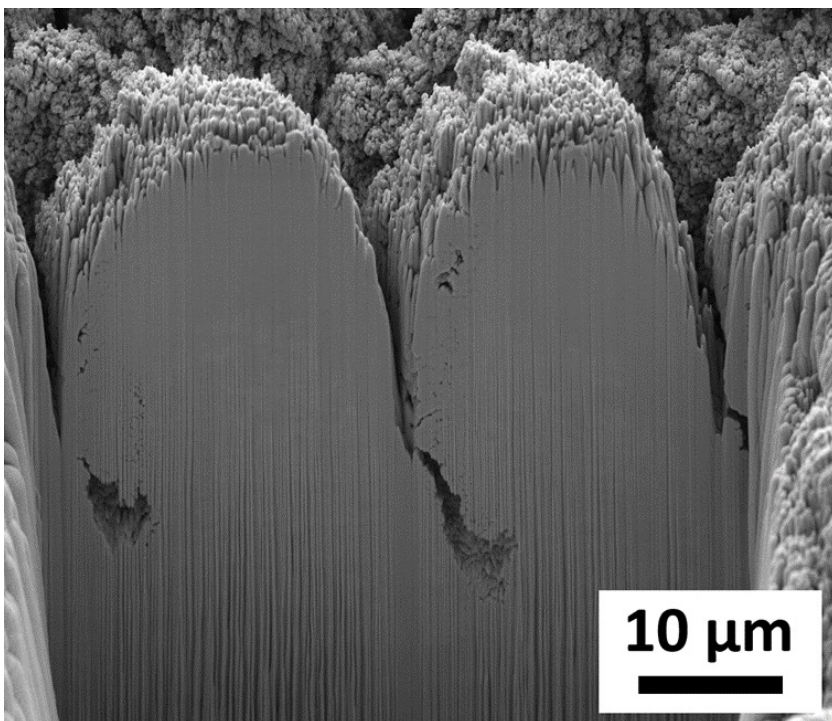


Antimicrobial surfaces for implantable cardiovascular devices. Current Opinion in Biomedical Engineering 23, 100406 (2022)

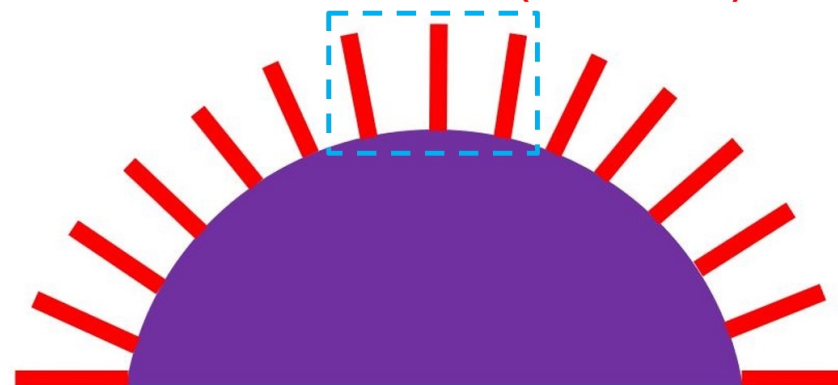
- Implantable devices are highly prone to infection and can even lead to death
- New methods to prevent this are critically required

## Motivation

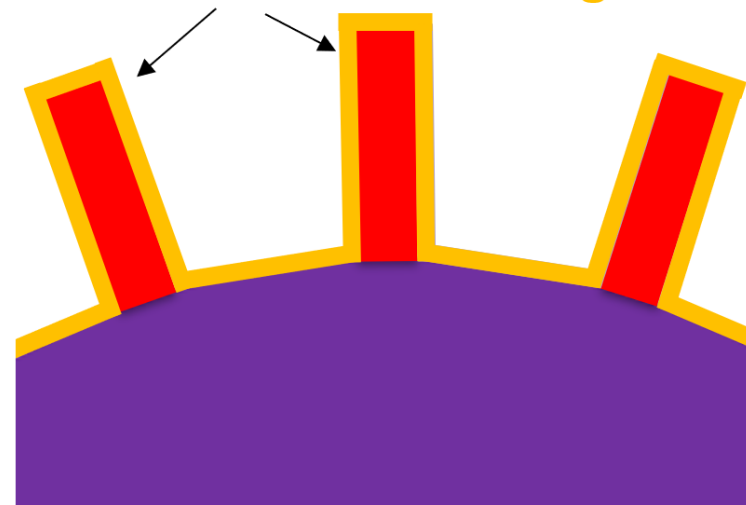
- ❑ **Femtosecond-laser Hierarchical Surface Restructuring (HSR™)**
  - Largely enhancing the electrochemical surface area & electrochemical performance
- ❑ **Antibacterial Antibacterial Coating on HSR™**
  - Forming ultrathin and conformal antibacterial coating on HSR™
  - Having minimal impact on the HSR™ electrochemical performance.



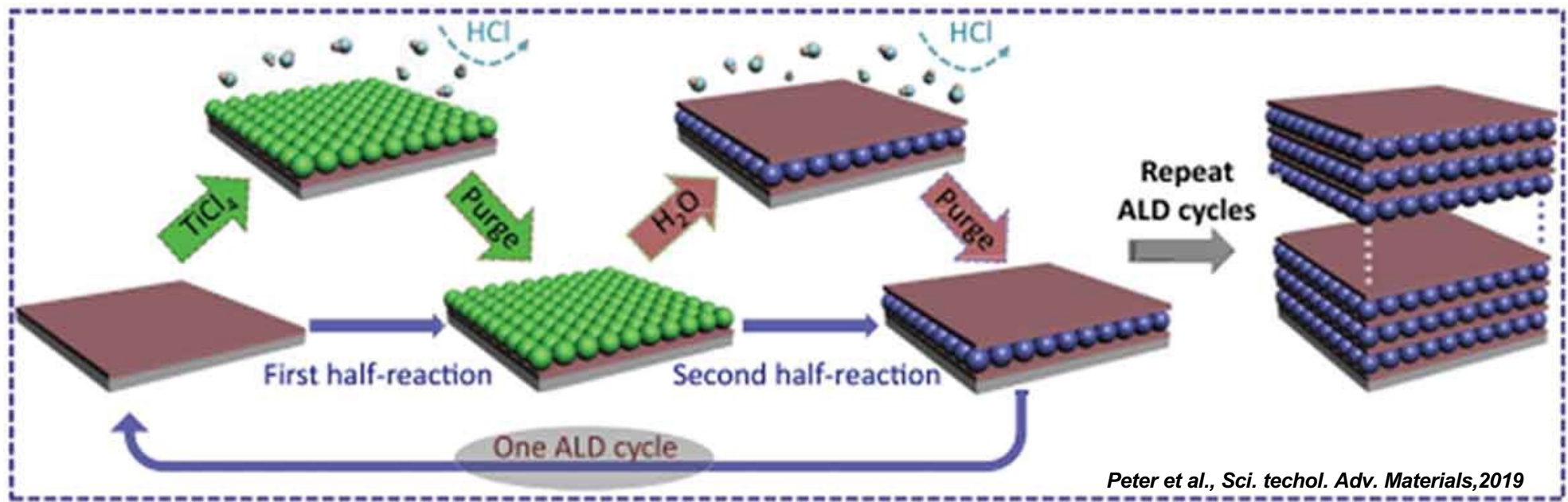
Nano-structure ( $10^{-7}$ - $10^{-9}$  m)



Antibacterial ALD Coating



# Atomic Layer Deposition



## Advantages of ALD Technique

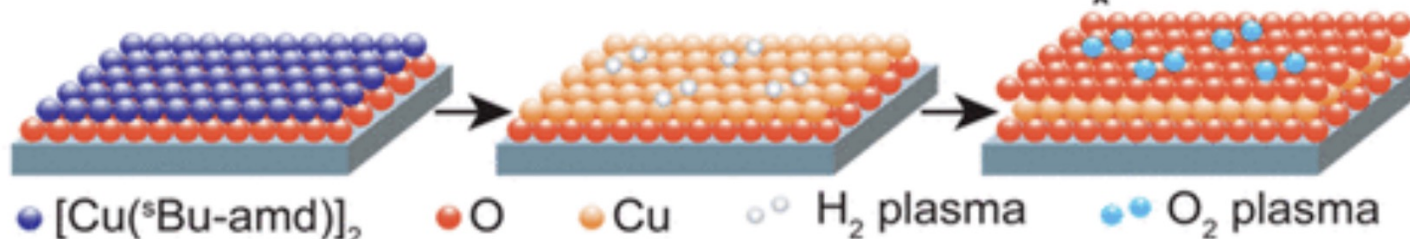
- ❖ High quality films
- ❖ Conformality
- ❖ Uniformity
- ❖ Step coverage

## Disadvantages of ALD Technique

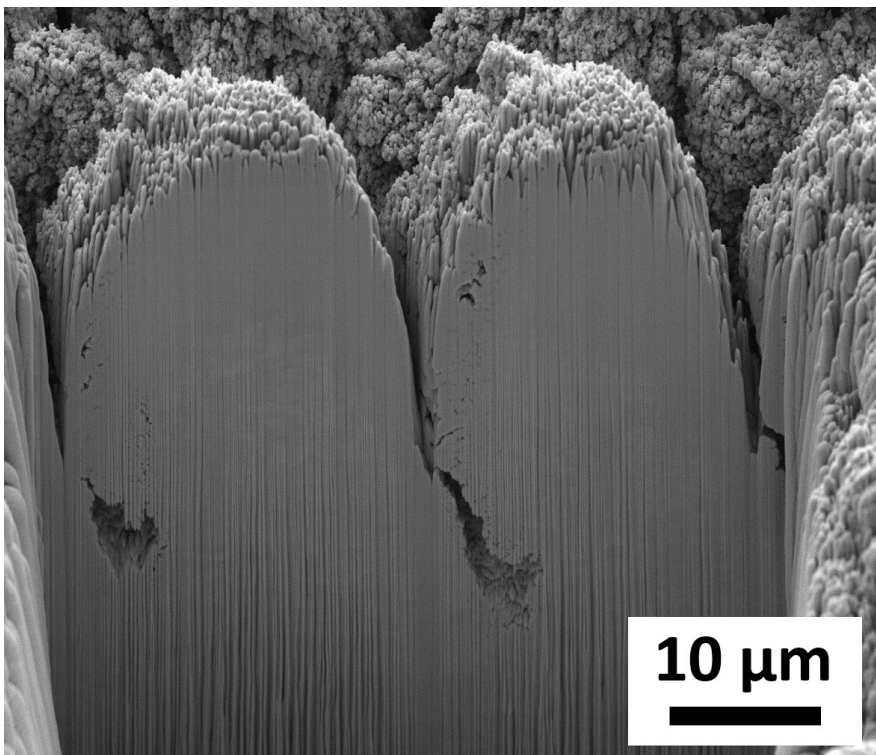
- ❖ Slow Process
- ❖ Precursor Chemistry

# ALD of CuO and Cu<sub>x</sub>O on Pt-10Ir HSR™ electrodes

## Plasma-Enhanced ALD of CuO<sub>x</sub>



*Lenef et al., The Journal of Physical Chemistry, 2021*



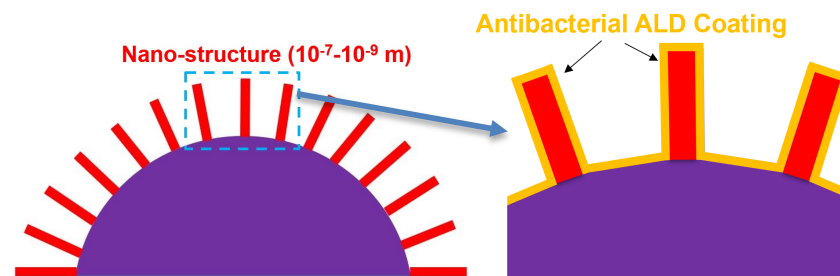
**\*No study available for**

- Antibacterial properties and
- Electrochemical properties of ALD Cu<sub>x</sub>O

**\*Cu<sub>x</sub>O ALD using PEALD is available**

PEALD is not ultra-conformal on Hierarchical surfaces

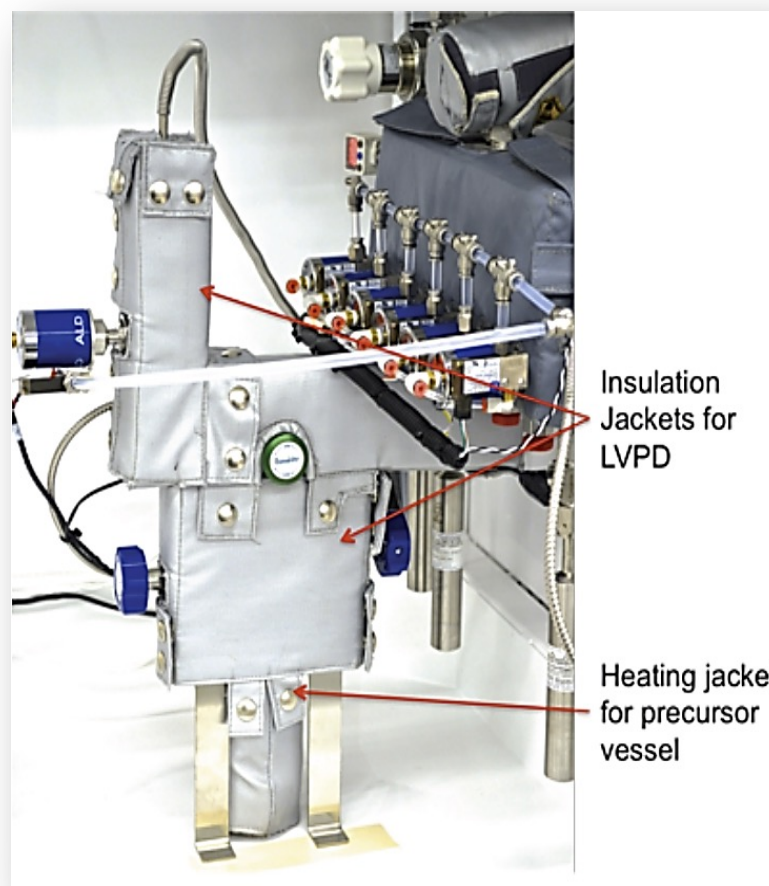
**\*Ozone based ALD – conformal coatings**



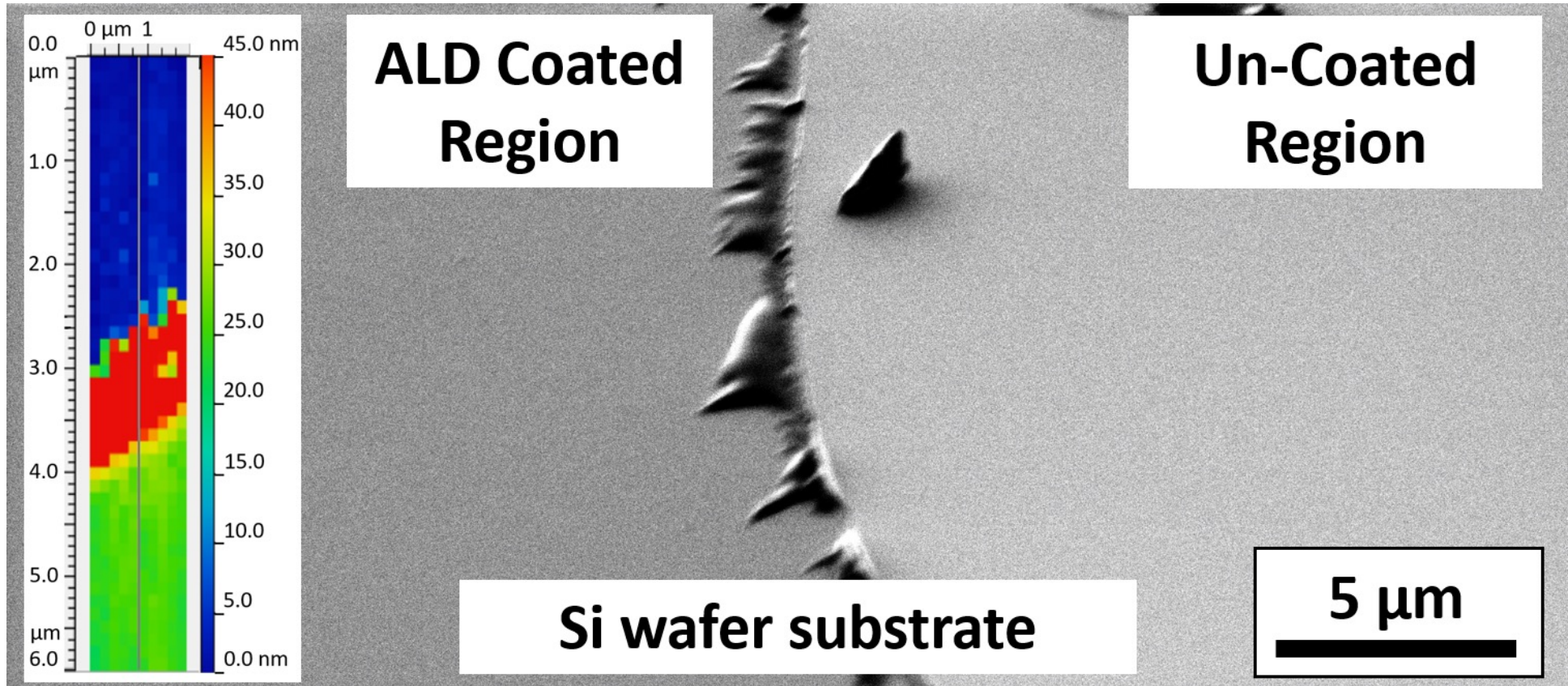
## ALD System Introduction



- Ultratech Fiji G2 ALD system
- Precursor-([Cu(s Bu-amd)]<sub>2</sub>)
- LVPD temp. 125°C
- Three types of samples- Si, Pt10Ir, HSR™-Pt10Ir
- Masked samples - distinguish coated region



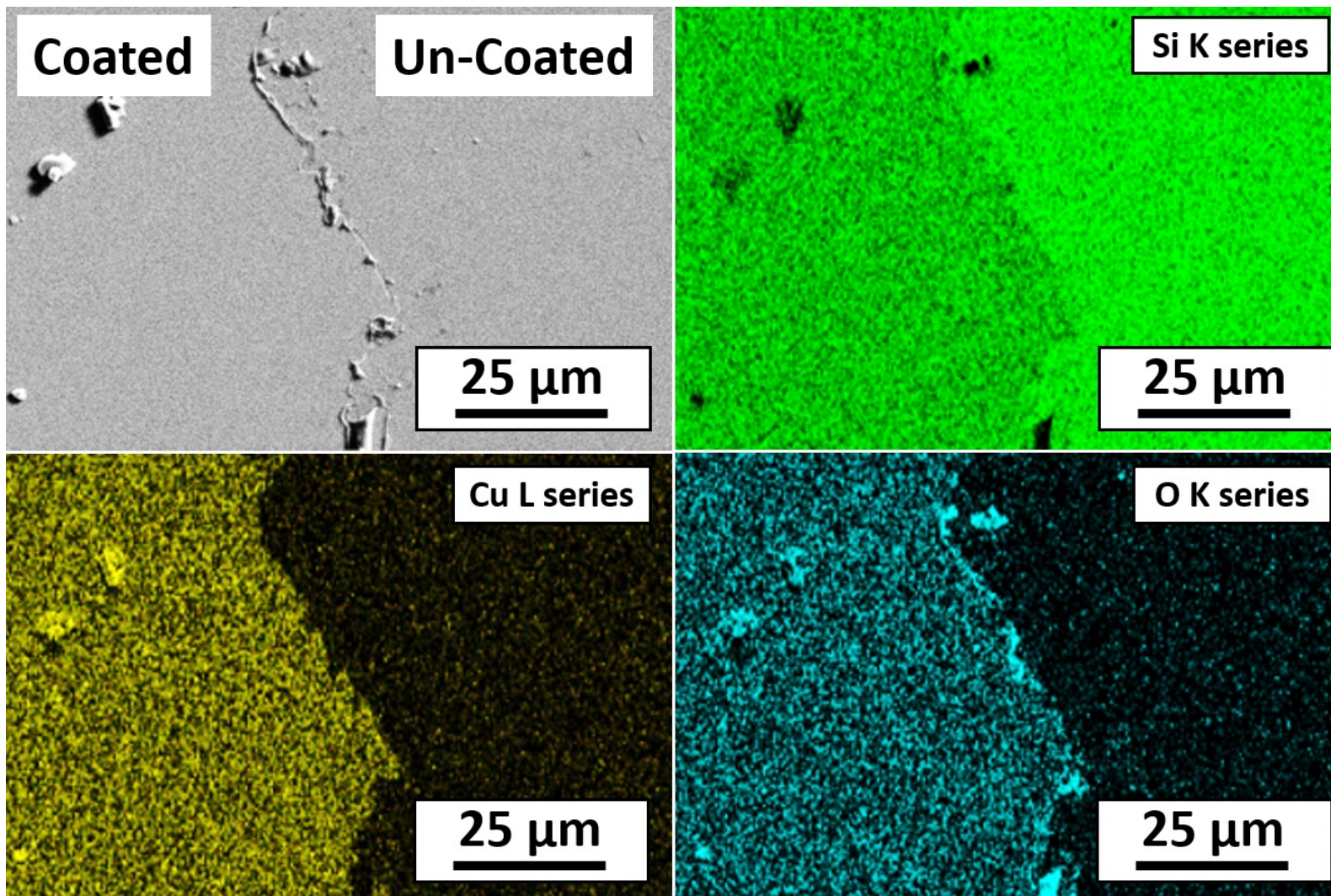
## SEM/AFM profile across masked boundary



- Visible color contrast in coated region
- AFM topography plot confirms the thickness of coating around 24nm

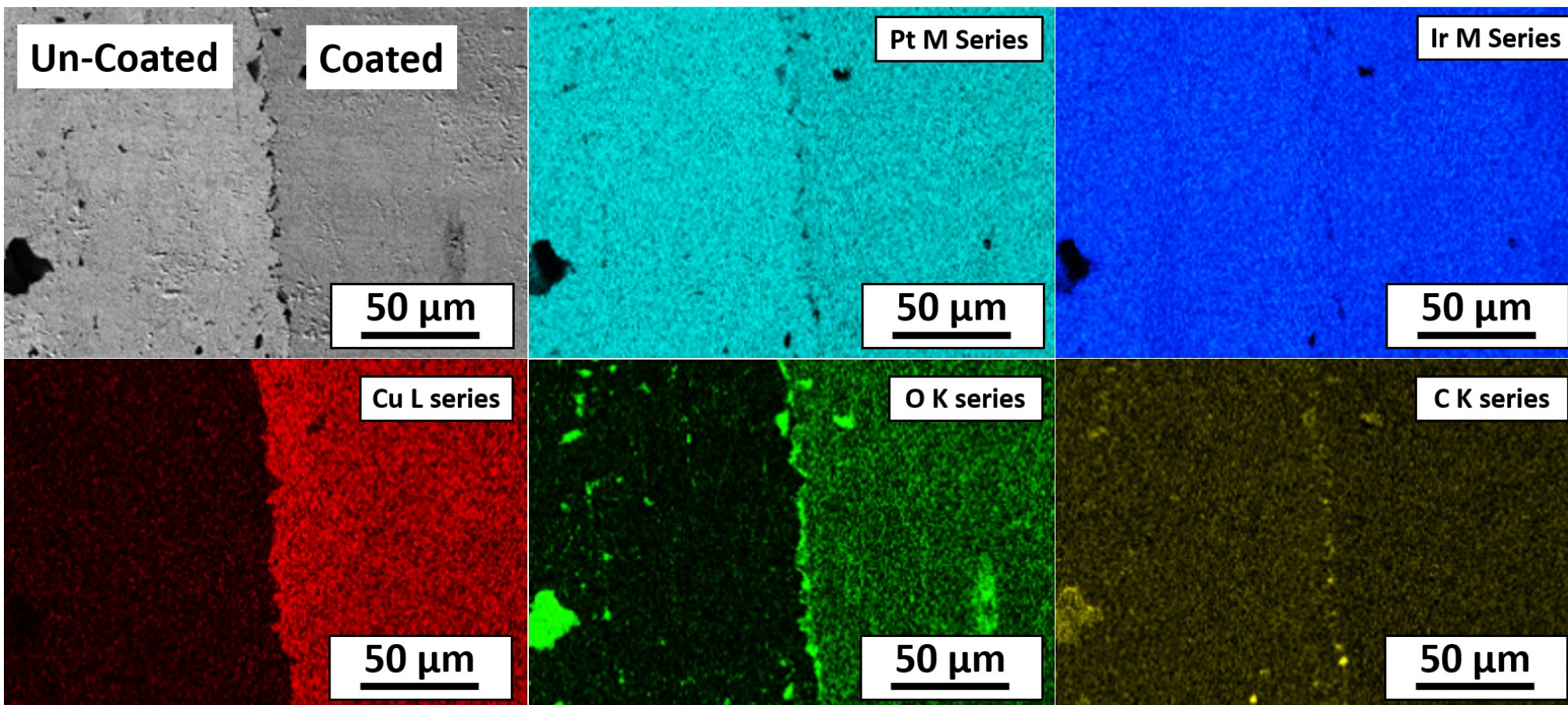
# Compositional Mapping

## Si Wafer substrate



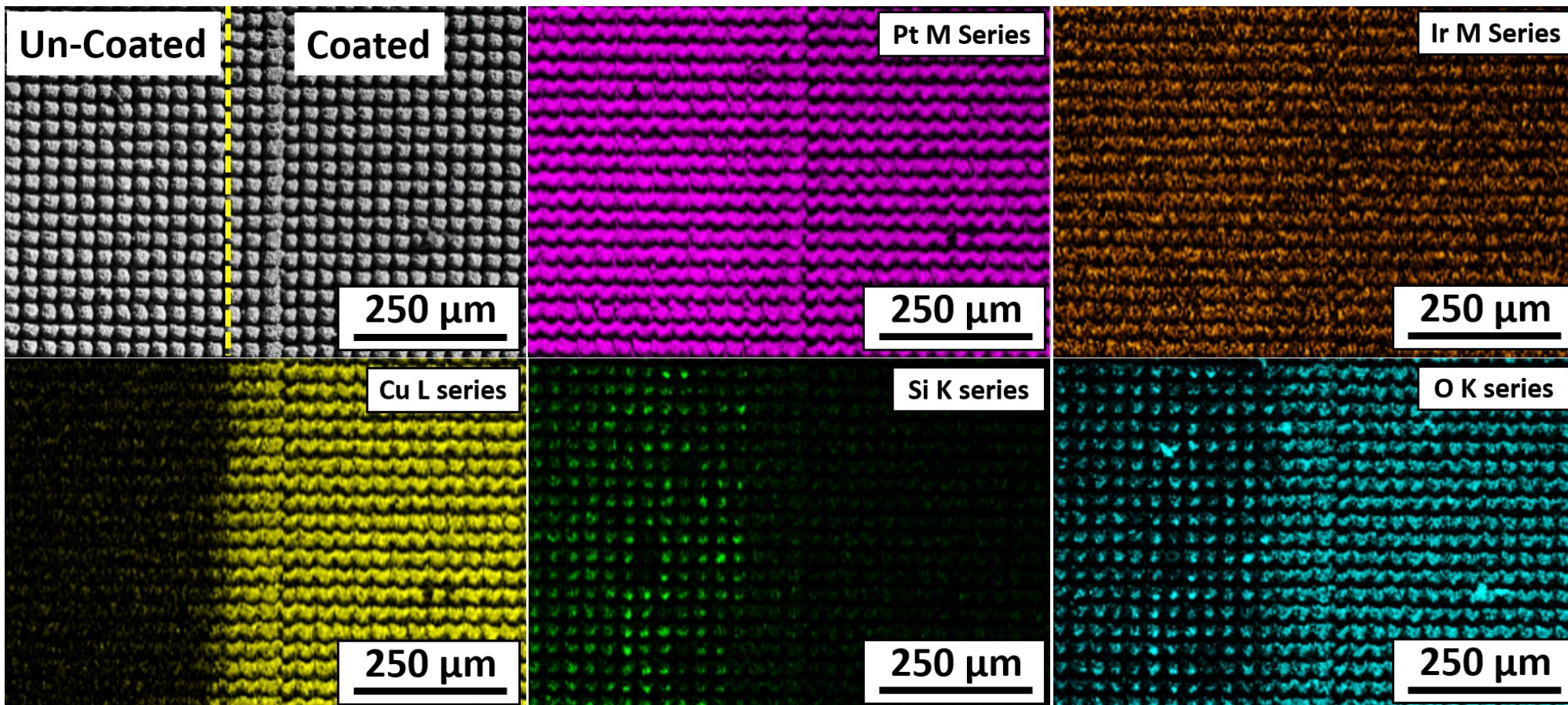
# Compositional Mapping

## Flat Pt10Ir substrate

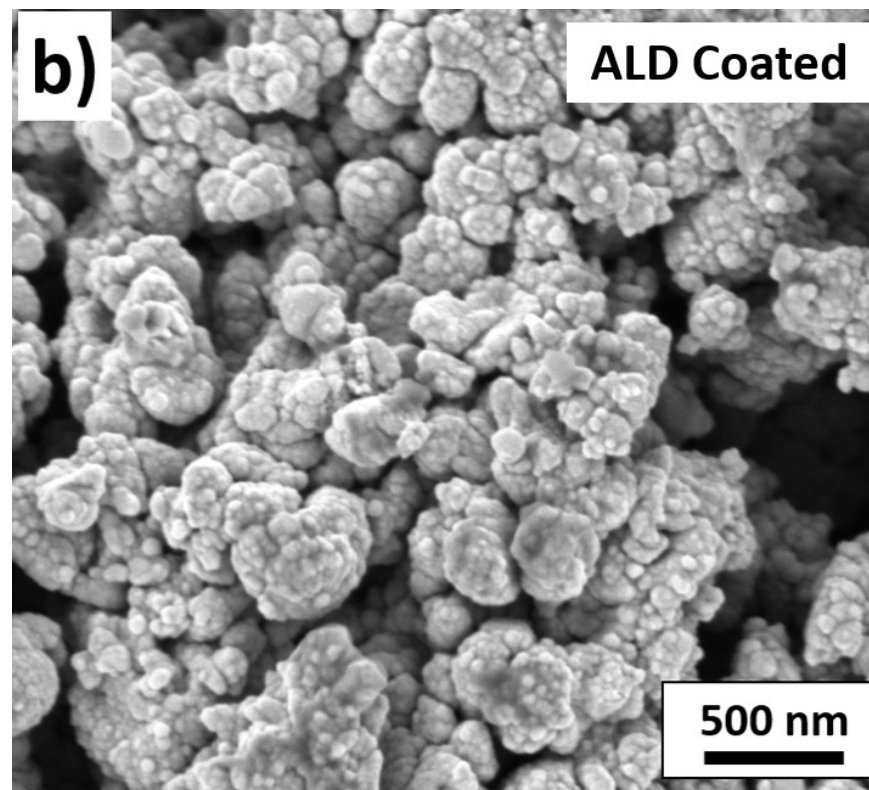
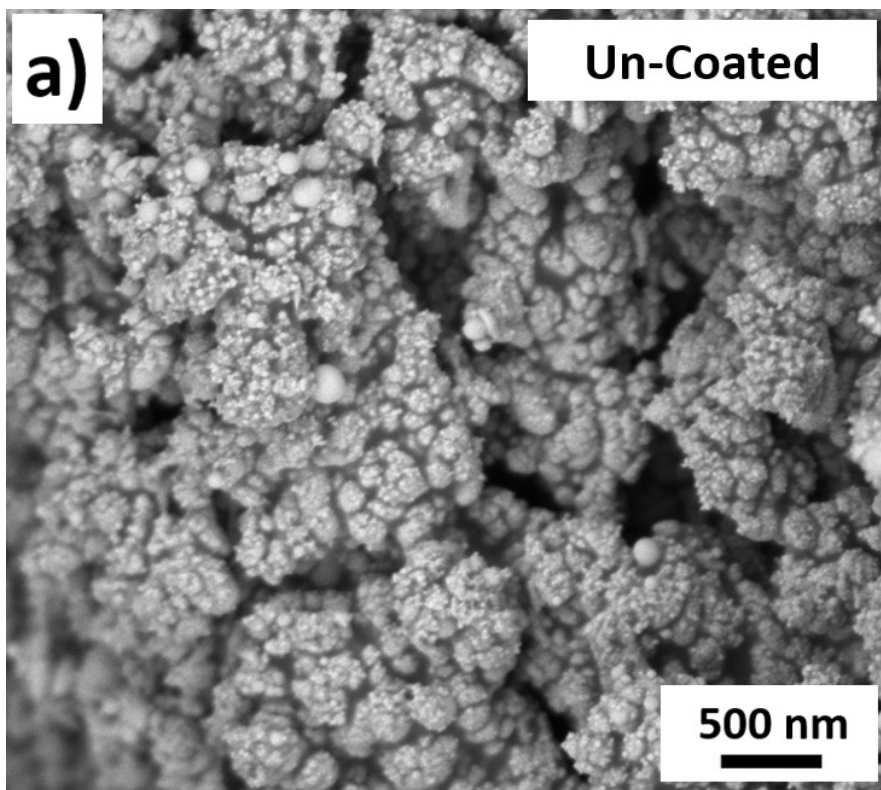


# Compositional Mapping

## HSR™ Pt10Ir substrate

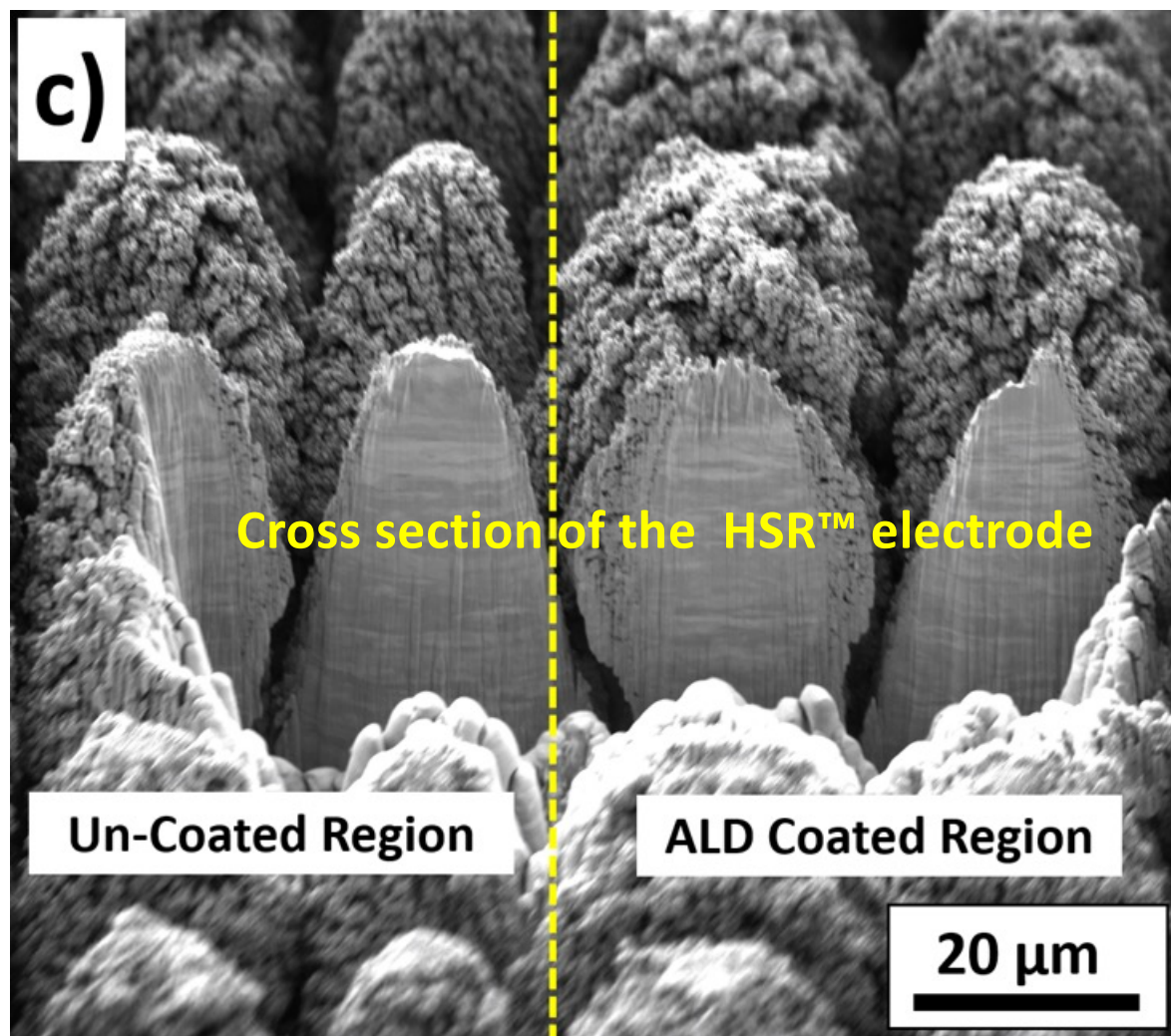


## Microstructural Results



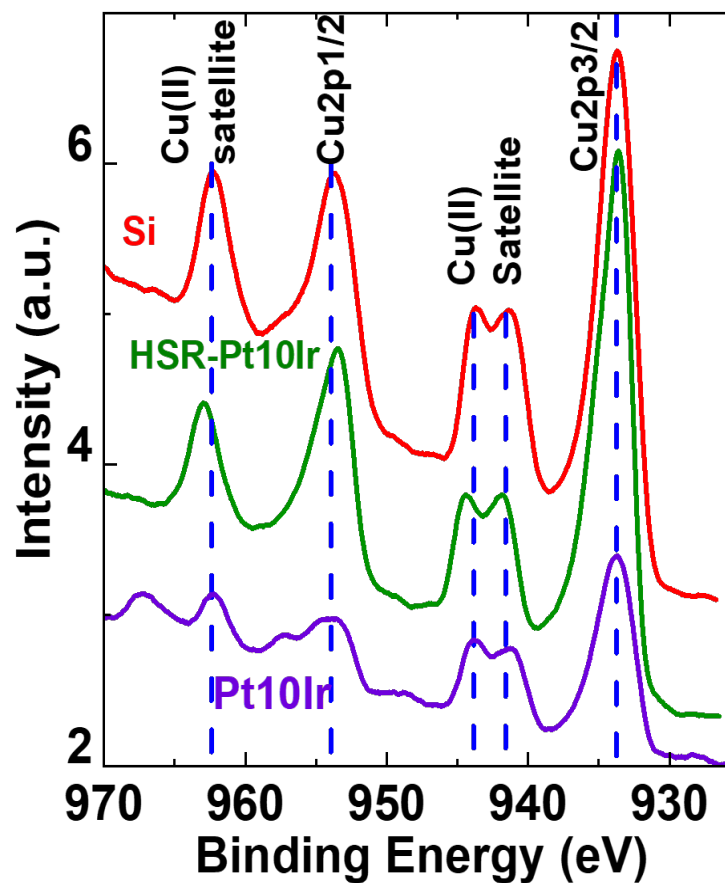
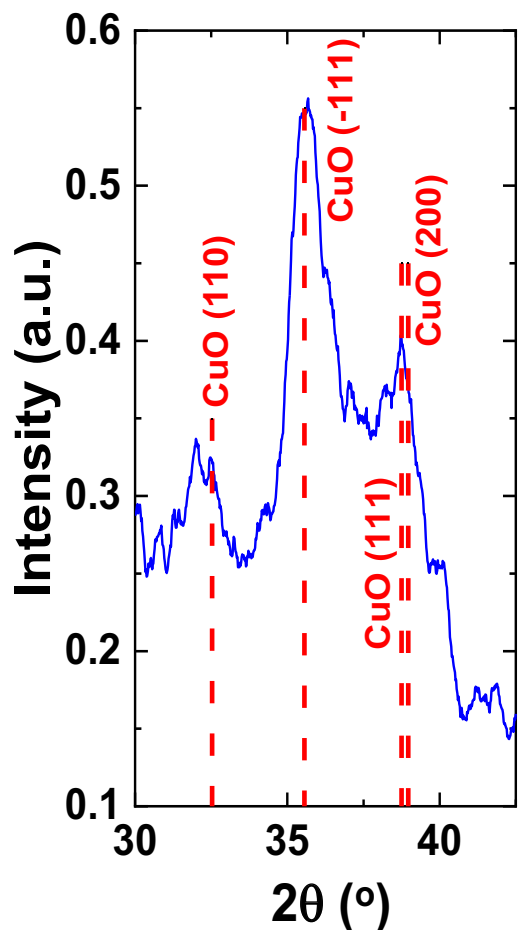
- CuO coating is conformal and uniform
- Nano-sized features of the HSR™ surface are slightly blunted after deposition

## Microstructural Results



# Thin Film Structure

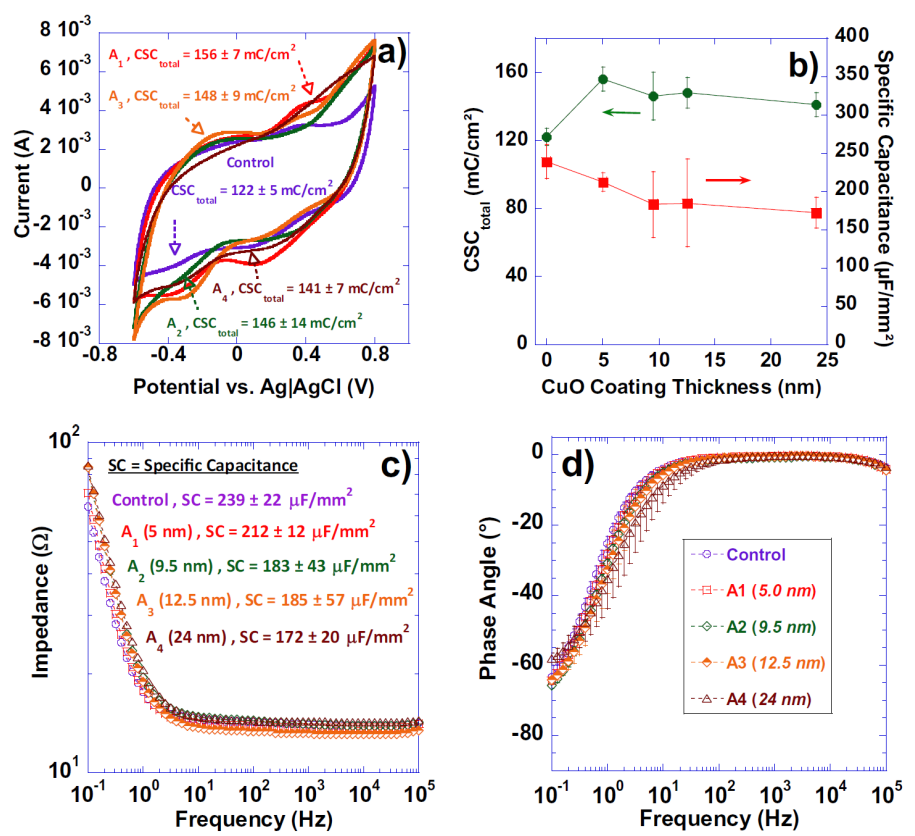
## XRD/XPS Spectra of $\text{Cu}_x\text{O}$ coated electrode



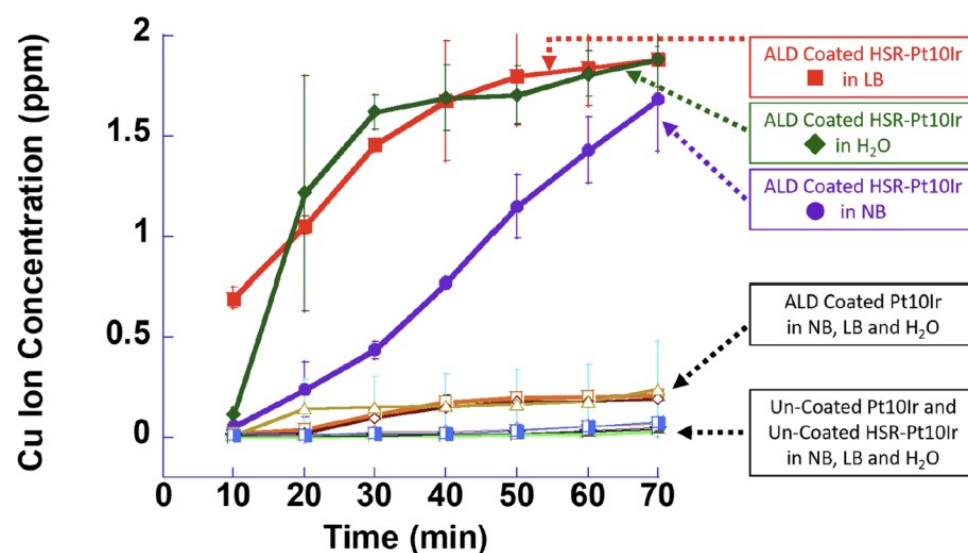
# Electrochemical Properties

As film thickness increases:

- Charge storage capacity increases
- Impedance is un-affected



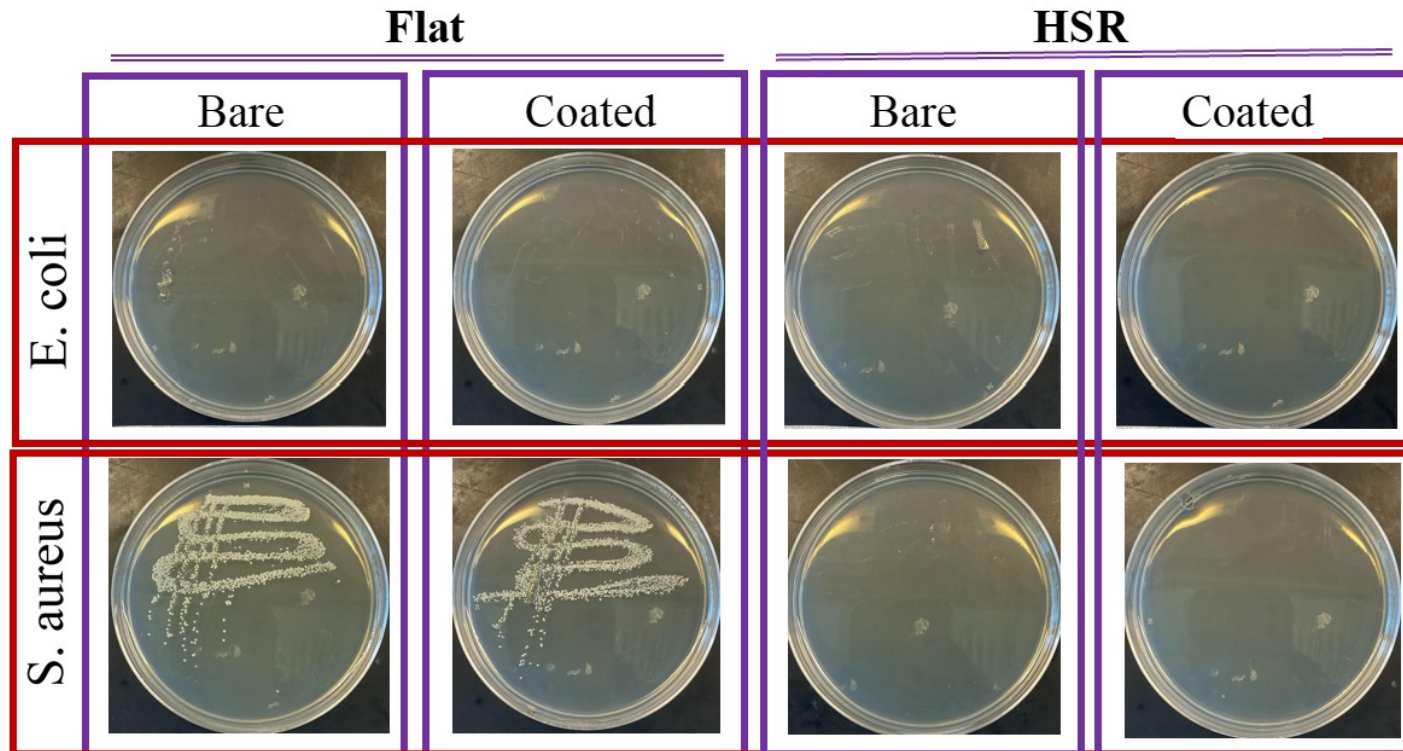
- HSR<sup>TM</sup> electrodes restructuring increase the Cu ion release rate due to the increase in surface area
- CuO coating releases Cu into surrounding medium



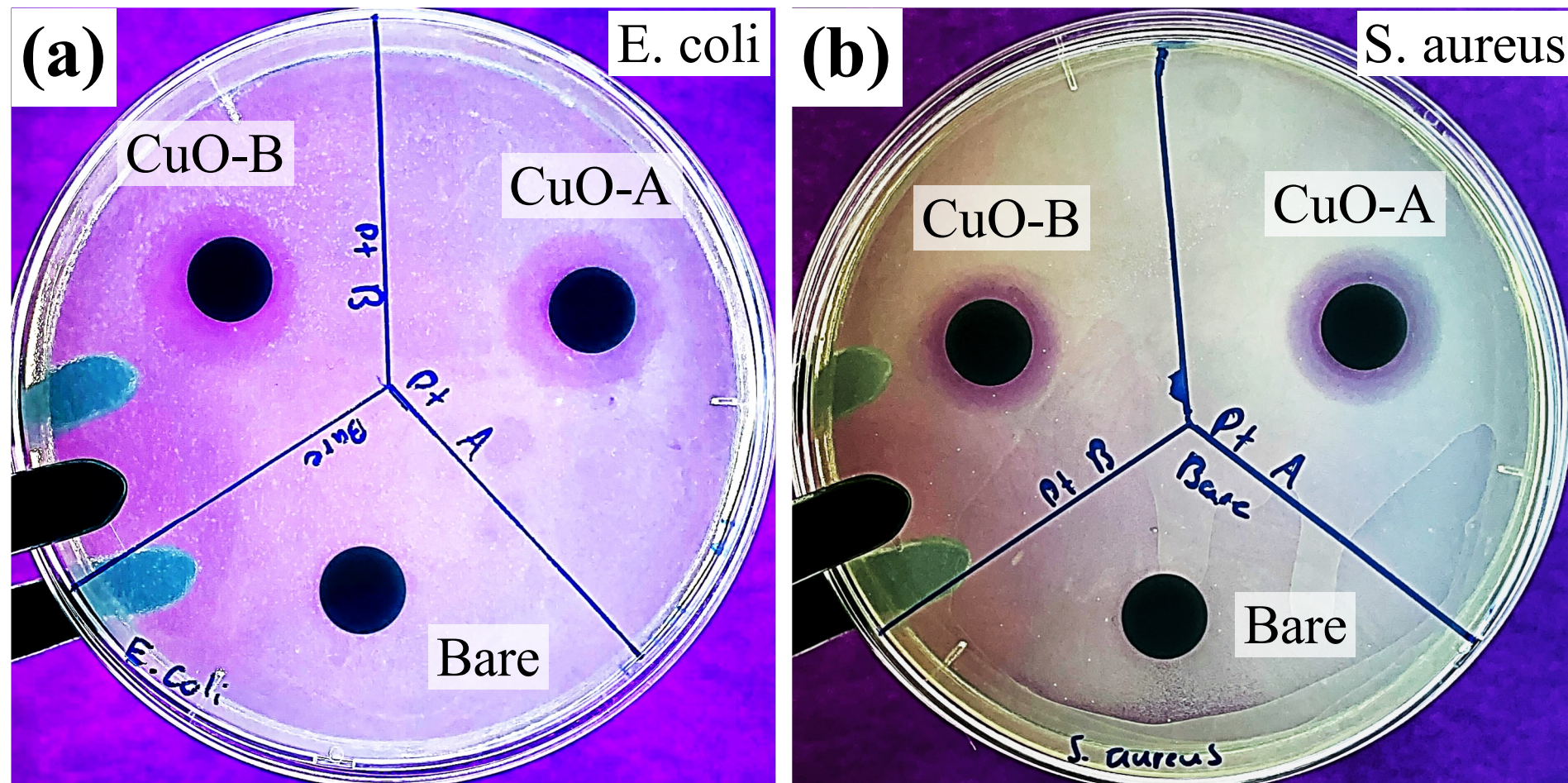
## Antibacterial Measurements

### Surface swapping test

- Both coated and uncoated HSR™ electrodes were found capable of rupturing gram positive (E.Coli) and gram negative (S.aureus) bacteria because of sharp morphology.
- Just the morphology of HSR™ Pt10Ir sample is capable to fight bacteria



- Zone of inhibition method required
- Kirby Bauer method to assess coating effectiveness



## Concluding Remarks and Future Scope

- Hierarchical surface restructuring increases electrochemical surface area of the electrodes
- CuO ALD coated electrodes are promising to reduce infection
- CuO ALD coated electrodes can kill E. coli and S. aureus in direct contact
- Electrode impedance is not affected by the ALD coating
- Charge storage capacity of the electrodes is slightly increased
- We will be working on other metal oxides deposition to see antibacterial effectiveness.

## Acknowledgements




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