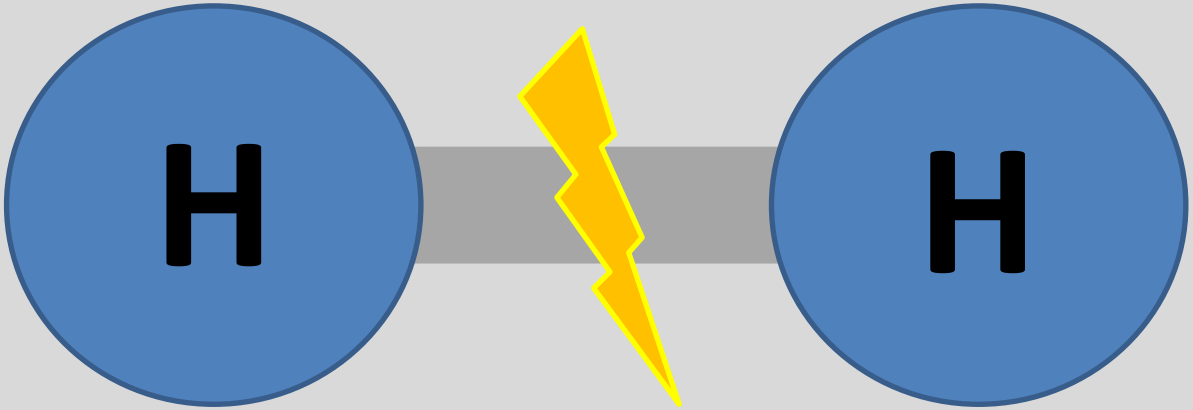
# Transition Metal Sulfide Heterostructures for Hydrogen Evolution Reaction

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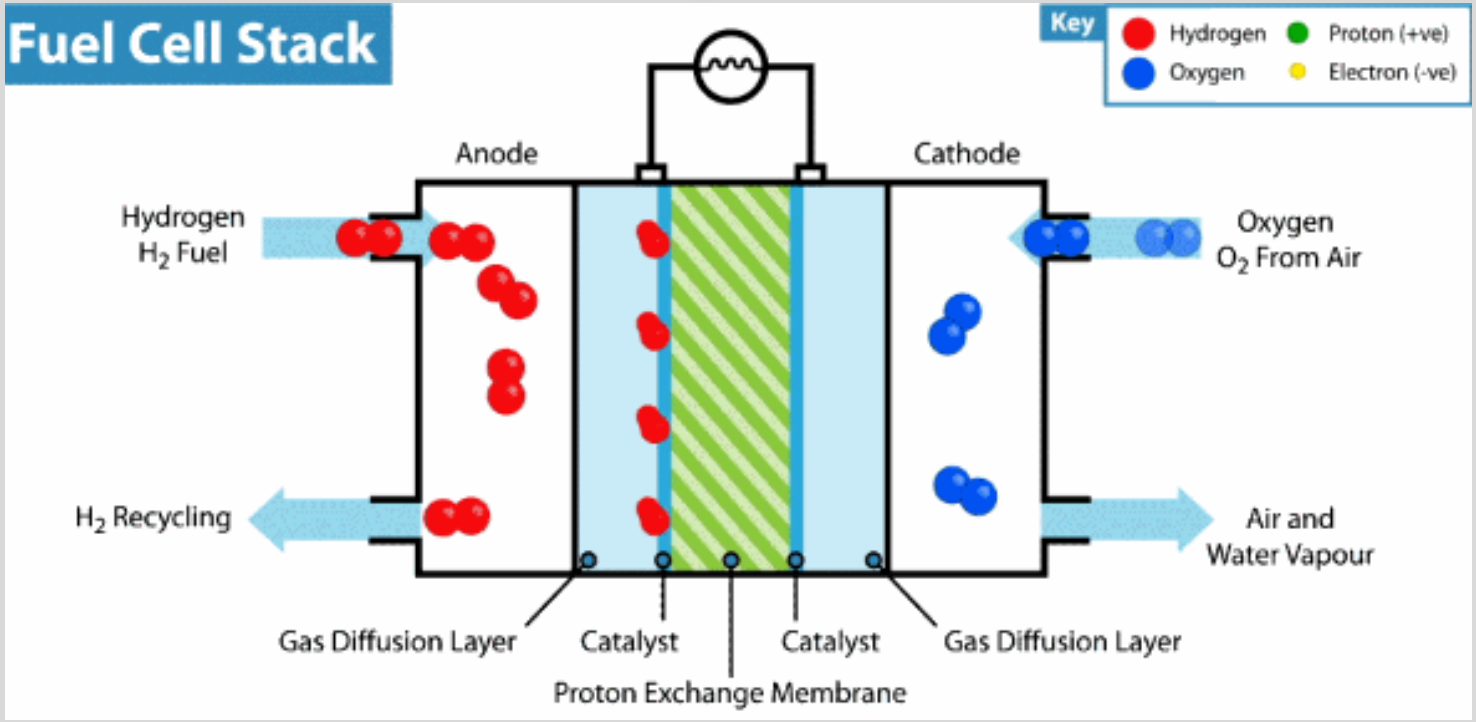
# The Hydrogen Solution



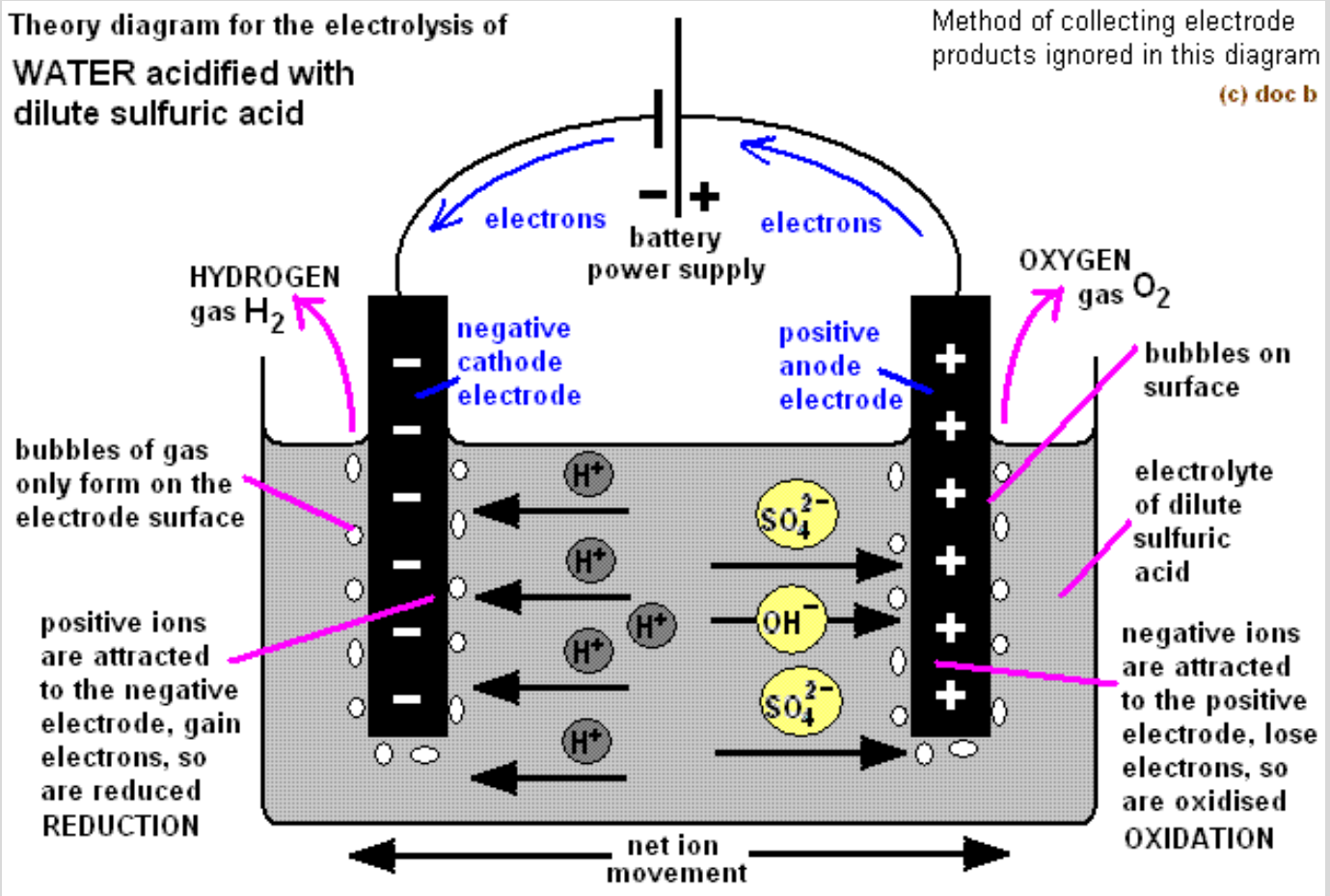

Pros:

- Hydrogen is most abundant element in the universe
- High energy content (rocket fuel)
- No pollutants produced (only byproducts are Heat +$H_2O$

Cons:

Non-competitive due to inefficient production

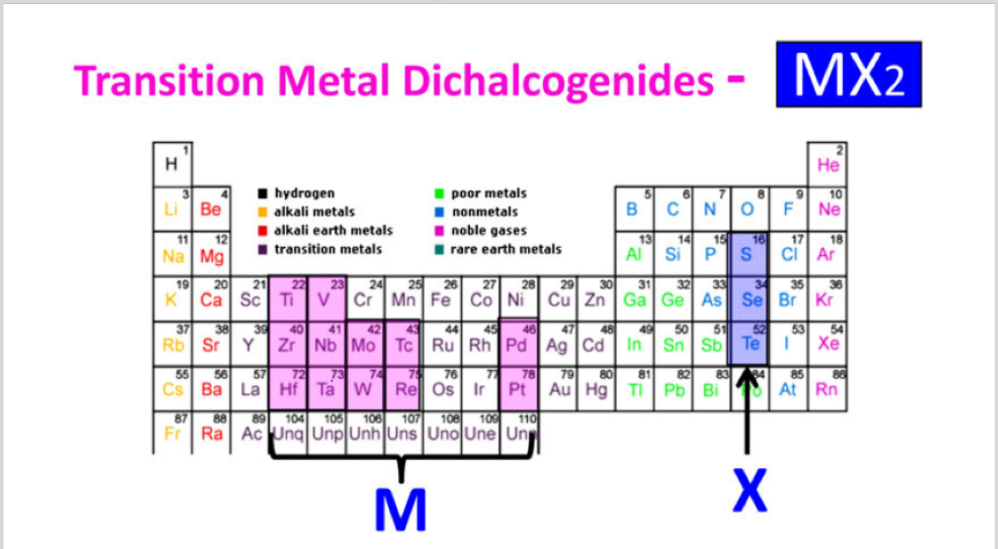
# Electrolysis

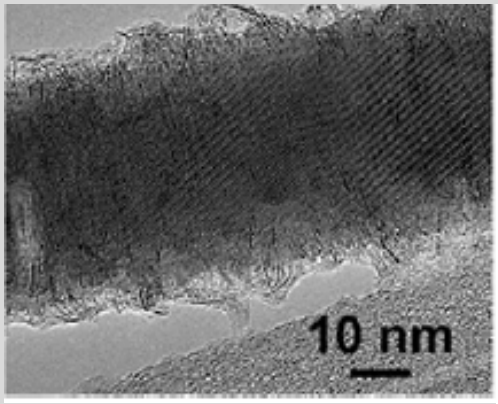


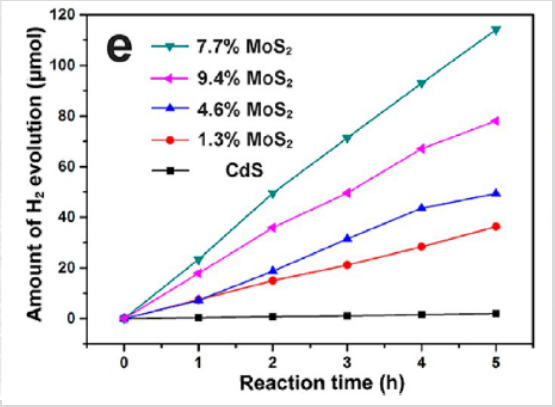

- Hydrogen evolution reaction occurs by means of splitting an acid into its components
- Requires a great deal of energy to operate reaction

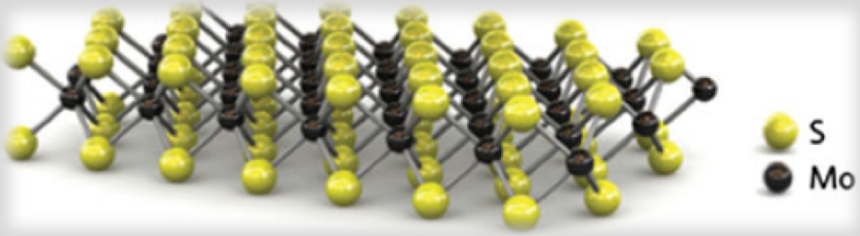
# TMDs as Catalysts for the Production of $H_2$

- Transition Metal Dichalcogenides (TMDs) are a family of layered semiconductors
  - Excellent electronic, optical, and mechanical properties
- $MoS_2$ has good stability and high catalytic activity along edges
- Has been shown to be effective in photocatalytic HER



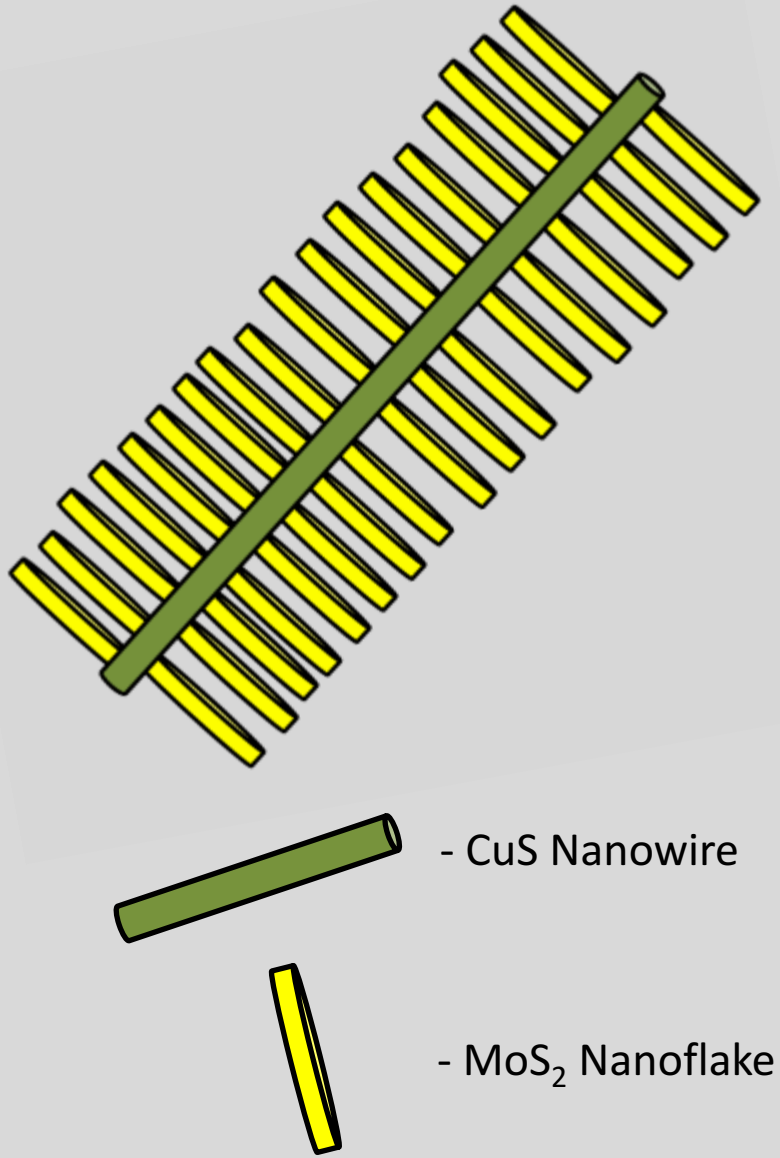




Chen, Junze, et al. Journal of the American Chemical Society (2017).

# Project Goal

- Use a morphological approach to create a heterostructure with high $MoS_2$ edge concentration to promote efficient HER
- Nanowire base structure with flake/wing-like protrusions
- Synthesize by facile vapor deposition process

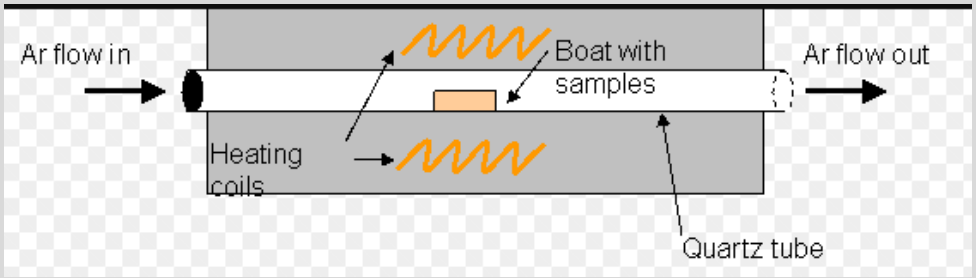


# Synthesis of $Cu_xO$ Nanowire Template

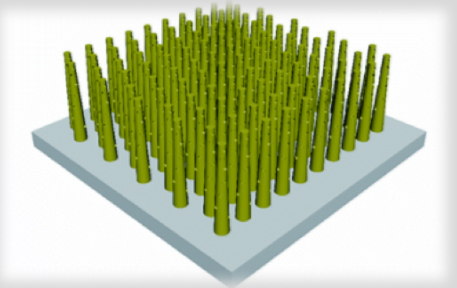
*Sample Prep*

- Sonicate in Acetone - 5 minutes
- Sonicate in Isopropyl Alcohol - 5 minutes
- Sonicate in DI water - 5 minutes
- Submerge in $HNO_3$ - 30 seconds
- Dry in $N_2$

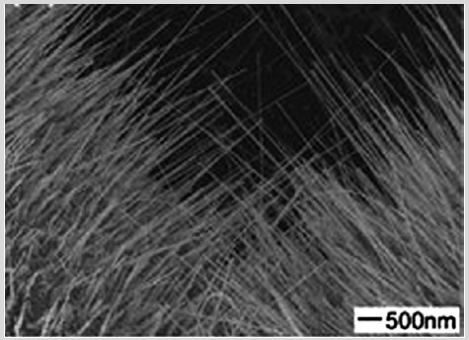
Begin with thin copper sheet




Thermal Oxidation ($N_2$ environment)



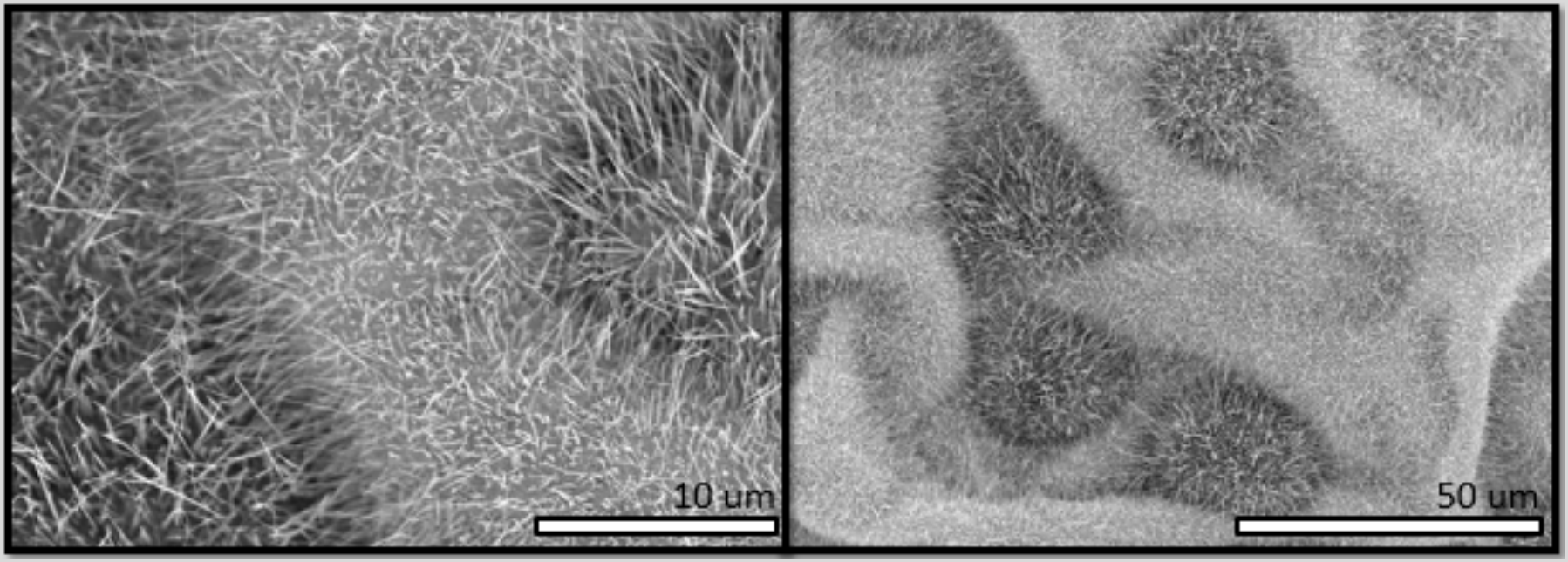
$CuO/Cu_2O$ nanowires




Zhang, Q et. al, (2014). *Progress in Materials Science*, *60*, 208-337.

# $Cu_xO$ Nanowires

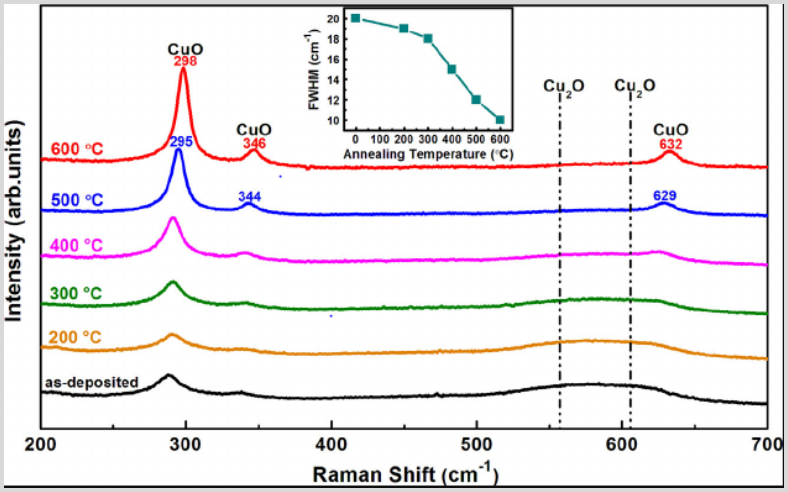
- Thermal oxidation of pure Cu substrate at 410°C for 5 hrs



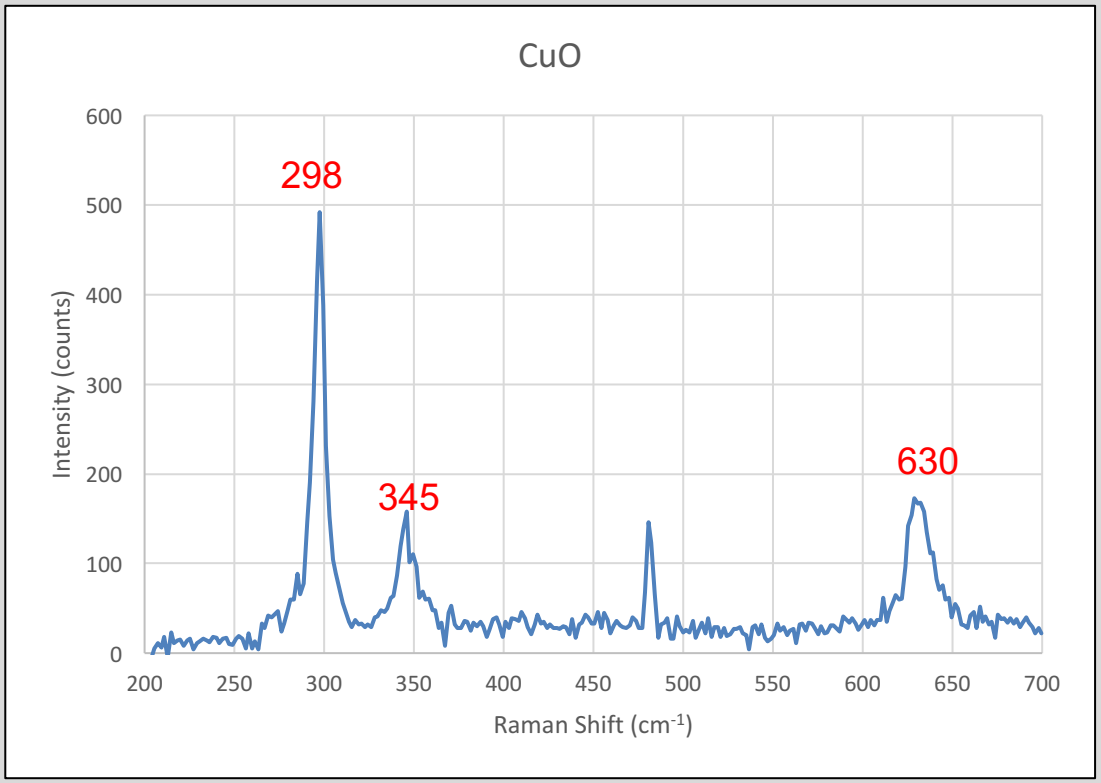
# CuO Nanowires

- Raman spectroscopy can measure the vibrational modes in a material which can be used to identify materials

- Spectra obtained compares well with reference
  - Same Raman stretching/bending modes detected

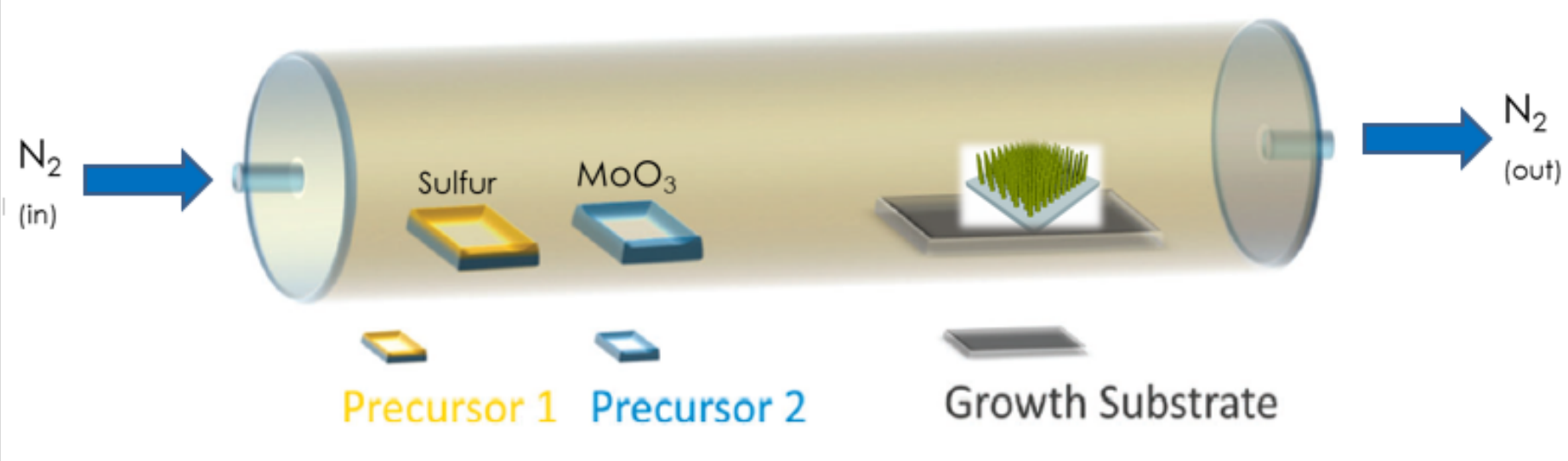


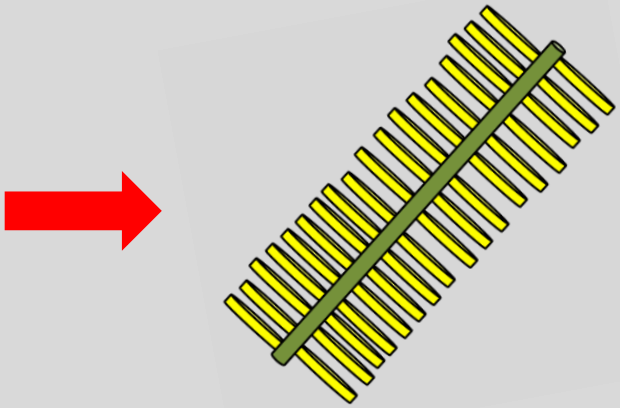

Akgul, Funda Aksoy, et al. "Influence of thermal annealing on microstructural, morphological, optical properties and surface electronic structure of copper oxide thin films." *Materials Chemistry and Physics*



# Heterostructure Synthesis

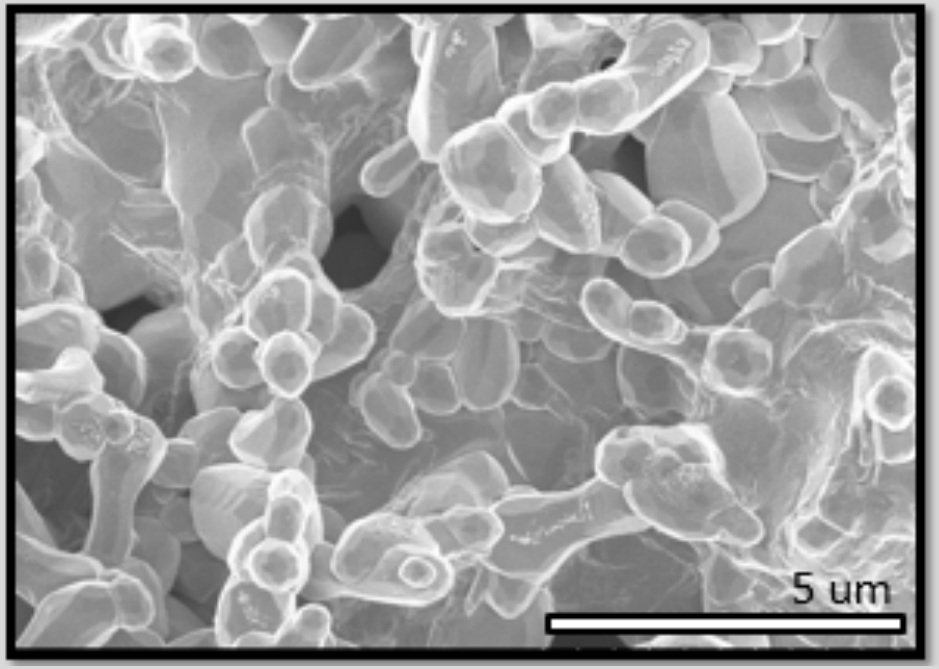
## Chemical Vapor Deposition(CVD)

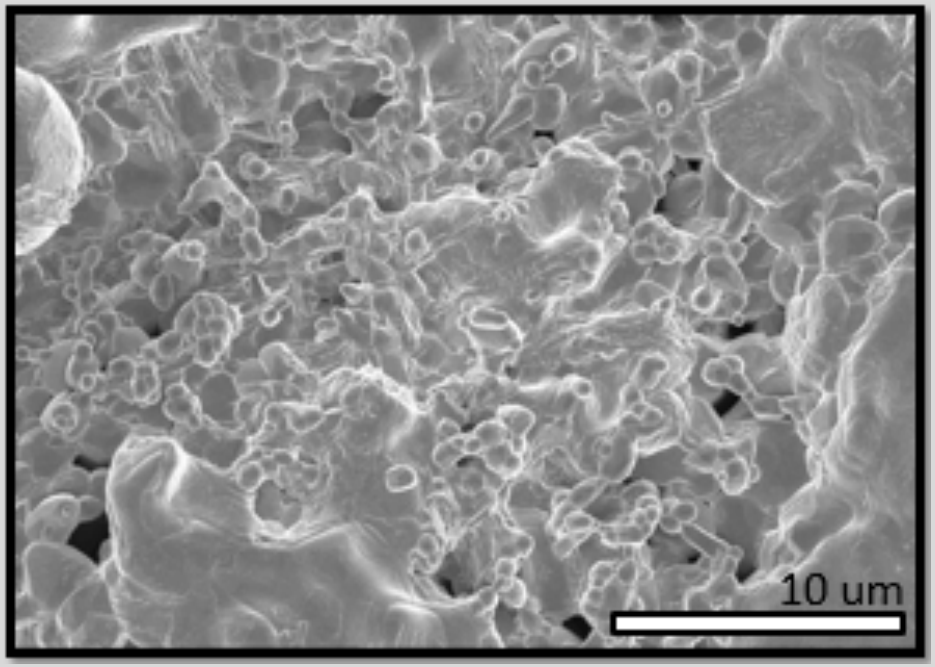


# Initial $MoS_2$ Growth Attempts

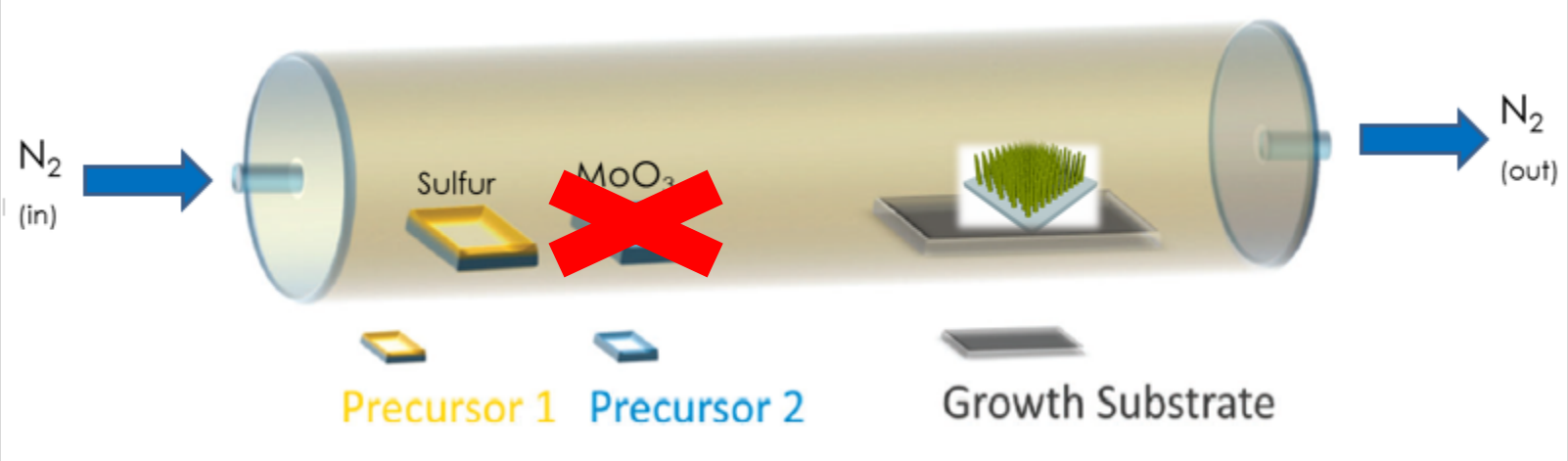
- Attempted standard $MoS_2$ growth w/ CuO nanowires at 650°C for 30 mins w/ $MoO_3$ and S precursors
- Unwanted globular structures appeared



# Sulfurization Experiments

- To test the nanowire stability, the CuO nanowires were subjected to sulfurization tests at varying temperatures
- Only sulfur upstream (no $MoO_3$)



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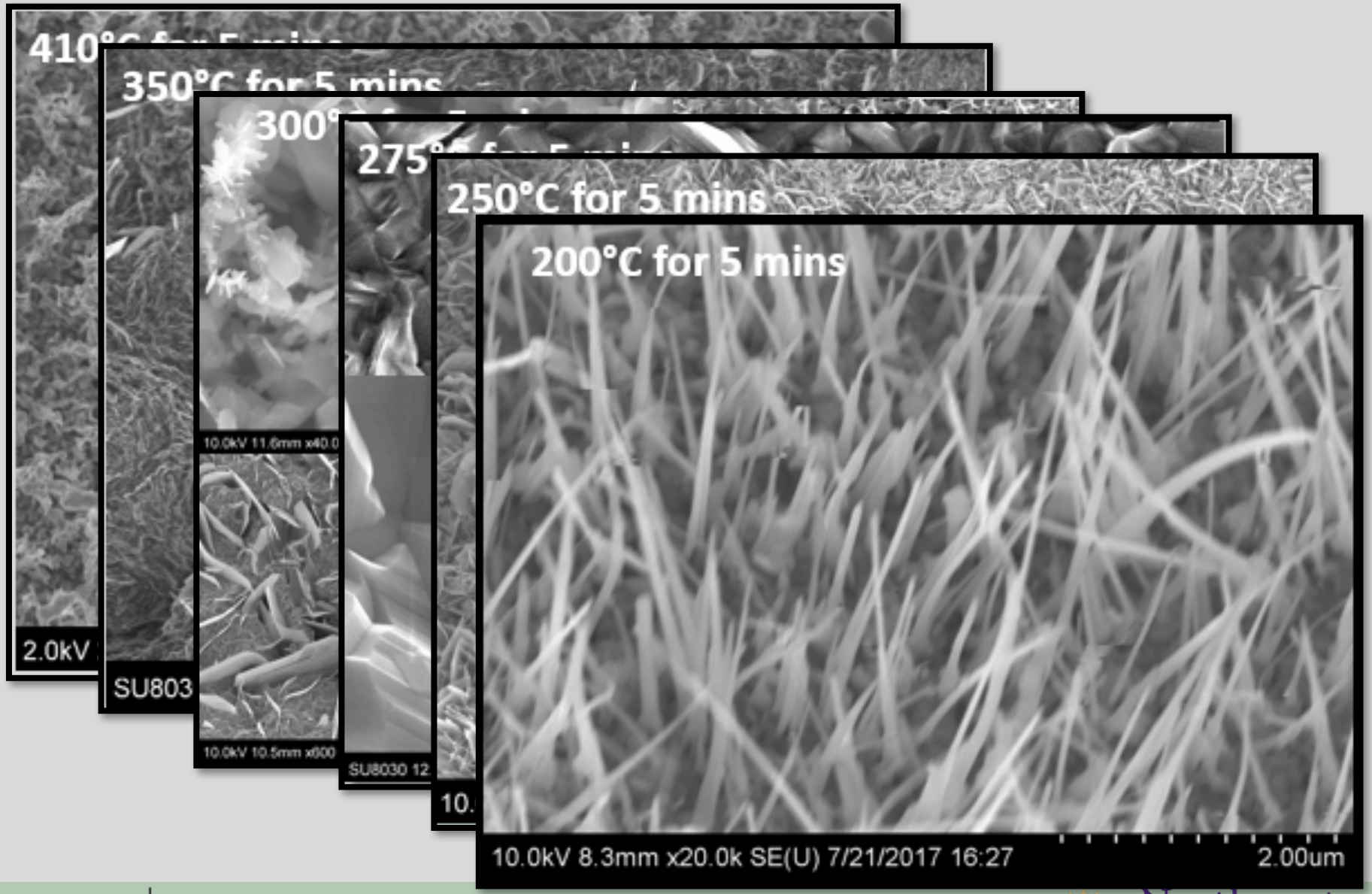
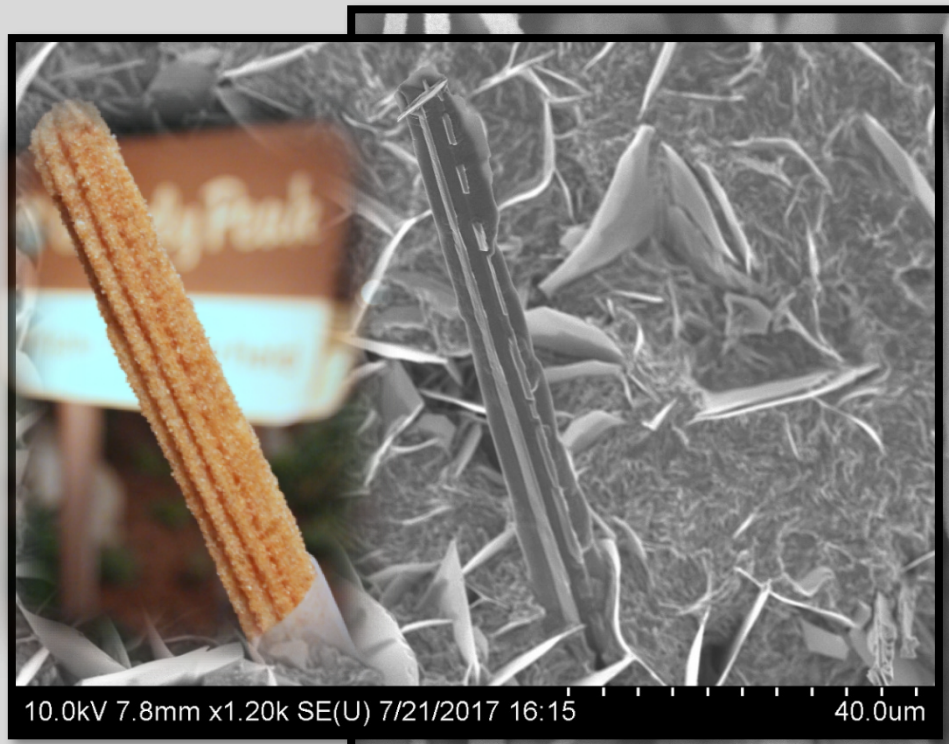
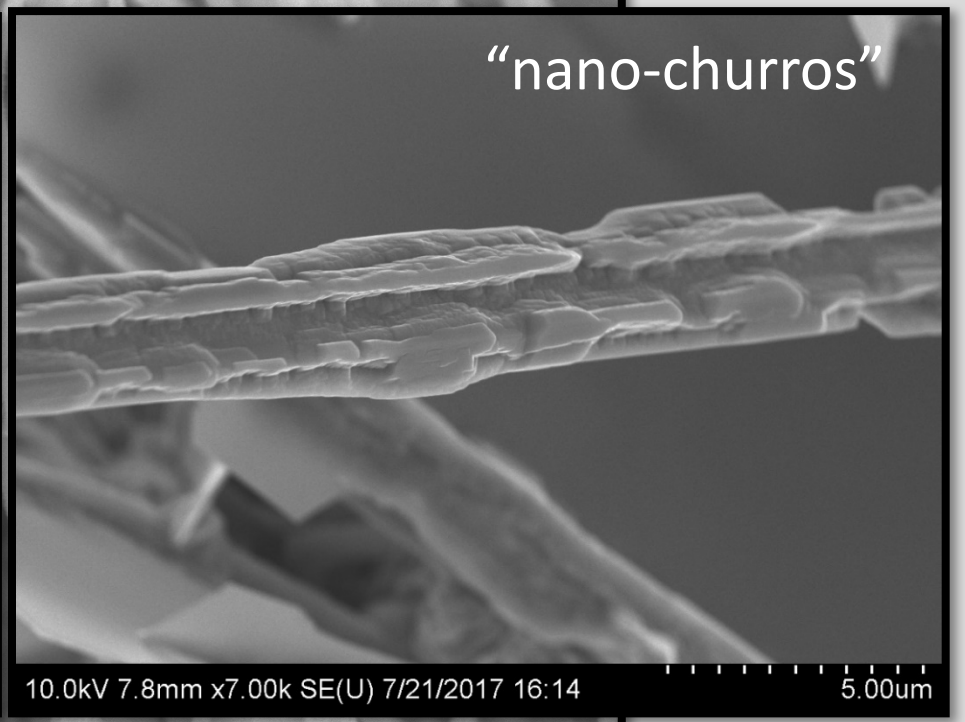


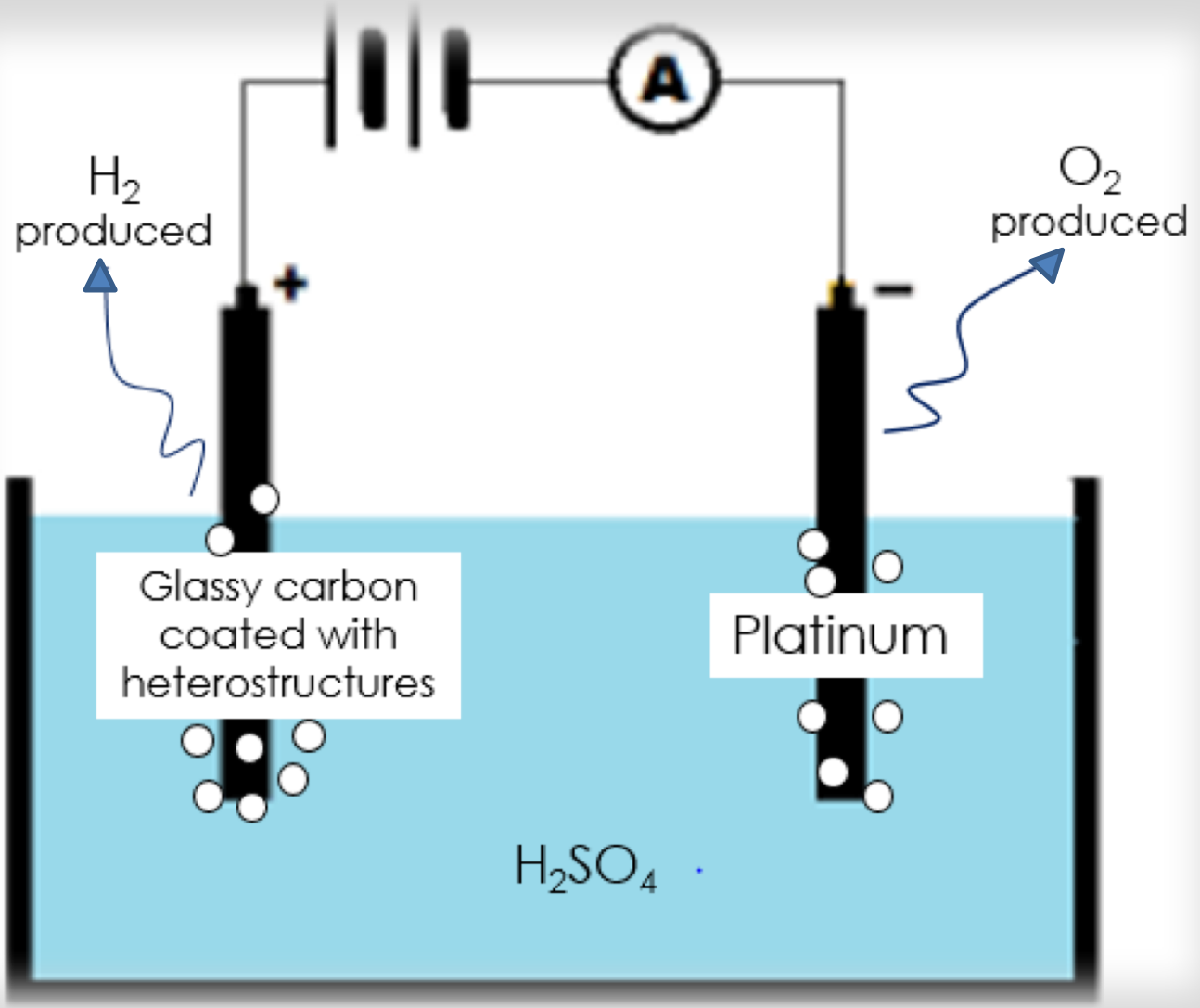
# Heterostructure Synthesis

- Deposited 1 nm thick layer of Mo on to $Cu_xO$ nanowire surface using electron beam evaporator
- Sulfurized sample at 300°C for 5 mins
- Synthesis of rod-like structures with fins parallel to rod-axis (high edge concentration)



# Hydrogen Evolution



# Future Work

- Coat more samples of CuO nanowires with nanometer thick layer of Mo using electron beam evaporator
- Understand link between growth parameters and structural morphology
  - Try to recreate the "nano-churro" structures achieved before in greater quantity
- Further analyze samples using Raman, XPS, and XRD
- Measure  HER using three - electrode electrochemical system test
  - Cyclic voltammetry test (CV)
  - Electrochemical Impedance Spectroscopy (EIS)
  - Generate Tafel slope to show overpotential

# Acknowledgements

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SHyNE Soft and Hybrid Nanotechnology Experimental Resource

NUANCE Atomic and Nanoscale Characterization Experimental Center

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