3D-printed spectroelectrochemical cell for in-situ Raman spectroscopy measurements of oxygen evolution reaction for nanocarbide electrocatalysts

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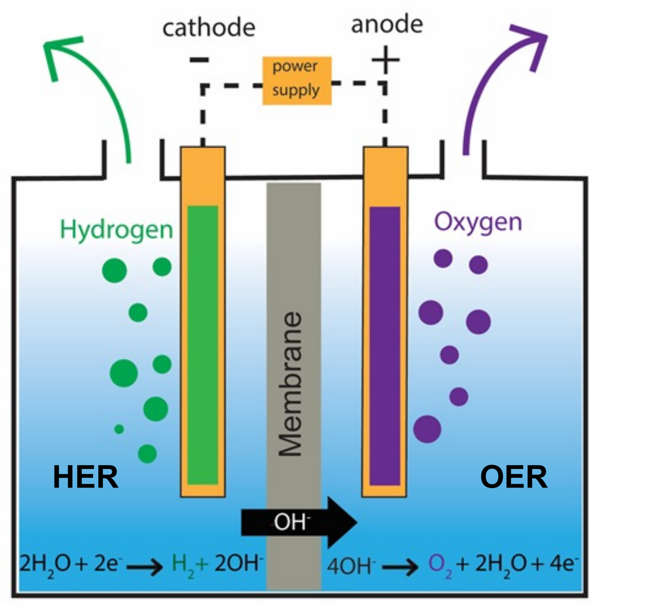
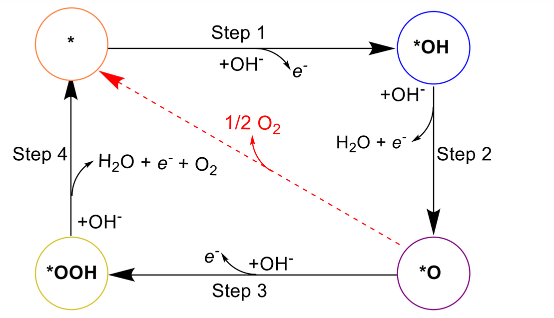
Motivation

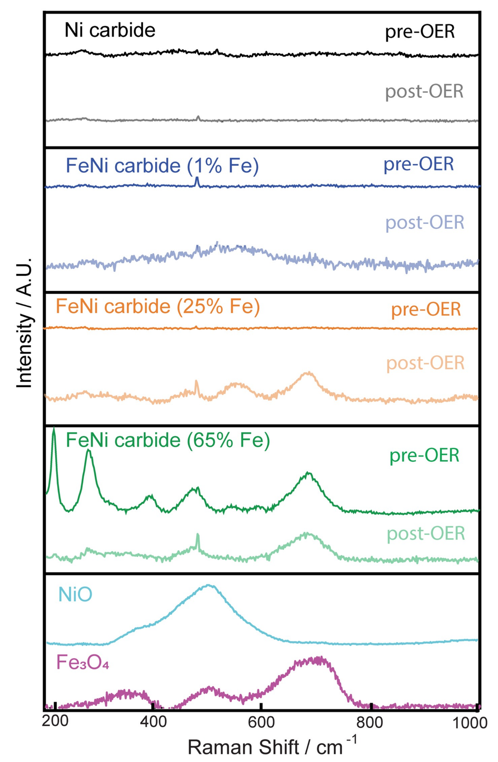
Exploring renewable ways to produce hydrogen for fuel cell technologies is crucial for reduction of CO_2 emissions in our atmosphere. Electrochemical water splitting is a promising method to ensure sustainable hydrogen production and is comprised of two half-reactions: the oxygen evolution reaction (OER) and the hydrogen evolution reaction (HER).

We focus on the electrocatalytic OER, as this requires the transfer of four electrons, which is kinetically sluggish.





Ex-situ Raman spectroscopy allowed us to visualize oxide transformation post-OER catalysis.



Limitation: Ex-situ Raman spectroscopy did not offer real-time analysis to visualize rapid surface oxide changes.

Project Motivation: To analyze real time surface oxide formation during OER catalysis using electrochemical in-situ Raman spectroscopy.

Project Aim: To develop a 3D printed spectroelectrochemical cell for in-situ Raman Spectroscopy measurements that corrects our main obstacles:
- Blocking out environmental noise
- Optimizing laser focal length

Electrochemical in-situ Raman Spectroscopy

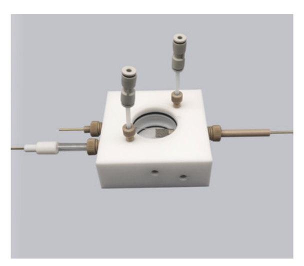
In-situ Raman Spectroscopy will allow us to monitor the effects of surface oxidation and how it impacts electrocatalytic performance. An in-situ setup is ideal for real-time monitoring of electrocatalytic reactions.

Commercial cells for in-situ Raman spectroscopy instruments can range from $1,000 – $3,000 USD

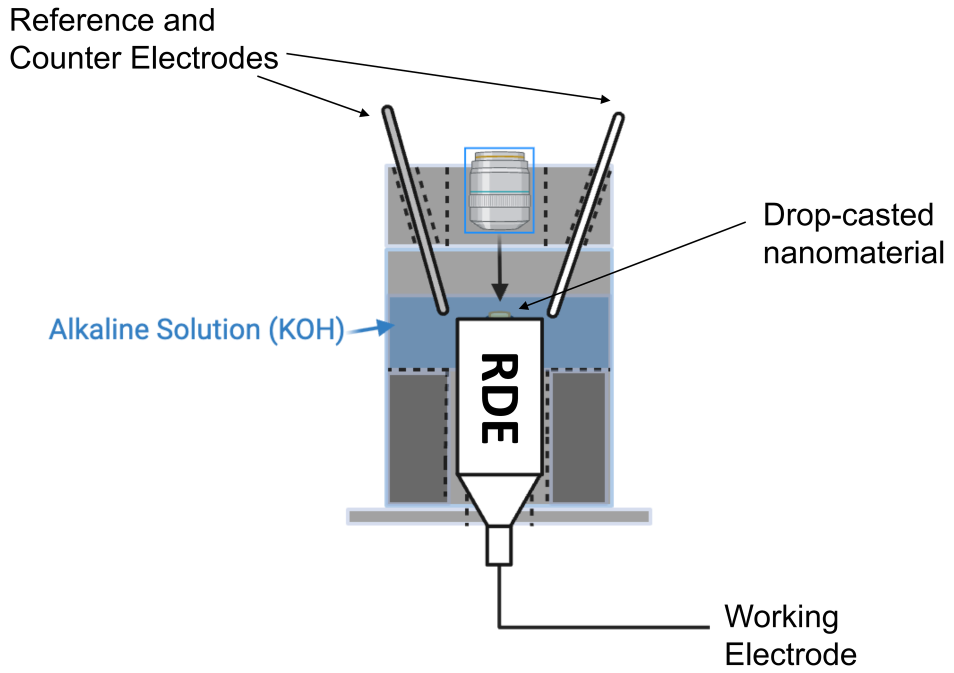
MSE supplies LLC: $6,620

DEK Research: $1,820

DEK Research: $2,250



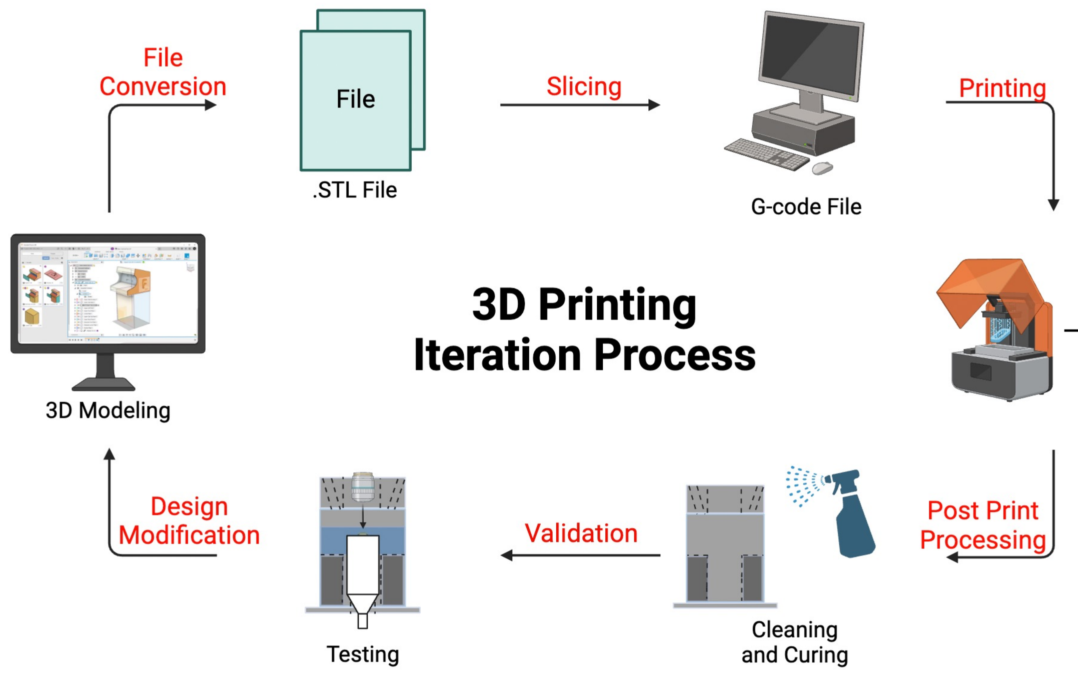
3D printing: a lower cost alternative for developing spectroelectrochemical cells.



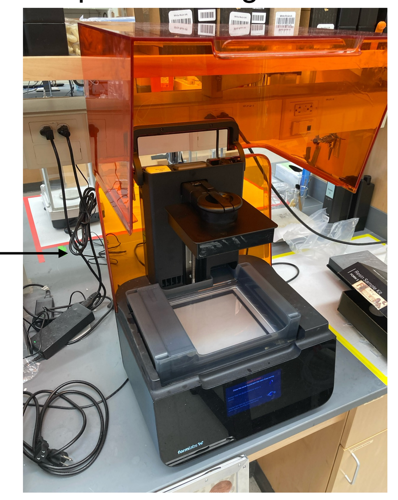
Design of Spectroelectrochemical Cell

3D printing and methodology

The step-by-step iterative process used in developing our 3D model design



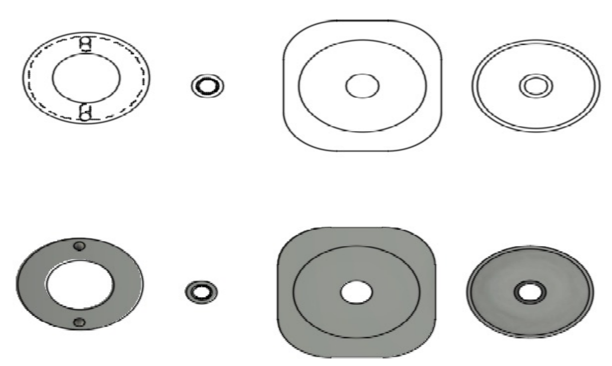
Form 3 by Formlabs used for all 3D printed designs shown.



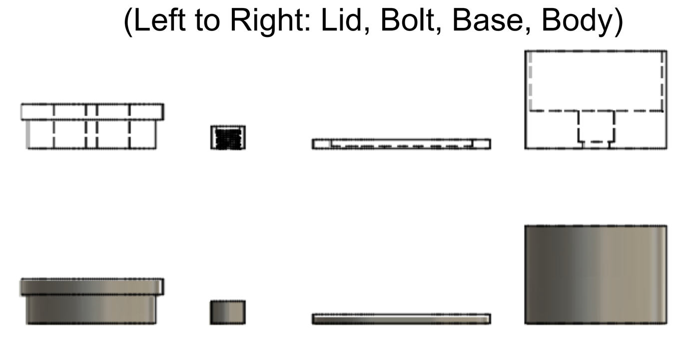
Low Force Stereolithography (LFS)

3D printing of parts separated

Top view of design parts separated. (Left to Right: Lid, Bolt, Base, Body)

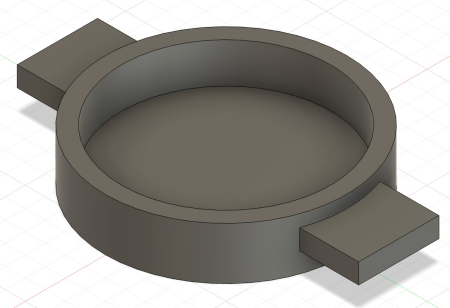


Side view of design parts separated. (Left to Right: Lid, Bolt, Base, Body)

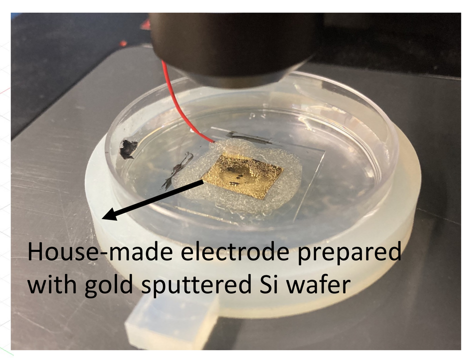


Stage 1 Design

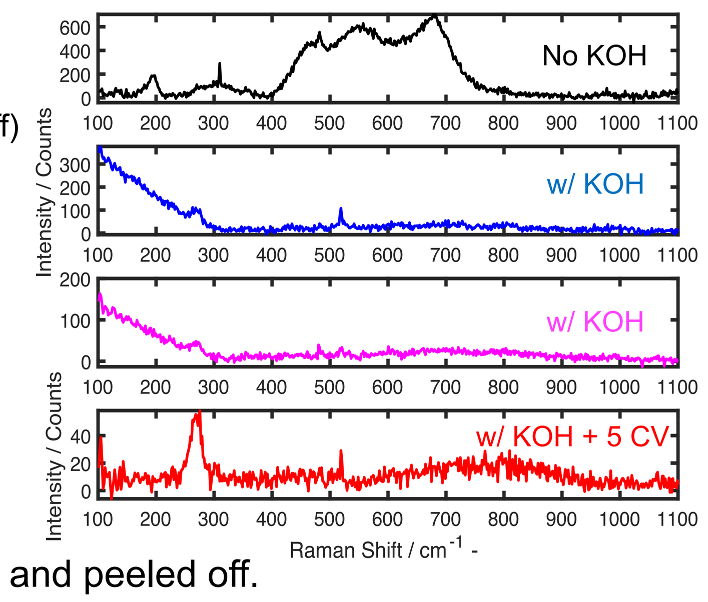
Petri dish holder design



In-situ Raman Spectroscopy experiment after 10 CVs (Note: almost all the material has fallen off)



Raman spectra for 25% FeNiC

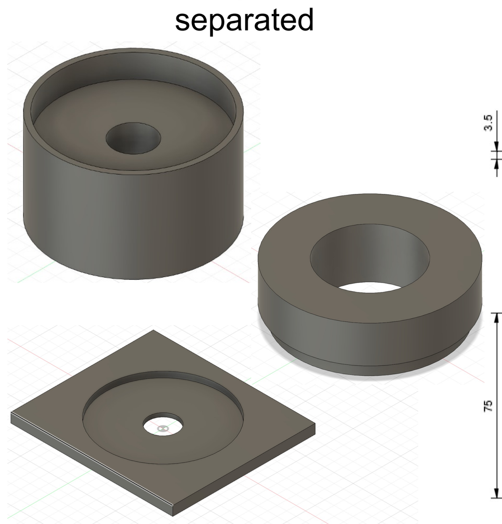


Limitations:
- The gold sputtered electrode surface was unreliable and peeled off.
- Air from the vents in the Raman lab caused the worsening of S/N in Raman spectra.

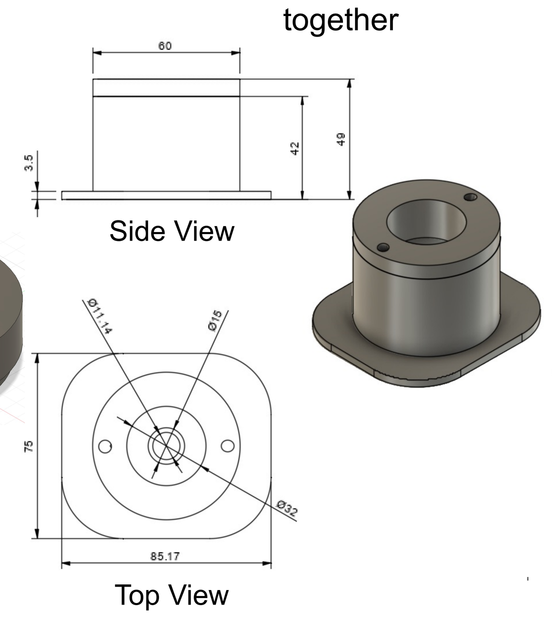
Improvements needed: Stable gold electrode surface and protect setup from environmental noise.

Stage 2 Design

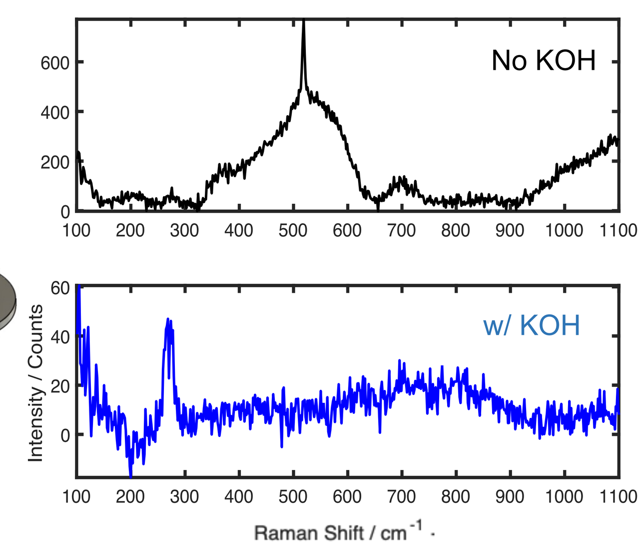
Rotating Disk Electrode based design with parts separated



Design specifications when parts are put together



Raman spectra for 100% NiC



Limitations:
- Noise was still present when solution was added.

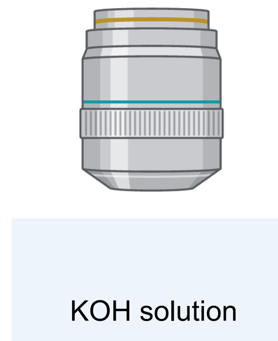
Improvements needed:
- R&D of improving S/N once electrode is immersed in solution.

Spectroelectrochemical Cell Modifications

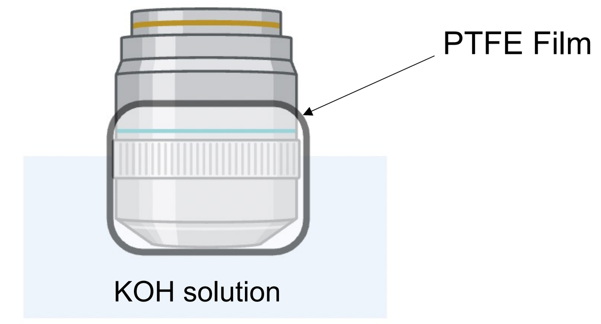
PTFE (Teflon) Film

Our Goal: Improve the ability to focus on samples in solution by wrapping a PTFE film over the laser and positioning the laser in solution

Our current design concept with laser out of solution



New design concept with laser cover by PTFE film and in solution

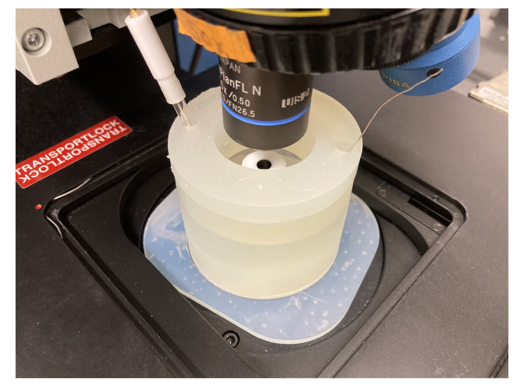


Volume Optimization

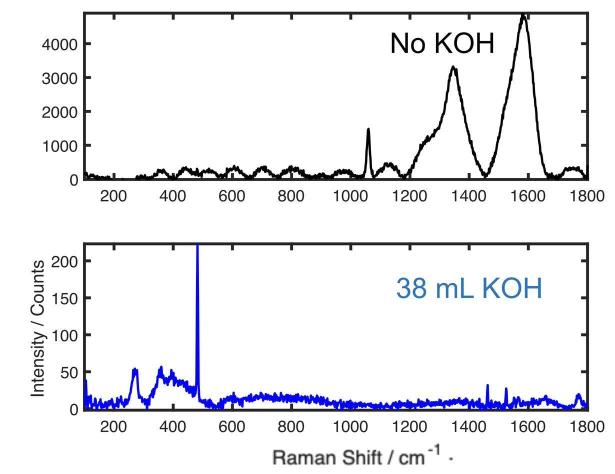
Our Goal: To develop standard protocol for using the finalized 3D design.

Solution can reduce incident light intensity and decrease collection efficiency of scattered photons.

In-situ Raman spectroscopy experiment setup



Raman spectra for 45% FeNiC Volume Test



Conclusions and Future Work

Conclusions:

- Development of a 3D spectroelectrochemical cell improves in-situ Raman spectra quality by:
  - Reduction of environmental noise, enhancing signal clarity and reliability
  - Preventing material degradation during electrochemical oxidation
  - Improved laser positioning stability, ensuring consistent and accurate data acquisition from a fixed position throughout the experiment duration
- Addition of solution prevents ability to accurately focus on nanomaterial and further aid is required
- 3D printing is a viable solution over expensive commercial cells

Future Work:

Finish design of in-situ model and make further improvements on focus → Test quality and repeatability of Raman spectrum → Test for standard protocols for using the design

References and Group Information

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