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| 13. SUPPLEMENTARY NOTES | | | | | |
| 14. ABSTRACT This project relates to generation of novel electrocatalysts (ECs) exploiting the principal investigator's published results on low platinum group metals (PGM) of 10 wt. % F doped Mn _{0.8} Ir _{0.2} O ₂ :10F, EC for oxygen evolution reaction (OER) and CoxCu _y Pz _z S (CCPS), ECs for hydrogen evolution reaction (HER) as promising ECs for proton exchange membrane water electrolysis (PEMWE) including hydrogen evolution reaction (HOR) and oxygen reduction reaction (ORR) electrocatalysts meeting the required targets of >1W/sq. cm@0.7V rated power and proton exchange membrane fuel cell (PEMFC) target performance of > 300 mA/sq. cm @0.8V. Specifically the work entails fabrication of large area electrodes with the above developed ECs utilizing theoretically predicted electrocatalyst loadings, electrode thickness and porosity for utilization in MEA fabrication for PEMFC and PEMWE. Proper experimental correlations between the electrode layer thickness, electrocatalyst loadings and electrode structures with performance, durability, mass and charge transport and polarization losses were established to optimize the electrode layer thickness, porosity and appropriate ECs loadings to achieve the targeted hydrogen and oxygen production rate to meet the desired targets. | | | | | |
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Accomplishments

What were the major goals and objectives of the project?

This project was initiated to synthesize and study the electrochemical performance of novel reduced precious group metal (PGM) containing as well as PGM-free electrocatalysts for acid mediated water electrolysis. The project was successfully completed with demonstration of the synthesis of bi-functional electrocatalysts. The major goal of the project is to engineer novel high performance electrocatalysts (ECs) for acid mediated proton exchange membrane (PEM) based water electrolysis (PEMWE) to generate chlorine free oxygen and hydrogen that will then be used to drive the PEM based hydrogen fuel cell (PEMFC). Accordingly, the project entails tasks for synthesizing, designing, and engineering novel oxygen evolution reaction (OER), hydrogen evolution reaction (HER) electrocatalysts for PEMWE as well as new and novel electrocatalysts for oxygen reduction reaction (ORR) and hydrogen oxidation reaction (HOR) for driving the PEMFCs. These novel ECs comprising low platinum group metal (PGM) and earth abundant PGM-free metal containing systems have been developed by the principal investigator and his team and are already published in peer reviewed archival journals. The electrocatalysts will be made into electrodes and tested for PEMWE as well as in PEMFC. Optimized designs for the electrocatalyst loadings, electrode design of thickness, porosity as well as ionomer configurations will be obtained by multi-physics and computational fluid dynamics (CFD) based theoretical modeling that will be incorporated into the experiments. All of the developed electrocatalyst systems will be systematically characterized for structure, microstructure and electrochemical response. The electrochemical response will be compared with current state of the art PGM based systems (e.g., Pt, IrO₂, etc.). Finally, the systems will be assembled into membrane electrode assemblies (MEAs) and tested for PEMFC and PEMWE performance to compare with current state of the art systems and determine the performance metrics.

Specifically, the work involves fabrication of large area (~5 square centimeter(cm²)) electrode with the principal investigator's (PI's) developed ECs utilizing theoretically predicted electrocatalyst loadings, electrode thickness and porosity for utilization in MEA fabrication for PEMFC and PEMWE. Proper experimental correlations between the electrode layer thickness, electrocatalyst loadings and electrode structures with performance, durability, mass and charge transport and polarization losses will be established to optimize the electrode layer thickness, porosity and appropriate ECs loading to achieve the targeted hydrogen (H₂)+oxygen (O₂) production rate with input energy of less than 40kWh/kg hydrogen (H₂) for PEMWE as well as achieve the target currents (> 300 mA/cm² @0.8V and rated power equal or exceeding ≥1 W/cm² @ 0.7V) for PEMFC. Single layer MEA and stacked cells will be assembled using optimized large area OER/HER electrodes for PEMWE subsystem to deliver H₂+O₂ fuel with the desired flow rates. Similarly, MEA configurations and designs utilizing optimized ORR/HOR electrodes will be implemented in fabricating PEMFC to achieve the targeted power and energy. Effects of temperature, pressure, current density, cell potential and water purity on MEA performance, and

durability using the PI's developed test protocol will also be studied. Furthermore, detailed characterization of the ionomer/catalyst interaction, including site blocking, and local transport phenomenon at the interface of the MEA as well as MEA degradation mechanisms will be studied to optimize the single layer and stacked MEA designs for both PEMWE and PEMFC. In the last quarter of the project, fuel supply from assembled PEMWE with PEMFC will deliver the targeted power (~40W) using compact systems. The project was successfully completed.

The project comprised three tasks and three milestones as outlined below:

Year 1, Month 1 (M1)-Month 4 (M4), Quarter 1 (Q1) – Quarter 2 (Q2):

Task 1.0: Theoretical computational fluid dynamics (CFD) based identification of optimal electrode parameters including optimal electrocatalysts (ECs) loadings, coating thickness and electrode area to achieve the targeted performance of all of the synthesized electrocatalysts (ECs) for PEMFC and PEMWE meeting the targets of (>300 mA/square centimeter (cm²) @0.8V) and rated power (equal or exceeding (\geq) 1W/cm² @ 0.7V).

Task-1.1: 5 square centimeter (cm²) area electrode fabrication of PEMWE and PEMFC using theoretically identified optimized electrode parameters (thickness and porosity) utilizing different ink formulations.

Subtask-1.1.1: Electrode structure, composition, microstructure, and particle size distribution characterization.

Subtask-1.1.2: Evaluation of the effect of electrode thickness on mass and charge transfer as well as activity and selectivity of the electrocatalysts (ECs). Optimization of OER/ORR/HER/HOR electrode thickness, porosity and electrocatalyst loading based on activity and durability.

Milestone 1.1: Demonstrate large area electrode (~5cm²) of OER/HER for PEMWE and ORR/HOR electrode for PEMFC exhibiting targeted performance of (>300 mA/cm² @0.8V) and rated power (equal or exceeding \geq 1W/cm² @ 0.7V) and sustained 5000h electrolysis and fuel cell operation without degradation.

Year 1, M5-M8, Q2-Q3:

Task 2.0. Multi-physics and CFD based identification of fuel production rate in PEMWE, and flow rates/pressures in the PEMFC to achieve the targeted performance and rated power.

Task 2.1: Fabrication MEAs with optimized 5cm², OER/HER electrodes for PEMWE to ensure sufficient fuel production and supply rates.

Task 2.2: Fabrication MEAs with optimized 5cm^2 , ORR/HOR electrodes for PEMFC to ensure high activity and durability of PEMFCs to generate the targeted performance and power ($>300\text{ mA/cm}^2$ @ 0.8V) and rated power (equal or exceeding (≥ 1) W/cm^2 @ 0.7V).

Subtask-2.1.1: Study the effect of temperature, fuel pressure, current density, and cell potential on MEA performance and durability.

Subtask-2.1.2: Understanding the degradation mechanism of the electrode layer and MEAs under cell operating conditions as well as develop mitigation strategies.

Task-2.2: Optimization of MEA design utilizing optimized ECs based electrode structures.

Milestone 2.1: Demonstrate a single cell large area electrode ($\sim 5\text{cm}^2$) MEA for PEMWE and PEMFC exhibiting targeted performance ($>300\text{mA}$, @ 0.8V) for sustained 5000h @ 1A/cm^2 electrolysis and fuel cell operation without exhibiting degradation meeting the desired targets as outlined in the technical approach.

Year 1, M9-M12, Q3-Q4:

Task 3.0. Computational identification of multi-layer stack designs for PEMWE and PEMFC to obtain targeted fuel rates and desired target power (40W), respectively using minimal stack layers.

Task 3.1: Fabrication of theoretically predicted stacks with the desirable number of MEAs to generate the targeted fuel production rates and achieve the desired power to be delivered.

Task 3.2: Combine the developed PEMWE and PEMFC subsystems described in the technical approach to achieve the desired 40W power system.

Task 3.3: Determine and analyze the costs of energy consumption and fuel production in the fabricated PEMWE combined with the energy production costs during the fabricated PEMFC operation.

Task 3.4: Final analysis of collected data and compilation of the results, completion and submission of final quarter findings and annual reports.

Milestone 3.1: Demonstrate a compact system comprising of PEMWE and PEMFC subsystem able to deliver 40W .

The project began with initiating Task 1 and completion of all the subsequent tasks planned to be completed within the one-year project. However, some of the tasks had to be modified and altered following discussions with the Program Manager, Dr. Maria Medeiros. These changes and alterations are described below.

What was accomplished towards achieving these goals?

The project was initiated, and all the experiments and modeling studies have been completed. During the twelve-month duration of the project, the following studies were initiated and there were changes and modifications done to the original plan of work which are described below.

The main aim of the present project is to design a small-scale system comprising of a portable stacked PEMFC combined with fuel supplying PEMWE capable of providing the targeted DC power (40 W) to drive the underwater unmanned vehicle (UUV).

Thus, a suitable electrode design of stacked cells in PEMFC along with PEMWE needs to be demonstrated.

Accordingly, the following milestones were designed and planned to be completed:

Milestone 1 (M1-M4): Demonstrate large area electrode ($\sim 5\text{cm}^2$) OER/HER ECs for PEMWE and ORR/HOR ECs electrode for PEMFC exhibiting targeted performance of $> 300\text{ mA/cm}^2@0.8\text{V}$ and rated power $> 1\text{W/cm}^2@0.7\text{V}$, sustained 5000h electrolysis and fuel cell operation without degradation.

Milestone 2 (M5-M8): Demonstrate single cell large area electrode ($\sim 5\text{cm}^2$) MEA for PEMWE and PEMFC giving apt performance ($>300\text{mA}$, $@0.8\text{V}$, $>1\text{W/cm}^2@0.7\text{V}$), sustained 5000h electrolysis and fuel cell operation without degradation.

Milestone 3 (M9-M12): Demonstrate a compact system comprising of PEMWE and PEMFC subsystem able to deliver 40W power.

However, during the second quarterly review meeting held on July 27, 2021, recommendation was made by Dr. Medeiros to explore possible development of the Unitized Regenerative PEMFC (UR-PEMFC) utilizing the principal investigator's (PI's) proprietary and patented, two ***bi-functional*** electrocatalysts: PGM-free Cu-Mn-O:F for OER/ORR and low PGM-containing Co-Ir alloy for HER/HOR reactions.

Following this recommendation, the present project objectives were slightly modified in terms of selection of the main electrocatalysts planned for simultaneous use in PEM fuel cell and PEM water electrolyzer sub-units.

Exploiting the PI's developed bi-functional electrocatalysts the goal is to explore construction of UR-PEMFC with targeted performance values and reduced price per Watt of output cell power as well as per kg of generated H₂/O₂ per the **Table 1** and **Table 2** below:

| Characteristics | PEM fuel cell (single cell) | PEM water electrolysis |
|-----------------------------|-------------------------------|----------------------------|
| Performance | ~300 mA/cm ² @0.7V | < 40kWh /kg H ₂ |
| Rated maximum power density | ≥ 1.5W/cm ² | |
| Durability | 5000 h @ rated power | 5000 h @1.45V |

Table 1: Performance and durability for PEMFC and WE

| Characteristics | unit | Cathode Cu _{1.5} Mn _{1.5} O ₄ :10F | Anode Co _{0.6} Ir _{0.4} | Target (2020) |
|-------------------|--------------------|--|--|------------------|
| Performance @0.7V | mA/cm ² | ~280 | ~900 | 300 |
| Durability | hours | 48h | 24h | 5000 |

Table 2: Electrode performance and durability with cycling

Accordingly, the current project tasks to be completed were as follows:

Year 1, Month 1 (M1)-Month 3 (M3), Quarter 1 (Q1):

Task 1.0: Theoretical and computational identification of optimal electrode parameters of optimal ECs loadings, coating thickness and electrode area to meet target response of 300mA/cm² @ 0.7V and maximum power density ≥ 1.5 W/cm² for all synthesized ECs for UR-PEMFC (**Tables 1, 2**).

Milestone 1.0: Develop a reliable and robust multi-functional computational model capable to identify optimal electrode parameters for UR-PEMFC.

Year 1, Month 4 (M4)-Month 6 (M6), Quarter 2 (Q2):

Task-1.1: Laboratory scale 5 cm² area electrode will be fabricated using theoretically identified optimized electrode thickness and porosity utilizing different ink formulations to achieve the targeted bifunctional electrocatalysts performance. **Subtask-1.1.1:** Electrode structure, composition, microstructure, and particle size distribution characterization will be performed.

Milestone 1.1: Demonstrate the feasibility of electrode (~5cm²) fabrication with desired electrode loading and porosity to achieve the targeted performance (**Table 1**).

Year 1, Month 7 (M7)-Month 9 (M9), Quarter 3 (Q3):

Task-1.2: A detailed electrochemical impedance spectroscopy analysis will be carried out to evaluate the effects of, **Subtasks-1.2.1** electrode thickness and **Subtasks-1.2.2.** porosity on mass and charge transfer of the electrocatalysts (ECs).

Milestone 1.2: Optimization of the electrode thickness and porosity with minimum charge transfer resistance while achieving the targeted performance.

Year 1, Month 10 (M10)-Month 12 (M12), Quarter 4 (Q4):

Task 1.3: A detailed electrochemical performance study using cyclic voltammetry (CV) and linear sweep voltammetry (LSV) will be conducted on optimized electrodes developed in Task 1.2 to evaluate effects of electrode thickness (**Subtask 1.3.1**), and porosity (**Subtask 1.3.2**) on activity and durability of the synthesized bifunctional electrocatalysts (ECs). Annual report submission.

Go/No-Go: Demonstrate laboratory scale electrode ($\sim 5\text{cm}^2$) exhibiting targeted performance and sustained 5000h electrolysis and fuel cell operation without degradation.

All of the above tasks were initiated and completed. All the bifunctional electrocatalysts of solid solution Co-Ir alloy ($\text{Co}_{0.6}\text{Ir}_{0.4}$) for HER and HOR, and solid solution electrocatalyst of $\text{Cu}_{1.5}\text{Mn}_{1.5}\text{O}_4:10\text{F}$ (CMO:10F) for OER and ORR (OER/ORR) were synthesized and their performance was demonstrated. Recommendations were also made by the program manager, Dr. Maria Medeiros to modify the CMO:10F composition for achieving improved ORR activity. These studies are planned for the continuation of the grant which is currently on-going. Additionally, the goal is to demonstrate the performance of these two bifunctional electrocatalysts in 1 cm^2 ; 5 cm^2 ; 40 cm^2 and possibly, 120 cm^2 MEA in both water electrolyzer as well as in PEM fuel cell operation. Finally, the goal is to demonstrate the function of these two bifunctional electrocatalysts in a unitized regenerative PEMFC. These studies are planned in the continuation of the grant which is currently on-going.

What opportunities for training and professional development did the project provide?

A diverse group of undergraduates including a summer undergraduate minority research intern from University of Texas, Austin, Kiet Luan, high school students and Ph.D. students worked as integral members of the principal investigator (PI) and co-principal investigator's (Co-PI's) research team. In particular, Matthew Criado is an exceptional PhD student that works in the PI's laboratory in the field of energy storage materials and proton exchange membrane (PEM) water electrolysis. Further, efforts will be made to recruit students from upper-level middle schools as well to expose them to the scientific understanding of water electrolysis in general and PEM water electrolysis combined with PEM fuel cells, in particular. The PI is planning therefore, not only make considerable efforts to recruit under-represented and minority students throughout the duration of this grant but also provide unique opportunities for these aspiring students to get exposed to the state-of-the-art facilities available in the PI's laboratory.

How were the results disseminated to communities of interest?

The results will be presented in various conferences for rapid release of the advances made to the diverse community comprising materials scientists, chemical engineers, electrical engineers,

electrochemists and solid-state chemists. The principal investigator (PI) and co-principal investigators (Co-PI's) are also active in the American Ceramic Society (ACerS), Materials Research Society (MRS), American Institute of Chemical Engineers (AIChE) and the Electrochemical Society (ECS) wherein they have been instrumental in organizing new technical symposia focusing on batteries, supercapacitors, and fuel cells. Advances will be made in the basic understanding of the synthesis, fabrication, interface stability and reactions, including changes in the microstructure and ensuing electrochemical reactions, as well as comparison with theory. Results of these studies will be published in peer reviewed archival journals. Significant achievements made during this project will be posted on the internet website: <http://www.engr.pitt.edu/Chemical/facstaff/kumta.html>; the university homepage of the PI. The website will also serve as a laboratory notebook site and hence, will also act as a medium for exchanging the results and initiating stimulating discussion between the individual group members.

What plans were executed during the current reporting period to accomplish the goals and objectives?

The project was initiated with experimental and modeling studies. During the past 12 months, all the tasks outlined above were executed in the project and the results are described in the sections to follow.

What honors or awards were received under this project in this reporting period?

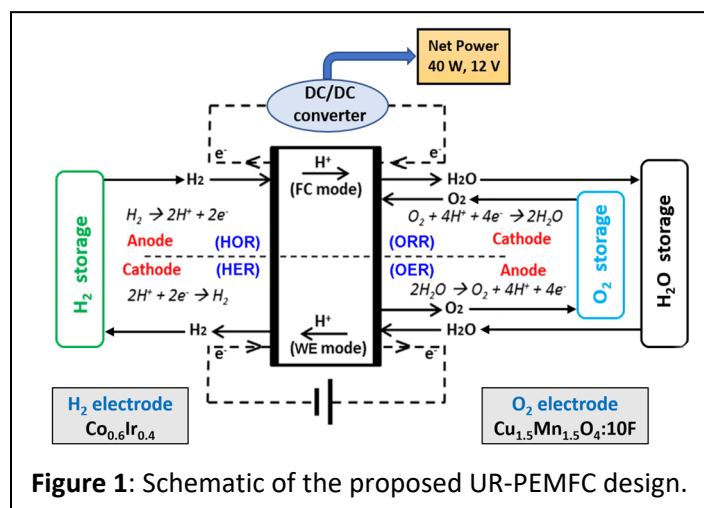
Nothing to report.

The technical targets of the present project for unitized regenerative fuel cell (URFC) in the PEMFC mode and PEMWE mode are shown in **Table 3**.

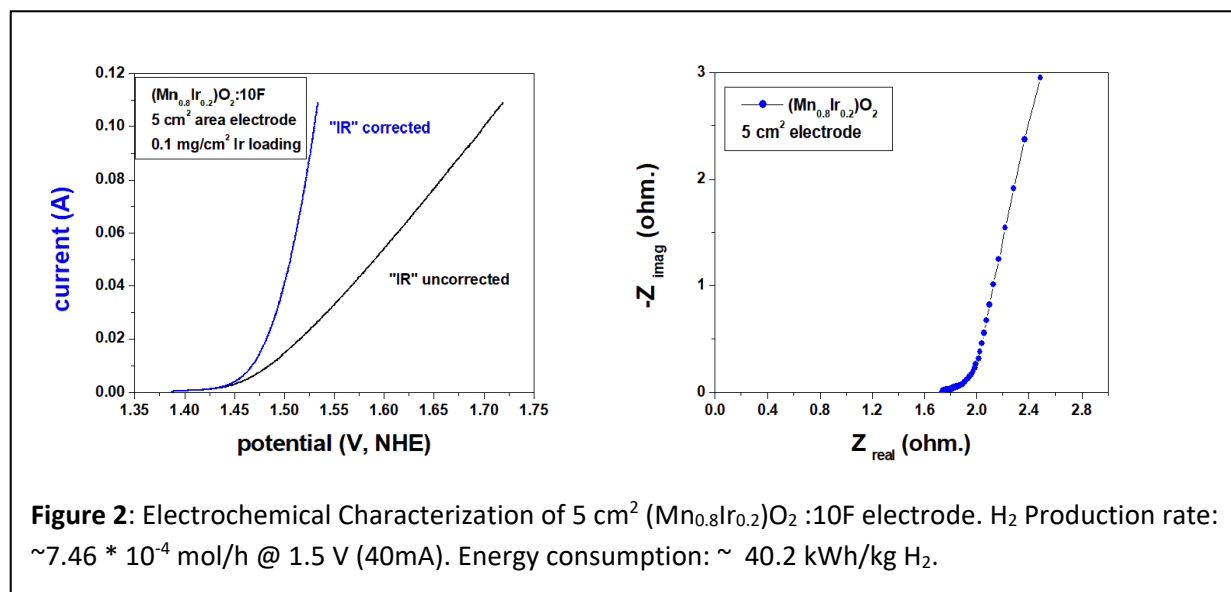
| Characteristics | PEMFC (single cell) | PEMWE |
|------------------------|--|------------------------------|
| Performance | 300mA/cm ² @0.8V; Rated power:>1W/cm ² @0.7V | ~ < 40 kWh/kg H ₂ |
| Durability | 5000 h @ 1A/cm ² | 5000 h @1.45V |

Table 3: Technical targets for PEMFC and PEMWE in the present project.

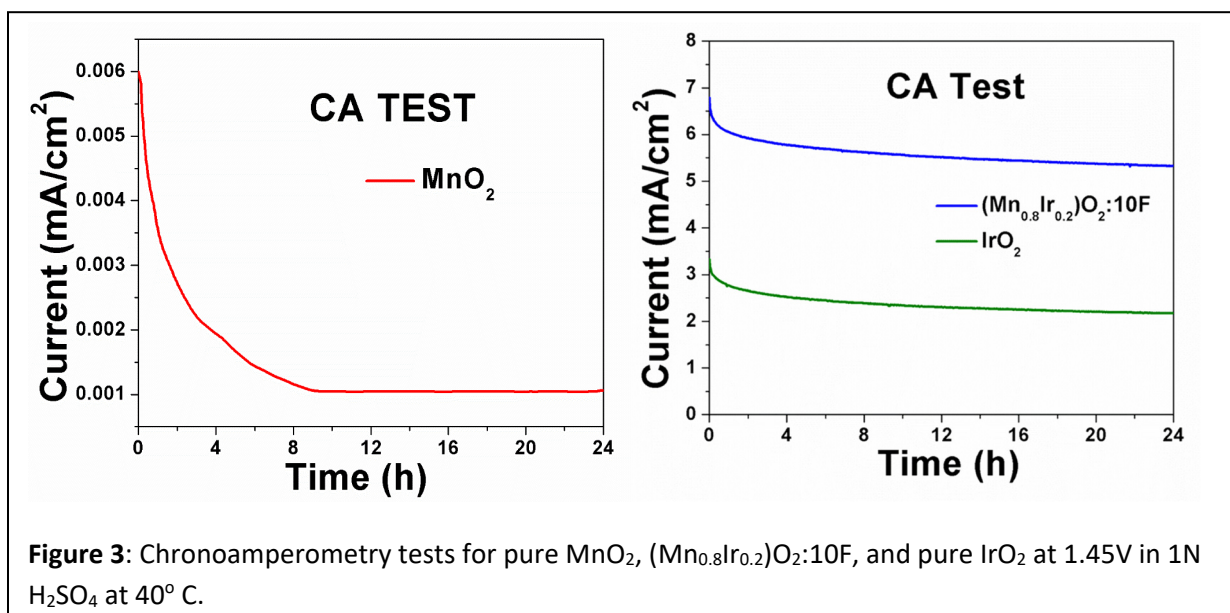
Figure 1 shows the schematic of the proposed UR-PEMFC design that will be made and executed.



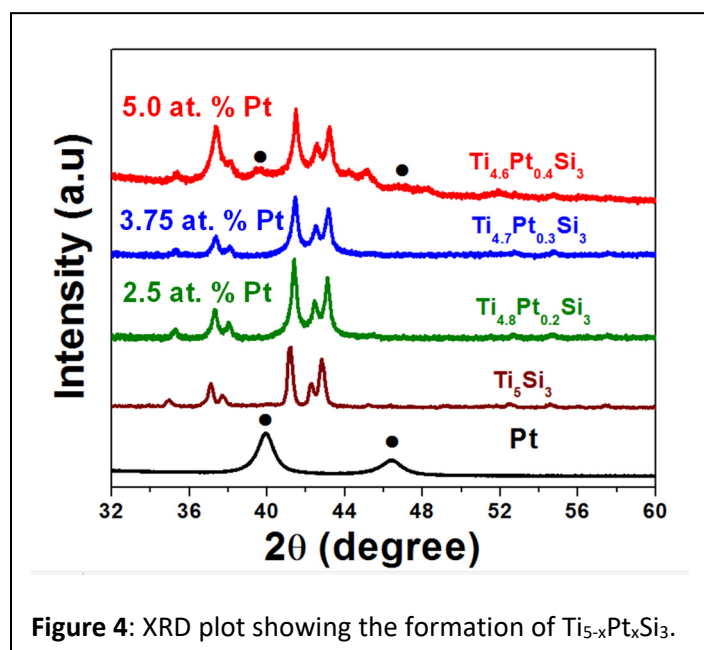
The design involves utilizing the bi-functional electrocatalysts identified for HER/HOR and OER/ORR as indicated above. These include solid solution alloys of $\text{Co}_{0.6}\text{Ir}_{0.4}$ alloys for HER/HOR and solid solution electrocatalysts of $\text{Cu}_{1.5}\text{Mn}_{1.5}\text{O}_4:10\text{F}$ for OER/ORR. The bifunctional electrocatalysts will be utilized to create membrane electrode assemblies (MEAs) for both PEM-based water electrolysis and PEM-based fuel cells.



As part of the original project milestones and tasks outlined earlier, the PI initially demonstrated efficacy of synthesized electrocatalysts for OER applications. Accordingly, the PI and the team synthesized solid solution electrocatalyst of $(\text{Mn}_{0.8}\text{Ir}_{0.2})\text{O}_2 :10\text{F}$ and characterized it for generation of hydrogen following the PI and the team's published protocol (Shrinath Dattatray Ghadge, Prasad Prakash Patel, Moni Kanchan Datta, Oleg I. Velikokhatnyi, Ramalinga Kuruba, Pavithra M. Shanthi and Prashant N. Kumta, Fluorine substituted $(\text{Mn},\text{Ir})\text{O}_2:\text{F}$ high performance solid solution oxygen evolution reaction electro-catalysts for PEM water electrolysis, RSC Adv., 2017, 7, 17311). As shown in **Figure 2**, the synthesized electrocatalyst exhibits a current of 40 mA at 1.5V when tested in $1\text{N H}_2\text{SO}_4$ for a 5 cm^2 working electrode. This corresponds to a hydrogen production rate corresponding to $\sim 7.46 \times 10^{-4} \text{ mol/h}$ of hydrogen at 1.5V giving a current of 40 mA . This also relates to an energy consumption of $\sim 40.2 \text{ kWh/kg H}_2$. The system also shows reduced charge transfer resistance.



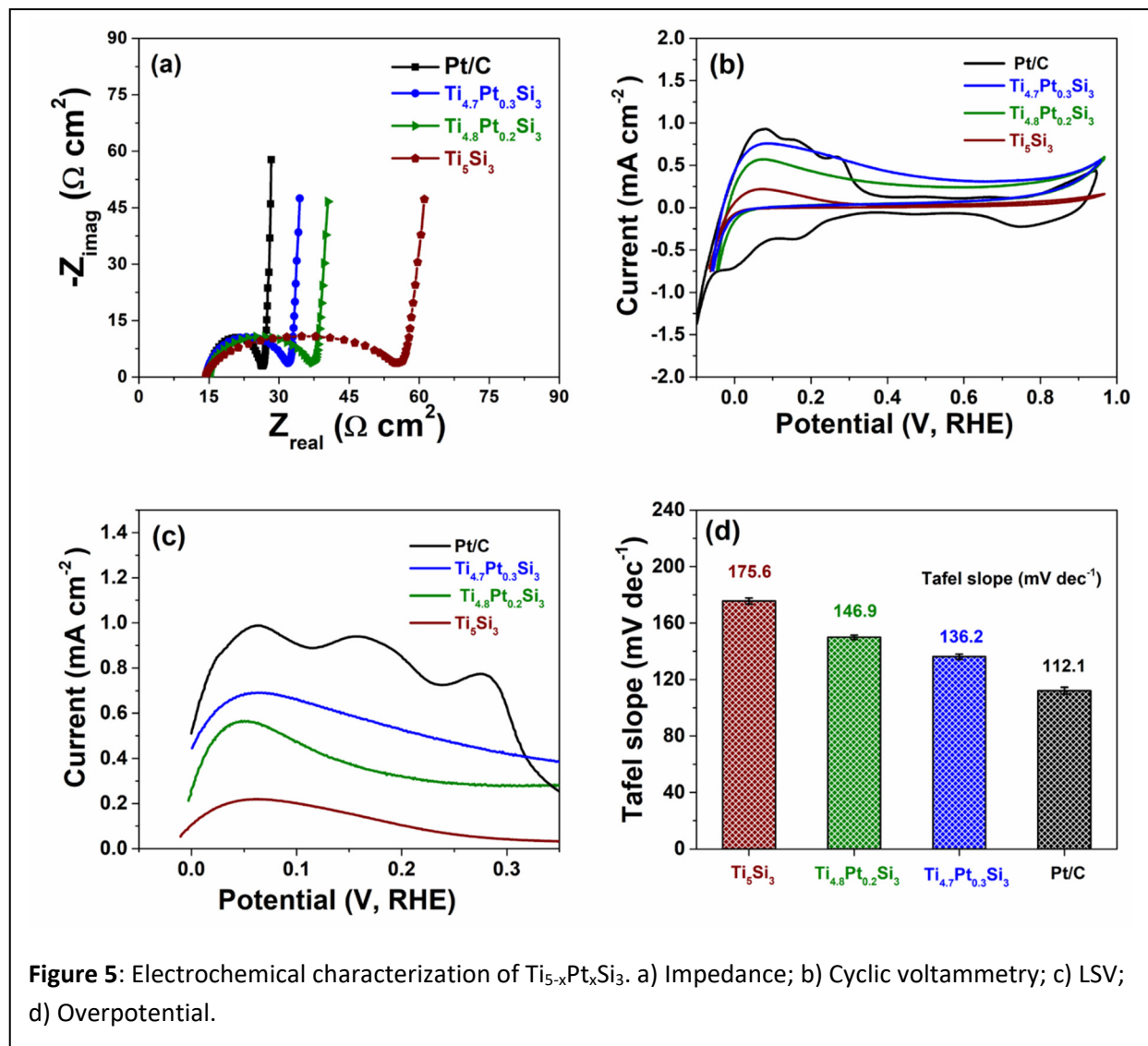
Stability of the solid solution electrocatalyst of $(\text{Mn}_{0.8}\text{Ir}_{0.2})\text{O}_2:10\text{F}$ was also tested by performing chronoamperometry (CA) tests. The results shown above in **Figure 3** shows that the synthesized electrocatalyst of $(\text{Mn}_{0.8}\text{Ir}_{0.2})\text{O}_2:10\text{F}$ is much more stable than in-house synthesized IrO_2 at 1.45V in 1N H_2SO_4 at 40°C. On the other hand, pure MnO_2 is extremely unstable undergoing rapid dissolution as shown in **Figure 3** at 1.45 V in 1 N H_2SO_4 at 40°C.



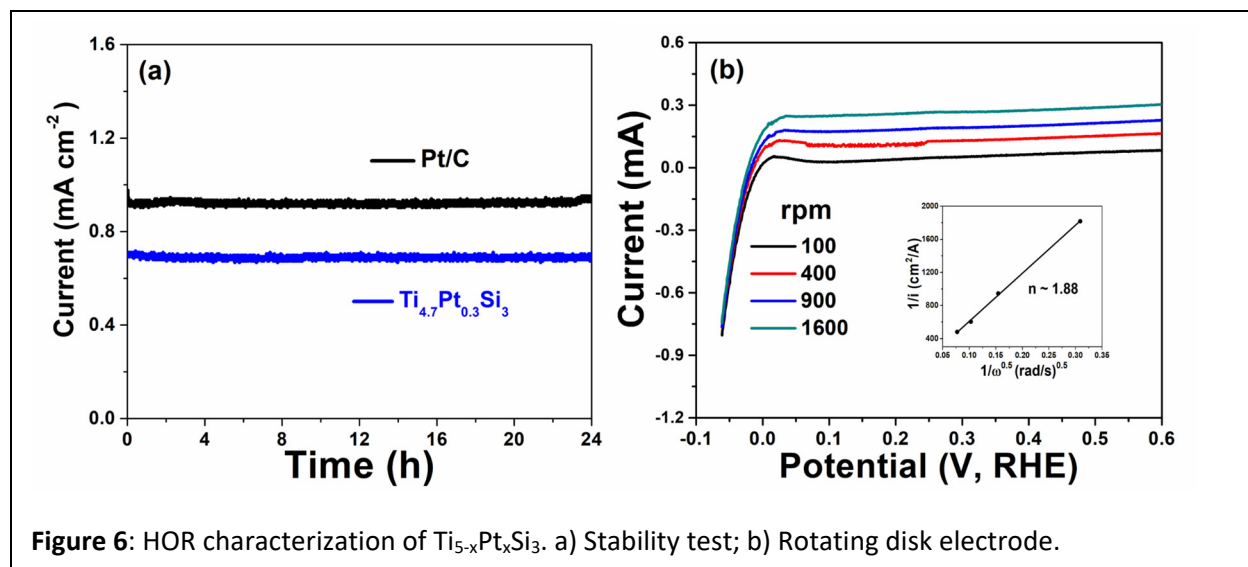
In the pursuit of alternative and promising hydrogen oxidation reaction (HOR) electrocatalyst system, the PI and the team also identified a new electrocatalyst in the Ti-silicide family. Correspondingly the team identified and synthesized Ti_5Si_3 , the XRD plot for which is shown in **Figure 4**. The titanium silicide was then intentionally doped with Pt to achieve the desired electrochemical activity and stability. Correspondingly, these systems were synthesized by mechanical milling. The system could incorporate a maximum amount of 3.75 at. % beyond which there is phase separation displaying the separated peaks of Pt. As shown in

Figure 5, the system shows excellent electrochemical response for HOR with the optimal system, $\text{Ti}_{4.7}\text{Pt}_{0.3}\text{Si}_3$ showing the best response of lower charge-transfer resistance of R_{ct} of $16 \Omega\text{-cm}^2$

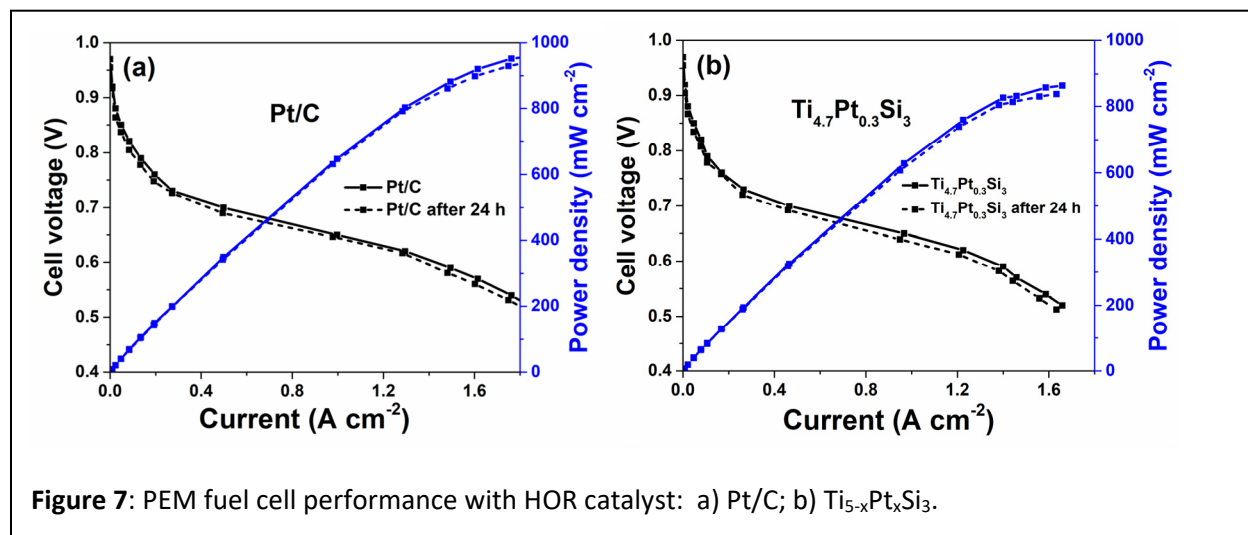
similar to Pt showing $14 \Omega\text{-cm}^2$ while displaying a current density of $0.75 \text{ mA}\cdot\text{cm}^{-2}$ similar to that of Pt which displays a current density of $0.9 \text{ mA}\cdot\text{cm}^{-2}$.



The corresponding Tafel slopes for the various synthesized undoped and Pt-doped Ti-silicides are shown in **Figure 5**. The $\text{Ti}_{4.7}\text{Pt}_{0.3}\text{Si}_3$ electrocatalyst again displays a lower Tafel slope ($136.2 \text{ mV dec}^{-1}$) commensurate with a reduced overpotential compared to the undoped silicide, Ti_5Si_3 ($175.6 \text{ mV dec}^{-1}$) electrocatalyst and in the same range as that of Pt/C of $112.1 \text{ mV dec}^{-1}$.

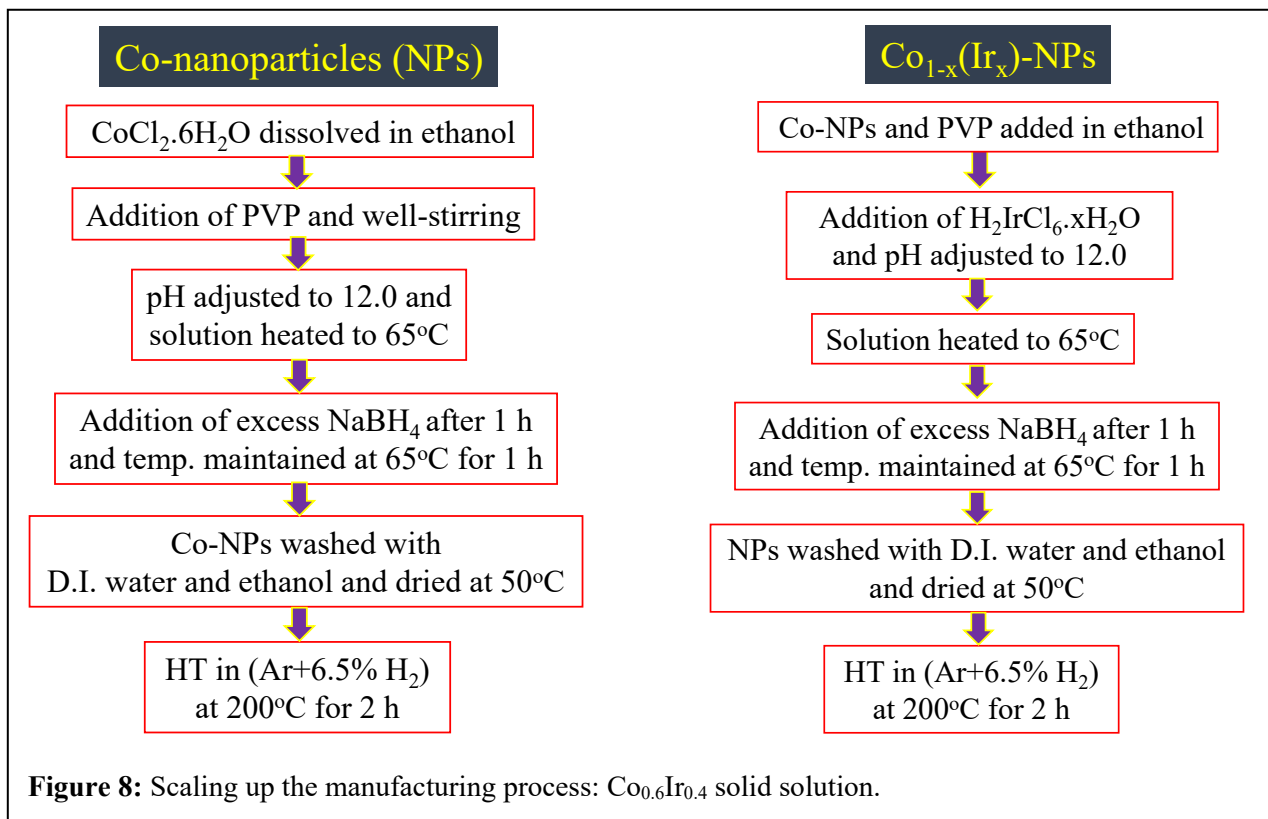


The electrochemical stability of the synthesized $\text{Ti}_{4.7}\text{Pt}_{0.3}\text{Si}_3$ electrocatalyst performed at 0.05 V shown in **Figure 6** also shows comparable electrochemical stability as that of Pt/C. Rotating ring disk electrode characterization tests conducted shows that the number of electrons produced is ~ 1.88 validating the two-electron pathway for creating the hydrogen ions via oxidation of hydrogen.



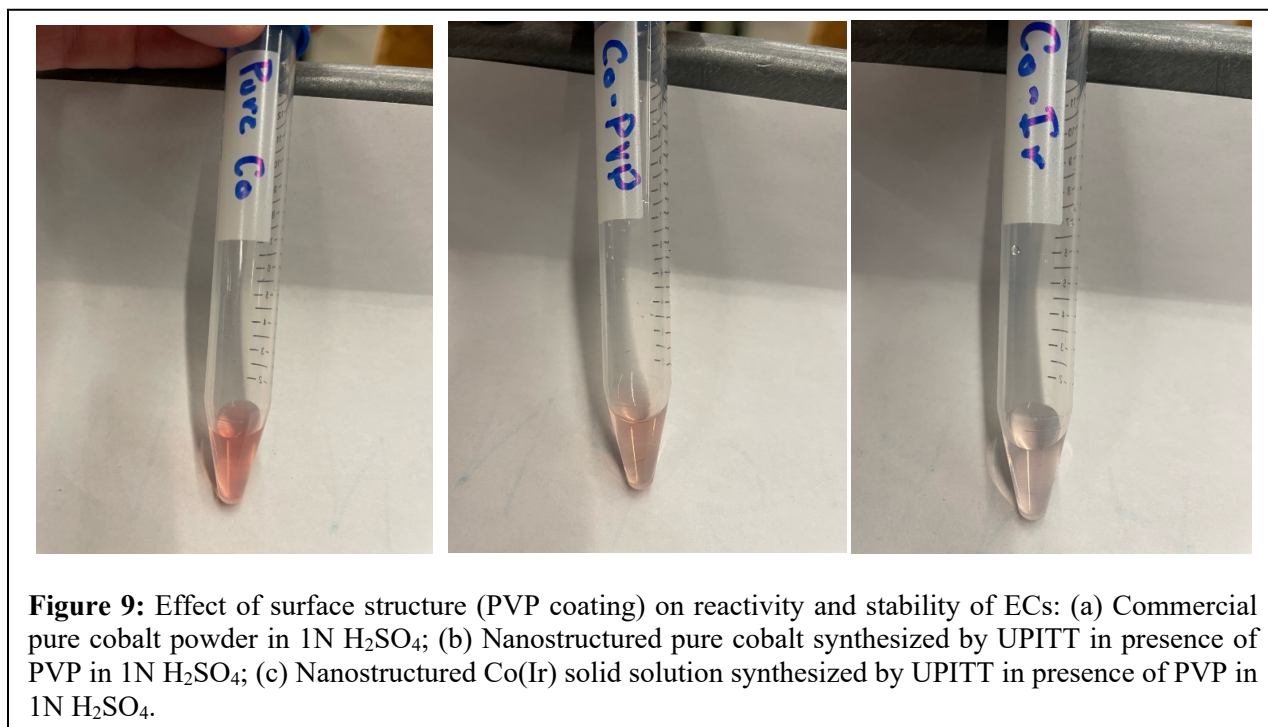
The novel electrocatalyst system of $\text{Ti}_{4.7}\text{Pt}_{0.3}\text{Si}_3$ was also tested in a single fully assembled PEM fuel cell to evaluate its performance in a single full PEMFC and compare against the state-of-the-art single full cell of PEMFC using Pt/C electrocatalyst. Thus, as shown in **Figure 7**, the performance of $\text{Ti}_{4.7}\text{Pt}_{0.3}\text{Si}_3$ used as the anode and Pt/C used as the cathode in the single full PEMFC is displayed in **Figure 7(b)**. **Figure 7(a)** shows the corresponding performance of the single full PEMFC using Pt/C as the anode and cathode electrocatalysts. Hydrogen was passed at 200 ml min^{-1} while oxygen was flown at 300 ml min^{-1} at a temperature of 80°C . As shown in **Figures 7**, the power density of the fuel cell using $\text{Ti}_{4.7}\text{Pt}_{0.3}\text{Si}_3$ electrocatalyst is comparable to the fuel cell using Pt/C as the electrocatalyst electrodes. Accordingly, the PEMFC using Pt/C electrocatalysts

shows the maximum power density of 960 mW/cm² while the single PEMFC using Ti_{4.7}Pt_{0.3}Si₃ electrocatalyst shows a maximum power density of 855 mW/cm² indicating a comparable electrochemical response of the synthesized Ti_{4.7}Pt_{0.3}Si₃ electrocatalyst.



Efforts were made to synthesize solid solution alloy electrocatalysts of Co_{0.6}Ir_{0.4} alloys using solution-based synthesis approach to generate nanoparticles of the alloy. **Figure 8** shows the synthesis scheme that was followed to generate the nanoparticles of Co following which the Ir nanoparticles are generated by reduction of the chloride salt using sodium borohydride. The reduction reaction creates Ir nanoparticles on the surface of the initially synthesized Co nanoparticles. Subsequent heat treatment of the Co-Ir nanoparticles in Ar-6.5%H₂ results in the formation of the desired Co_{0.6}Ir_{0.4} nanoparticles in line with the PI and the team's published work (Prasad Prakash Patel, Moni Kanchan Datta, Oleg I. Velikokhatnyi, Prashanth Jampani, Daeho Hong, James A. Poston, Ayyakkannu Manivannan and Prashant N. Kumta, Nanostructured robust cobalt metal alloy-based anode electro-catalysts exhibiting remarkably high performance and durability for proton exchange membrane fuel cells, *J. Mater. Chem. A*, 2015, **3**, 14015-14032). The use and presence of polyvinylpyrrolidone as a surfactant is highly critical for the formation of nanoparticles as well as preserving the stability of the synthesized alloys in sulfuric acid. This is clearly evidenced from the color of the solution when the synthesized particles are dispersed in 1N H₂SO₄. As shown in **Figure 9**, the pure Co nanoparticles that were commercially obtained are completely soluble (notice the blue color) in 1N H₂SO₄. On the other hand, when the Co nanoparticles are synthesized in the presence of PVP, it can be seen from **Figure 9** that the particles are now sparingly soluble in 1N H₂SO₄. This clearly indicates the surface stabilizing action

of PVP used as a surfactant. Moreover, the nanostructured $\text{Co}_{0.6}\text{Ir}_{0.4}$ solid solution Co(Ir) alloys



synthesized also in the presence of PVP used as a surfactant are now insoluble when dispersed in 1N H_2SO_4 . To validate the solubility of Co in 1N H_2SO_4 , inductively coupled plasma optical emission spectroscopy (ICP-OES) was used to determine the presence of Co in the sulfuric acid solution. **Figure 10** validates the color observed in **Figure 9** clearly showing the reduced amount of Co present in the sulfuric acid solution when the nanoparticles are synthesized in the presence of PVP. In fact, the solid solution alloy of $\text{Co}_{0.6}\text{Ir}_{0.4}$ synthesized in the presence of PVP shows presence of ~ 1 ppm of Co while pure Co dispersed in 1N H_2SO_4 shows almost 6 ppm of Co dissolved in the electrolyte solution (see yellow highlighted region in **Figure 10**). The result clearly shows the excellent chemical stability of the nanostructured $\text{Co}_{0.6}\text{Ir}_{0.4}$ solid solution alloy in 1N H_2SO_4 . Further optimization of the surface structure with the optimal amount of PVP needed is currently ongoing to optimize and obtain maximum reactivity of the synthesized electrocatalyst with excellent stability and durability.

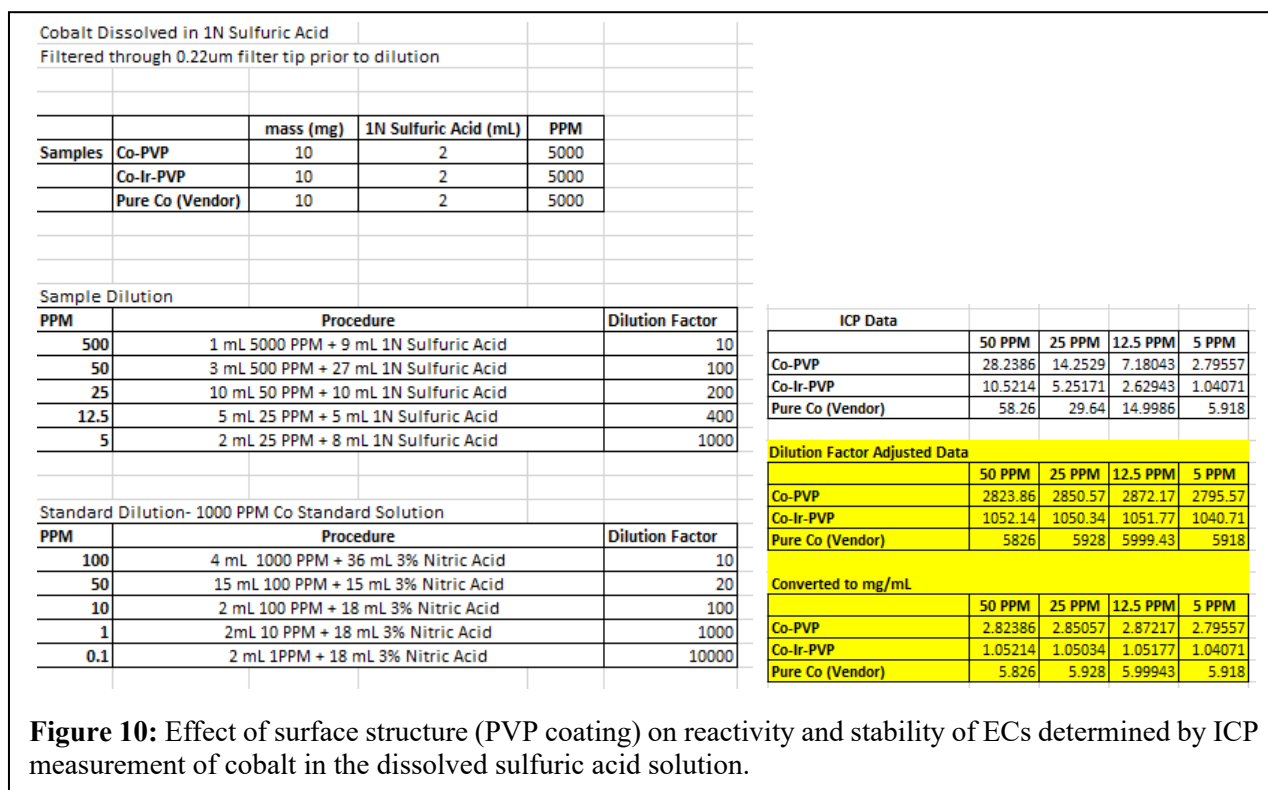


Figure 10: Effect of surface structure (PVP coating) on reactivity and stability of ECs determined by ICP measurement of cobalt in the dissolved sulfuric acid solution.

Following the analysis of the synthesized solid solution alloy of $\text{Co}_{0.6}\text{Ir}_{0.4}$ above indicating the use of polyvinylpyrrolidone (PVP) imparting the desired surface stability to the synthesized alloys, efforts were made to test the influence of PVP on the electrochemical activity as well. The results of the electrochemical testing conducted on the synthesized Co(Ir) solid solution alloys to assess the performance of these electrocatalysts for HOR and HER in acidic conditions are discussed in the sections to follow.

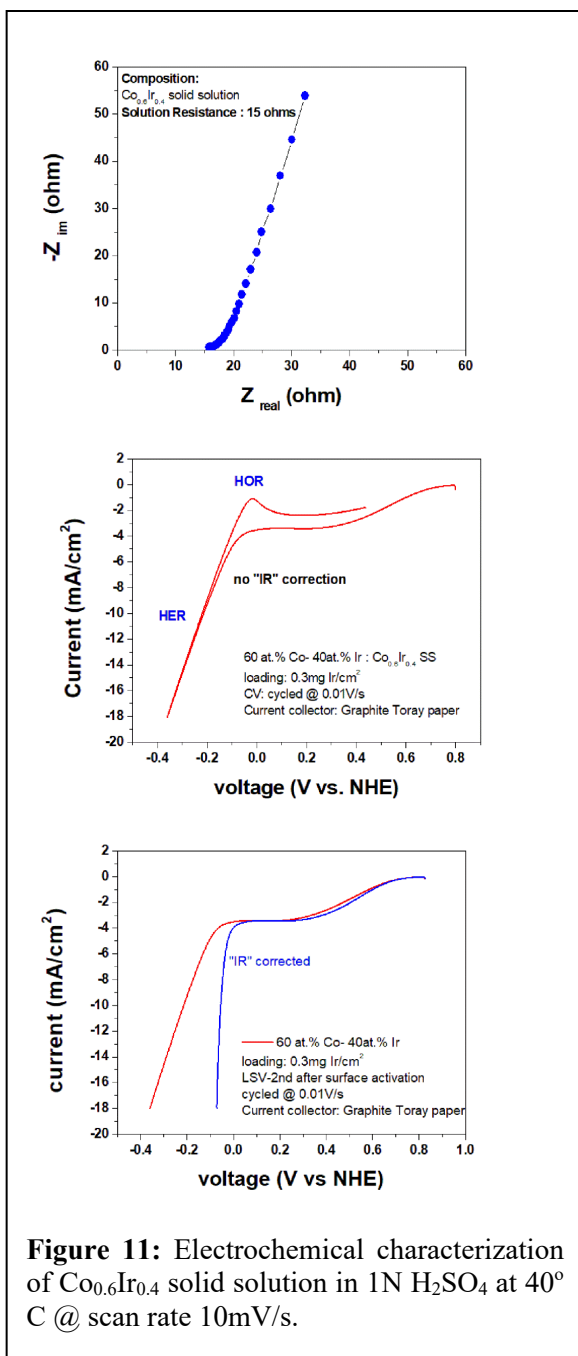
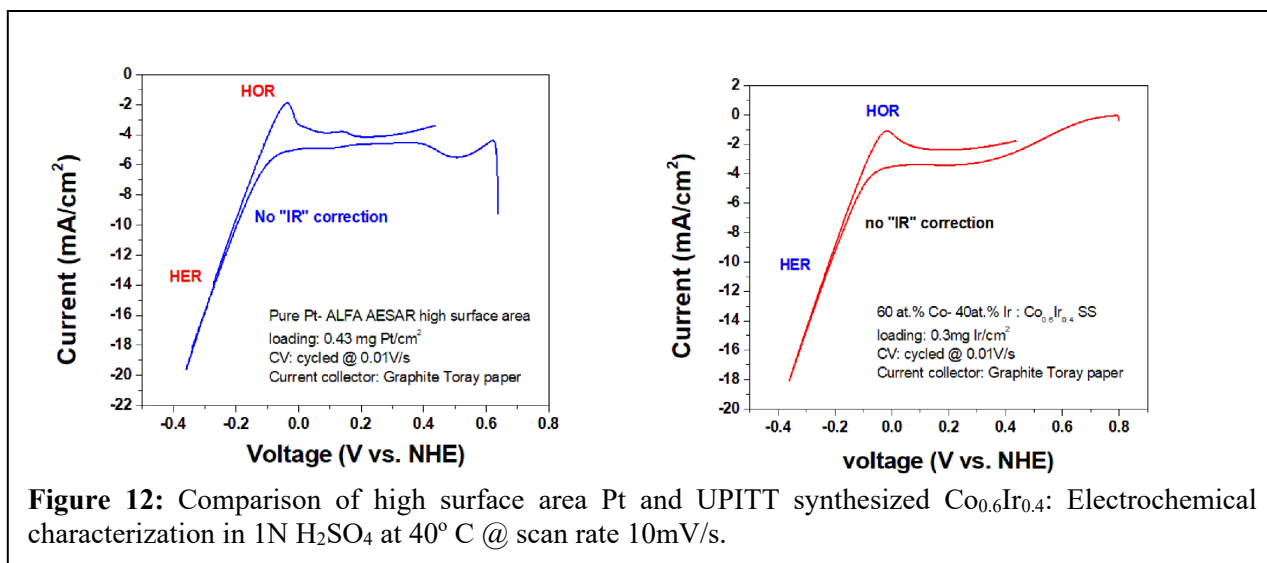


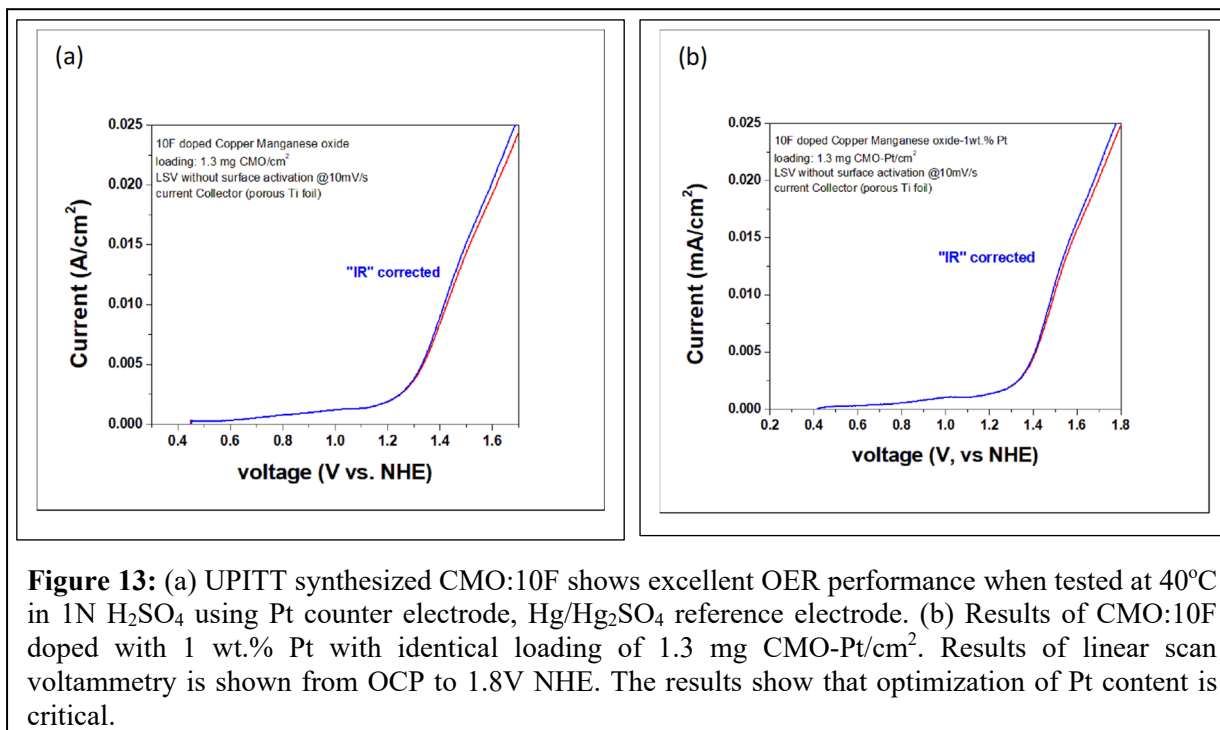
Figure 11 shows the results of the HOR, and HER electrochemical activity tests conducted on the synthesized solid solution $\text{Co}_{0.6}\text{Ir}_{0.4}$ alloys. The tests were done using 0.46 mg/cm^2 loading of the synthesized $\text{Co}_{0.6}\text{Ir}_{0.4}$ alloy corresponding to an Ir loading of 0.3 mg/cm^2 . The electrocatalysts were tested at 40°C in 1N H_2SO_4 using Pt as the counter electrode and $\text{Hg}/\text{Hg}_2\text{SO}_4$ as the reference electrode. The figure clearly shows that hydrogen is produced at 0.36V potential albeit without any IR correction corresponding to $\sim 14 \text{ mA}/\text{cm}^2$ which is comparable to the current generated by Pt black with a loading 0.43 mgPt/cm^2 . Furthermore, as shown in **Figure 12**, the current at -0.36V with no IR correction versus NHE is $\sim 14 \text{ mA}/\text{cm}^2$ for an overpotential of 8mV. This result matches that for the control of high surface area Pt/C black of specific surface area of $\sim 30.4 \text{ m}^2/\text{g}$ giving also a similar current density of $\sim 14 \text{ mA}/\text{cm}^2$ for an identical overpotential of 8 mV versus NHE but at a lower Co (Ir)loading of 0.46 mg/cm^2 corresponding to 0.3 mg/cm^2 , Ir loading.

The $\text{Co}_{0.6}\text{Ir}_{0.4}$ solid solution alloy also displays very good HOR response. As shown in **Figure 11** and **Figure 12**, the current at 0V versus NHE for the $\text{Co}_{0.6}\text{Ir}_{0.4}$ solid solution alloy is 2.8 mA/cm^2 comparable to that of Pt/C black which shows 3 mA/cm^2 . The Ir loading for the $\text{Co}_{0.6}\text{Ir}_{0.4}$ alloy is 0.4 mg/cm^2 while that of Pt/C black is 0.4 mg/cm^2 . These results therefore indicate that the nanoparticles of $\text{Co}_{0.6}\text{Ir}_{0.4}$ solid solution alloy display both HOR and HER performance matching that of Pt/C black for a lower loading

of 0.3 Ir compared to that of 0.43 mg/cm^2 for Pt/C black loading.

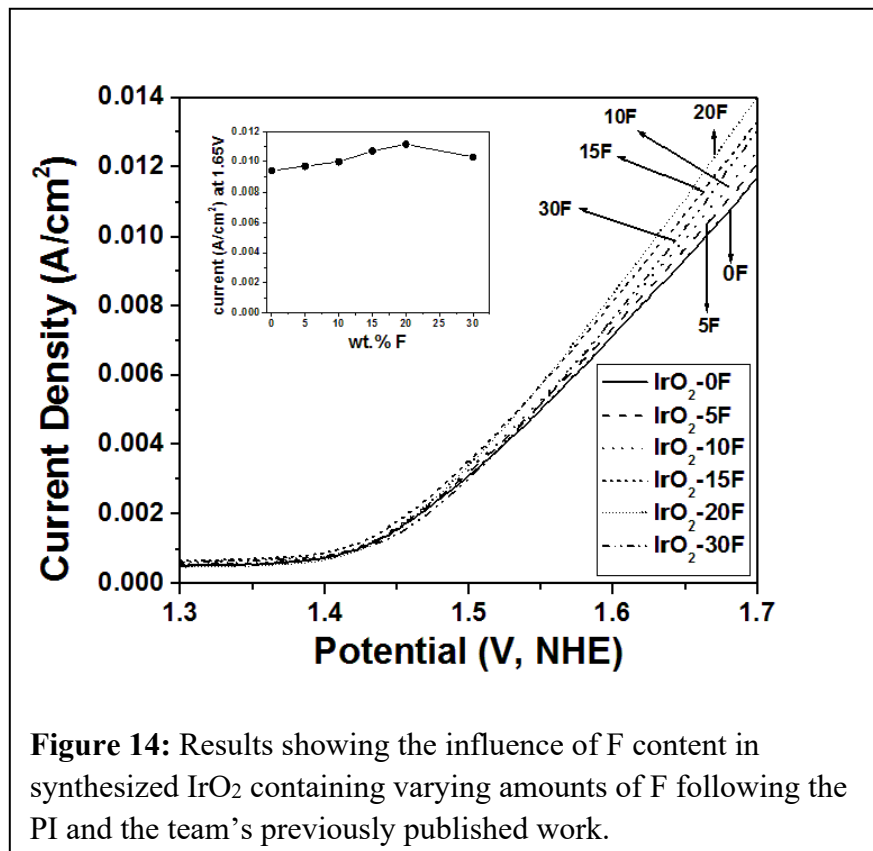


The result above validates the discussion showing the comparable HOR and HER performance of the chemically synthesized nanoparticles of $\text{Co}_{0.6}\text{Ir}_{0.4}$ solid solution alloy to that of Pt/C black. The results above also validate and illustrate the role of PVP in controlling the electrochemical activity of the solid solution alloy of $\text{Co}_{0.6}\text{Ir}_{0.4}$. There is a need to control the time-temperature-mass to achieve optimal carbon coating on the surface of the $\text{Co}_{0.6}\text{Ir}_{0.4}$ alloy. These studies are currently on-going to achieve better performance than high surface area Pt/C black.



Finally, efforts were made to chemically synthesize the bifunctional oxygen evolution reaction (OER) and oxygen reduction reaction electrocatalyst of $\text{Cu}_{1.5}\text{Mn}_{1.5}\text{O}_4$:10F following the PI and the team's published work (Prasad Prakash Patel, Moni Kanchan Datta, Oleg I. Velikokhatnyi, Ramalinga Kuruba, Krishnan Damodaran, Prashanth Jampani, Bharat Gattu, Pavithra Murugavel

Shanthi, Sameer S. Damle and Prashant N. Kumta, Noble metal-free bifunctional oxygen evolution and oxygen reduction acidic media electrocatalysts, Nature Scientific Reports, DOI: 10.1038/srep28367). To improve the performance of the base CMO:10F electrocatalyst, doping of 1 wt. % Pt into CMU:10F was also attempted. Results of the electrochemical testing are shown



in **Figure 13**. Results show that the synthesized CMO:10F shows excellent OER performance while the ORR performance testing is current ongoing. The F content is very critical for achieving the desired and optimal electronic conductivity for achieving the required preferred electrochemical response and stability. Incorporation of Pt is also vital for enhancing the ORR electrochemical performance. As shown in **Figure 14** following the PI and the team's published work (Karan Kadakia, Moni Kanchan Datta, Prashanth H. Jampani, Sung Kyoo Park, and

Prashant N. Kumta, Novel F-doped IrO₂ oxygen evolution electrocatalyst for PEM based water electrolysis, J. Power Sources, **222** (2013) 313-317), the F content in IrO₂ is vital for achieving improved current density and electrochemical response. Similarly, the incorporation of Pt will be critical for further enhancing the ORR electrochemical activity. The influence of Pt and F in controlling the electrochemical response is further elaborated and discussed in the section below.

Computational study of the present project:

The experimental study on $\text{Cu}_{1.5}\text{Mn}_{1.5}\text{O}_4:10\text{F}$ demonstrated excellent OER catalytic activity but relatively low ORR catalytic performance. To improve the ORR catalytic properties the first-principles density functional theory (DFT) computational study has been conducted to provide a theoretical basis for experimental synthesis of the catalyst with enhanced ORR catalytic activity. The main idea was to dope the basic $\text{Cu}_{1.5}\text{Mn}_{1.5}\text{O}_4:10\text{F}$ with certain amount of Pt infused into the crystal lattice.

Currently, in the theory of catalysis there is a well-known concept which suggests a gravity center of d-electronic band of the material as a single qualitative descriptor of the electrocatalytic activity for ORR and OER reactions. Using DFT methodology the first-principles calculations of the electronic structure of $\text{Cu}_{1.5}\text{Mn}_{1.5}\text{O}_4$ pure and doped with 10 wt. % of F along with the standard electrocatalysts – pure Pt and IrO_2 have been conducted and the results are shown on **Figure 15**.

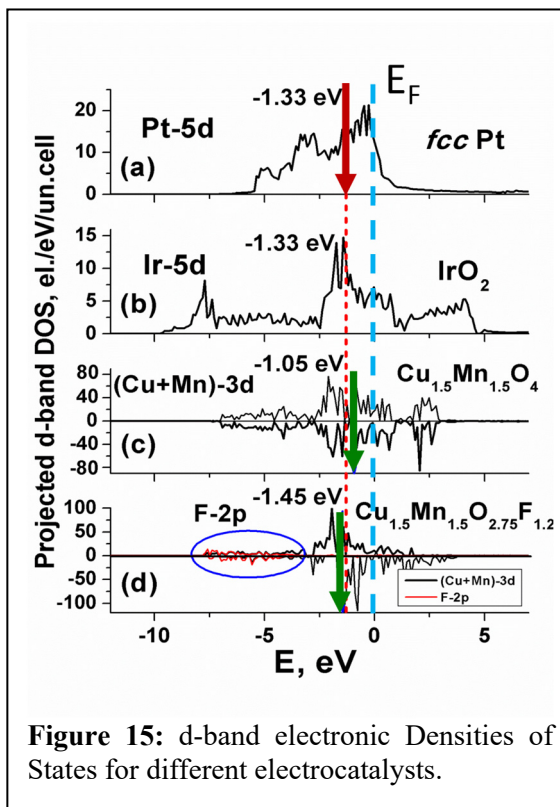


Figure 15: d-band electronic Densities of States for different electrocatalysts.

The DFT results demonstrate that the d-band centers for Pt and IrO_2 (marked with red dashed line and red arrow) locate both at -1.33 eV below Fermi level (E_F denoted with blue dashed line at 0 eV). Also, one can see that d-band center for undoped $\text{Cu}_{1.5}\text{Mn}_{1.5}\text{O}_4$ is located at -1.05 eV, while the same d-band center for F-doped $\text{Cu}_{1.5}\text{Mn}_{1.5}\text{O}_4$ is located at -1.45 eV. To improve the catalytic activity of these materials there is a need to bring the d-band center position into the vicinity of the corresponding value for pure Pt (-1.33 eV). Introduction of Pt into $\text{Cu}_{1.5}\text{Mn}_{1.5}\text{O}_4:10\text{F}$ should move the d-band center to the right towards the Pt d-band center position of -1.33 eV. Another approach to move the d-band center closer to the Pt d-band center, is to decrease the F-content.

Thus, Pt-doping along with optimization of F-content would certainly improve the electrocatalytic activity of $\text{Cu}_{1.5}\text{Mn}_{1.5}\text{O}_4:10\text{F}$ for both ORR and OER reactions. The corresponding experimental synthesis of the Pt-doped Cu-Mn-O-F oxides and optimization of the Pt and F contents is currently on-going and will be reported during the continuation of the present project.

COMSOL computational approach

1-D MEA model

The Finite Element Analysis computational method implemented in the software package COMSOL Multiphysics may help better understand effects of different geometric, physical, and electrochemical parameters on the overall performance of the PEM fuel cell and water electrolyzer, such as electrode layer thickness, catalyst loading, feeding flow rate, etc.

The simplified 1-D model used in the present project is shown on **Figure 16**. The modeled MEA consists of PEM membrane sandwiched between cathodic and anodic electrodes each containing Gas Diffusion Layer (GDL), microporous layer, and electrocatalytic layer. This model was used to calculate the polarization curves (applied current density vs. voltage generated by the fuel cell).

Figure 17 and **Figure 18** demonstrate the dependencies of the polarization curves on different parameters and the physical and electrochemical properties of MEA in PEMFC and in the water electrolyzer operation. Such plots will enable researchers to identify optimal geometric parameters and material characteristics needed for the design and

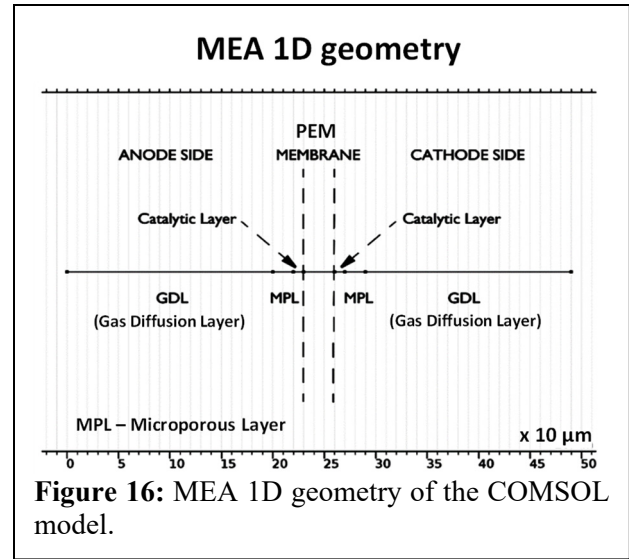


Figure 16: MEA 1D geometry of the COMSOL model.

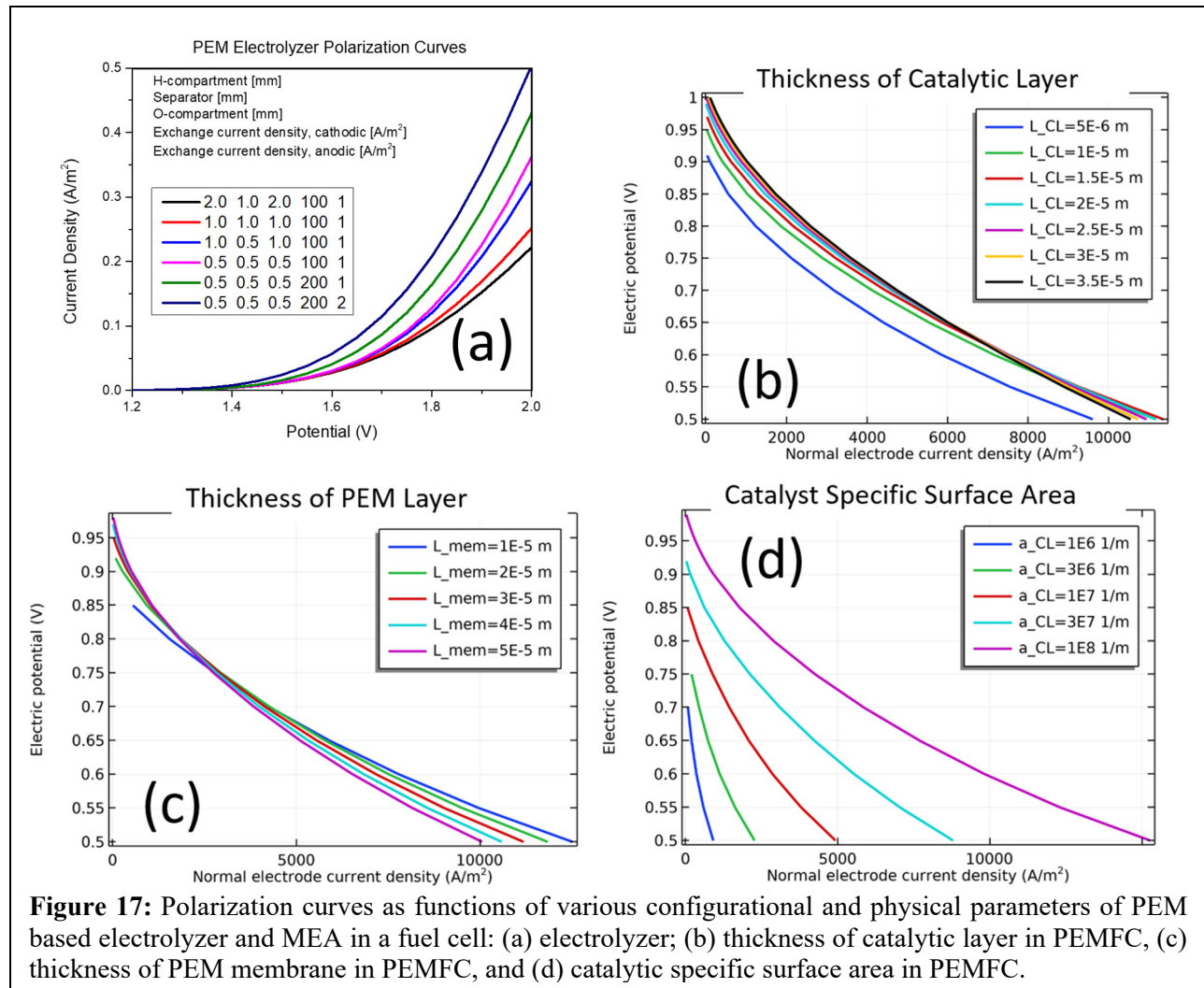
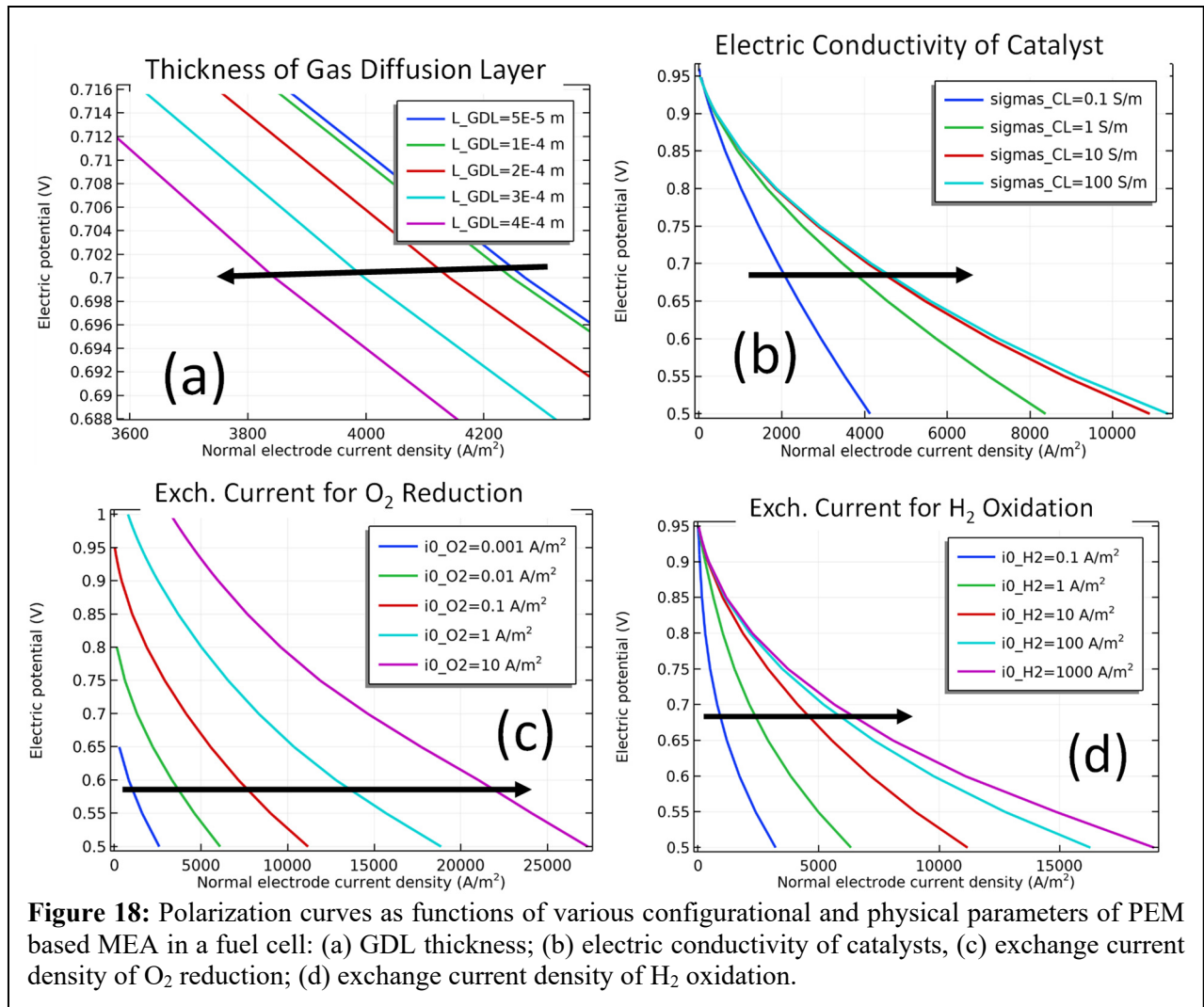


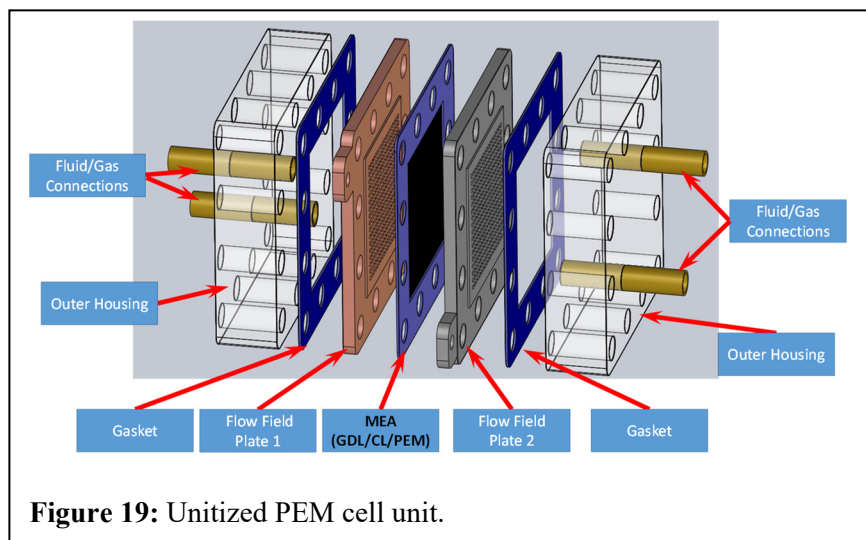
Figure 17: Polarization curves as functions of various configurational and physical parameters of PEM based electrolyzer and MEA in a fuel cell: (a) electrolyzer; (b) thickness of catalytic layer in PEMFC, (c) thickness of PEM membrane in PEMFC, and (d) catalytic specific surface area in PEMFC.



development of the fuel cells and water electrolyzers to achieve the given target performance rates collected in **Tables 1-3**. The role of the specific surface area of the electrocatalyst layer in the electrochemical performance of the PEMFC will be discussed in more detail later in the present report.

3-D PEMFC serpentine model

Based on the results obtained for 1-D MEA model the PI and the project team has developed more comprehensive 3-D model containing MEA with flow



field channels on both sides. This model shown in **Figure 19** and **Figure 20** will be scaled up for fabrication of larger MEA areas for use in single as well as in the stacked configurations. The general design of the model shown in **Figure 20** reflects a low temperature fuel cell with serpentine flow field patterns with a counter-flow type of flow, such that the oxygen and hydrogen inlet flow streams

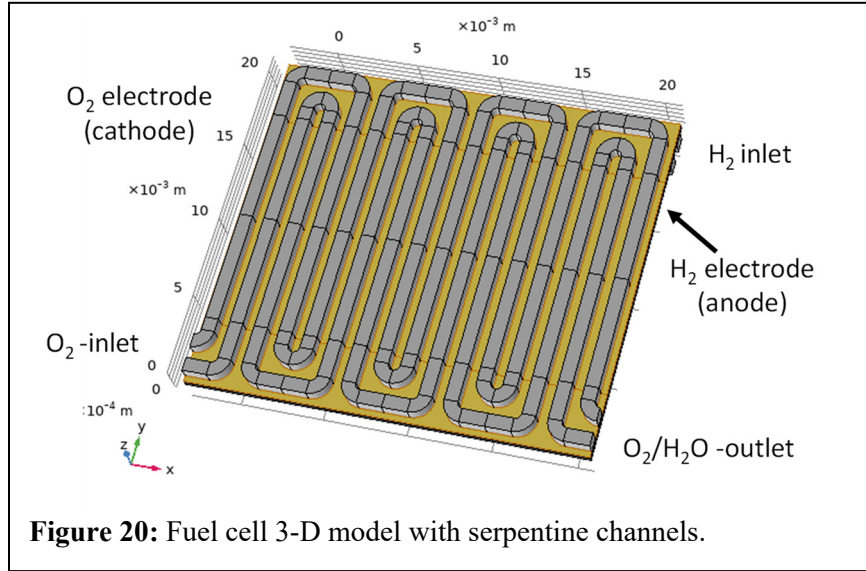


Figure 20: Fuel cell 3-D model with serpentine channels.

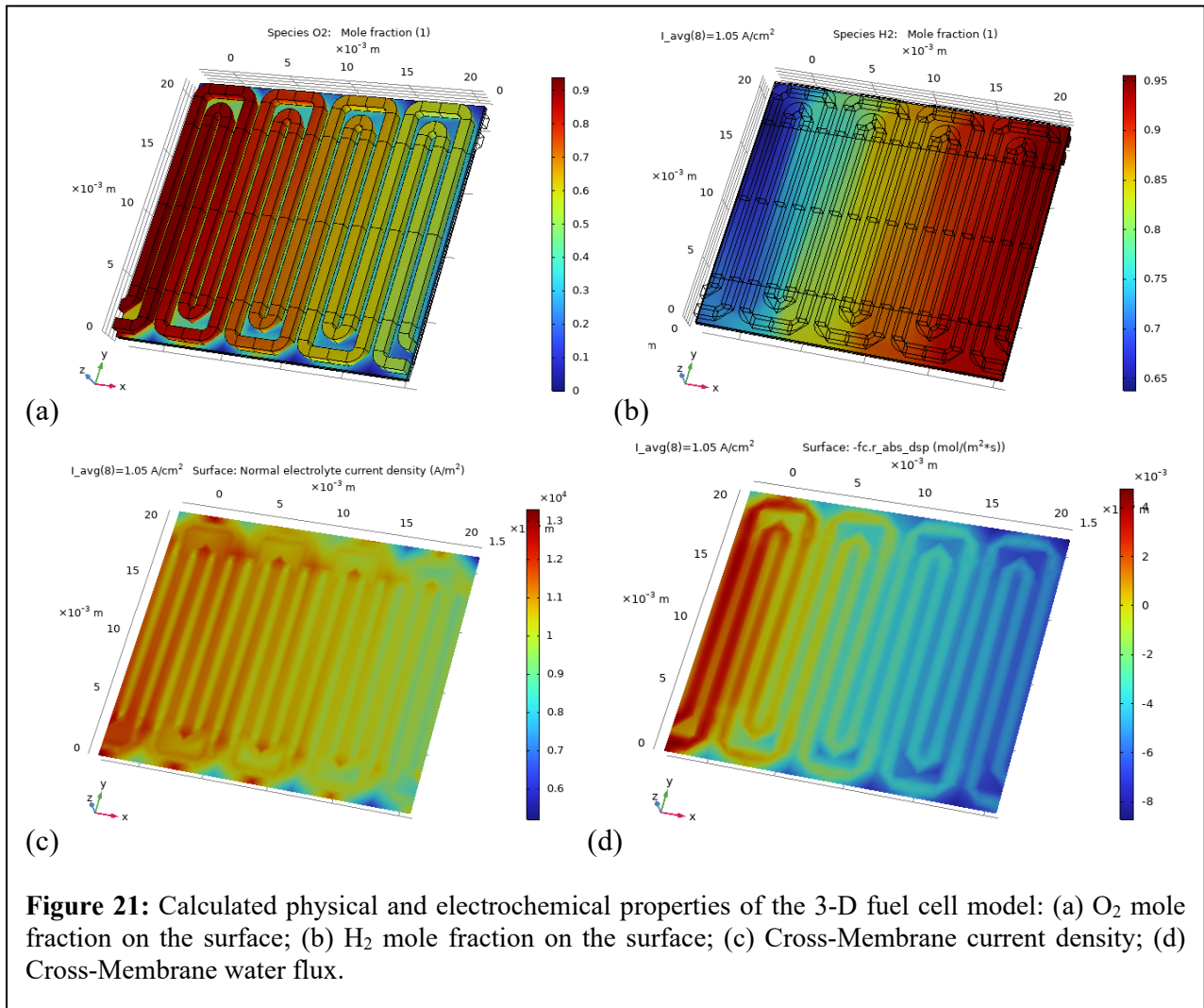


Figure 21: Calculated physical and electrochemical properties of the 3-D fuel cell model: (a) O₂ mole fraction on the surface; (b) H₂ mole fraction on the surface; (c) Cross-Membrane current density; (d) Cross-Membrane water flux.

are located at opposite sides of the cell.

All the present results are obtained for the model utilizing a 5cm^2 MEA area. The model solves for the charge balance in GDL and the membrane and for the mass transfer as molar fractions and momentum transport, such as pressure and velocity in the gas phase on each side of the membrane (see **Figure 16**). Also, the membrane transport of water is included in the model.

Figure 21 thus, demonstrates the distribution of gases and water flows as well as the current density during the functioning and operation of the fuel cell. In **Figure 21 (a)**, and **(b)** one can see the streamline of oxygen **Figure 21(a)** and hydrogen **Figure 21 (b)** as well as the corresponding molar fractions for current density of 1 A/cm^2 on the cathode and anode surfaces. The molar fractions decrease towards the outlets. Also, the graphs **Figure 21 (c)** and **Figure 21 (d)** in **Figure 21** demonstrate a cross-membrane current density and water flux, correspondingly. One can see that at different locations in the membrane the water flux has different directions because of different humidity conditions.

Figure 22 demonstrates the polarization curves calculated for the different geometric and electrochemical parameters using the abovementioned 3-D serpentine model of the PEMFC. These polarization curves are calculated for different working temperature, thicknesses of the electrocatalyst layers, various specific surface area (SSA) of the electrocatalyst, and the different exchange current densities for oxygen reduction. The topmost curve on the graph corresponds to working temperature of 60°C , electrocatalytic layer thickness of $20\mu\text{m}$, SSA of $\sim 40\text{ m}^2/\text{g}$ and an exchange current density for ORR of 0.2 mA/m^2 .

At these parameters, which are very realistic, the power density corresponds to $\sim 0.9\text{ W/cm}^2$. Moreover, these calculations have been performed with a coarse mesh of the 3-D model for faster estimation of the general trends and effects of the parametric variations.

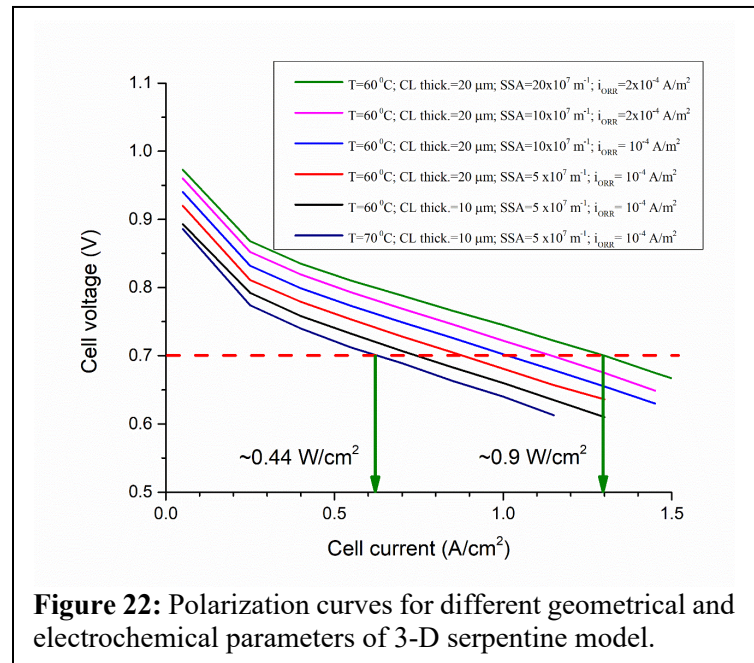
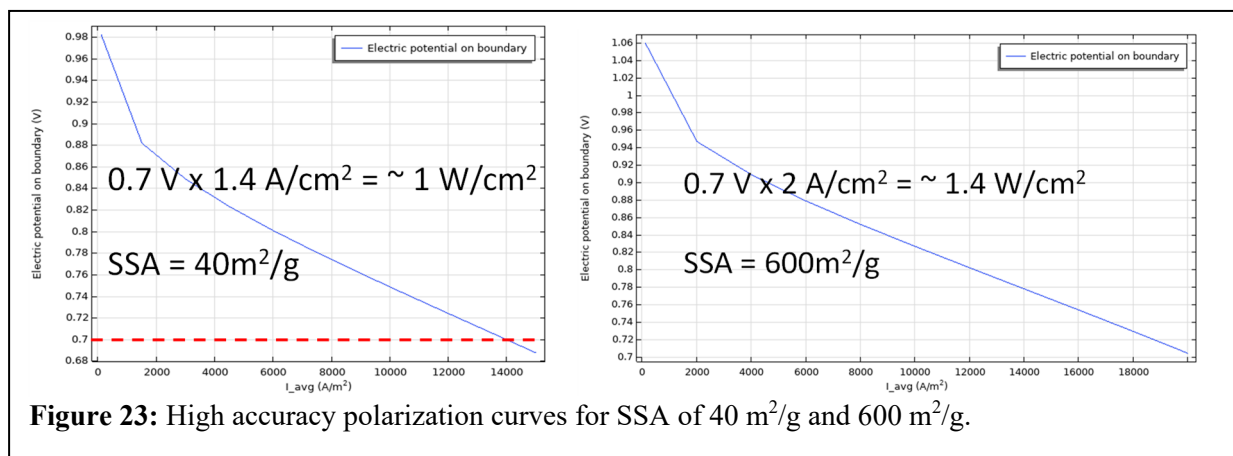


Figure 22: Polarization curves for different geometrical and electrochemical parameters of 3-D serpentine model.

Calculation of the polarization curve with a highly dense mesh provides the result shown on the left graph of **Figure 23** and corresponding to the power density of $\sim 1 \text{ W/cm}^2$ which is the target value in the present project (see **Table 3**). Also, for very high surface area of $\sim 600 \text{ m}^2/\text{g}$ the model gives the power density of $\sim 1.4 \text{ W/cm}^2$ (right graph) matching the values of rated maximum power density desired in **Table 1**. This SSA value is however, still realistic, since in earlier work of the PI and the team, the project team obtained the SSA value for another electrocatalyst material discussed earlier in this report (see **Figure 2**) – $(\text{Ir}_{0.2}\text{Mn}_{0.8})\text{O}_2$ of over $700 \text{ m}^2/\text{g}$. However, that material was synthesized in the nanorods morphology (Shrinath Dattatray Ghadge, Oleg I. Velikokhatnyi, Moni K. Datta, Pavithra M. Shanthi, Susheng Tan, Krishnan Damodaran, and Prashant N. Kumta, Experimental and Theoretical Validation of High Efficiency and Robust Electrocatalytic Response of One-Dimensional (1D) $(\text{Mn},\text{Ir})\text{O}_2:10\text{F}$ Nanorods for the Oxygen



Evolution Reaction in PEM Based Water Electrolysis, ACS Catal. 2019, **9**, 2134-2157) while in **Figure 2**, the material was synthesized in thin film form. It is therefore possible to generate electrocatalysts exhibiting high specific surface areas using scalable approaches matching that of the calculations shown in **Figure 23**.

Thus, there is a large room for improvement of the electrochemical and electrocatalytic properties of the electrocatalyst materials which will result in achieving the targeted performance characteristics of the novel PEMFC and PEM-based water electrolyzer developed in the current project. Also, the computational model has provided critical processing parameters, such as various molar and mass flow rates of oxygen and hydrogen needed to realize the optimal performance of the designed PEMFC and PEMWE.

Future Work:

Continuation of the current project is ongoing with the plan to incorporate the optimized bifunctional electrocatalyst in a single membrane electrode assembly (MEA) that can be used for PEM-based water electrolyzer as well as in a PEM-based fuel cell. Thus, the proposal plan is to create a unitized regenerative PEM-based fuel cell (UR-PEMFC) utilizing the principal investigator's (PI's) proprietary and patented, two **bi-functional** electrocatalysts: PGM-free Cu-Mn-O:F for OER/ORR and low PGM-containing Co-Ir alloy for HER/HOR reactions. The goal is to generate large MEAs of 1 cm^2 , 5 cm^2 , 40 cm^2 as well as 120 cm^2 to be implemented in both water electrolyzer as well as in PEM fuel cell operation. Finally, the goal is to use theoretical

computational modeling studies to determine the optimal electrocatalyst, water electrolyzer and PEMFC design for the experimental validation to ultimately demonstrate the function of these two bi-functional electrocatalysts in a unitized regenerative PEMFC (UR-PEMFC). These studies are planned in the continuation of the grant which is currently on-going.

References:

1. Shrinath Dattatray Ghadge, Prasad Prakash Patel, Moni Kanchan Datta, Oleg I. Velikokhatnyi, Ramalinga Kuruba, Pavithra M. Shanthi and Prashant N. Kumta, Fluorine substituted (Mn,Ir)O₂:F high performance solid solution oxygen evolution reaction electro-catalysts for PEM water electrolysis, *RSC Adv.*, 2017, 7, 17311.
2. Prasad Prakash Patel, Moni Kanchan Datta, Oleg I. Velikokhatnyi, Prashanth Jampani, Daeho Hong, James A. Poston, Ayyakkannu Manivannan and Prashant N. Kumta, Nanostructured robust cobalt metal alloy-based anode electro-catalysts exhibiting remarkably high performance and durability for proton exchange membrane fuel cells, *J. Mater. Chem. A*, 2015, 3, 14015-14032.
3. Prasad Prakash Patel, Moni Kanchan Datta, Oleg I. Velikokhatnyi, Ramalinga Kuruba, Krishnan Damodaran, Prashanth Jampani, Bharat Gattu, Pavithra Murugavel Shanthi, Sameer S. Damle and Prashant N. Kumta, Noble metal-free bifunctional oxygen evolution and oxygen reduction acidic media electrocatalysts, *Nature Scientific Reports*, DOI: 10.1038/srep28367.
4. Karan Kadakia, Moni Kanchan Datta, Prashanth H. Jampani, Sung Kyoo Park, and Prashant N. Kumta, Novel F-doped IrO₂ oxygen evolution electrocatalyst for PEM based water electrolysis, *J. Power Sources*, **222** (2013) 313-317.
5. Shrinath Dattatray Ghadge, Oleg I. Velikokhatnyi, Moni K. Datta, Pavithra M. Shanthi, Susheng Tan, Krishnan Damodaran, and Prashant N. Kumta, Experimental and Theoretical Validation of High Efficiency and Robust Electrocatalytic Response of One-Dimensional (1D) (Mn,Ir)O₂:10F Nanorods for the Oxygen Evolution Reaction in PEM Based Water Electrolysis, *ACS Catal.* 2019, 9, 2134-2157.

Technology Transfer

Nothing to report. The PI and the team, however, anticipate several technological findings and intellectual property to be generated that will be submitted as invention disclosures with subsequent filings of provisional and non-provisional patent filings as needed in consultation with the innovation institute at the University of Pittsburgh and the program manager with the continuation of this project, currently ongoing.

Participants

There are no limits on the number of participants you list for this section; however, you must list participants who have worked one person month or more for the project reporting period. Students are not included in this section.

Have on hand the following information for each participant to enter into the report:

1. Type: Most senior project role

2. Prefix (optional) Dr.
3. First Name Prashant
4. Last Name Kumta
5. Middle Name (optional): Nagesh
6. Suffix
7. Nearest person month worked (a person month equals approximately 160 hours of effort, regardless of funding source) 1
8. National Academy Member? (N)
 9. Country if participant is a foreign collaborator;
10. Country(ies) of international travel and duration of stay.

11. Type: Most senior project role

12. Prefix (optional) Dr.
13. First Name: Moni
14. Last Name: Datta
15. Middle Name (optional): Kanchan
16. Suffix
17. Nearest person month worked (a person month equals approximately 160 hours of effort, regardless of funding source) 1
18. National Academy Member? (N)
 19. Country if participant is a foreign collaborator;
20. Country(ies) of international travel and duration of stay.

21. Type: Most senior project role

22. Prefix (optional) Dr.
23. First Name: Oleg
24. Last Name: Velikokhatnyi
25. Middle Name (optional): I

- 26. Suffix
- 27. Nearest person month worked (a person month equals approximately 160 hours of effort, regardless of funding source) 1
- 28. National Academy Member? (N)
 - 29. Country if participant is a foreign collaborator;
- 30. Country(ies) of international travel and duration of stay.

31. Type: Most senior project role

- 32. Prefix (optional) Dr.
- 33. First Name: Ramalinga
- 34. Last Name: Kuruba
- 35. Middle Name (optional):
- 36. Suffix
- 37. Nearest person month worked (a person month equals approximately 160 hours of effort, regardless of funding source) 1
- 38. National Academy Member? (N)
 - 39. Country if participant is a foreign collaborator;
- 40. Country(ies) of international travel and duration of stay.

Students

Please enter in the number of students for each of the following categories: “Number of undergraduate and graduate STEM participants” and “Number of participants that received a STEM degree”

Matthew Criado, PhD student, Bioengineering Department, University of Pittsburgh

The data entered here should cover the reporting period of one year.

Products

No products have been generated during this reporting period.

Below is the information detailed for each product submission:

1. **Publications (publication reference information (article title, authors, journal, date, volume, issue) can be automatically entered using a DOI):** Nothing to report in this term.
2. **Conference Paper:** Nothing to report in the duration of this project.
3. **Book:** Nothing to report in the duration of this project.

4. **Book Chapter:** Nothing to report in the duration of this project.
5. **Thesis:** Nothing to report in the duration of this project.
6. **Website:** Nothing to report in the duration of this project.
7. **Other Products:** Identify any other significant products that were developed under this project.
Describe the product and how it is being shared.: Nothing to report in the duration of this project.
8. **Patents:** Nothing to report in the duration of this project.

End of Template