Exploring Electrochemical Technology: A Perspective on the ASEE/NSF Small Business Postdoctoral Research Diversity Fellowship

Julie N. Renner and Kathy E. Ayers

Abstract—The American Society for Engineering Education administers a postdoctoral fellowship program supported by the National Science Foundation, encouraging PhD recipients to conduct research in small businesses for 1-2 years. This is a relatively new and unique program where the fellow gains valuable hands-on industry experience while simultaneously small companies enjoy PhD-level work at an affordable cost. To date, the official website is the sole source of public information about this program, with very few first-hand experiences described. This paper summarizes the research and professional activities of a postdoctoral fellow working for Proton OnSite, a leader proton exchange membrane (PEM) water electrolysis systems. The information will help graduate students make educated career decisions.

Index Terms—electrochemical devices, multidisciplinary engineering, small business, women in engineering.

I. INTRODUCTION

Industrial positions constitute a small percentage of the postdoctoral workforce. A 2008 NSF InfoBrief estimated 8% of engineering postdocs worked in for-profit or nonprofit companies or organizations.1 While data on industrial postdocs is scarce, the experience offers a variety of potential benefits including exposure to team-oriented and collaborative environments, access to industry contacts and resources, and the opportunity to gain managerial experience.2 In addition, industry postdoctoral positions come with financial benefits, sometimes including bonuses and stock options. While many of the above can also be attained in academia, postdoctoral positions in industry are often an overlooked option.

The American Society for Engineering Education (ASEE) and the National Science Foundation (NSF) have created a program to support postdoctoral training in small businesses.3 Companies with active Phase II Small Business Innovation Research (SBIR) awards are eligible to participate. Fellows write research proposals for the small business to review before accepting them into the program. Companies benefit financially, paying only a modest amount toward the fellow’s stipend and for a small administrative fee, while fellows have the opportunity to participate in industrial research. Fellows are assigned a mentor at the company, and semi-annual reporting is required throughout the fellowship to document progress made toward project goals.

The program offers many special advantages. Fellows come with their own funding, which can allow them some freedom to explore their own research topics and learn techniques in a new field. Additionally, the required mentorship ensures fellows are getting adequate support, while simultaneously ensuring that the company expectations are satisfied. Finally, because the fellow is in a smaller company, they have the opportunity to be a part of different aspects of the business, including but not limited to manufacturing, quality control, sales, business development, and customer service.

One of the program goals is to recruit postdoctoral candidates from underrepresented groups to work in small businesses.3 Minority groups are underrepresented in postdoctoral positions,4 and women are underrepresented in industry management positions.5 This article outlines the experience of one female postdoctoral fellow working for Proton OnSite, located in Wallingford, CT. Proton OnSite is the world leader in proton exchange membrane (PEM)-based electrolysis systems, and is well-established in the marketplace for industrial applications. Postdoctoral activities including research, mentoring, management, proposal writing and networking are described. The information will provide context for the type of experience that can be gained in an industry postdoctoral position, and will better equip students to make career decisions upon graduating.

II. RESEARCH AND DEVELOPMENT

The research fellow was given the opportunity to be highly involved with catalyst studies at Proton OnSite as well as proof-of-concept experiments to support proposal writing activities. A summary of the major results from these activities is presented below.

A. Catalyst Fundamentals

The fellow worked with the production team to audit the catalyst processing techniques at Proton OnSite and identify...
important parameters. It was hypothesized that catalyst surface charge had a significant impact on the characteristics of the resulting material. A simple and inexpensive titration method was employed to screen surface charge. Exploring effects of the extreme cases, it was found that catalysts which caused a large basic shift in pH also resulted in different morphology and processing behavior compared to catalyst with an acidic shift (Fig. 1).

Based on these results, a technique was explored to control the charge on the catalyst particles. A catalyst lot with high base contamination was subjected to an acid treatment and rinsed. Titrations of the treated lot indicated that the charge could be changed and the processing behavior could be controlled. In a separate experiment, Brunauer-Emmett-Teller (BET) analysis indicated that after the acid treatment the catalyst had a negligible decrease in surface area (less than 5%).

Scanning electron microscope (SEM) images were taken of the base contaminated catalyst, and of the same catalyst treated with acid (Fig. 2). Both samples were dried and sifted with the same protocol. Fine particles are reduced after the acid treatment.

The performance of a membrane electrode assembly (MEA) manufactured with acid treated catalyst was evaluated in a 25 cm² test cell at 50°C and compared to Proton’s baseline data (Fig. 3). The results indicate that the performance is not affected by the acid treatment of the catalyst.

The impact of the research includes an inexpensive screening technique which prevents catalyst lots with suboptimal surface charge from entering the processing pipeline. This research also demonstrated control the surface charge for future processing needs.

B. Electrodes for Low-Cost, Alkaline Exchange Membrane (AEM)-based Water Electrolysis

Over the past decade it has been realized that anion exchange membranes (AEMs) can be used as a solid state electrolyte, enabling AEM fuel cells and other devices. Compared to PEMs, the technology is less developed, but AEMs are advantageous because they 1) enable low-cost materials of construction, 2) they allow the utilization of a wider array of low-cost catalysts, and 3) in Proton’s experience, AEM materials are stiffer and easier to handle than PEM membranes of similar thickness. This makes AEMs more robust to normal processing than PEM materials, and therefore thinner membranes can be used. Because of these distinct cost advantages, efficiency penalties due to AEMs having intrinsically lower ion conductivity are expected to be mitigated. It is for these reasons AEM development is included in Proton’s technology roadmap.

Currently, there are no viable alternatives to noble metal-based catalysts in PEM-based electrolysis. Proton works with a variety of academic partners (for example, Illinois Institute of Technology and Northeastern University) who investigate AEM technology and appropriate catalysts. However, catalyst activity and stability, electrode structure and manufacturing are all still active areas of research. Since AEMs have a lower heat tolerance than PEM materials, traditional heating and pressing to make MEAs will not work. In this work, the fellow had the opportunity to independently conceptualize and explore a solution-based metal deposition technique for AEM-based electrolysis, which does not require excessive heating, or pressing. To Proton’s knowledge, this is the first time this technique has been explored for this specific application.

Cobalt-based catalyst was directly plated on anode gas diffusion layers (GDLs) to make gas diffusion electrodes (GDEs). These GDE samples were built in a 5 cm² test cell with a standard noble metal electrolysis cathode. Testing was conducted at 50°C in an anode feed configuration. The non-noble metal electrode showed similar initial performance to the standard noble metal electrode, demonstrating proof-of-concept for the deposition technique (Fig. 4).
purified inlet streams, potentially allowing air to be the N₂ source. In addition, because electricity is used to drive the reactions, integration with renewable energy sources (e.g., wind or solar) becomes more plausible.

There are several papers involving electrochemical production of ammonia.⁹ PEMs are well-established and have been recently incorporated into a number of ammonia synthesis devices.¹⁰ However, the reported performance is low and can be partly attributed to the need for selective catalysts. One problem with using PEM-based devices is the acidic environment limits durable catalyst options to expensive noble metals. In addition, ammonia is a weak base, and it is expected that it readily reacts with acidic membranes to reduce proton conductivity and speculatively, membrane lifetime. Recently, AEMs have been successfully demonstrated in ammonia fuel cells,¹¹ but there is no significant published work on AEM utilization for ammonia synthesis to date. The fellowship allowed access to multiple experts in cell design and exploratory work to take place proving an AEM-based ammonia production cell is feasible.

An AEM-based ammonia production cell was designed and built for proof-of-concept experiments. Fig. 6 shows the schematic of the electrochemical cell. The feed gas stream is humidified nitrogen gas. The N₂ and water (H₂O) present in the feed stream combine with electrons at the cathode to form hydroxide ions (OH⁻) and ammonia. The key enabler in the device is the AEM which selectively conducts OH⁻ to the anode where the ions form oxygen (O₃) and H₂O. The end result is an ammonia enriched stream depleted of small amount of N₂ and H₂O. Electrolysis catalysts were used, which were neither selective to ammonia nor optimized for stability but served as a proof-of-concept demonstration.

Polarization data showed current was achieved at potentials lower than the water electrolysis theoretical voltage in the initial performance test (Fig. 7). This is evidence that ammonia production is occurring. Degradation of performance was apparent with the earliest curve having the best performance, and an increase in activation energy occurring as the cell is operated. Samples taken during the initial testing period and after several hours of operation were analyzed for ammonia content using a colorimetric assay.¹² The assay shows that the AEM-based technology is capable of producing ammonia (Fig. 8).
The fellow also supported government reporting activities for multiple SBIR projects. This was a synergistic role, providing the company with project support in a concrete way, but also exposing the fellow to multiple technologies in a short amount of time.

B. Small Business Upper Management Exposure

In the first year of the fellowship, the research mentor organized one-on-one interviews with the research fellow and the upper management at Proton OnSite. During the interviews, the research fellow learned what the person’s role was at the company. The fellow was encouraged to ask detailed questions about the importance of the role, including how the role was executed, what experiences helped them in their career, and if they had any advice for the fellow. This effort has fostered a greater understanding of how a small business operates and how each part of the company fits into the overall vision.

C. Project Management and Team Building

The research mentor encouraged the fellow to assume a Project Manager role in a Phase II STTR with Brookhaven National Labs which investigates alternative electrode manufacturing and core-shell catalysts to reduce the noble metal content by an order of magnitude in proton exchange membrane (PEM)-based water electrolysis. The research fellow’s responsibilities included collaborating with the investigators at Brookhaven, managing and prioritizing project tasks, organizing team meetings and providing direction to engineers to achieve the milestones of the project. The research fellow was also encouraged to travel to Brookhaven to meet the collaborators, and attend a symposium to further establish a network.

In addition, the research fellow was asked to be part of multiple teams in a technical support role by conducting laboratory experiments, supporting government reporting activities, acting as an academic liaison, and providing technical guidance to engineers. This experience provided a broad technical exposure, while simultaneously building a teamwork mentality within the company culture.

D. Mentoring

The research fellow also had the opportunity to mentor multiple undergraduate interns with industry-based research projects. The fellow was responsible for defining the project plans for these students, as well as managing their activities and tracking their progress. The fellow spearheaded a co-op program with the University of Connecticut Department of Chemical and Biomolecular Engineering, where students participate in defined industry-based projects for credit. The program is being piloted this spring, with two students participating. One student is working on identifying and developing new catalyst characterization techniques to understand catalyst processing behaviors. The end result will be official company work instructions for the techniques developed. Another student is designing, building and testing an electrodeionization unit using Proton hardware. The end result of this project will be proof-of-concept data to be included in future research proposals or publications.

Fig. 7. Polarization data of an AEM-based ammonia production cell.

These results have supported multiple proposals, showing, proof-of-concept for a new and promising electrochemical cell design for ammonia production, as well as a direction for future development activities.

III. SMALL BUSINESS EXPERIENCES

A. Proposal and Report Writing

The above results were included in multiple SBIR/STTR proposals where the technical write up was spearheaded by the fellow. The mentor provided feedback and scaffolding for the proposal writing effort. Through this effort, the fellow also had the opportunity to interact with academic partners to organize the scope and technical objectives of the proposals. This allowed the fellow to have guidance from professionals who have had experience and success in proposal writing.
E. Networking

Financial assistance is given to the fellow through the fellowship program to attend conferences, allowing the fellow to travel at a significantly reduced cost to the company. The fellow was able to meet academic partners who later became collaborators on proposals. Additionally, the fellow interfaced with local universities to understand current research and potential future collaborations, as well as equipment and testing capabilities.

IV. CONCLUSIONS

While industry postdocs are relatively rare, this report demonstrates the experience can include many professional advantages, including the opportunity to conduct cutting edge research, write proposals, manage projects, direct research, obtain valuable contacts, and understand business structure. Postdocs can have a tangible positive impact on companies by supporting manufacturing, and can also have long term impact by developing emerging technologies.

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REFERENCES