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14. ABSTRACT The grid-scale energy storage symposium created a forum for scientists and engineers interested in mechanical, chemical, and electrochemical methods for storing energy at large-scales. Attendees included academic, governmental, and industrial researchers, allowing the symposium to showcase a breadth of work that went from the most exploratory fundamental research to the pragmatic industrial product-focused development. The symposium featured work from institutions such as Toyota, Ceramtec, City University of New York, Sandia National Laboratories, Trinity College, and Pacific Northwest National Laboratory. Talks went across two days.

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Report Title

Final Report: Grid-Scale Energy Storage (Symposium EE8)

ABSTRACT

The grid-scale energy storage symposium created a forum for scientists and engineers interested in mechanical, chemical, and electrochemical methods for storing energy at large-scales. Attendees included academic, governmental, and industrial researchers, allowing the symposium to showcase a breadth of work that went from the most exploratory fundamental research to the pragmatic industrial product-focused development. The symposium featured work from institutions such as Toyota, Ceramtec, City University of New York, Sandia National Laboratories, Trinity College, and Pacific Northwest National Laboratory. Talks went across two days, and MRS will be publishing the proceedings of the meeting in MRS Advances, which is a new peer-reviewed journal from MRS.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
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TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
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TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 20.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received Book

TOTAL:

Received

Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

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FTE Equivalent:	
Total Number:	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:..... 0.00

Names of Personnel receiving masters degrees

NAME
Total Number:

Names of personnel receiving PHDs

NAME
Total Number:

Names of other research staff

NAME PERCENT SUPPORTED
FTE Equivalent:
Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Symposium EE8 summary and program attached.

Technology Transfer

Report to Army Research Office (ARO)

MRS 2016 Spring Meeting, Phoenix, AZ, March 28-31, 2016

Symposium EE8: Grid-Scale Energy Storage

Jun Wang (A123 Systems LLC, Waltham, MA)

Mitchell Anstey (Sandia National Laboratories, Livermore, CA)

Sagar Mitra (Indian Institute of Technology, Bombay, India)

Babu Chalamala (Sandia National Laboratories, Albuquerque, NM)

Summary

The grid-scale energy storage symposium created a forum for scientists and engineers interested in mechanical, chemical, and electrochemical methods for storing energy at large-scales. Attendees included academic, governmental, and industrial researchers, allowing the symposium to showcase a breadth of work that went from the most exploratory fundamental research to the pragmatic industrial product-focused development. The symposium featured work from institutions such as Toyota, Ceramtec, City University of New York, Sandia National Laboratories, Trinity College, and Pacific Northwest National Laboratory. Talks went across two days, and MRS will be publishing the proceedings of the meeting in *MRS Advances*, which is a new peer-reviewed journal from MRS.

Objectives

Energy storage at the scale useful for applications in the electric grid is a difficult but important challenge that we need to address. It is also a contemporary problem, brought on by the shift to alternative energy sources that are dependent on season, weather, and day-night cycles, which has been spurred on by the availability of low-cost photovoltaics and wind energy technologies. Balancing the grid at the utility and consumer level is needed, and therefore, a large toolkit of storage options is required to address specific grid architectures. Additionally, grid-scale energy storage is essential for remote areas with limited grid connectivity such as many islands, scientific and military outposts. A wide array of systems level and materials challenges must be addressed in grid scale energy storage. Chemistry and materials science are continually developing next-generation technologies that improve upon previous iterations. Integration and power management systems tailored to each new storage technology must then be built and implemented.

This symposium highlighted advances in grid storage technology at all scales and stages of development to generate a top-level view of the field. Specifically discussed in this symposium were new advances that address safety issues through materials, low-cost redox-active materials for electrolytes, new electrode materials and architectures, large-scale variants of nonstandard storage systems, systems integration, advanced models for lifecycle analysis, and cost-benefit analyses for storage systems.

Research Highlights

The presentations were divided into several areas:

- (1) Energy Storage Safety and Reliability
- (2) Technologies Beyond Lithium-Ion
- (3) Frontiers of Lithium-Ion Chemistry
- (4) Redox Flow Batteries

Christopher J. Orendorff from Sandia National Laboratories kicked off the symposium on Tuesday afternoon by delivering an invited talk entitled, “A material science approach to improve energy storage safety”, which is a key area of focus for symposium EE8. There exists growing concern over the safety and reliability of large scale systems (>10 kWh) based on lithium-ion energy storage technologies. It is evident that control strategies at the cell-level (current interrupt, positive thermal coefficient devices, fusing, etc.) may not be effective at the battery pack or system-level and may, in fact, be more detrimental to battery safety. Additionally, control solutions at the system-level (battery management systems, active cooling, packaging, design) are often costly, complex, and impact both volumetric and gravimetric energy density.

To strike a balance in improving battery safety while mitigating potential negative impact to system performances, Orendorff outlined a complementary approach to engineered solutions which focused on safety improvements and enhancing abuse tolerance at cell component level, such as electrolytes and separators. The Sandia team has developed advanced electrolyte salts based on anion binding agents (ABAs) that minimize the consequences of thermal runaway while maintaining electrochemical performance comparable to LiPF₆-based electrolytes. They have demonstrated the utility of a co-solvent approach with hydrofluoro ether (HFE) solvents for non-flammable electrolytes during a cell vent failure. Common coating strategies based on Al₂O₃ atomic layer deposition (ALD) and LiMPO₄ (M = transition metal) solution phase coating were also discussed with some performance trade-offs.

There were three presentations by David Rosewater of Sandia National Laboratories, Fuminori Mizuno from Toyota Research Institute of North America, and Summer R. Ferreira also from Sandia National Laboratories who discussed various areas of greatest need based on impact to the safety of deployed systems. They also identified necessary resources to address these challenges in different ways, some at cell level and others at systems level.

The second theme of this EE 8.2 was “Technologies Beyond Li-ion” which explored new electrode materials and new battery technology other than Li-ion. High quality invited and technical papers were presented, with topics covering poly sulphate based Na-ion electrode materials, Sodium-Iodine battery, Na-NiCl₂ battery and Zn-MnO₂ based electrochemical storage systems. In the beginning, Prof. Prabeer Barpanda from Indian Institute of Science, Bangalore, introduced for the first time his work on sodium-iron sulphate chemistry for grid storage

applications. He has shown an interesting perspective of new sulphate anion based electrochemistry against sodium ion, and this new chemistry could be a game changer in the future. Sai Bhavaraju from Ceramtec Inc, South Salt Lake, Utah, United States, described the development of sodium-iodine battery using NaSICON membranes that can operate at a significantly lower temperature. This battery technology needs further investigation and optimization and can be a groundbreaking solution for large-scale applications. The session ended with four technical presentations by various students from different universities in the United States. The session was a great experience to see many professionals of different disciplines together in one place, sharing their knowledge and ideas on energy storage science and technologies.

In area of focus (3) on Frontiers of Lithium-Ion Chemistry, Prof. Susan Odom from the University of Kentucky presented her work on organic materials, phenothiazines, which have the potential to act as overcharge protection additives and also as electrolytes for redox flow batteries. SEI formation is a well-known process in standard lithium-ion battery operation; however, using aqueous electrolytes does not normally allow for this process to occur leading to degradation and a reduction in performance metrics. Prof. Chunsheng Wang from the University of Maryland, in collaboration with the Army Research Laboratory in Adelphi, presented new work on “water-in-salt” electrolyte compositions that allow for new mechanisms of SEI formation using TFSI-based salts. This is certainly a ground-breaking result that will give cheaper options for battery assembly. Representing A123 Systems, Dr. Jun Wang presented a wide array of A123’s business and refocused efforts on transportation storage systems, specifically highlighting new work in silicon anodes for lithium-ion batteries. Prof. Amitava Choudhury from the Missouri University of Science and Technology showed his group’s work on new materials based on iron cathode materials with mixed-anion compositions. This session finished with one of the most prominent researchers in grid-scale energy storage, Prof. Sanjoy Banerjee from CUNY. His experience in both research and implementation of new battery technologies came in the form of a talk that touched upon cost comparisons of alkaline batteries at large installations and where new technologies or existing technologies have to improve to compete with the \$0.02 kWh figure as achieved through pumped hydro.

In area of focus (4), Vincent Sprenkle represented Pacific Northwest National Laboratory and its large library of vanadium-based redox flow battery chemistry. He showed the sizable testing facility that features 20-cell stacks running vanadium cells of varying purity to demonstrate viability of their new mixed acid electrolyte system. Travis Anderson of Sandia National Laboratories spoke to his ionic liquid flow system and the synthesis of new membranes for non-aqueous systems. The lack of appropriate membranes is most likely one of the biggest drawbacks to any research in non-aqueous flow batteries, and Anderson showed that a custom-made material developed by Cy Fujimoto, also from Sandia, increases conductance without allowing the large crossover of ions that normally leads to fast self-discharge. A fellow Sandia researcher, Leo Small, then gave a very interesting talk on mixed electrolyte ionic liquid systems

that pair lithium as an anode material, which is a very unique idea in flow battery systems. Some very interesting engineering approaches to battery design were shown by Prof. Daniel Steingart from Princeton University. He advocated for less “over optimization” and moving to a zinc-bromine system where degradation mechanisms are prevalent but fully reversible. This is a design-first approach that shows what a multidisciplinary approach can achieve when science and engineering combine.

In regards to the ARO grant, the organizers used this funding to pay for registration fees for both the organizers and invited speakers. These speakers were selected based on several criteria such as the impact of their advancements, their reputation as a scientist, and innovation of their work. Additionally, three graduate students from universities both domestic and international were given travel stipends to allow them to gain valuable experience presenting their work to a scientific audience.

Contributions to the Discipline

Symposium EE8 was a relatively small program compared to other symposia with much broader scopes; yet, it undoubtedly brought together an interdisciplinary group of researchers and engineers working on both fundamental materials research and device-related materials engineering topics, in an effort to help them to appreciate the breadth of the field, address current challenges, promote collaboration, generate ideas and identify future applications. The symposium offered researchers a forum out of their routines to critically examine and cross-fertilize each other’s work which might not be so straightforward otherwise.

The number of abstracts submitted to symposium EE8 was about 25, out of which 20 papers survived the critical review process mandated by MRS. As summarized in the area of focus above, it clearly validated the cross-fertilization idea which was further embraced by the scope of those 20 papers making it to the final stage. It is the organizers’ firm belief that the ideas and collaborations fostered by the symposium EE8 will drive the successful adoption of these materials for new applications in the energy storage space.

Future Directions

Energy sustainability remains a critical issue our society has to face. MRS has long recognized that and embraced the challenge in every meeting, and other technical societies such as ECS (Electrochemical Society), ACerS (American Ceramic Society) and ACS (American Chemical Society) have also shown consistent effort in promoting those ideas. It is safe to predict that this trend is expected to continue at a higher level of momentum in the foreseeable future. To that end, future symposia will carry on the topic of new battery materials research, system design and evaluation at multiple scales. Given the fast changing and growing competition of the energy storage field and enormous challenges associated with smart grid and energy efficiency, new materials and technologies are continually spurring new advances in the field. We hope this symposium could stimulate a major breakthrough in any one of the areas

touched upon by speakers participated in symposium EE8, which could potentially change the energy storage landscape in an unprecedented way. The net benefit will further facilitate the mass adoption of advanced energy storage technologies as viable solutions to grid scale energy economy. Without pointing to any specific examples, many of those advances may very well be aligned with the interest of the US Army for a number of applications.

Acknowledgements

We gratefully appreciate support from the Army Research Office (ARO), which was critical to the success of this symposium. We also want to thank MRS staff for supporting the application of this ARO grant.



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Symposium EE8—Grid-Scale Energy Storage



2016 MRS Spring Meeting & Exhibit

March 28-April 1, 2016

Phoenix, Arizona

2016-03-29

Symposium EE8

Hide All Abstracts

Symposium Organizers

- Mitchell Anstey, Sandia National Laboratories
- Babu Chalamala, Sandia National Laboratories
- Sagar Mitra, Indian Institute of Technology Bombay
- Jun Wang, A123 Systems LLC

Support

- Army Research Office

EE8.1: Energy Storage Safety and Reliability

- Chair: Mitchell Anstey
- Chair: Jun Wang
- Tuesday PM, March 29, 2016
- PCC North, 100 Level, Room 121 C

1:30 PM - *EE8.1.01

A Material Science Approach to Improving Energy Storage Safety

Christopher J. Orendorff¹, Ganesan Nagasubramanian¹, Kyle R Fenton¹, Eric Allcorn¹.

¹, Sandia National Labs, Albuquerque, New Mexico, United States.

Hide Abstract

As large scale (> 10 kWh) lithium-ion energy storage systems are being fielded for electric utility storage, transportation, and defense technologies, concerns over the safety and reliability of these systems is increasing. For battery storage, there is evidence that control strategies at the cell-level (current interrupt, positive thermal coefficient devices, fusing, etc.) may not be effective at the battery pack or system-level and may, in fact, be more detrimental to battery safety. Moreover, control solutions at the system-level (battery management systems, active cooling, packaging, design) are often costly, complex, and impact energy density (Wh/kg and Wh/L). For the high volume, commercial space, it is important that adoptable strategy to improve safety not have any negative impact battery performance parameters.

A complementary approach to engineered solutions is materials development focused on safety improvements and enhancing abuse tolerance of lithium-ion chemistries. Targeted solutions can be developed to address the most pervasive safety challenges. We have developed advanced electrolyte salts based on anion binding agents (ABAs) that minimize the consequences of thermal runaway while maintaining electrochemical performance comparable to LiPF₆-based electrolytes. We have demonstrated to utility of a co-solvent approach with hydrofluoro ether (HFE) solvents for non-flammable electrolytes during a cell vent failure. We have demonstrated that advance coating strategies based on Al₂O₃ atomic layer deposition (ALD) and LiMPO₄ (M = transition metal) solution phase coating can be employed to not only improve cycle life, but also provide thermal stability to active materials. This presentation will highlight these efforts to develop enhanced materials solutions to the safety challenges and address any performance trade-offs.

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

2:00 PM - EE8.1.02

Advanced Methodology for the Development of Safe and Reliable Energy Storage Systems

David Martin Rosewater¹.

¹, Sandia National Laboratories, Albuquerque, New Mexico, United States.

Hide Abstract

Safety and reliability of grid scale energy storage systems represent a critical challenge for large scale adaption of battery technologies in the electricity industry. Materials play a major role in determining the safety and integrity of system. While we have sufficient qualitative understanding of the materials failure modes, we need to move from phenomenological models to predictive models. Over the last few years, we have done extensive testing of a number of battery technologies in configurations ranging from 5KW to a large 1MW system to understanding the performance characteristics of these systems in grid tied environments. This research has determined that advanced hazard analysis techniques are able to provide the granularity of design constraints needed to ensure system safety. Therefor safety and reliability can be assured through a combination of systematic testing and automated control that enforces the system's design constraints. This presents an advanced methodology for the design and development of energy storage systems.

2:15 PM - EE8.1.03

Ionic Liquids for Lithium-Gas Energy Storage

Fuminori Mizuno¹, Kensuke Takechi¹, Charles A Roberts¹, Nikhilendra Singh¹, Paul T Fanson¹, Tangqiumei Song^{1 2}, Samuel Seo², Joan F Brennecke².

¹, Toyota Research Inst of NA, Ann Arbor, Michigan, United States; ², University of Notre Dame, Notre Dame, Indiana, United States.

Hide Abstract

Ionic liquids present a promising new technology for cost-efficient separation of CO₂ from post-combustion flue gas¹. Their numerous combinations of cations and anions enable tuning of chemical and physical properties for CO₂ capture. Additionally, we have suggested a new approach to energy storage using ionic liquids with CO₂. Mixing of concentrated CO₂ can create substantial electrical energy in a metal-oxygen battery system². Coupled with fundamental knowledge of gas solubility in ionic liquids, we propose a new energy flow diagram³.

An extension of ionic liquid based lithium-CO₂ and lithium-O₂ batteries is the direct use of exhaust gas from internal combustion engines. Here we present preliminary results for Li-air batteries using model lean burn automobile exhaust gas (NO, CO, C₃H₆, and excess O₂). We recently found that the operating voltage/capacity under the lean burn condition was higher than the ones measured under a reference condition having the same O₂ concentration⁴. This finding indicates that the residual gas components (NO, CO, C₃H₆) play a significant role in enhancing the operating voltage/capacity. In addition to being applied in primary batteries, this concept is particularly noteworthy because it presents a practical solution to use metal-air batteries under ambient air conditions. In this presentation, we will investigate the physical properties of ionic liquids exposed to various gas conditions, and then explain their Li-air battery performances.

References

- [1] J. F. Brennecke et al., *J. Phys. Chem. Lett.*, 1 (2010) 3459.
- [2] K. Takechi et al., *Chem. Commun.*, 47 (2011) 3463.
- [3] S. Seo et al., submitted for publication (2015).
- [4] F. Mizuno et al., submitted for publication (2015).

2:30 PM - EE8.1.04

Safety Research and Development Challenges in Stationary Storage Systems

Summer R. Ferreira¹.

¹, Sandia National Labs, Albuquerque, New Mexico, United States.

Hide Abstract

With increased deployment of fielded demonstrations of grid storage systems, there is now more attention being paid to safety concerns and challenges. By collaborating with industry stakeholders, as well as other labs and organization conducting R&D in battery technology we have prioritized the research and development challenges around storage system safety. This talk reviews what were identified as the areas of greatest need based on impact to the safety of deployed systems. The necessary resources to address these challenges have been assessed and considered in developing a safety program. Previous work that is informing where gap areas exist currently, and best paths toward both identifying risks and mitigating them is reviewed. This includes developments around storage safety within the electric vehicle community.

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

2:45 PM -

BREAK

Hide Abstract

EE8.2: Technologies beyond Li-Ion

- Chair: Babu Chalamala
- Chair: Sagar Mitra
- Tuesday PM, March 29, 2016
- PCC North, 100 Level, Room 121 C

3:00 PM - *EE8.2.01

New Sodium Iron Sulfate Chemistry for Grid-Scale Power Storage: Materials Perspective

Prabeer Barpanda¹, Debasmita Dwivedi¹.

¹, Indian Institute of Science, Bangalore, India.

Hide Abstract

In the quest to design improved rechargeable batteries, new cathode materials are incessantly pursued. In this case, polyanionic materials offer a rich treasurehouse to unveil novel compounds having robust three-dimensional

frameworks offering thermal/ chemical stability. Guided by the inductive effect principle, the redox potential of polyanionic cathodes can be tuned by playing with the electronegativity of constituent anion units. In this case, highly electronegative SO_4 -based compounds are shown to deliver the highest redox potential (e.g. LiFeSO_4F operating at 3.9 V vs. Li/Li^+) [Nature Materials, 10, 772, 2011]. Recently, Barpanda et al have discovered a novel $\text{Na}_2\text{Fe}_2(\text{SO}_4)_3$ alluaudite structured cathode delivering over 100 mAh/g along with high rate kinetics and cycling reversibility [Nature Communications, 5, 4358, 2014]. It marks the highest $\text{Fe}^{3+}/\text{Fe}^{2+}$ redox potential (ca. 3.8 V vs. Na/Na^+). However, its preparation is cumbersome demanding (i) extended milling and annealing of precursor mixture (350 °C for 24 h) and (ii) usage of pure anhydrous FeSO_4 , which is not readily available. Also spontaneous dissolution of SO_4 -species rules out any water-based synthesis. We have developed two new methods (1) Pechini route and (2) spray drying methods; both using distilled water as reacting media directly taking hydrated FeSO_4 precursors. Also, non-aqueous reacting media such as ionic liquids and polymers can be used to prepare this new compound. We will describe these new synthetic routes to obtain alluaudite high-voltage cathode material for sodium-ion batteries. These alternate routes enable us (i) synthesis at low temperature of 300 °C, (ii) formation of in-situ carbon coating and (iii) production of homogeneous nanoscale particles. These synthesis routes have been successfully extended to other 3d homologues in this family with the observation of polymorphism in some cases. We will describe salient features of these low-temperature syntheses, crystal structure and final electrochemical performance of high-voltage alluaudite insertion materials for batteries. The scalability of these synthesis routes and possible application of sodium iron sulfate alluaudites in remote area grid storage will be discussed.

3:30 PM - EE8.2.02

Development of Sodium-Iodine Battery Unit Cell with Tubular NaSICON Membrane for Large-Scale Energy Storage

Sai Vishnubhotla Bhavaraju

1, , Ceramtec Inc, South Salt Lake, Utah, United States.

Hide Abstract

The nation can utilize effective large scale electrical energy storage and delivery devices for powering the grid. Sodium batteries are attractive for these applications due to their high performance available at low costs. Toward this end, Ceramtec, Inc., is developing an intermediate temperature (~ 120 - 180 °C) secondary sodium- iodine battery that meets this criteria. In this battery, we combined proprietary NaSICON ceramic membrane that has been shown to have high sodium-ion conductivity with a sodium metal anode (one of the least expensive and energy dense metals) and liquid state iodine cathode. The present Na- I_2 batteries have high theoretical capacities (581 Wh/kg) and cell voltages (2.8 V), inherently safe, and are expected to have long cycle life (>5000 cycles) while meeting the module level cost target of < \$200/kWh.

At this time, long-term cycling results are being obtained using 28 Wh sodium-iodine cell with molten salt catholyte formulation, carbon cathode current collector, molten sodium anode, and a tube type NaSICON separator. These data were collected up to C/4 charge-discharge rate and a NaSICON current density corresponding to 50 mA per sq.cm. of membrane area. These cells were cycled 200 times at 60% Depth of Discharge (DOD) with energy efficiency of 85%. In summary, we have demonstrated proof-of-concept for a long term and efficient performance of Na & I_2 full cells at practical DOD, C-rates and energy efficiencies. We recently started to construct and test scaled-up 100 Wh unit cells with tubular NaSICON membranes with the aim of demonstrating commercial size unit cells. We will be presenting up to date performance results from our 28 Wh & 100 Wh prototype unit cell testing.

3:45 PM - EE8.2.03

Advanced Intermediate Temperature Na-NiCl₂ Battery Technologies

Guosheng Li, Xiaochuan Lu¹, Vincent L Sprenkle¹.

1, , Pacific Northwest National Lab, Richland, Washington, United States.

Hide Abstract

Recently, molten-sodium (Na) beta-alumina batteries (NBBs) have been considered one of the most attractive stationary electric energy storage systems, which are crucial to stimulate the growth of renewable energy resources and to improve the reliability of electric power grids. Here we demonstrate for the first time that planar Na-NiCl₂

batteries can be operated at an intermediate temperature of 190°C with ultra-high energy density. A specific energy density of 350 Wh/kg was obtained for planar Na-NiCl₂ batteries operated at 190°C over a long-term cell test (1000 cycles). The high energy density and superior cycle stability are attributed to the slower particle growth of the cathode materials (NaCl and Ni) at 190°C. The results reported in this work demonstrate that planar Na-NiCl₂ batteries operated at an intermediate temperature could greatly benefit this traditional energy storage technology by improving battery energy density, cycle life and reducing material costs.

4:00 PM - EE8.2.04

Surface Reactions in Aqueous Sodium-Ion Batteries for (Micro)Grid Applications

Mona Shirpour¹, Xiaowen Zhan¹.

¹, , Univ of Kentucky, Lexington, Kentucky, United States.

Hide Abstract

Aqueous sodium-ion batteries may solve the cost and safety issues associated with the energy storage systems for the fluctuating supply of electricity based on solar and wind power. Aqueous, or water-based, sodium-ion batteries are promising candidates for grid storage applications because they offer multiple cost savings using less expensive electrode materials, much cheaper electrolyte solutions compared to the lithium-ion cells, and less costly manufacturing conditions. They also have the extra advantage of being safer for grid applications[1], and higher cycling efficiency due to the higher ionic conductivity of the aqueous electrolyte. In the water-based cells, both the anode and cathode should undergo redox processes approximately within the voltage stability region of water. Most of the anode materials[2] for sodium-ion batteries, including carbon-based compounds and titanates[3] have a very low operating voltage vs. sodium, and fall in the region of hydrogen evolution. NaTi₂(PO₃)₄ is perfectly located at the lower border of the water voltage stability region and operates at 2.1 V versus Na/Na⁺. We have developed an aqueous sodium-ion battery based on a NASICON-type anode material, and a fluorophosphate cathode material. The specific energy of the cell is 50Wh/Kg, which is larger than the specific energy of similar cell with organic electrolyte, but the cell capacity fades quickly especially when cycled slowly. For this presentation, we will discuss our recent results on the search for electrically and electrochemically stable electrode materials for aqueous sodium-ion batteries. The mechanism responsible for the severe capacity fading upon cycling will also be discussed.

References:

1. Li, Z., Young, D., Xiang, K., Carter, W. C., Chiang, Y.-M. *Advanced Energy Materials* 2013, 3, 290.
2. (i) Palomares, V., et al., *Energy & Environmental Science* 2012, 5, 5884; (ii) Kim, H., et al., *Chemical Reviews* 2014, 114, 11788; (iii) Slater, M. D., et al., *Advanced Functional Materials* 2013, 23, 947.
3. (i) Senguttuvan, P., et al., *Chemistry of Materials* 2011, 23(18), 4109; (ii) Shirpour, M., Cabana, J., Doeff, M. *Energy & Environmental Science* 2013, 6, 2538; (ii) Shirpour, M., Cabana, J., Doeff, M. *Chemistry of Materials* 2014, 26, 2502.

4:15 PM - EE8.2.05

Study of Electrodeposited Zinc Morphology in Rechargeable Alkaline Batteries

Xia Wei¹, Alexander Couzis¹, Sanjoy Banerjee¹.

¹, , The City College of New York, New York, New York, United States.

Hide Abstract

Rechargeable zinc-nickel flow-assisted batteries are attractive in applications like, frequency regulations, UPS inverters and hybrid electrical vehicles, etc. due to their advantages, such as, cheap, capable of rapid charge and discharge and high energy density etc. However, the challenges that rechargeable zinc-anode batteries are facing are short lifespan time due to non-uniform zinc deposition during charging, hydrogen evolution that comprises energy efficiency and zinc corrosion during battery resting, which leads to further capacity loss. We developed an indicator that can monitor the transition of zinc morphology during battery charging. Zincate concentration in the electrolyte, flow velocity and current density were varied over a wide range. The results from SEM and 3D X-ray computer tomography, etc. show that the ratio between the effective current density and the limiting current density (current density ratio), which is directly related to the zincate concentration at the electrode surface, determines the zinc morphology. Furthermore, we investigated the causes of transition of zinc morphology. Compact zinc deposits are found to have a fine-grained, bright finish and the highest anodic efficiency. Electrochemical impedance spectroscopy

(EIS) proves that compact zinc corresponds to the minimum in the half-cell resistance.

Anode substrate is a parameter that also influences the zinc morphology. We studied the effects of various novel metal substrates on zinc morphology over battery cycling. Results from electrochemical techniques, chemical characterization and cell cycling will be presented. In order to alleviate the self-discharge rate and increase the lifespan of zinc-anode batteries, we investigated the impacts of additives on zinc morphology and the cell performance.

4:30 PM - EE8.2.06

Advanced Alkaline MnO₂-Zn Batteries: Accessing the Second Electron Capacity

Gautam G. Yadav¹, Joshua W. Gallaway¹, Michael Nyce¹, Sanjoy Banerjee¹.

¹, Chemical Engineering, City College of New York, New York, New York, United States.

Hide Abstract

Manganese dioxide (MnO₂) and zinc (Zn) are one of the most abundant, safest and cheapest materials available. Together, they are found in common household batteries like Duracell, Energizer, etc. as small cylindrical alkaline cells. These cells or batteries are used as primary batteries, i.e., as single use batteries, where the entire capacity of the battery is delivered once and then discarded. The disadvantage of primary batteries is that it takes a lot of energy to produce the battery than the energy that can be actually obtained from it, and also, it creates environmental waste. However, the manufacturing of primary cells has still been rampant due to the cost of manufacturing MnO₂-Zn cells being very cheap. In terms of improving the overall energy efficiency, reducing waste and maintaining its cost advantage, it makes good sense, economically and environmentally, to make MnO₂-Zn cells rechargeable. However, the main deterrent to this direction has been the fundamental material and chemical problems of the main raw components, i.e., MnO₂ and Zn.

Manganese dioxide can theoretically deliver a capacity of approximately 617mAh/g. It delivers this capacity through a 2 electron electrochemical reaction (each electron providing around 308mAh/g). MnO₂ has been found to be rechargeable when the capacity has been limited to around 5-10% of the 617mAh/g. It suffers a crystal structure breakdown as more of the capacity is accessed, and it inherently forms electrochemical irreversible phases. If the entire 2 electron capacity can be accessed then theoretically it can reach energy density numbers near lithium-ion batteries. Similar problems are associated with the zinc electrode, where higher utilization of its capacity causes dendrite formation, shape change and formation of inactive zinc oxides that ultimately lead to electrode failure. These are the main deterrent to a cheap and safe battery that could be a disruptive technology in the energy storage field.

At the City College of New York, we have made a breakthrough in accessing the second electron capacity by altering the crystal structure of manganese dioxide through dopants and novel and cheap synthesis routes. We have cycled the MnO₂ electrode to well over 3000 cycles at rates that are of interest in the battery community. Also, in this talk I will go through the various breakthroughs that have been made at the City College of New York with regards to solving other fundamental problems and the scope of these batteries in the current market.

4:45 PM - EE8.2.07

Highly Flexible and Transparent Solid-State Supercapacitors Based on RuO₂/PEDOT Hybrid Ultrathin Films

Chuanfang (John) Zhang¹, Thomas Higgins¹, Jonathan Coleman¹, Valeria Nicolosi¹.

¹, Trinity College Dublin, Dublin, Ireland.

Hide Abstract

The ever-increasing demanding of flexible, portable electronics requires advanced energy storage devices, in particular supercapacitors. Traditional transparent supercapacitors were achieved by decreasing the electrode thickness. However, this method would induce the percolation effect.¹⁻³ On the other hand, increasing the electrode thickness would promote the specific capacity (or capacitance) at the expense of electrodes transmittance. Therefore it's very necessary to develop ultrathin films with highly pseudocapacitive material and conductive pathways. Here we report on a RuO₂/PEDOT hybrid ultrathin film by a simple aerosol-jet spray technique. The RuO₂ provides high pseudocapacitance while PEDOT provides electric double-layer capacitance as well as efficient electrons pathways. It was found that RuO₂ loading and electrode thickness have great effects on the transmittance and sheet resistance of

the thin film, which could be well explained and fitted via the percolation theory.^{1,3} Under a critical RuO₂ loading (below 50 wt.%), percolation problems could be avoided. By loading 40 wt.% RuO₂, the hybrid thin film demonstrated a transmittance of 94%, low sheet resistance, small time constant and high areal capacitance (1.4 mF cm⁻²). Moreover, the composite thin film (40 wt.% RuO₂) indicates a high electrical conductivity ($\sigma_{DC} = 755$ S/cm), which is much higher than most of the solution-based graphene and carbon nanotube thin films. The solid-state symmetric supercapacitors were assembled, demonstrating fast-rate responses and retained 100% of initial capacitance after 10000 cycles. Solid-state asymmetric supercapacitor was also assembled and exhibited a voltage window of 1.2 V and a transmittance of 84%. Furthermore, thin films with large area (100 cm²) were also sprayed and assembled into asymmetric solid-state supercapacitor, demonstrating its scalability. Such a highly transparent supercapacitors with excellent electrochemical performances could find possible applications in many flexible and transparent electronics.

References

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2016-03-30

Symposium EE8

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EE8.3: Frontiers of Li-Ion Chemistry

- Chair: Mitchell Anstey
- Chair: Travis Anderson
- Wednesday AM, March 30, 2016
- PCC North, 100 Level, Room 121 C

8:00 AM - *EE8.3.01

Electron-Donating Phenothiazines for Energy Storage Applications

Susan A. Odom¹, Matthew Casselman¹, Aman Preet Kaur¹, Corrine Elliott¹, Chad Risko¹.

¹, University of Kentucky, Lexington, Kentucky, United States.

Hide Abstract

Phenothiazines have seen widespread use as stable electron-donating organic compounds with generally stable oxidized states, making them an amenable core for functionalization toward electrochemical energy storage applications. With phenothiazine itself as a starting material, functionalization of the 3, 7, and 10 positions is facile, providing options to modify redox potentials and improve stability in both the neutral and singly oxidized (radical

cation) states. Additionally, this ring system can be built from arylamines and arylbromides, allowing for the production of compounds with even more functionalization, including incorporating groups at the 1 and 9 positions, as well as the entire periphery of the aromatic core. My group has prepared a dozens of phenothiazine derivatives using both synthetic strategies and has used DFT calculations to predict the structural and electronic properties of even more potential products. This presentation focuses on our characterization of the properties of different classes of phenothiazines, both from an experimental and computational perspective, and includes results from incorporation into lithium-ion batteries as electrolyte additives for overcharge protection and into stationary non-aqueous redox flow batteries as catholytes. In many cases, computational studies verify what we have observed in experimental settings, and often guides our design of next-generation materials.

8:30 AM - *EE8.3.02

Water-in-Salt Electrolyte Enables High Voltage Aqueous Li-Ion Chemistries

Chunsheng Wang¹, [Liumin Suo](#)¹, Kang Xu², Oleg Borodin².

1, , Univ of Maryland, College Park, Maryland, United States; 2, , Army Research Lab, Adelphi, Maryland, United States.

Hide Abstract

Li-ion batteries still cause safety, environmental and cost concerns, which mostly arise from the non-aqueous electrolytes. The use of aqueous alternatives is limited by their narrow electrochemical stability window (1.23 V), which sets an intrinsic limit on the practical voltage and energy output. We report a highly-concentrated aqueous electrolyte, whose window was expanded to ~3.0 V with the formation of an electrode/electrolyte interphase. A full Li-ion battery of 2.3 V using such aqueous electrolyte was demonstrated to cycle up to 1000 times, with nearly 100% Coulombic efficiency at both low (0.15 C) and high (4.5 C) rates.

9:00 AM - EE8.3.03

Evaluation of Binders for High Capacity Si/Graphite Composite Anode

[Jun Wang](#)¹, Ronnie Wilkins¹, Paul Gionet¹, Paul Graham¹, Derek C. Johnson¹.

1, , A123 Systems LLC, Waltham, Massachusetts, United States.

Hide Abstract

Loss of accessible active material is a typical failure mode for the high capacity anode due to significant volume change during cycling, which is particularly critical for Si/graphite based anodes. This necessitates careful design of binders to ensure sufficient electrode integrity for better cycle life.

The intent of this study was to explore the impact of binder composition on electrode properties and cell performance. Various commercial grade binders, such as carboxyl methylcellulose (CMC) and styrene butadiene rubber (SBR), Polyvinylidene fluoride (PVdF), polyacrylic acid (PAA), and polyacrylonitrile (PAN), were investigated. The high capacity anode system utilized in this investigation was a Si/graphite composite anode as Si is a high volume expansion alloying material that is gaining considerable interest. The study demonstrated that aqueous based binders, such as CMC/SBR and PAA, exhibited low adhesion strength but high first cycle coulombic efficiency (CE), whereas a PVdF based anode typically showed high adhesion strength but low first cycle CE. Interestingly, a NMP (N-Methylpyrrolidone) based hybrid binder exhibited the desired balance between adhesion strength and first cycle CE. A Si/graphite anode formulated with the hybrid binder of PVdF/PAA demonstrated properties associated with the individual binder; that is the coating had an adhesion strength more indicative of a PVdF based binder with a first cycle CE representative of a PAA binder. Highlighting the synergy between the binders is the approximate 6.7% first cycle CE increase was seen with the hybrid binder for the same anode, resulting in more cell capacity when compared to the PVdF based counterpart.

Specific details regarding the study and investigation into additional hybrid binder systems will be presented as this approach is expected to be applicable to other combinations such as PVdF/PAN, PAN/PAA, and PVdF/CMC.

9:15 AM - EE8.3.04

New Iron-Based Mixed-Polyanionic Compounds as Cathode Materials for Rechargeable Lithium- and Sodium-Ion Batteries for Grid Applications

[Amitava Choudhury](#)¹, Hooman Yaghoobnejad Asl¹.

1, , Missouri University of Science and Technology, Rolla, Missouri, United States.

Hide Abstract

Polyanion compounds (phosphates, sulfates, borates, and silicates) of transition metals are receiving an ever increasing attention as potential positive electrode in the secondary alkali ion batteries. They possess certain advantages over the traditional transition metal oxides due to their higher inherent safety and structural stability granted by the covalent bonding in the polyanion. The vast possibilities of creating a diverse 2D layered and 3D framework crystal structures through the incorporation of a different secondary anion into the polyanion compound and subsequent tuning of the cell voltage has not been explored much. In this context polyanion compounds of iron can be competitive for grid applications where safety, long cycle and calendar life, environmentally friendliness and low cost of the cathode materials supersede the need of high specific energy and energy density constraints of mobile applications.

In this paper we are presenting new polyanion compounds of iron and will demonstrate their ability to function as the cathode materials for low-cost and scalable fabrication of lithium- and sodium-ion batteries with good current capabilities and cycle-life. This work will introduce three recently discovered mixed polyanionic compounds of iron: $\text{Li}_3\text{Fe}(\text{HPO}_3)_3\text{Cl}$, $\text{Li}_x\text{Fe}(\text{H}_2\text{O})_2\text{B}(\text{PO}_4)_2\cdot\text{H}_2\text{O}$, and $\text{LiFePO}_4\text{NO}_3$; a report on their structure and electrochemical activities with respect to Li- and Na-ion cells. All these compounds show average voltage of 3 V and experimental specific capacity of about 100 mAh/g. The last compound, $\text{LiFePO}_4\text{NO}_3$, is a first member of a newly discovered family of mixed polyanionic compounds combining nitrate and phosphate, where it allows the study of the effect of variation of secondary polyanion on the cell voltage and performance. Moreover for the same compound reversible Na^+ intercalation proved to be feasible which makes it a possible candidate for Na-ion battery cathode material. The presentation will cover the crystal structure of the respective compounds as solved through single-crystal or synchrotron powder X-ray diffraction as well as their chemical, compositional and electrochemical studies employing spectroscopic techniques including Mossbauer spectroscopy and galvanostatic charge/discharge tests.

9:30 AM - *EE8.3.05

An Overview of Energy Storage Options for Large Scale Storage

Sanjoy Banerjee¹, Joshua W. Gallaway¹.

1, The Energy Institute and Chemical Engineering Department, City University of New York, New York, New York, United States.

Hide Abstract

Batteries will be the electricity storage option of choice for emerging grid applications provided costs and charge-discharge performance can be made comparable to pumped hydro storage, together with low hazards and environmental impact to enable widespread deployment. Pumped hydro storage is relatively inexpensive, typically adding about \$0.02/kWh to the cost of electricity for the storage component. It accounted for 99% of worldwide storage capacity prior to 2012 and has become the benchmark for other grid storage technologies, although its implementation is limited. Batteries are a more versatile storage option, allowing for a wide range of sizes, from hand-held devices to massive grid-integrated installations. They can be deployed easily and placed at the end of the distribution pipeline, thus enhancing overall round trip efficiency for grids by reducing transmission and distribution losses.

The purpose of this talk will be to give perspective on the future of battery storage technologies, especially with regard to the performance needed for large scale storage materials. There is considerable confusion about the basic costs for manufacture and other performance characteristics of the various battery options, as data is not usually presented by vendors in comparable or consistent form. Also different storage applications would differ in their requirements such as cycle life: 300 charge-discharge cycles might be appropriate for home power backup, while 1000 cycles might be needed for industrial UPS and cell tower support and 2000 for large-scale solar firming. With all types of batteries, higher cycle life is achieved by incorporating more active material in the battery electrodes, essentially allowing a less-demanding depth of discharge. Thus longer-lived batteries have a higher sales price. This will be discussed with respect to the various battery options, such as lead-acid, lithium-ion, and alkaline, all of which are actually umbrella terms including a range of more specific designs.

10:00 AM -

BREAK

Hide Abstract

EE8.4: Redox Flow Batteries

- Chair: Sagar Mitra
- Chair: Susan Odom
- Wednesday AM, March 30, 2016
- PCC North, 100 Level, Room 121 C

10:30 AM - *EE8.4.01

Next Generation Flow Batteries

Wei Wang¹, Vincent L Sprenkle¹.

¹, Pacific Northwest National Lab, Richland, Washington, United States.

Hide Abstract

Redox flow batteries (RFBs) have increasingly being recognized as a prominent candidate for large-scale energy storage due to their unique advantages of high safety, decoupling of power and energy, long lifespan, quick response, and potentially low cost. This presentation describes the recent progress of the advanced redox flow battery technologies developed at Pacific Northwest National Laboratory. On the component level, new developments on the high energy density electrolyte, high selective membrane, and catalytic electrode will be reported. New redox flow battery chemistry based on soluble low-cost redox active materials will be discussed, which eliminate the need for expensive metal-based redox couples. Compared with other reports in this area, the new redox flow battery technology demonstrated higher voltage, energy density, rate capability, and improved cycling stability.

11:00 AM - *EE8.4.02

Advances in Materials for Ionic Liquid Flow Batteries

Travis Mark Anderson¹, Leo J. Small¹, Harry Pratt¹, Cy Fujimoto¹.

¹, Sandia National Laboratories, Albuquerque, New Mexico, United States.

Hide Abstract

The utilization of ionic liquids in flow batteries enables unique advantages such as intrinsic conductivity, higher operating voltages, and amelioration of vapor pressure issues, but it also introduces technical challenges. Ionic liquids typically have higher viscosities than aqueous-based electrolytes, and they are considerably more expensive. To circumvent the aforementioned issues, we have invented a family of metal-based ionic liquids that are prepared in a single-step process using low cost and earth abundant commercial precursors. This metal-based ionic liquid flow battery requires an ion-selective membrane to provide ionic conductance between electrodes while preventing crossover of electroactive species. However, most commercial components for flow batteries are designed for aqueous use. In order to determine if commercially available anion exchange membranes might be suitable for use in our system, we measured area-specific conductances and through-plan conductivities of a number of materials. Concurrently, we also synthesized and tested Diels Alder polyphenylene membranes with three different ionic contents and tested them in the ionic liquid flow battery. These membranes displayed a significant increase in conductivity compared to most of the commercial membranes we tested. The membrane with an intermediate ion content displayed high Coulombic efficiency, but the electrochemical yield decreased over time due to crossover. Improvements in the ionic selectivity and ionic conductivity are currently underway and are expected to increase both the electrochemical yield and voltage efficiency. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

11:30 AM - EE8.4.03

Dialkyl Sulfide-Based Electrolytes for Nonaqueous Redox Flow Batteries

Leo J. Small¹, Harry Pratt¹, Cy Fujimoto¹, Travis Mark Anderson¹.

1, , Sandia National Laboratories, Albuquerque, New Mexico, United States.

Hide Abstract

Here we present recent work in the development of dialkyl sulfide based ionic liquid electrolytes for nonaqueous redox flow batteries (RFB) aimed at grid-scale energy storage. We demonstrate complexation of various redox-active metal salts by dialkyl sulfides and the resulting performance of these anolytes and catholytes in a nonaqueous RFB. While decreased viscosities and moderate conductivities allow for use without dilution in solvent, mismatched viscosities, and the resulting pressure across the membrane influence RFB performance. The real time performance of the electrode-electrolyte-membrane system is monitored using impedance spectroscopy, providing insights into degradation mechanisms and identification of superior anion exchange membranes for this electrolyte system. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

11:45 AM - EE8.4.04

Low-Cost, Pump-Free, Membrane-Free and Highly Dynamic ZnBr₂ Batteries for Grid Scale Storage

Daniel Steingart¹, Shaurjo Biswas¹.

1, , Princeton Univ, Princeton, New Jersey, United States.

Hide Abstract

Bromine cathodes are attractive as an electrochemical oxidant storage media because they have high energy densities and rate capabilities, combined with long cycle lives on a reactant basis. To date, the engineering difficulties of containing and maintaining the support structure in a flow battery have bromine flow cells from reaching their maximized cost potential. In this project, we use uncomplexed bromine in passively cooled closed cells, leveraging the relative density of bromine to exploit gravity-based separation with lossy carbon foam electrodes. Preliminary data indicates that a static ZnBr₂ system can be built for a cost << \$50/kWhr and the "self discharge" behavior of Br₂ (aq) attacking plated Zn can be exploited for cell "self maintenance". By removing the pumps and membranes from a traditional ZnBr₂ system, we are removing the majority of the cost and failure points.

Despite not having pumps nor membranes, we demonstrate an unoptimized cell design that has a coulombic efficiency of > 95%, and an energy efficiency of greater than 70% at a cost of under \$50/kWhr with a C/5 cycling basis. This performance is without irreversible damage to the battery with stable performance for over 100 cycles and counting. In this presentation we will discuss the cell design, materials processing and performance relationships addressed, and potential routes for addressing remaining failure mechanisms of polybromide formation and carbon degradation.

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