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ENERGY & ENVIRONMENT DIVISION

Exploratory Technology Research Program for Electrochemical Energy Storage

Executive Summary Report for 1992

K. Kinoshita, Editor

October 1993

ENERGY & ENVIRONMENT DIVISION

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EXPLORATORY TECHNOLOGY RESEARCH PROGRAM FOR ELECTROCHEMICAL ENERGY STORAGE

EXECUTIVE SUMMARY REPORT FOR 1992

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October 1993

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EXECUTIVE SUMMARY

The U.S. Department of Energy's Office of Propulsion Systems provides support for an Electrochemical Energy Storage Program, that includes research and development (R&D) on advanced rechargeable batteries and fuel cells. A major goal of this program is to develop electrochemical power sources suitable for application in electric vehicles (EVs). The program centers on advanced systems that offer the potential for high performance and low life-cycle costs, both of which are necessary to permit significant penetration into commercial markets.

The DOE Electrochemical Energy Storage Program is divided into two projects: the Electric Vehicle Advanced Battery Systems (EVABS) Development Program and the Exploratory Technology Research (ETR) Program. The EVABS Program management responsibility has been assigned to Sandia National Laboratory (SNL), and the Lawrence Berkeley Laboratory* (LBL) is responsible for management of the ETR Program. The EVABS and ETR Programs include an integrated matrix of R&D efforts designed to advance progress on selected candidate electrochemical systems. The United States Advanced Battery Consortium (USABC), a tripartite undertaking between DOE, the U.S. automobile manufacturers and the Electric Power Research Institute (EPRI), was formed in 1991 to accelerate the development of advanced batteries for consumer EVs. The role of the ETR Program is to perform supporting research on the advanced battery systems under development by the USABC and EVABS Program, and to evaluate new systems with potentially superior performance, durability and/or cost characteristics. The specific goal of the ETR Program is to identify the most promising electrochemical technologies and transfer them to the USABC, the battery industry and/or the EVABS Program for further development and scale-up. This report summarizes the research, financial and management activities relevant to the ETR Program in CY 1992. This is a continuing program, and reports for prior years have been published; they are listed at the end of this Executive Summary.

The general R&D areas addressed by the program include identification of new electrochemical couples for advanced batteries, determination of technical feasibility of the new couples, improvements in battery components and materials, establishment of engineering principles applicable to electrochemical energy storage and conversion, and the development of air-system (fuel cell, metal/air) technology for transportation applications. Major emphasis is given to applied research which will lead to superior performance and lower life-cycle costs.

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* Participants in the ETR Program include the following LBL scientists: E. Cairns, K. Kinoshita and F. McLarnon of the Energy and Environment Division; and L. DeJonghe, J. Evans, J. Newman, P. Ross and C. Tobias of the Materials Sciences Division.

The ETR Program is divided into three major program elements: Exploratory Research, Applied Science Research, and Air Systems Research. Highlights of each program element are summarized according to the appropriate battery system or electrochemical research area.

EXPLORATORY RESEARCH

The objectives of this program element are to identify, evaluate and initiate development of new electrochemical couples with the potential to meet or exceed advanced battery and electrochemical performance goals. Research was conducted on new versions of the **Zn/NiOOH cell** and a novel **Na/polymer cell**. Each of these cells is considered to be an attractive candidate for EV applications, and should provide high performance at ambient or near-ambient temperatures. A Zn/NiOOH battery would exhibit higher performance and lower costs, compared to the MH/NiOOH battery; and a Na/polymer battery would lower cost, compared to a Li/polymer battery.

- LBL has observed that the Zn/KOH/NiOOH cell capacity retention and NiOOH electrode coulombic efficiencies are superior in cells containing NiOOH electrodes loaded at 1.3 g/cm^3 void space, compared to cells containing NiOOH electrodes loaded at 1.6 g/cm^3 void space.
- A subcontract was awarded to the Advanced Energy Systems Division of Acme Electric Corporation on the basis of a Request-for-Proposals to extend the development of Zn/NiOOH cell technology. The objectives of this project are to evaluate the LBL electrolyte composition for extending the cycle life of Zn/NiOOH cells and to develop Zn/NiOOH battery technology for EV applications. Formation cycles of cells containing sintered Ni positive/plastic-bonded Zn negative or fiber-bonded Ni positive/plastic-bonded Zn negative showed that good cell capacities can be obtained, but indicate that some technical issues must be resolved to achieve the state-of-the-art performance obtained at LBL.
- Cycle testing of Na/PEO/$Na_{0.6}CoO_2$ cells is underway at LBL. Over 100 cycles were obtained at 0.5 mA/cm^2 to 50% capacity and up to 200 cycles at somewhat lower depths of discharge.

APPLIED SCIENCE RESEARCH

The objectives of this program element are to provide and establish scientific and engineering principles applicable to batteries and electrochemical systems; and to identify, characterize and improve materials and components for use in batteries and electrochemical systems. Projects

in this element provide research that supports a wide range of battery systems — alkaline, metal/air, flow, solid-electrolyte and nonaqueous. Other cross-cutting research efforts are directed at improving the understanding of electrochemical engineering principles, minimizing corrosion of battery components, analyzing the surfaces of electrodes, and electrocatalysis. **Electrode Characterization** studies are an important research element for the successful development of rechargeable electrodes for advanced secondary batteries. Efforts are underway to determine the role of surface morphology on mass transport which may affect the morphology of electrodeposits. Advanced spectroscopic techniques are utilized to investigate the chemical state of electrode materials during charge/discharge cycling.

- LBL is extending the study on the effect of protrusions on mass transport to electrodeposited surface layers, and the coalescence of electrolytic gas bubbles which may affect the morphology of electrodeposits. A mathematical model has been developed that uses the Boundary Integral Element Method (BIEM) to understand the hydrodynamics of flow over protrusions. Electrochemical cells to study this phenomena and to observe the coalescence of electrolytic gas bubbles were designed and fabricated.
- Brookhaven National Laboratory (BNL) has used extended X-ray absorption fine structure (EXAFS) and X-ray absorption near-edge spectroscopy (XANES) to study manganese oxides compounds, Bi-doped manganese oxide and $Li_x MnO_2$. EXAFS data were obtained at both the Mn K edge and the Bi L_3 edge for Bi-doped manganese oxide that was discharged in 9 M KOH. At the end of the one-electron discharge the manganese oxide is highly disordered, while on completion of the two-electron discharge, the product reverts back to an ordered phase.

Improved Components for Alkali/Sulfur Cells, such as superior alternatives to the high-temperature sulfur-polysulfide electrode for Na/S cells, are under investigation.

- LBL demonstrated that the performance of sulfur electrodes is markedly improved by the addition of phosphorus. The study showed that the best performance in Na/S cells was obtained with a P/S ratio of 0.143 in the range 0.143 to 1.17. Equilibrium electromotive force (EMF) measurements showed that at least three three-phase regions were present in the Na-P-S ternary system at 350 and 400°C. A Na/S_xP_y battery would exhibit ~11% higher specific energy than a comparable Na/S battery.

Corrosion Processes in High-Specific-Energy Cells are under investigation, and the aim is to develop low-cost container and current-collector materials for use in nonaqueous, alkali/sulfur and molten-salt cells.

- Illinois Institute of Technology (IIT) is evaluating the quality of Mo_2C coatings obtained by plasma-enhanced chemical vapor deposition (CVD). X-ray photoelectron spectroscopy (XPS) analysis indicate that the films deposited on a glass or steel substrate contain a significant fraction of oxygen. The use of hydrogen and methane as a carrier gas is being evaluated to minimize the oxygen activity in the reaction chamber.

- The Environmental Research Institute of Michigan (ERIM) has prepared TiN-coated Al containment materials by sputter-deposition techniques for corrosion testing in molten Na_2S_4 at 350°C. After three weeks in the polysulfide melt a conductive surface film was still present on the sample coated with TiN. Final analyses of the physicochemical properties of the coating have not been completed.

- Johns Hopkins University has extended the studies on the passivation and anodic behavior of iron and 1018 carbon steel in $LiAsF_6$/dimethoxyethane (DME). In a nominally dry $LiAsF_6$/DME solution containing 50-100 ppm H_2O, the dissolution of Fe and the electropolymerization of the DME solvent are believed to play a major role in the repassivation mechanism. In addition, the electropolymerization reaction is dependent on the presence of $LiAsF_6$; no polymerization is observed in its absence or when other salts are present instead.

Components for Ambient-Temperature Nonaqueous Cells, particularly metal/electrolyte combinations that improve the rechargeability of these cells, are under investigation.

- Case Western Reserve University (CWRU) has used *in situ* spectroscopic techniques and thermal analysis to study the Li/organic electrolyte interface. X-ray photoelectron spectroscopy (XPS) studies indicate that the interactions between Li and perdeuterated tetrahydrofuran (TDF) are strong, leading to substantial structural modifications. Thermal desorption studies with TDF revealed that Li can dehydrogenate TDF to form LiD as one of the predominant products. This type of information is needed to develop a Li electrode/electrolyte combination that exhibits reversible behavior.

- CWRU is utilizing *in situ* EXAFS and XANES to investigate electrochemical phenomena in Li/polymer electrolyte and Li/FeS_2 cells. Initial effort has centered on studies of Li/FeS_2 cells that operate at room temperature. During discharge Li is inserted into FeS_2, and the corresponding EXAFS spectra revealed a gradual loss in the fine structure as the amount of Li is increased.

Cross-Cutting Research is carried out to develop mathematical models of electrochemical systems and to address fundamental problems in electrocatalysis and current-density distribution; solutions will lead to improved electrode structures and performance in batteries and fuel cells.

- LBL has developed mathematical models to understand transport and kinetic phenomena occurring in selected electrochemical systems. Models have been developed to predict the behavior of Li/polymer electrolyte/metal oxide cells during charge and discharge. Comparisons were made between cells that contain a Li metal negative and Li-intercalated carbon negative.
- LBL has used ion implantation to modify the properties of Ni electrodes for alkaline batteries and grid materials for lead-acid batteries. Co-implanted Ni electrodes appear to have a higher coulombic storage capacity than comparable electrodes without the addition of Co. On the other hand, Cu implantation has the opposite effect. Implantation of Ti in Pb and Pb-4%Sb grids reduces the corrosion current in 5 M H_2SO_4 by a factor of about 30-fold and 70-fold, respectively.
- LBL has observed that the highest activity for the methanol electrooxidation on Pt-Ru alloys occurs on an alloy with a surface composition of 10 at% Ru. On these alloys, the adsorption of methanol is believed to occur on the three-fold Pt surface sites where the hydrogen of the methanol is oxidized. The remaining CO fragment diffuses to the neighboring Ru where it undergoes oxidation. These studies help point the way to the development of a stable, high-performance direct-methanol fuel cell (DMFC).
- LBL has observed that the performance of dispersed Pt-Ru for the electrooxidation of vaporized methanol in 72 wt% Cs_2 CO_3 reached a maximum at a temperature of 10 to 15°C below the boiling point of the electrolyte. This performance level is the highest ever observed for a DMFC with low catalyst loading.

AIR SYSTEMS RESEARCH

The objectives of this program element are to identify, characterize and improve materials for air electrodes; and to identify, evaluate and initiate development of metal/air battery systems and fuel-cell technology for transportation applications.

Metal/Air Cell Research projects address bifunctional air electrodes, that are needed for electrically rechargeable metal/air (Zn/air, Fe/air) cells; and novel alkaline Zn electrode structures, that could be used in either electrically recharged or mechanically recharged cell configurations.

- CWRU has completed an extensive 8-year (1984-1992) study of oxygen electrocatalysis on various transition-metal macrocyles and oxide catalysts. This effort resulted in a better understanding of O_2 reduction and evolution on these electrocatalysts in acid and alkaline electrolytes. The significant accomplishments of the research are summarized.
- Eltech Research Corporation is investigating the viability of graphitized carbon blacks and metal oxides as electrocatalyst supports in bifunctional air electrodes for electrically rechargeable Zn/air cells. Graphitized carbon blacks of both Monarch 120 and Shawinigan acetylene black appear to be suitable electrocatalyst supports. Electrochemical tests of $NiCo_2O_4$/Monarch 120 in small cells at MATSI achieved over 350 cycles (discharge 4 h at 10 mA/cm^2, charge 8 h at 5 mA/cm^2).
- LBL has operated a Zn/air cell which employed a reticulated Zn electrode structure under natural convection. A small laboratory cell (77 cm^2 air-electrode active area) was operated at 20 mA/cm^2 at an average cell voltage of 0.9 V. Measurements with a laser-Doppler velocimeter showed that the electrolyte velocity under natural convective flow during cell discharge was 0.1 mm/s. This kind of Zn/air cell would be simpler to operate and more reliable than other Zn/air cells with flowing electrolyte.
- LBL evaluated several procedures to prepare $La_{0.6}Ca_{0.4}CoO_3$ which has shown promise as an electrocatalyst in bifunctional air electrodes. The amorphous citrate precursor method yielded fairly pure powders with limited crystallinity and BET surface areas up to 18 m^2/g. A preliminary cycle life of 45 cycles in an electrolyte of 45 wt% KOH and 40 g Zn^{2+}/l was obtained with the electrocatalyst in gas-diffusion electrodes, which may be limited by flooding of the electrode and/or slow degradation of the electrocatalyst.

Fuel Cell Research at Los Alamos National Laboratory (LANL) includes research in several areas of electrochemistry, theoretical studies, fuel-cell testing, fuel processing, and membrane characterization. Major achievements of the fuel-cell program during 1992 are listed below:

- LANL has found that processing membrane-electrode assemblies (MEA) with Nafion membranes that contain tetrabutyl ammonium are more robust than those obtained from the Na^+ and H^+ forms.

- Life tests of up to 4000 h have been achieved by LANL with only a 10% performance loss with membrane/electrode assemblies with thin-layer catalyst layers in polymer electrolyte fuel cells (PEFC) at 80°C.
- A new membrane from DuPont (Nafion 5L) was evaluated by LANL. It showed a protonic conductivity of about 20% higher than that for Nafion 117. Its water uptake was also about a factor of 2 larger than that for Nafion 117, membrane C, and an experimental Dow membrane.
- LANL has clarified the influence of CO_2 on the performance of anodes in PEFCs. The study showed that the reverse shift reaction involving CO_2 and the electrochemical reduction of CO_2 both produce CO which poisons the anode.
- Permeation of CH_3OH through Nafion was studied by LANL in PEFCs. Permeation rates equivalent to 200 mA/cm^2 were determined with 45% CH_3OH in the fuel stream.
- LANL obtained self diffusion coefficients of 1.4×10^{-5} cm^2/s and 2×10^{-5} cm^2/s for H_2O and CH_3OH, respectively, in Nafion using nuclear magnetic resonance (NMR).
- BNL is utilizing X-ray absorption spectroscopy (XAS) to study the correlation between the electronic properties of carbon supported Pt/Cr, Pt/Co and Pt/Ni alloy catalysts and their electrocatalytic activity for oxygen reduction. The results indicate that alloying Pt enhances the performance of the oxygen electrode in a PEFC operating at 95°C and at 5-atm pressure.

MANAGEMENT ACTIVITIES

During 1992, LBL managed 15 subcontracts and conducted a vigorous research program in electrochemical energy storage. LBL staff members attended project review meetings, made site visits to subcontractors, and participated in technical management of various ETR projects. LBL staff members also participated in the following reviews, meetings, and workshops:

- Fuel Cells for Transportation Meeting, Washington, D.C., January 6-7, 1992
- IECEC Planning Meeting, San Diego, CA, January 23-24, 1992
- Zn/Air Review Meeting, Sandia National Laboratories, Albuquerque, NM, February 4, 1992
- USABC Technical Advisory Committee Meeting, EPRI, Palo Alto, CA, February 11, 1992
- Power Sources Manufacturers Association - Battery R&D Committee Meeting, Colorado Springs, CO, February 13-14, 1992

- 203rd American Chemical Society National Meeting, San Francisco, CA, April 5-10, 1992
- 1992 Spring Meeting - Materials Research Society, San Francisco, CA, April 27-May 1, 1992
- EPRI/GRI Fuel Cell Workshop, San Francisco, CA, April 29-30, 1992
- 181st Meeting of the Electrochemical Society, St. Louis, MO, May 17-22, 1992
- Fuel Cells for Transportation Meeting, Santa Fe, NM, June 9-11, 1992
- Workshop on Advanced Battery Technology R&D, Willowbrook, IL, June 16-17, 1992
- 4th Quarterly Progress Review of EPRI Contract for Development of Sealed Bipolar Zn/NiOOH Batteries, Energy Research Corporation, Danbury, CT, July 10, 1992
- 27th Meeting of IECEC, San Diego, CA, August 3-7, 1992
- SNL/DOE Zinc/Air Battery Development Program Kickoff Meeting, Westinghouse Electric Corporation, Pittsburgh, PA, August 21, 1992
- Power Sources Manufacturers' Association Meeting, Tinton Falls, NJ, September 2, 1992
- 43rd Meeting of the ISE, Cordoba, Argentina, September 19-23, 1992
- 182nd Meeting of the Electrochemical Society, Toronto, Canada, October 11-16, 1992
- Supercities Conference, San Francisco, CA, October 27, 1992
- Annual Automotive Technology Development Contractors' Coordination Meeting, Dearborn, MI, November 2-5, 1992
- Final Review of the ERC Nickel-Zinc Battery Development Project with EPRI, Palo Alto, CA, December 4, 1992

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ANNUAL REPORTS

1. "Technology Base Research Project for Electrochemical Energy Storage - Annual Report for 1991," LBL-32212 (June 1992).
2. "Technology Base Research Project for Electrochemical Energy Storage - Annual Report for 1990," LBL-30846 (June 1991).
3. "Technology Base Research Project for Electrochemical Energy Storage - Annual Report for 1989," LBL-29155 (May 1990).
4. "Technology Base Research Project for Electrochemical Energy Storage - Annual Report for 1988," LBL-27037 (May 1989).
5. "Technology Base Research Project for Electrochemical Energy Storage - Annual Report for 1987," LBL-25507 (July 1988).
6. "Technology Base Research Project for Electrochemical Energy Storage - Annual Report for 1986," LBL-23495 (July 1987).
7. "Technology Base Research Project for Electrochemical Energy Storage - Annual Report for 1985," LBL-21342 (July 1986).
8. "Technology Base Research Project for Electrochemical Energy Storage - Annual Report for 1984," LBL-19545 (May 1985).
9. "Annual Report for 1983 - Technology Base Research Project for Electrochemical Energy Storage," LBL-17742 (May 1984).
10. "Technology Base Research Project for Electrochemical Energy Storage - Annual Report for 1982" LBL-15992 (May 1983).
11. Technology Base Research Project for Electrochemical Energy Storage - Report for 1981," LBL-14305 (June 1982).
12. "Applied Battery and Electrochemical Research Program Report for 1981," LBL-14304 (June 1982).
13. "Applied Battery and Electrochemical Research Program Report for Fiscal Year 1980," LBL-12514 (April 1981).

LIST OF ACRONYMS

AES	Auger electron spectroscopy
AFM	atomic force microscopy
BIEM	Boundary Integral Element Method
BNL	Brookhaven National Laboratory
CMD	chemical manganese dioxide
CVD	chemical vapor deposition
CWRU	Case Western Reserve University
DME	dimethoxyethane
DMPEFC	direct methanol polymer electrolyte fuel cell
DOE	Department of Energy
EMD	electrolytic manganese dioxide
EMF	equilibrium electromotive force
EPRI	Electric Power Research Institute
ERIM	Environmental Research Institute of Michigan
ETR	Exploratory Technology Research
EV	electric vehicle
EVABS	Electric Vehicle Advanced Battery Systems
EXAFS	extended X-ray absorption fine structure
FT	Fourier transforms
FTIRRAS	Fourier transform infrared reflectance absorption spectroscopy
HOPG	highly ordered pyrolytic graphite
HREM	high-resolution electron microscopy
IECEC	Intersociety Energy Conversion Engineering Conference
IIT	Illinois Institute of Technology
ISE	International Society of Electrochemistry
LANL	Los Alamos National Laboratory
LBL	Lawrence Berkeley Laboratory
LEED	low energy electron diffraction
LEIS	low energy ion scattering
MEA	membrane electrode assemblies
NSLS	National Synchrotron Light Source

NMR	nuclear magnetic resonance
OPG	ordinary pyrolytic graphite
PDS	photothermal deflection spectroscopy
PECVD	plasma-enhanced chemical vapor deposition
PEFC	polymer electrolyte fuel cells
PEM	proton-exchange membrane
PEO	poly(ethylene oxide)
PTFE	polytetrafluoroethylene
pzc	point of zero charge
RDE	rotating disk electrode
RFP	Request for Proposal
SCE	saturated calomel electrode
SEM	scanning electron microscopy
SERS	surface-enhanced Raman spectroscopy
SFUDS	Simplified Federal Urban Driving Schedule
SNL	Sandia National Laboratories
SRPE	solid redox polymerization electrode
SSRL	Stanford Synchrotron Radiation Laboratory
STM	scanning tunneling microscopy
TBAP	tetrabutyl ammonium phosphate
TDF	perdeuterated tetrahydrofuran
TEM	transmission electron microscopy
TGA	thermogravimetric analysis
THF	tetrahydrofuran
TMPP	tetramethoxyphenyl porphyrin
TPD	temperature programmed desorption
UHV	ultrahigh vacuum
UPD	underpotential deposition
USABC	United States Advanced Battery Consortium
XANES	X-ray near edge absorption spectroscopy
XAS	X-ray absorption spectroscopy
XPS	X-ray photoelectron spectroscopy

SUBCONTRACTOR FINANCIAL DATA - CY 1992

Subcontractor	Principal Investigator	Project	Contract Value (K$)	Term (months)	Expiration Date	Status in CY 1992*
EXPLORATORY RESEARCH						
Advanced Zn/NiOOH Cells						
Acme Electric Corp.	M. Anderman	Secondary Zn/NiOOH	193	12	9-93	C
APPLIED SCIENCE RESEARCH						
Lawrence Berkeley Laboratory	E. Cairns, L. DeJonghe, J. Evans, K. Kinoshita, F. McLarnon, J. Newman, P. Ross and C. Tobias	Electrochemical Energy Storage	1765	12	9-92	C
Electrode Characterization						
Brookhaven National Laboratory	J. McBreen	Battery Materials	95	12	9-92	C
Corrosion Processes in High-Specific Energy Cells						
Illinois Institute of Technology	R. Selman	Corrosion Resistant Coatings	132	12	8-92	T
Environmental Research Institute of Michigan	T.Hunt	Secondary Batteries	105	12	9-92	C
Johns Hopkins University	J. Kruger	Corrosion	30	12	8-92	T
Components for Ambient-Temperature Nonaqueous Cells						
Case Western Reserve University	D. Scherson	*In Situ* Studies	86	15	8-92	C
University of Pennsylvania	G. Farrington	Polymeric Electrolytes	50	12	7-92	T
AIR SYSTEMS RESEARCH						
Metal/Air Cell Research						
Eltech Research Corporation	E. Rudd	Oxygen Electrodes	125	12	8-92	C
Case Western Reserve University	E. Yeager	Air Electrodes	150	12	4-92	T
Fuel Cell R&D						
Los Alamos National Laboratory	S. Gottesfeld	Fuel Cell R&D	1200	12	9-92	C
Brookhaven National Laboratory	J. McBreen	Fuel Cell Research	94	12	9-92	C

* C = continuing, T = terminating

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