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Executive Summary Report for 1984

TECHNOLOGY BASE RESEARCH PROJECT FOR
ELECTROCHEMICAL ENERGY STORAGE

May 1985

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EXECUTIVE SUMMARY REPORT

FOR 1984

TECHNOLOGY BASE RESEARCH PROJECT FOR ELECTROCHEMICAL ENERGY STORAGE

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EXECUTIVE SUMMARY

The U.S. Department of Energy's Office of Energy Systems Research provides continuing support for an Energy Storage Program, which includes R&D on advanced electrochemical energy storage and conversion systems. A major goal of this program is to develop electrochemical power sources suitable for application in electric vehicles and/or electric load-leveling devices. The program centers on advanced secondary batteries and fuel cells 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 Exploratory Technology Development and Testing (ETD) Project and the Technology Base Research (TBR) Project. ETD Project management responsibility has been assigned to Sandia National Laboratory (SNL), and the Lawrence Berkeley Laboratory (LBL) is responsible for management of the TBR Project. The ETD and TBR Projects include an integrated matrix of research and development efforts designed to advance progress on several candidate electrochemical systems. The role of the TBR Project is to perform supporting research for the advanced battery systems under development by the ETD Project, and to evaluate new systems with potentially superior performance, durability and/or cost characteristics. The specific goal of the TBR Project is to identify the most promising electrochemical technologies and transfer them to industry and/or the ETD Project for further development and scale-up. This report summarizes the research, financial, and management activities relevant to the TBR Project in CY 1984.

General problem areas addressed by the project 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 assessment of fuel-cell technology for transportation applications. Major emphasis is given to applied research which will lead to superior performance and lower life-cycle costs.
The TBR Project is divided into three major project elements: Exploratory Research, Applied Science Research, and Air Systems Research. Highlights of each project element are summarized according to the appropriate battery system or electrochemical research area.
Exploratory Research

The objectives of this project 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 projects were conducted on ambient-temperature lithium cells, novel Na/S systems, and a thermally-regenerative system.

Ambient-Temperature Lithium Cells

- Duracell, Inc. has identified the Li/LiAlCl₄−SO₂/C system for further development of an all-inorganic ambient-temperature rechargeable lithium battery. Small prototype cells (0.4 Ah, 100 Wh/kg) were fabricated and tested. A cycle life of 60-90 cycles at 100% depth-of-discharge (DOD) has been achieved, and cells on partial discharge (60% DOD) achieved a few hundred cycles.

- EIC has found that the stability of lithium in sulfolane-based electrolytes is good during storage at temperatures up to 70°C. However, lithium electrodes show inadequate cycling efficiencies in these electrolytes due to various factors. Further effort on this project has been discontinued because of the problems encountered with electrolyte purification and poor cycling efficiency of the lithium electrode.

Sodium/Sulfur Systems

- Experiments at the University of Tennessee on Na/β"-alumina/SCl₃⁺ in AlCl₃-NaCl cells indicate a compromise between high specific energy and long cycle life may be required to achieve a viable battery system. "Sulfur-rich" cells showed high specific energy but the performance degraded with cycling.

- Dow Chemical Company has developed a sensitive technique involving anion chromatography that can detect sulfur corrosion of glass electrolytes of as little as 10⁻⁶ cm of the glass surface. Reaction rates and kinetics of sulfur attack on 16 glasses were determined at 300° and 400°C. The standard Dow D406 glass was found to be the most resistant to attack at 300°C, but it was rapidly attacked at 400°C.
Thermally-Regenerative System

- Experiments are underway at Ford Motor Company to improve the performance and endurance of electrodes for a thermally-regenerative electrochemical system using sodium (sodium heat engine). Encouraging results were obtained with several cermet electrodes which were produced by sintering of porous $\beta''$-alumina layers on commercial $\beta''$-alumina tubes, followed by impregnation with molybdenum salt or by flame spraying $\beta''$-alumina and metal powder.

Applied Science Research

The objectives of this project 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, molten-salt, and non-aqueous. Other projects are directed at research on improving the understanding of electrochemical engineering principles, corrosion of battery components, surface analysis of electrodes, and electrocatalysis.

Alkaline Cells

- Experiments at LBL are directed at developing practical means of reducing zinc species solubility in strong alkaline electrolytes. The addition of Ca(OH)$_2$ to the zinc electrodes does not appear to enhance its cycle life.

- Lockheed Missiles and Space Co. has concluded from their studies on porous zinc electrodes in alkaline electrolytes that, to prevent shape change to the zinc electrode during cycling, cell designs should be implemented which maintain uniform lateral current distribution. These modifications could include (i) graded porosity in the separator material, (ii) graded ohmic resistance or porosity of the substrate, and (iii) graded porosity of the counter electrode.
SRI International has examined the effect of various additives (Li$^+$, Co$^{2+}$, Fe$^{2+}$, F$^-$, ZnO, and PbO) on the charge-discharge behavior of Zn and Ni electrodes in alkaline electrolyte. The addition of F$^-$ ions was found to decrease the O$_2$-evolution overpotential on the Ni electrode and decrease the capacity of the Zn electrode, while the addition of PbO may have some beneficial effect.

Raman spectroscopy studies of zincate species in KOH at Argonne National Laboratory revealed a spectrum indicative of the presence of Zn$^{2+}$ ions and [Zn(OH)$_4$]$_2^+$ complexes, where n > 1.

Ohio State University has completed measurements on the thermodynamic activity of water in the binary KOH-H$_2$O system at 25°C.

Sandia National Laboratory has found that sulfonated polysulfones show encouraging performance as separators for zinc/ferricyanide cells. These separators showed no weight-loss after exposure to alkaline-ferricyanide electrolyte at 50°C for one week, and they exhibited a low resistance ($1 - 5$ ohm-cm$^2$) and a low rate of ferricyanide permeation ($10^{-5}$ mmol/h/cm$^2$). An average efficiency of 75% was achieved with these separators for over 200 cycles in cell tests.

A number of membranes (polyvinyl alcohol cross-linked with crown ether) have been fabricated by Brigham Young University. They exhibit conductivities ranging from 0.02 to 0.033 mho/cm; these values are approaching the target of 0.01 mho/cm.

Hybrid separators developed by Pinnacle Research Institute appear to possess ion selectivity that is comparable to that of ion-exchange membranes.

The development of supported liquid-membrane separators for Zn/NiOOH cells at Castle Technology Corp. was discontinued due to a change in the priorities for the TBR Project.
**Zinc/Halogen Cells**

- The morphology of zinc electrodeposited from 1.0 M ZnCl₂ (0.7 < pH < 4.6) was investigated at LBL by studying the initial stages of deposition. Kinetic measurements using a potential-step method yielded exchange current densities of -1 mA/cm². The density of zinc nodules increased with surface potential at a rate from 200 to 1000 nodules/mm²-mV.

- Illinois Institute of Technology has concluded from their modeling studies that the direct effect of convection on tip growth rate during deposition of zinc nodules is quite small, since tip growth is kinetic rather than mass-transfer controlled. However, the predicted growth rates are smaller that the experimental results by one to two orders of magnitude.

- Brookhaven National Laboratory (BNL) has found that the additions of stoichiometric or excess stoichiometric amounts of AlCl₃ to ZnBr₂ electrolytes has a favorable effect on the deposition zinc at a zinc electrode. It was observed that the electrode kinetics were slowed, a fine-grain deposit was produced, and dendrite growth was inhibited.

- Lawrence Livermore National Laboratory (LLNL) has completed measurements of the mutual diffusion coefficient (25°C) for the ZnCl₂-H₂O system over the concentration range 0.004996-7.2489 mol/kg H₂O.

**Molten-Salt Cells**

- Argonne National Laboratory has completed development of the electrode performance-evaluation system. Evaluation of negative electrodes of the family LiₓAl₁₋ₓSiₙ indicate that the composition with n = 0.5 has higher specific power that Li-Si electrodes and higher specific energy than Li-Al electrodes. Tests of a dense FeS₂ electrode in LiCl-KCl at 410°C indicated a factor of two increase in capacity utilization at high current density (100 mA/cm²) and a cell resistivity that is about 50% lower than that of conventional FeS₂ electrodes.
Thermogravimetric analysis by Gould, Inc. indicates that FeS$_2$ electrodes must be operated at $285^\circ$C or lower to avoid sulfur evolution. The loss of sulfur from FeS$_2$ alone is about 70 times greater than that from FeS$_2$ in a eutectic LiCl-KCl electrolyte.

Stanford University has examined numerous lithium-based systems for negative electrodes; these include Li-Al, Li-Sn, Li-Sb, Li-Bi, Li-Zn, Li-Cd and Li-Pb. It has been found that a number of these binary systems exhibit high chemical diffusion coefficients at near ambient temperatures, so that their electrode reactions should be quite rapid.

**Components for Alkali/Sulfur Cells**

- Experiments at LBL suggest that the asymmetric polarization observed with sodium electrodes can be minimized or eliminated by the addition of a chemical oxygen scavenger, which reacts with Na$_2$O at an appreciable rate to yield an insoluble oxide and sodium metal.

- Experimental studies at Rockwell International indicate that ZrO$_2$-toughened $\beta''$-alumina should contain between 10-15 vol% ZrO$_2$ to obtain the optimum strength and ionic conductivity.

- A family of glasses with compositions analogous to NASIGLAS has been synthesized by the Massachusetts Institute of Technology. The conductivity at $350^\circ$C for several of the sodium-based glasses is as high as $10^{-4}$ ohm$^{-1}$cm$^{-1}$.

- A novel electrochemical method was developed at the Massachusetts Institute of Technology which is capable of simultaneously determining the electrolytic and chemical stabilities of solid electrolytes. This method indicated that the glass composition Li$_2$O-B$_2$O$_3$, which contained 2 mol% Al$_2$O$_3$, behaved as a perfect lithium-ion electrolyte over a wide range of lithium activity (up to 0.08 at $380^\circ$C) and temperature (320-400°C).
• LBL has developed a successful procedure for making and blowing lithium-chloroborate glass into thin-walled sealed tubes.

• Argonne National Laboratory has completed evaluation of 24 soda-rich glasses in the soda-alumina-zirconia-silica system for use in Na/S cells. Static corrosion tests in sodium, sulfur, and sodium polysulfide at 400°C for 1000 hr indicated that four of the glasses have relatively little weight-loss (0.8 mg/cm²) in all three environments. Further evaluation of these four materials in Na/S cells is underway.

• LBL is developing mathematical models that are useful for understanding the operation of Na/S cells. The mathematical models provide improved understanding of the operation of the sulfur electrode and the transport processes in sodium-polysulfide melts.

Corrosion Processes in High-Temperature, High-Sulfur Activity Molten Salts

• LBL has employed static corrosion and dynamic polarization tests to characterize the behavior of Mo, Cr and 70% Cr-30% Fe in both sulfur-sodium polysulfide and sulfur-lithium polysulfide melts. The dynamic polarization behavior is found to depend strongly on the identity of the cation in the molten-salt mixture, as well as the metal.

• Argonne National Laboratory has examined the corrosion stability of numerous metals and alloys in sulfur and sodium polysulfide at 350°C. Their results indicate that these materials show vastly different corrosion rates in the two sulfur environments. It appears that materials with corrosion stability (e.g., Al, Ni-Cr-Mo-W and Fe-Cr-Mo alloys) in sulfur may not be as stable in sodium polysulfide and vice-versa.

• Illinois Institute of Technology has deposited a nickel interlayer on low-carbon steel by conventional electroplating and electroless nickel plating. Experiments are underway to coat these samples with molybdenum and to test their stability in a high-sulfur-activity environments.
Components for Ambient-Temperature Lithium Cells

- Surface layers formed on lithium in LiClO\textsubscript{4} with < 1 ppm H\textsubscript{2}O have been analyzed by LBL. From observations using IR spectroscopy, ESCA analysis, and in-situ X-ray diffraction analysis, it was concluded that Li\textsubscript{2}CO\textsubscript{3} and a polymeric compound are formed.

- UV-visible spectroscopy experiments of iodine and bromine solutions at LBL have demonstrated that the stable form of the halogen in propylene carbonate is the trihalide.

- The University of Pennsylvania has found that MgCl\textsubscript{2} complexes of poly(ethylene oxide) (PEO) exhibit a conductivity at 60°C that is comparable to that of the LiCF\textsubscript{3}SO\textsubscript{3}-PEO complex. These new complexes may be a general group of high-conductivity solid electrolytes for rechargeable lithium batteries.

- Efforts at Case Western Reserve University to develop multi-block copolymers for use as membranes in lithium batteries was discontinued due to difficulties encountered in the synthesis of these materials.

Cross-Cutting Research

- Illinois Institute of Technology has completed a draft "Bibliography of Convective Transport Correlations." Suggestions by reviewers have been incorporated in the draft copy, and the final report will be available in 1985.

- LBL has developed models that are useful in the identification of electrochemical parameters. These models will aid in the design and scale-up of electrochemical systems; they are used to (i) calculate the concentration, potential, and current distribution in thin-gap flow cells, (ii) predict important operating characteristics of LiAl/FeS\textsubscript{2} cells, and (iii) predict the effect of redistribution of active material on the cycle life of Zn/NiOOH batteries.
• An electrolyte cell was constructed by LBL which allows observation of coalescence phenomena at gas bubbles generated on microelectrodes. Preliminary results indicate that for bubbles smaller than 1 mm in diameter, the coalescence event is completed within $10^{-5}$ s. Earlier observations by researchers which showed that bubbles could touch without immediate coalescence may have resulted from slight axial misalignment in their electrochemical cells.

• Low-energy electron diffraction (LEED) analysis has been used at LBL to obtain fundamental understanding of the processes occurring at electrode surfaces. The LEED analysis was applied to investigate the anodic reconstruction of metal surfaces and the electrodeposition/dissolution of metals.

• Porous electrodes containing transition-metal sulfides (CuS, MoS$_2$ and CoS), carbon black and PTFE were fabricated at LBL. Experiments are underway to determine the kinetic parameters for the sulfide/polysulfide redox couple.

Air Systems Research

The objectives of this project 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 Battery Research

• Results obtained by pyrolysis gas chromatography-mass spectroscopy at Case Western Reserve University indicate that heat-treatment to 850°C of metal macrocycles supported on carbon produces volatile fragments that do not contain nitrogen. Data obtained by transmission electron microscopy and Mossbauer spectroscopy suggest that the iron and cobalt tetramethoxyphenyl porphyrins, which are adsorbed on carbon, decompose at 800°C to produce the corresponding oxides. It appears that the supported metal oxides have high catalytic activity for oxygen reduction.
• Experiments at LBL indicate that the catalytic effect of metal oxides on carbon corrosion in alkaline electrolytes does not involve a redox mechanism. Extensive testing of graphitized furnace blacks has identified graphitized Sterling R as the only material with a corrosion rate significantly lower than that of acetylene black.

• Energy Research Corporation has found perovskites of the composition $\text{LaNiO}_3$ and $\text{La}_{0.5}\text{Sr}_{0.5}\text{CoO}_3$ show promising performance for bifunctional air electrodes.

• Experimental work on potential transients within fluidized-bed electrodes and moving-bed electrodes has been completed at LBL. This study showed that the phenomenon referred to as "bubbling" (development of regions in the electrode that are almost free of particles) occurs with zinc-coated copper particles, but it does not occur with Sorapex particles.

• Development work on the iron/air battery at Westinghouse Electric Corp. has been discontinued due to the limited life and performance of their bifunctional air electrode containing carbon blacks.

Aluminum/Air Battery R & D

• The major thrusts of the Aluminum/Air battery program were on (i) air cathode development, (ii) hydrargillite crystallization and separation, (iii) integrated experiments involving a coupled cell/crystallizer, (iv) cell design, and (v) development of improved aluminum anodes. Major achievements of the program include:

- Two experiments were performed at LLNL in which a single cell was coupled to a crystallizer. Although the experiments lasted only 5 hr (at nominally 120 A), a great deal of important systems-design information was obtained.

- Experiments at Eltech Systems, Electromedia, and Case Western Reserve University have identified RB (de-ashed) and Norit Ultra-carbon, both containing 5-10% CoTMPP, as promising air cathodes.
Two proprietary aluminum anodes developed by Alcan show promise with an open-circuit corrosion rate of 0.12 mg/cm²/min and an open-circuit potential of 1.84 V.

Alcan has obtained some significant results on the agglomeration of hydrargillite. Their measurements indicate that particle agglomeration to >40 μm occurs under "low turbulence" conditions in reasonably short times (2 hr).

Fuel Cell Research

Los Alamos National Laboratory (LANL) has conducted research in several areas of electrochemistry, theoretical studies, fuel-cell testing, fuel processing, and membrane characterization, as well as managed subcontracts on fuel cell R&D. Major achievements of the fuel-cell program include:

- Theoretical studies of the PtO and Pt₂ molecules were conducted at LANL. These studies indicate that a charge transfer of 0.34 electrons takes place from the Pt atom to the O atom; and the Pt-Pt bond length in Pt₂ is 2.33 Å, which is less than that for bulk Pt.

- A six-station, H₃PO₄ fuel-cell test stand was completed at LANL to investigate the effects of rapid current pulsing, slow load-current ramping, and temperature cycling on electrode performance.

- Rutherford back scattering and X-ray photoelectron spectroscopy verified that the activity decay observed with electrodes tested for 2000 hr in the six-station test stand was due to Pt redistribution in the cell. Pt from the cathode appears to migrate and deposit on the anode.

- LANL investigated the mechanism of methanol steam reforming on active catalysts. The oxygen atom in CO₂ is provided by water and not methanol, apparently occurring by rapid oxygen exchange on the catalyst surface.
A 20-kW fuel-cell system was delivered by Energy Research Corporation, and tests of the system have started at LANL. The original system efficiency of 30% decreased to 26%, but overall the system performed reasonably well.

United Technologies Corporation has observed that heat-treated macrocyclic (iron tetramethoxyphenylporphyrin) supported on carbon exhibits stable performance in half-cell test in H₃PO₄ at 140°C. However, the electrocatalyst decays in performance in full-cell tests; the decay mechanism has not been identified.

An alternative membrane to Nafion that was tested by the General Electric Company showed encouraging performance. Over 1200 hr of operation were accumulated using this membrane in a cell at 180°F, 30 psig O₂ and 30 psig H₂.

The University of Virginia was successful in quenching disorder in a 25 at% Cr-75 at% Pt sample. Previous investigators reported that it was not possible to retain disorder in samples of this composition.

HREELS (High Resolution Electron Energy Loss Spectroscopy) studies at BNL showed that various adsorbed species involving Pt-O and Pt-C bonds are formed when H₂O, O₂, CO, and CH₄ interact with the (111) face of a Pt single crystal.

Kinetic studies at LBL using the rotating ring-disk electrode technique indicate that the current for oxygen reduction in dilute Cs₂CO₃ is higher than that in KOH at the same concentration. Because Cs₂CO₃ solutions are a CO₂-rejecting electrolyte, the favorable kinetics for oxygen reduction that was observed is encouraging for the development of alkaline fuel cells using alternatives to alkali-hydroxide electrolytes.
Planned Activities in 1985

New research projects planned include:

1. An RFP will be issued for research on novel concepts for metal/air batteries.

2. New research projects to address corrosion in electrochemical systems will be considered.

Management Activities

During 1984, LBL managed 36 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 TBR projects. LBL staff members participated in the following reviews, meetings, and workshops:

• Sodium-Beta"-Alumina Review Meeting, SNL, Albuquerque, NM, March 29, 1984.
• Spring Meeting of the Electrochemical Society, Cincinnati, OH, May 6-11, 1984.
Workshop on Conducting Polymers, Rochester, MI, June 4-8, 1984.


23rd Annual Conference of Metallurgists, Quebec City, Canada, August 19-23, 1984.


Fall Meeting of the Electrochemical Society, New Orleans, LA, October 7-12, 1984.


Lead Center Coordination Meeting, LBL, Berkeley, CA, November 7, 1984.

Aluminum/Air Battery Subcontractors Meeting, LLNL, Livermore, CA, November 8, 1984.


Milestones for Technology Base Research Project

Milestones accomplished in Fiscal Year 1984 by the TBR project include:

- A decision was made not to pursue engineering development of the iron/air battery at Westinghouse Electric Corp. due to problems with the present state-of-technology. The corrosion stability of the carbon support that is presently used in their bifunctional air electrode is not adequate to achieve a cycle life of 1000 cycles, and improved electrocatalysts with high activity and stability are not available.

- Development of two computer codes, "SIMPLE" and "M32.bas" by LLNL has been completed. These codes are used to analyze crystallization parameters in the crystallizer of the aluminum/air cell. The computer code has been expanded to take into account the effect of particle size distribution on the crystallizer-cell interaction.

- Fuel-cell endurance tests of macrocyclic electrocatalysts at United Technologies Corporation was recommended based on the promising results obtained in short-term tests. Iron tetramethoxyphenyl porphyrin, which was heat-treated at 850°C, exhibited an intrinsic activity higher than that of Pt on a metal-weight basis to a specific current of 300 mA/mg.

- Rockwell International has found that the addition of tetragonal-ZrO₂ (up to 25 vol%) to β''-alumina increased its fracture toughness by up to 66%, and its strength by a factor of two or three. Based on these results and data obtained at LBL on this transformation-toughened β''-alumina, it was recommended that the project at Rockwell International be continued.

- A review of the Duracell, Incorporated program on the development of an all-inorganic rechargeable lithium battery was held to assess their technology. The results of their research are sufficiently encouraging that it was recommended that this battery system continue to receive DOE support.

- Hamilton Standard Electrochemical (formerly a part of the General Electric Company) is investigating proton-exchange membrane/electrocatalyst assemblies (M&E) with the goal of reducing the cost of the membrane and electrocatalyst, while still maintaining the performance levels presently achieved with Nafion and high loadings of Pt electrocatalyst (4 mg/cm²). The results to date are encouraging, and they indicate that low-cost M&Es are attainable. Based on these developments, it is recommended that the development of low-cost membrane/electrocatalyst assemblies continue.
Acknowledgement

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# Subcontractor Financial Data - CY 1984

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