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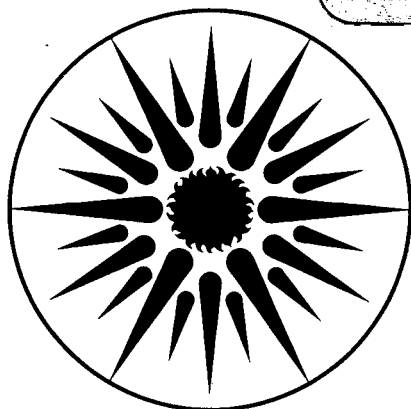
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ON ADVANCED SECONDARY BATTERIES

by

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Introduction

The U.S. Department of Energy (DOE) provides continuous 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 that offer the potential for high performance and low life-cycle costs, both of which are necessary to permit significant penetration into commercial markets. Widespread introduction of such technologies could ease the worldwide demand for petroleum-based fuels. The DOE Electrochemical Energy Storage Program was the subject of a recent conference, and abstracts/summaries are compiled in References 1 and 2.

The DOE Electrochemical Energy Storage Program is divided into two projects: the Exploratory Technology Development and Testing (ETD) Project, which is described in Reference 3, and the Technology Base Research (TBR) Project, which is summarized in Reference 4. 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 (conducted by a number of corporations, national laboratories and universities) 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.

Supporting research activities include engineering-science research, encompassing electrode morphological/chemistry studies, transport phenomena, electrocatalysis, and modeling; and materials research, which includes the study and characterization of electrolytes (ceramic, molten-salt, glass, polymeric), separators, electrodes, and other cell components, as well as efforts to mitigate corrosion problems.

Types of advanced secondary batteries and cells under consideration include high-temperature cells, flow batteries, metal/air batteries, and ambient-temperature lithium cells. High-temperature cell research encompasses the identification of new electrolytes for Na/S cells, the characterization of molten-salt cells, the development of corrosion-resistant current-collector and cell-container materials, and the study of new solid electrolytes for Li/S cells. Flow battery (Zn/Br₂, Zn/Cl₂) research efforts are focussed on the identification of conditions leading to smooth, compact zinc deposits, as well as cell modeling. Metal/air battery R&D activities include the engineering development of mechanically-rechargeable Al/air batteries, and research on O₂ electrodes, Zn electrodes, and separators. Ambient-temperature lithium cell work encompasses the development of rechargeable Li/SO₂ cells, the characterization of new liquid organic electrolytes, and the study of novel polymeric electrolytes.

High-Temperature Cells

Significant improvements are needed in the reliability of and manufacturing processes for the β'' -alumina electrolyte used in Na/S batteries. On-going research efforts at LBL employ electrochemical, microscopic, and acoustic-emission testing to characterize the degradation phenomena that eventually lead to fracture of the electrolyte and short-circuiting of the cell. Four types of degradation are observed: (i) chemical coloration (associated with partial reduction of the electrolyte), (ii) sodium precipitation in the interior of the electrolyte (Mode II degradation), (iii) crack initiation and propagation (Mode I degradation), and (iv) attack of the electrolyte by sulfur species. Recent work (5) has shown the importance of the grain size of β'' - Al_2O_3 material to its resistance to Mode I degradation.

The Rockwell International Science Center is studying methods to fabricate transformation-toughened β'' - Al_2O_3 electrolytes. Rockwell has found (6) that the addition of up to 15 vol% tetragonal ZrO_2 as a second phase to β'' - Al_2O_3 results in a material with improved mechanical properties, when compared to single-phase β'' - Al_2O_3 , without a significant increase in ionic resistivity. ZrO_2 additions result in increased fracture toughness and strength, associated with a tetragonal-monoclinic phase transformation in the stress field of flaws, as well as control of grain growth of the β'' - Al_2O_3 phase.

The Massachusetts Institute of Technology is synthesizing new crystalline electrolytes with high sodium-ion conductivity. The crystal structures are characterized by impedance measurements and neutron-diffraction analysis to provide information on the distribution and

transport of mobile ions. The crystal structure of the NASICON system ($\text{Na}_{1+x}\text{Zr}_2\text{Si}_x\text{P}_{3-x}\text{O}_{12}$) has been determined (7), but NASICON is unstable in sodium at elevated temperatures. New compositions, with structures identical to that of NASICON but without the presence of phosphorous (which is linked to the degradation observed in molten sodium), are being synthesized. Argonne National Laboratory (ANL) is evaluating (8) a series of glasses in the quaternary system soda-alumina-zirconia-silica, which represent glass analogs of NASICON. Compositions have been identified which exhibit a sodium-ion resistivity as low as 130 ohm-cm at 300°C, which is about two orders of magnitude lower than that of the sodium-borate glass employed by Dow Chemical Co. (9) in their hollow-fiber Na/S cell technology.

The University of Tennessee has evaluated (10) intermediate-temperature 4.2V Na/S(IV) cells, in which the sulfur species are dissolved in NaCl-AlCl_3 molten salt at 180-250°C. Studies of laboratory cells have demonstrated cycle lives of more than 1000 cycles, and the capability of two-plateau reduction of S^{+4} to S^{-2} .

Argonne National Laboratory and Stanford University are evaluating components for high-temperature molten-salt cells that offer the potential for very high performance (200 Wh/kg, 200 W/kg). Ternary lithium alloys, such as Li-Al-Si and Li-Al-Sb, are being studied in detail, and their phase diagrams have been determined. The Li-Al-Si alloys appear to offer a favorable compromise between high capacity and low polarization over a wide range of Li content (11). Positive-electrode materials, such as FeS_2 , NiS_x , and a number of tunnel-structure and layer-structure ternary oxides, are also under study (12,13).

Gould Research Center has completed investigations of the thermal management of LiAl/FeS cells (14) and LiAl/FeS₂ cells (15). The temperature coefficient of the emf of 6-Ah LiAl/FeS₂ cells was found to be positive at all measured states of charge, which indicates that the electrochemical discharge reaction is endothermic. Calorimetric measurements of heat generation by 54-Ah LiAl/FeS₂ cells showed that the discharge process is endothermic during the upper (FeS₂) voltage plateau and exothermic during the lower (FeS) plateau. LBL has developed (16) a general energy balance equation, which is useful for estimating the cell thermal characteristics required for the design and thermal management of high-temperature molten-salt cells. LBL previously developed a complete mathematical model (17) of the LiAl/FeS cell, and present efforts are directed toward a model that describes the thermal and discharge behavior of the LiSi/FeS₂ cell.

Several organizations are evaluating new materials that may be useful for positive-side current collection and containment in Na/S and Li alloy/FeS₂ cells. ANL is evaluating a series of alloys containing Ni, Fe, Cr, Mo, etc. for their resistance to corrosion in sulfur and sodium polysulfides. The Illinois Institute of Technology is applying new methods of chemical vapor deposition and molten-salt electrodeposition to form corrosion-resistant coatings of TiN, MoC and Mo on base metals. LBL has employed static corrosion and dynamic polarization tests (18) 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.

Both glassy and crystalline lithium-ion conductors are being evaluated for possible use in Li/S secondary cells. The Massachusetts Institute of Technology has characterized (19) series of glasses in the $\text{Li}_2\text{O-LiCl-B}_2\text{O}_3$ system, and some glasses have demonstrated lithium-ion conductivity as high as $0.02 \text{ ohm}^{-1} \text{ cm}^{-1}$ at 300°C . LBL is evaluating the stability of several of these glasses in Li alloy/S cells. Stanford University has synthesized a number of crystalline lithium ion-conducting solid electrolytes that may be stable in contact with lithium. The crystalline compound $\text{Li}_2\text{Zr}(\text{PO}_4)_2$ exhibits (20) a lithium-ion conductivity approaching $10^{-2} \text{ ohm}^{-1} \text{ cm}^{-1}$ at 300°C .

Flow Batteries

The reliability and performance of zinc/halogen flow batteries depend strongly on the degree to which the morphology of electrodeposited zinc can be controlled during battery recharge. The zinc quality, including the presence of unwanted dendrites, nodules and striations, is strongly dependent upon mass-transfer conditions, electrolyte composition (including the presence of additives and impurities), current density and substrate effects.

LBL has investigated (21) the effect of hydrodynamic flow and other process variables on the macromorphology of electrodeposited zinc at a rotating-disk electrode and in flow channels. Scanning electron microscopy (SEM) and in-situ time-lapse motion picture photography were used to characterize the electrodeposited zinc. The results support the hypothesis that zinc striations are the result of the strong dependence of nucleation rate on overpotential in zinc-halide systems. The Illinois Institute of Technology is determining the growth rate of zinc

electrodeposits on rotating concentric cylinder electrodes, using SEM and in-situ optical microscopy to study the morphology of zinc dendrites and nodules. Corrosion of zinc growths by dissolved halogen is being studied experimentally and computationally (22). Brookhaven National Laboratory has identified crystal orientation and electrolyte composition (23) to be important factors controlling the extremely fast kinetics of the zinc deposition reaction. The kinetics are about an order of magnitude faster in 3M ZnBr₂ than in 3M ZnCl₂, and about an order of magnitude faster on the basal plane than on the plane perpendicular to the basal plane.

LBL has developed (24) design equations to permit economic comparisons of flow-through and flow-by porous electrodes, and a mathematical model of a thin-gap flow cell is under development. Lawrence Livermore National Laboratory (LLNL) has completed measurements (25) of the mutual diffusion coefficients of high-purity aqueous ZnCl₂ solutions, and diffusion coefficients of the ternary system ZnCl₂-KCl-H₂O were measured. The behavior of the ternary system is very complex, with some compositions exhibiting negative cross-term diffusion coefficients.

Metal/Air Cells

The aluminum/air system (26) offers the possibility of a high-specific-energy battery with rapid "rechargeability" through mechanical insertion of aluminum anodes, periodic withdrawal of Al(OH)₃ product, and replenishment of H₂O. Recent efforts, coordinated by LLNL, have focussed on the development of wedge-shaped cells to permit full utilization of aluminum anodes, evaluation of processes to permit the separation of Al(OH)₃ from the alkaline electrolyte, and the development of

non-platinum catalysts for use in air electrodes. A full-scale (600 cm²) wedge cell, which employs solution-side current collection for the wedge-shaped aluminum anodes, has been successfully tested, and the crystallizer/separator system demonstrated control of aluminate concentration. ELTECH Systems Corporation, as prime industrial subcontractor, is pursuing development of the Al/air battery system. ELTECH has evaluated a number of air cathode structures and catalysts, and electrodes catalyzed with cobalt tetramethoxyphenyl porphyrin have demonstrated (27) both performance and lifetime superior to those demonstrated by Pt-catalyzed cathodes.

Case Western Reserve University has synthesized various phthalocyanines (28) and porphyrin (29) compounds and evaluated their performance as catalysts for oxygen reduction and generation. These materials have been characterized by a wide range of electrochemical, spectroscopic and microscopic techniques, and some have shown very high catalytic activity for oxygen reduction in both alkaline and acidic electrolytes, as well as long-term stability in alkaline electrolytes. These efforts have also led to improved understanding (30) of the mechanisms of oxygen reduction on various catalysts.

Corrosion of the carbon substrate material has been identified (27,31) as a fundamental process limiting the life of carbon-based air electrodes. LBL has employed (32) microscopy and ¹⁴C radiotracer studies to characterize the corrosion behavior of various types of carbons, and it was found that the carbon microstructure is an important fundamental property. Energy Research Corporation is evaluating non-carbon-based structures for use as bifunctional air electrodes. Perovskite

compounds (33) have shown good catalytic activity for both reduction and evolution of oxygen, as well as long-term stability.

The Westinghouse Research and Development Center has completed tests on 40-cm² bifunctional air electrodes for use in secondary Fe/air batteries (34). A new catalyst, which is a mixture of the hydroxides of Ni, Fe and Co, has shown good performance.

Alkaline Cells

LBL has employed a computer-controlled test system (35) to study the cycle-life performance of model electrochemical cells containing polymer-bonded alkaline zinc electrodes. X-ray, microscopic and chemical analyses are used to characterize the cycled electrodes. It has been found (36) that zinc electrodes cycled in alkaline-fluoride and alkaline-borate electrolytes, in which the solubility of zinc-bearing species is reduced to about 25% of that in "standard" 31% KOH electrolyte, exhibited substantially reduced rates of cell capacity decline.

Lockheed Missiles and Space Corporation (37) and LBL (38) have developed model single-pore cells that permit simultaneous microscopic observation of the electrode surface and recording of the potential profile. Local precipitation of oxidized zinc species at a zinc anode has been correlated with local changes in electrode potential.

SRI International is combining (39) metal-electrochemistry and semiconductor-electrochemistry techniques to characterize the oxide films on Ni, Fe and Zn electrodes in alkaline electrolytes. Comparisons are made between film-free metal surfaces, single-crystal metal oxides, and thick electrodeposited oxide films. The role of various additives

(to the electrode and/or the electrolyte) has been elucidated. The Ohio State University (OSU) has completed (40) a study of the thermodynamic properties of various metals (Fe, Ni, Zn, Al and Li) in concentrated KOH, NaOH and LiOH as a function of temperature. OSU also completed (41) a study in which the degradation of porous alkaline nickel electrodes is correlated with changes in their AC impedance behavior.

Several laboratories are pursuing the development of improved membrane separators for secondary alkaline zinc batteries. Brigham Young University is fabricating anion-exchange membranes containing potassium salts of crown-ether compounds which are stable in strongly alkaline electrolytes. It is anticipated that membranes such as these will exhibit transport properties that slow (or reverse) the zinc material redistribution (shape change) associated (42) with osmotic flow of electrolyte between the anode and cathode compartments of secondary alkaline zinc cells. Pinnacle Research Institute and RAI, Inc. have collaborated (43) to fabricate and evaluate hybrid battery separators using radiation-grafting techniques. These materials demonstrate the low electrolytic resistivity characteristic of microporous separators, yet they offer good permselectivity against zincate ion. Testing of hybrid separators in alkaline-zinc cells is in progress. Castle Technology Corporation has completed an evaluation (44,45) of supported-liquid membranes, materials in which highly specific organic transport reagents are contained within microporous separators, in secondary alkaline zinc cells. Supported-liquid membranes display ionic resistances as low as 0.4 ohm-cm^2 , and they almost completely inhibit transport of zincate ions. Cycle-life tests showed that cells containing one layer of a supported-liquid membrane exhibited lifetimes comparable to those of

cells containing three layers of standard Celgard* microporous separator.

Ambient-Temperature Lithium Cells

Duracell International, Inc. has studied (46) the cell $\text{Li/LiAlCl}_4\text{-SO}_2/\text{CuCl}_2$, which has a theoretical specific energy of 550 Wh/kg when the 1-electron (upper plateau) reduction of CuCl_2 is utilized. More than 200 deep-discharge cycles have been obtained, and an overcharge mechanism (breakdown and recombination of LiAlCl_4) has been identified. EIC Laboratories, Inc. has completed a study (47,48) on the use of sulfolanes in rechargeable lithium batteries. Techniques for the purification of sulfolane were developed, but lithium cycling efficiencies were not sufficiently high to warrant continued investigation.

LBL has employed electrochemical, microscopic and ellipsometric techniques (49) to characterize the protective surface layers that form on lithium electrodes in organic electrolytes such as propylene carbonate and 2-methyl-tetrahydrofuran. It has been recently found (50) that the presence of residual water in propylene carbonate greatly affects the properties of the surface layer. LBL has also evaluated the iodine/iodide couple as a potential positive-electrode reaction for use in organic electrolytes. The complex stoichiometry of this reaction has been analyzed using the theory of cyclic voltammetry (51).

* Celanese Corporation

The University of Pennsylvania has completed studies (52,53) on the stability and electrochemistry of polyacetylene, which has potential application as an electrode material in batteries using non-aqueous electrolytes. The results show that polyacetylene is very sensitive to oxidation, the efficiency of the oxidation/reduction process is strongly dependent on the depth of charge/discharge, and a polyacetylene-based battery would exhibit low specific energy. Case Western Reserve University has completed evaluation (54) of a process to synthesize novel copolymer membranes for secondary lithium batteries. Block copolymers, in which one block is amorphous and lithium-ion solvating, and the second block is crystalline, should give strong films with high permeability. Multiblock copolymers were prepared, and the resulting film was flexible and strong (77 kg/cm² tensile strength).

Future Trends

The Technology Base Research Project will continue to support a balanced research effort for high-temperature cells. Emphasis will be placed on the development of new electrolytes and high-performance, corrosion-resistant components for use in solid-electrolyte Na/S and Li/S cells, as well as molten-salt-electrolyte Li alloy/metal disulfide cells. The Rockwell process to produce transformation-toughened sodium beta"-alumina electrolytes will be evaluated for compatibility with practical manufacturing methods.

The aluminum/air battery project is advancing to the development stage, and management responsibility will be transferred to Sandia National Laboratory's Exploratory Technology Development and Testing Project.

Supporting research for flow batteries, metal/air batteries, alkaline cells, and ambient-temperature lithium cells will continue, as well as exploratory research to identify new, high-performance couples.

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