

DEVELOPMENT OF A SEALED BIPOLAR Li-ALLOY/FeS₂ BATTERY FOR ELECTRIC VEHICLES

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ABSTRACT

Rapid progress is being made in the development of a bipolar Li-alloy/FeS₂ battery with molten LiCl-LiBr-KBr electrolyte and MgO powder separator. Such a battery would be operated at 400-425 °C and is expected to attain high performance and long cycle life (>1000 cycles). An overcharge tolerant cell design safeguards the Li-alloy/FeS₂ cells in the bipolar battery and allows charge equalization with simple trickle charging. New ceramic materials have been developed for use as the peripheral hermetic seals required for electrolyte containment in bipolar cells. A stack of four bipolar Li-alloy/FeS₂ cells with these seals has been operated for over 150 cycles. Projected performance for a prototype sealed bipolar Li-alloy/FeS₂ battery is 191 Wh/kg specific energy and 500 W/kg peak specific power through 80% depth of discharge. Outstanding electric-vehicle performance can be expected from such a battery.

INTRODUCTION

Research on the Li-alloy/FeS₂ molten-electrolyte battery has recently focused on the development of a sealed bipolar version. To maintain the electrolyte molten, cells are normally operated at temperatures of 375-425°C. The high-temperature operation provides advantages, as well as engineering and materials development challenges. The objective of this research is the development of a Li-alloy/FeS₂ battery that will attain high performance (~200 Wh/kg and 500 W/kg) and long cycle life (>1000 cycles) with the potential for low-cost fabrication. Electrolyte-starved (ES) cell operation yields high performance and enables application of a low-cost MgO-powder separator to replace the BN felt separator. These cells are both overdischarge tolerant and overcharge tolerant (OCT), both of which are important for practical operation of the bipolar battery. New materials have fostered development of hermetic seals which are resistant to the corrosive molten-salt environment. The sealed bipolar battery will substantially increase battery performance and still maintain long life.

Improvements in cell electrochemistry came about from a change in molten-electrolyte composition, to 25 mol % LiCl-37 mol % LiBr-38 mol % KBr (m.p. 320°C), and use of a more densely loaded FeS₂ electrode containing CoS₂.¹ These cells are operated at lower temperature than earlier cells (about 400°C) and only on the upper voltage plateau (U.P.). Cell tests have demonstrated excellent capacity stability for >1000 cycles. A prismatic bicell (12 Ah and 100 cm² separator area) operated at a 2-h discharge rate and a 4-h charge rate achieved 1027 cycles and 7000 h,² after which operation was voluntarily terminated. The cell retained greater than 80% of initial capacity.

Overdischarge and overcharge tolerance for the cells is handled by modifications to the Li-alloy electrode. Cell capacity is limited by the Li-alloy electrode capacity. These "lithium-limited" cells avoid excessive expansion of the U.P. FeS₂ electrode from being discharged into the lower voltage plateau. An U.P. FeS₂ cell test of over 900 cycles demonstrated the long-term stability of a "lithium-limited" cell in which a Li-Al electrode is polarized by 300 mV to end each discharge.² Overcharge tolerance is achieved by abruptly increasing the lithium activity of the lithium-alloy electrode near the end of charge capacity to establish a "lithium-shuttle" self-discharge mechanism.³

A major challenge for bipolar battery development is the electrolyte containment. Our innovative design approach, in which each cell in the bipolar stack is hermetically sealed, promises to provide long-term stability (capacity and coulombic efficiency) and overcharge tolerance. The unique feature of the bipolar battery is that the positive and negative electrodes share a common current collector. Because of this arrangement, measures have to be taken to avoid the appreciable self-discharge that would result from an electrolyte path connecting the negative and positive electrodes. Earlier work on bipolar batteries concentrated on forming gaskets between the two electrode assemblies to retain the electrolyte; these batteries were short-lived. In our present bipolar cell design, a peripheral seal for each cell is formed prior to cell/battery assembly.

Li-ALLOY/FeS₂ CELL ELECTROCHEMISTRY

Li-alloy/FeS₂ cell electrochemistry has been modified as we sought to replace the BN felt separator with an MgO powder separator, which has the potential for lower costs and greater thermodynamic stability in overcharge-tolerant U.P. FeS₂ cells. However, the MgO powder separator, since it is not physically stable under flooded electrolyte conditions, must be operated in an "electrolyte-starved" cell configuration. In our efforts to develop an electrolyte-starved, overcharge-tolerant cell, we fabricated and tested LiAl + 10 mol % Li₅Al₃Fe₂/U.P. FeS₂ cells (22.5-Ah capacity) with LiCl-LiBr-KBr electrolyte and 100-cm² MgO powder separators. These cells demonstrated performance comparable to that of the flooded cell with BN felt separator. To increase the ionic conductivity of the electrolyte-starved cell, which is normally about 30% lower than that of the flooded cell, the electrolyte composition was shifted off-eutectic to a LiCl-rich composition (in mol %, 34 LiCl-32.5 LiBr-33.5 KBr), which has a 25% higher ionic conductivity than that of the eutectic at 425 °C.⁴ This higher ionic electrolyte conductivity approximately compensates for the reduced electrolyte content of the electrolyte-starved cell. Therefore, the area specific impedance (ASI₀) of the electrolyte-starved cells (1.1 Ω cm²) is comparable to that of the flooded cell (1.0 Ω cm²). The capacity

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utilization of the electrolyte-starved cells after more than 50 cycles (>85% at discharge current density of 50 mA/cm²) is now slightly greater than that of the flooded cell.

The effect of conductive additives to the positive electrode (NiS₂, CoS₂) was studied to evaluate the improvement in electrode performance in the LiCl-rich electrolyte. In cells containing the new electrolyte and no electrode additive, about 85% utilization of theoretical FeS₂ capacity was seen at the 50 mA/cm² discharge rate, and, in general, utilization was 10% lower than that seen in similar cells containing an additive. In addition, the impedance of an U.P. FeS₂ electrode without additive (see Fig. 1) was about 10-15% higher (1.1 Ω cm²) than that of an electrode containing 10 mol % NiS₂ (0.9-1.0 Ω cm²). The increased impedance here appears to be electronic rather than ionic in nature; thus, the ASI at 45 ms (the electronic impedance) is about 0.6 Ω cm² for electrodes with no additive, compared to 0.5 Ω cm² for the electrodes with 10 mol % NiS₂ additive. The ASI vs. depth of discharge (DOD) curve for U.P. FeS₂ electrodes with or without additive remains flat during the bulk of the discharge, however. The electrolyte-starved U.P. FeS₂ electrode without conductive additives may have adequate performance for some applications, but noticeable improvement results from just 10 mol % NiS₂ additive. Thus, we have attained satisfactory performance without use of the expensive CoS₂ additive.

CELL OVERCHARGE TOLERANCE DEVELOPMENT

The recent development of overcharge-tolerant Li-alloy/FeS₂ cells was accomplished by an innovative application of a non-destructive self-discharge mechanism, the "lithium-shuttle mechanism."^{3,5} In general, the lithium-shuttle mechanism involves diffusion of lithium metal species across the separator to chemically discharge the positive electrode. The chief controlling element of the mechanism is the lithium activity of the negative electrode, while the cell operating temperature and electrolyte composition are also contributing factors. With this design, lithium shuttle rates of ≥2 mA/cm² develop at Li-alloy electrode potentials of -150 to -200 mV (vs. α + β Li-Al reference electrode) in LiCl-LiBr-KBr at 400 °C. A Li-Al₅Fe₂ alloy with a potential of -260 mV (vs. α + β Li-Al reference electrode) has been applied to engineering development of overcharge-tolerant cells. Altering the electrolyte composition (e.g. LiCl-KCl or LiF-LiCl-LiBr) and cell operating temperature (>400 °C) imposes new limits upon the lithium-shuttle rate, up to 10 mA/cm². Generally, the lithium shuttle rate is sufficient for charge equalization by a trickle-charge procedure.

Tests of overcharge-tolerant monopolar FeS₂ cells (25-Ah capacity) with reference electrodes (Ni/Ni₃S₂) have verified that the lithium-shuttle mechanism provides sufficient levels of overcharge tolerance. Such cells have operated over 200 cycles with high performance and stable overcharge tolerance. In Fig. 2, the electrode potentials vs. percent of capacity utilization for a U.P. FeS₂ cell [LiAl + 10 mol % Li₅Al₅Fe₂/LiCl-LiBr-KBr (MgO)/FeS₂] are given for a charge-discharge cycle. A bulk charge at 25 mA/cm² to 2.03 V was followed by a trickle charge of 3.0 mA/cm². As seen in Fig. 2, the Li-alloy electrode undergoes about a 200 mV transition near full-charge capacity. This is crucial to ensure cell longevity. The U.P. FeS₂ electrode indicates negligible change in potential during the overcharge period; that is, it is protected from the deleterious effects of

overcharge polarization. The self-discharge rate undergoes a stepwise increase (a 20-fold increase by the lithium-shuttle mechanism) to become equal and opposite in effect to the trickle-charge rate. In this test, the trickle-charge period (8 h) charged 15% more coulombs into the electrode than the rated capacity, and the extended trickle charge did not contribute to additional capacity in the subsequent discharge. A coulometric and galvanic analysis of the overcharge-tolerant cell design indicated approximately 0% charge acceptance for a trickle charge of 3.0 mA/cm² for the U.P. FeS₂ cell at 425 °C.

To aid technology transfer, one electrolyte-starved, overcharge-tolerant LiAl + Li₅Al₅Fe₂/FeS₂ type cell was fabricated with electrode and MgO separator plaques that we had pressed by Westinghouse Oceanic (Cleveland, OH). The performance of this cell at 425 °C was outstanding: 92% utilization at a discharge current density of 50 mA/cm² and an ASI_{15s} of 1.2 Ω cm². Concurrently, overcharge tolerance tests indicated that 3-mA/cm² trickle charge rates were tolerated for over 10% additional charge capacity without positive electrode polarization. A near 100% coulombic efficiency was attained for normal charge-discharge cycles before and after the overcharge tolerance tests. As seen in Fig. 3, the ASI curve for the positive electrode as a function of depth-of-discharge (DOD) is virtually flat out to 80% DOD. The impedance values observed at the different potential relaxation times (t = 45 ms to 15 s) indicate that approximately 50% of the electrode impedance is electronic rather than ionic. Such low cell impedance is highly desirable for power-demanding applications such as the electric vehicle. Approximately 95% of cell capacity is available with sufficient power for vehicle acceleration. The demonstrated electrode performances support the projected specific energy of 175 Wh/kg and specific power of 200 W/kg for a 200-Ah prototype monopolar cell design.

Application of the lithium-shuttle mechanism for cells in a bipolar battery stack permits cell capacity equalization by a simple trickle-charge procedure, without use of electronics or cell voltage monitoring. For a typical 2.00 V charge cutoff, a cell with a two-phase negative electrode (Li-Al plus Li-Al₅Fe₂) yields a unique combination of overcharge capacity and overcharge tolerance. The overcharge capacity safeguards the cell and permits unmonitored bulk charge at 20-50 mA/cm² with a battery voltage limit. A bipolar battery can thus be charged with a voltage that is 2.00 V multiplied by the number of cells in the battery (e.g., a 100-cell battery would be charged at 200 V). At completion of a bulk charge, the "stronger" half of the battery cells would attain the overcharge capacity state. (The conventional electronic charger/equalizer, which monitors the voltage of each cell, would have ended the bulk charge when the first cell attained a charge voltage cutoff of 2.00 V.) After the bulk charge, battery cell equalization is accomplished by a battery trickle charge of about 2.5 mA/cm². The "stronger" cells, which have been safeguarded by the overcharge capacity, are charged at approximately 0% coulombic efficiency because the trickle-charge rate balances the self-discharge rate. The "weaker" cells, on the other hand, accept additional capacity at about 2.5 mA/cm² rate over a 3-6 h period and undergo an additional 5-10% increase in capacity. The battery is effectively charge equalized when all the battery cells have reached the overcharge capacity state.

SEALED BIPOLAR CELL DEVELOPMENT

Peripheral hermetic seals are required for the bipolar cell in our design approach, while only a feedthrough seal is required for the monopolar cells. Our bipolar cell development has concentrated on formation of these seals.⁶ These hermetic seals in the Li-alloy/FeS₂ cells have certain chemical and mechanical requirements that must be met by the component materials. For long-term operation, the seal must maintain an electrically insulating, gas-tight bridge between the metal components, usually molybdenum at the positive side and steel at the negative side. Structural integrity requires coefficients of thermal expansion (CTE) for ceramic and metal components to be sufficiently matched to tolerate a temperature range of 750 to 1000 °C for seal formation and room temperature to 450 °C for cell assembly and operation. Additionally, the electrically insulating component must be chemically stable in molten salts that have either high sulfur or lithium activity. We are developing new sealant materials, which are excellent electronic insulators and bond tenaciously to metals (Fe, Ni, Mo) and ceramics (MgO, BN), even after exposure to molten salt containing lithium alloy or iron disulfide at 400–450 °C.

The mechanical strength of the sealant bond to various metals was evaluated metallographically to determine adhesion and wetting angles. Three substrate materials were examined: molybdenum, steel, and TiN-coated steel. In general, good-to-excellent wetting (i.e., wetting angles from 25° to <10°) was observed. Sealant coverage was generally >95% of the available area. Metallographic examination of the sealant/substrate interface indicated excellent wetting on steel, with wetting angles approaching 0°. Our efforts to improve sealing and bonding onto molybdenum have been quite successful. Initially, the sealant exhibited good wetting (25° wetting angle) on molybdenum, but the sealant tended to break away. A modified sealant displayed improved wetting onto molybdenum with better adhesion. The modified sealant has a coefficient of thermal expansion of about $5.0 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$, which approximates that of molybdenum.

To verify the good performance of hermetic seals formed from the new materials, we tested seals in Li-alloy/U.P. FeS₂ monopolar and bipolar cell designs. Chemical stability of the sealant materials was verified by post-test X-ray diffraction and differential thermal analysis. A feedthrough seal in a monopolar cell was successfully tested over 1000 h and 100 cycles at 400 °C. During this time, the seal maintained a resistance greater than 100 k Ω , which is considered excellent for FeS₂ monopolar cell applications.

In our bipolar cell development, the hermetic seal is formed at the periphery prior to cell assembly⁷ (Fig. 4). Here, a ceramic ring is sealed to molybdenum on one side to form the FeS₂ electrode housing, and a steel assembly is sealed to the other side of the ring to form the Li-alloy electrode housing. Cold-pressed pellets of electrodes and separator materials make up the cell. In tests of the hermetic peripheral seal, a LiAl + 10 mol % Li₅Al₅Fe₂/U.P. FeS₂ bipolar cell (3-cm dia, 0.25-Ah capacity) has been operated over 175 cycles and 400 h and has exhibited high performance and >99% coulombic efficiency. Following 50 short discharge cycles, the U.P. FeS₂ electrode attained >80% utilization over the next 125 deep discharge cycles. Cell capacity was limited by the negative electrode capacity. Following voluntary termination, post-test analysis of the cell confirmed

the stability of the metal-to-ceramic seals after 400 h of operation.

The peripheral seal has been continually improved with over two dozen sealed bipolar FeS₂ cells tested thus far. With emphasis upon ceramics research and seal assembly procedure, a graded CTE seal has been developed. As a result, coefficients of thermal expansion for the metal/ceramic interfaces have been approximately matched for both steel and molybdenum housings. Six improved seals for the bipolar cell (2.5-cm ID) were prepared with similar ceramic compositions and processing methods. One seal was successfully vacuum leak checked at a vacuum of 100 μm , which was the limit of the test fixture capability. Another seal was used to build a sealed bipolar Li-alloy/FeS₂ cell, SBPN-14, which has operated over 450 cycles and 2000 h. Cell SBPN-14 has retained at least 90% of its initial capacity. Its ASI value of 0.55 ohm cm² indicates potential for outstanding power performance for the sealed bipolar Li-alloy/FeS₂ battery. The remaining four seals were used to fabricate sealed bipolar Li-alloy/FeS₂ cells for a four-cell stack.

The outstanding performance of our bipolar FeS₂ cells is indicative of the benefits of the uniform current distribution across the surface of the electrode and the low internal impedance afforded by the bipolar cell configuration. The observed bipolar cell impedance of 0.55 to 0.65 $\Omega \text{ cm}^2$ at 400 °C is only about 50% of that observed in a monopolar prismatic cell. Further, the weight contribution of hardware to the cell is minimized by the bipolar design. Active material thus accounts for about 50% of cell weight for the bipolar cell, compared to about 30% for the monopolar prismatic FeS₂ cell. Calculations based on our measured values of electrode performance and component weight breakdown for a prototype bipolar cell (75 to 150 Ah capacity) indicate that the bipolar configuration should improve specific energy by 30% and specific power by more than 100%. Additionally, we expect that the uniform current distribution present in the bipolar battery configuration will extend the cycle-life from 1000 to 2000 cycles.

The four-cell stack of sealed bipolar Li-alloy/FeS₂ cells (5-cm² cells) is now under test and has exceeded more than 150 cycles and 1300 h of operation. Bipolar stack capacity (0.45 Ah) is at least 90% that of individual cells. The stack is operated with a charge and discharge cutoff voltage of 8.2 and 5.4 V, respectively. As shown in Fig. 5, the bipolar Li-alloy/FeS₂ stack at cycles 100 and 101 is operated with 4-h charge and 2-h discharge rates. Development of a suitable method for integrating cells into a stack is a future task. This initial bipolar cell stack was assembled from four individual cells stacked with nickel felt pads and voltage leads between the cells. A special Al₂O₃ tube was prepared to align the cells in the stack and support the voltage taps. The average stack discharge voltage of approximately 6.5 V greatly exceeds the decomposition voltage of the molten salt electrolyte at 3.2 V. Thus, this stack demonstration served as an excellent test of our seal technology. A special cyclor was built to monitor the individual cells in the stack and determine the efficacy of the overcharge tolerance for the Li-alloy/FeS₂ cells. The stack attained >90% utilization of theoretical capacity at the 4-h discharge rate. The stack impedance at 0.7 ohm cm² of cell area is within 130% of that of individual cells, in spite of the added intercell contact resistance. Improvements in stack integration may reduce stack impedance to less than that of cells, because of greater uniformity in intercell contact.

A substitute for the molybdenum current collector in the FeS_2 electrode is of interest for the electric vehicle application. Such a component would lower battery materials cost and probably simplify seal fabrication for a steel/ceramic/steel configuration. A TiN coating or Mo coating on a low-cost steel substrate has prospects for the FeS_2 current collector. The simple configuration of a bipolar current collector further improves application prospects. Samples of TiN-coated (by PVD) stainless steel were obtained from Richter Precision Inc., East Petersburg, PA. Pore-free coatings were identified from out-of-cell corrosion tests. However, an apparent mismatch in CTE between the TiN coating and stainless steel resulted in coating cracks at the edge of the sample. We are currently testing additional samples of a four-ply TiN-coating on substrates which have a CTE approximating that of TiN (e.g., steel, nickel, Hastalloy B). Initial results are encouraging, with TiN-coated steel samples exceeding 200 h of corrosion testing.

BIPOLAR BATTERY DESIGN

Prototype bipolar Li-alloy/ FeS_2 battery development is expected to proceed with testing of 15-cm dia cells. These cells have a capacity of 75 to 150 Ah. A 22-cell stack would provide 36-V modules, which would be series-connected in one housing for the full battery voltage, i.e., 220 V. As shown in Fig. 6, the battery housing includes a vacuum/multifoil insulated case. This case technology is the outgrowth of ongoing Li-alloy/FeS battery development.⁸ An electric vehicle propulsion system may consist of three of the batteries in parallel and remain below weight and volume limitations. Generally, larger batteries are required to attain the power capability necessary for vehicle acceleration, with specific power to energy ratio (SpP/SpE) at about one. For the bipolar battery design, electrode thicknesses can be adjusted to achieve the desired SpP/SpE. A projected SpP/SpE of about three for the bipolar Li-alloy/ FeS_2 battery would provide sufficient power such that the vehicle could be operated from just one of three parallel-connected batteries. This is an interesting option for advanced electric vehicle design.

An outstanding feature of the bipolar Li-Alloy/ FeS_2 battery is its high energy density, about 500 Wh/L. Cell density is at about 3.0 g/cm³, compared to 1.0-1.5 for Na/ β -alumina-type cells. The monoblock construction (a single weldment) afforded by the bipolar battery design reduces volume, as well as weight due to battery cases and other hardware. As shown in Fig. 6, the sealed bipolar Li-alloy/ FeS_2 battery is designed with almost no free volume internally. The intercell connections that normally extend battery volume are internal to the cell stack. Whereas thermal management requirements for most battery systems exclude a monoblock design approach, the Li-alloy/U.P. FeS_2 battery possesses a significant entropic cooling on discharge to aid thermal management.⁹ Therefore, in this system, entropic heating occurring during battery charging can be dealt with passively by controlling charge rate or actively (by a cooling blower) that is part of an auxiliary charging system. Because of the entropic cooling during discharge and significantly reduced internal resistance of the bipolar battery, the battery operates at near isothermal conditions even with high power demands for electric vehicle acceleration.

Performance projections for a five-passenger electric car have been made. Assuming a bipolar battery specific energy of 191 Wh/kg (3-h rate) and specific power (19 sec pulse) of 500

W/kg at 80% DOD with comparable weight limitations, the bipolar Li-alloy/ FeS_2 could provide a 400 km range, about double the vehicle range of a Na/S battery powered car (Fig. 7). Due to the high density of the Li-alloy/ FeS_2 cell, a much greater portion of its battery weight is energy-storage cell weight. This compactness enhances battery power as well. Since the bipolar battery configuration also minimizes internal power losses, such as intercell connection resistance, its high specific power even at 80% DOD offers vehicle acceleration comparable to that of the internal-combustion gas engine (Fig. 8). Again, its flat cell impedance vs. discharge capacity curve enables excellent vehicle acceleration through the vehicle range.

CONCLUSION

Development of a bipolar Li-alloy/ FeS_2 battery based on our innovative design of hermetically sealed cell is progressing. Tests of new ceramic materials under development for forming the metal-to-ceramic bonds of the bipolar peripheral seal indicate long-term stability. Ceramic compositions have been formulated to match their CTE with that of mating metal components. Sealed bipolar cells have exceeded 450 cycles and 2000 h of operation. The high performance of these bipolar FeS_2 cells reflects the improvement expected from uniform current distribution and low internal impedance, 0.55 to 0.65 Ω cm². Currently, a four-cell bipolar stack (6.5 V) has operated over 150 cycles and 1000 hours with performance at least 90% of cell-level performance.

The bipolar Li-alloy/ FeS_2 battery has unusually high energy density, about 500 Wh/L. A high degree of compactness is possible without adversely affecting battery thermal management. A strong entropic cooling effect of the U.P. FeS_2 electrode in discharge reduces the requirements for active thermal management. High energy density of the bipolar cells enables battery performance to be >80% of that achieved by individual cells, which is considerably higher than that for Na/ β -alumina-type batteries. The prospective electric vehicle performance with a bipolar Li-alloy/ FeS_2 battery is impressive: vehicle acceleration comparable to that of a gasoline engine car and vehicle range of 400 km.

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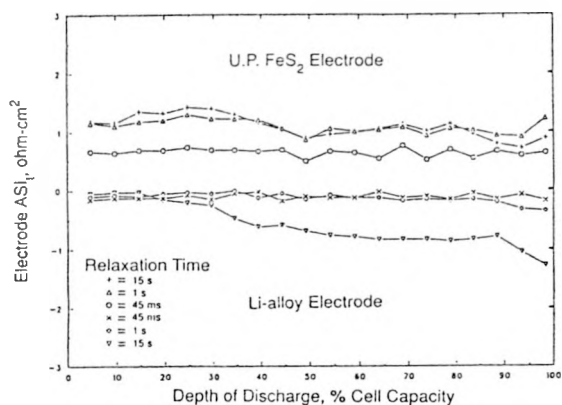


Fig. 1. Half-Cell ASI Measurements of Electrolyte-Starved U.P. FeS₂ Cell (no additives) Having LiCl-rich LiCl-LiBr-KBr at 425 °C.

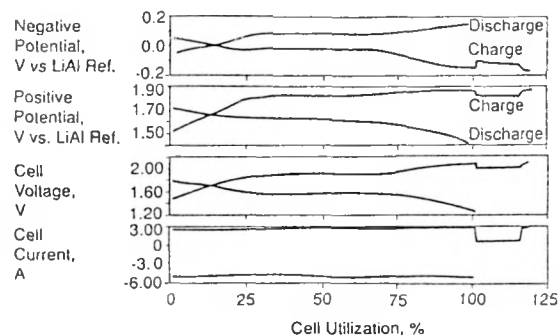


Fig. 2. Overcharge Tolerance of U.P. FeS₂ Cell Operated at 425 °C and Overcharged by 15% at 3 mA/cm². No polarization of U.P. FeS₂ (positive) electrode indicates overcharge safeguard.

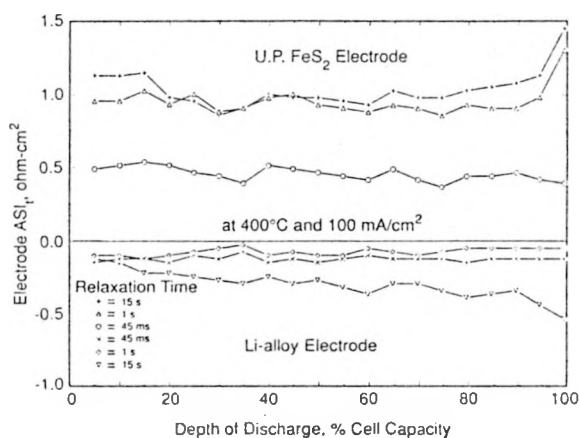


Fig. 3. Half-Cell Impedance Measurements of Electrolyte-Starved U.P. FeS₂ Cell Operated at 400 °C and 100 mA/cm².

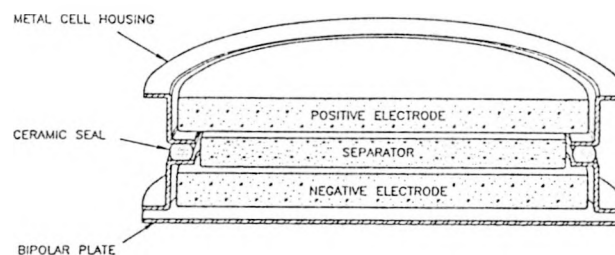


Fig. 4. Sealed Bipolar Cell Design for Li-Alloy/FeS_x Battery. Seal is Formed Prior to Cell Assembly.

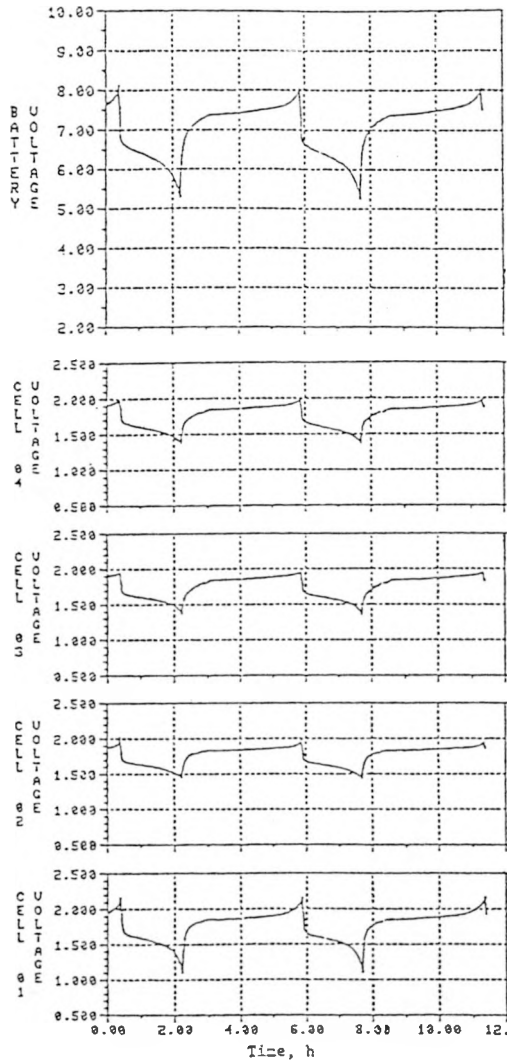


Fig. 5. Operating Characteristics of Sealed Bipolar Li-alloy/ FeS_2 Battery Stack (Voltage in volts) vs. Time at Cycle 100 and 101.

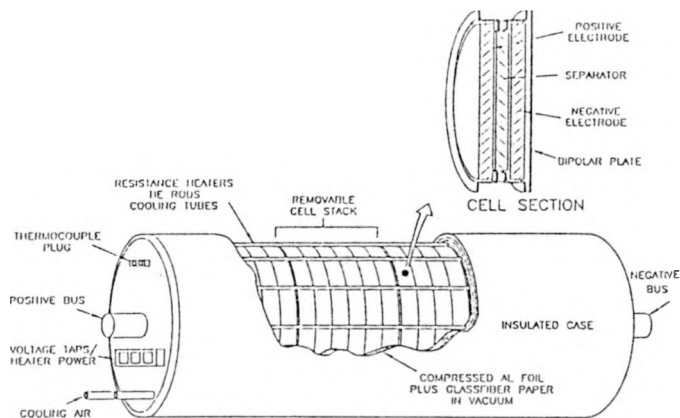


Fig. 6. Schematic of Li/ FeS_2 Bipolar Battery within a Vacuum/Multifoil Insulated Housing.

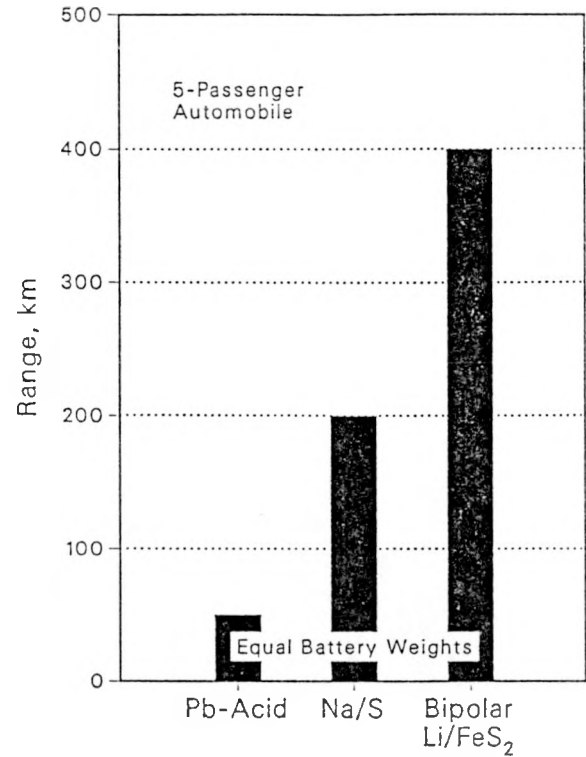


Fig. 7. Projected Vehicle Range for a 300 kg Battery.

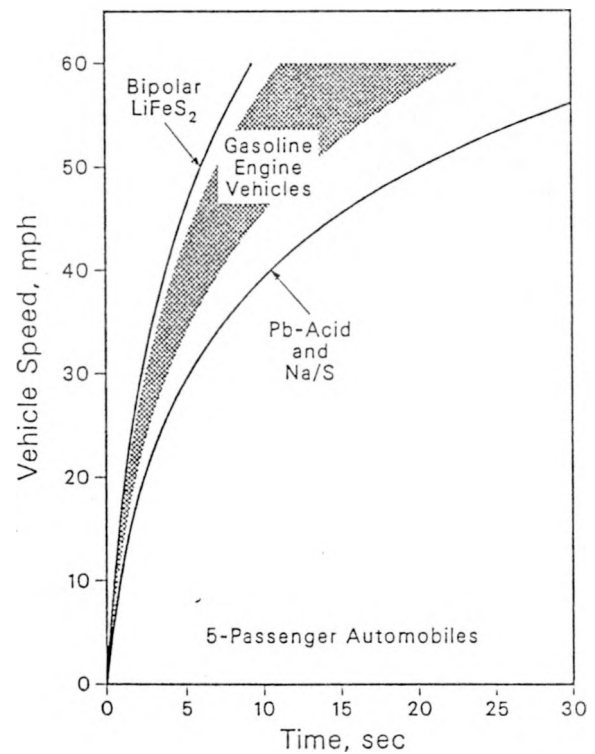


Fig. 8. Projected Vehicle Acceleration with Prospective Electric Car Batteries at 80% Depth-of-Discharge.