

Lithium–aluminum/iron sulfide batteries*

G.L. Henriksen and D.R. Vissers

Electrochemical Technology Program, Chemical Technology Division, Argonne National Laboratory, 9700 South Cass Avenue, Argonne, IL 60439 (USA)

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Abstract

Lithium-alloy/metal sulfide batteries have been under development at Argonne National Laboratory since 1972. ANL's technology employs a two-phase Li alloy negative electrode, low-melting point LiCl-rich LiCl–LiBr–KBr molten salt electrolyte, and either an FeS or an upper-plateau (UP) FeS₂ positive electrode. These components are assembled in an 'electrolyte-starved' bipolar cell configuration. Use of the multi-phase Li alloy (($\alpha + \beta$)-Li–Al and Li₅Al₅Fe₂) negative electrode provides *in situ* overcharge tolerance that renders the bipolar design viable. Employing LiCl-rich LiCl–LiBr–KBr electrolyte in 'electrolyte-starved' cells achieves low-burdened cells that possess low area-specific impedance, comparable with that of flooded cells using LiCl–LiBr–KBr eutectic electrolyte. The combination of dense UP FeS₂ electrodes and low-melting electrolyte produces a stable and reversible couple, achieving over 1000 cycles in flooded cells, with high power capabilities. In addition, a new class of stable chalcogenide ceramic/sealant materials was developed. These materials produce high-strength bonds between a variety of metals and ceramics, which make fabrication of lithium/iron sulfide bipolar stacks practical. Bipolar Li–Al/FeS and Li–Al/FeS₂ cells and four-cell stacks using these seals have been built and tested for electric vehicle (EV) applications. When cell performance characteristics are used to model full-scale EV and hybrid vehicle (HV) batteries, they are projected to meet or exceed the performance requirements for a large variety of EV and HV applications. In 1992, the US Advanced Battery Consortium awarded contracts to ANL and SAFT America to continue the development of the bipolar Li–Al/FeS₂ battery to meet their long-term criteria. Both ANL and SAFT are working together to refine this technology for EV applications and scale it up to larger stacks and fully integrated battery modules.

Operating principles

The development of high temperature cells containing lithium and sulfur was initiated at Argonne National Laboratory (ANL) in the late 1960s [1, 2]. Efforts to develop cells containing liquid lithium, elemental sulfur, and molten salt electrolytes were abandoned in the early 1970s because of the difficulties associated with controlling and containing the active materials of both electrodes [2, 3]. Solid lithium–aluminum alloy anodes and solid metal sulfide cathodes replaced the lithium and sulfur-active materials in 1972–1973 [4–6]. A variety of metal sulfides were examined: copper, iron,

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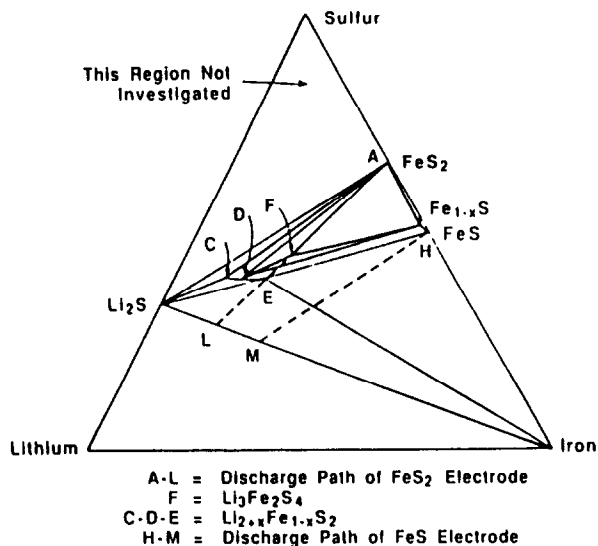


Fig. 1. Phases in the Li-Fe-S system at 450 °C.

result in enhanced reversibility for the FeS₂ electrode, especially when operating in the 400–450 °C temperature range with the LiCl–LiBr–KBr electrolyte.

Complete discharge of an FeS electrode is relatively simple in an all-lithium-ion electrolyte and involves only one intermediate phase:



When electrolytes containing significant levels of potassium ions are used, LiK₆Fe₂₄S₂₆Cl (J-phase) tends to form via a series of chemical reactions [12]. The presence of J-phase appears to hinder the kinetics of the FeS electrode. The extent of J-phase formation is dependent on the quantity of K⁺ in the electrolyte, and a change from eutectic to lithium-rich electrolyte compositions, for either the LiCl–KCl or the LiCl–LiBr–KBr electrolyte, eliminates or significantly suppresses J-phase formation.

Lithium–aluminum alloys have been extensively studied as negative electrode materials. A phase diagram for this alloy system is provided in Fig. 2 [13]. The (α + β)-region of the phase diagram covers the compositional range from 9 to 47 at.% lithium. This two-phase alloy is within 300 mV of the pure lithium metal potential, but remains solid at cell-operating temperatures (400–450 °C). The theoretical capacity of the 47 at.% lithium alloy (within the (α + β)-region) is 0.74 Ah/g. As discussed later, other alloys are mixed with the (α + β)-Li–Al to achieve a negative electrode that provides *in situ* overcharge tolerance to Li–Al/FeS_x cells.

Two types of separator systems have been used successfully in this battery. The initial choice was boron nitride fiber, used either in the form of a woven fabric or non-woven felt [14, 15]. This material works well as a separator, but is fairly expensive. Also, as overcharge tolerance alloys, which have higher lithium activity, were introduced into cells, the chemical stability of boron nitride became a concern. In recent years magnesia powder has replaced boron nitride [14–17]. The magnesia powder is intimately mixed with electrolyte powder and cold pressed to form MgO/electrolyte plaques. The plaques are suitable for use only in electrolyte-starved cells, where the electrolyte

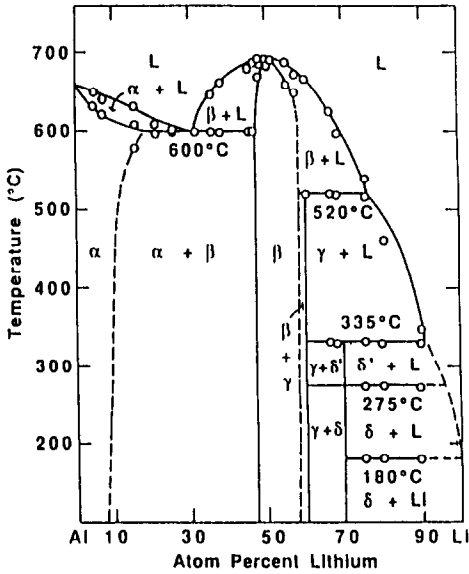


Fig. 2. Lithium-aluminum phase diagram.

content of the plaque is 70 to 80 vol.%. Magnesia has been shown to be stable in the presence of the higher lithium activity.

During normal operation of Li-Al/FeS_x cells, the coulombic efficiency is generally quite high (>97%), and the corresponding self-discharge rate is 0.1 to 0.2 mA/cm². Self-discharge likely occurs by the transport of species containing lithium metal, dissolved in the electrolyte, across the separator and chemical reaction at the FeS_x electrode. For a given lithium activity, this rate is dependent on the electrolyte, operating temperature, separator thickness, and structure of the porous separator.

Due to the high ionic conductivities of molten salt electrolytes (1 to 3 S/cm) and the fast electrode kinetics at these temperatures, Li-Al/FeS_x cells can achieve high voltaic efficiencies, while operating at relatively high current densities. For these reasons, Li-Al/FeS_x batteries are capable of high power densities, even at relatively high depths-of-discharge (DODs).

One concern with high temperature batteries is life. Both the Li-Al/FeS and UP Li-Al/FeS₂ chemistries have exhibited >1000 cycles of life. Therefore, electrode reversibility and stability have been demonstrated. Also, chemical and electrochemical stability tests have shown that the non-active materials used in fabricating state-of-the-art cells are sufficiently stable to provide ten years of calendar life.

In the area of battery reliability, the Li-Al/FeS_x technologies possess attractive characteristics. Both Li-Al/FeS and Li-Al/FeS₂ cells have a history of consistently developing low-resistance internal short circuits upon reaching end-of-life, even when end-of-life occurs prematurely. This feature allows the design of battery systems containing long strings of series-connected cells and the parallel connection of these strings at full battery voltages. This is possible because all other series-connected cells can continue to operate normally while the charge and discharge current passes through prematurely failed cells. In the case of Li-Al/FeS, a 36-V 20-kWh battery, containing prismatic cells, remained operational even after 44% of its cells reached end-of-life

[18]. Less information is available on the reliability of Li–Al/FeS₂ batteries, but four-cell stacks have continued to operate after half of the cells failed.

Another typical concern with high-temperature batteries is safety. Although extensive safety tests have not been conducted on the Li–Al/FeS_x technologies, ANL has a 20-year history, working with a variety of industrial partners, in the development of these technologies without experiencing any significant safety problems [19]. One of the most dramatic tests was an unplanned event that occurred with a 20-kWh Li–Al/FeS prismatic battery. This battery developed an internal short circuit and subsequently discharged all of its stored energy in a 20-min period. Although significant internal heating occurred, the battery skin temperature only reached 130 °C, and the case was not breached. In planned safety tests, unprotected individual prismatic Li–Al/FeS cells were held at operating temperature while being subjected to a drop test that simulated a 30-mph (49-km/h) collision. Although the cell cases breached and their hot contents spewed out on the ground, no explosion, fire, or release of noxious gas occurred.

The excellent safety records for Li–Al/FeS_x technologies likely result from the inherent safety characteristics of these systems. For both systems, the active materials and the reaction products are solids – not liquids or gases – and are therefore more readily contained upon breaching the cell case. Secondly, all the solid reactants are intimately coated with molten salt electrolyte. This coating provides a protective film that prevents violent reactions with atmospheric oxygen or water vapor upon breaching of the cell case. Also, because these are high-temperature batteries, the battery cases will be engineered to provide a high degree of thermal insulation and thereby will provide an additional degree of protection from mechanical abuse as well.

Current status

In the late 1970s and during the 1980s, prismatic Li–Al/FeS cells and batteries were under development by ANL and several industrial firms for electric van and utility load-leveling applications. A cut-away view of a prismatic Li–Al/FeS cell is provided in Fig. 3. The design is similar to conventional lead/acid prismatic cells, which employ flat-plate electrodes that are parallel-connected with internal overhead bus bars to achieve the desired cell electrode area for meeting the power requirements of the application. Stored energy per cell is controlled by adjusting electrode thickness. Electrode separation was maintained using boron nitride felt – a flexible porous ceramic cloth that was chemically and electrochemically stable to the cell environment. Full-size cells of this type for electric van applications were developed and evaluated in 36-V 20-kWh battery packs [18] (see Fig. 4). As part of this effort, fairly sophisticated thermal enclosures were developed. These thermal enclosures employed alternative layers of aluminum and glass fiber paper, compressed between an inner and outer steel wall. The inner and outer steel walls were joined to form a sealed annulus, which was evacuated to produce a vacuum-insulated jacket. Batteries of this type could be engineered to meet the energy and power requirements of electric van applications, but the design was not suitable for higher power/energy (P/E) ratio electric-vehicle (EV) or hybrid-vehicle (HV) applications.

In parallel, a low-level R&D program was being conducted at ANL to develop the core technology for a higher performance Li–Al/FeS₂ battery. The major developments emanating from this program are summarized in Table 1. Achievement of more than 1000 cycles life in 1986 on flooded-electrolyte prismatic cells employing a dense UP cathode was a major breakthrough in that prior Li–Al/FeS₂ cells lost significant capacity over the first 100 cycles of operation. It is believed that the electrolyte, lower operating

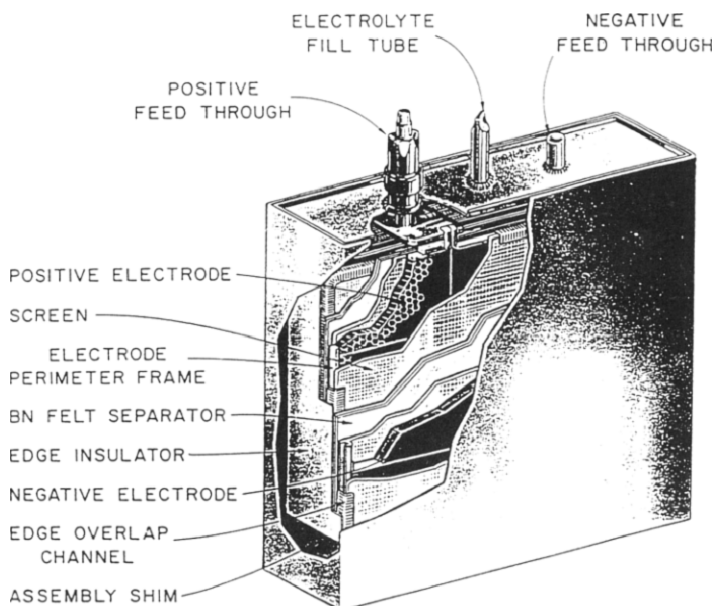


Fig. 3. Cut-away view of multi-plate prismatic Li-Al/FeS cells.

temperature, dense cathode, and upper voltage plateau operation all contributed to the enhanced reversibility and stability of the FeS_2 electrode.

During the late 1980s, several approaches for achieving a stepwise enhancement of the negative electrode lithium activity were studied as methods for dramatically increasing the self-discharge rate near the end of charge. As described earlier, self-discharge occurs by the transport of dissolved lithium across the separator, where it reacts chemically with the iron sulfide active material. A significant increase in this self-discharge reaction, caused by the increased lithium activity, would permit continued charging of a fully charged cell, at or below this enhanced self-discharge rate, without overcharging and damaging the cell. This stepwise increase in the self-discharge rate provides a degree of overcharge tolerance that can be used for balancing cells in a series-connected array of cells. This was successfully demonstrated in 1988. State-of-the-art cells utilize Al_3Fe_2 as an additive to the negative electrode [12]. As the $(\alpha + \beta)$ -Li-Al alloy begins to undergo transition to the β -alloy during the later stages of charging, the Al_3Fe_2 becomes involved electrochemically, producing Li- Al_3Fe_2 solid alloys that possess lithium activities which provide a 20-fold increase in the self-discharge rate of the cell. This permits extended charging at rates of 2 to 3 mA/cm^2 without overcharging fully charged cells, while cells at lower states-of-charge continue to approach the fully charged state. This development allowed serious consideration of bipolar battery designs for both the Li-Al/FeS and Li-Al/FeS₂ technologies.

The higher lithium activity of the overcharge tolerant alloy, however, prohibits the use of BN separators. While MgO separators appear to be sufficiently stable in the presence of this higher lithium activity, they require the use of an electrolyte starved cell configuration. The development of a lithium-rich LiCl-LiBr-KBr electrolyte in 1989 resulted in an electrolyte of higher conductivity and the ability to achieve cell impedance and power levels comparable with those of flooded cells employing the LiCl-LiBr-KBr eutectic electrolyte.

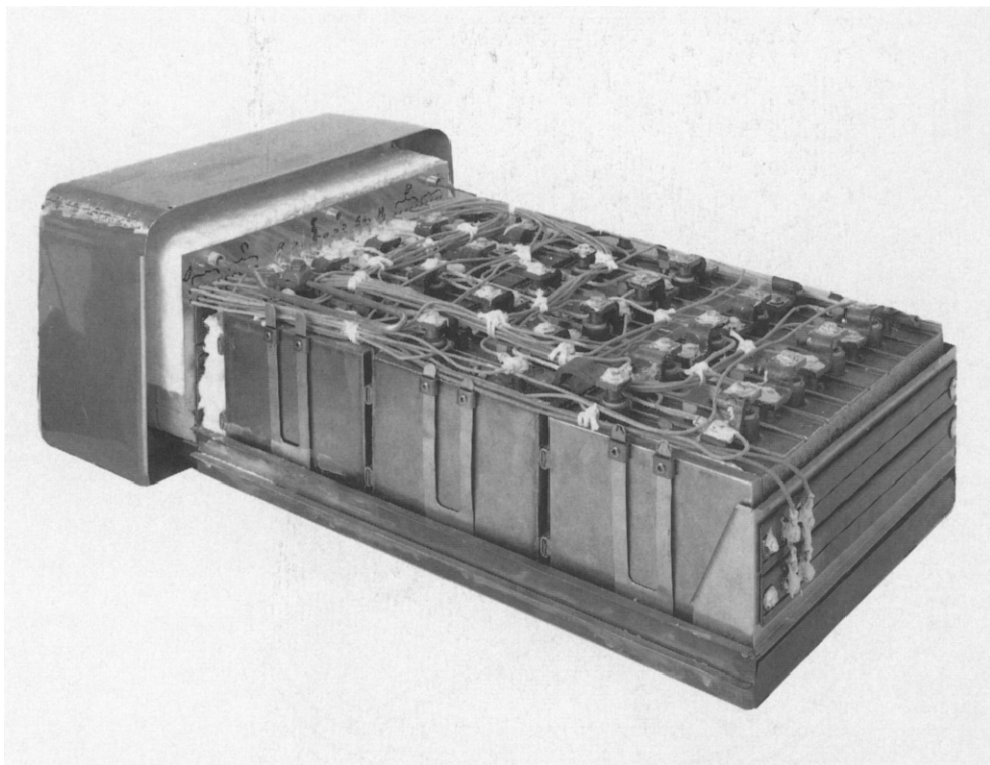


Fig. 4. 36-V 20-kWh battery module comprised of 27 prismatic cells.

TABLE 1

Major technical advances in the development of bipolar Li-Al/FeS₂ cells

Time frame (Year)	Major technical advance	Practical implication
1986	Low-temperature electrolyte and upper plateau dense FeS ₂ cathode	Achieves >1000 cycles
1988	Electrochemical overcharge tolerance	Makes bipolar design viable
1989	Lithium-rich electrolyte in starved cell	Enhances performance
1990	Chalcogenide seal material	Makes bipolar design practical

The development that resulted in a switch from prismatic cells to bipolar cells occurred in 1990. A new class of ceramic materials, which can be engineered to be either electrical insulators or conductors, were identified. These materials exhibit strong and chemically stable bonds to a variety of materials [20]: ceramics, metals, graphite, etc. Since their discovery in 1990, these chalcogenide-based sealant materials have been engineered to seal the periphery of 3-cm cells and then were successfully scaled up to 13-cm diameter bipolar cells and stacks. An exploded view of a four-cell bipolar stack is provided in Fig. 5. Typical performance data from 13-cm diameter bipolar cells are provided in Table 2.

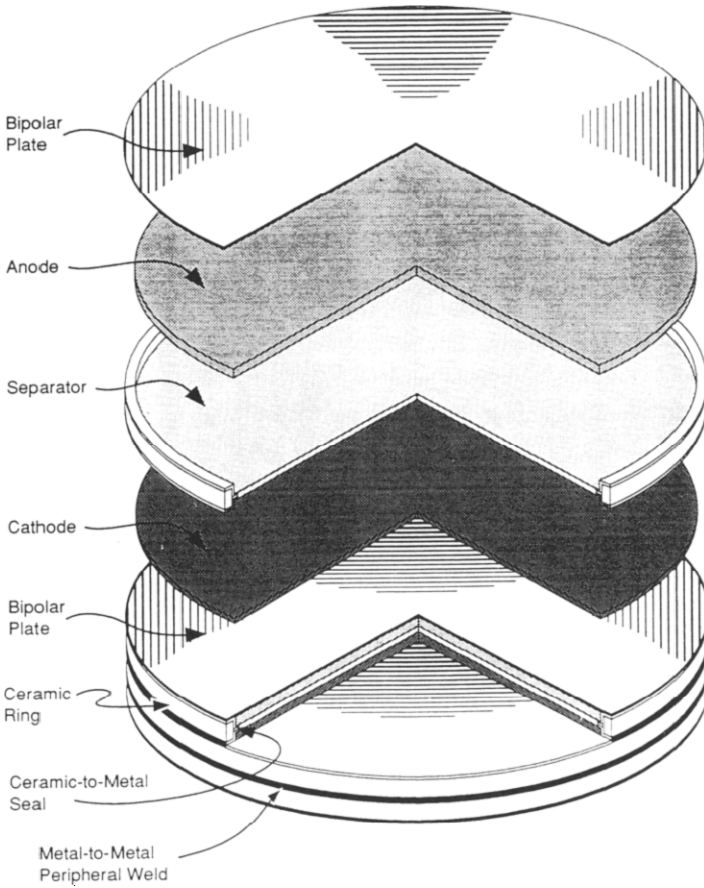


Fig. 5. Exploded view of four-cell bipolar Li-Al/FeS_x stack with MgO separator.

TABLE 2

Specific energy and power of 13-cm diameter bipolar cells

Cell technology	Specific energy (Wh/kg at W/kg)	Specific power at 80% DOD ^a (W/kg)
Li-Al/FeS	130 at 25	240
Li-Al/FeS ₂	180 at 30	400

^aDOD = depth-of-discharge.

In 1992, the US Advanced Battery Consortium (USABC) awarded separate contracts to ANL and SAFT America to work jointly on developing the bipolar Li-Al/FeS₂ battery technology to meet their long-term criteria. Their mid-term and long-term criteria for advanced batteries are summarized in Tables 3 and 4. The R&D projects at SAFT and ANL are being conducted in an integrated manner. Argonne's technology was transferred to SAFT during the first phase of the project, and the two groups are currently working together to advance the technology and scale up the hardware

TABLE 3

US Advanced Battery Consortium primary criteria for mid-term and long-term advanced battery technologies

Primary criteria	Mid-term	Long-term
Power density (W/l)	250	600
Specific power at 80% DOD/30 s (W/kg)	150 (200 desired)	400
Energy density at C/3 discharge rate (Wh/l)	135	300
Specific energy at C/3 discharge rate (Wh/kg)	80 (100 desired)	200
Life (years)	5	10
Cycle life at 80% DOD (cycles)	600	1000
Power and capacity degradation (% of rated spec)	20	20
Ultimate price (US \$/kWh) (10000 units at 40 kWh)	< 150	< 100
Operating environment (°C)	- 30 to 65	- 40 to 85
Recharge time (h)	< 6	3 to 6
Continuous discharge in 1 h (no failure)	75% (of rated energy capacity)	75% (of rated energy capacity)

to large stacks and fully integrated modules. The SAFT contract value is US \$17.3 million, while the ANL contract value is US \$7.3 million. Both contracts run through December 1995.

Technical challenges

The basic technology appears to be sound. Although room for further refinements and improvements exist in many areas, the real challenges are associated with: (i) scaling up the technology to larger stacks and fully integrated battery modules; (ii) reducing component costs; (iii) establishing adequate materials' supplies, and (iv) converting laboratory-scale fabrication processes to effective and efficient pilot-scale processes. Meanwhile, it is essential to implement quality control throughout the production process to ensure a reproducible product of adequate quality.

The technology demonstrated by ANL in 1991-1992 employed specialty materials and laboratory-scale processes. One of the major challenges is to identify or develop suppliers that can provide acceptable materials in the quantities needed for hardware scale-up and demonstration, as well as for future commercialization. In some instances, it may be necessary to develop new processes for maintaining adequate purity and/

TABLE 4

US Advanced Battery Consortium secondary criteria for mid-term and long-term advanced battery technologies

Secondary criteria	Mid-term	Long-term
Efficiency (%) C/3 discharge 6-h charge	75	80
Self-discharge	<15% in 48 h	<15% per month
Maintenance	no maintenance; service by qualified personnel only	no maintenance; service by qualified personnel only
Thermal loss (for high temperature batteries)	3.2 W/kWh; 15% of capacity in 48-h period	3.2 W/kWh; 15% of capacity in 48-h period
Abuse resistance	tolerant; minimized by on-board controls	tolerant; minimized by on-board controls

or reducing cost, as the demand increases. Also, much of the original processing was developed at the laboratory scale for implementation in a controlled atmosphere glove box. These processes need to be reviewed with regards to effective scale-up for pilot-plant manufacturing. It is anticipated that some operations can be transferred from an argon glove box environment to a dry room environment, while other operations may need to be scaled up within a more controlled environment.

Another major challenge is to scale up to larger hardware without experiencing significant performance or life degradation. Some of the key areas to monitor with these battery systems are: (i) quality of the peripheral seal; (ii) temperature variations between cells and modules, and (iii) mechanical loading of the cell stacks. It is important to retain the electrolyte within each of the individual sealed cells. Although some gas permeability appears acceptable, electrolyte permeability could lead to current shunting external to series-connected cells within a module enclosure and loss of efficiency and performance. Therefore, the peripheral seals must be of sufficient quality to prevent electrolyte permeability. Use of starved-electrolyte cells is an advantage here, but the seals must still be impermeable to the electrolyte. In the area of minimizing temperature variations between cells and modules, this is a challenge that lends itself to an engineering solution. The real challenge with thermal management is to keep it simple and inexpensive. The degree of sophistication required for the thermal management subsystem will be dictated, to some extent, by the requirement set for the maximum heat rejection rate. For the Li-Al/FeS₂ battery, this rate is established by the maximum recharge rate.

A third challenge is to reduce some of the component costs of the system. State-of-the-art Li-Al/FeS₂ cells employ molybdenum as the current collectors for the FeS₂ electrodes. The switch from prismatic to bipolar cells significantly reduced the amount of molybdenum per kWh of stored energy, but the thin-sheet molybdenum used as the bipolar plate remains a high-cost component. Cost reductions can be achieved in several ways: (i) redesign to further reduce molybdenum in cells; (ii) develop a lower-

cost process for producing thin-sheet molybdenum, and/or (iii) develop a lower-cost substitute material.

The challenges associated with successfully developing a bipolar Li-Al/FeS battery are quite similar. However, the component cost issue for molybdenum is eliminated, because a steel or stainless-steel bipolar plate would be adequate for use with FeS. On the other hand, FeS is not a naturally occurring mineral, like FeS₂, so it is likely to have a higher cost. Also, more emphasis should be directed at enhancing the power capability of the Li-Al/FeS system at >80% depth-of-discharge (DOD).

Commercial impact

The principal market for bipolar Li-Al/FeS_x batteries is high-performance EVs beyond the year 2000. Other potential applications include HVs, specialty battery applications (e.g., pulsed power for military uses), and stationary energy storage (e.g., load leveling). The US Department of Defense supported the development of bipolar Li-alloy/metal sulfide batteries for use in pulse-power applications [21, 22]. That technology is quite similar to one being developed for EV applications and indicates the capability of this technology to be re-engineered for applications that require extremely high P/E ratios. The requirements for a battery in a HV application can vary rather dramatically, as shown by the information in Table 5 [23], but the P/E ratios lie between those of the EV and the pulse-power specialty applications. A dual-mode HV possesses some range capability operating on the battery alone, while the battery in a power assist HV is used to level the power profile of a separate power device (heat-engine, fuel cell, etc.) on the vehicle. Stationary energy storage applications are likely to require P/E ratios in the 1–2 range and, therefore, are similar to the requirements for an electric van.

In 1990, ANL developed a computer-aided battery design and performance model that utilizes laboratory performance data in the design of full-scale bipolar EV batteries, of the type illustrated in Fig. 6 [12]. The model accounts for the weight and volume of every component needed to produce a fully integrated battery and thereby establishes final dimensions, as well as the total weight and volume for each full-scale battery. Using these weights, volumes, and laboratory performance levels, this model permits the evaluation of alternative packaging concepts. Figure 7 provides a projected battery performance band for bipolar Li-Al/FeS₂ batteries. The Figure plots battery specific energy as a function of specific acceleration power. The circles enclosed by the shaded area are performance levels projected by the model for batteries designed to meet various P/E ratios, using different component designs and battery packaging concepts.

TABLE 5

General Motors' requirements for energy storage devices in two types of hybrid vehicles

Parameter	Hybrid vehicle type	
	Power-assist	Dual-mode
Energy (kWh)	0.5–2.0	6–8
Power (kW)	50–70	100
Weight (kg)	22.7	136
Life (cycles)	100000	2000
Cost (US \$)	300–500	800

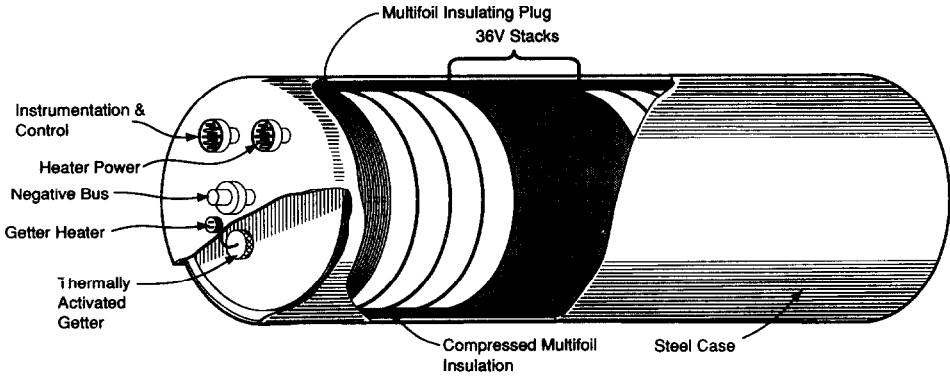


Fig. 6. Cut-away view of bipolar Li-Al/FeS_x integrated battery module with vacuum multi-foil thermal enclosure.

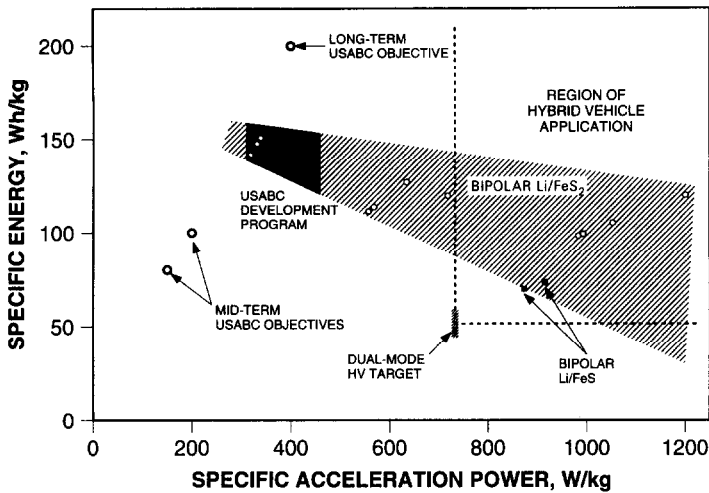


Fig. 7. Projected bipolar Li-Al/FeS_x battery performance for electric-vehicle and hybrid-vehicle applications.

The dark area of the band is the area being addressed by the USABC EV battery development contracts. The dual-mode HV target of General Motors (GM) is identified on the Figure and battery designs with higher specific energy and higher specific acceleration power would meet the GM performance requirements in a lighter weight package than specified. The bipolar Li-Al/FeS₂ battery is projected to readily meet and surpass the dual-mode HV requirements using several packaging concepts. Although somewhat lower in performance, bipolar Li-Al/FeS batteries are also projected to be acceptable for dual-mode HV applications.

The market sizes for EVs and HVs in the years beyond 2000 are impossible to project with any degree of certainty, but the US government is working together with the US automobile industry in the Partnership for New Generation Vehicles (PNGV) to develop personal-use vehicles that possess triple the fuel economy of today's vehicles powered by internal combustion engines. It is highly probably that HVs will play a

major role in approaching or achieving this goal. A battery system with the capability and design flexibility to meet the performance requirements of most EV and many HV applications could have a significant commercial impact on the US and world transportation sectors. Bipolar Li-Al/FeS_x batteries, especially the Li-Al/FeS₂ system, possess these performance capabilities and a high degree of design flexibility. These batteries can be readily repackaged to meet a wide range of P/E ratios and packaging constraints.

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