

Li/SO₂ rechargeable batteries*

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Abstract

Various systems of Li/SO₂ rechargeable batteries are reviewed based on electrolyte salts, types of negative electrodes, and types of electrolytes. Development and progress in these systems are briefly described. Electrolyte properties, electrode problems, and cell performance and features are discussed.

1. Introduction

During the last two decades, intense research and development of high-energy-density battery systems have been conducted by numerous groups throughout the scientific world. These activities were initiated by the needs for military applications, space exploration, electric vehicles, environmental protection, electronic equipment, and consumer-products' miniaturization. As a result of a worldwide search for significantly smaller and lighter (high-energy-density) batteries, the first practical Li/SO₂ primary cells were developed in the early 1970s [1–3]. Since then, the Li/SO₂ system has become the most highly developed primary lithium battery system and for over a decade the one most widely used for military and industrial applications.

Although the use of liquid sulfur dioxide as an electrolyte solvent in batteries with alkali-metal anodes had been discussed in Schasl and McDonald's paper [4] as early as 1948, the actual Li/SO₂ primary battery system was developed by Meyers *et al.* in their 1969 milestone patent [1]. Since then, continuous effort and attention have been given to improving battery safety and performance [5–13]. However, Li/SO₂ rechargeable cells experienced little attention and progress [14–17], even though the rechargeability of the Li/SO₂ system was first suggested by Maricle and Mohns [18] in 1971. The lack of interest and progress was ascribed to difficulties in recharging the Li electrode and the belief [19] that reduction of SO₂ was irreversible due to the insolubility of the Li₂S₂O₄ discharge product in SO₂ electrolytes. This

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situation did not change for almost eight years until Schlaikjer [20], as well as Dey *et al.* [21–23], made advances in the development of new electrolytes that have greatly improved the cycling behavior of the Li electrode in SO₂ electrolytes.

In the early 1980s, progress in selecting the proper electrolyte salts and cosolvents in liquid sulfur dioxide renewed industrial and governmental interest in Li/SO₂ rechargeable cells. For example, Ballard Research Inc. has worked on developing an Li/SO₂ rechargeable system for Amoco (formerly Standard Oil of Indiana). They adopted an electrolyte consisting of LiClO₄ and a supporting electrolyte salt composed of a compound whose cation is selected from a group of metal ion complexes, or the nitrogen or phosphorus organic cations [24, 25]. BTC Engineering Ltd (BTCE) has developed an LiAl/SO₂ rechargeable system for the Canadian Department of National Defence [26]. Duracell International has been involved in the rechargeable Li/SO₂ cells under contracts with the United States' Army and Department of Energy. They employed a highly conductive, liquid solvate (e.g., Li(SO₂)₃AlCl₄) as an electrolyte and a solid positive electrode (e.g., CuCl₂) in their system [27–29]. Various cells based on these types of liquid solvate electrolytes have been evaluated by Duracell [30–43] and by U.S. Army Electronics Technology & Devices Laboratory [44–48] researchers. In addition, Fey *et al.* at National Central University and Chung-Shan Institute of Science and Technology, Taiwan, have focussed on electrolyte properties of LiAlCl₄ and LiGaCl₄ in liquid sulfur dioxide solution at various temperatures and related cycling behavior of Li/SO₂ cells [49–54].

The main components of an Li/SO₂ rechargeable system are a cylindrical, nickel-plated steel case containing an Li foil anode, a cathode, including a current collector comprised of a particularly high surface area material, an electrolyte salt solution with sulfur dioxide as the major cathode depolarizer, and a co-solvent if it is required.

Apart from rechargeability, the main features of the primary and secondary Li/SO₂ batteries are not expected to be much different. Batteries based on the Li/SO₂ primary system have significant advantages over existing conventional batteries [55–59]; for example, high specific energy (300 W h kg⁻¹), high rate capability, long shelf life (5–10 years), high cell voltage (~3 V), excellent voltage regulation, good low temperature performance (at -40 °C with 50% capacity retention), and good capacity retention after storage at elevated temperatures.

A variety of lithium salts such as Li₂B₁₀Cl₁₀ [21, 22, 27], LiClO₄ [24, 25], LiGaCl₄ [21, 22, 27], and LiAlCl₄ [31–33], has been used and studied in various Li/SO₂ rechargeable batteries. A review solely dealing with these Li/SO₂ rechargeable batteries has not yet been reported except for some short, scattered discussions [60–62].

The objective of this paper is to review the work being done on this system in research and development laboratories and to review the electrolyte properties and cell performance of the above-mentioned lithium salts in Li/SO₂ rechargeable cells. In addition, this paper presents our findings on the

stability, rechargeability, redox behavior, and ionic transport phenomenon of the Li/SO₂ cells containing the LiAlCl₄ or LiGaCl₄ electrolyte solution at various temperatures. Due to page limitation, the review will not be exhaustive.

The discussion of rechargeable Li/SO₂ batteries in this review will center around the work developed by Duracell International and Standard Oil of Indiana (Amoco). Also, some comments will be made on the approaches and contributions of the Power Sources Division, U.S. Army Electronics Technology and Devices Laboratory.

2. Li/Li₂B₁₀Cl₁₀, SO₂/C cells

This was the first SO₂-based, all-inorganic, ambient temperature rechargeable lithium battery system developed by Duracell and reported in a 1979 patent by Schlaikjer [20] whose invention has spurred various activities to develop a practical Li/SO₂ rechargeable battery. Dampier and Kalivas at GTE also worked on this system [63].

The system with Li₂B₁₀Cl₁₀/SO₂ electrolyte showed complete reversibility of the SO₂ reduction and no dendrite formation on lithium plating. The cell has an OCV near 2.9 V and good conductivity of 13.8–24.2 mS cm⁻¹ in a 0.60 N solution over a temperature range of -20 to 70 °C [20]. The electrochemical oxidation of B₁₀Cl₁₀²⁻ anions was reported by Bowden [64].

The prototype cell showed poor cycle life, with a drastic decline in cell capacity on cycling due to the formation of an insoluble solid, Li₂S₂O₄, within the cathode pores during a discharge cycle. The saturation of the cathode with the dithionite salt generated large internal stresses in the carbon electrode matrix and caused irreversible damage during cycling. The cell can achieve in excess of 100 cycles on shallow cycling, but only 20–40 cycles on deep cycling [65]. The system was primarily limited by the degradation of the porous carbon electrode and the prohibitively expensive electrolyte salt, Li₂B₁₀Cl₁₀.

3. Duracell's Li/SO₂ rechargeable cells with solvate electrolytes

This is also a totally inorganic, rechargeable, nonaqueous cell system and can be further described in terms of three types:

(a) Cell 3.1 Li/LiAlCl₄:*x*SO₂/C cells

(b) Cell 3.2 Li/LiAlCl₄:*x*SO₂/CuCl₂ cells

(c) Cell 3.3 Li/LiAlCl₄:3SO₂, Cosolvent/C or CuCl₂ cells.

In general, the system contains a lithium anode, an LiAlCl₄:*x*SO₂ (where *x* = 3 or 6) solvate electrolyte with low SO₂ vapor pressure [39], and a microporous Tefzel separator [66]. When CuCl₂ is used, the positive electrode is made of a conductive carbonaceous material having an apparent bulk density of about 80 g l⁻¹ [31] and CuCl₂ accounting for at least 60% of

its weight [32]. The cells have an OCV range between 3.3 and 3.5 V and discharge at about 3.2 V.

The discharge product is not dithionite, but the products are LiCl and an LiClAl(OSO-C)_3 complex which allows the cathodic reaction to be completely reversible [40]. The chemical composition of the Li surface film in the Li/SO₂ cell has been considered to be solely Li₂S₂O₄ [67], although fresh Li surfaces form Li₂O and Li₂S at low levels of SO₂ exposure [68]. However, recent SEM, IR, and XPS results obtained by Abraham and Chaudhri [69] have indicated that the Li surface film in the Li/SO₂ cell is a complex mixture of products including Li₂S and several Li sulfuroxy compounds which may be Li₂S₂O₄, Li₂SO₃, Li₂S₂O₅ and Li₂S_nO₆.

The conductivities of $\text{LiAlCl}_4 \cdot x\text{SO}_2$ electrolytes are very high, about 0.1 S cm⁻¹ at ambient temperatures, which are compatible with those of aqueous electrolytes [39]. Other liquid SO₂ solvates of the tetrachloroaluminates of sodium, calcium, and strontium also show high conductivities. The addition of one of these solvates to LiAlCl₄·3SO₂ can lower the freezing point of the electrolyte considerably. The internal pressure of the electrolyte during discharge was found to increase with discharge [33]. The liquid SO₂ solvates of LiAlCl₄ have been studied recently by IR, AA, ESCA, X-ray fluorescence [70], CV [44, 71], and Raman spectroscopy [72]. The electrolyte provides an overcharge protection mechanism which is based on the oxidation of AlCl₄⁻ followed by the recombination of Cl₂ with Li and the reaction of LiCl with AlCl₃ [41]. However, chlorine was found to react with the separator material, poly(propylene), causing internal cell shorting. Thus, microporous Tefzel is used as a resistant separator material.

Although the solvate electrolyte is highly conductive, Cell 3.1 has been reported to yield low capacities when Shawinigan carbon black was used as cathodes [36]. This is probably due to the low surface area and low conductivity of Shawinigan carbon black. Kuo *et al.* discovered that the capacity of cathodes such as AgCl may be increased several times by the addition of small amounts (e.g., 10–12%) of the carbonaceous material such as Ketjenblack EC [31]. As a result, Cell 3.2, using CuCl₂ as a cathode, was developed by Duracell researchers [41]. Gopikanth recently evaluated several new composite solid cathode materials such as AgCl₂ with CoBr₂ or NiBr₂ for Li/SO₂ rechargeable batteries and found that the energy densities of cells with these composite electrodes, which show no capacity fading, are 8–10 times higher than those obtained by other lithium rechargeable systems [43].

Mammone and Binder at LABCOM have done extensive cyclic voltammetric work on the Li(SO₂)₃AlCl₄ electrolyte [44] and the one containing added halogens [71]. They found that adding Cl₂ to the electrolyte significantly reduces passivation of the cathode during reduction, and that adding soluble Br₂ substantially improves both carbon cathode discharge and charge performance. Further, they studied the cells (Cell 3.3) containing various percentages of pure SO₂Cl₂ and discovered that adding up to 25% pure SO₂Cl₂ to the electrolyte greatly enhances room temperature cell capacity to 50 mA h cm⁻² compared with 10 mA h cm⁻² for baseline cells [73].

The advantages and disadvantages of cells in this system have been listed [74]. Duracell has manufactured practical cells of 2/3A, AA, and C sizes. Prototype 2/3A, AA, and C-size spirally wound, Li/porous carbon cells filled with the $\text{LiAlCl}_4/\text{SO}_2$ electrolyte have been built. Prototype 2/3A-size [38] and C-size [40] wound cells have achieved energy densities of 100 W h kg^{-1} and 134 W h kg^{-1} , respectively.

4. Amoco's Li/SO_2 rechargeable cells with 'second kind' negative electrodes

Amoco made approaches to the problems associated with lithium plating, cell safety, and rechargeability, which were different from Duracell and others. In their patent [24], Prater *et al.* have developed a rechargeable, nonaqueous, ambient-temperature cell using a negative electrode of the second kind (analogous to the Pb/PbSO_4 electrode in the lead-acid battery), which is a metal/passivating film interface where the potential determining ion in solution is in equilibrium with a solid salt phase. Since lithium salt is precipitated on the lithium anode, the morphology changes and lithium dendrite formation by the reduction of lithium cations in solution are thus avoided. The system can deliver about four times the volumetric energy density of either lead-acid or nickel-cadmium at $C/3$ (e.g., sub C prototypes deliver 1.8 A h at the $C/3$ rate) [75].

A specific example is the $\text{Li}/\text{LiClO}_4 + \text{TBAP}-\text{SO}_2/\text{C}$ cell wherein the anode is an electrode of the second kind due to a proper combination of electrolyte components such as a solvent (e.g., SO_2), a supporting electrolyte salt, R^+X^- (e.g., TBAP), and an active metal salt, M^+X^- (e.g., LiClO_4) [24]. A significant feature of this system is its ability to induce the M^+ ion produced during discharge to form a precipitate on the anode, and thereafter to induce the ion to return to metal form during charge. Although high amounts of M^+ ion are required to provide reasonable current densities and charge rates, a high concentration of M^+ in solution can result in asymmetrical deposition of the anode metal during charge (analogous to dendrite formation) [24]. Thus, optimizing the proper concentration levels of M^+ cation and X^- anion is important in order to achieve good cell performance and to avoid shape-change problems.

The system has encountered certain unknown phenomena; so-called 'bleeding' and 'yellowing' effects. 'Bleeding' is probably caused by self-discharge of the anode due to the presence of conducting impurities on the anode surface. The red material streaming from the lithium electrode is believed to be forms of $\text{SO}_2:\text{S}_2\text{O}_4^{2-}$ complexes [24]. 'Yellowing' occurred unpredictably in a liquid sulfur dioxide solution substantially saturated with LiClO_4 and about 0.2 molal TBAP. Additives such as iodine, LiI , LiBr , R_4NX (where R: alkyl; X: halogen) were reported to be stabilizers [25].

5. BTCE's Li(Al)/SO₂/C cells

In an attempt to combine the best features of a lithium alloy and a liquid cathode, BTCE has designed and tested a small, hermetically-sealed metal cell of the LiAl/SO₂ system. The cell consists of a stainless steel can of diameter 2.2 cm, and height 2 cm, containing an LiAl anode (two electrodes in parallel) and a carbon/Teflon cathode (three electrodes in parallel) and a proprietary SO₂-based electrolyte. A Celgard separator was used between the electrodes [76].

The LiAl electrodes were made by either an electrochemical method in 1 M LiAsF₆/PC or electrodeposition of lithium onto the 0.813 mm thick aluminum electrodes. The electrolyte was primarily sulfur dioxide with an organic co-solvent, LiAsF₆, and a special additive. The details of the cell configuration and testing as well as the experimental methods have been described [26, 76].

In spite of the low cycle life, the LiAl/SO₂ system has enjoyed the following advantages: a flat discharge curve with a voltage of 2.4 V, good retention performance at low temperatures, low self-discharge rate, and high anode melting range (600–700 °C). The use of LiAl alloy may not only increase cell safety but may also improve electrolyte stability. However, the system is still in the early developmental stage.

6. Li/LiAlCl₄ or LiGaCl₄, SO₂/C cells (solution type)

In an effort to understand and improve the poor high-temperature storability of LiAlCl₄:xSO₂ solvate electrolytes in Duracell Li/SO₂ rechargeable cells, Fey *et al.* have investigated the electrolyte properties and cell performance of LiAlCl₄ and LiGaCl₄ electrolytes in liquid sulfur dioxide at various temperatures [49–54]. The investigation was necessary due to the facts that lithium metal corrosion decreases as SO₂ concentration increases, and that lithium is most stable in liquid LiAlCl₄:xSO₂ solvates at elevated temperatures in electrolytes with higher SO₂ content [38].

Both LiAlCl₄ and LiGaCl₄ in liquid sulfur dioxide over a temperature range –25 to 40 °C are temperature dependent and have a transition temperature near 25 °C based on our conductivity measurements and cyclic voltammetry [50, 52] as shown in Fig. 1(a) and (b), respectively. Above this transition temperature, the electrolyte of interest starts to show abnormal conductivity and redox properties, adversely affecting cell performance at elevated temperatures. This may explain why the electrolyte LiAlCl₄:3SO₂ has considerably limited cell operation and storage time at near ambient temperature.

For either electrolyte, below 25 °C the conductivity increases with increasing temperature while above 25 °C the conductivity decreases with increasing temperature. This phenomenon implies that a change in the ionic structure of the electrolyte occurs near transition temperature and results in two different ionic transport mechanisms. The cause of the decrease in

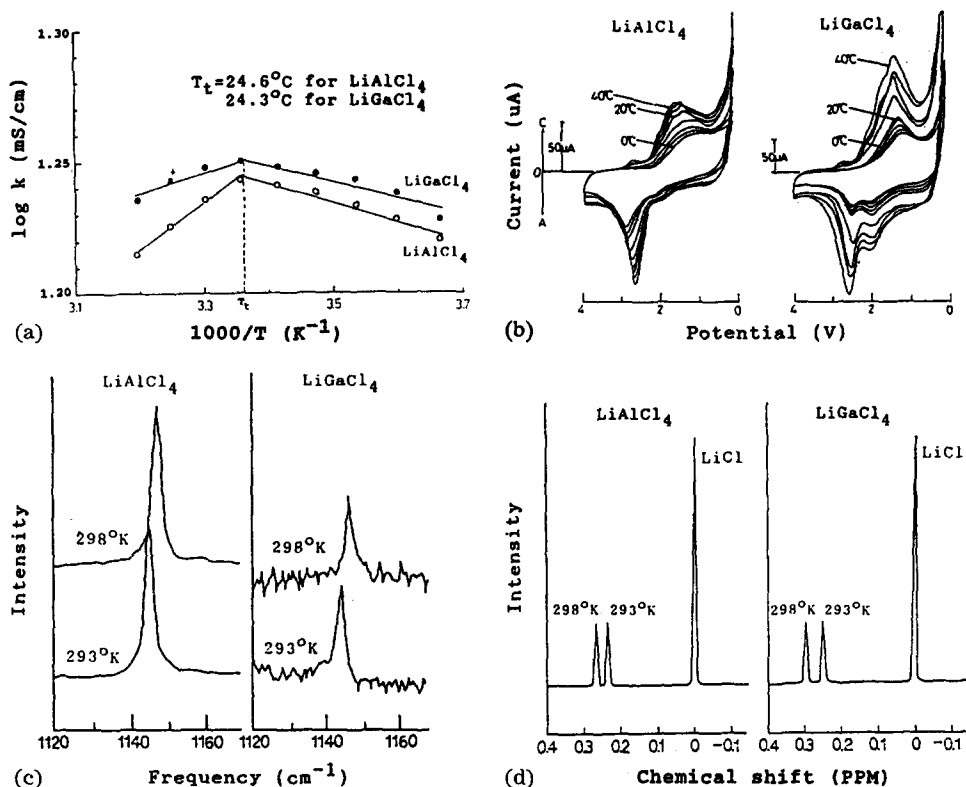


Fig. 1. Temperature dependence of 0.2 M LiAlCl_4 and LiGaCl_4 complexes in sulfur dioxide solution. (a) Arrhenius plots of the electrolyte conductivity vs. temperature [52]; (b) cyclic voltammograms from 0 to 40 °C [52]; (c) Raman spectra at 293 and 298 K [53]; (d) ^7Li NMR spectra at 293 and 298 K [53].

conductivity above 25 °C is probably the formation of ion-pairings or intimate ionic aggregates at high temperatures [77].

The ionic structural change of either electrolyte was clearly observed in our recent work in Raman spectroscopy and ^7Li nuclear magnetic resonance (NMR) [53] as demonstrated in Fig. 1(c) and (d), respectively. The intense S–O stretching Raman band of LiAlCl_4 or LiGaCl_4 solvate in SO_2 solution appears at 1146 cm^{-1} [78]. A distinct change in the frequency of this band was observed near 25 °C, indicative of changing solvation number in the ionic structure of the solvates of interest. The effects of cycle life and depth of discharge on Raman spectroscopy of the electrolytes, as well as the effects of SO_2 solvation on the cell performance, have been discussed. On the basis of the magnitude and direction of the ^7Li chemical shifts and the measurement of the spin-lattice relaxation time T_1 of $^7\text{Li}^+$ in liquid sulfur dioxide, we have studied the Lewis basicity of the solvent [79], the primary solvation numbers of cations, the ion–solvent and ion–ion interactions [80]. The details of both Raman and NMR work in connection with ionic structure, solvation numbers

of cations, and models for the ion-solvent configuration of $\text{Li}(\text{SO}_2)_x\text{AlCl}_4$ and $\text{Li}(\text{SO}_2)_x\text{GaCl}_4$ are also discussed [53].

Both concentration and temperature significantly affect the electrolyte properties of LiAlCl_4 and LiGaCl_4 in liquid sulfur dioxide [50]. We found that the temperature effect on the conductivity of the salts of interest dominates in the dilute solution, while the concentration effect plays an important role in the concentrated solutions [50]. LiGaCl_4 , with better conductivity behavior, is more stable than LiAlCl_4 .

On the basis of the OCV values at 3.2 V for the $\text{Li}/\text{LiAlCl}_4$, SO_2/C cell and 2.8 V for the $\text{Li}/\text{LiGaCl}_4$, SO_2/C cell, the discharge products on carbon electrodes at room temperature are LiCl and $\text{Li}_2\text{S}_2\text{O}_4$ for LiAlCl_4 and LiGaCl_4 electrolytes, respectively. However, the cathodic product is $\text{Li}_2\text{S}_2\text{O}_4$ instead of LiCl when the concentration of LiAlCl_4 sulfur dioxide solution falls below 1.0 M [54]. The effects of DOD and cycling on impedance parameters and the effects of electrolyte concentration on OCV and cell performance were discussed [54]. The Li/SO_2 cell with LiGaCl_4 electrolyte has reached 76 cycles and shown a capacity seven times greater than that with LiAlCl_4 electrolyte [54].

7. Conclusions

In spite of almost twelve years of intensive research, the Li/SO_2 rechargeable battery system is still at the prototype development stage. As reviewed, no theoretical model has yet emerged to enable one to determine, *a priori*, the performance of a given electrolyte or the type of electrode required for a particular battery system. Further research and development is required:

- (i) to obtain more safety data;
- (ii) to improve the cycle life and charging rate;
- (iii) to study electrolyte effects on conductivity, the dielectric constant, and the transport mechanism;
- (iv) to increase the capacity of positive electrodes and their rate capability upon recharge;
- (v) to confirm electrode structure and products.

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