# CALCIUM THIONYL CHLORIDE CELLS: ANODE STUDIES AT LOW TEMPERATURES

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## Summary

Calcium/Ca(AlCl$_4$)$_2$–oxyhalide/carbon cells have been studied because of their high energy density and purported safety advantages over lithium oxyhalide cells. However, calcium anode corrosion and poor cell performance at low temperatures have impeded development of this system. At −30 °C, severe anode polarization is responsible for cell failure. Performance of pure calcium and calcium alloys in Ca(AlCl$_4$)$_2$–thionyl chloride electrolyte was explored. It was found that best cell performance was achieved when the components included a pure calcium anode, 1 M Ca(AlCl$_4$)$_2$–SOCl$_2$ electrolyte containing 7% sulfur dioxide, and a carbon cathode composed of a blend of high and low surface area carbons. At −30 °C, anode polarization is due largely to increased electrolyte viscosity and related effects. Addition of SO$_2$ alleviates these effects to some extent. Raising the discharge temperature to −20 °C significantly improves cell performance.

## Introduction

The calcium thionyl chloride cell chemistry has been widely explored as an alternative to lithium thionyl chloride for use in high energy battery applications [1 - 14]. It is believed that the high melting point (839 °C) and inability to plate calcium metal in thionyl chloride-based electrolytes should afford safety advantages over lithium based systems. Preliminary studies support these claims [5, 6, 9].

Calcium thionyl chloride cell capacities are less than those for analogous lithium thionyl chloride cells, and observed open circuit potentials (2.9 - 3.2 V) are approximately 0.6 V below thermodynamic potentials. Cathode capacity is less than that in analogous lithium systems because of clogging by a more glassy CaCl$_2$ deposit. Cathode and electrolyte modifications [1 - 4, 11, 12, 14] have increased cathode capacity and load voltage. Unlike lithium anodes, at moderate current densities calcium anodes are

severely polarized at low temperatures (*e.g.*, −30 °C). The two biggest challenges remaining are protecting the anode from corrosion during storage and improving low temperature performance. Reasons for these short-comings have yet to be fully explained or resolved.

This paper examines attempts to improve anode performance at −30 °C. Cathode and electrolyte modifications are also discussed.

## Experimental

Redistilled thionyl chloride (EM Industries), and high purity aluminum chloride and calcium chloride (Anderson Physics) were used to prepare $Ca(AlCl_4)_2$–$SOCl_2$ electrolyte. Laboratory cells contained a 1 $cm^2$ anode and cathode separated by a woven glass separator. A smaller Li reference electrode was located between the anode and cathode, slightly offset to one side. Anodes were mechanically scraped to expose the shiny metal surface, and welded to a nickel tab. Cathodes were constructed as described previously [15] using Shawinigan 50% compressed acetylene black (Gulf), acetone-washed Black Pearls 2000 (Cabot), Ketjenblack EC DJ-600 (Akzo Chemie America, Chicago, IL) or mixtures of these. Calcium (99.99%) and calcium alloys (98%/2% lithium, 90%/10% antimony, 80%/20% strontium, 80%/20% barium, 95%/5% silver) were purchased from Pfizer. Fluorinated Shawinigan (1.01%) and Ketjenblack (5.13%) were purchased from Electrosynthesis Co. (Amherst, NY). Cells were assembled in an argon-filled (<10 ppm $H_2O$) glove box and filled with 7 ml of electrolyte. Sulfur dioxide was bubbled into the electrolyte, resulting in a solution containing approximately 7% $SO_2$ by volume. Cell discharges were computer controlled with Hewlett Packard Model 86B and 85B computers in line with Princeton Applied Research potentiostat/galvanostats models 173/176 or 276 and Fluka model 8842A digital multimeters. Temperatures were controlled in Tenney chambers. Experiments run at 20 °C began six hours after cell assembly. Discharges at all other temperatures were performed after allowing the cell to equilibrate for two hours at room temperature followed by fifteen hours at the temperature of interest. Constant current discharges at 5 mA $cm^{-2}$ were used throughout.

## Results

### Electrolyte

In unpressurized cells, 1.0 M $Ca(AlCl_4)_2$–$SOCl_2$ electrolyte provided best performance when it contained $SO_2$ ($\approx 7\%$ V/V). Cathode load voltages (*versus* a lithium reference) increased, and cathode life (capacity) was extended.

### Cathode performance

At room temperature, calcium cell performance is limited by the carbon cathode. Generally, at −30 °C calcium thionyl chloride cells

containing 1.0 M $Ca(AlCl_4)_2$–$SOCl_2$ with "standard" Shawinigan carbon black cathodes cannot support load voltages above 2.0 V when discharged at 5 mA cm$^{-2}$ (Fig. 1). Even so, cell performance is not a cathode problem. The cathode sustains a steady, high voltage (*versus* Li) long after anode failure (Fig. 2). Cathodes constructed from a mixture of 75% Shawinigan black and 25% acetone-washed Black Pearls 2000 showed improved performance by combining the catalytically active, high surface area of Black Pearls with the structural integrity of Shawinigan. At −30 °C, cathode load voltage was improved by about 0.1 V. Since cell voltage polarization is primarily due to the anode, the use of catalysts in the cathode would be expected to have only a slight effect (at the cathode) on reducing cell polarization.

Cells with mixed carbon cathodes operated at voltages above 2.0 V for about one minute. Addition of ≈ 7% (V/V) $SO_2$ (by bubbling $SO_2$ gas through the electrolyte) occasionally sustained the load voltage above 2.0 V for an additional two minutes (Fig. 1). Even though this is the most efficient combination of electrolyte and cathode tested, the cathode is utilized for less than 1% of its possible life (Table 1). In no case did the addition of $SO_2$ in combination with a Shawinigan cathode give load voltages higher than 2 V.

High rate (20 mA cm$^{-2}$) room temperature discharges using fluorinated carbons have shown improved cathode performance. Low temperature studies using mixtures (75%/25%) of 1.01% fluorinated Shawinigan, 5.13% fluorinated Ketjen Black, and the same carbons without fluorination, performed slightly less well than the 75% Shawinigan/25% Black Pearls
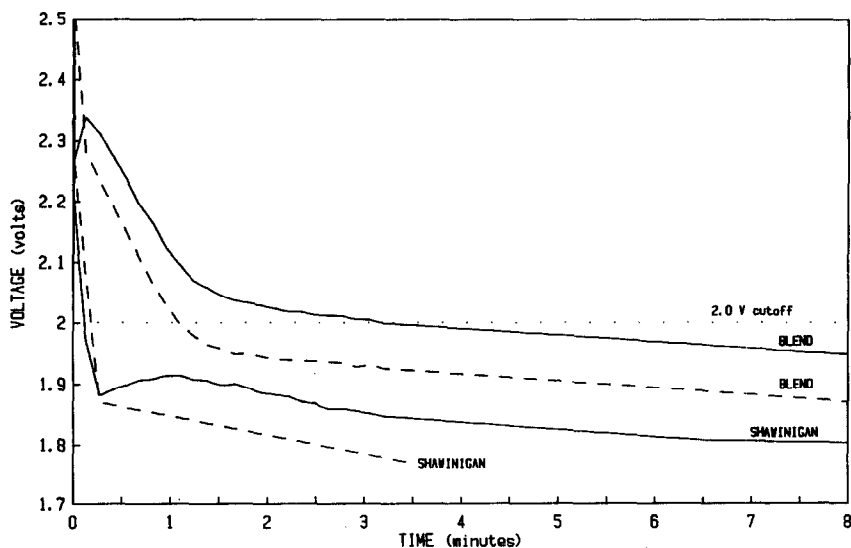


Fig. 1. Ca/1 M $Ca(AlCl_4)_2$–$SOCl_2$ cell discharge at −30 °C, 5 mA cm$^{-2}$ load. Shawinigan *vs.* 75% Shawinigan/25% Black Pearls cathode. − − −, baseline electrolyte; ———, $SO_2$-containing electrolyte (7% v/v).
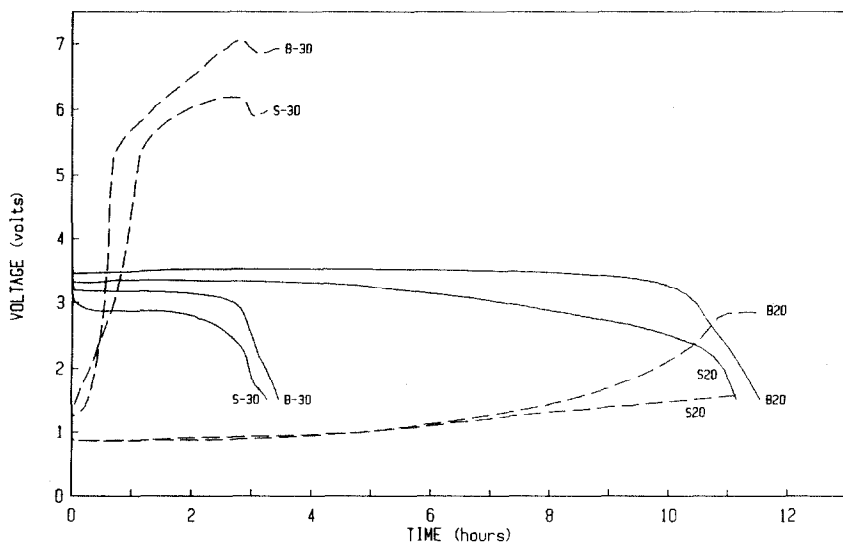
Fig. 2. Anode and cathode potentials *vs.* Li reference at 20 °C and −30 °C. − − −, calcium anode; ———, Shawinigan (S) or 75% Shawinigan/25% Black Pearls blend (B) cathode. Ca/1 M Ca(AlCl$_4$)$_2$–SOCl$_2$, 5 mA cm$^{-2}$.

TABLE 1

Ca/SOCl$_2$ cell performance at −30 °C

| Cell | Cathode life to 2 V (mA h cm$^{-2}$) | Cell life to 2 V (mA h cm$^{-2}$) | Cathode utilization (%) |
|---|---|---|---|
| 1 | 13.5 | 0 | 0 |
| 2 | 20.3 | 0 | 0 |
| 3 | 17.55 | 0.09 | 0.5 |
| 4 | 20.25 | 0.18 | 0.9 |
| 5 | 14.85 | 0.04 | 0.27 |

1, Shawinigan; 2, Shawinigan, SO$_2$ in electrolyte; 3, Shaw./Black Pearls mix; 4, Shaw/BP mix, SO$_2$ in electrolyte; 5, Fluorinated Shaw./fluorinated Ketjen mix.

cathodes. Therefore, at low temperature and low current density there seems to be no advantage in using fluorinated carbons. The Shawinigan/ Black Pearls cathode blend appeared to be superior with respect to total time of cathode utilization and load voltage.

*Anode performance*

Several attempts were made to understand and improve calcium anode performance at −30 °C, where it is the limiting electrode (Fig. 2).

Meitav and Peled [6, 9] attributed high anode resistance to the solid electrolyte interphase (SEI) of CaCl$_2$ on calcium, which has a very low

transport number for $Ca^{2+}$ ions. We attempted to modify the SEI to allow better $Ca^{2+}$ ion transport and/or to retard corrosion by using alloys of calcium with either barium, strontium, antimony, lithium or silver. Calcium alloys have been studied by others [7, 10, 12], with Sb, Zn [10] and Li [7] alloys showing better current-carrying capability than pure calcium. None of the alloys used in this study showed low temperature performance comparable with that of pure calcium. Figure 3 shows that pure calcium and the alloys appear to give roughly the same load voltages at $-30$ °C. A sharp anode polarization starts shortly after cell discharge begins and then levels off at potentials between 5 and 9 V (*versus* Li). Antimony showed severe corrosion during room temperature discharge, thus low temperature performance was not studied.

At room temperature these calcium alloys performed much more poorly than pure calcium, and upon cell disassembly, the calcium alloy anodes were observed to be severely corroded. In some cases almost no metal remained attached to the nickel current collector. Anode behavior (Fig. 4) under these conditions showed that during the first 2 - 3 h the alloys were generally undistinguishable from pure calcium (continually polarized by nearly 1 V). After 3 - 5 h of discharge the alloys began to polarize more severely. The only deviation from this description was the strontium alloy which behaved much like pure calcium for the first 6 h. A simple explanation for the severe polarization of the alloys is that since corrosion was high and pieces were breaking off from the anode, effective current density at the anode was continually increasing. These results imply that the anode should consist of the highest purity calcium metal obtainable.
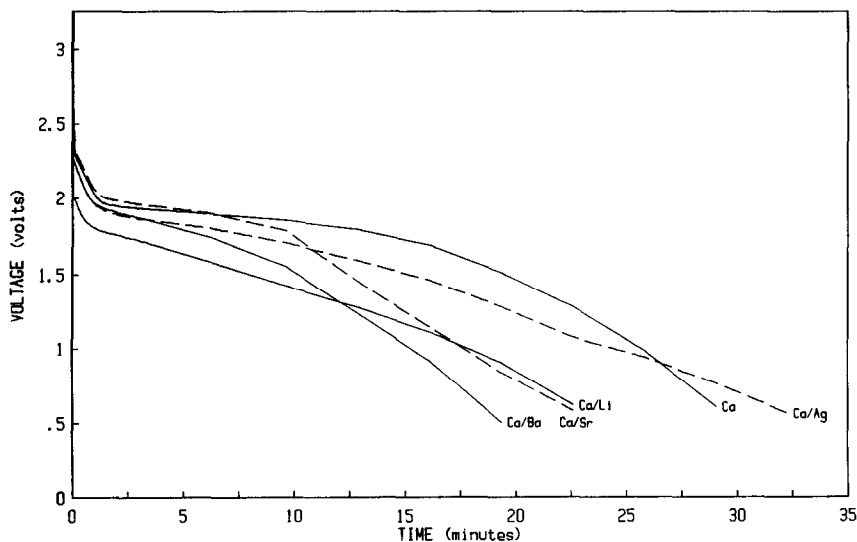


Fig. 3. Cell discharge at $-30$ °C, 5 mA cm$^{-2}$ of cells containing 75% Shawinigan/25% Black Pearls cathodes, 1 M Ca(AlCl$_4$)$_2$–SOCl$_2$ electrolyte, and anodes of pure Ca, Ca/Ba, Ca/Sr, Ca/Li and Ca/Ag alloys.
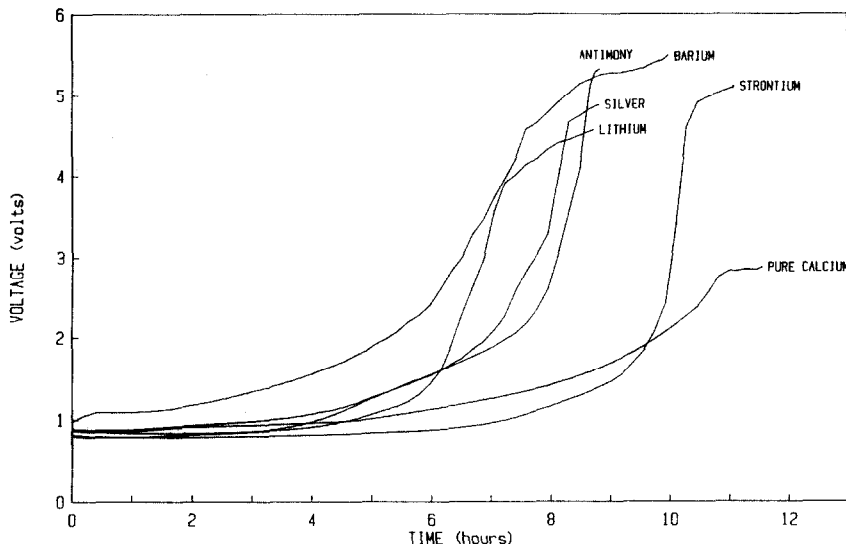
Fig. 4. Anode polarization (*vs.* Li reference) of pure calcium and various calcium alloys, 1 M $Ca(AlCl_4)_2$–$SOCl_2$, 75% Shawinigan/25% Black Pearls cathodes, 5 mA cm$^{-2}$, 20 °C.

Visual observation of calcium and calcium alloy coupons immersed in 1.0 M $Ca(AlCl_4)_2$–$SOCl_2$ electrolyte in sealed tubes for 60 days (20 °C) showed corrosion of all samples, with only small differences between samples. A lithium sample in the same electrolyte showed no signs of corrosion. Even if some protection from corrosion is afforded by one or more alloys, cell discharge results presented here indicate that performance will suffer.

Another attempt to modify the SEI was by soaking the calcium anode overnight in $SOCl_2$ prior to cell assembly. Since Ca corrosion is lower in neat $SOCl_2$ it was hoped that the $CaCl_2$ film might "grow" more slowly and form a more uniform $CaCl_2$ lattice, free from sulfur [16] or $Al^{3+}$ ions in the electrolyte which may cause defects. Unfortunately, low temperature performance of these pretreated anodes was undistinguishable from unteated calcium anodes.

An additional attempt to control corrosion involved the use of yttrium. Since the ionic radius of $Y^{3+}$ is close to that of $Ca^{2+}$ it was hoped that addition of small amounts of $Y(AlCl_4)_3$–$SOCl_2$ might cause an exchange reaction whereby $YCl_3$ would form in place of, or as part of, the $CaCl_2$ SEI. This might change ion transport and corrosion characteristics of the SEI. Earlier work [17] with $SrCl_2$ provides some basis for this hypothesis. Conductivity of $SrCl_2$ is due only to anion transport. When $Y^{3+}$ was incorporated into $SrCl_2$ it was found that conductivity and anion diffusion were decreased. If yttrium is likewise able to decrease anion diffusion through $CaCl_2$, this should retard or prevent corrosion reactions at the calcium metal surface. However, $YCl_3$ salt was not very soluble in $AlCl_3$–$SOCl_2$ solutions. Electroplated yttrium or an alloy might be useful.

Interaction of the alloying metal with the cathode was studied with the 98%Ca/2%Li alloy. Following room temperature discharge, the cathode was rinsed in $SOCl_2$ to remove excess salt or other soluble materials and then the insoluble precipitate was extracted with water and analyzed for Li by atomic absorption. It was expected that Ca would predominate since it is the overwhelming metal in the anode, and the only cation in the electrolyte. Based on the small amount of Li found, it was concluded that more than half of the cations deposited at the cathode came from the electrolyte. It is implied that metal cations (other than calcium) from calcium alloys will have a negligible effect on the cathode. This result might be important if other electrolyte salts are used since different cathode precipitates would form, possibly affecting cathode capacity.

### Model for anode performance and polarization

Unlike LiCl formed on lithium anodes in oxyhalide electrolytes (which allows transport of $Li^+$ ions), $CaCl_2$ film formed on calcium allows almost no $Ca^{2+}$ ion transport (although anion transport is allowed). If calcium anode performance at low temperature is dictated primarily by the SEI, then one should be able to significantly change or otherwise incorporate defects into the $CaCl_2$ SEI, resulting in altered anode performance. None of the alloys had a significant effect on anode performance at $-30$ °C. Since the SEI was necessarily different in each case, and no beneficial change in performance was observed, one may conclude that the SEI was not the predominant controlling factor for low temperature performance.

A logical explanation is that electrolyte changes such as increased viscosity, decreased salt solubility, reduced conductivity, and lowered ion diffusion ultimately limit anode performance. Upon cell discharge there is, initially, a large increase in anode polarization as the $Ca^{2+}$ ion concentration increases locally, due to low diffusion away from the anode. As the concentration of ions increases there is a saturation and/or precipitation of salt at the anode (due to $AlCl_4^-$ or $Cl^-$ nearby). In the absence of counter ions the local increase of $Ca^{2+}$ ions creates a positive charge on the anode surface, drawing anions towards it. Concentration effects become more exaggerated and precipitation of calcium salt continues. Figure 5 gives good evidence for this since, initially, there is some anode polarization. Yet, after a short time, it sharply increases as local ion concentrations build up. This further increases solution viscosity and decreases diffusion. The SEI no doubt contributes to this scenario but is not of major significance since at 20 °C the anode is not as severely or sharply polarized. At 20 °C, electrolyte viscosity is much lower and there is more rapid diffusion of ions. If the anode was SEI-controlled, both $-30$ °C and $+20$ °C anode polarization curves should look similar.

In $Li/SOCl_2$ cells there is very little anode polarization and it remains constant with time. However, in $Ca/SOCl_2$ cells the calcium anode is polarized by nearly 1 V (*versus* Li, 20 °C) at the onset of discharge and increases during discharge. If we assume that room temperature calcium
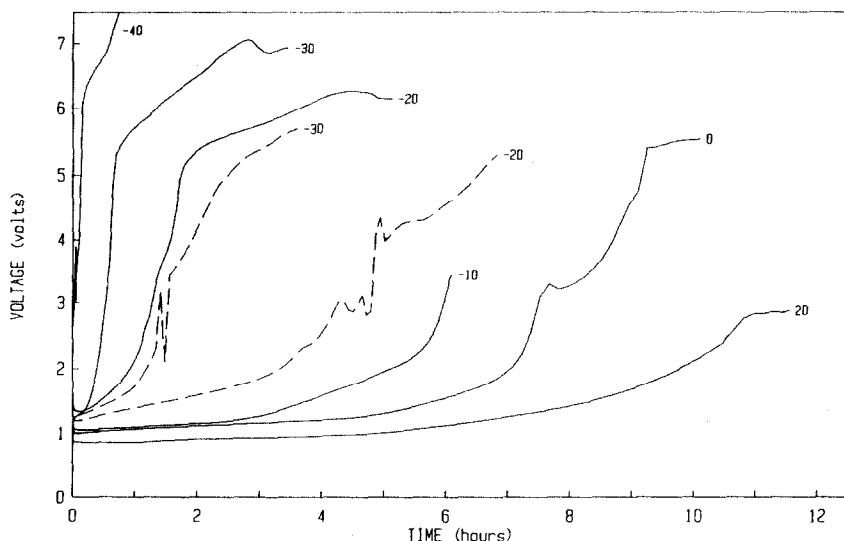
Fig. 5. Calcium anode polarization (*vs.* Li reference) as a function of temperature. 1 M Ca(AlCl$_4$)$_2$–SOCl$_2$, 75% Shawinigan/25% Black Pearls cathodes, 5 mA cm$^{-2}$. — — —, SO$_2$ containing electrolyte.

anode polarization is attributable mainly to the SEI, then low temperature polarization can be envisioned as an additive effect due to increased salt concentration and viscosity coupled with the room temperature SEI effect.

The solubility of Ca(AlCl$_4$)$_2$ in SOCl$_2$ at various temperatures was studied by preparing solutions of 1.0 M, 1.29 M, 1.5 M and 1.8 M Ca(AlCl$_4$)$_2$–SOCl$_2$ electrolyte placed in Teflon-lined screw cap tubes containing CaCl$_2$ crystals as seeds. The sealed tubes were placed in a Tenney chamber and the temperature slowly lowered to observe at what temperature and concentration salt would precipitate out of solution. No precipitate was noticed down to −40 °C, even after standing overnight. At −50 °C, the 1.0 M, 1.29 M and 1.5 M solutions froze completely, without salting out. The 1.8 M solution froze slowly (without salting out), beginning at the CaCl$_2$ seeds at the bottom, yet it only froze halfway up the tube after 16 h. Upon warming, all tubes melted back to liquids. Certainly, no added salts would have dissolved at low temperatures since preparing these solutions at room temperature is difficult. However, it was surprising that no salt came out of solution. A possible explanation for this comes from the work of McDonald [18]. He studied LiAlCl$_4$–SOCl$_2$ solutions at low temperature and concluded that the system behaves as a mixture of two miscible liquids (SOCl$_2$ and LiAlCl$_4$·2SOCl$_2$) rather than a salt solution. Only one phase transition was observed as the liquid completely solidified with no salting out. Our observations of Ca(AlCl$_4$)$_2$–SOCl$_2$ could be similarly explained.

Evidence of the importance of salt concentration is shown in Figs. 6 and 7, where cells with varying concentrations of electrolyte were discharged
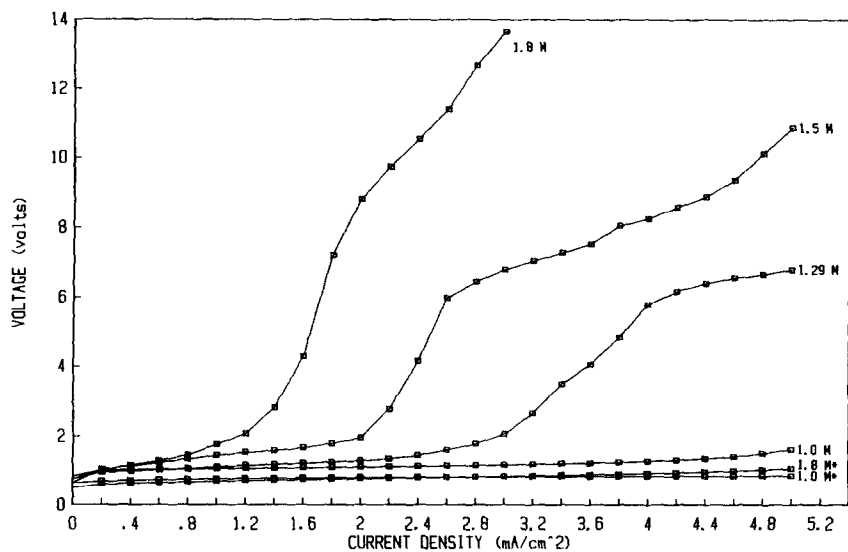
Fig. 6. Calcium anode polarization (vs. Li reference) as a function of current density and electrolyte concentration. Steps at each current density lasted 2 min. 1 M Ca(AlCl$_4$)$_2$– SOCl$_2$, 75% Shawinigan/25% Black Pearls cathodes, −30 °C (except where noted by * = 20 °C).
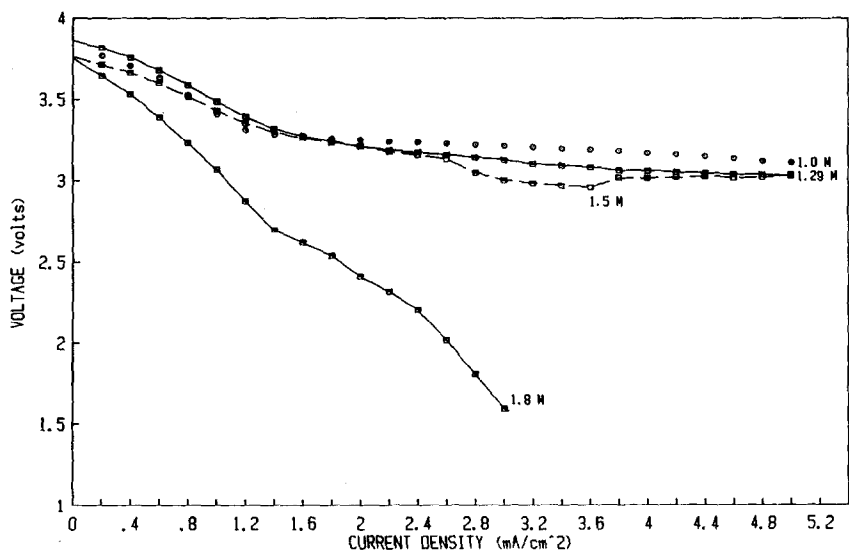


Fig. 7. Effect of electrolyte concentration on cathode polarization (vs. Li reference) at −30 °C. Each current step lasted 2 min. 75% Shawinigan/25% Black Pearls cathodes.

at either 20 °C or −30 °C, at current steps of 0.2 mA cm$^{-2}$ of 2 min duration from 0.2 up to 5 mA cm$^{-2}$. In 1.0 M electrolyte, cathode polarization at both temperatures is small and fairly constant throughout the entire range of

current densities. Anode polarization is not severe and very slowly increases with current density at 20 °C. At −30 °C, polarization is slightly greater but not severe.

In 1.8 M electrolyte, anode polarization at 20 °C is comparable with that in 1.0 M electrolyte. However, at −30 °C there is severe polarization, even at very low current densities. In addition, cathode performance is degraded and triggers the circuit to open before 5 mA cm$^{-2}$ is reached (hence, data are shown only to 3.0 mA cm$^{-2}$). Intermediate anode polarization values are observed in 1.29 M and 1.5 M electrolyte concentrations. After the −30 °C polarization routine was completed, cells were left at open circuit while anode and cathode potentials were monitored. Recovery of the cathode potential was identical in 1.0 M and 1.8 M electrolytes; however, anode potential recovery was noticeably different between these concentrations. Anodes in 1.0 M electrolyte immediately returned to close to open circuit value, implying that polarization resulted mostly from *IR* drop. In 1.8 M electrolyte, the anode remained polarized on open circuit and required several minutes to approach "true" open circuit potentials. The slow return to OCV is consistent with diffusion related effects. It is concluded that at high salt concentrations, diffusion and viscosity have a pronounced effect, determining cell performance.

As noted earlier, SO$_2$ added to the electrolyte allowed cell load voltages to remain above 2 V for a few minutes, due to reduced cathode polarization and reduced anode polarization. Improved anode behavior is attributed to a beneficial cosolvent effect which increases conductivity and decreases viscosity (but not to SO$_2$ changing the SEI on the calcium). NMR studies in sulfuryl chloride (unpublished data) suggest that calcium ions are solvated by sulfur dioxide, probably in much the same way as Li$^+$ is preferentially solvated by SO$_2$ in LiAlCl$_4$–SOCl$_2$ electrolyte [19]. This would allow for some dissolution and diffusion of Ca$^{2+}$ formed at the anode. Perhaps higher SO$_2$ concentrations would allow even better performance but would require the use of a pressurized system.

One unexplained observation occurred during low temperature discharge with SO$_2$-containing electrolyte. At both −20 °C and −30 °C, when the anode became polarized 2 - 3 V *versus* lithium, 1 V fluctuations occurred in a fairly regular pattern during a 1 h period, after which anode polarization proceeded "smoothly" (Fig. 5). The cathode showed no such perturbations, so the effect was not due to gas evolution at the cathode. One might speculate that SO$_2$ pressure breaks open areas of the SEI or somehow changes the high concentrations of salt forming at the anode.

*Cell startup and temperature effects*

Cathodes made from Shawinigan/Black Pearls mixtures or fluorinated carbon mixtures allowed cell voltages above 2 V for about 1 min. Addition of SO$_2$ extended cell life of blended Shawinigan/Black Pearls cathodes another minute or so, but was of no benefit to cells containing Shawinigan cathodes. Below −10 °C, cells had virtually no useable life, unless SO$_2$ was

added to the electrolyte (Table 2). Raising the temperature from $-30$ °C to $-20$ °C (in electrolyte containing $SO_2$) provided dramatically improved cell life (*cf.* Fig. 1 with Figs. 8, 9). At $-20$ °C, there was about a half hour of cell life (above 2 V). The effect of $SO_2$ on cell life diminishes with increasing temperature.

Voltage delay upon initial load in fresh cells does not appear to be a problem (Figs. 1 and 8). It is important to note that cell voltage remained above 2.0 V and degraded below this voltage rather than climbing up from below this value. Therefore, as long as initial load voltages are sustained above 2 V for a short period (as the data support) one could expect better performance as the battery warms.

TABLE 2

| Temp. (°C) | Capacity to 2.0 V (mA h cm$^{-2}$)* |
|---|---|
| 20 | 38.42 |
| 0 | 22.08 |
| $-10$ | 13.75 |
| $-20$ | 0.14 (2.53 with $SO_2$ added) |
| $-30$ | 0.1   (0.18 with $SO_2$ added) |
| $-40$ | 0 |

*75% Shawinigan/25% Black Pearls cathodes, 5 mA cm$^{-2}$.
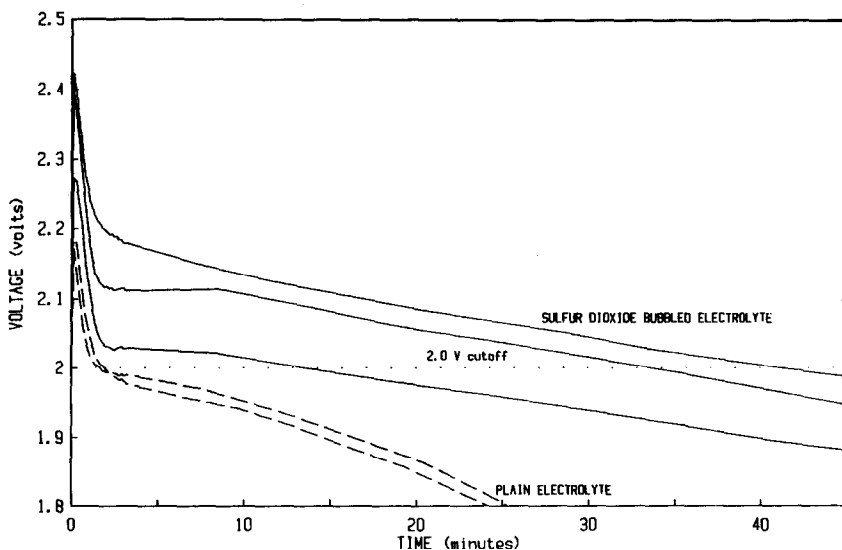


Fig. 8. Ca/1 M Ca(AlCl$_4$)$_2$–SOCl$_2$/75% Shaw./25% Black Pearls cells discharged at $-20$ °C, 5 mA cm$^{-2}$. Baseline electrolyte (— — —) *vs.* $SO_2$ containing (7% v/v) electrolyte (———).
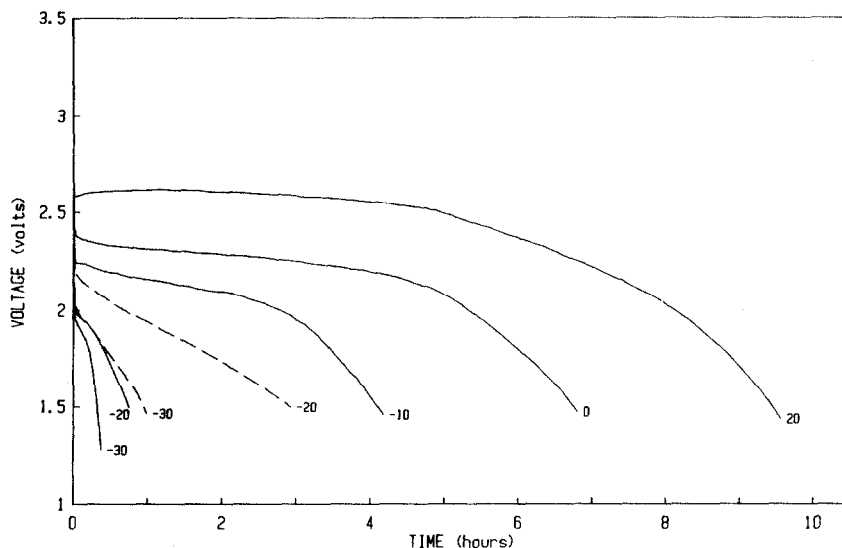
Fig. 9. Cell discharge as a function of temperature and $SO_2$ addition. Ca/1 M Ca(AlCl$_4$)$_2$–SOCl$_2$/75% Shaw./25% Black Pearls, 5 mA cm$^{-2}$. – – –, $SO_2$ bubbled through electrolyte (7% v/v); ——, plain electrolyte.

## Conclusions

Poor low temperature performance of calcium thionyl chloride cells is attributed to the calcium anode. Use of pure calcium anodes, 1 M Ca(AlCl$_4$)$_2$–SOCl$_2$ electrolyte with $SO_2$ bubbled through it, and a cathode blend composed of 75% Shawinigan and 25% acetone-washed Black Pearls may provide performance above 2 V for a few minutes at −30 °C (at 5 mA cm$^{-2}$).

Changing the calcium anode SEI via the use of calcium alloys had virtually no effect on anode performance. Factors controlling anode behavior at low temperature are related to electrolyte salt solubility, viscosity, and electrolyte conductivity.

At −30 °C, as electrolyte concentration was increased, anode polarization severely increased while cathode performance was unaffected except at very high salt concentrations. Upon return to open circuit, calcium anodes in 1 M Ca(AlCl$_4$)$_2$–SOCl$_2$ electrolyte showed immediate return to OCV (*IR* effects implied), whereas in 1.8 M electrolyte several minutes were required to return to OCV (diffusion effects implied).

Addition of $SO_2$ to the electrolyte decreased anode polarization at −30 °C, by solubilizing salt, increasing conductivity, and decreasing viscosity. If the temperature is raised to −20 °C, load potentials above 2 V for over 30 min are possible at 5 mA cm$^{-2}$.

Voltage delay for fresh cells did not appear to be a problem since initial load voltages at 5 mA cm$^{-2}$ were above 2 V at all temperatures.

At −30 °C we are at the threshold of acceptable cell performance for the calcium thionyl chloride system. Improvements shown at −30 °C may seem marginal, but the heat generated in a spirally wound cell will quickly raise the internal cell temperature and thus improve capacity. A battery package of 5 - 10 cells should produce significant thermal heating, allowing the battery to sustain itself. As heating progresses, the load voltage and capacity characteristics will simultaneously be improved.

Once storage concerns (corrosion) are addressed, it appears that this system will be a viable alternative to lithium chemistries for ultra-safe battery applications.

## Acknowledgements

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