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Hydroxide-Limited Silver-Zinc Cells

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14. ABSTRACT

Silver-zinc cells under certain circumstances can be limited in capacity by the amount of hydroxide ion added during cell activation. This report examines the reasons for this limiting condition and suggests techniques to determine whether cells are limited by their hydroxide content. In addition, the report found that the projected cell capacities as a function of wet-stand duration followed the same capacity trend line as a group of cells that were affected by hydroxide-limitation problems that resulted in low ampere capacities during cell discharge.

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1. Background

Silver-zinc cell configurations that were examined for the electrolyte requirements of that chemistry are not new. The foundational studies by Shepherd[1] employed assembled flat plate cells consisting of a single zinc sheet placed between two silver-oxide electrodes. The cells, with different current densities and electrolyte concentrations, were discharged until the zinc electrode became passivated due to the insufficient rate of transport of hydroxide ions to the surface of the zinc electrode. At this point, the electrochemical processes stopped. The experimental results were recorded as ampere-minutes of capacity per square inch of zinc electrode per gram of potassium hydroxide. Shepherd's experiments and results are best understood by understanding the reaction sequence at the surface of the zinc electrode. The authors in the book edited by Fleischer and Lander[2] suggest the following possible reaction sequences during discharge based on their studies of the zinc electrode in alkaline solutions.

First, the zinc is oxidized via a two-electron reaction, resulting in the formation of zinc oxide or hydroxide on the surface. Next, hydroxide ions from the electrolyte solublize the zinc oxide or hydroxide with the formation of a soluble zincate ion. These steps are shown in Sequences 1 and 2.

Sequence 1. $Zn + 2OH^- = ZnO + H_2O + 2e^-$

followed by $ZnO + H_2O + 2OH^- = Zn(OH)_4^{-2}$

or

Sequence 2. $Zn + 2OH^- = Zn(OH)_2 + 2e^-$

followed by $Zn(OH)_2 + 2OH^- = Zn(OH)_4^{-2}$

Figure 1 is illustrative of the results of the Shepherd study as reported in Ref. 2, Ch. 23. The significant increase in capacity at lower current densities is a consequence of the improved mass transport environment in these situations. At the higher current densities, it is more difficult for the hydroxide ions to reach the surface of the zinc electrode where it solublizes the sparingly soluble zinc intermediates forming on the surface of the zinc. Hydroxide ions reach the surface of the zinc electrode via diffusion, convection, and migration. When the rate at which the hydroxide ions arrive at the surface is exceeded by the rate at which the insoluble zinc intermediates are formed, the electrochemical reaction abruptly ceases. The extensive studies by Shepard into this topic were to aid in the design of a silver-zinc cell with the appropriate amount of hydroxide ion and surface area of zinc. To extend the discharge time of the zinc electrode, higher concentrations of hydroxide were used, and zinc electrode structures with more surface area were manufactured. For a specific application, the amount and concentration of potassium hydroxide is very important.

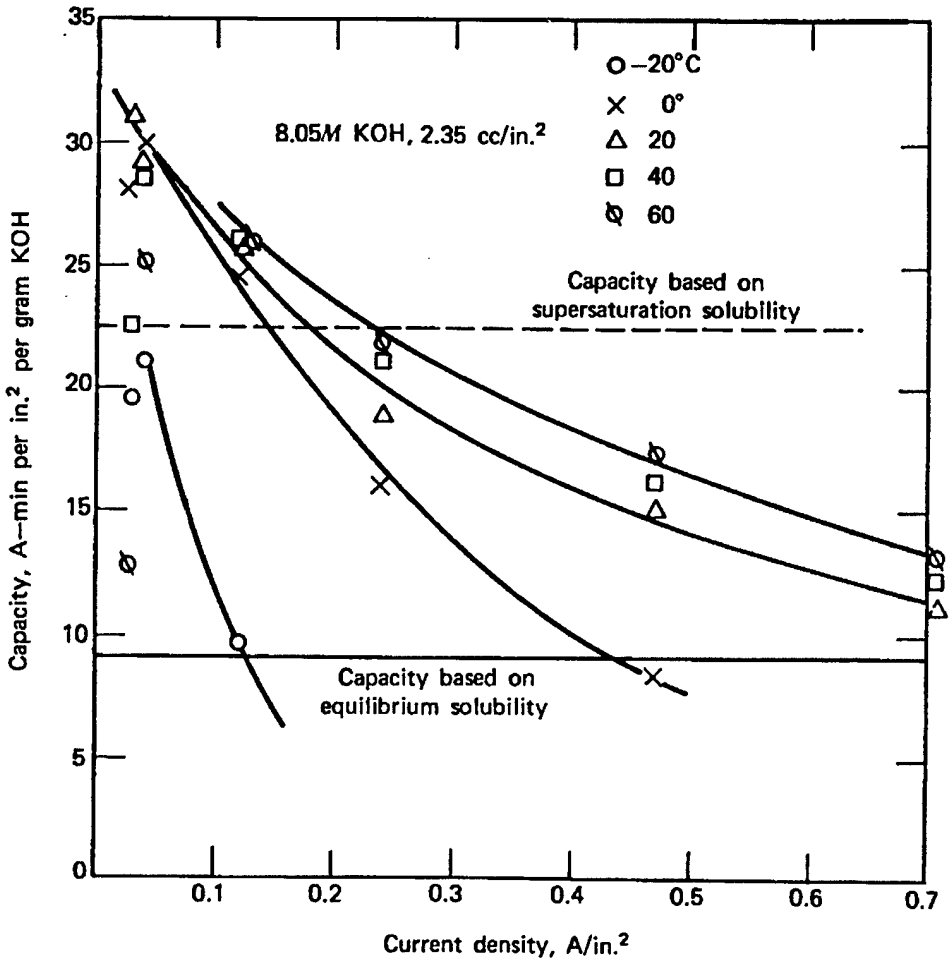

Figure 1. Cell capacity as a function of current density.

Recently, a group of silver-zinc cells were constructed that at first appeared to be limited by the amount of remaining zinc at the time the cells were discharged. A closer examination of all of the available data suggested that the cells were actually limited in their discharge time by the amount of hydroxide ion that was available at the time the cells were discharged following their wet-stand periods. A review of the reactions taking place during the wet-stand period as they relate to the hydroxide availability at the end of the wet-stand period was undertaken to investigate the possible existence of cells that were hydroxide limited at the time of discharge. In conjunction with that study, several suggested techniques are outlined to help determine the exact cause of the capacity limitation experienced during cell discharge following a wet-stand period. The corrosion characteristic of the zinc electrode material is an important factor in causing premature termination of cell discharge. The actual cause of the premature failure is the passivation of the zinc surface due a deficiency of hydroxide ion.

2. Introduction

Silver-zinc batteries are used in many different sizes on all launch vehicles. The silver-zinc chemistry has been the system of choice since the beginning of the space program for applications where high-energy-density primary (non-rechargeable) batteries are needed. Silver-zinc batteries have not been free of problems, but like any other device operating at the edge of its performance limit, constant vigilance must be applied to their design, development, manufacture, storage, and use.

Launch vehicle batteries from different cell manufacturers come in sizes ranging from less than 1.0 A-h to greater than 200 A-h. These sizes are dictated by the application for which they are intended. There are a variety of uses on a typical launch vehicle or upper stage. This results in different design guidelines for these different applications. The mission duration of the booster stage is only a few minutes so the batteries for that application do not have any long-term loads to supply. They are used to send a pulse of current to initiate the explosive material in the bolts used to separate the different stages of the overall launch vehicle. Another application that requires only a very short current pulse is the detonation of the explosives onboard the launch vehicle. This only occurs when a launch attempt is not going correctly and, based on the decision of the range safety officer, the mission must be terminated. Batteries are also used to operate pumps, adjust the vectoring of rocket motors, and supply instrumentation power to an assortment of measuring devices used during the vehicle's flight. The aforementioned requirements take advantage of the very high current pulse capability of silver-zinc cells. At the other end of the spectrum, upper stages often transport satellites from a LEO orbit to a GEO orbit or wherever the final destination might be. The transit time can be many hours or weeks during which telemetry and heater loads must be supplied. For these applications, the high energy density capabilities of these batteries are required.

These two different classes of application give rise to two different cell design methodologies. However, silver-zinc cells have two important features that require careful consideration as they relate to the original cell design.

1. The zinc anode material, even though it is amalgamated to reduce its reactivity with the potassium hydroxide electrolyte, reacts in a manner that results in the formation of hydrogen gas, the depletion of zinc capacity, the consumption of hydroxide ions, and the consumption of water molecules.
2. During the electrochemical discharge of the zinc electrode, hydroxide ions and water molecules are also consumed as the insoluble intermediate zinc species are solublized.

Common to all silver-zinc cell types is the fact they are typically built with a larger capacity of zinc, in terms of ampere-hours, relative to the capacity of silver oxide. The cells are referred to as silver limited since the silver oxide should be exhausted prior to the exhaustion of the zinc. Due to the consideration of characteristics 1 and 2 above, the concentration and amount of electrolyte is another important consideration in each cell design. The concentrated solution of potassium hydroxide not

only provides ionic conductivity between the zinc and silver oxide electrodes, but the hydroxide ions are consumed as part of the chemical and electrochemical reactions at the zinc electrode.

Although most silver-zinc cells used in aerospace applications are silver limited when they are first built, the self-discharge reactions that take place during the wet-stand period can cause the cells to become either zinc limited or hydroxide limited when the cell is finally discharged. For this reason, it is important to fully understand the electrolyte requirements and reactions within the different cell designs. This is accomplished by knowing the hydroxide requirements of the different chemical and electrochemical reactions involved with this cell chemistry. These requirements are impacted by the corrosion rate of the zinc electrode. This is related to the effectiveness of the amalgamation process used to reduce the corrosion rate of the zinc electrode during the wet-stand period prior to use.

3. Silver-Zinc Cell Designs

3.1 Limiting Conditions in Silver-Zinc Cells

The performance of silver-zinc cells can be limited in a number of different ways. They are typically designed to be limited by the amount of silver oxide built into the positive electrodes. In cases where there have been excessive amounts of zinc corrosion, cells can become limited in their performance by the remaining amount of zinc in the negative electrodes. In cases where there is an insufficient supply of hydroxide ions at the surface of the zinc electrodes to solublize the zinc oxide or hydroxide forming on the surface of the zinc during discharge, the surface will become passivated and useful discharge will stop. This deficiency of hydroxide ion can be caused by an insufficient amount of hydroxide in the electrolyte or an insufficient rate of transfer of hydroxide ions to the surface of the zinc electrodes. The early studies by Shepherd (Figure 1) showed how the capacity of an electrode could vary from 10 to 30 ampere-minutes per square inch per gram of KOH as the current density was varied from 0.7 A/in.^{2} to 0.05 A/in.^{2} In his experiments, Shepherd used sheet zinc electrodes. A fourth condition that can result in limiting the performance of a silver-zinc cell is an insufficient amount of electrolyte volume needed to properly wet the surfaces of the electrodes.

3.2 Reactions Within Silver-Zinc Cells

The reactive materials in silver-zinc cells designed for delivering maximum amounts of ampere-hour capacity are silver peroxide and amalgamated elemental zinc. The possible electrochemical reactions during cell discharge are represented by the following three reactions.

$$Zn + AgO = ZnO + Ag \tag{1}$$

$$Zn + AgO + H_2O = Zn(OH)_2 + Ag \tag{2}$$

$$Zn + AgO + H_2O + 2\ OH^- = Zn(OH)_4^{-2} + Ag \tag{3}$$

Since the reaction products in reactions (1) and (2) are only slight soluble, the surface of the zinc would quickly become covered with an insoluble material, and the electrochemical reaction would cease. Reaction 3 is the generally accepted overall reaction sequence during the discharge of the zinc electrode. Although the hydroxide ions are able to solublize the surface film forming on the zinc electrode (Figure 2), there are solubility limits associated with zincate ion. Electrolyte solutions can become supersaturated with zincate ion. Ultimately, zinc hydroxide will precipitate from solution as per reaction (4).

$$Zn(OH)_4^{-2} = Zn(OH)_2 + 2OH^- \tag{4}$$

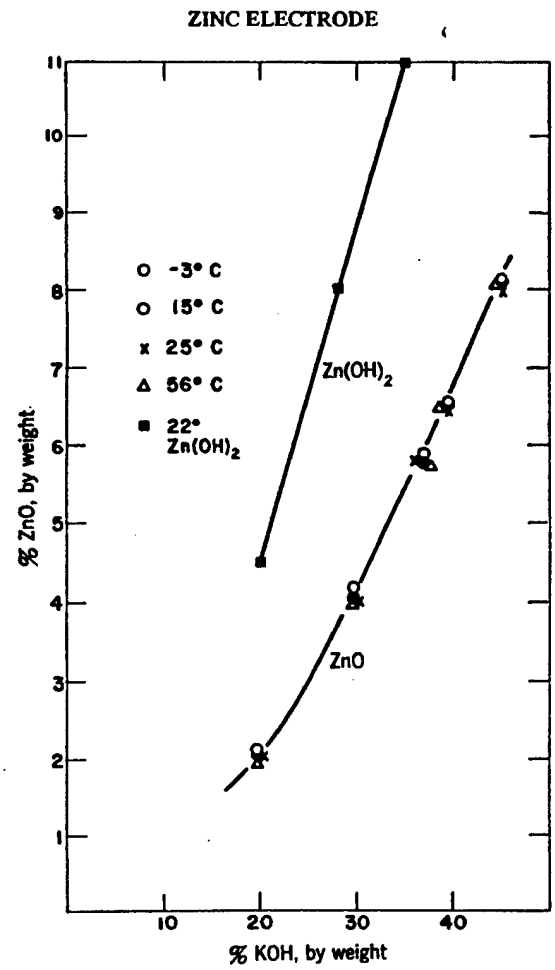

Figure 2. Solubility of ZnO and $Zn(OH)_2$ as a function of KOH concentration (Ref. 2, Ch. 3).

To be noticed is the release of hydroxide ions as the zinc hydroxide is precipitated into the bulk electrolyte. During the wet-stand period following the addition of electrolyte to the cells, several chemical reactions are possible between the zinc and the electrolyte. These reactions are listed in the following corrosion reactions.

$$Zn + 2H_2O = H_2 + Zn(OH)_2 \tag{5}$$

$$Zn + 2H_2O + 2OH^- = H_2 + Zn(OH)_4^{-2} \tag{6}$$

The initial reaction during the corrosion of zinc during the wet-stand period will be reaction (6). At the point where the solubility limit of the zincate ion is reached, the corrosion will proceed as suggested by reaction (5) or by reaction (6) followed by reaction (4), with the net reaction again described by reaction (5). Whereas reaction (6) consumes hydroxide ion, reaction (5) does not.

3.3 Performance in Actual Cells

As suggested by the studies of Shepherd, by having high-surface-area zinc electrodes and high concentrations of hydroxide ions, one would expect that zinc electrodes can be discharged to completion without experiencing problems related to hydroxide deficiencies. However, this is not always the case. Whether the solution becomes saturated or supersaturated with zincate ions, the formation of zincate ions consumes some of the available hydroxide ion. Reaction (4) will replenish some of the hydroxide to the electrolyte solution as the zinc hydroxide is precipitated away from the surface of the zinc electrodes. This permits the cell discharge reaction to proceed to completion provided enough hydroxide ion has been provided to the cell during activation.

The impact of zinc corrosion during the wet-stand period can alter the performance of cells that, by virtue of their design, are either hydroxide limited or close to being hydroxide limited. Since the naturally occurring corrosion reactions during the wet-stand period [reactions (5) and (6)] generate hydrogen commensurate with the amount of zinc that is corroded, the amount of zinc capacity that is lost can be estimated by collecting the volume of hydrogen exiting the cell during the wet-stand period. If the cell design is originally hydroxide limited, it will become more so based on the loss of hydroxide ion via reaction (6). As soon as the solubility limit for zincate is reached, there will not be any further reduction in available hydroxide as the corrosion reaction now continues as per reactions (5) or (6) followed by reaction (4).

3.4 Helpful Tests for Hydroxide Limitaions

Several routine tests can be applied to silver-zinc cells to determine whether or not hydroxide limitations are present at the time the cells are discharged. Some of these tests require a reference electrode that measures the individual electrode potentials relative to a reference potential such as a Hg/HgO reference electrode, whereas others require a combination of chemical analysis and corrosion measurements using a technique that measures the volume of hydrogen gas liberated. The Appendix B of Reference 3 contains several useful procedures that can be helpful in carrying out the techniques listed in this section. In a cell that is fully discharged, there should be undischarged zinc in the zinc electrodes if the cell is still silver limited. To differentiate the presence of remaining amounts of zinc due to hydroxide limitations and simply a normal situation of the cell being silver limited requires additional testing. The following paragraphs outline several suggested testing techniques.

Technique 1. Following a wet-stand period, a cell is discharged while monitoring the voltage between the zinc electrode and a Hg/HgO reference electrode. If the polarization at the zinc electrode signals the end of the discharge under a constant resistive load, either excessive amounts of zinc have been corroded such that the cell is no longer silver limited or the zinc electrode has become polarized due to insufficient transport of hydroxide ion to the surface of the zinc electrode. In both situations, the reaction at the zinc electrode will change to the evolution of oxygen gas. To distinguish between the depletion of zinc and the passivation of the zinc surface, two methods may be used. In the first method, the discharge rate would be reduced by a factor of 10. Any further discharge of a zinc-depleted electrode would not result in much additional capacity. If the zinc electrode was passivated due to the inability to transport hydroxide ion to the surface of the zinc electrode at a sufficient rate, further capacity should be available at the reduced rate of discharge. The second method depends on

the presence of undischarged zinc as well as undischarged silver still remaining in the case of zinc passivation. The remaining silver could be detected by discharging sample silver electrodes against fresh zinc electrodes in an excess amount of electrolyte.

Technique 2. Following a wet-stand period and cell discharge, the cell must be disassembled in a dry box to preclude the reaction of the zinc electrodes with the oxygen in the air. One or more zinc electrodes are immersed in an excess quantity of electrolyte and discharged against fresh silver electrodes. If the zinc electrodes are fully depleted, minimal amounts of capacity would be available. If the zinc electrodes had failed due to an insufficient rate of hydroxide movement to the surface or the cell was still silver limited, then additional amounts of zinc capacity would be available in the presence of excess electrolyte. In situations were large amounts of hydroxide ion are available, all of the zinc oxide or hydroxide can be solublized and the only thing left of the zinc electrode will be the silver substrate. As in Technique 1, the silver electrodes should be examined for remaining silver capacity.

Technique 3. The presence of undischarged zinc can also be determined by the addition of dilute acid that will result in the evolution of hydrogen gas. The volume of hydrogen gas can be directly related to the presence of zinc. Here again, care must be taken to preclude the loss of elemental zinc to air oxidation. As before, the silver electrodes must be examined for remaining silver capacity.

Technique 4. Based on the information in the preceding sections, the capacity trends during the wet-stand period can also be used to determine whether or not a cell design is hydroxide limited. In normal silver-limited cells, the capacity will degrade with time depending on the reactivity of the slightly soluble silver oxides with the oxidizable functional groups in the cellulosic material of the separator. If the capacity of cells were determined after different increments of time during the wet-stand period, the results might look similar to the trend, as suggested by Figure 3.

The slope of the line is related to the reactivity of the silver ions with the cellulosic separator material. At higher temperatures, the slope would be steeper. The cells are designed such that at the highest

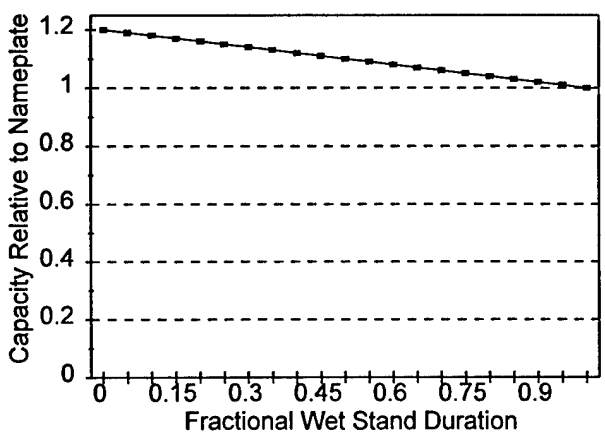

Figure 3. Capacity trend of normal silver-limited cells during the wet-stand period.

expected temperature, the cells would still be able to deliver the specified amount of usable capacity at the end of the specified wet-stand period.

In situations that are initially hydroxide limited, the capacity trend data from actual cells would be closer to the trend shown in Figure 4. In this trend plot, the capacity deteriorates at a rate that now depends on the rate of zinc corrosion. This rate will depend on the characteristics of the amalgamated zinc electrode material and the wet-stand temperature. When the saturation limit for the zincate ion is reached, the capacity trend line will level off since the corrosion reaction no longer consumes hydroxide ion. It is assumed here that the cells were initially silver limited.

In Figure 5, data from a group of cells that was affected by hydroxide limitations shows the same capacity trend as the theoretical one in Figure 4. After zinc had corroded to the point where 8 L of hydrogen had been generated, there is only a minimal additional drop in capacity to 1.3 V.

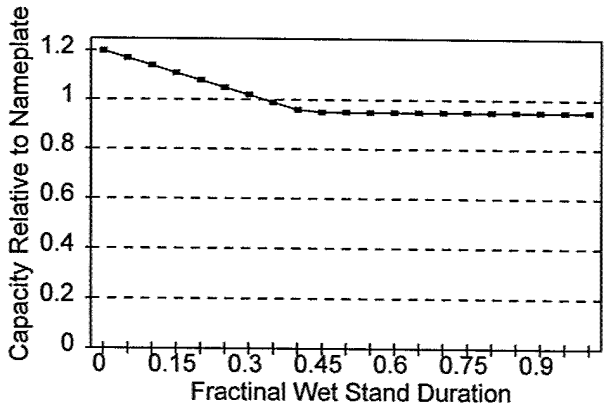

Figure 4. Capacity trend in cells that are hydroxide limited.

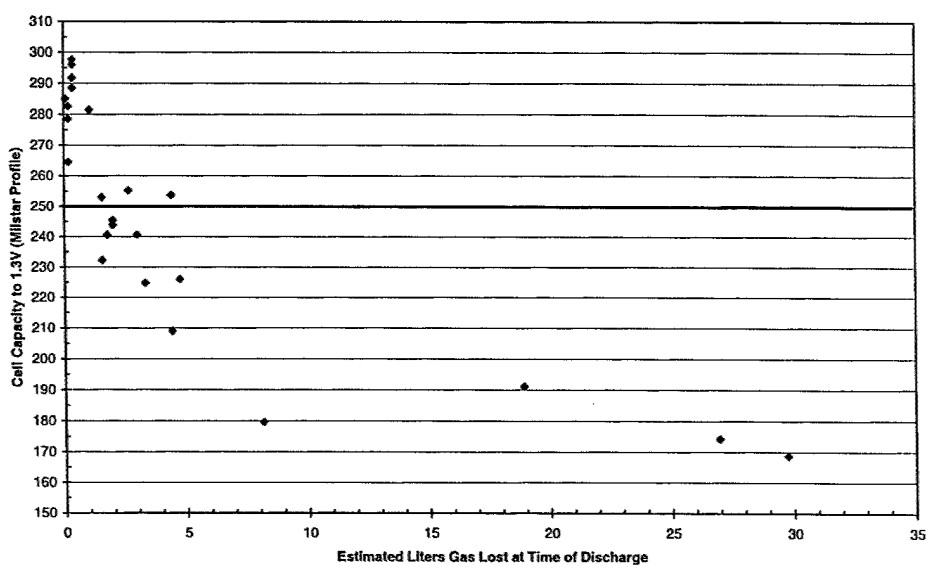

Figure 5. Capacity trend for cells affected by hydroxide limitations.

There are other possibilities that will not be plotted here. As an example, the cell may start out to be silver limited and not yet hydroxide limited. As the zinc corrodes, it may consume a sufficient amount of hydroxide such that the cell becomes hydroxide limited partway through the wet-stand period. It is essential that whatever limits the capacity of a silver-zinc cell, the capacity remaining at the end of the wet-stand period must be at or above the specified ampere-hour requirement for that cell design.

3.5 Sources of Hydroxide-Limited Cells

Some cells, by nature of their application, are designed to be hydroxide limited to minimize the amount of electrolyte added during the activation procedure. Batteries where the current pulsing requirements and not the ampere-hour requirements are of prime importance may turn out to be hydroxide limited. In these designs, the amount of electrolyte can be set during a series of cell discharges following the required wet-stand period. Using this technique, a series of cells are constructed with different amounts of electrolyte and then discharged following the end of the required wet-stand period. The cell design is then finalized by selecting the electrolyte quantity that permits the cell to deliver the required amount of capacity at the end of the wet-stand period.

In cell designs where the cell case is undersized for the amount of electrolyte added during the activation process, spewing from the exit valve can occur during the wet-stand period. In these situations, the amount of electrolyte is often reduced at the risk of converting the cell design to one that is hydroxide limited.

The techniques listed in the preceding paragraphs can be subject to a major source of error. If an increase in the rate of zinc corrosion occurs between the zinc electrode material that was used in the original test sequence, subsequent cell builds may suffer an increased rate of capacity loss of zinc material and/or the cell may undergo zinc passivation prior to the delivery of the required amount of capacity. If the cell would still be silver limited, this would not necessarily cause a problem if the cell did not become hydroxide limited. However, if the increased zinc corrosion converted the cell to being hydroxide limited, significant capacity shortfalls would be expected. In a situation where the saturation limit for zincate had already been exceeded, the impact of increased zinc corrosion would be described by reaction (5), which does not further deplete the hydroxide content of the cell. In this case, only zinc and water would be depleted as a consequence of the increased zinc corrosion. If the cell was originally dry in terms of liquid content, the extra loss of water might result in a cell that is subject to premature failure due to cell dry out.

Other cell designs that emphasize the amount of usable capacity over extended periods of discharge are also sensitive to limitations caused by insufficient amounts of liquid volume and/or hydroxide. As with cells that are designed for pulsing applications, cells for extended discharge applications are designed based on historical zinc corrosion rates and the desire to have high energy by minimizing the amount of electrolyte. In these cells, reaction (6) describes the corrosion process up until the solubility limit of zincate ion is reached. After that, reaction (5) can be assumed to take place. If the cell design was close to being hydroxide limited and the zinc corrosion rate was higher than expected, the cell may be hydroxide limited at the time of discharge. In this case, one would expect the useable cell capacity to drop linearly with time once the zinc corrosion caused a crossover to where the cell is now hydroxide limited. With increased corrosion, this capacity drop would be expected to cease at

the point where the corrosion reaction no longer consumes hydroxide ion, that is, reaction (5). The rate of capacity drop would depend on the rate of zinc corrosion.

If the cell design is silver limited, the cell capacity will initially remain constant as the zinc is corroded. As more zinc is corroded, and there is a continual loss of zinc, water, and hydroxide ion, the cell can become either hydroxide limited or zinc limited. Cells that are hydroxide limited or tend to become hydroxide limited can be converted back to designs that are silver limited by the addition of more hydroxide ion. To be able to do this, the cell must have enough void volume to accommodate the extra amount of electrolyte volume.

4. Summary

The chemical and electrochemical reactions involved with silver-zinc cells involve the participation of hydroxide ions as well as water molecules. As a consequence of this participation in the chemical and electrochemical reactions within the cell, the cell performance can become limited by the amount and concentration of electrolyte added during the activation procedure. These cells are referred to as hydroxide limited. Cells are typically designed to be silver limited, but some designs become hydroxide limited following extended periods of wet stand when excessive rates of zinc corrosion are present. This results in reduced amounts of cell capacity at the time the cells are discharged. To guard against this problem, care must be exercised during the amalgamation process to minimize the rate of reaction between the zinc and the electrolyte. Also, adequate free volume must be provided in the cell case to allow the use of an adequate amount of electrolyte.

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