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Special Tests and Destructive Physical Analyses Procedures as Used by The Aerospace Corporation with Nickel-Hydrogen Cells

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30 November 1992

Prepared for

THE NASA BATTERY STEERING COMMITTEE
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Engineering and Technology Group

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THE AEROSPACE CORPORATION
El Segundo, California

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ABSTRACT

The destructive physical analysis (DPA) of electrochemical devices is an important part of the overall test. Specific tests have been developed to investigate the degradation mode or the failure mechanism that surfaces during the course of a cell being assembled, acceptance tested, and life-cycle tested. The tests that have been developed are peculiar to the cell chemistry under investigation. Tests are often developed by an individual or group of researchers as a result of their particular interest in an unresolved failure mechanism or degradation mode. This report addresses a series of production, operational, and storage issues that have been addressed by the Electrochemistry Group at The Aerospace Corporation, mostly under the direction of Dr. Albert Zimmerman. As a result of these investigations, as well as associated research studies carried out to develop a clearer understanding of the nickel oxyhydroxide electrode, a series of unique and useful specialized tests has been developed. Some of these special tests have been assembled into this report to describe the methods that have been found to be particularly useful is resolving a wide spectrum of manufacturing, operational, and storage issues related to nickel-hydrogen cells. The general methodology of these tests is given here with references listed to provide the reader with a more detailed understanding of the tests. In Part I, the tests are classified according to the sequencing, starting with the impregnation of the nickel plaque material and culminating with the storage of completed cells. In the Part II of the report, the details of the wet chemical procedures that have been found to be useful because of their accuracy and reproducibility are given. At the end of Part II the equations used to make the appropriate calculations are listed.

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CONTENTS

FIGURES

TABLE

INTRODUCTION

Nickel-hydrogen technology has matured considerably over the last several decades. Experience with different manufacturers and cell designs has generated a number of different types of questions and problems. A variety of novel post-test analyses have been developed in attempts to understand and then resolve these problems and questions. The Aerospace Corporation has encountered a number of these issues as part of their support of Air Force and NASA programs. This document brings together a series of articles stemming from our experience in the field. Some have already appeared in the open literature, and some are currently internal documents that have yet to be converted into publications in the open literature. They were written to describe the specialized testing developed by Dr. Albert Zimmerman and his associates resulting from their investigations into a series of performance issues. The reader is also referred to the recent writings of H. Lim (Hughes Aircraft) and H. Vaidyanathan (Comsat Corporation). Both these researchers have developed their own set of unique analytical techniques as they investigated performance issues in nickel-hydrogen cells that were part of their own studies.

The tests described herein are arranged along the path a cell would follow as it progresses from the unimpregnated plaque, to the finished plate, through acceptance testing as a completed cell, then storage, cycling tests, and finally DPA following testing or failure.

Viewed in this sequence, the problems that have been associated with nickel-hydrogen cells can roughly be divided between:

1. Manufacturing issues prior to assembly.
2. Cell assembly and acceptance problems.
3. Capacity or performance degradation during storage.
4. Performance degradation during life-cycle testing.

Each one of these major categories is discussed further in order to make the significance of the specialized tests more meaningful. References are made to works written describing the techniques developed to resolve several critical performance issues. Several references discuss different aspects of the same problem. Table 1 is a listing of several tiers of issues that have been found in nickel-hydrogen cells.

Rejections of individual components during the manufacturing process usually result from workmanship issues and will not be addressed in this report. The rejections that will be covered often result from component screening tests that are done as part of the quality assurance process. Rejections at this phase are usually due to quality problems associated with the nickel electrode plaque manufacturing process or the impregnation process. These can result in poor nickel plate capacity and could affect the ultimate performance or life cycle of a completed cell.

Table 1. Historical Nickel-Hydrogen Issues

IMMEDIATE SYMPTOMS
- Rejection during manufacture
- Capacity loss during storage
- Loss of usable capacity during test
- Increase in overcharge requirements
- Loss of case-to-electrode isolation

LOSS OF USEABLE CAPACITY
- Increase in second plateau (Delta V = 0.6 V)
- Increase in internal resistance
- Other?

INCREASE IN INTERNAL RESISTANCE
- Breakup of sinter material
- Conversion to nonconductive form of active material
- Oxidation of sinter to a non-cobalt-containing film
- Hydrogen electrode flooding
- Electrolyte maldistribution
- Nickel electrode contamination

INCREASE IN OVERCHARGE REQUIREMENTS
- Internal short circuits
- Other?

CHANGES OVER THE COURSE OF THE CYCLING
- Amount of precharge
- Percent utilization
- Internal resistance
- Electrolyte concentration
- Hydrogen pressure

QUALITY OF INITIAL COMPONENTS
- Cathode
- Anode
- Separator
- Electrolyte
- Feed throughs

DESIGN FEATURES
- Electrolyte quantity
- Electrolyte concentration
- Stack compression
- Stack expansion
- Precharge adjustment
- Current density
- Loading level of active material
- Sinter character
- Cobalt level

A charge retention test is usually among the tests applied to a completed cell. It typically consists of a 72-hr charged stand and is designed to identify cells having low-grade short circuits. There can also be seal leaks, weld leaks, and other mechanical quality issues, but the capacity as a function of temperature and charge retention parameters are the major electrochemical factors that have been used to evaluate the quality of a cell.

Degradation following extended periods of storage has affected a number of cell types. The understanding and ultimate solution of this problem is critical to the wider use of this technology. The fuller understanding of this situation has resulted from unique chemical and electrochemical studies directed toward the nickel electrode, its chemistry, its electrochemistry, and its reactivity with oxygen and hydrogen gas.

Performance degradation during life-cycle testing is to be expected, but the rate of this degradation is often indicative of an inappropriate cell design or a latent component quality problem that went undetected during manufacturing and acceptance testing.

Although the issues listed in Table 1 are extensive, they are not intended to be an exhaustive collection of problems that have been observed. Fortunately, the majority of the problems have been traced to the nickel electrode. Some of these problems, and the analytical techniques developed to screen for them will now be addressed.

PART I — SPECIALIZED TESTING FOR NICKEL-HYDROGEN PERFORMANCE-RELATED ISSUES

A. MANUFACTURING ISSUES PRIOR TO ASSEMBLY

Nickel plaque material is typically specified according to the desired degree of porosity. This number averages about 80%, usually lower for plaque made by the slurry process and somewhat higher for plaque made by the dry-powder process. Plaque material can be improperly made and still have the proper value of overall porosity. Desirable plaque material has a particular pore size distribution that coincidentally has an associated value for the porosity percentage. However, the reverse is not true. That is, material with a certain overall porosity will not necessarily have the proper pore size distribution. Material that has been sintered improperly (too high a temperature and too high a belt speed for instance) can result in a product that has a surface layer made up of very fine pore sizes. The interior of this material can be more porous than usual due to being under-sintered such that, on an overall basis, the material has the specified porosity. If this situation is not detected, impregnation problems can result. The technique[1] that has been developed and used to detect and measure this and other anomalous sinter conditions is similar to the principle used in the Scanning Tunneling Microscope (STM).

No published papers are as yet available in the open literature, but a brief description of this technique can be given that outlines the principles on which it is based. A test specimen consisting of a sample of sinter material, about two inches long, is first mounted in epoxy, cross-sectioned, and polished smooth. The sample is next mounted on a translation stage capable of movement in the x and y directions, and having a movement resolution of at least 0.1 μm. A platinum metal probe having a finely cut point is attached just above the sample to another translation stage capable of movement in the z direction. A 10-V power supply is then connected in series with a 10 kΩ resistor between the sample and the probe. The power supply wire is connected to the sinter sample using silver-filled paste. As the probe is lowered to touch the sample, conductivity is measured between the probe and the sample if the probe contacts a sinter partial. If the probe contacts an epoxy-filled void region, no conductivity is observed. To scan the pores in a given cross-sectional region of the sinter requires 5000 to 7500 points across the thickness of a 700 to 800 μm thick sample. The distance between measurements is typically 0.1 to 0.15 μm, and the probe tip radius is typically about 0.1 μm. Following the collection of information across 30 to 40 scans of the thickness of the plaque, the information is analyzed using a personal computer routine. The results can provide a quantitative measure of the homogeneity of porosity and pore size distribution across the width of the sinter.

Impregnated plaque material is usually called plate. The amount of active material deposited either chemically or electrochemically into the plaque can be specified in terms of grams per cubic centimeter of void or in terms of grams per square cm of frontal area of plate. It is typically measured according to the weight pickup of plaque material that has a given porosity. Here again, it is generally assumed that the active material has been deposited evenly throughout the interior of the plate. Finished plate is tested for usable capacity per unit area as a way of measuring the suitability of the plate for inclusion into the assembly process for the cell. Problems arise when active material has been disproportionately deposited near the surface of the plate. This type of plate will have very low porosity near the surfaces and thus poor mass transport characteristics. This type of plate generally has poor capacity per unit of frontal area. The technique[2] used to verify the existence of this maldistribution is called profiling. By using an ion microprobe analysis across the width of the electrode, the loading profile of active material can be detected. Figure 1 shows the results obtained during these studies on an electrode from the Hubble Space Telescope (HST) program. The areas selected for analyses across the width of the electrode were 40 by 50 µm. Both the cobalt and nickel content were determined to verify that it was active material. Scanning electron microscope photos of this same electrode (Figure 2) qualitatively show the presence of higher loading at the front and back surfaces.

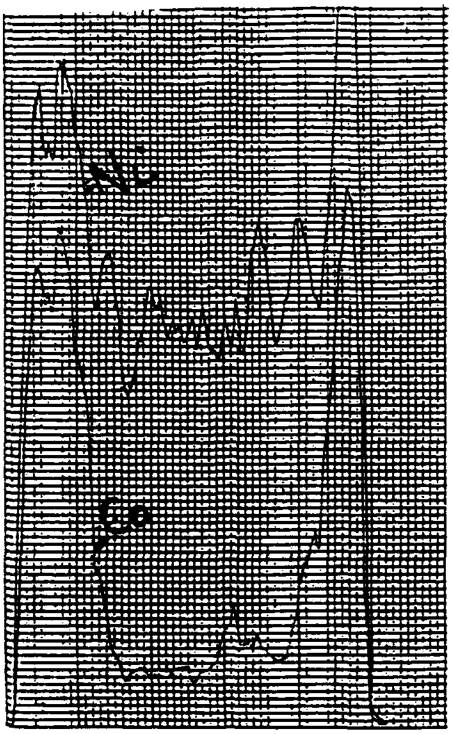

Figure 1. Nickel and cobalt loadings showing high surface concentrations.

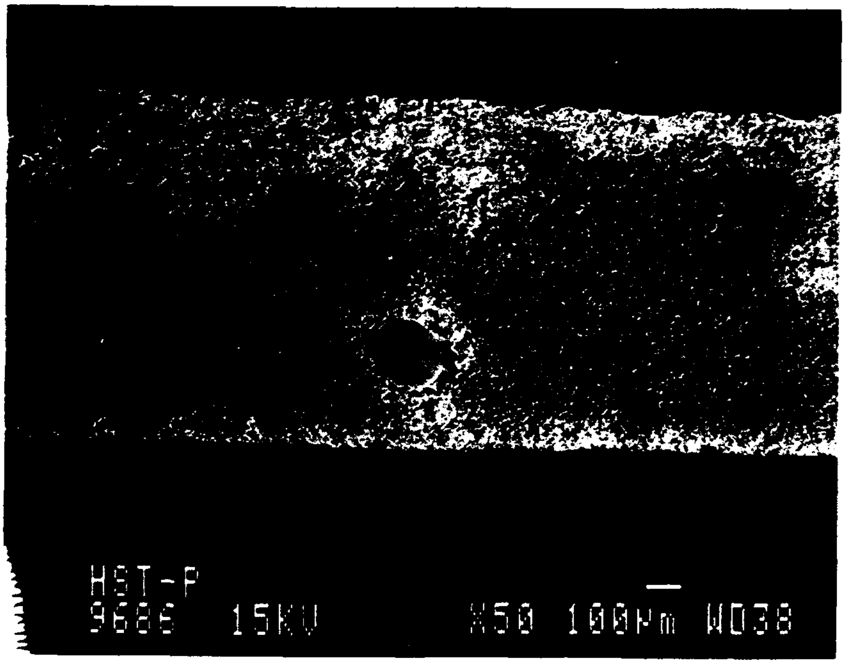

Figure 2. SEM photo indicating higher surface loadings.

B. CELL ASSEMBLY AND ACCEPTANCE PROBLEMS

The intent of screening tests is to ensure that only quality plaque material that has been converted into properly impregnated plates is assembled into finished cells. Following assembly into cells and the addition of electrolyte, an activation procedure that is particular to the manufacturer is carried out. Following this, cells typically undergo (among many other tests) a 72-hr capacity retention test. The purpose of this test is to identify cells that have low-level short circuits. The rate of capacity loss as a function of temperature and hydrogen pressure is reasonably well known. This permits the capacity remaining after the 72-hr stand test to be evaluated against the known rate of self discharge in the absence of any short circuits. Unfortunately, this test has not been found to be as sensitive as required to exclude cells that had potentially significant short circuits. Reference 3 describes the method as reported by Zimmerman and coworkers that permits the accurate measurement of low-level short circuits that might otherwise have gone undetected using the typical capacity retention requirements of a 72-hr stand test.

The test is based on the fact that at very low rates of charge, a cell without any short circuits would tend to have a more rapid increase in its voltage and capacity than a cell with a short circuit. In the actual test, as described in full detail in Ref. 3, a cell is first fully and completely discharged to 0.0 V. Next, the cell is charged by stepping the voltage in increments of about 50 mV to 1.0 V and then back down to 0.0 V. The cell is held at each voltage level for 8 hr to assure that a steady-state condition has been established. The current at the end of the 8-hr waiting period is recorded. If large numbers of cells are to be tested, modified procedures are suggested to analyze for short circuits. These screening tests may be used either on a routine basis for screening, or used to optimize the cell activation procedure. This test can also indicate the amount of hydrogen or oxygen in the discharged cell by the shape and magnitude of the current vs. voltage (I/V) curve.

Another technique introduced in Ref. 3 is called electrochemical voltage spectroscopy (EVS). This technique is also described in Refs. 4 through 7. Reference 4 gives an excellent discussion of the technique. This technique uses a computer to carry out a very slow cyclic voltammogram. Where the more usual cyclic voltammogram sweeps through a complete cycle in a few seconds or less, an EVS scan can take as long as several days. By cycling at such a slow rate, any mass transport limitations are eliminated, and quasi-steady-state conditions will be present for even very slow reactions. Typically, a 1-cm^2 sample is used in a flooded cell. Since these tests are associated with alkaline cells, a KOH solution is used as the electrolyte, and a Hg/HgO electrode serves as the reference. The starting potential, the ending potential, the size of the voltage steps, and the criteria for going to the next voltage level are all preselected using the computer program. Once started, the experiment is fully under the control of the computer. As the data is collected, the amount of charge passed (dQ/dV) at each voltage step is plotted as a function of the applied potential. References 4 through 7 give a more complete explanation of this very useful technique, as well as describe the instrumentation and computer software needed to carry out the test. This technique is also mentioned in later portions of this report since it is very useful in evaluating residual charge in nickel electrode samples as well as impurities, or new electroactive species.

A further test has been found to be useful in detecting low-level loss of isolation between the metallic portions of the stack and the case wall. There are no current references on this technique. It is based on the fact that the cell case in contact with hydrogen gas is an easily polarized hydrogen electrode. If the potential between the positive terminal and the case is measured with an extremely high-impedance voltmeter, it should be exactly the same as the reading between the two cell terminals if there is complete isolation. If there is the least amount of metallic continuity between the stack and the case, the "hydrogen" electrode of the wall will be highly polarized, and the reading will be lower than the cell potential. In several instances, this low-level loss of isolation has resulted in case corrosion sufficient to cause a leak in the pressure vessel. This simple screen test is capable of detecting such a condition prior to failure. Gross short circuiting problems have occurred from time to time as a result of wire barbs or electrode leads touching the cell wall, but these are very easily detected by existing acceptance tests, and can often result in severe damage to the cell stack.

C. CAPACITY OR PERFORMANCE DEGRADATION DURING STORAGE

Once cells pass the acceptance-level screening tests, a new set of problems can beset them. If the cell has been designed with an excess of hydrogen gas relative to the amount of nickel active material, it is referred to as a cell with hydrogen precharge. When these cells are fully discharged, the residual (precharge) hydrogen gas can react with the active material within the positive electrode. When the positive electrode is above about 0.5 V relative to the hydrogen electrode, it is protected from being reduced by hydrogen. However, when the electrode is fully discharged, the hydrogen can be oxidized on the nickel sinter material and electrochemically reduce the discharged active material down to the metallic state. In this situation, the nickel sinter is acting as a hydrogen electrode. The active material at this depth of discharge and in the immediate vicinity of the nickel sinter substrate is a solid solution of nickel hydroxide containing from 5 to 10% of cobalt hydroxide. This material is reduced to a mixture of metallic nickel and metallic cobalt. Upon recharging the cell, a mixture of nickel and cobalt oxides are formed, which are no longer in solid solution. This material will have modified electrochemical performance and can significantly reduce the capacity of the remaining normal material.

Reference 5 describes the unique analytical technique that was developed to establish the existence of a cobalt-depleted layer of active material adjacent to the conductive nickel plaque and, at the same time, a cobalt-enriched region away from the substrate. This reference describes a sophisticated technique using a modern energy-dispersive X-ray microscope (EDAX). The cobalt content was profiled across a number of pores within an electrode that had shown severe capacity loss following storage under conditions where the residual hydrogen was present in a fully discharged cell. Once this had been demonstrated, it was further shown[5] that the "segregated cobalt" material is present as a mixed oxide with nickel. This oxide can be separated from the normal active material since it is insoluble in dilute acetic acid. This analysis is described in the analytical portion of this report.

One response to the problem of capacity loss in hydrogen precharge cells during storage is to not store them under conditions where they are discharged and shorted. Keeping the positive electrode in the partially charged condition (cell voltage above about 0.5 V) will prevent this type of reaction. Another approach to the cobalt segregation problem is to design the cell to have excess positive material. This is referred to as nickel precharge. Although this arrangement precludes the reduction of the positive material to the metallic state, it does permit the oxidation of the platinum material used as catalyst in the hydrogen electrode. Soluble platinum species, possibly in the +4 valence state ($Pt(OH)_6^{=}$), move to the nickel electrode, and have been found in the form of mixed nickel-cobalt-platinum oxides. This phenomenon and the analytical techniques used to verify its presence are discussed more fully in Reference 6. Although the presence of this material is not known to be detrimental to the performance of the cell, it is capable of compromising the normal nickel precharge, and its long-term effects are not fully known at this time. Here again, platinum dissolution can be prevented by not allowing the potential of the hydrogen electrode to be pulled up to that of the partially charged nickel electrode, i.e., keeping the cell potential above 0.5 V during storage.

D. PERFORMANCE DEGRADATION DURING LIFE-CYCLE TESTING

At this point, cells have passed all their quality and acceptance tests and have been stored properly. Several problems have been detected once testing, or testing and storage periods, have taken place. One is associated with the loss of the nickel precharge that had been built into the cell during their construction. Under ideal conditions, when a cell with positive precharge is discharged, all of the hydrogen will be consumed prior to the exhaustion of the charged nickel material. If the discharge were to continue, the negative electrode would change from oxidizing hydrogen gas to oxidizing the electrolyte to generate oxygen. There would be an abrupt drop in the cell voltage of that electrode at this point (about 1.6 V). The positive electrode would continue to discharge until exhaustion and then convert to evolving hydrogen. If the cell had been built with hydrogen precharge and it were to be fully discharged, the positive electrode would begin to evolve hydrogen at the same rate that the negative electrode was consuming hydrogen; thus, the cell voltage would be much closer to zero compared to the case where the sum of the electrode reaction would be the electrolysis of water. This is one way of determining whether a cell has a hydrogen precharge or a nickel precharge.

Another less aggressive method of determining whether a cell has a net positive or negative precharge uses a cell that has been discharged and shorted down for several hours and then placed at open circuit for several more hours. The cell is then given a slow recharge at about the C/100 rate for several minutes. If the cell contains a hydrogen precharge, the negative electrode will already be at the hydrogen potential, and the cell voltage will quickly rise to about 1.4 V. On the other hand, if the cell still has a positive precharge, the negative electrode will be in an oxidized state and close to the oxygen electrode potential. It will have a much lower starting voltage at the beginning of recharge as the oxides are reduced prior to hydrogen evolution. This is explained more fully in Ref. 2.

A third method involves the disassembly of a cell following a normal 20°C capacity cycle. In this method, a cell is fully discharged and then shorted across a 0.25 Ω resister for a day. After cutting open the cell, using the usual methods, an electrode is discharged against a nickel sheet using a power supply. In this flooded beaker test (more fully outlined in Part II Paragraph C.), a Hg/HgO reference electrode should be used. If there is positive precharge remaining, the electrode will continue to discharge nickel material and then convert with an abrupt change in half-cell potential when hydrogen evolution begins.

Knowledge of the precharge condition is important because it has been found that cells that are originally built with positive precharge, are very easily converted to where they have an apparent negative precharge. Certain procedural sequences are suspect in being able to convert cells with positive precharge into cells that act as if they have negative precharge. When cells are allowed to self discharge, the remaining active material is distributed differently from a cell that had been electrically discharged. Subsequent electrochemical discharge of the remainder of the active material will result in "pockets" of charged active material located away from the conductive sinter material that can not be discharged at the normal current densities used for cell discharge. Since the potential of the nickel electrode is controlled by the material at the interface between the fully discharged material and the nickel sinter, the potential will be low, and the nickel electrode will appear to be fully discharged. However, since there still is some undischarged active material electrically isolated from the nickel sinter, there will be an equivalent amount of unconsumed hydrogen. Under these conditions, the remaining hydrogen can react with the fully discharged portions of the active material adjacent to the nickel sinter in the way that results in classical cobalt segregation. The amount of temporary and permanent capacity loss that takes place in these situations is not entirely predictable, but conditions that result in the conversion of a positive precharge cell into a negative precharge cell should be avoided.

Pockets of active material can be analyzed for in several ways. If the cell is disassembled, samples of plate material can be ground up and subjected to a chemical analysis technique that measures the residual oxidizing power of the material. A second technique is to carry out a very slow reduction scan using the EVS technique already described. A third method involves the complete cell if the presence of undischargeable active material is suspected as a consequence of a reduction of the cell's measured capacity. A recharge using a standard charging sequence should restore the active material to its normal charged form, which is also very much more conductive than the fully discharged form. A subsequent electrochemical discharge at a normal rate, followed by a series of progressively lower discharge rates to lower and lower cutoff voltages should reveal the cell's full capacity. When active material is discharged electrochemically, discharge ideally begins from the outside of the particles and progressively works in towards the conductive sinter particles. This leaves behind very little undischarged active material. The difference in these two capacity values is indicative of the amount of active material that had been in an undischargeable condition following the previous sequence of self discharge and discharge. This test should be carried out without an extended intervening storage period, which could lead to permanent capacity losses.

PART II — FAILURE ANALYSIS PROCEDURE FOR NICKEL ELECTRODES IN NICKEL-HYDROGEN CELLS

A failure analysis procedure has been developed by The Aerospace Corporation to aid in determining the underlying causes for degraded nickel electrode performance in nickel-hydrogen battery cells. The procedure is a two part analysis. The first part of the procedure consists of electrochemical measurements that determine the performance characteristics of the nickel electrode. Performance variables are identified through flooded capacity measurements using standard charge and discharge rates, measurements of residual capacity, and Electrochemical Voltage Spectroscopy (EVS). In the second part of the procedure, the electrode is chemically analyzed to determine the overall chemical components and makeup of the positive plate (cobalt doping level, loading level, sinter weight, porosity, water content, etc.) The procedure for the chemical analysis is described below.

A. PRIOR TO THE ELECTROCHEMICAL TEST

Prepare a 1-cm^2 sample cut from the nickel electrode. Record the weight of the sample. The surface area should be calculated and recorded with the measurements estimated to 0.1 mm. A micrometer is used to determine the sample thickness. Several thickness measurements (estimated to 0.0001 in.) should be made across the width of the sample and the average recorded.

B. FLOODED ELECTROCHEMICAL CHARACTERIZATION TESTS

The test is done in plastic cells sealed in plastic bags to exclude the interaction of air with the KOH solution (~31%). The 1.0-cm x 1.0-cm samples are placed in a flooded cell equipped with a mercuric oxide reference electrode and nickel sheet counter electrode. The purpose of the test is to evaluate high-rate and low-rate utilization of the active material as well as the ease by which the nickel hydroxide can be converted to the beta- and gamma-phases of charged material using a standard cycling regime. First, however, the electrode must be completely discharged to recover any residual charge still present. This residual charge could be due to the cell not being fully discharged prior to disassembly or the presence of nickel precharge. The discharge sequence is as follows: 2 mA/cm^2 discharge to 0.5 V followed by a 0.2 mA/cm^2 to 0.4 V. This two-step discharge sequence will be referred to as "A". The electrode under these conditions is fully discharged, barring the presence of inaccessible active material within the electrode. A charging sequence of 2.0 mA/cm^2 for 16 hr is intended to minimize the amount of overcharge during this first portion of the test. The electrode is then discharged at 10 mA/cm^2 to a 0.6-V cutoff followed by the two-step discharge referred to as "A". A second, more aggressive, charging sequence of 2.0 mA/cm^2 for 32 hr follows. It is the intent of this step to convert a significant portion of the charged active material to the gamma phase. The final step is to discharge the electrode using the three-step sequence described in the previous step. The amount of capacity withdrawn during each portion of the two different discharge sequences permits the beta- and gamma-phase utilizations to be determined when coupled with the chemical analyses of the following sections. Inferences as to the nature of the problems that may be associated with the electrode can be made by comparing these utilizations to new electrodes or electrodes from control cells.

C. POST ELECTROCHEMICAL TESTS

Rinse the sample in fresh, deionized water until the presence of KOH can no longer be detected (a pH of about 7 as indicated by pH paper). Remove the sample and pat dry on absorbent paper. Let the sample air dry overnight. The weight and thickness should again be recorded.

Place the sample in a clean, weighed weighing bottle with a top. Place the sample, bottle, and top in a drying oven for 4 hr at 95°C. Remove the hot sample, bottle, and top and place in a desiccator overnight. The dry weight of the cooled sample should be recorded.

To remove the sinter from the screen, place the sample in a mortar, place the mortar on a large sheet of white paper, and place a finger glove on one finger. Using the gloved finger to hold the sample in place, the sinter can be separated from the screen with an X-ACTO knife. When finished, scrape clean the surface of the screen and record the weight of the screen. Collect and return any pieces of sinter that may have been thrown out of the mortar onto the paper.

The sample is now ready to be pulverized. Add a few drops of deionized water to the sample and begin grinding the slurry using the pestle. The result of the grinding should be a very fine slurry.

Measure out 85 ml of 10% acetic acid. Using deionized water and a small portion of the acetic acid as rinses, quantitatively transfer the ground slurry from the mortar and pestle into a 250-ml beaker. Measure out 0.75 gm of hydrazine sulfate. Add the remaining acetic acid and the hydrazine sulfate to the beaker along with a large Teflon-coated stirring bar. Cover the beaker with Parafilm, place on a stirring plate, and stir for 1 hr without heating. The solution should be clear at the end of the hour, with the active material in solution and the undissolved nickel sinter attached to the magnetic stirring bar.

Obtain two 250-ml volumetric flasks. Pour the contents of the beaker into the first flask (#1) while preventing the stirring bar from falling into the funnel. Do five rinses of the beaker and stirring bar and add the rinses to the first flask. Dilute the volumetric flask to the fill mark. This flask will contain the active material (nickel hydroxide and cobalt oxyhydroxide) in solution.

Add 70 ml of 0.15M nitric acid to the beaker containing the stirring bar. Cover the beaker with a watchglass, then heat and gently stir the beaker until all the black particles have gone into solution. Let the solution cool to warm, then filter it through a weighed Millipore filter. All the filtrations should be done under vacuum, using a Nalgene nylon membrane filter (47 mm diameter, 0.45 µm pore size) and a coarse porosity, fritted-glass filter support base. Transfer the solution from the filter flask into the second 250-ml volumetric flask. Dilute to the fill mark. This flask will contain the dissolved nickel sinter material. Remove the filter containing the insoluble nickel sinter residue and air dry, then place in a desiccator overnight. Record the weight of the residue.

D. ANALYSIS FOR NICKEL CONTENT

The nickel in the active material and the nickel sinter will be determined gravimetrically by chelation with dimethylglyoxime (DMG).

Volumetrically pipette into a 250-ml beaker a 40-ml aliquot from the dissolved nickel sinter solution or a 50-ml aliquot from the active material solution. Add 25 ml of 10% acetic acid to the solution in the beaker. Dilute to the 100 ml mark using deionized water. Neutralize with concentrated ammonium hydroxide to a pH ≈ 8. This is indicated by the first color change to a light-blue color.

Prepare a 0.04M solution of DMG. (To prepare the DMG solution weigh out the proper amount of DMG and dissolve in reagent grade ethanol.) Add 35 ml of the 0.04M DMG to the beaker, at which point a bright-red precipitate will appear. Cover the beaker with a watchglass and let sit for 10 min.

Quantitatively filter the solution through a weighed Millipore filter and rinse thoroughly. Remove the filter and air dry, then place in a desiccator overnight. Record the weight of the precipitate (wt ppt DMG).

E. ANALYSIS FOR COBALT CONTENT

The cobalt in the active material will be gravimetrically determined by chelation with 1-nitroso-2-napthol (1N2N).

Quantitatively transfer the remaining contents from flask #1 (the pre-rinse from the volumetric pipette used in the active material analysis), the final rinse from the volumetric pipette, and any rinse from the stopper into a 1000-ml beaker, and add 100 ml glacial acetic acid to the beaker.

Prepare a 0.05M solution of 1N2N. (The solvent used to prepare the 1N2N solution is 50% glacial acetic acid and 50% deionized water, with the 1N2N first being dissolved in the glacial acetic acid. The volumetric flask is then filled to the mark with deionized water.) Add 10 ml of the 1N2N solution to the beaker, cover with a watchglass, and then heat the solution to just below boiling for about 15 min. A reddish-brown precipitate should appear. Remove the beaker and let cool to very warm.

Quantitatively filter through a weighed Millipore filter, remove the filter to air dry, then place in a desiccator overnight. Record the weight of the precipitate (wt ppt 1N2N).

F. PROCEDURE FOR THE MAGNETIC SEPARATION OF ACTIVE MATERIAL FROM THE NICKEL SINTER

This is a procedure used to prepare a large sample of the active nickel material for analysis by magnetically separating the active nickel material from the nickel sinter. With no reducing agent added for the analysis of the insoluble residue from the active nickel material (i.e., hydrazine sulfate), the plate that the sample is cut from must be in a completely discharged state.

Cut a large sample (about 1/4 of the nickel electrode plate) from a rinsed and air-dried nickel electrode. Follow the procedure used above to remove the electrode material from the screen. Add a few drops of deionized water to the mortar, then grind the sample into a very fine slurry. Transfer the slurry into a 1000-ml beaker with deionized water.

Add 200 ml deionized water to the beaker. Place a large stirring bar in the beaker and stir vigorously for 10 min. Remove the stirring bar and wash the filings on the bar into a second 1000-ml beaker. Return the clean bar to the first beaker and again stir vigorously for 5 min. Remove the bar and again wash the filings into the second beaker.

Place the bar into the second beaker, add 100 ml deionized water and stir vigorously for 5 min. Remove the bar and wash the filings into a small beaker. Return the bar to the second beaker and repeat the process.

Combine the contents of the first and second beaker and filter through an unweighed Millipore filter. Remove the filter and let air dry overnight. Scrape the filtered material into a clean, weighed glass vial and record the weight of the active nickel material.

A portion of the active nickel material can be used for analysis by X-ray diffraction and semi-quantitative spectrographic analysis. Additional analysis can be performed to detect stable oxides or other acid-insoluble contaminants in the active material. This procedure follows.

G. PROCEDURE FOR ANALYSIS OF INSOLUBLE RESIDUE FROM ACTIVE NICKEL MATERIAL

Place a weighed amount (~ 0.5 g) of the separated active material in a 1000-ml beaker. Add 500 ml 10% acetic acid. Heat on a hot plate for 4 hr at about 70°C (no boiling). Let the solution cool to warm, then filter through a weighed Millipore filter. Remove filter and allow to air dry. Place the filter in a desiccator overnight and record the weight of the precipitate.

The insoluble residue can be analyzed by EDAX or spectrographic methods.

H. CALCULATIONS

(1) Ni Sinter wt (g) = (wt ppt DMG) (250/Vol aliquot) (0.20319)

(2) Ni Active mass wt (g) = (wt ppt DMG) (250/Vol aliquot) (0.20319)

(3) Co Active mass wt (g) = (wt ppt 1N2N) (250/Vol aliquot) (0.10241)

(4) $$\mathrm{Ni(OH)_2\ wt_{active}\ (g)} = \frac{\mathrm{(wt\ Ni\ active\ mass)\ (f.w.\ Ni(OH_2))}}{\mathrm{(a.w.\ Ni)}}$$

(5) $$\mathrm{CoOOH\ wt_{active}\ (g)} = \frac{\mathrm{(wt\ Co\ active\ mass)\ (f.w.\ CoOOH)}}{\mathrm{(a.w\ Co)}}$$

(6) Residual $\mathrm{wt_{active}}$ (g) = dry wt - [screen wt + Ni metal $\mathrm{wt_{sinter}}$ + $\mathrm{Ni(OH)_2}$ $\mathrm{wt_{active}}$ + CoOOH $\mathrm{wt_{active}}$]

(7) Ni metal $\mathrm{wt_{sinter}}$ (g) = (Ni sinter wt) + (Insoluble Ni sinter residue wt x 0.7858)

(8) $$\%\ \text{Water}_{\text{active}}\ (\%) = \frac{(\text{sample wt}_{\text{air dry}} + \text{residual wt}_{\text{active}}) - \text{dry wt}}{\text{wt}_{\text{active}}\ \text{Ni(OH)}_2 + \text{wt}_{\text{active}}\ \text{CoOOH}} \times 100$$

(9) $$\%\ \text{Porosity}_{\text{sinter}}\ (\%) = [1 - ((\text{Ni metal wt}_{\text{sinter}}/8.902\ \text{g/cm}^3)/\ \text{Volume (cm}^3))] \times 100\%$$

(10) $$\%\ \text{Porosity}_{\text{plaque}}\ (\%) = [1 - (((\text{Ni metal wt}_{\text{sinter}} + \text{screen wt})/8.902\ \text{g/cm}^3)/\ \text{Volume (cm}^3))] \times 100$$

(11) $$\%\ \text{Porosity}_{\text{electrode}}\ (\%) = [1 - [(((\text{Ni metal wt}_{\text{sinter}} + \text{screen wt})/8.902\ \text{g/cm}^3) + ((\text{Ni(OH)}_2\ \text{wt}_{\text{active}} + \text{CoOOH}_{\text{active}})/3.6\ \text{g/cm}^3)))/\ \text{Volume (cm}^3)]] \times 100$$

(12) $$\text{Loading level sinter (g/ccv)} = \frac{(\text{Ni(OH)}_2\ \text{wt}_{\text{active}} + \text{CoOOH wt}_{\text{active}}}{((\%\ \text{porosity}_{\text{plaque}} \times \text{Volume (cm}^3))/\ 100)}$$

(13) $$\text{Loading level sinter (g/cm}^2) = (\text{Ni(OH)}_2\ \text{wt}_{\text{active}} + \text{CoOOH wt}_{\text{active}}\ /\ \text{Area (cm}^2)$$

(14) $$\text{Sinter wt (g/dm}^2) = (\text{Ni metal wt}_{\text{sinter}} \times 100)/\ \text{Area (cm}^2)$$

(15) $$\%\ \text{Wt Co}_{\text{active}}\ (\%) = \frac{(\text{CoOOH wt}_{\text{active}})}{(\text{Ni(OH)}_2\ \text{wt}_{\text{active}} + \text{CoOOH wt}_{\text{active}})} \times 100$$

(16) $$\text{Theoretical Capacity (mAh)} = (\text{Ni(OH)}_2\ \text{wt}_{\text{active}} \times 289.06) + (\text{CoOOH}_{\text{active}} \times 291.52)$$

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   - Application of Electrochemical Voltage Spectroscopy
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   - Technique for detecting segregated cobalt species

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