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Separator Development and Testing of Nickel-Hydrogen Cells

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SEPARATOR DEVELOPMENT AND TESTING OF NICKEL-HYDROGEN CELLS

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ABSTRACT

The overall objective of the technology development program at the Lewis Research Center is to improve the components, design, and operating characteristics of Ni-H₂ cells and batteries. As part of this effort a separator development program was designed to develop a separator that is resistant to penetration by oxygen and loose active material from the nickel electrode, while retaining the required chemical and thermal stability, reservoir capability, and high ionic conductivity. The performance of the separators in terms of cell operating voltage was to at least match that of state-of-the-art separators while eliminating the present separator problems. The separators were submitted to initial screening tests and those which successfully completed the tests were built into Ni-H₂ cells for short term testing. The separators with the best performance will be tested for long term performance and life.

INTRODUCTION

Two types of separator materials are used in state-of-the-art (SOA) nickel-hydrogen (Ni-H₂) cells. They are fuel cell grade asbestos and zircar cloth (ZYK-15). It is known that the separator affects the voltage characteristics of the cells and the extent of rapid oxygen recombination damage to the negative electrodes (1). Therefore, it was necessary to take a closer look at the SOA separators, their properties and problem areas to determine the necessary characteristics for new, improved separators.

The problem areas identified for the two SOA separators included rapid oxygen recombination, and electrolyte management for zircar; and long-term degradation, electrolyte management, and possible unavailability of the materials for the asbestos separator. Separator characteristics were studied to establish those which make the separators perform effectively from a short-term point of view; those properties being resistance, electrolyte retention, pore size, porosity, electrolyte diffusion and bubble pressure. A set of standard requirements were identified which

will provide the necessary characteristics for short-term performance while solving the problems related with the long-term performance of SOA separators. All candidate separators were submitted to screening tests to measure the previously mentioned properties.

Since the zirconium oxide (ZrO₂) and asbestos fibers are the standard fibers used in SOA separators, our study started by characterizing separators composed of these two fibers in different compositions. Other fibers were studied and because of availability, their effect on final mat characteristics were studied initially in conjunction with asbestos fibers. Different fillers and binders were also studied, these were first screened to ensure chemical stability in the cell environment and the 31 percent KOH solution. Available commercial separators were also studied. Only one commercial separator, polysulfone, from Gelman Science (HT-200C) met most of our separator requirements and was recommended for cell tests.

The separators which met the design criteria were made up into Ni-H₂ stacks and put into pressure vessels to examine their voltage characteristics and cell performance. These separators were submitted to the same characterization tests as the baseline ZrO₂ cloth and asbestos. Three cells of each selected separator were tested and are being subjected to further cycling. Results of the characterization tests will be presented.

The separator program is a continuing effort to improve on separator characteristics. Different fabrication methods are presently being studied to improve separators. New fibers and commercial separators will be studied as they become available.

MATERIALS

The fibers studied were: (1) fuel cell grade asbestos (FCGA), (2) zirconium oxide (ZrO₂), and (3) pigmentary potassium titanate (PKT). The asbestos was used as a baseline since it is the SOA separator and readily available for inhouse screening tests. Asbestos fibers have been widely used in batteries

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and fuel cells and are known to degrade slowly at moderate temperatures ($>80^{\circ}\text{C}$). There are also environmental problems and uncertain availability. In addition, the asbestos separators have shown lower end of discharge voltages than the zircar cloth. Alternative fibers such as ZrO_2 and PKT looked promising. The zirconium oxide cloth as well as the ZrO_2 fibers are highly resistant to degradation, environmentally acceptable and readily available. The PKT fibers are also highly resistant to degradation and are commercially available but they have some environmental problems. These three fibers formed the basis of the separators to be tested. The fillers used were: (1) Titanium dioxide (TiO_2), (2) ceric oxide (CeO_2), and (3) zirconium oxide (ZrO_2). The TiO_2 filler was available in two different sizes U-110 (0.1 to 0.3 μm) and P25 (0.01 to 0.03 μm), which allowed us to compare the effect of particle size on final separator characteristics. The zirconium oxide was also available in two different particle sizes E10 (2 to 3 μm) and E20 (5 to 9 μm). All the filler materials were examined for weight loss at 100°C for 100 hr in 31 percent KOH.

A range of different commercial and experimental binders were evaluated. The binders used were the following:

- EBL-100 - Butyl latex rubber
- EXP - Experimental high strength butyl latex
- EP603A - EPDM rubber latex
- HL - Hypalon rubber latex
- ET-AA - Ethylene acrylic acid copolymer dispersion

All the binders proved to have high chemical stability in 31 percent KOH.

PREPARATION OF SEPARATORS

For making the separator matrices a sheet mold from Williams Apparatus Co. was used. The procedure for making the separators was as follows:

- (1) Calculation of necessary material weights for the specific separator composition and weighing of the materials.
- (2) Binder was dissolved in water and the fibers and fillers added while mixing.
- (3) The sheet mold was prepared by placing a screen on it and locking to form a close seal. Water was placed in the mold until almost full. Some water was drained by opening a drain valve to release trapped air.
- (4) The mixture was poured into the sheet mold and the drain valve was opened

after the mixture had settled, to drain excess water. It may be necessary to apply vacuum.

(5) The mold was opened and the separator was placed between layers of blotting paper and compressed on a press to force excess water out.

(6) Step 5 was repeated with new blotting paper and then the separator was taken out and air dried.

EXPERIMENTAL TEST PROCEDURES

State of the art separators as well as the separators developed in-house were characterized with respect to: resistivity, electrolyte diffusion, pore size, porosity, electrolyte retention, and bubble pressure. The screening tests as well as cell construction and characterization procedures were the following:

Electrical Resistivity

The electrical resistance of each separator was measured by the Direct Current Method. Two 1-1/2 in. x 1-1/2 in. samples of each separator were tested after soaking overnight in 31 percent KOH. A blank was run with no separator in the cell to measure cell resistance. This quantity was then subtracted from the resistance obtained with the separator in the cell, the difference being the separator resistance. The procedure was based on that given by J. J. Lander and R. D. Weaver in Ref. 2.

Electrolyte (OH^-) Diffusion

The permeability of separator matrices to the ionic constituents of the electrolyte is a contributing factor to the proper functioning of alkaline cells. The permeability of the separator reflects the effect of the microstructure on the diffusion and migration of the electrolyte in a similar manner as the separator affects the electrical resistance properties. A two compartment cell was used with the separator in between. One side of the cell was filled with distilled water and the other with 31 percent KOH solution. Samples of the water compartment were taken out at constant time intervals and titrated for its OH^- concentration. Using the concentration changes with time a diffusion rate for the hydroxyl ion was calculated. The method of determination of the electrolyte diffusion is based on that given by E. L. Harris in Ref. 2.

Pore Size

The size of the pores of the separator is a very important factor to be considered in characterizing the separators. A high pore

size is favorable because of comparatively free flow of ions between the electrodes but the individual pores must be small enough to prevent migration of the solid active materials. Also, pore sizes are important for proper electrolyte distribution in the cell. The pore sizes were measured using a wet method involving water flow through the separators as discussed in the procedure by L. M. Cooke and J. J. Lander in Ref. 2. Because some separators had pores too big to be measured by this method or due to lack of separator integrity some pore size measurements were obtained by use of a mercury porosimeter.

Porosity

The porosity is an important characteristic in the performance of separators in a battery. Percent electrolyte absorption was used as a measurement of porosity. Such measurement was obtained by determining the weight, diameter and thickness of the sample, before and after saturation with 31 percent KOH. The separators were cut in 5-cm-diameter circles and soaked overnight in the electrolyte. A slant press was used to drain each sample for 15 min and excess liquid was removed by pressing the sample between filter papers with a 1 kg weight for 30 sec before recording the saturated properties. The porosity of the separators can also be obtained by use of a mercury porosimeter

$$\text{Porosity} = \frac{W_w - W_d}{\rho_{\text{KOH}} * \frac{\pi}{4} L_w d_w^2}$$

W_w, W_d wet and dry weight of separators
 L_w thickness of wet separator
 d_w diameter of wet separator
 ρ_{KOH} density of KOH solution

Electrolyte Retention

Important separator properties to the operating characteristics of alkaline cells are electrolyte retention and absorption. These properties assume further significance in sealed cells with no free electrolyte present. A maximum amount of retained electrolyte is desired to achieve minimum cell internal resistance and to maximize cell capacity over a wide range of discharge rates. The procedure used to measure electrolyte retention is the same as the one to calculate pore size. The percent electrolyte retention is the ratio of final weight of the saturated separator less the initial weight over the initial weight of the dry separator.

Bubble Pressure

The separator material, usually 12-cm-diameter, was soaked overnight in water. The material was then placed between the cell plates, bolted together and the top reservoir filled with water. The air valve was then opened slowly until the first stream of bubbles was noticed and the pressure at this point recorded.

Cell Construction

Those separators that completed the screening tests with good overall characteristics were further tested in Ni-H₂ boiler plate cells. Both SOA and selected in-house separators were tested. Cells were built using two pairs of electrodes, each Ni electrode with a nominal capacity of 1.25 A-h. The cell configuration and components were that of the Air Force/Hughes cell, with electrochemically impregnated nickel electrodes and 10 mg/cm² platinum loaded hydrogen electrodes with the Teflon backing. Two polypropylene gas screens were used for the hydrogen gas flow field. The stack was filled with electrolyte by immersion into 31 percent KOH solution under vacuum. The assembled cells were evacuated and then pressurized with hydrogen to 50 psi. A set of three cells was built with each separator tested.

Cell Characterization

Cell capacities were determined for each individual cell during the cell formation cycles. Based on the theoretical capacities (2.5 A-h), a C/2 charge followed by a C/4 discharge was performed. A 10 percent overcharge was given with each charge and the cells were drained after each discharge. The cell capacities were taken as the capacity delivered to 1.0 V. This process was repeated until the cell capacities stabilized.

After the capacity determination, a set of characterization cycles were performed. Three different charge rates were used (C/4, C/2, C) each characterized with four different discharge rates (C/4, C/2, C, 2C). All cycles were given a 10 percent overcharge and a drain to minimize factors affecting the cell performance.

The cells were then cycled on a low earth orbit (LEO) regime at 80 percent DOD based on actual capacity delivered at C/4 rate to 1.0 V.

RESULTS AND DISCUSSION

A set of separator characteristics were chosen as goals for the development of separators. The new separators were to be superior to either zircar or asbestos by improving

the long term stability and performance while still meeting the characteristics which were considered necessary for good performance in Ni-H₂ cells. This set of characteristics is shown in Table I.

The approach for the separator development was to systematically vary fibers, filler, and binder quantity to study their effect in separator characteristics. Screening tests started by characterizing the standard fuel cell grade asbestos (FCGA) and the beater treated asbestos (BTA) (3) which incorporates 5 percent by weight of a butyl latex rubber (EBL-100) as a binder.

The effect of binder quantity on the properties of FCGA separators was investigated. The results are shown in Table II. Some of the FCGA characteristics could not be obtained because of loss of separator integrity. The porosity and pore sizes were obtained by use of a mercury porosimeter. It is clearly shown that as the amount of binder was increased the resistivity of the separator also increased.

It can also be seen that the in-house FCGA-5 percent EBL had a higher resistance than the commercial BTA, this is attributed to the process being much more uniform and controlled in a commercial run. The OH⁻ diffusion of the separators did not change significantly and the electrolyte retention decreased slightly with the addition of the binder as did the porosity. It was determined that the appropriate level of binder should be 5 percent by weight to improve physical integrity while minimizing its effect on resistivity.

For every set of tests run, a FCGA-5 percent EBL sample was made to compare the results of each set with a standard. This method was considered adequate since variation in the different runs were assumed to affect all separators in the same relative manner.

The next step in the study was to examine the effect of different binders on the properties of FCGA separators. Ten percent instead of 5 percent of each of the binders was used in order to intensify their effect. The binders tested and the results obtained are shown on Table III. The volume resistivity for all binders are relatively close except for that of the ethylene acrylic acid whose resistivity was almost half that of the other binders. For this binder the resistivity and bubble pressure were the only tests performed due to an extremely large amount of electrolyte absorbed which made it impossible to handle after wetting with KOH. This is considered a good characteristic since the amount of binder could be increased still maintaining low resistance, but its physical strength

requires improvement. The tests show no direct relation between pore size and electrolyte retention as is usually the case because of the different binder characteristics. The nature of the binders dictates the hydrophobic or hydrophilic characteristics which will influence electrolyte retention. This difference disappears if the same binder is used. Since the total characterization of the binders did not reveal another which was better than the EBL-100, it was chosen as the base binder for the rest of the studies.

The effect of adding ZrO₂ fibers to FCGA was then studied. The amount of ZrO₂ fibers was increased from 0 to 80 percent by weight while keeping 5 percent by weight EBL-100. This data is shown in Table IV. No significant effect is seen on resistivity and OH⁻ diffusion while the electrolyte retention, porosity and bubble pressure decreased with increasing amount of ZrO₂ fibers. This was expected and can be explained by looking at the average pore sizes of the separators. As the pore sizes increased the bubble pressure decreased because less pressure is necessary to force the air through the pores. Also as the pores get bigger, capillary forces decrease diminishing the electrolyte retention. This seems to indicate that a low amount of ZrO₂ fibers is desirable.

Pigmentary potassium titanate (PKT) fibers were also tested. This data is shown in Table V. The amount of PKT was only increased to 50 percent by weight since the bubble pressure decreased dramatically. Porosity and electrolyte retention increased considerably and the pore sizes remained stable. The bubble pressure was the only characteristic which was negatively affected. When looking at interaction between fibers these studies will only point out possible trends and interactions with the particular fibers. And verification of interactions within other fibers and fillers is necessary once the composition of an improved separator is being optimized.

Different fillers like ceric oxide (CeO₂), zirconium oxide (ZrO₂) and titanium dioxides (TiO₂) were studied and their effect on properties of FCGA separators were determined. These data are shown in Table VI. The CeO₂ had a negative effect on OH⁻ diffusion. This filler did not seem to mix well with the asbestos remaining mostly on one side of the separator. The zirconium oxide E20 and E10 both increased the resistance while decreasing slightly the bubble pressure and had no effect on the other characteristics. The two titanium dioxides P25 and U110 greatly increased the OH⁻ diffusion of the separators which should result in higher rate capability in the cell. Also the bubble pressures were high and separator strength was improved.

Therefore the Ti_1O_2 fillers were considered the best candidate to try to increase bubble pressure and electrolyte diffusion capabilities of separators with no FCGA fibers in them.

Separators with only PKT fibers and a binder were pursued but a low amount of binder (5 percent) was not enough to give the separator integrity. Therefore the amount of binder was increased to 10 percent by weight to keep resistivity low while increasing strength of the separators. Extreme caution had to be exercised when making and handling separators of PKT fibers. Otherwise small cracks on the separators developed resulting in low bubble pressures while other separator characteristics were excellent.

Further studies determined the optimum composition to be 80 percent PKT-20 percent ZrO_2 with 10 percent EBL-100 as a binder. PKT fibers were also deposited on ZrO_2 cloth. The characteristics of these separators along with the SOA separators and the HT-200C (polysulfone) are shown in Table VII. These separators described above were recommended for cell testing based on the screening characteristic study and electrolyte management studies as reported in Ref. 3.

CELL TEST RESULTS

A data base was established to compare cell performance of new separators against SOA separators. FCGA, BTA, and ZrO_2 cloth were tested in cells. The mid-point discharge voltages for a C rate charge, C rate discharge are shown in Table VIII.

The zircar cloth had higher mid-point voltages than the asbestos separators, this is expected due to the more open structure of the separator. BTA tests were repeated along with the other separators recommended for testing. This data is also shown in Table VIII. The initial characterization (only C rate data is shown) showed higher mid-point voltages for the two in-house separators than either asbestos separator. Although the voltages were comparable or lower than the zircar cloth separators it is still considered very good for a close separator with high bubble pressure. The polysulfone separator showed mid-point voltages comparable to the asbestos separators. Table IX shows end of discharge voltages for all separators tested at 250 cycles.

Plots of charge and discharge for 80 percent PKT-20 percent ZrO_2 , BTA, PKT on ZrO_2 cloth and polysulfone HT200-C separators for a C rate charge and C rate discharge are shown in Figs. 1 to 4.

CONCLUSIONS AND RECOMMENDATIONS

The in-house characterization study showed the following:

- (1) A minimum amount of binder should be used to give physical integrity to the separators keeping the resistivity to a minimum.
- (2) Binder characterization did not reveal a binder better than EBL-100, but further studies in ET-AA emulsion are recommended due to its electrolyte retention capabilities and low resistivity.
- (3) Separators made of ZrO_2 fibers only cannot be obtained due to lack of integrity and separators with PKT only are too brittle for commercial production.
- (4) Fillers can be used to improve bubble pressure and other characteristics of the separators but an increase amount of fillers also increase separator brittleness. An optimization study has to be performed for every separator of interest.
- (5) Screening studies showed two separators which met characteristic goals. These were 80 PKT-20 ZrO_2 - 10 percent EBL and PKT on ZrO_2 cloth. These separators were recommended for cell testing.
- (6) Cell characterization of the in-house separators showed good cell voltage with the mid-point voltages better than the FCGA and BTA separators. Therefore these separators are recommended for further cycling in cells purchased from a vendor with modifications to test separator effect free of electrode or design effects on separator and cell performance.

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TABLE I. - SEPARATOR PROPERTIES - GOALS

Resistivity, Ω -cm	<5
Electrolyte retention, percent	≥ 100
Porosity, percent	>50
Bubble pressure, psi	>20
Electrolyte diffusion, moles/cm ² min	$\geq 1 \times 10^{-5}$
Oxidation resistance, percent weight loss	≤ 5

TABLE II. - EFFECT OF BINDER QUANTITY ON SEPARATOR CHARACTERISTICS

Separator	Volume resistivity, Ω -cm	OH ⁻ diffusion $\times 10^{-5}$, moles/cm ² min	Electrolyte retention, percent	Porosity percent	Pore size, \AA	Bubble pressure, psi
FCGA	3.0	a_____	a_____	^b 77.3		24
BTA	2.4	5.30	134.2	69.8	1941	>30
FCGA-5% EBL	3.8	5.05	136.6	61.7	2686	>30
FCGA-10% EBL	11.41	4.27	133.4	53.4	1367	>30
FCGA-20% EBL	11.1	4.59	131.6	55.6	1960	>30

^aCannot be determined due to lack of separator integrity.

^bDetermined using the mercury porosimetry method.

TABLE III. - EFFECT OF BINDER ON FCGA SEPARATORS

Separator	Volume resistivity, Ω -cm	Electrolyte retention, percent	Porosity, percent	Pore size, A	Bubble pressure, psi
FCGA-10% EBL	5.4	133.9	64.9	1636	>30
FCGA-10% EPA	6.1	119.5	77.9	1391	>30
FCGA-10% EXP	6.5	132.0	66.5	2630	>30
FCGA-10% EHL	5.2	78.3	44.0	1661	29
FCGA-10% EAA	3.3	-----	-----	-----	>30

TABLE IV. - EFFECT OF ZRO₂ ON FCGA SEPARATORS

Separator	Volume resistivity, Ω -cm	OH ⁻ diffusion $\times 10^{-5}$, moles/cm ² min	Electrolyte retention, percent	Porosity, percent	Pore size, A ^o	Bubble pressure, psi
FCGA-5 EBL	1.9	7.86	121.5	61.8	2596	19
80% FCGA-20% ZrO ₂ -5 EBL	2.4	4.52	120.9	61.9	2710	16
50% FCGA-50% ZrO ₂ -5 EBL	2.6	6.04	102.4	57.4	3204	5
20% FCGA-80% ZrO ₂ -5 EBL	2.1	6.85	80.6	54.2	7514	2

TABLE V. - EFFECT OF PKT ON FCGA SEPARATORS

Separator	Volume resistivity, Ω -cm	OH^- diffusion $\times 10^{-5}$, moles/cm ² min	Electrolyte retention, percent	Porosity, percent	Pore size, \AA	Bubble pressure, psi
FCGA-5 EBL	1.5	7.53	137.9	63.9	2123	30
10 g FCGA-2 g PKT-5% EBL	2.0		196.7	78.0	2092	17
5 g FCGA-5 g PKT-5% EBL	2.9	1.29	203.3	79.8	2178	1

TABLE VI. - EFFECT OF FILLER ON FCGA SEPARATORS

Separator	Volume resistivity, Ω -cm	OH^- diffusion $\times 10^{-5}$, moles/cm ² min	Electrolyte retention, percent	Porosity, percent	Pore size, \AA	Bubble pressure, psi
10 gr FCGA-5 EBL	1.5	7.53	137.9	63.9	2123	30
10 gr FCGA-2 gr CeO ₂ -5% EBL	1.0	1.41	149.4	68.0	2683	20
10 gr FCGA-2 gr E20-5% EBL	5.8	6.90	122.9	62.2	2335	20
10 gr FCGA-2 gr E10-5% EBL	4.8	6.35	129.4	61.9	2609	18
10 gr FCGA-2 gr P25-5% EBL	3.7	23.41	112.8	68.4	1284	>30
10 gr FCGA-2 gr U110-5% EBL	3.1	17.20	129.3	69.8	1003	>30

TABLE VII. - PROPERTIES OF SEPARATORS CELL TESTED

Separator	Resistivity, Ω -cm	Electrolyte diffusion $\times 10^{-5}$ moles/cm ² min	Electrolyte retention, percent	Porosity, percent	Bubble pressure, psi
Goal	<5	≥ 1	≥ 100	≥ 50	> 20
ZrO ₂ cloth	1	a	≥ 150	^b 82.5	4
FCGA	3	a	^a	^b 77.3	24
BTA	2	5.3	134	70	> 30
PKT-10% EBL on ZrO ₂ cloth	3		100	67	20
80% PKT-20% ZrO ₂ -10% EBL	2	10.2	171	69	> 30
HT-200 C	7	13.9	152	56	> 30

^aCannot be determined due to lack of separator integrity.

^bDetermined using the mercury porosimetry method.

TABLE VIII. - CHARACTERIZATION TESTS

[C rate charge/C rate discharge].

Separator	Average mid-point voltages,
1-FCGA	1.18
2-BTA	1.17
3-ZrO ₂	1.20
4-BTA(2)	1.18
5-80% PKT - 20% ZrO ₂	1.20
6-PKT on ZrO ₂ cloth	1.20
7-HT 200-C	1.18

Note 1: Separators 1 to 3 were tested with one pair of electrodes, one gas screen for hydrogen gas access. Separators 4 to 7 were tested with two pairs of electrodes, two gas screens for hydrogen access.

TABLE IX. - END OF DISCHARGE VOLTAGES
 UNDER LEO REGIME AT 250 CYCLES

Separator	Average end of discharge voltages,
FCGA	1.09
BTA	1.11
ZrO ₂ cloth	1.13
BTA(2)	1.14
80 PKT - 20ZrO ₂	1.14
PKT on ZrO ₂ cloth	1.15
HT 200-C	^a 1.09

^aOnly one cell remains.

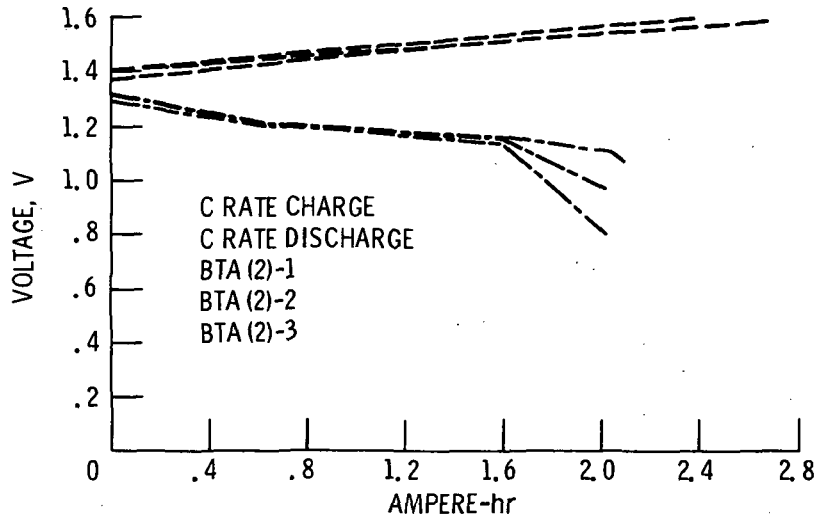


Figure 1. - Voltage versus ampere hours for BTA (2). Cell construction, 2-AH cells/standard.

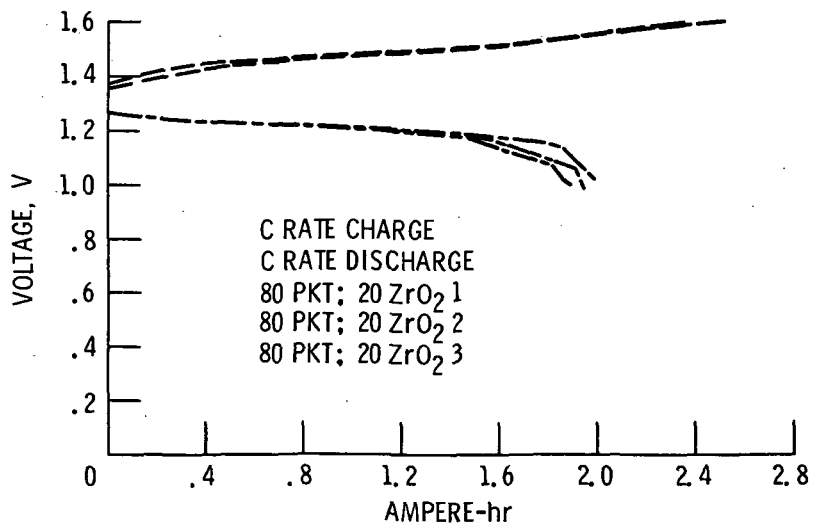


Figure 2. - Voltage versus ampere hours for 80 PKT on 20 ZrO₂.

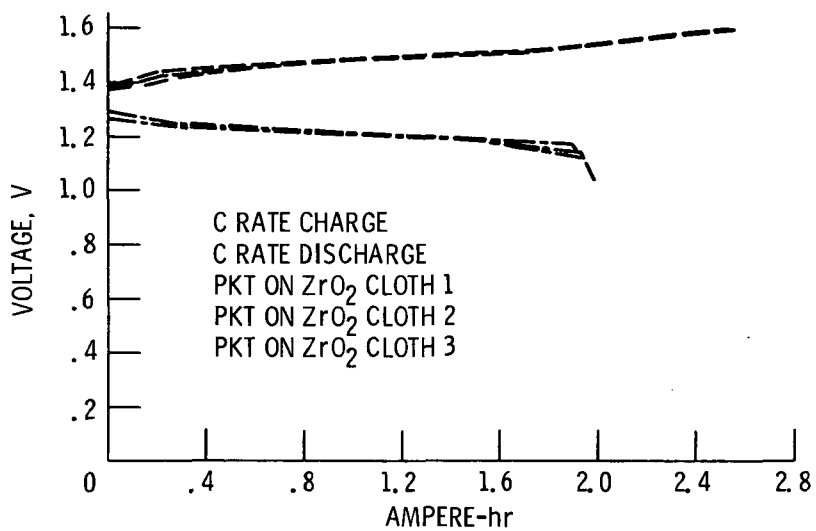


Figure 3. - Voltage versus ampere hours for PKT on ZrO₂ cloth.

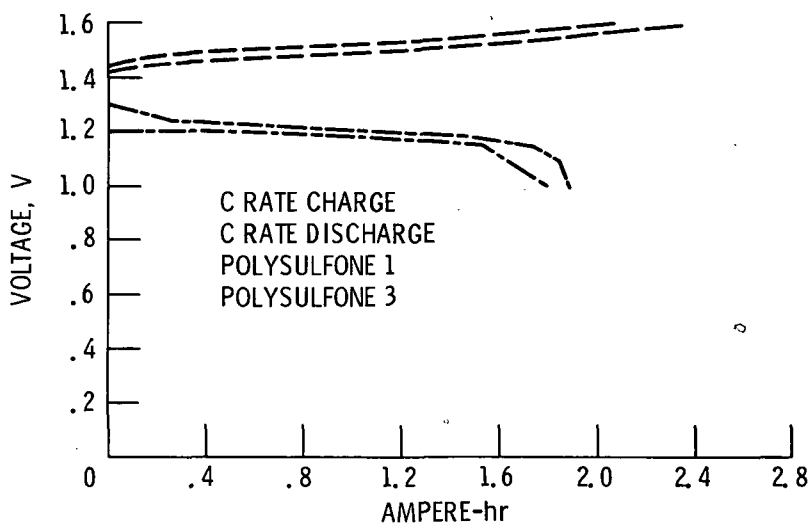


Figure 4. - Voltage versus ampere hours for polysulfone.

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