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EIGHTH QUARTERLY REPORT

On

ALKALINE BATTERY SEPARATOR
CHARACTERIZATION STUDIES

(28 June 1969 - 28 September 1969)

J. J. Kelley
S. Orenstein

For

GODDARD SPACE FLIGHT CENTER

CONTRACT NAS 5-10418

Contracting Officer: John Comstock
Technical Monitor: T. H. Hennigan

Prepared by
ESB INC. RESEARCH CENTER
Yardley, Pennsylvania 19067

For

GODDARD SPACE FLIGHT CENTER
Greenbelt, Maryland 20771

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ALKALINE BATTERY SEPARATOR CHARACTERIZATION STUDIES

by

J. J. Kelley and S. M. Orenstein

ABSTRACT

The performance of grafted polyethylene and polyvinyl alcohol separators equals that of cellophane in Ni-ZnO cells and surpasses cellophane in Ag-CdO tests. Measurements of silver permeability for membranes received since the Seventh Quarter agree with previously reported zincate diffusion measurements.

The number of absorbent separators studied in the past two quarters has increased to eighteen. Characterization of three new materials is presented along with electrolyte absorption and oxidative resistance measurements for all test materials. Duplicate three-plate, "sealed" Ni-Cd test cells containing five absorber materials have completed 1000 cycles. Behavior of the unique cell configuration is described.

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1.0 INTRODUCTION

Three absorber and four membrane materials were received and included in the test program during this quarter. A brief description of these materials appears in Table 1.

An additional sample of Southwest Research Institute grafted and crosslinked polyethylene has been received. The conditions used to make the film are apparently aimed at including more grafted acrylate in material with the same crosslink density. The reaction parameters for Runs 362 and 359 may be found in Reference (1). Two small samples of material resulting from the efforts of Monsanto Research are also available. The material is an ethylene-methacrylic acid copolymer developed for heat sterilizable Ag-Zn batteries. Details of the material development may be found in Reference (3). Last is a sample of inorganic matrix separator developed for heat sterilizable Ag-Zn batteries at Westinghouse Research. Reference (4) deals with the fabrication of the zirconia-polysulfone coating on non-woven polypropylene base material.

Characterization of membrane materials received since the Seventh Quarter is nearing completion. Silver and zincate permeability, electrical resistance and cell testing for all materials is contained in this and the preceeding quarterly reports.

TABLE 1. NEW MATERIALS

MEMBRANE MATERIALS

<u>Style - Number</u>	<u>Composition</u>	<u>Manufacturer</u>
362 GX	Radiation grafted and crosslinked polyethylene	Southwest Research Institute
C-58-74-1	Zirconia - Polysulfone coated on EM476	Westinghouse Elec. Corp.
MRCD90 MRCD91	Ethylene-Methacrylic Acid Copolymer	Monsanto Research

ABSORBER MATERIALS

304 PO	Non-woven Polypropylene	GAF - American Felt Co.
Nylon Taffeta	Woven Nylon Cloth	Howard Textile Mills
RAI 476	Corona discharge treated EM 476	Kendall Mills via RAI Research

Absorber materials received during this quarter are also shown in Table 1. Although woven materials have not been widely used in alkaline batteries, a sample of nylon taffeta is being characterized. In addition to its possible battery applications, the regularly spaced fiber pattern may be useful in defining the material properties which affect improved performance. A sample of EM 476 has been subjected to the same Corona discharge surface treatment given Pellon 2505 samples tested during the Seventh Quarter. The most obvious result of the surface treatment is the formation of pinholes by fusion of the fibers. The American Felt Company of GAF, Inc. has supplied a sample of 304 PO, polypropylene felt. Since this material is not a production item, it may also be referred to as WEX 1242. The tests performed on 304 PO so far, show similar properties to other polypropylene felts evaluated.

2.0 SEPARATOR SCREENING TESTS - MEMBRANE MATERIALS

2.1 Silver Permeability

The flux of dissolved silver was determined by diffusion of radioactive argentate across the sample membrane. The method has been described in the Second Quarterly Report and Reference (2). Table 2 shows the rate of absorption, flux and Fick's Law diffusion coefficient for the membranes presently being tested. The order of ionic flux is the same for silver and zinc:

RAI 191 < Monosol 3-001-3 < Monosol 1-001-3 < 4-359 GX < 362 GX.

In addition, the permeability of silver and zinc are within an order of magnitude, in spite of the low solubility of silver in KOH (0.36×10^{-3} M). The combined effect of low silver solubility and high flux is reflected in the diffusion coefficient, D, which is 10^8 times greater for silver than zinc.

The same behavior towards silver and zinc permeability is apparent for all of the grafted and crosslinked polyethylene samples tested so far. Data for previously tested samples may be found in the Sixth Quarterly Report.

2.2 New Materials

The properties of new materials appear in Table 3. Three of the four materials shown are formulated for use only after in situ sterilization (see Table 3). Hence, data was taken after bomb sterilization

TABLE 2

SILVER PERMEABILITY OF MEMBRANE MATERIALS

	f - Flux Rate moles/cm ² -sec x 10 ⁸	D - Diffusion Coef. ¹ cm ⁻¹ sec ⁻¹ x 10 ¹⁰	Absorption Rate gms AgO/in ² -hr x 10 ⁸
RAI 191	0.18	1.75	0.93
4-359GX	0.65	6.95	4.86
362GX	1.35	12.7	1.44
1-001-3	0.48	4.75	3.81
3-001-3	0.46	4.55	7.21

¹ Ag₂O concentration of 0.36×10^{-3} M

TABLE 3

PROPERTIES OF NEW MEMBRANE MATERIALS

	362GX	C58-74-1S ¹	M90S ¹	M91S ¹
Thickness (cm x 10 ³)				
dry	2.54	10.2 ²	³	³
in 40% KOH	3.38	12.7	12.95	11.7
Zincate Permeability in 1M ZnO-KOH				
f (moles/cm ² sec x 10 ⁸)	7.3	1.96	0.129	0.094
D (cm ² /sec x 10 ¹⁰)	2.47	25.7	1.72	1.14
A. C. Resistance				
Ω -cm	19.8	35.2	43	42.5
Ω -cm ²	0.067	0.447	0.556	0.497

¹ S, indicates bomb sterilized material.

² Nature of sterilized material did not permit drying without loss of inorganic matrix. Thickness given is for unsterilized material.

³ Dry material is puckered. Representative measurements cannot be made.

at 135°C for 69 hours in the presence of excess 40 percent electrolyte.

The ethylene-methacrylic acid copolymers from Monsanto, M90 and M91, are much like Permion 2291. Low zincate flux is achieved at the expense of electrical resistance ($0.5 \mu\text{-cm}^2$). The literature available (3) indicates that difficulty was encountered in forming membranes thinner than those under test. The polymer is hot pressed between Teflon sheets. Films that were less than 0.003 inch could not be removed from the Teflon sheets. The samples of M90 and M91 were puckered when dry, probably from spot adhesion to the Teflon.

The sample of C-58-74-1 was difficult to handle after sterilization. Even when wet, the inorganic coating did not adhere well to the EM 476 base. Electrical resistance before sterilization is much greater than $500 \mu\text{-cm}$. After sterilization, the specific resistance is comparable to the Monsanto membranes or Permion 2291 (i.e., still rather high). In addition, the zincate flux is an order of magnitude greater than the Monsanto or Permion materials.

Of the two Southwest Research Institute materials tested in the past two quarters, 362 GX has the lowest electrical resistance. However, zincate and silver permeability remain quite high. As with 4-359 GX, zinc clusters were formed on the solution side of the mercury-membrane electrode during zincate diffusion tests. These clusters were collected and spectroscopically analysed for mercury. Since no mercury could be detected, zinc must have penetrated the membrane allowing electrodeposition

from the bulk solution. The poor zinc stopping power of these materials should be verified in zinc penetration tests planned for the next quarter.

The ion-exchange capacities of 4-359 GX and 362 GX are 4.20 and 4.65 meq/gram, respectively. This was determined by converting a one gram sample of membrane to the acid form, washing and drying. The sample is accurately weighed and equilibrated in a measured amount of standardized solution of base. An aliquot of the equilibrating solution is titrated and the ion-exchange capacity of the membrane is calculated from the decrease of (OH^-) concentration. It can be seen from Ref. 1 that an additional 60 feet of polyethylene film and higher reaction temperatures were encountered in making 359 GX than with 362 GX. Since the concentration of acrylic acid in the grafting solutions was the same, a slightly higher ion-exchange capacity would be expected for the 362 GX because of the higher acrylic acid to polyethylene ratio.

3.0 ABSORBER MATERIALS

3.1 Properties of Absorber Materials

The electrolyte absorption and tensile strength related properties of absorber materials are shown in Table 4. The test procedures are described in the Second Quarterly Report. Tensile strength samples were cut with a template and razor knife instead of a die. This procedure has been found more convenient and reproducible.

Since Pellon 2505 is successfully used as the absorbent in Ag-Zn, Ag-Cd, and Ni-Cd batteries, we may assume that the tensile strengths of Table 4 are more than adequate. As expected, massive degradation of the absorbers tested is not observed in the oxidative resistance test. Two absorber materials have been eliminated from the test program. The first is RD 651 asbestos from Score, Inc. The 100 psi tensile strength is too low to allow wet handling and the material has been assayed at 0.2 percent Fe. Although this is low for an asbestos material, it need not be tolerated where iron-free materials are available. The second material removed is Pellon 301. This was erroneously listed in the Seventh Quarterly Report as a polypropylene material. We have since been informed that this material is neither polypropylene nor intended for use as a battery separator.

The values for porosity shown in Table 4 are calculated from electrolyte absorption data by:

$$\% \text{ Porosity} = \frac{G}{dV_w} \times 100$$

where, G, is the weight of electrolyte of density, d, contained in a volume, V_w , of wet material. An investigation of methods for determining porosity and electrolyte absorption is underway and results will be reported in the next quarter.

TABLE 4

PROPERTIES OF ABSORBER MATERIALS IN 40% KOH

	RAI Treated Pellon 2505 WO				2505ML	RD651 ¹
	E-T&S	E-T	E-S	U-T&S	U-T	U-S
Electrolyte Absorption $\left(\frac{\text{gms}}{\text{cc dry vol}} \right)$	1.22	1.28	0.93	1.35	1.14	0.91
% Electrolyte Retained	100	100	100	100	100	100
% Porosity	77.9	83.5	58.4	86.8	74.1	58.8
Tensile Strength (psi) dry	300	280	380	370	390	370
after 24 hrs in 40% KOH	310	220	280	380	310	380
Oxidative Resistance (psi) 168 hours	290	250	230	360	320	300
336 hours	230	220	200	290	250	270

TABLE 4 (contd)

	EM476	RA1476	E1451	M1450	M1410	M1401	E1488	309PO	Nylon Taffeta
Electrolyte Absorption $\left(\frac{\text{gms}}{\text{cc dry vol}} \right)$	0.30	0.29	0.65	0.46	0.50	0.20	0.40	0.70	0.35
% Electrolyte Retained	100	98	100	100	100	100	99	-	-
% Porosity	20.6	22.9	47.3	39.2	38.2	19.7	27.3	50.2	25.1
Tensile Strength (psi) dry	3930	-	3630	1640	1160	3450	3830	510	12,400
after 24 hrs in 40% KOH	4630	5420	3160	1410	1310	4020	5640	760	15,140
Oxidative Resistance (psi) 168 hours	3240	4540	2960	1560	1400	4180	6060	890	14,040
336 hours	4190	5030	3410	1660	1340	3790	5500	650	12,400

¹ No further testing planned.

3.2 New Materials

The properties of RAI 476, GAF 304 PO and Howard nylon taffeta are shown in Tables 4 and 5. The wettability of 304 PO is typical of non-woven polypropylene, though its resistance is lower than similar materials tested. The Howard nylon taffeta is the only woven material tested so far. Uniform thickness, resistance and air permeability are available at the expense of uniform wetting. When the taffeta material is placed in the wicking apparatus, random areas wet while others appear dry. The estimated wicking rate (see Table 5) is slow compared to non-woven materials. The performance of this material in three-plate Ni-Cd cell tests may help identify those properties important in cell operation.

The properties of RAI 476 are largely the same as EM 476. Wicking height and rate, and wet-out time are slightly faster for the RAI treated material, however, this material is slightly thinner than EM 476 and its resistance (in Ω -cm²) is slightly lower. A sample of the original material was not supplied by RAI and the observed differences between treated and untreated material could easily be variations between manufacturing lots. Unlike the RAI treated Pellon materials investigated during the Seventh Quarter, the Kendall material was not scoured before treatment. The treated material, therefore, retains any fiber treating agents applied by the manufacturer. Since EM 476 is a thin material, it is not unusual to find voids and pin-holes in the structure. Microscopic examination of RAI 476 revealed numerous pinholes resulting from fusion of the fibers.

TABLE 5

PROPERTIES OF NEW ABSORBER MATERIALS IN 40% KOH

	RAI 476	GAF 304 PO	Nylon Taffeta
Wicking Height cm/0.5 hr	< 8.3	7.9	- 1
Wicking Rate min/cm	3.5	4.8	0.041 ¹
Wet-out Time (min)	1.7	4.28	8.9
Thickness (cm x 10 ³) dry	9.0 ± 0.5	27.2 ± 0.5	5.08
in 40% KOH	7.6 ± 0.5	27.3 ± 0.7	5.08
A. C. Resistance Ω-cm	15.0	3.5	17.4
Ω-cm ²	0.114	0.319	0.089
Air Permeability (sec/300 cc)	21	5.33	105

¹ Material does not wick uniformly. Wicking rate is estimated from elapsed time for appearance of electrolyte at the top (8 cm height) of the fixture.

The same condition and appearance was found in the above-mentioned RAI-treated Pellon materials. There is no doubt that these pinholes are a result of the treatment process.

4.0 CELL TESTING

4.1 Membrane Materials - Ni-ZnO and Ag-CdO

Six of the eight membrane samples on hand were assembled in duplicate Ni-ZnO and Ag-CdO cells. The MONSANTO M 90 and M 91 were omitted because insufficient material was available. Details of the cell construction, electrode capacities and cycling parameters are given in the First Quarterly Report. A summary of life cycling data and failure modes is given in Table 6.

The first three samples listed in Table 6 performed as well as cellophane in Ni-ZnO cells. Failure of both 3-001-3 cells and one of the 4-359 GX tests was a direct result of dendrite penetration of the separator face. The second 4-359 GX, Ni-ZnO test was characterized by erratic open circuit and discharge voltages, normally associated with partial shorting. Conclusive evidence for shorting could not be found during autopsy of the cell. The same is true for both Ni-ZnO cells with 1-001-3 PVA. The remaining three membranes tested apparently have very little zinc stopping power. The one RAI 191 cell to complete 30 cycles, evidently recovered from a dendritic short. Capacity dropped from 1.07 Ahr on Cycle 9 to 0.53 Ahr on Cycle 10. The cell recovered to a maximum of 0.89 Ahr on the sixteenth cycle and completed the 30-cycle test with 52 percent of its initial capacity. Erratic behavior of open circuit and discharge voltages confirm shorting of this cell at the time noted.

TABLE 6

SUMMARY OF MEMBRANE MATERIAL CELL TESTS¹

Sample	Cycles to Failure ²		Cycles to Last Successful Charge		% Capacity on Last Successful Discharge		Mode of Failure ⁴	
	Ni-ZnO	Ag-CdO	Ni-ZnO	Ag-CdO	Ni-ZnO	Ag-CdO	Ni-ZnO	Ag-CdO
1-001-3	21, 23	22, 20	30, 30	30, 22	35, 43	40, 40	EP, EP	EP, EP
3-001-3	24, 19	18, 5	24, 19	24, 5	66, 76	8, 130	P, P	S, P
4-359GX	24, 24	30, 30	24, 30	30, 30	46, 38	70, 80	P, EP	- -
362GX	12, 7	29, 11	14, 7	30, 12	80, 80	40, 12	P, S	E, P
RAI 191	9, 8	30, 30	30, 8	30, 30	52, 85	50, 55	EP, P	- , -
C 58-74-1S ³	3, 1	8, 12	6, 1	11, 3	12, 100	25, 10	P, P	P, P

¹Duplicate cells each, for Ag-CdO and Ni-ZnO tests.²Failure defined as cycles to less than 50% initial capacity or first evidence of short (e.g., low OCV, excess self-discharge, failure to accept charge, etc.).³Bomb sterilized material.⁴Mode of failure legend; P, dendrite penetration at top edge or face;
S, edge shorting at folds;

E, electrode failure

EP, apparent electrode failure accompanied by low OCV and/or discharge voltage;

-, no entry indicates cell performing satisfactorily at end of test.

Performance in the Ag-CdO system was slightly better. Two materials, 4-359 GX and RAI 191, completed 30 cycles with satisfactory performance. MONOSOL 1-001-3 duplicated the behavior of the Ni-ZnO tests, offering marginal performance and evidence of shorting. The three samples remaining, 3-001-3, 362 GX, and C 58-74-1S, shorted by silver penetration.

The data indicates that 1-001-3, 4-359 GX, and RAI 191 could equal or better the performance of cellophane in silver-zinc test cells. Further study of the data will be made along with recommendations for further testing.

4.2 Absorber Materials - Ni-Cd

Duplicate, sealed, Ni-Cd cells were assembled with two polypropylene and three dynel absorber materials. Details of the cell construction are given in the Seventh Quarterly Report.

The 4 Ahr, Ni-Cd packs used to fabricate test cells were taken apart and each electrode tab was numbered so that the original pack could be re-assembled at any time. The packs were then assembled in their original configuration and placed in suitable jars. Electrolyte (31 percent KOH) was added and after a 16-hour soak, the procedure of Table 7 was carried out. The data for the fourth and fifth cycles was used to rate the individual plates for C/2 discharge capacity to one volt. Such a five cycle treatment also simulates the manufacturer's recommendations for conditioning the cells before putting them into service. For assembly of ten test cells, three

TABLE 7

PROCEDURE FOR PREPARATION OF 4 AH Ni-Cd PACKS

1. Form for 16 hours at 1A. Record ECV.
2. Discharge at 2A to 1.0 V. Record Ahr output.
3. Charge at 1A to 125 percent of previous output. Record ECV.
4. Repeat Steps 2 and 3 until discharge #4.
5. Discharge as in Step 2. Record Ahr to 1.0 V but do not turn off.
continue discharge at 2A. Record Ahr to 0.0 V (full cell).
DO NOT TURN OFF, continue discharge to 0.0 V (E Cd) vs Ni Reference.
6. Recharge to 125 percent of TOTAL output in Step 5.
7. Proceed for the fifth discharge as in Step 5. DO NOT RECHARGE.
8. Remove Ni Plates and substitute nickel foil electrodes.
Shim and top up electrolyte (31 percent KOH) as needed.
9. Wash Ni Plates with deionized water until wash water which has
stood for 2 hours or more has a pH of 7 or less. Dry at $\sim 70^{\circ}\text{C}$
in a vacuum for 2-3 hours. Cool and store in a dessicator.
10. Charge the cadmium plates against the nickel foil electrodes
for 2 hours at 1A. Record ECV (E Cd vs Ni Ref.).
11. Wash, dry and store the Cd plates as in Step 9.

packs were treated. The average of the three packs gave a Cd:Ni ratio of 1.88:1 and 0.565 Ahr and 1.06 Ahr capacities for nickel and cadmium plates at the C/2 rate. A total of ten absorber test cells were assembled from the washed and dried plates. The six "outside" negatives were not used in the test cell assembly. Formation at the C/3 rate to 125 percent of rated capacity preceded the cycle life test.

A specially designed clamping jig was used to retain the bank of ten test cells. A pneumatic clamping cylinder was set to apply one atmosphere of pressure to the cell face. The cells were separated by 0.25 inch thick aluminum plates. Extensions were made for the cell terminals to avoid accidental shorting or grounding to the clamping jig. The cells were then put into service on a 90 minute orbit cycle at C/2 discharge, C/3 charge and 25 percent overcharge. A summary of cycling data appears in Table 8.

With the exception of the first EM 476 test, there is no apparent difference between the five materials. Typical performance is characterized by a slow decrease of end of discharge voltage (EDV) with cycle life. End of charge voltage (ECV) is seen to rise to a gassing plateau within 300 cycles and is followed by slowly increasing ECV. These conditions prevail until an apparent "memory effect" simultaneously increases ECV and decreases EDV. The above description fits the expected performance of a flooded cell. The excepted cell, containing EM 476, performed with sealed cell characteristics and showed no "memory effect". Since a minimum of twenty additional cells will be assembled and cycled, further analysis of the data will be deferred until a larger sample is available.

TABLE 8

SUMMARY OF THREE PLATE Ni-Cd CYCLING

Voltage - Millivolts

Sample	Cycle		50	100	200	300	400	500	600	700	800	900	950
	EDV	ECV											
EM476	1195	1450	1175	1455	1160	1155	1455	1155	1160	1160	1160	1160	1160
													1470
E1451	1180	1495	1180	1535	1175	1160	1150	1135	1120	1125	1115	1095	1080
													1750
E1488	1175	1475	1185	1540	1190	1170	1165	1150	1135	1125	1075	1025	945
													1818
M1450	1195	1525	1180	1560	1195	1170	1155	1145	1135	1115	1090	1065	1060
													1750
M1450	1190	1495	1185	1535	1190	1185	1160	1160	1155	1145	1125	1110	1100
													1715
M1450	1195	1460	1175	1485	1195	1185	1175	1155	1155	1145	1130	1110	1105
													1715
M1450	1175	1460	1155	1470	1170	1175	1165	1155	1155	1145	1135	1115	1105
													1735
M1450	1180	1480	1170	1505	1185	1170	1155	1145	1135	1115	1090	1065	1055
													1765

continued

TABLE 8 (contd)

Cycle		50	100	200	300	400	500	600	700	800	900	950
Sample												
M1410	EDV	1185	1175	1180	1175	1165	1160	1165	1155	1150	1145	1145
	ECV	1465	1485	1515	1615	1645	1655	1665	1655	1650	1670	1675
	EDV	1190	1170	1165	1165	1160	1165	1155	1140	1140	-	-
	ECV	1445	1465	1530	1600	1645	1620	1635	1640	1650	-	-

5.0 FUTURE WORK

During the Ninth Quarter, the remaining membrane tests will be completed. With data on zinc penetration, pore size and tortuosity, electrolyte absorption, tensile strength and oxidative resistance, recommendations for further testing in Ag-ZnO batteries will be made.

Cell testing of absorber materials will be continued. In addition to testing 'as received' materials, cell tests with surfactant free and surface treated materials are planned. Investigation of organic and inorganic matter present in absorbers will be conducted. Studies of alternate methods for determining electrolyte absorption will also be made.

6.0 LITERATURE CITED

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