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SEPARATOR MATERIAL FOR RESERVE-PRIMARY ZINC/AIR BATTERIES

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ABSTRACT

It has been found that separator materials commonly used in zinc/air batteries do not wet readily when placed in contact with alkaline electrolyte at -40°C. They are, therefore, unsuitable for use in a reserve-primary zinc/air battery which must be capable of rapid activation and operation at any temperature in the range -40° C to 50° C.

A glass-Vinyon separator which meets the requirements of this type of battery has been developed.

RÉSUMÉ

Les matériaux généralement utilisés comme séparateurs dans les générateurs zinc/air présentent une mauvaise mouillabilité en milieu alcalin à des températures aussi basses que -40° C. Ils ne sont donc pas acceptables dans les générateurs primaires zinc/air du type amorçable aux températures variant de -40° à 50°C.

Nous avons mis au point un séparateur composé de verre et de Vinyon qui satisfait aux besoins de ce type de générateur.

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INTRODUCTION

During the development of a reserve-primary zinc/air battery to power the AN/TRN-30 beacon (1), it was found that the very long time required for the battery to reach an operational voltage after activation at -40° was due to the poor wettability of the dynel separator at that temperature. If the battery was activated at room temperature, cooled to -40° and then put on load, the working voltage was attained instantaneously. However, if the 33% KOH electrolyte at -40° was added to a battery at the same temperature the time required for the battery to come up to open circuit voltage (OCV) was about five minutes and the voltage decreased below the minimum acceptable level upon application of the load and did not recover for a further ten to fifteen minutes. These observations were unexpected as a number of commercially available dynel, polypropylene and nylon materials have been used successfully as separators in zinc/air batteries. However, in the normal configuration of the reserve-primary zinc/air, the battery is activated by the addition of water which forms the electrolyte by leaching out powdered KOH from the porous zinc anode. Thus activation of temperature below the freezing point of water is not possible.

The company developing the battery attempted to overcome this wettability problem by substituting filter paper for the dynel separator. Initial results were encouraging but during evaluation of the batteries it was found that the quality of the paper was not uniform. Some samples would shrink and tear when wetted with electrolyte allowing the formation of anode to cathode short circuits. Other samples remained whole and performed satisfactorily (2).

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In view of the foregoing it was decided to survey a variety of available materials to determine if any were suitable for the desired application. The ultimate goal was to obtain a separator which would ensure rapid activation of a zinc/air bicell at any temperature within the range -40° to 50° C, rapid activation being defined as (a) attainment of a stable OCV greater than 1.4V within 1 minute of the addition of electrolyte, and (b) attainment of a working voltage greater than 0.9V within 30 seconds of the initiation of a current drain equivalent to a current density of 15 mA/cm², the drain being initiated not more than 5 minutes after the addition of electrolyte. In addition the material would be required to remain in satisfactory physical condition during the full discharge of the bicell.

EVALUATION OF AVAILABLE SEPARATOR MATERIALS

With most zinc/air batteries the separator material is made into a bag to enclose the zinc anode but there are advantages to applying the separator directly to the air cathode. This construction eliminates the entrapment of air in the separator bag which sometime occurs during activation. In the preparation of the cathode a hydrophobic film is pressed onto the air side and the pressing of a separator film onto the other side could easily be incorporated in the manufacturing process. In some cases the two films could be applied simultaneously.

The first step in the test program was to press a piece of the candidate material onto the electrolyte side of an air cathode which was then mounted vertically over an opening in a lucite box equipped with a nickel screen counter electrode. The preparation of the carbon-based air cathode having silver as the electrocatalyst has been described previously (3). The lucite box and a beaker containing sufficient electrolyte to fill the box were set in an environmental chamber at -40° C. A mercury-mercuric oxide reference electrode was placed so that the luggin capillary was immersed in the beaker. After four hours of cold soaking, the reference electrode was positioned in the lucite box in such a manner that the luggin capillary touched the candidate material and the electrolyte was added to the box. If the air cathode reached a stable OCV within 15 minutes of the addition of electrolyte this time was recorded and 2 minutes later a current of 150 mA (26.4 mA/cm²) was impressed and the cathode potential plotted as a function of time. A polarization of not more than 70 mV greater than that exhibited under the same conditions by a cathode without a separator was considered to be acceptable. If a stable OCV was not attained, the sample was discarded.

The following materials were investigated:

153MST5	- laquered cellulose film made by Du Pont of Canada Ltd.
Celgard K-72-4	- microporous polypropylene by Celanese Plastic Co.
Webril E 1451	 non-woven polypropylene fabric from Kendall, Fiber Products Division.
Webril E 1489	- non-woven dynel fabric from Kendall.
RA Grade 202	- Filter paper made by Reeve Angel.
RA 934AH	- Glass fiber filter by Reeve Angel.
	72-4, E1451 and E1489 have all been used successfully as ther zinc/air systems.

The results of the survey are summarized in Table I.

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TABLE I

Effect of Commercial Separator Materials on Air Cathode Performance at -40°

Separator	Application Conditions	Time	Cathod	e Potent	ial (V)
		to OCV (min)	0	1	5 (min)
None		<0.5	-0.036	-0.029	-0.028
153MST5	Heat-sealed at perimeter	>15	-	-	-
Celgard K-72-4	Pressed at 100°C, 112 kg/cm ²	>15	-	-	-
Webril E 1451	Pressed at 100°C, 112 kg/cm ²	>15	-	-	-
RA 202 (1 layer)	Cold pressed at 112 kg/cm ²	<0.5	-0.062	-0.060	-0.054
RA 202 (2 layers)	Cold pressed at 112 kg/cm ²	<0.5	-0.093	-0.092	-0.092
Webril E 1489	Pressed at 125°C, 112 kg/cm ²	<0.5	-	-	-2.0
Webril E 1489	Cold pressed at 112 kg/cm ²	<0.5	-2.3	-2.0	-1.0
202 + E 1489	Cold pressed at 112 kg/cm ²	>15	-	-	-
202 + E 1451	Pressed at 125°C, 112 kg/cm ²	>15	-	-	-
RA 934AH	Pressed at 125°C, 112 kg/cm ²	<0.5	-0.045	-0.028	-0.029

Neither the 153MST5 nor the polypropylene films would wet sufficiently to permit the air cathode to reach a stable OCV within 15 minutes of the addition of the electrolyte. The dynel (E 1489) did allow a rapid attainment of OCV but not enough of the cathode surface was in contact with the electrolyte for the maintenance of the impressed current and the cathode polarized completely. Cold application of the dynel to the electrode gave slightly better results than hot but the cathode polarization remained too high for use in a zinc/air cell. Attempts to use a layer of RA 202 filter paper between either a polypropylene or dynel separator and the air cathode did not produce the wicking effect desired. The time to OCV was lengthened rather than reduced.

Only cathodes having either RA 202 filter paper or RA 934AH glass fiber filter as the separator gave acceptable electrochemical performance. Attainment of a stable OCV requires less than 0.5 minutes and the increase in polarization attributable to the separator was negligible. However, on disassembly of the cells, the glass fiber filter disintegrated and the filter paper separator was found to be torn. Thus none of the materials tested is suitable for use in a reserve-primary zinc/air battery.

DEVELOPMENT OF SUITABLE SEPARATOR MATERIALS

PRELIMINARY SURVEY OF GLASS-FILLED PAPERS

The DREO Pilot Plant is equipped to make papers having a wide variety of compositions. The hand sheet mold can be used to prepare 8 in. square sheets and is, therefore, well suited for an investigation of the effects of various parameters on the desired properties of the paper.

Initially this device was used to prepare samples of glass-wood pulp, glass-rayon and glass-Vinyon (a co-polymer of vinyl acetate and vinyl chloride) papers. In order to facilitate a comparison of the properties of these papers, the samples were all made with a basis weight of 90 g/m^2 .

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TESTS FOR WETTABILITY AND MECHANICAL STABILITY

Before applying a sample to an air cathode and performing the evaluation described in the preceding section, the material was first subjected to the following simple tests for wettability with electrolyte at -40° and for mechanical stability in electrolyte at 75°, a temperature typical of a battery during discharge at 50°C.

(a) Wettability: A sample of about 5 cm in diameter was glued to the lip of a petri dish which was placed in an environmental chamber along with a glass dropper and a beaker of electrolyte. After being equilibrated at -40° , drops of electrolyte were placed on the sample and its wettability judged to be poor, fair or good.

(b) Mechanical stability: A sample was pressed onto an air cathode which was then immersed in a beaker of electrolyte maintained at 75° by a hot plate After 4 hours the air cathode was removed and any tearing or change of shape noted.

Samples which were judged to have fair or good wettability at -40° and did not show tears or more than moderate shrinkage after exposure to electrolyte at 75° were subjected to the evaluation described in the preceding section.

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The results of this preliminary screening of glass-containing papers are recorded in Table II. In every case the time of OCV was less than 0.5 minutes and the cathode potential observed on application of the load remained unchanged for 5 minutes. The contribution of the separator to electrode polarization was obtained by subtracting the potential of a bare cathode (Table I) from the electrode potential observed after 5 minutes of operation.

The glass-Vinyon and glass-wood pulp (without resin) samples had acceptable wettability at -40° but the samples with either of the polymer additives did not. All samples except the 67/33 Glass/Wood Pulp withstood exposure to electrolyte at 75° without significant changes in shape despite the fact that the glass content dissolved under these conditions. For the 50/50 Glass/Vinyon and 50/50 Glass/Wood Pulp, the contribution of electrode polarization was considerably less than the maximum allowable of 70 mV at -40° and 26.4 mA/cm².

During these studies it was noted that the glass-wood pulp papers tore very easily when wet with electrolyte while the glass-Vinyon samples did not. As the glass-Vinyon samples were equal or superior to the others in terms of wettability at -40° , mechanical stability at 75° and contribution

TABLE II

Evaluation of Various Glass-Filled Papers

Composition % w/w	Wettability at -40°C	Shrinkage at 75°C	Contribution to Polarization (mV)*
50/50 Glass/Vinyon	good	none	19
50/50 Glass/Wood Pulp	fair	none	29
50/50 Glass/Wood Pulp plus 6 Parez Resin	poor	none	
50/50 Glass/Rayon plus 2 Polyvinyl Alcohol	poor	none	-
67/33 Glass/Wood Pulp	good	disintegrated	

* At -40° and a current density of 26.4 $\rm mA/cm^2$

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to polarization, all further studies were limited to this type of paper.

DEVELOPMENT OF GLASS-VINYON SEPARATORS

Small quantities of paper were made to investigate the effects of varying the fiber diameter of the glass, the ratio of glass to Vinyon and the basis weight.

Type 475 glass from Johns-Manville of the following fiber diameter

Code 102 0.2 µm Code 104 0.2-0.4 µm Code 106 0.5-0.75 µm Code 108 0.75-1.59 µm

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were used along with Vinyon HH (No. 23, Bright, 3 denier, ¼ inch) from FMC Corporation.

Samples which met the wettability at -40° , mechanical stability in electrolyte at 75°, low contribution to cathode polarization as well as reasonable wet and dry strength requirements were tested in a zinc/air bicell. The bicell, shown in Figure 1, consists of a central cavity in which is placed a 2.7 g porous zinc anode and on either side of which is positioned an air cathode in such a way that the side covered with the candidate separator sample is facing the zinc. With the appropriate plastic frames and rubber gaskets, the device can be clamped together to form a leak-free electrochemical cell. The cathodes are joined by soldering a strip of silver-coated copper to the cathode tabs and the bicell is activated by the introduction of 33% KOH electrolyte through a filling port into the central cavity. The bicell and the preparation of porous zinc anodes will be described in detail in a later report.

To evaluate a separator sample the bicell was placed in an environmental chamber at -40° and after 2 hours activated with electrolyte which had also been equilibrated at that temperature. Five minutes after activation the cell was discharged by the impression of a constant current of 200 mA (16 mA/cm² on each electrode surface) from a power supply. The circuit was arranged so that a decrease in cell voltage below 0.90V stopped the discharge and the interval timer used to record the duration of the discharge to the nearest 0.1 minute. In all cases an OCV of greater than 1.4V was reached before all of the electrolyte had been added to the cell. The five minute delay before applying the load was required for the chamber to return to the designated temperature from the fluctuation caused by opening the chamber door to activate the cell.

Separator evaluations were carried out in a similar manner with the environmental chamber controlling the temperature at 50°. With every discharge a fresh zinc anode and a newly prepared set of cathodes equipped with pieces of the material to be tested were used.



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From previous work it was known that the duration of discharge at -40° under the above conditions was 128-130 minutes for a bicell having RA 202 filter paper as the separator material provided the paper did not tear. Thus any separator permitting a discharge of 130 minutes or more was considered to be acceptable for use at -40° . At 50° an absence of voltage fluctuations which might indicate the presence of intermittent internal anode to cathode shorting and a discharge time of at least 500 minutes (75% utilization of the zinc in the electrochemical discharge reaction) were used as the acceptability criteria.

The results of the investigation of glass-Vinyon papers are summarized in Table III and discharge curves at -40° and 50° for bicells equipped with Sample 8 separator material shown in Figure 2.

EFFECTS OF FIBER DIAMETER

Papers containing glass fibers of the largest diameter (Code 108) failed to wet when exposed to electrolyte at -40° and as the particle diameter was decreased, an increasing number of samples failed the test for mechanical stability in electrolyte at 75° C. Adjustments in the percentage of glass used and the basis weight gave samples containing Code 106, 104 or 102 which passed not only the tests mentioned above but contributed little to electrode polarization and permitted zinc/air bicells to be discharged for the required times at -40° and 50° C.

The tensile strength of a paper sample was also affected by the diameter of the glass fiber particles. When a constant percentage of glass and a constant basis weight were maintained, the tensile strength increased as the fiber diameter decreased (compare samples 3, 7 and 16).

EFFECTS OF GLASS CONTENT AND BASIS WEIGHT

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For any given glass fiber diameter, samples having a glass content higher than a certain value failed the test for mechanical stability in electrolyte at 75°C. The highest permissible content was 50% for Code 104 glass. The tensile strength was also found to decrease with increasing glass content.

Most of the studies on the effects of glass content and basis weight on electrochemical measurements (polarization and discharge times) were performed with papers made from Code 104 glass. The longest discharge times at -40° were obtained with 40% glass and basis weight 114 g/m² (sample 8) and 50% glass and basis weight 84 g/m² (sample 10).

In the limited range of papers made with Code 102 glass, a bicell equipped with sample 14 (30%, basis weight 91) gave a -40° discharge nearly



TABLE III

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Properties of Glass-Vinyon Papers

							0	110	Lin	00			2						
Time**	nin			562		557	500	546	546	582	570	579			531	513	test		
Discharge Time** -40° 50°	nin	lity test	lity test	154	lyte at 75°	146	134	142	152	134	150	139	lyte at 75°	lyte at 75°	169	131	lyte at 75°	lyte at 75°	
Contribution to		failed wettability test	failed wettability test	21	failed electrolyte at 75°	27	20	28	30	п	19	23	failed electrolyte at	failed electrolyte at	25	17	failed electrolyte at 75°	failed electrolyte at 75°	
Burst	li/in ²			5.2	4.1	6.0		5.2		3.0	3.6	5.6	3.6	4.7	6.2	5.3	5.3	4.9	
Tensile Strength	1b/in			2.3		3.1				1.3	2.0	2.4	1.6						
Tensile	lb/in			2.1	1.6	2.8		2.5		1.0	1.9	2.4	1.6	2.4	3.0	1.9	2.7	2.1	
Density	g/cm ³			0.229	0.235	0.222		0.250		0.246	0.234	0.244	0.247	0.255	0.239	0.238	0.249	0.277	
Thickness	mil			15.2	15.3	16.2		14.5	17.9	8.1	14.1	18.8	13.6	17.6	14.9	9.8	14.5	13.8	
Basis	8/m ²	75	151	88	92	92	99	92	114	51	84	117	86	114	91	59	92	87	
SS	*	40	40	40	50	30	40	40	40	50	50	50	60	60	30	40	40	20	
Glass	Code	108	108	106	106	104	104	104	104	104	104	104	104	104	102	102	102	102	
Sample		1	2	3	4	2	9	1	•	6	10	п	12	13	14	15	16	11	

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** For a Zn/Air bicell with a 2.7g anode discharged at 200 mA (16 mA/cm²).

* At -40° and a current density of 26.4 mA/cm².

40 minutes longer than one equipped with sample 15 (40%, basis weight 59).

Although ten of the seventeen different paper samples prepared met the prescribed criteria for acceptability as a separator material, it is evident that some performed better than others, particularly during discharge of bicells at -40°, and that performance was related to glass fiber diameter, glass content and basis weight. As the limiting factor in the overall capabilities of zinc/air batteries is their poor performance at low temperature (Figure 2), this factor has been emphasized more than the length of discharge at 50°. None of the bicells discharged at 50° showed signs of penetration of the separator by zinc. Such penetration would cause internal shorts and would result in fluctuations of the cell voltage whereas all cells displayed very smooth discharge curves (Figure 2). Neither was there any visible evidence of separator disintegration when bicells that had been discharged at 50° were disassembled. Despite the fact that measurements of the loss of weight of the separator during the exposure to electrolyte at this temperature indicated that all of the glass had dissolved, the remaining material was untorn and sufficiently dense to prevent the formation of internal shorts in the cell.

MACHINE-MADE GLASS-VINYON PAPERS

Laboratory hand-sheets were useful for evaluating a number of different papers but machine made paper would be required for battery manufacture. The DREO Experimental Paper Machine is capable of producing papers of this type in a continuous roll.

Because large quantities of materials are required for a machine run, it was decided that only two or three sample formulations would be used. From a careful examination of the results with laboratory hand sheets it was decided to prepare the following papers:

> 30% Code 102 glass - 70% Vinyon at basis weight of 70 and 90 g/m². 40% Code 104 glass - 60% Vinyon at basis weight 70 g/m².

Aqueous suspensions of these fiber mixtures were stored in separate tanks and pumped to the machine, where a uniform fiber web was deposited on a moving screen which allowed the water to drain away. The wet web was transferred to a moving felt, and after being lightly pressed between rolls, was led through the drying cabinet where it was passed around six heated drums. On leaving the cabinet the paper was collected on a take-up roll.

The run, which was designated Run 216, was started with the fiber stock made from Code 102 glass. When conditions had stabilized such that the basis weight of the product was about 70 g/m² Sample 1 was taken, the start-up paper discarded and the collection of separator material on Roll 2 started. After about 2.5 lb of paper had been collected a second sample was taken and the speed of the machine decreased to produce a heavier paper. The transition paper was discarded and after conditions had again stabilized, a second produce roll (Roll 4) was collected. Samples 3 and 4 were taken from the

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beginning and end of this roll. Supply to the machine was then switched to the other fiber formula and a third product roll (Roll 6) produced. Samples 5 and 6 were taken from the beginning and end of this roll.

Thus Run 216 resulted in the production of three different separator materials.

Roll 2. 2.53 lb of 30% Code 102 glass of basis weight 70 g/m^2 . Roll 4. 2.50 lb of the same formula but basis weight 90 g/m^2 . Roll 6. 1.12 lb of 40% Code 104 glass of basis weight 95 g/m^2 .

The physical properties of these papers, including those calculated by averaging the values measured for samples taken at the start and finish of each roll, are recorded in Table IV. In addition the %Ash for each sample is given. As the ash represents the glass content of the sample, these figures confirm the stated glass/Vinyon ratio and demonstrate the uniformity of the composition of the materials produced.

For comparison, physical data supplied by the manufacturer for Webril E 1489 are also included in Table IV. This dynel is the material originally selected for use in a reserve primary zinc/air battery (1) but which was rejected because of its poor wettability at -40°. As it was considered necessary to have a double layer of E 1489 for a suitable separator, the weight per bicell of separator would have been comparable with that for the glass-Vinyon papers but the thickness would have been halved. The extra 7 mil thickness of paper separator is not considered to significantly affect good bicell design. The tensile strengths of the paper samples are slightly less than that of the dynel when measured in the machine direction but greater in the cross direction. Therefore the paper is expected to be as resistant to tearing during battery manufacture and operation as the dynel.

The electrochemical properties of air electrodes and bicells equipped with machine-made glass-Vinyon separators are given in Table V.

It is evident that the discharge times at -40° are considerably shorter than would be expected from the results recorded in Table III. Because of a breakdown of the environmental chamber normally used, all of the discharges listed in Table III were carried out in an unfamiliar chamber while those recorded in Table V were done in the repaired chamber. A calibration of the thermometers in the two chambers with a Fluke 2100A Digital Thermometer and an iron/constantan Type J thermocouple revealed some discrepancy in their readings. The results in Table III were actually obtained at -39.2° and those in Table V at -40.6° .

In order to demonstrate that this difference in temperature accounted for the differences in discharge times, a bicell was made up with the separator material designated as Sample 14 in Table III and discharged in the repaired chamber. The results, which are included in Table V, show that the discharge time was reduced from 169 minutes at -39.2° to 139 minutes at -40.6° . As sample 14 had approximately the same composition and basis weight as the material on Roll 4, it is evident that hand-made and machine materials are equally satisfactory as separators for a reserve-primary zinc/air system.

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Material from Roll 4 is presently being used as the standard separator

TABLE IV

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Properties of Machine-Made Glass-Vinyon Papers

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						Ten	sile S	Tensile Strength*	:h*		
Sample	61a	Glass	Basis Wt	Thickness	Density	Dry MD CD	r E	M	Wet CD	Ash	
	Code	%	g/m²	mi.1	g/cm ³	1P/	th.	1b/in	in	%	
1	102	30	71.	13.	0.218	3.0	2.5	3.2	2.8	29.1	
2	102	30	68.	12.	0.225	3.2	2.4	3.3	2.4	29.1	
9	102	30	87.	15.	0.229	4.0	3.1	4.0	3.4	29.2	
4	102	30	92.	16.	0.231	4.4	3.2	4.8	3.5	28.3	
5	104	40	.66	16.	0.241	3.5	2.5	3.7	2.8	39.2	
9	104	40	94.	15.	0.245	3.4	2.5	3.5	3.0	39.5	
Rol1 2	102	30	70.	12.	0.222	3.1	2.5	3.3	2.6	29.1	
Rol1 4	102	30	.68	15.	0.230	4.2	3.1	4.4	3.4	28.7	
Roll 6	104	40	.96	16.	0.243	3.4	2.5	3.6	2.9	39.4	
E 1489	(dyne]	lel)	44.	4.		4.8	1.6				
¥		ifne Di	ne Direction.								
C	CD = Cross		Direction.								

TABLE V

Sample No.	Contribution to Polarization*	Discharge -40°	Time** 50°C
	mV	min	mir
Roll 2	23	124	520
Roll 4	29	137	560
Roll 6	24	137	520
14†	25	139	530

Glass-Vinyon Separators on Air Electrodes and in Bicells

* At -40° with a current density of 26.4 mA/cm^2 .

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** For a Zn/Air bicell with a 2.7g anode discharged at 200 mA (16 mA/cm²).

+ Composition the same as Sample 14 in Table III.

in an investigation aimed at optimizing the zinc anode structure and electrolyte composition to be used in this type of battery. More than fifty bicells have been discharged at temperatures throughout the range -40° to 50° C without encountering any cases of separator failure.

CONCLUSIONS

1. No commercially available material was found to be suitable for use as a separator in a reserve-primary zinc/air battery. Either poor wettability when exposed to potassium hydroxide electrolyte at -40°C or shrinkage and tearing during use at higher temperatures eliminated all the candidate samples tested.

2. An investigation of glass-filled papers prepared at the DREO Pilot Plant showed that glass-Vinyon papers had the required properties of wettability and strength.

3. The length of discharge of a zinc/air bicell equipped with glass-Vinyon separator material was found to depend on the glass fiber diameter, the percentage glass and the basis weight of the paper.

4. Machine-made glass-Vinyon paper of an optimized composition performed well as a separator in bicells discharged over the temperature range -40° to 50° C.

ACKNOWLEDGEMENT

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It has been found that separate batteries do not wet readily when play at -40°C. They are, therefore, unsuital battery which must be capable of rapi temperature in the range -40°C to 50° A glass-Vinyon separator who of battery has been developed.	ced in contact with a ble for use in a rese d activation and oper C.	lkaline electrolyte rve-primary zinc/ai ation at any
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March 19, 1977

To: All Holders

Defence Research Establishment Ottawa DREO Report No. 756 Separator material for reserve-primary zinc/air batteries, by W.A. Armstrong and J.A.Wheat.

page 1, 1st. paragraph, 16th line - should read "Thus activation at temperatures below the freezing..."

Wintel

for D.A. Hackie, Librarian for Director General