HIGH CYCLE LIFE 30 AH
SEALED SILVER OXIDE-ZINC CELL
DEVELOPMENT

JET PROPULSION LABORATORY
CONTRACT NO. 952472

FIRST QUARTERLY REPORT
(FEBRUARY 14, 1969 TO
MAY 14, 1969)

ESB REPORT NO. E-23-69

This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, as sponsored by the National Aeronautics and Space Administration under Contract NAS7-100.

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EXIDE MISSILE AND ELECTRONICS DIVISION
RALEIGH, NORTH CAROLINA

16 JUNE 1969

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TECHNICAL CONTENT STATEMENT

This report contains information prepared by ESB Incorporated, Exide Missile and Electronics Division, under Jet Propulsion Laboratory subcontract. Its contents is not necessarily endorsed by the Jet Propulsion Laboratory, California Institute of Technology, or the National Aeronautics and Space Administration.

ABSTRACT

Work during this quarter included cell pack design, new separator evaluation, initial development of wedge shaped teflonated negative plate and procurement of cycle life test panels. Cells have been designed and are in production for a cycle life experiment requiring 90-70% to 50% depth of discharge (DOD) cycles. Twelve cell design modifications include the prime variables:

- Weight ratio ZnO to silver
- Concentration KOH
- Negative electrdde shape: flat vs wedge
- Method of plate wrap
- Type of separator: RAI 2291, FSC,irradiated EM-476 and EM-470.

TECHNICAL SUMMARY

The object of the work is to develop a silver oxide-zinc sealed cell capable of 90 50% depth of discharge cycles (1 cycle per day) after a 7-month interplanetary trip to Mars and 1-2 months charged stand before launch.

Initial work has involved setting up a sixty cell cycle life experiment consisting of 50 70% plus 40 50% DOD cycles to test twelve different designs having the prime variables:

- Weight ratio ZnO to silver: 1.4:1 and 1.2:1.
- Concentration KOH electrolyte, 90% saturated with ZnO: 45% and 41%.
- Negative electrode shape: Flat and wedge (thicker at plate top).
- Mode of plate wrap: "Z" or accordion wrap, positive wrap, negative wrap and both plates wrapped.
- Type of separators: RAI 2291, fibrous sausage casing, EM-476 irradiated, EM-470.

Cycling will involve the following:

- An automatic cycling circuit to handle each significant design modification on a 22 hour charge - 2 hour discharge regime for 90 days.
- Modified constant potential charge (setting of 1.94 volts per cell) with the current limited.

- 70% depth of discharge of 30 AH until first cell capacity drops below 21 AH, then 50% depth of discharge or 15 AH to 90 cycles or failure.
- Periodic 100% DOD cycles during cycling as a measure of negative plate erosion.

Cell designs were completed during the quarter and plates manufactured, including wedge shaped negatives, demonstrated conformance to drawings. Test circuits were designed and parts are on order for 10 separate circuits for the 12 design variations. Cycling testing will demonstrate whether any of the test designs represent major improvements over the cell design now in use by comparison to a 9-cell row assembly of Mariner '69 cells cycled under the same conditions.

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I. INTRODUCTION

ESB Incorporated, Exide Missile and Electronics Division was awarded JPL Contract 952472 for the continued development of float type Mariner cells and 18 cell batteries. This work was redirected by Modification 1 to design and development of sealed high cycle life 30 ampere-hour silver oxide-zinc cells for the 1973 Viking Mars mission. Cells are to be capable of four 20-70% cycles in 15 days plus 90 or more charge/discharge cycles at 50% depth of discharge (DOD) over a period of 10 months. Six different cell designs have been completed as the first step in cell development and are detailed in sections to follow. JPL requirements in the form of design goals and design considerations are presented in the Appendix.

II. TECHNICAL DISCUSSION

A. Cell Designs Selected. - The 30 AH cell was designed into Mariner '69 type cell cases retaining single cell and plate dimensions and seal type. The experimental design variables for 12 different 30 AH cell designs are presented in Table I and include two electrolytes, two ZnO/Ag ratios, five separator systems, and three wrapping styles.

1. Electrolyte Concentration. - Two concentrations - 45% and 41% 90% saturated with ZnO - were selected for test. The upper concentration is desirable for long life to retard Ag ion diffusion from positives to negatives and oxidative hydrolysis of cellulosic membranes. The lower concentration gives better charge and discharge voltages and ampere-hour efficiencies at the C/2 rate at 50°F. The effect of the concentration change is expected to be greater for the separator systems having proportionately larger fractions of irradiated polyethylene membranes.

2. ZnO/Ag Active Material Ratio. - Lander[1] has observed that the cycle life of 24 AH sealed Ag-Zn cells increases from 300 to 1140 cycles on a 2 hour orbit at 25% DOD and 75°F in 45% KOH when the molar ratio of ZnO/Ag is increased from 1/1 to 2/1. Charkey[2] has observed that the cycle life of 5 AH nickel-zinc cells containing 35% KOH and tested at 70% DOD on 8 hour charge/1 hour discharge/1 hour rest increased from 50 to 175 cycles when the ratio of molar capacities (-)/(+) was increased from 0.88 to 1.68. The ratio effect, according to Strauss [3], is a function of the true depth of discharge of the negative plates, and cycle life increases with decreasing depth of discharge. On the basis of the above data and hypotheses the following ratios were selected and approved by JPL:

Ratio By Weight (ZnO/Ag)	Molar Ratio (ZnO/Ag)
1.2/1	1.59/1
1.4/1	1.86/1

Ratio 1.4/1 or 1.86/1 is almost twice that of Mariner '69 cells (0.76 by weight or 1.0/1 by molar ratio) now at JPL. Both 1.2/1 and 1.4/1 ratios are considered more than adequate to give 90 50% DOD cycles and will give a measure of the ratio on delivered voltage and energy density.

3. Separators Wraps. - Three wrapping styles are to be tested. Table I shows a positive wrap for designs 2 and 3, negative wrap for designs 1, 4, and 5, and a combined wrap for design 6. Each wrapping style places the reservoir of electrolyte at a different location in the cell:

Wrap Style	Location of Reservoir
o Positive Wrap	Around Negatives
o Negative Wrap	Around Positives
o Combined	Between 3rd and 4th layer of RAI 2291 of design 6

For long term cycling on a 22 hour charge and 2 hour discharge the cell reaction and osmosis will favor transfer of water from positive to negative compartments, raising electrolyte levels around negatives, lowering levels around positives, tending to starve positive and polarize the cell. The small pore diameter of RAI-2291 should aggravate this condition. On the basis of this hypothesis capacity should be maintained best for the system with least fluctuation of levels and least concentration changes at positives and negatives. A predicted ranking applicable for present specification requirements would be negative wrap better than combined wrap better than positive wraps. One cell of each test group will be manufactured in a clear cell jar material to permit observation of levels during cycling.

4. Separator Type.- Test separators will be two absorbers - irradiated Kendall EM-476 obtained from RAI Research Corporation and Kendall EM-470; and two membranes - Visking fibrous sausage casing and RAI 2291. Table II lists the properties of the test membranes and DuPont 193 PUDO cellophane, the membrane of present Mariner type cells. Table III lists EMED data and literature properties of the proposed test absorbers and non-irradiated Kendall EM-476 used in Mariner '69 cells.

The combination of separator systems in the cell designs of Table I is intended to determine the system most capable of retarding silver, zinc penetration, and negative plate erosion and to improve both cycle and wet life over the present Mariner '69 cell system.

5. Common Design Features. - In the sixty test cells now under

construction the following design features are common to all cells:

• Active silver weight	123 g
• Active silver density	69 g/in^3
• Number of positive plates	8 or 10
• Negative active mix	
ZnO	90%
HgO	3%
Teflon	7%
• Negative process	Sintered
• Negative plate density	45 g/in^3
• Negative grid (2/plate)	2/0 distorted
• Pack tightness	1.25 psi minimum
• Cell jar material	ABS
• Cell jar sealant	Epoxy and ABS cement
• Zinc moss block	Turned over top edge of separator system

Of the design features above the changes from the Mariner '69 design expected to improve cycling capability are the decrease in active material density (positive and negative), the reduction in percent HgO in the negative mix, and the sintered teflonated plate process.

B. <u>Cell Design Relationships</u>. - Cell designs 1 through 6 of Table I were based on a common set of design equations. The relative thickness of negative and positive active material (T-) and (T+) was obtained from

$$T- = \left(\frac{d+}{d-}\right) \circ \left(\frac{W}{f-}\right) \circ \; T+ = 1.71 \; (W)T+ \qquad \text{[Equation 1]}$$

where d_+ = positive active material density = 69.4 gm/in^3

d_- = negative active material density = 45.0 gm/in^3

W = ratio by weight ZnO/Ag = 1.40 or 1.20

F_- = fraction ZnO in negative active material.

Cell pack components were fitted into the cell jar by addition of all <u>wet</u> thicknesses and equating to cell length (C.L.) at the jar bottom:

$$C.L. = N_{+}T_{+} + N_{-}T_{-} + N_{M}T_{M} + N_{A}T_{A} + N_{G+}T_{G+} + N_{G-}T_{G-} + S \quad [\text{Equation } 2]$$

where M = separator membrane

A = separator absorber

G = grid (+) or (-) allowance

S = shim thickness

N = number of components of a particular type

C. Process Development. - Plates were manufactured to prints based on the two design equations above. Table IV summarizes positive and negative plate thicknesses designed and achieved for cell designs 2 through 6. Negative plate thicknesses are values measured after sintering. Tolerances on negative plates were ±3 mils on thickness and +0 -30 mils on width and height. New tooling was found to be necessary to prevent loss of active material from negative plate edges.

The wedge shaped negative plates of design 1 required process and tooling development. Final plates were to print and had the following thicknesses:

Type	Plate Top Mils ±3	Center Mils ±3	Bottom Mils ±3
Full	76	51	26
Half	38	26	14

Wet cell pack thickness and shim thickness were initially calculated using equation (2) and wet thickness values of RAI 2291 and FSC (45% KOH) at 40 oz/in^2 applied pressure obtained from Figure 1 and unpublished data. Wet thickness values of absorbers EM-476 irradiated and EM-470 used were obtained at only one applied pressure, i.e. 2 oz/in^2 on a Randall-Stickney gage. To standardize cell pack tightness recalculations were made using the composite wet thickness of each separator system determined at 40 oz/in^2 on a Randall-Stickney gage after 120 hours in 45% KOH. See Figure 2 and Table V. Wet thickness at 40 oz/in^2 was chosen as the lower limit design thickness since at this pressure the major wrinkles in the system have been smoothed out but some free electrolyte remains between layers. Composite separator system wet thickness values times the number of systems in each design were used in equation (2) in place of the membrane and absorber terms to determine final shim thickness. Designs 2 and 5, containing larger quantities of FSC, have pack tightness values of 20 ounces per in^2 minimum without shims, depending on KOH concentration.

D. <u>Cycle Life Testing</u>. - Ten test circuits have been designed and parts placed on order. The sixty 30 AH cells will be cycled through 90 cycles per Table VI after an eleven day stand at 140°F to simulate a long term interplanetary trip. Cycling will require a 22 hour charge, 2 hour discharge with a modified constant potential charge and a constant resistance discharge. Test parameters are given below for the initial 70% DOD and final 50% DOD cycles:

DOD	Charge		Discharge	
% (C = 30 AH)	CP Volts/Cell (±.01)	Current Limit Amps (Minimum)	Current Amps	Capacity AH
70	1.94	1.0	10.5	21.0
50	1.94	.72	7.5	15.0

Cycling will begin with charge after a discharge depth corresponding to encounter power demands. When the 5- cell groups no longer deliver the required capacity to 1.0 volts on the lowest cell, the depth of discharge will be reduced from 70 to 50% and cycling continued to 90 cycles total. Six 100% depth capacity measuring cycles will be performed during cycling approximately 10 cycles apart as a measure of negative plate erosion. Following cycling representative cells from each group will be dissected to determine failure modes. This test phase will begin early in the second quarter of work.

E. Noryl Thermoplastic as Cell Case Material. - Noryl, General Electric Company polyphenylene oxide modified with polystyrene, is being evaluated as a new case material to replace ABS. General purpose grade Noryl 731 molds readily, has excellent heat resistance, low cost and good mechanical properties. Properties of Noryl 731 are compared to ABS (presently being used in cell cases) in Table VII. Butt tensile strength of epoxy and solvent cemented specimens of Noryl 731 will be investigated. This case material will only be considered if excellent case molding and sealing processes can be obtained.

III. QUALITY ASSURANCE PROVISIONS

During the first quarter of 1969, Quality Assurance submitted to JPL the draft of the ESB Quality Assurance Plan for the Viking Program. The JPL Quality Assurance representative reviewed and made comments and recommendations on the proposed ESB Quality Assurance plan. These recommendations have been incorporated in this program plan.

To give adequate Quality Control coverage of the statistical experiments for the program, Amendment A to QAS-251 was written. Quality Assurance Specification No. 251 is a general document intended to cover the quality and traceability requirements of a complex statistical experiment. Amendment A qualifies QAS-251 for the Viking experiment. It describes the percent inspection, the color coding for traceability and the particular attribute to be verified during inspection. Amendment A was released on a 30 day tentative approval for review for all operating groups.

IV. CONCLUSIONS AND RECOMMENDATIONS

- Six 30 AH cell design types have been drafted and manufactured to plate stages.
- Plate processes have demonstrated dimensions in agreement with design tolerances.
- Noryl 731 is a promising substitute for ABS case materials to improve high temperature characteristics and increase burst pressures, provided reliable sealing processes can be developed.
- Wet separator system thicknesses have been measured over the pressure range 0 - 120 oz/in^2.

V. NEW TECHNOLOGY

No items of new technology have been developed during this period.

VI. REFERENCES

1. John J. Lander - Sealed Ag-Zn Batteries, AIAA Paper 64-749, September 1-4, 1964.
2. A. Charkey - Performance Characteristics of Nickel-Zinc Cells, 23rd Annual Power Sources Conference, 22 May 1969.
3. Howard J. Strauss - General Theoretical Principles of Cell Design, Electrochemical Society Paper, Montreal Meeting, 1968.
4. J. Kelley, NASA Contract NAS5-10418, 2nd Quarterly Report, Table 5.
5. T. H. Meltzer, Alkaline Battery Separator Study, 3rd Quarterly, NAS5-2860, October 11, 1962.
6. Ibid, 8th Quarterly Report.
7. J. Kelley, NASA Contract NAS5-10418, 5th Quarterly Progress Report, Tables II and III.
8. EMED Evaluation Data.
9. W. Jakobi, NASA Contract NAS5-1045, Final Report Table IX.

APPENDIX I

1. Design Goals - JPL Contract 952472 Modification 1. - The minimum goals for the battery cells shall include the following:

(A) Capable of ninety-six (96) or more charge/discharge cycles at 20% DOD to 70% DOD over a period of ten months or more as shown below:

Cycle	Depth of Discharge	Lapsed Time
1	70%	1 Day
2	20%	2 Days
3	20%	5 Days
4	20%	15 Days
5	70%	7 Months
6 to 96 or more	50%	7 Mo. 1 Day to 10 Mo. (1 Cycle/Day)

(B) Capable of one (1) to two (2) months room temperature (75°F) stand after activation but before cycling as required in paragraph (A) above.

(C) Capable of the requirements in paragraph (A) above when operated in a plane 180° from normal and/or while being rotated.

(D) Capable of meeting the environmental requirements set forth in JPL Specification TS500437, Revision B, entitled "Type Approval and Flight Acceptance Test Requirements and Preacceptance Test Limits (Assembly Level) Mariner Mars '69 Flight Equipment, General Specifications For."

(E) The nominal 18 cell energy shall be 750 W/hr. when discharged at 75°F at a rate of 15 amperes.

(F) Capable of operating as specified in paragraph (A) above in the temperature range of 50° to 100°F.

(G) Capable of delivering 75% of the rated capacity during discharge at 50°F at a rate of 15 amperes. The cut-off voltage shall be 1.43 volts per cell.

(H) Capable of recharge in twenty-four (24) hours.

(I) Sealed cell design capable of operating during continuous exposure to space vacuum for one (1) year.

2. Design considerations shall include, but not necessarily be limited to, the investigation and analysis of the following:

(A) Separator configuration on both positive and negative electrodes.

(B) Means of preventing growth of zinc over or out of its separator compartment.

(C) New types of separators.

(D) Cell characteristics as a function of electrode thickness during charge and discharge.

(E) Numbers of layers of separator material.

(F) Electrolyte concentration.

(G) Concentration of negative plate additives.

(H) Electrolyte quantity.

(I) Active material ratio.

(J) Maximum overcharge capability.

(K) Constant current versus constant potential charge methods.

(L) Methods of accelerated testing.

TABLE I

SIXTY 30 AMPERE-HOUR CELL EXPERIMENTAL DESIGN

Contour of Negative Plate	Electrolyte Concentration (90% Sat. ZnO)	ZnO/Ag Weight Ratio	Separator System					
			Positive Wrap		Negative Wrap			Combined
			1L EM-470 4L FSC	1L EM-470 4L RAI-2291 2L FSC	1L EM-476I 6L RAI-2291		1L EM-470 4L RAI-2291 2L FSC	3L RAI-2291 + 1L EM-476I 4L RAI-2291-
Wedge (1)	45 (SS-246 Type 7)	1.2:1			5			
Standard	45 (SS-246 Type 7)	1.2:1	5	5	5		5	
Standard	41 (SS-246 Type 5)	1.2:1	5	5		5	5	5
Standard	41 (SS-246 Type 5)	1.4:1				5		5
		Total Cells	10	10	10	10	10	10
		Design No.	2	3	1	4	5	6

(1) Thicker at plate top.

TABLE II

PROPERTIES OF SEPARATOR SYSTEM MEMBRANES DRY OR WET IN 31% KOH

Test Parameter	Unit	193 PUDO	FSC	FAI-2291
Thickness, dry	Mils	1.0	2.9-3.5	1.2
wet		3.0	7.0-8.6	1.3
Electrolyte Absorption	g/cc	3.4	3.2 (5)	0.5
Dimensional Changes (roll) W	% $\left(\frac{Dw-Dd}{Dd}\right)100$	6	0	3
L		-3	-10	3
T		200	150	10
Electrical Resistance	a.c. ohm-cm^2	0.065	.17	0.35-0.69
	ohm-cm	8.6	8.2 (6)	100-200
Tensile Strength 72°F	psi			
Dry		12,210 (4)	14,570 (6)	1850 (7)
Wet		4,100 (24 hr. in 40% KOH)	6,800 (15 minutes in distilled water)	1350 (24 hr. in 40% KOH)
Silver Permeability	$\frac{\text{Moles Ag}}{cm^2 \text{ sec.}} \times 10^{10}$	35.5	--	4.8
Silver Absorption	$\frac{\text{g Ag} \times 10^6}{in^2 \text{ hr.}}$	62.7	--	0.73
Zinc Diffusion	$\frac{\text{Moles Zn} \times 10^7}{cm^2 \text{ sec.}}$	2.7	--	0.08
Zinc Penetration (40% KOH)	hrs/cm x 10^{-2}	2.0	2.9	10.7
	minutes/mil	80	193	----
Zinc Absorption	K value	0.87	--	0.84 (40% KOH)
Tortuosity		2.6	2.1-2.5	
Pore Size	Å	13	15-16	1.4-2.3 (40%)
Oxidation Resistance	% retained tensile strength	0	30-40	100

Note Dw = Wet dimension in inches
Dd = Dry dimension in inches

TABLE III

PHYSICAL PROPERTIES OF POSITIVE PLATE ABSORBERS (7)

Test Parameter	Unit	EM-476 Non-Irradiated Polypropylene	EM-476 Irradiated Polypropylene	EM-470 (309) Dynel
Thickness, Dry	Mils	4.0	4.0	1.0
Wet		4.0	4.0	1.0
Weight, Dry	mg/in^2	23	23	1.2
Electrolyte Absorption	$\left(\frac{Ww-Wd}{Wd}\right)$ 100	220 40% KOH	177 (8) (40% KOH)	90 (9) (25% KOH)
Electrical Resistance 40% KOH	$ohm\text{-}cm^2$	0.39	--	--
Air Permeability	Seconds to pass 300 cc	20	--	2.7
Wettability Spreading Time, 30% KOH	Seconds	321	--	7.5
Wicking Height 40% KOH	Inches	1.6 (at 30 minutes)	2.0 (8) (at 40 minutes)	0
Wettability to Minimum Resistance	Minutes 40% KOH	51	--	0.5

Note: Ww = Wet weight in grams
Wd = Dry weight in grams

TABLE IV

ACTIVE MATERIAL AND PLATE THICKNESS

ZnO To Silver Weight Ratio	Positives*		Negatives* By Design Type									
			2		3		4 & 1		5		6	
			Full	Half	Full	Half	Full	Half	Full	Half	Full	Half
1.2:1	Active Material	23			48	24	48	24	48	24	48	24
	Plate	25			51	26	51	2	51	26	51	26
	Active Material	29	59	30								
	Plate	31	62	31								
1.4:1	Active Material	23					55	28			55	28
	Plate	25					58	30			58	30

(*) Thickness in mils.

Tolerances: ±1.0 on positives

±3.0 on negatives.

TABLE V

WET THICKNESS OF COMPOSITE SEPARATOR SYSTEMS AFTER
120 HOURS IN 45% KOH

Composite Separator System	Cell Design Number	Wet Thickness (40 oz/in^2) Mils
4 L FSC 1 L EM-470	2	36.6
4 L RAI 2291 2 L FSC 1 L EM-470	3 & 5	23.5
6 L RAI 2291 1 L EM-476 Irradiated	1 & 4	10.6
7 L RAI 2291 1 L EM476 Irradiated	6	12.2

Instrument: Randall-Stickney Platform Thickness Gage.

TABLE VI

60 CELL TESTING PROFILE

Event	Cells Per Group									
• Formation charge	60									
• 1st cycle discharge, 15 amps 75°F										
• Recharge										
• Eleven day stand at 140°F and top off charge										
• Discharge, 100% DOD										
• 50 autocycles, 22/2, 70% DOD plus 2-100% DOD cycles	5	5	5	5	5	5	10	5	5	10
• 40 autocycles, 22/2, 50% DOD plus 3-100% DOD cycles	5	5	5	5	5	5	10	5	5	10
• Dissection and inspection	2	2	2		2		4	2		2
• Balance to be left on cycling (time and circuits permitting)	3	3	4	4	4	4	6	4	4	8
Design Type	1		2		3		4	5		6

TABLE VII

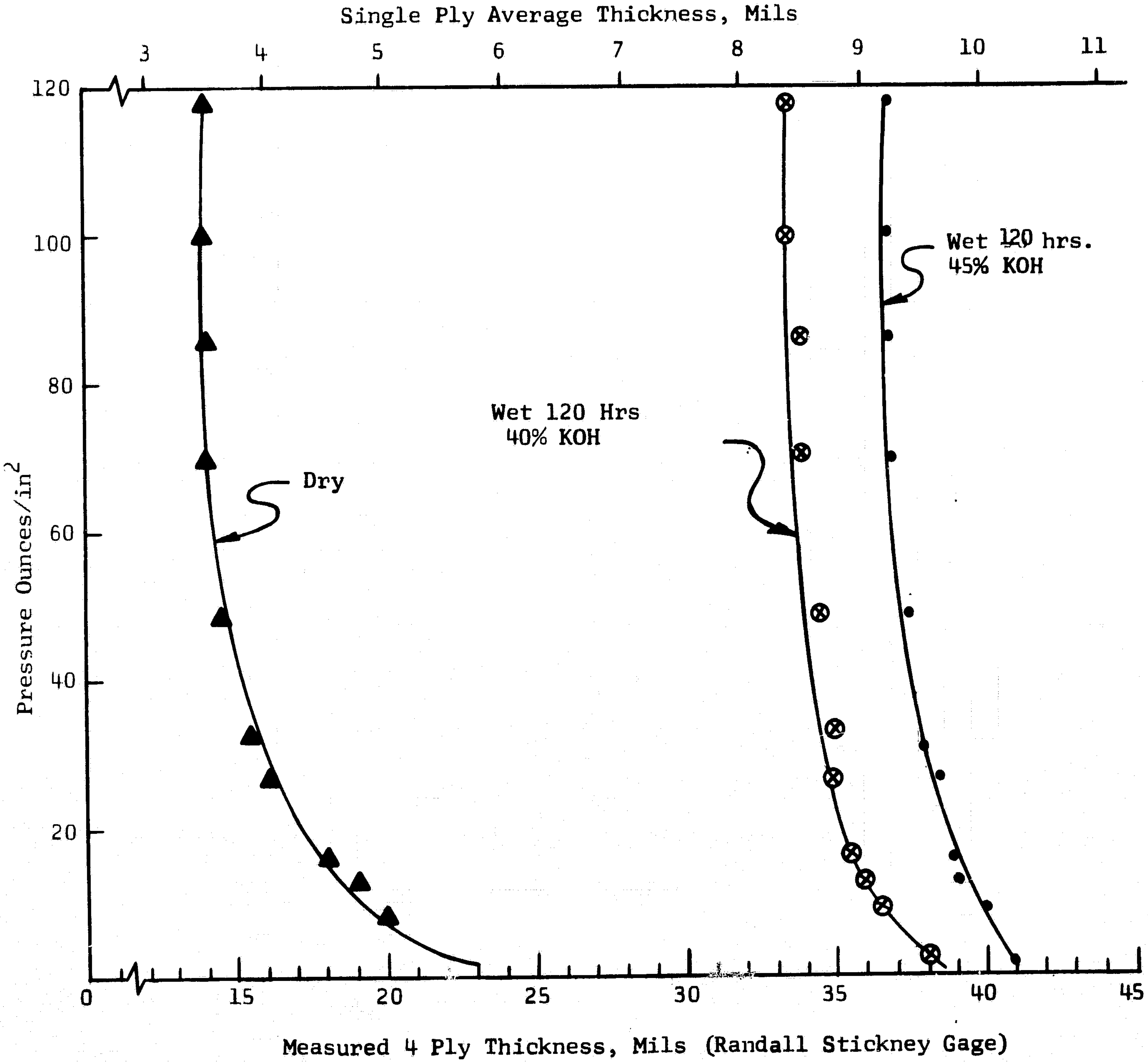
TYPICAL PROPERTIES OF NORYL 731 AND ABS HIGH IMPACT MOLDING MATERIAL

Property	ASTM No.	Temp. °F	Unit	ABS T- Grade	Noryl 731
Tensile Strength	D638	73 200	psi	5,900	9,600 6,500
Compressive Strength	D695	73	psi	7,900	16,400
Flexural Yield Strength	D790	73 160 -40	psi	9,600 5,500 14,800	13,500 10,000 15,000
Tensile Modulus	D639	73	psi	300,000	355,000
Flexural Modulus	D790	73 160 -40	psi	320,000 230,000 370,000	360,000 260,000 380,000
Elongation	D638	73	%	5-60	20-30
Specific Gravity	D792	73	--	1.04	1.06
Water Absorption (24 hr.)	D570	73	%	.40	.07
Thermal Conductivity	C-177		BTU/1 hr./ ft²/°F/in.		1.5
Linear Coefficient of Thermal Expansion	D696-44	--	in/in/°F	5.4×10^{-5}	3.3×10^{-5}
Heat Deflection Temperature 66 psi 264 psi	D648	--	°F at 10 mils deflection	 206 191	 279 265
Cost, Molding Pellets	--	--	$/lb.	.39	.70

Sources: General Electric Company Product Data Sheet CDX-38B (5-68-20M) and Marbon Data Sheet 157-M-1569.

FIGURE 1

WET AND DRY THICKNESS OF FIBROUS SAUSAGE CASING AT VARYING PRESSURES



Note: Average one ply dry thickness was 3.0 mils measured on a hand micrometer with pressure applied to the point where the micrometer clutch slipped.

FIGURE 2

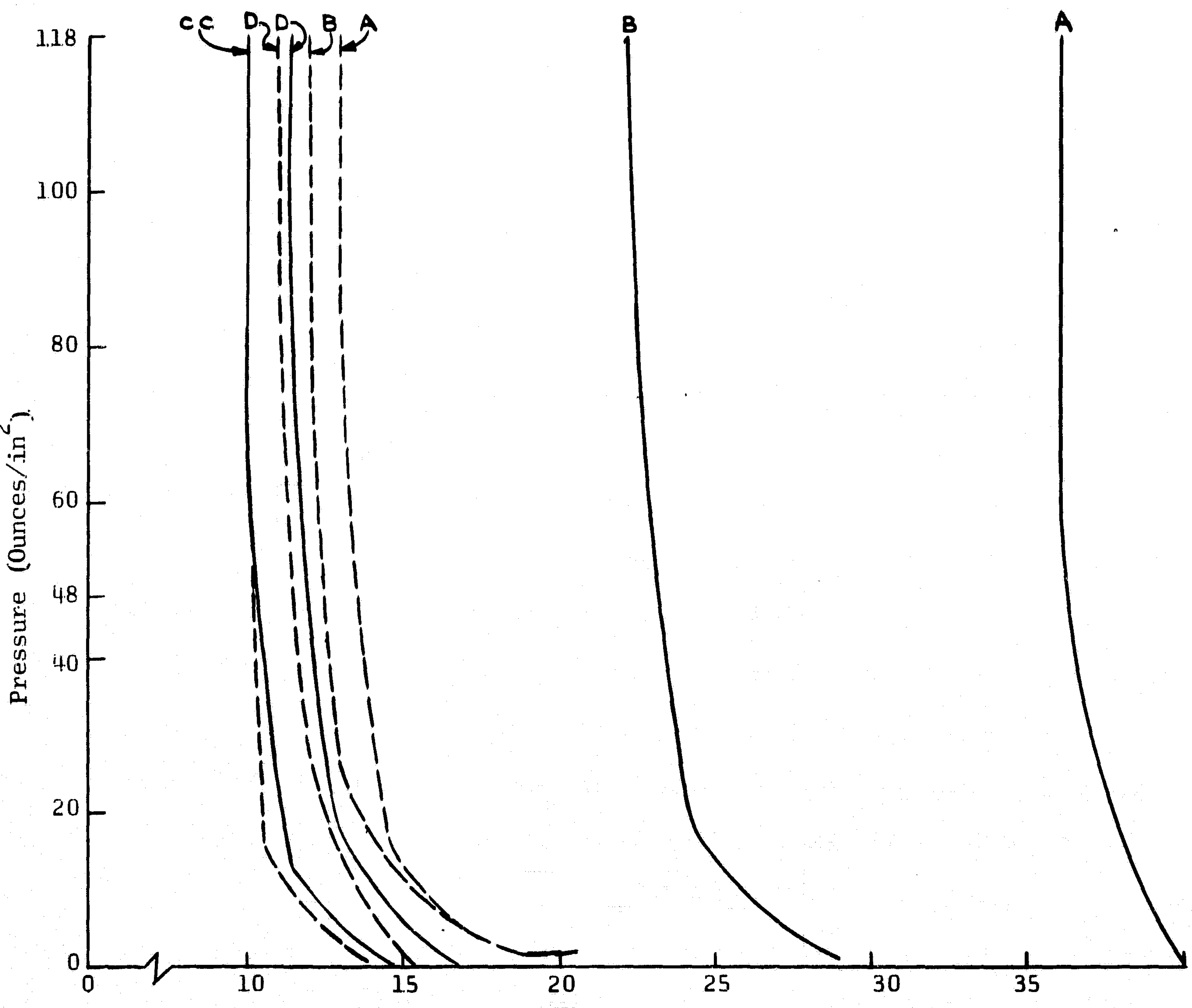
EFFECT OF PRESSURE CHANGE ON SEPARATOR SYSTEM THICKNESS

Separator Systems:

A. 4-FSC, 1 EM-470
B. 4-RAI 2291, 2-FSC, 1 EM-470
C. 6-RAI 2291, 1 EM-476I*
D. 7-RAI 2291, 1 EM-476I*

(* Irradiated)

------Dry ———— Wet 120 hrs. 45% KOH



Thickness (Mils) Measured
(Randall-Stickney Gage)