FORD/ERDA SODIUM-SULFUR BATTERY DEVELOPMENT

PHASE II

Progress Report No. 22

For Period: July 1 - July 31, 1977

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TASK 1 - ELECTRIC VEHICLE DEVELOPMENT AND DEMONSTRATION

A. Research Staff, Ford Motor Company

No work done on this task this period. EV cells built and tested are described in Task 5 of this report.

TASK 2 - DEVELOPMENT AND DEMONSTRATION OF A 1 MW LOAD LEVELING BATTERY

A. Research Staff, Ford Motor Company

Personnel, Dr.'s Minck and Mikkor, spent the month of July at Aeronutronic Division, supporting the test facility build (Na/S) and consequently were not in a position to support battery concept studies or testing at Ford, Dearborn.

Heat transfer studies are continuing.

- Radiation heat flow for cell cooling is being investigated.
- Various module arrangements are being studied for effective convection cooling for minimizing cooling duct volume.

TASK 3 - CONTAINER AND SEAL DEVELOPMENT

A. Research Staff, Ford Motor Company

The results of static corrosion testing of coupons, in duplicate, using high purity sodium polysulfides are as follows:

Material	Polysulfide	Time	°C	Weight Change mg/cm ²
Molybdenum (Foil, as received)	Na ₂ S ₄	8 weeks	400	+0.45
	Na ₂ S ₄	10 weeks	400	+0.50
Ti-30% Mo (sheet, as rec'd)	Na ₂ S ₄	6 weeks	400	-253
Ti-30% Mo (oxidized one hour at 450°C)	Na ₂ S ₄	2 weeks	400	-165
	Na ₂ S ₄	4 weeks	400	-228
Ti-0.2% Pd (oxidized one hour at 450°C)	Na ₂ S ₄	2 weeks	400	-48
	Na ₂ S ₄	4 weeks	400	-54

The corrosion test data shows that Ti -30% Mo and Ti -0.2% Pd, whether examined as received or oxidized one hour in air at 450°C, are extensively corroded and not suitable for container use. The remaining fourteen molybdenum coupons under test were removed in early August. Subsequent analysis is underway to determine the rate of molybdenum dissolution.

-Oxidized chromium-nickel plated E-Brite is being evaluated to determine how chromium thickness and oxidation conditions influence the corrosion resistance and conductivity. Initial four-point measurements show acceptable values of less than one ohm cm².

-Climax Molybdenum was contacted concerning preparation of explosive wire coating molybdenum on the inside of tubes.

B. Aeronutronic Division, FACC

Local chrome-plating vendors were evaluated for their ability to provide a uniform chrome-plating with a low crack density. After several attempts, a successful vendor was found. This vendor is now plating a lot of 25 sulfur containers which will be carefully evaluated.

The test of the second A-70 type seal was discontinued after 360 hours

at 370°C. The helium-leak rate had stabilized at 4×10^{-7} std. cm³/sec. Upon cooling to 25°C, the leak rate was less than 1×10^{-8} std. cm³/sec.

Sample coatings of Tiodize-CC400 were prepared on glass slides for thickness and resistance measurements. The average thickness of these samples was 0.015 mm (0.0006 in.). The resistivity averaged 0.25 Ω cm which results in a calculated resistance of $\sim 0.4 \mu\Omega$ cm² through the coating which is negligible compared to other components of cell resistance.

Samples of Chemfilmed 3003 aluminum were also coated with Tiodize CC-400 and were thermally cycled between room temperature and 400°C with no spalling of the coating.

Preliminary sheet resistivity measurements were made on Tiodize CC-400 on Chemfilmed 3003 aluminum. After heat treating the sample for five days at 425°C in a dynamic vacuum (1.5 mm Hg) the resistance did not change. However, a resistance increase of almost two orders of magnitude was observed after heating a sample in air for five days at 400°C, which indicates the desirability of baking out in vacuum or inert atmosphere.

Arrangements are being made for Chemetal to coat the inside surfaces of six sulfur containers with molybdenum using a CVD process. Delivery of the coated containers is expected in September.

Three A-70 type sulfur containers were fabricated from E-Brite tubing, Chromized and Tiodized. These containers were sent to Ford.

Six 1018 steel sulfur containers were fabricated. Four of these containers were chrome plated and two were Chromized. All have been Tiodized.

TASK 4. DEVELOPMENT OF CERAMIC ELECTROLYTE AND SEAL TECHNOLOGY

A. Department of Materials Science and Engineering - University of Utah

4.1 Pre-Pilot Electrolyte Development and Evaluation

4.1a Production of Standard Tubing. The pre-pilot batch plant is in operation and capable of producing β "-alumina tubing at the required levels. Standard tubing (1.5 cm and 3.35 cm OD and up to 25 cm long) is supplied unsealed, sealed to α -alumina tubular headers, or sealed at a rate consistent with the supply of machined α -alumina disk headers. This month ten large diameter (3.35 cm) tubes were sent to Aeronutronic for Na-S cell construction. These tubes were sealed to α -alumina disk headers using the new sealing station which was designed, constructed and placed into operation this month. Before additional sealed tubes are sent, we will await word from Aeronutronic concerning the construction and operation of Na-S cells with these sealed large diameter electrolytes. Approximately 100 large diameter tubes are in inventory awaiting sealing. This inventory cannot become much larger because of limited storage space.

Since the inception of the contract in February 1976, 250 standard tubes have been supplied (20 in the month of July). 50 of these tubes have been set aside and delivered to Ford for shipment to ERDA. The shipment schedule for tubing is outlined in Table 1.

4.1b Fabrication of Tubing with a Nominal 3 cm OD. To date approximately 170 tubes (3.2-3.4 cm OD) with wall thicknesses between 2.5 and 3.0 mm have been green formed by isostatic pressing at 40-55 kpsi and sintered horizontally to properties typical of the zeta process. The reader is referred to the May monthly report for a summary of typical dimensional characteristics and physical properties of the large diameter tubing. A low cost (\$2/lb) raw material (Reynolds ERCHPS) is presently used in the standard production of β "-alumina tubing.

TABLE 1

Pre-Pilot Production of Standard B"-Alumina Tubing

8.85% Na₂O, 0.75% Li₂O

<u>DATE SHIPPED</u>	<u>QUANTITY</u>	<u>NOMINAL DIMENSIONS (cm)</u>	<u>DESCRIPTION</u>
15 July 1976	11	1.5 x 20 x 0.1*	Unsealed
12 August 1976	5	"	"
27 August 1976	4	"	"
5 November 1976	10	"	"
5 November 1976	5**	"	Sealed to tubular headers***
18 November 1976	29	"	Unsealed
22 December 1976	10	"	Sealed to disk headers***
29 December 1976	5**	"	Sealed to tubular headers
13 January 1977	9	"	Sealed to disk headers
18 January 1977	5**	"	Sealed to tubular headers
19 January 1977	4	3.20 x 25 x 0.3	Unsealed
31 January 1977	12	1.5 x 20 x 0.1	Sealed to disk headers
3 February 1977	6	3.20 x 25 x 0.3	Unsealed
8 February 1977	5**	1.5 x 20 x 0.1	Sealed to tubular headers
15 February 1977	5	3.20 x 25 x 0.3	Unsealed
29 March 1977	5**	1.5 x 20 x 0.1	Sealed to tubular headers
4 April 1977	23****	1.5 x 20 x 0.1	Sealed to disk headers
11 April 1977	17	3.35 x 25 x 0.25	Unsealed
4 May 1977	5**	1.5 x 30 x 0.13	Sealed to tubular headers
6 May 1977	25	3.35 x 25 x 0.25	Unsealed
25 May 1977	10**	1.5 x 30 x 0.13	Unsealed
13 June 1977	11	1.5 x 20 x 0.1	Unsealed
20 June 1977	9	1.5 x 20 x 0.1	Unsealed

Table I
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<u>DATE SHIPPED</u>	<u>QUANTITY</u>	<u>NOMINAL DIMENSIONS (cm)</u>	<u>DESCRIPTION</u>
19 July 1977	10****	3.35 x 25 x 0.25	Sealed to disk headers
31 July 1977	<u>10**</u> 250	1.5 x 20 x 0.1	Unsealed

* (OD x length x wall)

** For eventual shipment to ERDA

*** α -alumina

**** Shipped to Aeronutronic Ford

4.1c Continuous Sintering and Sintering Without Platinum. Studies have been continued this month to determine the lifetime of pre-treated α -alumina tubes which are used to encapsulate β "-alumina electrolyte tubes during sintering. On a daily basis, several carriers containing β "-alumina tubes or powders are cycled back and forth from room temperature to the sintering temperature. Three carrier tubes have passed forty-seven heating and cooling cycles without any noticeable degradation (~442 hours above 1450°C).

Preliminary experiments have been completed in the use of ceramic V-setters to reduce the degree of ovality in large diameter (3.4 cm OD) tubing after horizontal sintering. Large electrolyte tubes have been batch sintered in pre-treated α -alumina encapsulation tubes by supporting them in V-setters. This type of support provides two-line contact along the length of the tube near its center. The results of these experiments showed that for sintered tubing (~2.4 mm wall) with sintered densities over 3.18 g/cm³, the degree of ovality can be reduced using this method. The max OD/min OD range for tubes batch sintered in this manner ranged from 1.000 to 1.015. (For current standard production tubing the range is 1.000 to 1.050). It is expected that this dimensional control using the V-setter technique will be the same or better in a continuous sintering operation.

4.1d Sealing. With the arrival last month of the seal specification, construction was completed on the furnace and the alignment rig for sealing alpha alumina plate headers to large diameter (3.4 cm) β "-alumina tubing. This first prototype seal station is now in operation. Ten sealed units have been sent to Aeronutronic. Five additional seal stations are currently under construction. By November 1 six stations will be in operation capable of sealing up to 18 electrolyte tubes per day.

The sealing of additional tubing is presently limited by the supply of B-2 seal glass with the correct composition. Since the glass (~10 lb) prepared at the Ford Glass Center was apparently off composition, small (4-1 lb) batches were prepared in early July at Utah. Samples were sent to Ford for analysis. Our present glass inventory will permit the sealing of 6-12 additional tubes.

4.1e Thermal Expansion Measurements. Measurements of the coefficient of thermal expansion were completed on polycrystalline β "- and α -alumina at temperatures between 300 and 800°C. The experimental values are

β "-alumina	$(8.22-8.35) \times 10^{-6}/^{\circ}\text{C}$
α -alumina	$(8.70-8.75) \times 10^{-6}/^{\circ}\text{C}$

The higher expansion coefficient for α -alumina relative to β "-alumina indicates that tensile and compressive stresses will arise in the α and β "-components after sealing. It may be significant that all seal failures noted to date occur in the α component.

4.1f Elastic Modulus Measurements. Elastic modulus measurements have been completed in polycrystalline β "-alumina ("zeta-processed") over a range of grain sizes (1-100 μm) and porosities (0.5-5%). These measurements indicate that

(1) the modulus is independent of grain up to 100 μm and thus microcracks do not develop during the processing of "zeta-process" β "-alumina providing the grain size is below about 100 μm .

(2) the modulus (E) is exponentially related to volume fraction porosity (P) by a relation of the form

$$E = E_0 \exp(-bP)$$

(3) A good value for the modulus of pore-free polycrystalline β "-alumina (zeta-process) is $30.4 \times 10^6 \text{ psi}$ ($2.10 \times 10^{11} \text{ N/m}^2$).

4.1g NDT Quality Control Development. An operating prototype involving the use of nickel band electrodes has been completed for resistivity measurements on 3.35 cm OD 25 cm long tubes. The design is similar to that described in previous reports for measurements on 1.5 cm OD by 20 cm long tubes. Measurements on twelve 3.35 cm OD tubes at 300°C and 30 MHz showed agreement with standard QC electrical measurements at 300°C within about 5%. This technique is subject to calibration for routine QC measurements in the pilot plant. Further work remains in the scaling up of this technique to pilot scale operation in the pilot plant.

B. Battery Ceramics Laboratory - University of Utah Research
Institute (UURI)

4.2 Pilot Plant Production of Electrolyte Tubing. Construction of the pilot plant in the Research Park began last month. The work is on schedule and should be complete, for the most part, by the end of August.

All pilot plant raw materials and equipment for the Phase II installation of the pilot plant process (through bisquing) are on order, except for two of the bisque furnaces (we wish to evaluate one of this type before ordering the last two) and the second spray dryer (we wish to evaluate larger spray drying runs using powder produced from the pilot plant process before ordering it).

TASK 5 - BATTERY (CELL) FABRICATION AND TESTING

A. Research Staff, Ford Motor Company

5.1 Test Facility Development. Operation of a ten-channel cycle controller has been corrected to eliminate occasional erroneous cycle reversals.

The second ten-channel unit is under construction.

Substantial but as yet undetermined damage to the computer control and data acquisition system occurred when the equipment was flooded with water as a result of workman's error while removing fire sprinklers in the Na-S Cell test room to lessen the sodium-water hazard. The computer equipment is being examined, but the time for its repair has not been determined. None of the cells under test at the time were destroyed by the accident, nor was any other test equipment damaged by the water.

5.2 Prototype Cell Development.

5.2a Prototype Cell Build. Two electric vehicle prototype cells were built: Al30-1 and Al30-2. These cells use 1.5 cm O.D. 8"-alumina, and 2.54 cm O.D. x 0.08 cm wall E-Brite, 26-1, sulfur containers. These cells have less capacity per unit area of 8"-alumina than the load leveling cells; namely, 0.37 Ah/cm² theoretical capacity. The two cells use WDF graphite felt electrodes with no void volume introduced into the felts. The standard compression seal with the α -Al₂O₃ header was used. Initial efforts at electroplating crack-free chromium onto the I.D. of the sulfur containers were unsuccessful. Therefore, Al30-1 and 2 were assembled with no Cr plate, but with Tiodize CC-400 (graphite-silicate) coated directly onto the E-Brite. Subsequently, a method

was found to successfully plate the containers, and the remaining six Al30 cells will have the standard plated/Tiodized containers.

Past efforts have indicated that cells partially filled with Al_2O_3 -coated graphite felt electrodes charge well into the two-phase region. However, the Al_2O_3 was not adherent and the high percent utilization could not be maintained. A new coating treatment has been devised, in which the Al_2O_3 loss in an adherence test drops from 100% to 2%. This treatment is being perfected and will be evaluated in future cells.

5.2b Prototype Cell Testing. The status of prototype cells under test is summarized in Table 5-1,2,3. One cell, Al30-1, was started and ten continued on test. All cells are operated at equal charge and discharge currents of 6 A (64 mA/cm^2), 350°C , and voltage limits of 2.5/1.5 volts, unless otherwise noted.

The performance of four Al20-series cells with chopped graphite fiber electrodes show highly variable initial results, three achieving partial two phase operation and all having cell resistances similar to other prototype cells. All have experienced some degradation in capacity and efficiency with life, but cell Al20-7 continues to operate with performance comparable to cells which employ the more expensive graphite felts.

Table 5.1

SUMMARY OF CELL TESTS

July 21, 1977

CELL CONSTRUCTION

No.	Treatment, Additives	CAPACITY, Ah		LIFE CHARACTERISTICS			Days	Cycles	% Decline (Gain) in		
		Theor.	Delivered Cycle #)	Rates, h Disc. Ch.	Efficiency Wh (out/in)				Capacity	Wh/kg	W/kg
40-2	Tiodize on AISI 446 Holes in Felt, No Treatment, No Additives	45.9	-	-	-	-	> 235	> 480	-	-	-
			36.6 (2)	6.1 6.1	82%				-	-	-
			33 (62)	5.5 5.5	82%				10	55	10
			32.1 (120)	5.4 5.4	82%				12	53	10
			26.1 (290)	4.3 4.3	82%				28	43	10
			31.2 (477)	5.2 5.3	86%				15	53	10
40-4	Tiodize on AISI 446 Holes in Felt, No Treatment, 1.7×10^4 ppm MoS ₂	45.9	-	-	-	-	> 226	> 605	-	-	-
			28.4 (2)	4.7 4.7	88%				-	46	10
			26.6 (50)	4.4 4.4	87%				6	43	10
			25.2 (130)	4.2 4.2	87%				11	41	10
			27.0 (340)	4.5 4.5	86%				5	44	10
			19.5 (504)	3.3 3.3	82%				31	31	10
40-5	Tiodize on AISI 446 Holes in Felt, No Treatment, 2.4 gms 446.	45.9	-	-	-	-	> 227	> 545	-	-	-
			30.2 (2)	5.0 5.0	88%				-	49	10
			28.1 (50)	4.7 4.7	87%				7	49	10
			26.7 (130)	4.5 4.5	84%				12	44	10
			26.4 (340)	4.4 4.4	84%				13	43	10
			26.5 (545)	4.4 4.4	83%				12	44	10

Table 5.2

SUMMARY OF CELL TESTS

July 21, 1977

CELL CONSTRUCTION

		CAPACITY, Ah				LIFE CHARACTERISTICS						
No.	Treatment, Additives	Theor.	Delivered (Cycle #)		Rates, h Disc. Ch.		Efficiency Wh (out/in)	Days	Cycles	% Decline (Gain) in Capacity	Wh/kg	W/kg
51-10	Cr Plate on E-Brite Oxidized, Holes in Felt, Untreated	101.1	-	-	-	-	-	>217	>405	-	-	-
			+30	(5)	5.0	5.0	88%			-	53	10
			+30	(24)	5.0	5.0	79%			-	47	9
			52.8	(95)	8.8	8.8	81%			-	100	11
			44.7	(190)	7.5	7.5	79%			-	84	11
			20.88	(405)	3.5	3.5	76%	-		37	11	
70-4	Inconel, Oxidized 1/2 Hr., Holes in Felt, Untreated 8 x 10 ppm of MoS ₂	202.2	-	-	-	-	-	>202	>228	-	-	-
			136	(2)	22.7	19.0	86%			-	199	9
			81.3	(9)	14.0	14.0	87%			40	114	8
			86.4	(84)	14.4	14.4	83%			36	120	8
			96.6	(167)	16.1	16.1	84%			29	130	8
			81.1	(228)*	9.7	13.6	80%	40	109	11		
70-4	E-Brite with Grafoil Lined, No Treatment, No Additives	81.0	-	-	-	-	-	>176	>422	-	-	-
			31.2	(2)	5.2	5.2	85%			-	56	11
			31.9	(94)	5.3	5.3	85%			(2)	57	11
			27.9	(208)	4.7	4.7	82%			11	49	11
			22.8	(422)	3.8	3.6	78%	27	39	10		

* $J_c = 64 \text{ mA/cm}^2$ $J_D = 89 \text{ mA/cm}^2$ † timed cycle, $J_c = J_D = 64 \text{ mA/cm}^2$

Table 5.3

SUMMARY OF CELL TESTS

July 21, 1977

CELL CONSTRUCTION

No.	Treatment, Additives	CAPACITY, AH				LIFE CHARACTERISTICS						
		Theor.	Delivered (Cycle #)		Rates, h Disc. Ch.		Efficiency Wh (out/in)	Days	Cycles	% Decline (Gain) in Capacity	Wh/kg	W/kg
120-4	E-Brite, chrome plate Tiodize, chopped graphite fibers 18.5 gms	101.1	-	-	-	-	-	>86	>92	-	-	-
			49.5	(2)	8.3	8.3	87%			-	91	11
			72.9	(17)	12.2	12.2	85%			(47)	136	11
			57.0	(56)	9.5	9.5	80%			(15)	104	11
			38.8	(92)	6.5	6.5	74%			(22)	68	11
120-5	Same as 120-4	101.1	-	-	-	-	-	>50	>60	-	-	-
			57.0	(2)	9.5	9.5	85%			-	106	11
			56.1	(21)	9.4	9.4	81%			2	100	11
			48.6	(60)	8.1	8.1	84%			15	88	11
120-6	Same as 120-4, except 37 gms chopped graphite fibers	101.1	-	-	-	-	-	>39	>29	-	-	-
			32.5	(2)*	3.9	5.5	87%			-	55	14
			29.8	(7)*	3.6	5.7	85%			8	49	14
			26.	(29)*	3.1	4.5	84%			20	43	14
120-7	Same as 120-6	101.1	-	-	-	-	-	>34	>26	-	-	-
			65.5	(2)*	7.9	11.6	85%			-	115	15
			62	(25)*	7.4	10.6	84%			5	108	15
130-1	E-Brite, Tiodize solid felt, no holes, 27 gms	34.7	-	-	-	-	-	>1	>3	-	-	-
			15	(2)**	3.2	3.3	85%			-	32	10

$$*J_c = \text{mA/cm}^2, J_{D_2} = 89 \text{ mA/cm}^2$$

$$**J_c = J_c = 50 \text{ mA/cm}^2$$

B. Aeronutronic Division, FACC

5.1 Test Facility Development

Thermal analysis of submodules indicates that thermal radiation can be a significant factor. For example, all of the heat generated in Mark-I cells adjacent to the walls (of a module box or oven) can be dissipated by radiation provided the walls are three Centigrade degrees less than the surface of the cells. The view factor -- the fraction of the radiating surface which is in direct view of another surface at a lower temperature -- is essentially unity for overall radiation from rows of cells (tubes) arranged in line, radiating to a parallel wall (plane). The view factors for direct radiation from cells in center rows, however, are much less than unity; consequently, heat dissipation solely by radiation would result in considerable nonuniformity of temperature between cells. Cells in the center of a submodule would be hotter than cells nearer the walls. A combination of forced convection and radiation probably is requisite in a practical cooling system.

Thermal analysis was conducted to size bus bars for transmitting power in and out of module boxes and ovens. Pure nickel probably is the most practical material for bus bars in experimental and prototype submodules, because it is extremely resistant to oxidation, retains its strength at elevated temperature and can be bonded easily to the nickel current collectors of individual cells. Power losses from nickel bus bars for 48-cell submodules probably will be about 200 watts.

Experience gained in this area is directly applicable to Task 2 concept studies.

5.2 Prototype Cell Development

During July, Aeronutronic built and tested its first Mark-I cells, cell A, B, and S-2. Due to the late deliveries of standard Mark I components, the cells utilized containers that had been previously prepared for use in early experimental work. The cells were cycled at constant currents of 13.7 to 27.4A (50 to 100 mA/cm²) at 350°C with voltage cut-off limits set at 1.5 and 2.5 volts.

Difficulty in start-up was encountered with all three cells, either due to gas trapped in the annulus between the safety tube and the electrolyte or due to delayed wetting of the β "-alumina by sodium. Cell A and B were inadvertently sealed with one atmosphere of air in the sulfur compartment. Cell B was overfilled with sulfur due to equipment malfunction, leaving inadequate space for the polysulfides within the sulfur container.

-Cell A

- Operated for 13 days and 20 cycles before failure.
- Restricted to single phase operation at current densities >50 mA/cm².
- High cell impedances (3-5 ohm/cm²) reflected in cycle efficiencies varying from 88% at 30 mA/cm² and 76% at 100 mA/cm².
- Cell performance data shown in Table 5.4.
- Cycle characteristics at various current densities is illustrated in Figure 5.1.
- Failure occurred at the seal area, β "-alumina header cracked and the reactants were forced out. Failure probably was aided by high pressures in the sulfur compartment at the end of the discharge cycle, due to the presence of air and reduced void volume. Containers were intact - x-rays indicated the β "-alumina tube also to be intact.

-Cell B

--Operated for four cycles before failure.

--Cycle capacity was 83 Ah (theoretical capacity of 156 Ah) with an energy efficiency of 85%.

--During the initial discharge cycle, difficulties were experienced with Cells A and B that resulted in high initial cell impedance. The problem appears to be associated with limited anode area either due to trapped gas in the annulus between the safety tube and the β "-alumina electrolyte or due to delayed wetting of the ceramic surface by sodium. The cell impedance has decreased steadily with cell operation.

Minor design changes can remedy this problem.

--Failure occurred at the seal area - the α -alumina header cracked and the reactants were forced out. X-rays indicated the δ "-alumina to be intact. The container was also intact.

It is interesting to note that even though reactants escaped from the cells at the time of failure, no fire or damage to the test equipment resulted.

Testing of the third cell, S-2 was begun.

TABLE 5.4

Performance of Cell A at 350°C, cycling under equal charge and discharge currents with voltage cut-off limits set at $V_H = 2.5$ volts and $V_L = 1.5$ volts. Theoretical capacity of cell is 143 Ah.

Current Density mA/cm ²	30	50	75	100
Cycle Capacity Ah	94	86	64	55
Rate Hours	11.9	6.5	3.2	2.1
Efficiency %	88	81	79	76

FIGURE 5.1