FORD/ERDA SODIUM-SULFUR BATTERY DEVELOPMENT

PHASE II

Progress Report No. 30

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TASK I - ELECTRICAL VEHICLE BATTERY DEVELOPMENT

1.1 Battery Design

A new computer code for cell design was completed which determines physical dimensions of the cell and ceramic, cell weight and number of cells in parallel for given power output, battery energy requirement and other inputted parameters.

The design code considers the effect of depth of discharge, reactant utilization, cell failure in a parallel string, non-uniform cell operation, cell aging, performance as a function of state of charge, losses due to cell connectivity and bus bars, battery voltage level and the required energy and power reserves.

The compact sodium core cell design stores all sodium within the 8"-alumina tube, has both electrical leads at the top of the cell, and incorporates a flangeless seal that allows cells to be densely packed. Cells in parallel can be in intimate contact, with series groups being separated by a minimum amount of electrical insulation.

Studies are being conducted for EV application to determine the most attractive module arrangement from the standpoint of weight, cost and packageability. It was assumed the battery would have a terminal voltage of 96 v, switchable to 48 v for starting, and that 70% utilization of reactants could be achieved.

Based on vehicle assumption of 1134 Kg (2500 lb), manual transmission, regeneration, rolling coefficient of 0.012 aerodynamic coefficient of 0.22123 and a 40 hp DC commutated electric motor, the preliminary cell and battery designs to meet a duty cycle of 100 mile CVS at 80% depth of discharge are as follows:

Cell Design

8" tube	dia. x length (cm)	2.56 x 36
Overall cell	dia. x length (cm)	3.55 x 37
Cell weight	(g)	630

Battery Design

Cells in parallel group	8
Groups in series per module	24
Modules in series or parallel as required	2
Cell total	384
Battery weight (Kg)	322
Battery housing L x W x H (cm)	97 x 64 x 57
Battery energy rating (Kwh)	41.3
Battery power rating (Kw)	35.7

The low power rating (steady state) was selected on the basis that increased power capability is available from NaS cells under pulse load conditions, as represented by the peak demand in the CVS driving cycle.

The mechanical support structures and thermal systems are now being studied.

TASK 2 - LOAD LEVELING BATTERY DEVELOPMENT

2.1 System Design

During this period, effort has been devoted to Manufacturing Feasibility Studies and Economic Analysis (Tasks 2.1.4 and 2.1.5). Studies have been undertaken to determine the manufacturing feasibility, to identify the requisite production equipment, and to estimate labor and material costs for a production rate of 10 million cells/year. Initial studies are based on the (slightly modified) Mark I cell design, but the analyses are formulated to accommodate future changes in cell design. There are no significant results to report at this time.

2.2 Component Development

Difficulties encountered during assembly of Mark I cells has lead to the analysis of dimensional tolerance stackup, evaluation of component and subassembly specifications, and consideration of stress levels in the alpha-beta alumina assembly. Appropriate drawings, jigs and specifications are being developed to eliminate interference conditions. Features requiring critical inspection have been identified.

During this initial phase of cell construction, many assemblies were broken near the alpha-beta alumina seal. Two major factors contributing to this scrapage have been: non-perpendicularity of alpha-beta alumina ceramic assemblies; and distortion of the alpha alumina washer (COORS slip cast AD 998 crucible cover) during flange sealing operations. Remedies are underway and include:

- 1) thicker washer (improved stiffness)
- 2) lowered bolting torques (~ 20 inch pounds) allowed by better polishing of washer surfaces.
- 3) tightened specifications for glass sealing operation.
- 4) "warm" cell filling to reduce thermal shock..

TASK 3 - CONTAINER AND SEAL DEVELOPMENT

3.1 Sulfur Container Material Development

A. Research Staff, Ford Motor Company

Oxidized Inconel 671, a commercial 48% chromium super alloy, has good corrosion resistance under static tests to the polysulfide melt. The effect of oxidation conditions on the nature and corrosion resistance of the oxide film is being determined. It was found that the air oxidation of Inconel 671 at 800, 900 and 1000°C for periods up to at least four hours follows parabolic oxidation kinetics. The rate constants are, respectively,

$$0.19, 2.1 \text{ and } 8.9 \times \frac{10^{-12} \text{ g}^2}{\text{cm}^4 \text{ sec}}$$

Previous data showed that although increasing the oxidation time (film thickness) at 800°C from 1/2 to 1 hour substantially reduced the corrosion rate, the resistivity increased from an acceptable 0.15 ohm cm²; a value somewhat high. Oxidation for 1/2 hour at 1000°C did not significantly change the corrosion resistance compared to the lower temperature condition, but the resistivity was unacceptably high. Exploratory test cells are being constructed using Inconel 671 as the sulfur container (Mark I otherwise). The initial three identical containers will be oxidized in air at 800°C for 1/2 hour.

To optimize the chromium-nickel alloy composition, high purity alloys of nominally 30, 40 and 50% chromium were vacuum melted, cast and hot rolled (1200°C) to strips 0.95 mm thick. To homogenize the samples and obtain a controlled phase distribution in each alloy, samples were annealed at 1200°C for 3-4 hours in helium and then quenched in ice water. Oxidation behavior and electrical resistivity measurements are being made on polished samples prior to selecting samples for corrosion testing. The 50% chromium alloy has a Kp of $0.41 \times \frac{10^{-12} \text{ g}^2}{\text{cm}^4 \text{ sec}}$ at 800°C in air, a value twice that obtained for Inconel 671.

B. Aeronutronic Division, FACC

Four aluminum-lined containers have been fabricated and exploratory cells utilizing these containers will go on test soon.

Work has begun on exploratory cells utilizing residual components from Phase II wherever possible. Paperwork for purchase of Phase III materials has been prepared and will be released as soon as Phase III funds become available.

C. University of Utah - Rutile Development Program

The rutile development program has progressed toward our goals of producing S-core current collectors where large metal seals pose the least problems. Since Nb-doped rutile is potentially the most economical material, we are moving toward this chemical system rather than the Ta-doped material. Because extensive studies are required, it is expected that the delivery of S-core current collections will precede by about 2-3 months the delivery of containers for Na-core cells. A summary of monthly accomplishments follows:

1. Expendable supplies for the production of rutile containers and current collectors have been ordered and some have been received.
2. Powders for the initial production of containers and current collectors have been formulated and tubular forms of Nb- and Ta-doped rutile are being pressed and sintered.
3. Niobium-doped samples of rutile have been annealed and shipped to Ford-Dearborn for static corrosion studies in molten polysulfide.
4. Microstructures have been obtained on several Nb-doped compositions. The specimens possess a rather uniform microstructure throughout. Presumably this slight improvement over Ta-doped material is due to the more rapid homogenization of Nb in TiO_2 .
5. An apparatus for measuring the electrical conductivity of rutile tube sections between 25° and 350°C has been fabricated. Measurements have been conducted on specimens fabricated this past summer.
6. A gas system for annealing conductive rutile has been designed and is ready for installation.
7. A sealing furnace for use in metal to rutile seal studies is being fabricated.
8. A "master curve" of resistivity as a function of Nb concentration in correctly prepared annealed samples is being prepared.

A full-time ceramic engineer (C. Venezilos, hired in November, 1977) is responsible for conducting the rutile development program. He reports to Dr. G. R. Miller.

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 is producing β "-alumina tubing (33.5mm O.D.) at a level which is consistent with the tube requirements at FACC. This month fifty-six large diameter (33.5mm) tubes were sent to FACC for Na-S cell construction. Twenty (20) assemblies were forwarded to Ford-Dearborn. Approximately 75 large diameter tubes are currently in inventory awaiting sealing. Pressing and sintering of β "-alumina has been kept at a minimum during the past month. This is a result of the combined Ford, FACC, Utah design review held at Newport Beach on 12/13/77. It was decided during the review meeting that production of standard tubing should be kept at low levels until new specifications were established for sintered tubing and sealed electrolyte assemblies.

Since the inception of the contract in February 1976, 461 standard tubes have been supplied (76 in the month of December). Sixty of these tubes have been set aside and delivered to Ford for shipment to ERDA. The shipment schedule for tubing is outlined in Table 4.1.

To date, approximately 370 tubes with wall thicknesses between 2.5 and 3.0 mm have been green-formed by isostatic pressing at 30-55 kpsi and sintered horizontally to properties typical of the zeta process.

TABLE 4.1

Pre-Pilot Production of Standard 8"-Alumina Tubing
8.85% Na₂O, 0.75% Li₂O

DATE SHIPPED	QUANTITY	NOMINAL DIMENSIONS (cm)	DESCRIPTION
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 0.2	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 20 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	"
13 June 1977	11	1.5 x 20 x 0.1	"
20 June 1977	9	1.5 x 20 x 0.1	"
12 July 1977	10****	3.35 x 25 x 0.25	Sealed to disk headers
1 August 1977	10**	1.5 x 20 x 0.1	Unsealed
10 August 1977	3****	3.35 x 25 x 0.25	Sealed to disk headers
12 August 1977	4*****	"	Unsealed
23 August 1977	3****	"	Sealed to disk headers
25 August 1977	3****	"	" " " "
8 September 1977	3****	"	" " " "
9 September 1977	10**	1.5 x 20 x 0.1	Unsealed
12 September 1977	6****	3.35 x 25 x 0.25	Sealed to disk headers
19 September 1977	8****	"	" " " "
22 September 1977	7****	"	" " " "
4 October 1977	7	"	" " " "
11 October 1977	6	"	" " " "
18 October 1977	20	"	" " " "
27 October 1977	28	"	" " " "
8 November 1977	27	"	" " " "
1 December 1977	20*****	"	" " " "
6 December 1977	29	"	" " " "
28 December 1977	27	"	" " " "
461			

* (OD x length x wall)

** For eventual shipment to ERDA

*** α -alumina

**** Shipped to Aeronutronic Ford (after 10-1-77 all tube shipments are sent to Ford-Aeronutronic unless indicated otherwise).

***** Send to Ford

4.1.b. Process Improvements. The two α -alumina V-setters received from Coors Porcelain Company were put to use this month. It was found that the ovality of sintered tubing can be drastically reduced by employment of these carriers during sintering. For ten tubes sintered in this manner the maximum O.D. to minimum O.D. ratio was: 1.011 ± 0.006 . It is expected that this result can be easily reproduced in large scale production. Consistent with budgetary limitations during this period of interim funding, we will gradually convert to sintering in which V-setters are used routinely.

During the past month, further studies were conducted on the S-type powders in an attempt to understand the necessity for dual sintering. X-ray diffraction studies on calcined powders revealed that the S-type soda and zeta components showed slightly different diffraction patterns than those derived from typical Meller alumina powders. The differences were due to the presence of residual α -alumina in the calcined zeta components and slight differences in relative amounts of the β , β'' and NaAlO_2 phases in the soda component. The S-type powder was then calcined at slightly different conditions so that its X-ray diffraction pattern was similar to that of Meller derived powders. Sintering studies were then performed to determine if these changes resulted in a lowering of the resistivity to acceptable values. It was found that the resistivities still remained high for single stage sintering cycles. Dual sintering resulted in a lowering of the resistivity to acceptable values. It can be concluded then that the intrinsic high resistivity associated with the single stage sintering of S-type powders is not solely due to the phases present (or relative amounts) after calcining. Further studies are planned in this area.

4.1.c. Sealing. Sealing is proceeding at a rate consistent with our ability to process sealed units which are acceptable at FACC. New specifications related to clearances for the sodium protection tube were discussed extensively at the review meeting held in Newport Beach. A "go-no-go" quality control device for determining minimum clearances for protection tubes inside tubular β -alumina electrolytes was fabricated at FACC and transferred to Utah. This new quality control device was implemented immediately upon receiving it at Utah. It was found that only 35% of the then sealed units would pass this test. As a result, 65% of all the tubing had to be re-sealed or reclaimed. The reasons for failure of the tubes to pass this test are several. They include the following (in order of importance): (1) excessive glass on the inside diameter of the disk, (2) slight off-centered sealing of the tube to the α -alumina disk, (3) warpage of the tube and (4) tube length being too short to clear the protection tube quality control device.

An extensive effort has been put forth to reclaim tubes which have initially failed the protection tube test. However, it has been found that some tubes require four to five re-sealing attempts before they can successfully pass the "go-no-go" test. It is certain that some of these sealing problems can be eliminated by slight modifications in the sealing alignment jigs and by using V-setter sintered tubing. However, these modifications will require time for design and implementation.

To summarize and emphasize, we presently find that to produce 100 acceptable sealed units, approximately 200-250 seal attempts alone must be made.

4.1.d. Pre-Pilot Plant Efficiencies. During the past month, accurate

statistics were kept to determine the rejection rates for powder production, pressed, cut, and bisqued tubing, sintered tubing, and sealed units. The following statistics were obtained:

1. Powder production: 93% by weight of all powder used as raw materials ends up as powder ready for pressing.
2. Pressing, cutting and bisquing: 8.2% of all green, isostatically pressed tubes are rejected prior to sintering. The greatest majority of these rejections is due to breakage during pressing.
3. Sintered tubing: 12.3% of sintered tubing is rejected due to either low densities, high resistivities, optical inhomogeneities, or failure to be leak tight.
4. Sealing: 13.1% of sealed tubing is rejected due to either (1) undetermined leakage near the seal or (2) inability of the sealed unit to pass the "protection tube clearance test".

In summary then, about 34% of all pressed tubing is rejected at various stages of the pre-pilot plant process.

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

4.2 Pilot Plant Electrolyte Development

This month has been devoted to installation and construction of several

facilities which include (1) calcining furnace, (2) ball mill, (3) screening equipment, and (4) pass-through sintering furnace. Specifically work was accomplished as follows:

1. The stairs and rails for the deep pit were completed.
2. Calcining furnace - formal drawings were completed.
 - a. The overhead plenum was modified.
 - b. The frame modification was completed.
 - c. Rail laying has started.
 - d. Casters are on all cars.
 - e. The furnace is ready to brick.
 - f. Parts for the drive mechanism have been ordered.
3. Ball Mill: the unit is installed and operational except for the timer. Planning of the access platform has begun.
4. Screening Room: a plan for the floor layout and equipment framework has been completed.
5. Pass-Through Sintering Furnace: the furnace was cut into three sections and moved to the pilot plant.

In anticipation of the Phase III effort, the pilot plant program, which will be transferred to Ceramtec, is now fully staffed. Recent staff additions include (1) Quality Control Manager, T. D. Hadnagy, (2) Quality Control Engineer, J. R. Rasmussen, (3) Supervisor of Plant Engineering, P. Smith, (4) Laboratory Technician, S. Young, and (5) Laboratory Assistant, B. Stevenson (part-time). With the exception of the Quality Control Manager who will report for work on February 1, 1978, all of the foregoing personnel are now on the job.

TASK 5. BATTERY (CELL) FABRICATION & TESTING

5.1 Production Facilities

A. Research Staff, Ford Motor Company

The work areas of rooms 2081, 2079, 2075 and 2071 have been rearranged for cell dissection and electrode treatment, buildup and drafting, testing, and ceramic preparation and seal development, respectively. All offices and work areas were painted and cleaned.

Design and construction of sodium and sulfur filling stations for high rate cell filling are underway.

5.2 Prototype Cell Build

A. Research Staff, Ford Motor Company

One sulfur core cell and two Mark I cells were built and delivered to test. The former featured a molybdenum wire braid/graphite felt disc electrode. Both Mark I cells were sulfur filled by casting directly into partially assembled cells and backfilling with oxygen prior to welding the end cap in place. This filling procedure allows the elimination of the fill tube which in the past was an area of very serious corrosion. This filling procedure will be used until hardware arrives that will allow further advancements.

One of the Mark I cells employed the standard felt disc electrode and will be one of three cells used to establish a data base to evaluate at least five other electrode configurations. The other Mark I built incorporated untreated chopped fibers as the electrode.

B. Aeronutronic Division, F.A.C.C.

During the months of November and December, a total of 24 cell assemblies were successfully completed and delivered to test, 36 cell assemblies were initiated and 13 failures were encountered. The cumulative production totals at various stages of assembly are given in Table 5.1

Receiving inspection of the electrolyte assemblies indicates the internal configuration of several of the tubes does not comply with the drawing requirements. In some instances, the combination of electrolyte tube bow, end taper and variation of end wall thickness results in the safety tube resting against the side of the electrolyte and leaves the possibility of the safety tube contacting the bottom of the electrolyte due to differences in thermal expansion when the cell is heated. A "go-no go" inspection tool is being designed and will be used in the receiving inspection of electrolyte assemblies. An inspection tool will also be provided for Utah.

Efforts are being made to reduce thermally and mechanically induced stresses in the region of the glass seal. Relocation of the metering holes to a point near the bottom of the safety tube is being evaluated as a means of

TABLE 5.1

CUMULATIVE CELL STATUS REPORT

I. Manufacturing	Phase II November 30, 1977			Phase III December 23, 1977		
A. Electrolyte Assemblies						
1. Received		136			29	
2. Carryover (Phase II)		-			29	
3. Bonded Storage (not inspected)		29			32	
4. Rejected		14			6	
5. Accepted		93			20	
	In Progress	Failed	Completed	In Progress	Failed	Completed
B. Cell Fabrication & Filling						
1. Assembly	4	5	84	0	1	23
2. Felt insertion & End Cap Weld	4	9	71	4	0	23
3. Bake Out	1	8	62	3	0	21
4. Sulfur Fill	5	5	52	9	4	13
5. Sodium Fill	0	2	50	9	4	0
II. Testing	In Progress	Failed	Completed	In Progress	Failed	Completed
A. Initial Heatup	0	9	40	0	0	0
B. Conditioning						
1. Initial "Trickle Discharge" (1-15mA/cm ²)	0	5	35	0	0	0
2. Deep Discharge at ~ 20mA/cm ²	2	6	27	0	0	0
C. Prolonged Automatic Controlled Tests						
1. Phase II Cells	14	13	-	5	9	-
2. Phase III Cells	-	-	-	0	0	0

reducing thermal stresses at the time of sodium filling. Decreasing the safety tube length and diameter is also being evaluated as a means for reducing the possibility of stresses on heatup related to the present close fit of the safety tube and the electrolyte. Cells are currently being assembled to evaluate the influence of these variables on the failure ratio observed during sodium filling, cell heatup, and testing.

Post-test examination of several cells after limited operation indicated that the sulfur was not wetting the graphite felt conductor disks in the upper third of the sulfur container. In an attempt to improve this condition, a procedure was developed to invert the cell after filling to allow the molten sulfur to flow thru all the graphite felt. The inversion resulted in an apparent increase in glass seal failures. Therefore, a decision was made to reinvert the cell and allow the sulfur to flow back thru the felt and freeze away from the glass seal. Neither the inversion or reinversion treatment indicated an improvement in graphite felt wetting. This experiment involved 20 cells.

5.3 Cell Testing

A. Research Staff, Ford Motor Company

Tests of two types were performed: EV cells subjected to pulsed loads and regeneration; and endurance of prototype cells.

The power and energy delivered during high current pulses are found to decrease sharply as the cell approaches the fully discharged state (melt composition of Na_2S_3). Peak power densities in excess of one watt per square centimeter of the ceramic electrolyte surface have been achieved for periods as long as 15 seconds, accompanied by a cell temperature increase of less than 10°C . The pulse power generally exceeds the peak value extrapolated from steady state data and can be delivered without creating excessive temperature rise or possible thermal run-away condition.

The ability of cells to accept charge during short duration high power pulses (regeneration) depends primarily on the recent operation history of the cell. Cells which had been recently discharged accept pulse charging very efficiently. The superior regeneration characteristics are believed due to the presence of discharge products near the ceramic electrolyte graphite felt electrode interface.

Two Mark I prototype LL cells (cells A140-1 and -2) were built and put on test. Results are included in Table 5.2 along with data from five cells under test at Aeronutronic Division FACC. A total of six standard Mark I cells are on test to establish baseline information. The charge and discharge current densities, 70/100 and 100/140 mA/cm^2 , represent 2 of 3 standardized current levels which satisfy the 7/5 hour ratio for charge/discharge times for the LL cycle and are to be generally employed throughout the testing program.

The energy delivered during discharge, energy density and cycle efficiency are data obtained during the stated cycle. The rechargeability of cells into the two phase region ($\text{S} + \text{Na}_2\text{S}_5$) is shown as a percentage of capacity for complete conversion of Na_2S_5 to sulfur. With the exception of cell A140-1 operating at lower currents, the Mark I cells fail to provide the needed two phase capacity.

TABLE 5.2
STATUS OF CELLS ON TEST

Cell No.	Cell Description	Design Deviation	Test Condition		Wh	Wh/kg	Efficiency $\frac{Wh_{out}}{Wh_{in}}$	Percent of Two-Phase Operation					Discharge Time (h)	Cycles	% Loss or (Gain) in Capacity	Remarks
			Temp. °C	Current Density J _{CW} /J _{DYS}				0-10	11-25	26-50	51-75	76-100				
	MARK I CELL		350	65/115	125	80	.75		X				4	250	< 10	GOALS
	1001		350	80/115	400	100	.75				X		5	2000	< 20	
M-3	Baseline Mark I		350	70/100	103	57.4	.779	X					2.1	119	--	Ceramic ID CABI 4C & D-4
M-4	Mark I		350	70/100	116	70.0	.744		X				2.5	102	--	CABI 13NG-5
S-71			350	100/141	50	25.6	.753	X					0.8	112	--	CABI 7LP-1
S-87			350	100/141	55	28.7	.719	X					0.9	99	--	EBE III 65-2
S89			350	100/141	55	28.9	.713	X					0.9	94	--	EBE III 65-1
A140-1			350	47/66	164	96.4	.81				X		5.2	103	--	
A140-2	Sulfur Electrode Dev.	cut fiber electrode,	350	25/25	63	37	.84			X			5.0	10	69	Terminated - cap loss.
A120-4		" 1.5x20 cm ceramic	350	64/64	48	49	.76	X					4.9	336	41	
A120-5		" "	350	64/64	52	52	.72	X					5.2	278	45	
A120-8		" "	350	64/64	47	47	.80	X					4.5	> 225	61	
OTHER CELLS																
130-2	EV	70 Wh theoretical capacity	350	50/50	24	32	.92	X					2.9	41	26	Used for pulse testing
A80-6	Sulfurcor	Mb Mesh current collector	350	12.3/12.3	--	--	.72	X					6.3	82	--	Very high cell resistance

The loss (or gain) in capacity represents the deterioration in ampere-hour capacity during the life of the cell under fixed test conditions. Because the 7/5 LL test mode was recently instituted for Mark I baseline cells, there is no initial reference for determining subsequent loss of these present cells.

The sulfur core cell exhibits extremely high internal resistance, restricting current densities to below 20 mA/cm².

Summary data for four exploratory cells that failed during this period are presented in Table 5.3

B. Aeronutronic Division, F.A.C.C.

Table 5.1 contains statistics indicating the number of assembled cells which survived the initial phases of electrical testing. The conditioning period is introduced to promote uniform operation within the cell. Defective cells generally become apparent during the conditioning stage, although some cell failures occur during the early cycles of the prolonged cycle test.

By the end of November, nine cells had failed during heating to the operating temperature and eleven during the conditioning period. The twenty cells built in Phase II which endured ten or more cycles are listed in Table 5.4. Note that most cells operate slightly into the two phase region at low current densities, but at higher currents, cell operation is limited to the one-phase region. As a result of the limited range of operation, the Wh capacities of the cells appear relatively reproducible. However, the energy delivered by most cells has not attained the values expected for the Mark I prototype design.

As of December 29, five Mark I cells remain on test. Test conditions were modified to reflect the 7h/5h ratio of charge/discharge times associated with the load leveling application. Data for these cells is presented in Table 5.2

During this period the "current-interrupt routine" was used occasionally. All cells are discharged and charged under a constant-current load. The control micro-processor can be programmed to stop (interrupt) the current for a fixed interval (11.25 seconds), measure the cell voltage at the end of the interrupted period, and compare it with the operating cell voltage just before the interrupt occurred.

Figures 5.1 and 5.2 present "interrupt data" during a full charge/discharge cycle for two of four cells so tested.

The "true" open-circuit voltage (O.C.V.), indicated on each plot (as two intersecting dash lines), was established by idling the cell for a prolonged period after charging and again after discharging.

The analysis indicates that the effects of concentration polarization are not removed completely in 11-1/4 seconds. The effect is less at 350°C than at 300°C.

Table 5.3

SUMMARY OF FAILED CELLS

December 21, 1977

CELL CONSTRUCTION

No.	Treatment, Additives	CAPACITY, Ah		Current Density mA/cm ²	Rates, h.		Efficiency Wh (out/in)	Days	Cycles	% Decline (Gain) in Capacity	Wh/kg	W/kg
		Theor.	Delivered (Cycle #)		Disc.	Ch.						
120-4	E-Brite, chrome plate Tiodize, chopped graphite fibers 18.5 gms	101.1	--		--	--	--	211	336	--	--	--
			49.5 (2)	64	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
			49.8 (139)	"	8.3	8.3	75%			0	88	11
			30.6 (191)	"	5.1	5.1	78%			38	53	10
			33.6 (245)	"	5.6	5.6	78%			32	58	10
			31.5 (311)	"	5.3	5.3	77%			36	54	10
			29.1 (334)	"	4.9	4.9	76%			41	49	10
120-5	Same as 120-4	101.1	--		--	--	--	188	278	--	--	--
			57.0 (2)	64	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
			45.6 (106)	"	7.6	7.6	80%			20	82	11
			42.3 (149)	"	7.1	7.1	78%			26	74	11
			35.7 (204)	"	6.0	6.0	77%			37	61	10
			36.2 (262)	"	5.2	5.2	72%			45	52	10
130-2	E-Brite, Tiodize	34.7	--		--	--	--	116	92	--	--	--
			18.1 (2)	50	3.8	3.8	92%			--	42	11
			13.4 (41)		2.9	2.9	92%			26	32	11
A140-2	Mark I design except chopped graphite fiber electrode	149.7	106 (2)	25	16.1	16.1	91.3	9	10		120	7
			53.3 (5)		8.1	8.1	86.5				60.5	7
			33.0 (9)		5.0	5.0	83.6				37	7

TABLE 5.4

SUMMARY OF ELECTRICAL PERFORMANCE -- ALL PHASE-II CELLS LASTING MORE THAN TEN CYCLES

Status at 0000, 26 November 1977

No.	Type	Test Conditions		Performance		Time, hrs.		Efficiency Wh (out/in)	Days	Cycles	% Decline (Gain) in Capacity	Energy Density Wh/kg	Power Density W/kg
		Temp., °C	Current Density mA/cm ²	Ah Delivered (cycle no.)	% Theore- tical Capacity	Disch.	Chg.						
A	NK-I, prototype	350	100	55 (12)	38.5	2.1	2.1	0.76	13**	19**	nil	80	12.0
X-2	NK-I, steel containers	290	50	104 (22)	72.4	8.0	8.1	0.78	19**	22**	nil	98	12.1
N-3	NK-I, mach. containers	350	50	71.9 (48)	47.7	5.5	5.3	0.87	49	48	nil	75.1	13.3
N-4	NK-I, mach. containers	300	50	71.4 (41)	50.5	5.5	5.7	0.77	35	41	nil	75.6	13.7
N-8	NK-I, mach. containers	300	50	79 (12)	54.2	6.1	6.1	0.75	16**	13**	nil	75	12.3
N-9	NK-I, mach. containers	350	100	18.5 (23)	28.8	1.4	1.4	0.74	13	23	nil	19.7	14.0
N-10	NK-I, mach. containers	350	50	62.3 (27)	44.1	4.9	5.0	0.80	27	27	nil	66.6	14.0
N-18	NK-I, mach. containers	350	50	60.6 (16)	42.7	4.7	6.8	0.79	27**	27**	nil	63.9	13.6
S-14	NK-I, sunk containers	300	50	60.9 (41)	41.0	4.7	4.8	0.83	35	41	nil	57.9	12.3
S-15	NK-I, sunk containers	350	50	94 (34)	63.3	7.2	10.8*	0.78	29**	33**	nil	85	11.8
S-16	NK-I, sunk containers	350	50	92 (25)	61.7	7.1	9.0*	0.77	28**	27**	nil	85	12.0
S-18	NK-I, sunk containers	350	50	83 (22)	61.4	6.4	6.4	0.76	27**	17**	nil	78	12.2
S-24	NK-I, sunk containers	350	50	65 (12)	48.7	5.0	5.6*	0.80	26**	15**	nil	61	12.2
S-25	NK-I, sunk containers	350	60	40.7 (22)	33.5	3.1	3.1	0.85	13	22	nil	41.4	13.4
S-26	NK-I, sunk containers	350	50	70.0 (27)	52.9	5.5	5.4	0.87	27	27	nil	69.8	12.7
S-31	NK-I, sunk containers	350	50	66.1 (42)	46.6	5.1	5.1	0.82	56**	55**	nil	63.2	12.6
S-69	NK-I, sunk containers	350	50	58.5 (27)	42.4	4.6	4.7	0.81	27	27	nil	56.3	12.2
S-71	NK-I, sunk containers	350	100	36.4 (29)	28.9	1.4	1.4	0.77	13	29	nil	32.5	23.2
S-86	NK-I, sunk containers	350	100	46.1 (15)	35.6	1.8	1.8	0.78	7	15	nil	38.6	21.4
S-87	NK-I, sunk containers	350	100	42.0 (27)	31.6	1.4	1.6	0.77	11	27	nil	34.6	21.6

* Charged at less than 50 mA/cm²

** Indicates cell which failed before 20 November 1977

FIGURE 1. CHANGE IN CELL VOLTAGE AFTER 11.25-SECOND CURRENT INTERRUPT

FIGURE 2. CHANGE IN CELL VOLTAGE AFTER 11.25-SECOND CURRENT INTERRUPT

5.4 Cell Dissection and Post Test Examinations

A. Research Staff, Ford Motor Company

Cells from the A40-series were dissected mainly to evaluate the condition and characteristics of the chrome/tiodize coating. These cells, the first to employ this particular coating system, had been on test for up to 319 days and had shown stable cycle capacity. It was observed that on some cells both the Tiodize and chrome layer locally had disappeared from the container surface. Observations are summarized in Table 5.5.

Cell 120-7 was partially dissected to evaluate the chopped fiber electrode distribution. Both the axial and radial distribution looked uniform. Some local separation of the fibers from the container wall was observed. The separation appeared to be caused by accumulation of corrosion products that are presently being analyzed.

B. Aeronutronic Division, P.A.C.C.

A cell-by-cell summary of reasons for termination of cells together with an analysis of the cause of failure is presented in Table 5.6. This table includes cells which failed during the period 1 September to 16 December 1977. It will be noticed from reading Table 5.5 that a large fraction of the cell failures have been attributed to cracks in the glass-seal area of the electrolyte-alumina disk assembly. Some of these failures (see S-7 in the Table) were described as "probably" caused by glass-seal failure. Uncertainty arises because the heat of reaction resulting from these failures causes a significant amount of cell damage which obscures the evidence of the reason for failure. There was little doubt, however, that glass-seal area failure was at fault (rather than, for example, failure of the electrolyte tubes away from the seal area). Observations indicate with reasonable certainty that damage to the upper half of the electrolyte tubes in many of these cases arises from the heat of reaction released when sodium that leaks through the glass seal finally reaches the sulfur which fills only the bottom 2/3 of the sulfur container. A common feature of all of the somewhat destructive heatup failures was that the damage had been restricted to the upper half of the sulfur container, in the area above the top of the sulfur level. In most of these cases the most severe damage to the electrolyte tube and bulging of the cell container occur right at the top of the sulfur level. The failure of the glass seal area itself was inferred from the fact that failures during cell assembly always occur at the glass seal and from the fact that a significant number of tested cells (X-3 and X-4, for example) were unambiguously found to have failed in the glass seal area.

5.5 Polysulfide Wetting of Felts

Graphite felts (WDF, Union Carbide) were coated with alumina solutions to improve their wettability by sodium polysulfide. Two different commercial aluminas at various concentration were used: namely, Catapal-SB (a white spray dried powder) and Alon D-30 (an acidic alumina solution).

TABLE 5.5 CELL DISSECTION SUMMARY

November & December 1977

<u>Cell Description</u>	<u>Operation Data</u>	<u>Post-test Observations</u>
A40-2	Days: 253 Cycles: 517 Cooled in Charged State Seal area broken (alpha) Nonfaradaic behavior	β" tube shattered. Protection tube pressed in. Na metal present in S container where felt was eaten away. Radial cracks in α disc. No Na in Na container.
A40-3 160 ppm FeS	Days: 174 Cycles: 224 Discharged while cooled down. Sulfur leak, probably loss of reactants. Decreasing capacity	Radial cracks in alpha disc. Appreciable Na level in Na container.
A40-4 1.7 x 10 ⁴ ppm Moly. Sulfide	Days: 231 Cycles: 611 Cooled in fully charged state in 2 phase region. Loss in cycle capacity.	α, β" tube glass seal broken. Appreciable Na level in Na container. Micro cracks on β" tube.
A40-5 2.9 gms of 446 S.S.	Days: 319 Cycles: 807 Cooled while in good operating condition and in fully charged state	Na container full of Na. α disc intact. Extensive loss of tioidize/chrome layer from sulphur container
Al20-7	Days: 108 Cycles: 100 Deeply discharged (due to a power failure)	Sulfur container wall split open. No contact observed existing between chopped graphite fiber and container wall due to presence of a layer of corrosion products and polysulfide. Thickness of the layer between 1 and 2 mm. See also note 6.

- Notes:
1. Sulfur containers for above A40 series cells had 0.13" E-brite wall plated with 1 to 2 mils of crack free chrome.
 2. Dissection strips from all A40 sulphur container walls showed definite signs of corrosion and blistering of coatings.
 3. Methanol extracts from A40-2, A40-3 and A40-4 cells sulfur containers were analyzed and were found to contain Fe, Cr, Si and Mo which supports above mentioned observation.
 4. Corrosion products accumulated on container walls are being analyzed.
 5. An unused sulfur container wall (chromeplated & tioidized) was examined under SEM. The tioidize layer seemed to contain crevices or cracks on the surface. Any definite conclusion regarding the nature of these faults on the tioidized surface could not be reached.
 6. Electron conductor in sulfur container was chopped graphite fiber. The distribution of graphite fibers did not seem to vary along the axis of the container. Corrosion products are being analyzed.

TABLE 5.6 SUMMARY OF CELL FAILURES

Cell Description	Operation Data	Post-Test Observations
S/N S-7 MK-1 Sunk Container Electrolyte EBE III 7703	Termination Date: 9-9-77 Temp: <350°C Days: 0 Cycles: 0 Terminated during heatup	Overheated spots near container seal and longitudinal split in container. Upper 1/2 electrolyte tube fractured. Probable cause of failure: Sodium leakage thru cracks in glass-seal area and subsequent heat from sodium-sulfur reaction caused most of the damage to the electrolyte tube and container.
S/N S-13 MK-1 Sunk Container Electrolyte EBE III 60-61#4	Termination Date: 9-9-77 Temp: 350°C Days: 7 Cycles: 0 Terminated due to zero cell voltage	Electrolyte tube was cracked near glass seal area. Probable cause of failure: See S-7
S/N S-10 MK-1 Sunk Container Electrolyte EBE III 35#1	Termination Date: 9-10-77 Temp: 350°C Days: 1 Cycles: 0 Terminated during first 13A discharge.	Leakage at sulfur container seal. Electrolyte tube was cracked throughout. Possible cause of failure: Thermal shock during sodium filling.
S/N S-12 MK-1 Sunk Container Electrolyte CABI 4EF#2	Termination Date: 9-16-77 Temp: 350°C Days: 0 Cycles: 0 Terminated during first 13A discharge.	Severe cracking of alumina disk. Cause of failure: Undetermined.
S/N X-1 MK-1 Sunk Container 1018 steel sulfur container, chromium plated and Ti-dized Electrolyte CABI 13AB#8	Termination Date: 9-19-77 Temp: <350°C Days: 0 Cycles: 0 Terminated during heatup	Hole burnt in sulfur container near flange. Probable cause of failure: See S-7
S/N X-3 MK-1 Sunk Container 1018 steel sulfur container, chromium plated and Ti-dized Electrolyte CABI 4AB#1	Termination Date: 9-19-77 Temp: <350°C Days: 0 Cycles: 0 Terminated during heatup	Sodium leaked through cracks in glass seal into graphite felt. Cause of failure: Cell short circuit.
S/N X-4 MK-1 Sunk Container Electrolyte CABI 13AB#6 1018 Steel Sulfur Container, chromium plate and Ti-dized	Termination Date: 9-26-77 Temp: 350°C Days: 7 Cycles: 0 Terminated due to excessive voltage drop during "trickle" discharge.	Sulfur leak through cracks in glass seal. Reaction products clogged fill holes in safety tube. Cause of failure: Cracking of glass seal.
S/N S-4 MK-1 Sunk Container Electrolyte CABI 13CE#2	Termination Date: 9-27-77 Temp: 350°C Days: 8 Cycles: 5 Terminated due to high resistance.	"Dry" graphite felt was found even at the bottom of the cell. Cracked glass seal. Probable cause of failure: Graphite felt was largely unwetted by sulfur or poly-sulfides.
S/N S-8 MK-1 Sunk Container Electrolyte CABI 13CE#4	Termination Date: 9-28-77 Temp: <350°C Days: 0 Cycles: 0 Terminated during heatup	Severe fracturing of top half electrolyte tube. Probable cause of failure: See S-7.
S/N S-22 MK-1 Sunk Container Electrolyte CABI 4AB#2	Termination Date: 9-28-77 Temp: 350°C Days: 0 Cycles: 0 Terminated during "trickle" discharge.	Sulfur leak through cracks in glass seal produced reactants that clogged fill holes in safety tube. Cause of failure: See X-4.
S/N S-11 MK-1 Sunk Container Electrolyte CABI 13CE#1	Termination Date: 9-30-77 Temp: <350°C Days: 0 Cycles: 0 Terminated during heatup.	Sodium leak through cracks in glass seal. Cause of failure: Cell short circuit.

TABLE 5.6 SUMMARY OF CELL FAILURES (Continued)

<u>Cell Description</u>	<u>Operation Data</u>	<u>Post-Test Observations</u>
S/N S-19 MK-I Sunk Container Electrolyte CAB113AB#2	Termination Date: 9-30-77 Temp: <350°C Days: 0 Cycles: 0 Terminated during heatup	Electrolyte tube severely fractured. Worst damage 40mm below seal. Probable cause of failure: See S-7.
S/N S-15 MK-I Sunk Container Electrolyte EBE11178#3	Termination Date: 10-10-77 Temp: 350°C Days: 29 Cycles: 35 Terminated due to loss of cell voltage.	Alumina disk broken upwards. Sodium remaining in safety tube only. Cause of failure: Cell overdischarged due to equipment malfunction.
S/N X-2 MK-I Sunk Container Electrolyte CAB113F#1A	Termination Date: 10-10-77 Temp: 290°C Days: 19 Cycles: 22 Terminated due to loss of cell voltage.	Alumina disk broken. Bottom of electrolyte tube badly broken. Cause of failure: Cell overdischarged due to equipment malfunction.
S/N M-1 MK-I Machined Container Electrolyte CAB14AAB#7	Termination Date: 10-10-77 Temp: <350°C Days: 0 Cycles: 0 Terminated during heatup	Cracks in electrolyte tube at center and near glass seal. Probable cause of failure: See S-7.
S/N S-16 MK-I Sunk Container Electrolyte CAB113F#1	Termination Date: 10-17-77 Temp: 350°C Days: 28 Cycles: 27 Terminated due to loss of cell voltage.	Electrolyte tube had cracks at glass seal at midpoint and a "U" shaped fracture at bottom. Polysulfides were found in safety tube annulus. Possible cause of failure: Equipment malfunction.
S/N M-2 MK-I Machined Container Electrolyte CAB14EF#4	Termination Date: 10-27-77 Temp: 350°C Days: 9 Cycles: 5 Terminated due to loss of capacity.	Leakage at sulfur container current collector weld. Glass seal was cracked. Failed due to loss of reactants.
S/N S-24 MK-I Sunk Container Electrolyte CAB14EF#3	Termination Date: 10-31-77 Temp: 350°C Days: 26 Cycles: 15 Terminated due to nonfaradaic condition.	Alumina disk broken upwards. Electrolyte tube had circumferential cracks at midpoint. Cause of failure: Cell was overdischarged due to equipment malfunction.
S/N S-18 MK-I Sunk Container Electrolyte CAB14AB#6	Termination Date: 11-1-77 Temp: 350°C Days: 27 Cycles: 17 Terminated due to non-faradaic condition observed beginning with cycle 17.	Chemical pressure had "imploded" the electrolyte tube in two areas 3 cm long near the midpoint and had bent the safety tube in that area. Cracks also found in glass seal area. Possible cause of failure: Equipment malfunction.
S/N M-5 MK-I Machined Container Electrolyte CAB113AB#3	Termination Date: 11-4-77 Temp: 350°C Days: 1 Cycles: 0 Terminated due to lack of cell voltage after heatup.	Cracks were found in the electrolyte tube near the seal area. Cause of failure: See X-3.
S/N M-11 MK-I Machined Container Electrolyte CAB115ABC#4	Termination Date: 11-7-77 Temp: 350°C Days: 1 Cycles: 1 Terminated because of apparent high cell impedance.	Cracks in glass seal. Blue residue in sulfur container. Possible cause of failure: Electrical conduction through crack provided alternate discharge path for cell.

TABLE 5.6 SUMMARY OF CELL FAILURES (continued)

<u>Cell Description</u>	<u>Operation Data</u>	<u>Post-Test Observations</u>
S/N S-68 MK-1 Sunk Container Electrolyte CABI4CH#8	Termination Date: 11-9-77 Temp: <350°C Days: 0 Cycles: 0 Terminated during experimental heating cycle.	Heat of reaction between sodium and sulfur burned a hole in the sulfur container and safety tube just below the flange area. Probable cause of failure: See S-7.
S/N M-8 MK-1 Machined Container Electrolyte EBEIII49#3	Termination Date: 11-10-77 Temp: 300°C Days: 16 Cycles: 13 Terminated due to high internal resistance.	Broken glass seal (probably occurred during cooldown). Sulfur coating on the electrolyte tube indicated that the cell had been charged. Sulfur blockage of electrolyte was cause of high impedance. Possible cause of failure. Equipment malfunction may have caused overcharging
S/N M-13 MK-1 Machined Container Electrolyte CABI22ABD#7	Termination Date: 11-10-77 Temp: 350°C Days: 3 Cycles: 0 Terminated due to high cell resistance	Cracks in glass seal. Reaction products had clogged fill holes in safety tube limiting sodium capacity to the amount contained outside the safety tube. Cause of failure: See X-4.
S/N M-15 MK-1 Machined Container Electrolyte CABI7AB#4	Termination Date: 11-17-77 Temp: 350°C Days: 10 Cycles: 4 Terminated due to low charge/ discharge capacity.	Sulfur container swollen at midpoint. Alumina disk broken upwards. Electro- lyte broken in seal area and at midpoint. Probable cause of failure: See S-7.
S/N S-72 MK-1 Sunk Container Electrolyte EBEIII36#3	Termination Date: 11-17-77 Temp: <350°C Days: 2 Cycles: 0 Terminated during heating	Cracks in glass seal area. Large cracks at 2/3 height electrolyte tube. Probable cause of failure: Cracking in glass seal area
S/N S-27 MK-1 Sunk Container Electrolyte CABI13HC#1A	Termination Date: 11-21-77 Temp: 350°C Days: 4 Cycles: 0 Terminated during "trickle" discharge.	Cracks in glass seal. Probable cause of failure: Short circuit through crack in glass seal.
S/N S-85 MK-1 Sunk Container Electrolyte CABI7CAD#3	Termination Date: 11-21-77 Temp: 350°C Days: 4 Cycles: 0 Terminated during "trickle" discharge.	Large crack in glass seal. Probable cause of failure: Short circuit through crack in glass seal.
S/N S-83 MK-1 Sunk Container Electrolyte CABI4AB#8	Termination Date: 11-27-77 Temp: 350°C Days: 5 Cycles: 0 Terminated at the beginning of programmed cycling.	Cracks in glass seal. Polysulfide reaction products clogging safety tube fill holes and partially filling the top 7 cm of the annulus between the electrolyte and the safety tube. Cause of failure: Cracks in glass seal.
S/N S-99 MK-1 Sunk Container Electrolyte CABI15ABC#1	Termination Date: 11-23-77 Temp: 350°C Days: 2 Cycles: 0 Terminated during "trickle" discharge.	Failure mode and observations almost identical to those of S-83 above.

TABLE 5.6 SUMMARY OF CELL FAILURES (continued)

Cell Description	Operation Data	Post-Test Observations
S/N M-18 MK-1 Machined Container Electrolyte CAB113D#3	Termination Date: 11-26-77 Temp: 350°C Days: 25 Cycles: 10 Terminated due to loss of cell voltage.	Alumina disk fractured and broken upwards. Longitudinal crack in S container 2.5 cm long starting 1 cm below container seal. Complete utilization of all available sodium except for the amount below the fill holes in the safety tube. Probable cause of failure: Cell over-discharged by short circuit in glass seal area, producing excessive amounts of polysulfides for S container capacity.
S/N S-31 MK-1 Sunk Container Electrolyte CAB113D#6	Termination Date: 12-1-77 Temp: 350°C Days: 57 Cycles: 53 Terminated due to loss of cell voltage.	Cracks in glass seal area. Blue, high-sodium material in top 4 cm of sulfur container. Cause of failure: Cracks in glass seal area.
S/N S-90 MK-1 Sunk Container Electrolyte EBE1184#2	Termination Date: 12-5-77 Temp: 350°C Days: 15 Cycles: 20 Terminated due to non-faradaic current flow.	Cracks in glass seal area. Cracks in electrolyte tube at about half height. Blue, high-sodium material filling 2-3 cm length of sulfur container annulus near cracks in electrolyte. Cause of failure: Unknown.
S/N S-96 MK-1 Sunk Container Electrolyte LBL11138-99#1	Termination Date: 12-14-77 Temp: 350°C Days: 24 Cycles: 38 Terminated due to loss of cell voltage.	Cracks in alumina disk. Leak of polysulfides from container seal. Alumina disk burst upward. Probable cause of failure: See Cell M-18 above.
S/N S-69 MK-1 Sunk Container Electrolyte CAB11C13#2	Termination Date: 12-14-77 Temp: 350°C Days: 38 Cycles: 36 Terminated because of non-faradaic current.	Cracks in alumina disk and in glass seal. Probable cause of failure: Cracks in glass seal.
S/N M-9 MK-1 Machined Container Electrolyte EBE11165#8	Termination Date: 12-16-77 Temp: 350°C Days: 23 Cycles: 23 Terminated due to apparently high internal resistance.	Cracks in alumina disk. Slight upwards bowing of alumina disk and cracks in glass seal. Probable cause of failure: Internal shorting due to cracks in glass seal.
S/N S-26 MK-1 Sunk Container Electrolyte CAB14GH#5	Termination Date: 12-16-77 Temp: 350°C Days: 38 Cycles: 37 Terminated due to loss of cell voltage.	Cracks in glass seal area. Blue, high-sodium material and metallic sodium fill top 7 cm of sulfur container annulus. Cause of failure: Cracks in glass seal area.
S/N S-25 MK-1 Sunk Container Electrolyte CAB17GN#1	Termination Date: 12-16-77 Temp: 350°C Days: 32 Cycles: 33 Terminated because of high internal resistance.	Small cracks in glass seal. Probable cause of failure: Cracks in glass seal.

Results were evaluated by weight pickup and by height rise experiments. The coated felts were dipped into sodium tetrasulfide at 350°C for 8 to 24 hours under vacuum. Felts coated with 5% Catapal SB, 0.5% polyol and bonding agent solution, wicked to about 2 inches from the liquid surface, but the experiment was not reproducible. Good surface wicking was observed, but the inner cross section of the felt was untouched. Experiments will continue with variations in the concentration of the surfactants and bonding agent. Felts with 5% Alon D-30 solutions did not show any significant wicking.

Soaking techniques for above experiments were changed from normal dipping to ultrasonic soaking. This did not enhance the wicking results for either alumina.

An alternate treatment process - surface oxidation is being tried.

Graphite felt was heated in air at 450°C for four hours to oxidize the surface of fibers and cooled down in helium atmosphere. The wicking experiments conducted with the above felt did not show any significant wicking of sodium tetrasulfide.

To achieve more complete surface oxidation the felts were heated at 450°C with O₂ flow and cooled in helium atmosphere. Wicking experiment with felts so prepared are in progress.

5.6 Na-Na Glass Cell Testing

Until Cerametek, Inc. is in a position to evaluate process modifications on ceramic electrolyte durability and performance by Na-Na cell tests, Ford will continue to perform these evaluations.

The resistivity of three 8"-alumina tubes, sintered without platinum, have been measured. The initial resistivity of the tubes at 300°C were 7.0, 7.0 and 8.9 ohm-cm with activation energy of 4.56 Kcal/mole. Data is presented in Table 5.7.

After operation at current density of 1A/cm² (92 amps) at 330°C for short periods the resistivity of tube JCSEI-61-10 dropped abruptly to a stable value of 4.9 ohm-cm at 300°C (3.50 Kcal/mole) until a sudden short circuit developed after 194 Ah/cm². The lower resistance, and its temperature dependence, is consistent with a model that the ceramic resistance is shunted by a fixed size sodium filled crack. This model would indicate an early ceramic failure.

Preliminary post mortem examination indicates pitted, cracked and discolored ceramic. The first test, tube EBE-99-10, ended prematurely due to equipment failure.

TABLE 5.7

Summary of Na-Na Test Results

<u>Ceramic Tube Number</u>	<u>Resistivity at 300°C ohm-cm</u>	<u>Activation Energy k cal/gm-mole</u>	<u>Ah/cm²</u>
EBE - 99 - 10	7.0	4.56	67
JCSEI - 61 - 10	7.0	4.56	
	4.9	3.50	194
JCSEI - 61 - 8	8.9	4.56	617 (on test)