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QUARTERLY REPORT

HYDROGEN-OXYGEN ELECTROLYTIC REGENERATIVE FUEL CELLS

by

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1. INTRODUCTION

This report reviews the progress made on the development of a regenerative hydrogen-oxygen fuel cell under NASA Contract 3-2781 during the period of 1 April 1966 through 30 June 1966. The program objective is the development of an electrolytically regenerative, hydrogen-oxygen fuel cell that will be superior in performance to currently available rechargeable batteries. The device under development consists of a cell stack that is utilized as an electrolyzer during charge periods and as a fuel cell during discharge periods. Integral gas storage tanks are used to contain hydrogen and oxygen gas that is generated during charge. Such a device offers advantages in the area of watt-hours-per-pound, high ambient temperature operation, and comparable cycle life than that which can be obtained from existing secondary batteries. Phase I consisted of a design development and testing of a nominal 75-watt, 44-watt-hour, 6-cell unit to demonstrate the feasibility of a multicell regenerative device. This phase has been completed. Phase II (as modified) consists of the investigation of the asbestos matrix, electrode combinations to improve cycle life, and the design and development of a 500-watt, 600-watt-hour, 34-cell unit of minimum weight for evaluation as a flight prototype.

2. SUMMARY

During this period primary emphasis was placed on the testing of single cells with various electrode and matrix structures to improve cycle life capabilities and to obtain a better understanding of modes of deterioration of cell performance. Single cell tests of the concentration type and conventional cycling type indicated that asbestos matrices are a major factor in the performance degradations that we have observed thus far. Fabrication techniques were established for forming matrices using potassium titanate as a substitute for asbestos. These new matrices have enabled cycle life improvements above what had been previously obtained with asbestos matrices. Corrosion tests of potassium titanate with molten potassium hydroxide have shown a relatively low rate of reaction exists.

3. TECHNICAL DISCUSSION

3.1 Single Cell Tests

Thirty two single cell tests were conducted during this period to evaluate the performance of various electrode and matrix structures. Test results and construction variables of these cells are summarized in Table 1.

3.1.1 O_2 Concentration Tests

Cells No. 170 and 173 were started in the last report period and consisted of concentration cells of the type described in the 7th Quarterly Report. Cell 170 was a continuous oxygen concentration cell test employing fuel cell type asbestos and gold coated platinized electrodes. The cell was run continuously for a period of 456 hours, at which time the test was discontinued to to excessive overpotentials. During the test period there was a gradual rise in voltage with time as shown in Figure 1. Analysis of a sample of electrolyte squeezed from the mat of the cell revealed a final KOH concentration of 26.25 percent. The electrodes and asbestos mat were relatively clean, with the exception that the porous nickel substrate appeared oxidized, especially on the electrode that was utilized on the oxygen evolution side of the cell.

Cell No. 173 consisted of gold coated platinized electrode and a mat fabricated from three layers of 0.020 inch potassium titanate paper. The cell was put on test as a cycling concentration cell, consisting of 35 minutes period. Figure 2 shows the mid-point voltage of the cell at each direction of current flow as a function of cycles. Figure 3 shows the voltage performance of the cell at various cycles. The test was discontinued after 200 cycles due to high overpotential. As can be seen, there was a gradual rising of voltage with cycling to the end of the test. Note also the difference in voltage as the cell reversed polarity, indicating a difference in the electrode structure and performance of the electrodes within the cell. When the cell was disassembled, the mat appeared extremely dry. A sample of electrolyte could not be obtained for analysis. This test, at the point of conclusion, represented a maximum number of cycles or operating time achieved in a cycling concentration cell and therefore justified the pursuing of potassium titanate matrices.

TABLE 1

SUMMARY OF SINGLE-CELL TESTS

Cell No.	O_2 Electrode		H_2 Electrode		Mat Thick (In.)	Mat Dry Wt. (gm)	Electrolyte		Comments	Results
	No.	Catalyst	No.	Catalyst			% KOH	Wt. (gm)		
170	64	14 mg Pt/cm^2	65	14	FC Asb. .060	27.0	40	31.09	Cont. Conc. cell	Run for 456 hrs to 1 V. end pt. Final KOH 26.25%.
173	A_u52	14 mg Pt/cm^2	A_u51	14 mg Pt/cm^2	3 layers .020 K.T. Paper	21.5	40	31.0	Cycling concentration cell.	Cycled 200 times to .95V end point.
175	-	20 mg Pt/cm^2	-	20 mg Pt/cm^2	FC Asb. .060	27.1	40	31.0	O_2 electrode pasted Pt Black on Nickel Screen	Cycled 10 times; Poor perform. Final KOH 29.15%
176	72	"	59	"	4 layers (.020) ACCO I	21.5	40	31.0	Am. Cy. Asb. Matl.	Cycled 4 times low discharge voltage. Final KOH 27.85%
177	Am.Cy.	9 mg Pt/cm^2	Am.Cy.	9 mg Pt/cm^2	FC Asb. .060	26.8	40	31.0	Acid washed electrodes.	Cycled 3 times Good perform. Disassembled to replace mat. Final KOH 32.75
178	A_u61	20 mg Pt/cm^2	A_u138	20 mg Pt/cm^2	FC Asb. .060	27.0	40	31.0	Tested as a Primary FC	Ran poor due to water transport difficulties.
179	A_u58	"	A_u57	"	FC Asb. .060	27.0	40	31.0	Tested as a Primary FC Pt. Nickel plaque elect.	Ran poor due to water transport difficulties.
180	Am. Cy.	9 mg Pt/cm^2	Am. Cy.	9 mg Pt/cm^2	FC Asb. Acid washed	30.0	40	31.0	Electrodes used in Cell 177.	Cycled 45 times Cell exhibited poor eff., fair perform. Final KOH 32%.

TABLE 1 (Cont.)

SUMMARY OF SINGLE-CELL TESTS

Cell	O_2 Electrode		H_2 Electrode		Mat	Mat	Electrolyte			
No.	No.	Catalyst	No.	Catalyst	Thick (In.)	Dry Wt. (gm)	% KOH	Wt. (gm)	Comments	Results
181	Am.Cy.	9 mg Pt/ cm^2	Am. Cy.	9 mg Pt/ cm^2	3 layers .020 KT paper	-	-	-	New O_2 electrode Old H_2 electrode Cycling conc. cell.	Cycled 32 times to 1 V. Showed gradual increase in voltage. Final KOH 32.9%.
182	85	20 mg Pt/ cm^2 nickel plaque	86	20 mg Pt/ cm^2 nickel plaque	75% KT 25% FC Asb.	20	40	32		Developed internal short on 12th cyc. Good perform. Final KOH 35.2%.
183	Am.Cy.	9 mg Pt/ cm^2	Am.Cy.	9 mg Pt/ cm^2	FC Asb. Acid Washed	30	40.15	31	Cont. conc. cell	Ran 140 hrs. to .95 V. Showed gradual rise in voltage. Final KOH 36.3%.
184	Am.Cy.	" "	Am.Cy.	" "	"	25	40.15	31	Cont. conc. cell. Electrodes from cell 180	Ran 72 hrs. to 1.2 V. Showed gradual rise in voltage. Final KOH 30.5%.
185	A_u90	14 mg Pt/ cm^2	A_u91	14 mg Pt/ cm^2	75% KT 25% FC Asb.	20.0	40	36	-	426 cycles slow gas cross leakage at 418 cycles.
186	Am.Cy.	9 mg Pt/ cm^2	Am.Cy.	9 mg Pt/ cm^2	" "	20.0	40	31	Cycling conc. cell.	320 cycles to 1.1 volts.
187	Am.Cy.	" "	"	"	100% KT	17.5	40.15	31	Old electrodes cont. conc. cell	Ran 27 hours to 1.1 V. Had high initial voltage rose rapidly. Final KOH 35.6%.

TABLE 1 (Cont.)

SUMMARY OF SINGLE-CELL TESTS

Cell No.	O_2 Electrode		H_2 Electrode		Mat Thick In.	Mat Dry Wt. (gm)	Electrolyte		Comments	Results
	No.	Catalyst	No.	Catalyst			KOH	Wt. (gm)		
188	Am. Cy.	9 mg pt/cm^2	Am.Cy.	9 mg pt/cm^2	100% KT	20.0	40	31	Cont. conc. cell. Old electrodes	High initial voltage. Test discontinued.
189	" "	" "	"	" "	"	19.9	40	31	-	Short in assembly
190	" "	" "	"	" "	"	19.5	40.15	31.0	Cont. O_2 Conc. Cell test	700 hrs. to 1.1 volts.
191	Felt Metal	14 mg Pt/cm^2	Felt Metal	14 mg Pt/cm^2	"	19.5	40.15	34.0	Cont. o_2 Conc. Cell test	840 hrs. to 1.1 volts
192	Plat. gold screen	20 mt Pt/cm^2	Plat. nickel plaque	20 mg Pt/cm^2	90% KT 10% Ab	20	40.0	33.0		330 Cycles. Slow gas cross leakage at 327 cycles.
193	Plat. gold screen	" "	"	"	" "	19.5	40.15	34.0	Cycling O_2 Conc. Cell Test	200 cycles to 1.1 volts.
194	Am.Cy.	9 mg Pt/cm^2	Am.Cy.	9 mg Pt/cm^2	100% KT	19.0	40.15	31.0	Used electrodes	Initial poor performance.
195	Am.Cy.	9 mg Pt/cm^2	Plat. nickel plaque	20 mg Pt/cm^2	90% KT 10% Ab	20.5	40.0	34.09		278 cycles, then developed high internal resistance
196	Am.Cy.	9 mg pt/cm^2	"	"	" "	20.0	40.15	34.0		60 cycles, slow gas cross leakage at 60th cycle.
197	Plat. gold screen	25 mg Pt/cm^2	Plat. nickel plaque	20 mg Pt/cm^2	90% KT 10% Ab	20.0	40.15	34.0		Volume balance problem. Test discontinued.
198	"	"	"	"	"	19.6	40.0	34.0		Still on test at 516 cycles, gradual degradation.
199	Am.Cy.	9 mg Pt/cm^2	Plat. nickel plaque	20 mg Pt/cm^2	90% KT 10% Ab	24.5	40.15	34.0		400 cycles. Still on test.

TABLE 1 (Cont.)

SUMMARY OF SINGLE-CELL TESTS

Cell No.	O_2 Electrode No.	O_2 Electrode Catalyst	H_2 Electrode No.	H_2 Electrode Catalyst	Mat Thick In.	Mat Dry Wt.(gm)	Electrolyte % KOH	Electrolyte Wt. (gm)	Comments	Results
200	Am.Cy.	9 mg Pt/ cm^2	173	20 mg Pt/ cm^2	90% KT 10% AB	24.59	40.15	34.09		Test discontinued due to cell imbalance.
201	Plt.92 Gold Screen	25 mg Pt/ cm^2	Plt.93 Gold Screen	25 mg Pt/ cm^2	Teflon Cloth Mat		40.0	32	O_2 Cycling Conc. Cell	When started cell voltage rose to 1.5 V. Discontinued
202	Am.Cy.	9 mg Pt/ cm^2	Plt. nickel plaque	20 mg Pt/ cm^2	90% KT 10% Ab	20.09	40.0	34.0		386 cycles. Still on test.
203	Am.Cy.	9 mg Pt/ cm^2 - Extra Pt.	172 Plt. nickel plaque	20 mg Pt/ cm^2	90% KT 10% Ab (with teflon)	22.59	40.0	34		30 cycles. Fair performance.
204	Am.Cy.	9 mg Pt/ cm^2	127 Pt. nickel plaque	20 mg Pt/ cm^2	100% KT	250	40.15	33.0		Slow gas cross leakage.

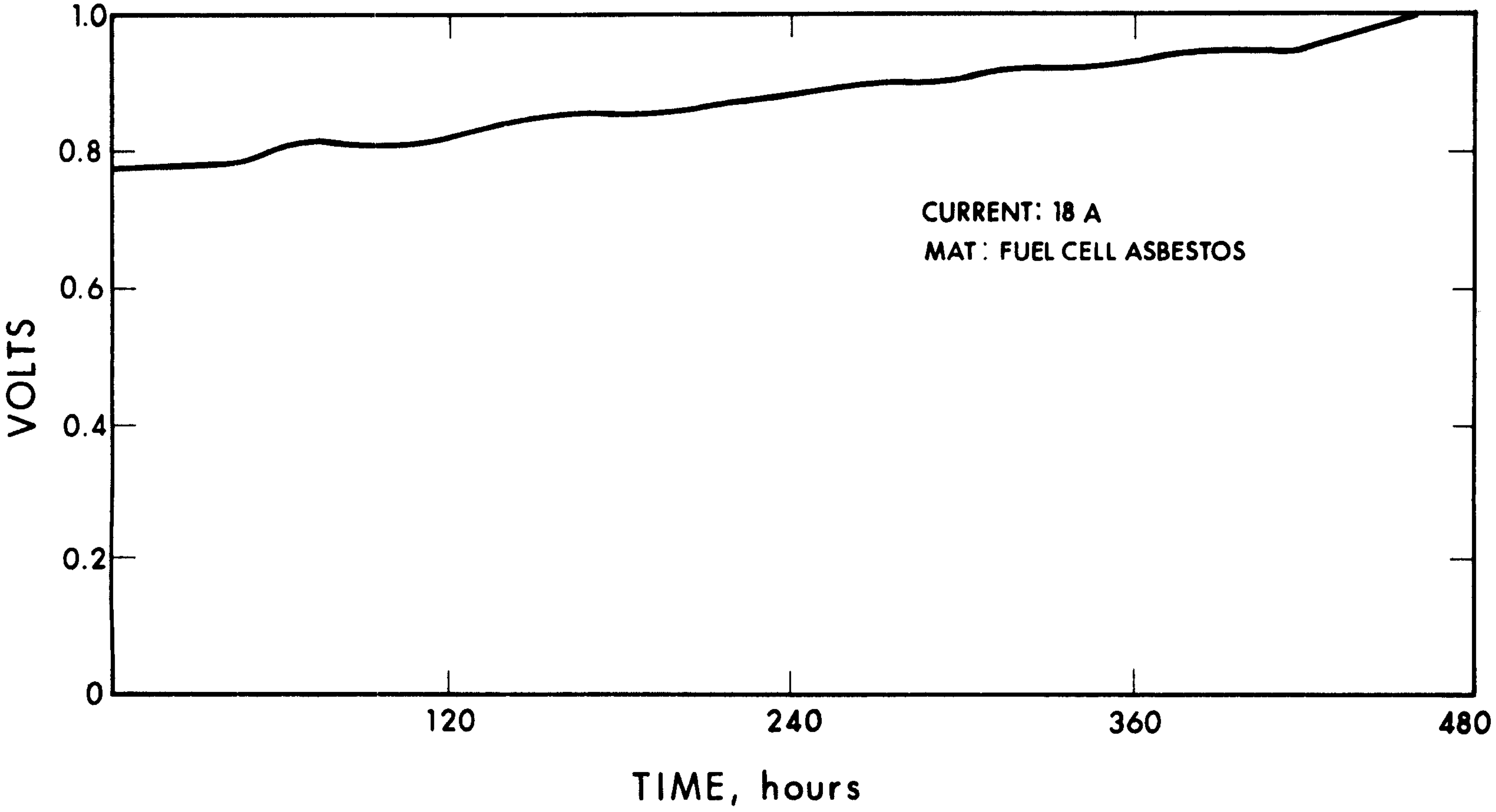

FIG. 1 CONTINUOUS O_2 CONCENTRATION CELL 170

TABLE 1 (Cont.)

SUMMARY OF SINGLE-CELL TESTS

Cell No.	O_2 Electrode		H_2 Electrode		Mat Thick In.	Mat Dry Wt. (gm)	Electrolyte		Comments	Results
	No.	Catalyst	No.	Catalyst			% KOH	Wt. (gm)		
200	Am.Cy.	9 mg Pt/cm^2	173	20 mg Pt/cm^2	90% KT 10% AB	24.59	40.15	34.09		Test discontinued due to cell imbalance.
201	Plt.92 Gold Screen	25 mg Pt/cm^2	Plt.93 Gold Screen	25 mg Pt/cm^2	Teflon Cloth Mat		40.0	32	O_2 Cycling Conc. Cell	When started cell voltage rose to 1.5 V. Discontinued
202	Am.Cy.	9 mg Pt/cm^2	Plt. nickel plaque	20 mg Pt/cm^2	90% KT 10% Ab	20.09	40.0	34.0		386 cycles. Still on test.
203	Am.Cy.	9 mg Pt/cm^2 - Extra Pt.	172 Plt. nickel plaque	20 mg Pt/cm^2	90% KT 10% Ab (with teflon)	22.59	40.0	34		30 cycles. Fair performance.
204	Am.Cy.	9 mg Pt/cm^2	127 Pt. nickel plaque	20 mg Pt/cm^2	100% KT	250	40.15	33.0		Slow gas cross leakage.

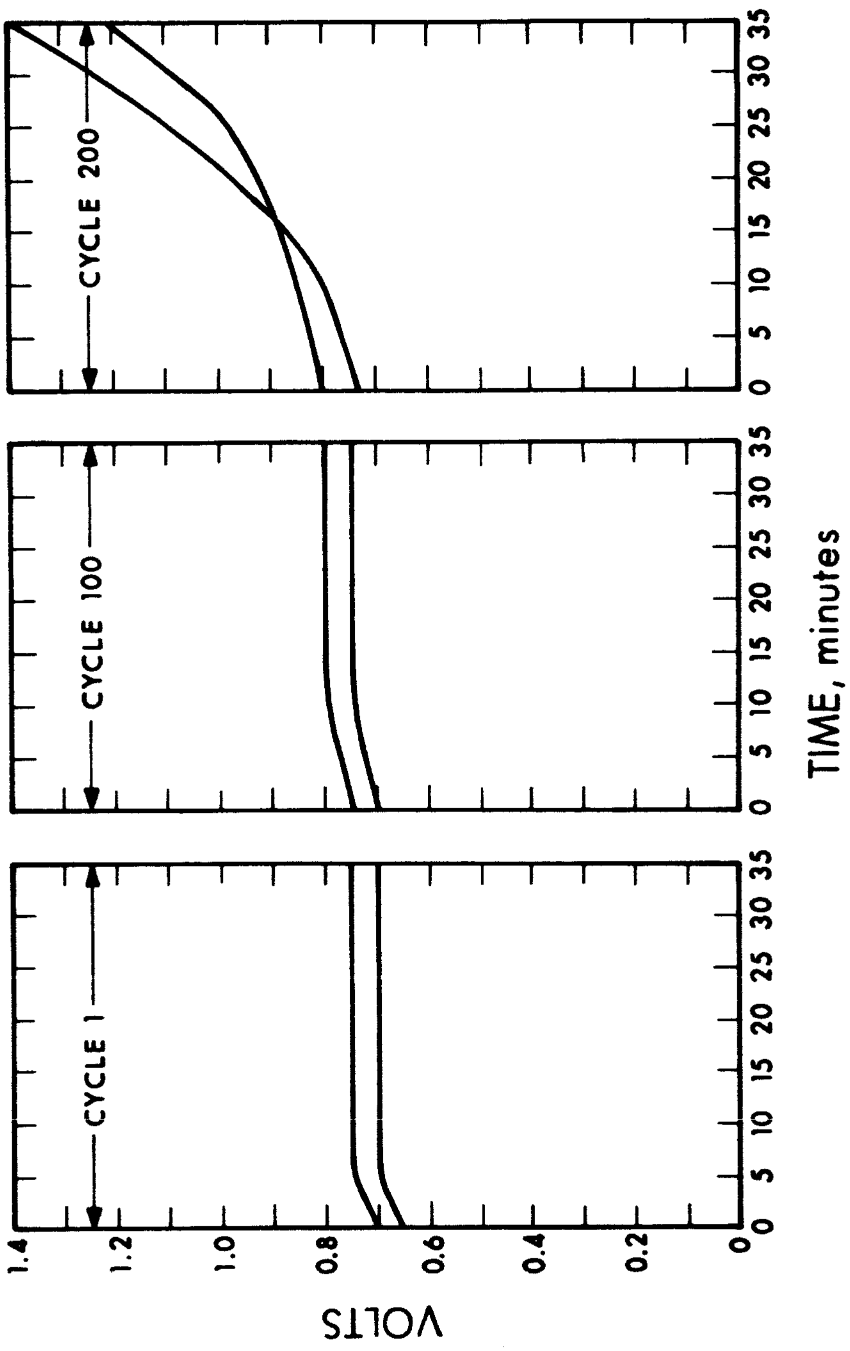

FIG. 3 CYCLING PERFORMANCE OF CELL 173

Cell No. 181 contained American Cyanamid type electrodes with a mat of three layers of 0.020 inch thick KT paper. One electrode employed was new, while the other electrode was one that had been previously used in other cell tests. The cell was subjected to test in a cycling concentration cell mode, and was tested continuously for 32 cycles. During the cycling, there was a gradual increase in voltage. The test was discontinued at the 32nd cycle when the voltage reached 1 volt. The poor performance could be attributed to the previously used electrode.

Cell No. 183 contained American Cyanamid type electrodes and a fuel cell grade asbestos mat that had been subjected to an acid wash process. The cell was tested in a continuous concentration cell mode and ran for a period of 140 hours to a 0.95 volt cutoff. During the period there was a gradual rise in voltage with time.

Cell No. 184 contained American Cyanamid type electrodes and an acid washed fuel cell grade asbestos mat identical in construction to Cell No. 183. This cell, too, was run in a continuous concentration cell mode and ran for a period of 72 hours to a 1.2 volt cutoff, showing a gradual rise in voltage with time. Final KOH of the cell was analyzed to be 30.5 percent.

Cell No. 186 was a cycling concentration cell run at 18 amps. (100 MA/cm^2) for 35 minutes at each polarity. Electrodes employed were American Cyanamid type AB6 containing 9 milligrams of platinum per cm^2 and the mat was 75 percent KT 25 percent asbestos. The potassium titanate paper used in Cells No. 173 and No. 181 was a sample of a material manufactured by DuPont that has been discontinued. This material has been replaced by a pigmentary potassium titanate and is presently the only form of potassium titanate available. The new material is essentially a powder consisting of particles approximately 0.2 microns in diameter and 10 microns long. Mats for the cells fabricated from pigmentary KT are made from a water slurry of the KT plus any additions desired. Forming is accomplished by filtering the slurry in a Buchner funnel. The filter cake (6-3/8" dia.) is then pressed to compact the mat. Asbestos was employed within the KT material for Cell No. 186 to add additional strength to the matrix since pure KT mats are extremely brittle. The cell was cycled for a total of 320 cycles, at which point the voltage reached 1.1 volts, and the test was discontinued. Additional moisture was added to the cell by filling the cell cavities with hydrogen and oxygen gas and discharging the gas into the cell. The cell was once again put on cycle and was run for an additional 10 cycles. The voltage of the cell after the addition of water, dropped to a lower level, then rose rapidly over the 10 cycle period to the high voltage level obtained when the test was stopped. The disassembled cell showed no visual signs of degradation, and the mat was clean and white in appearance throughout. The voltage performance of this cell is shown in Figure 4. Figure 4 shows a plot of the cell voltage mid points of each cycle as a function of cycle number. As can be seen, there is a difference between the voltage of the cell as the polarity shifts. Figure 5 shows the individual voltage performance at different cycles. As can be seen, there is a gradual increase in voltage with time, but this performance is far better than any previous performance obtained and indicates a very promising solution to degradation problems encountered in the past.

Cell No. 187 contained American Cyanamid type electrodes and a matrix made of 100 percent KT. This cell employed electrodes that had been previously used in a number of runs and that had possible contaminants on them. The cell ran in a test of the continuous concentration cell type for a period of 27 hours to 1.1 volt cut-off. The voltage of the cell was initially high and rose rapidly to the 1.1 volt level. Analysis of the KOH concentration in the mat revealed a 35.6 percent. The short run of this cell could be attributed to the old electrodes used in the cell.

Cell No. 188 also consisted of a cell employing old American Cyanamid electrodes with a 100 percent KT mat in a continuous concentration cell mode. This cell, too, exhibited initial high voltage and the test was discontinued.

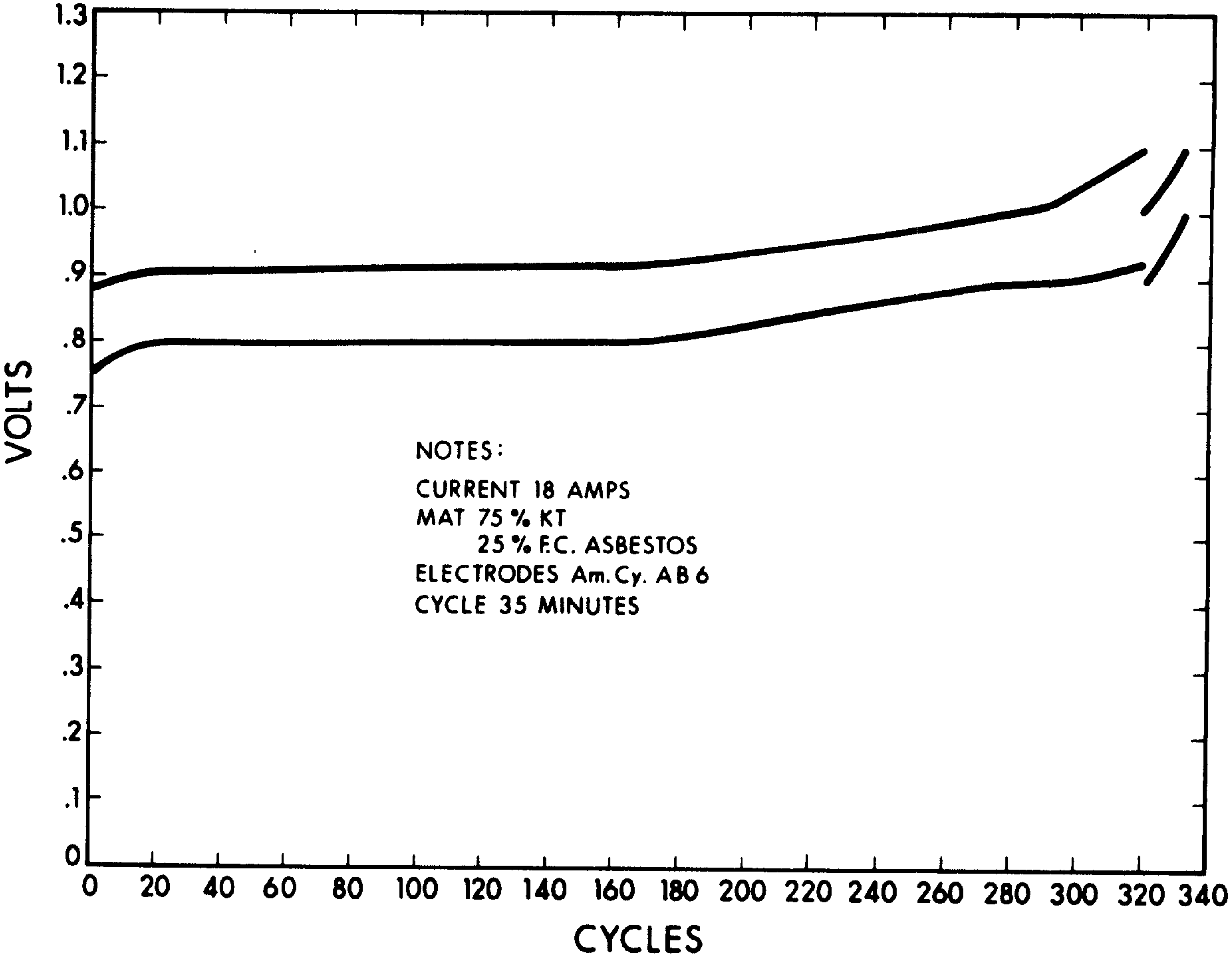

FIG. 4 CYCLING O_2 CONCENTRATION CELL 186 (MIDPOINT VOLTAGE)

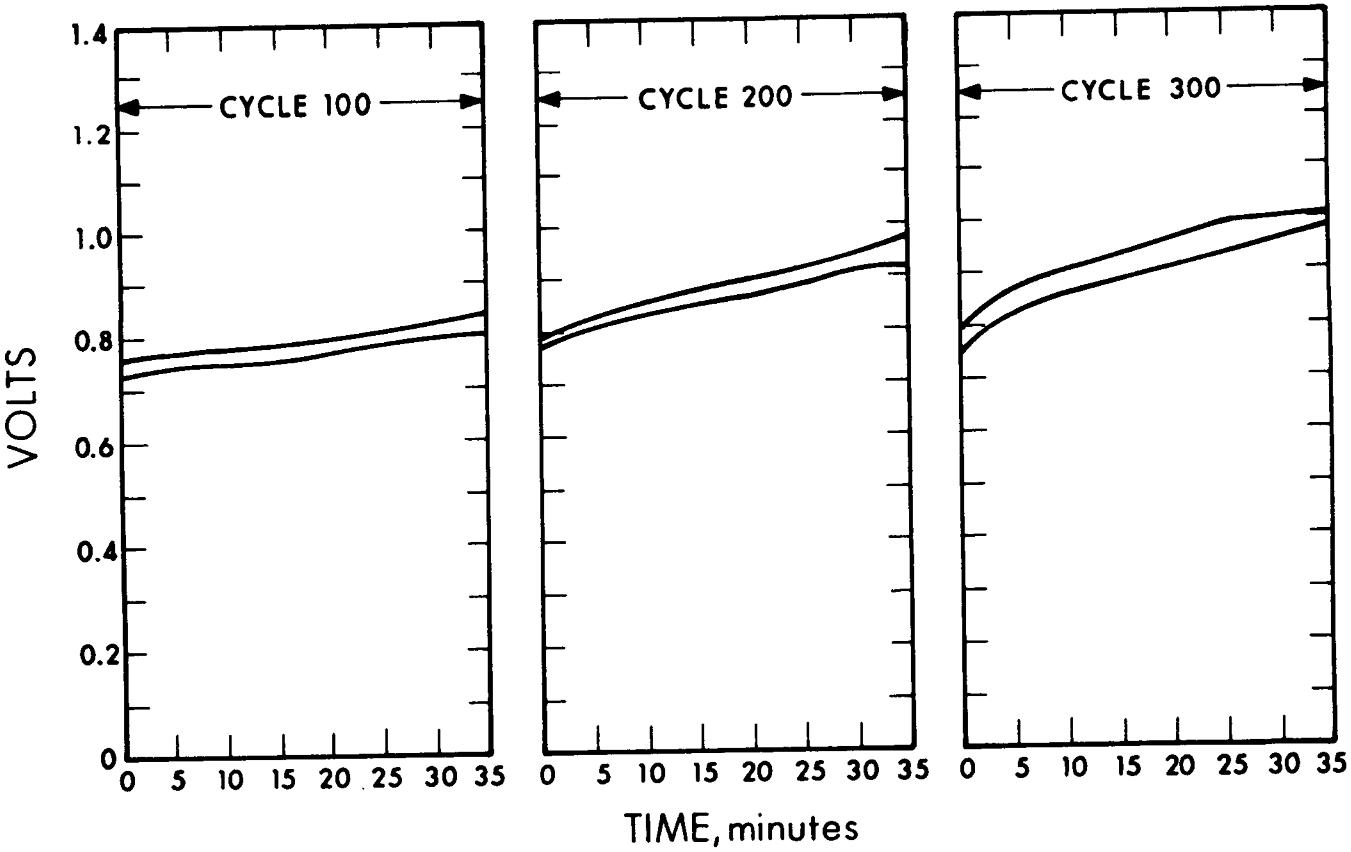

FIG. 5 CYCLING PERFORMANCE OF CELL 186

Cell No. 189 contained new American Cyanamid type electrodes with a 100 percent potassium titanate mat. This cell developed a short in assembly and was not subjected to test.

Cells Nos. 190 and 191 were continuous concentration cell tests. They both contained matrices composed of 100 percent potassium titanate. Cell 190 contained American Cyanamid type electrodes AB6, and Cell 191 contained Felt Metal Nickel electrodes that had been gold coated and platinized by electrodepositing platinum on the electrodes. Cell 190 ran for a period of 700 hours to a 1.1 volt cut-off point. Cell 191 ran for a period of 840 hours to 1.1 volt cut-off point. The results of these cells are shown in Figures 6 and 7. Throughout the test there was a gradual increase in voltage with time. The initial high point in voltage of cell 191 was due to a lowering of the oven temperature which was corrected. The life obtained with these two cells was far superior to that obtained in previous concentration cells with asbestos and shows the promising nature of potassium titanate matrices. From the results, it appears that a deteriorating mode still exists with potassium titanate matrices, but the rate of degradation is relatively low. Also to be considered is that in the concentration cell tests, there are two functioning oxygen electrodes--one in the discharge mode, and one in the charge mode.

Cell No. 193 consisted of two electrodes made of electroplating platinum black on a gold plated nickel screen. The cell employed a matrix of 90 percent potassium titanate, 10 percent asbestos, and was run in a cycling concentration mode. Figure 8 shows the mid-point voltage of the cell as cycling proceeded. Figure 9 shows the voltage of various cycles throughout the testing. As can be seen, there was a gradual rise in the voltage with cycling, and the latter cycles showed a more pronounced upward slope in the voltage over the time period, and the test was discontinued.

Cell No. 194 consisted of a used set of American Cyanamid electrodes with a 100 percent potassium titanate matrix. The cell was put on a continuous concentration cell test and rose very rapidly to 1.1

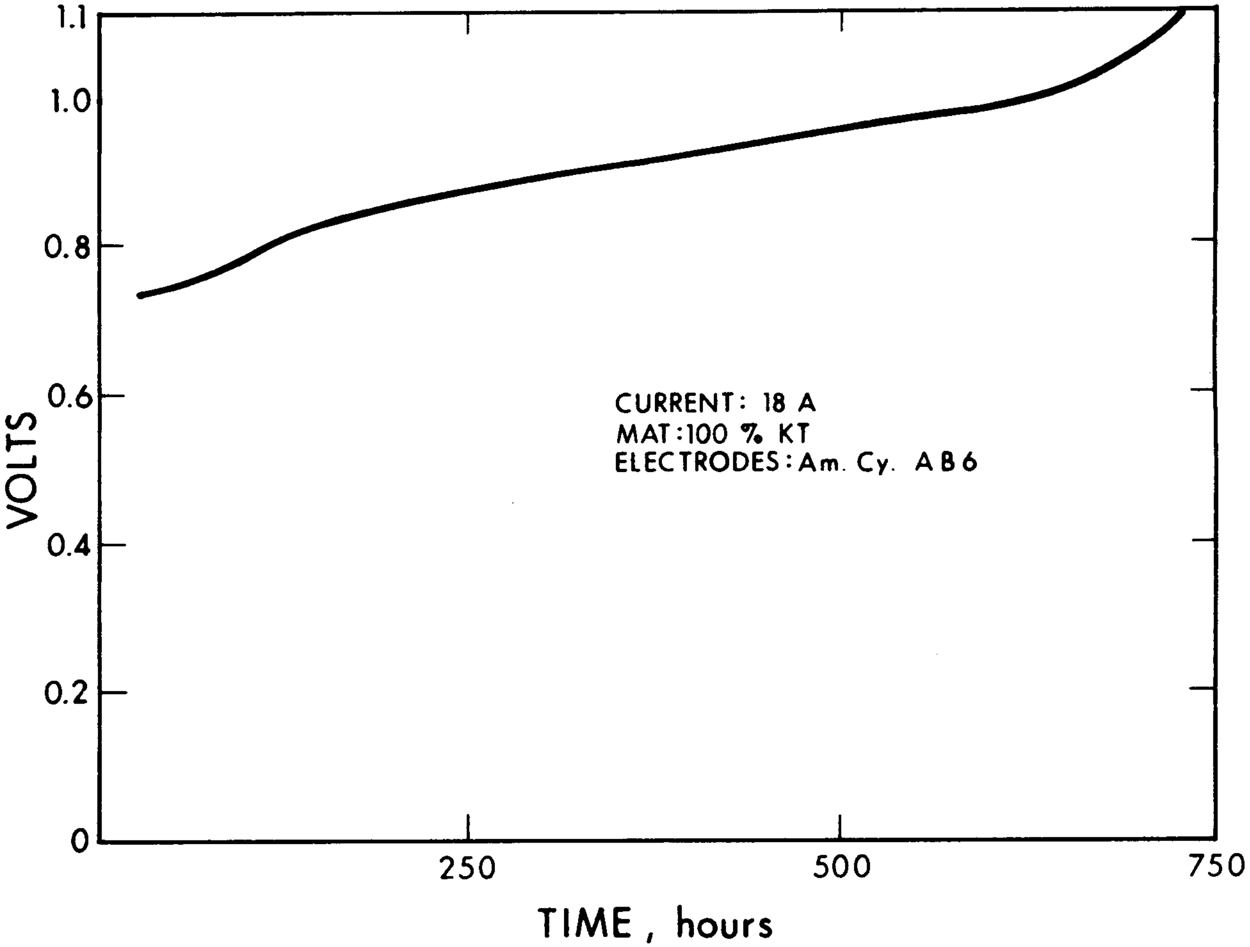

FIG. 6 CONTINUOUS O_2 CONCENTRATION CELL 190

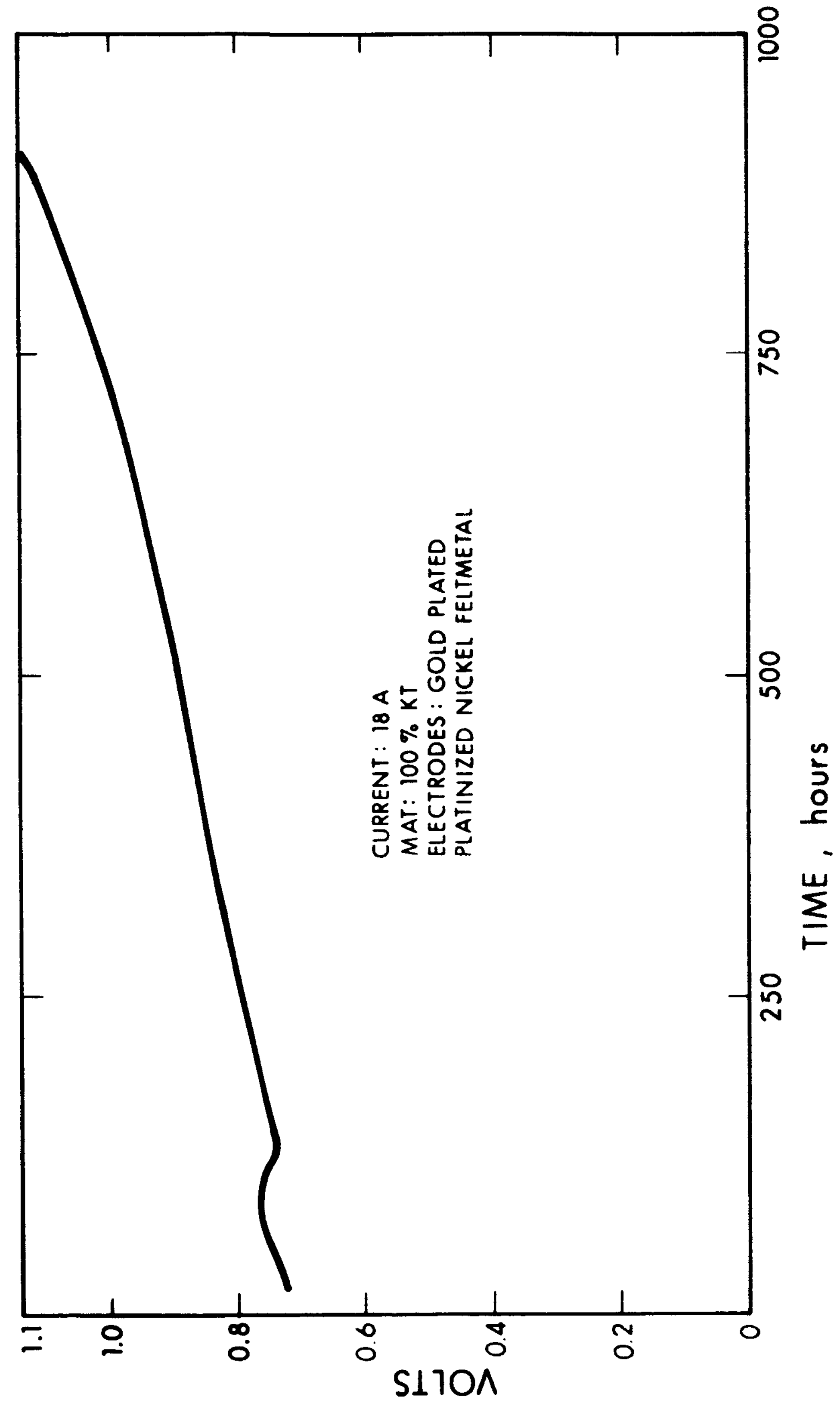

FIG. 7 CONTINUOUS O_2 CONCENTRATION CELL 191

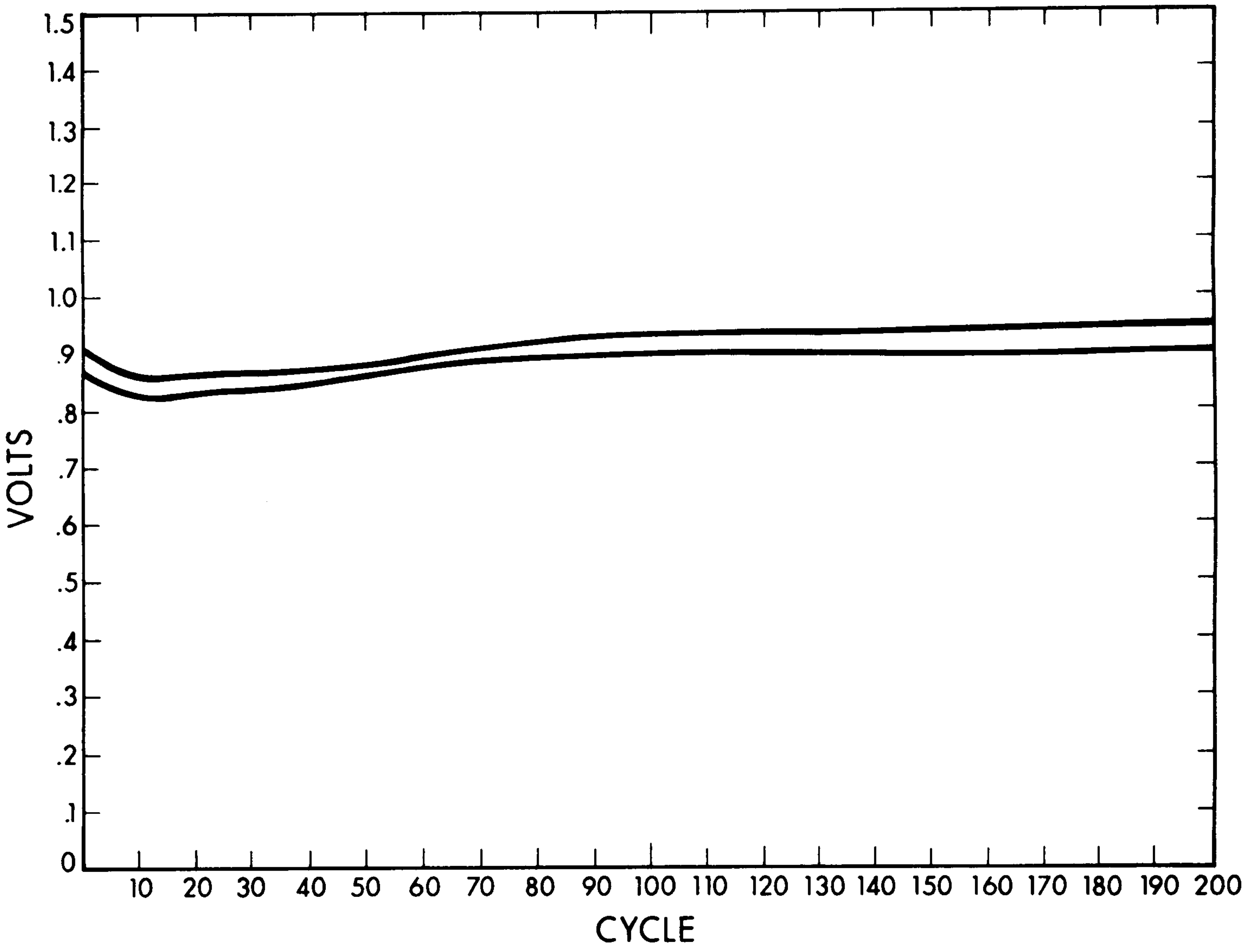

FIG. 8 SINGLE CELL 193 - CYCLING CONCENTRATION CELL 193 (MIDPOINT VOLTAGE)

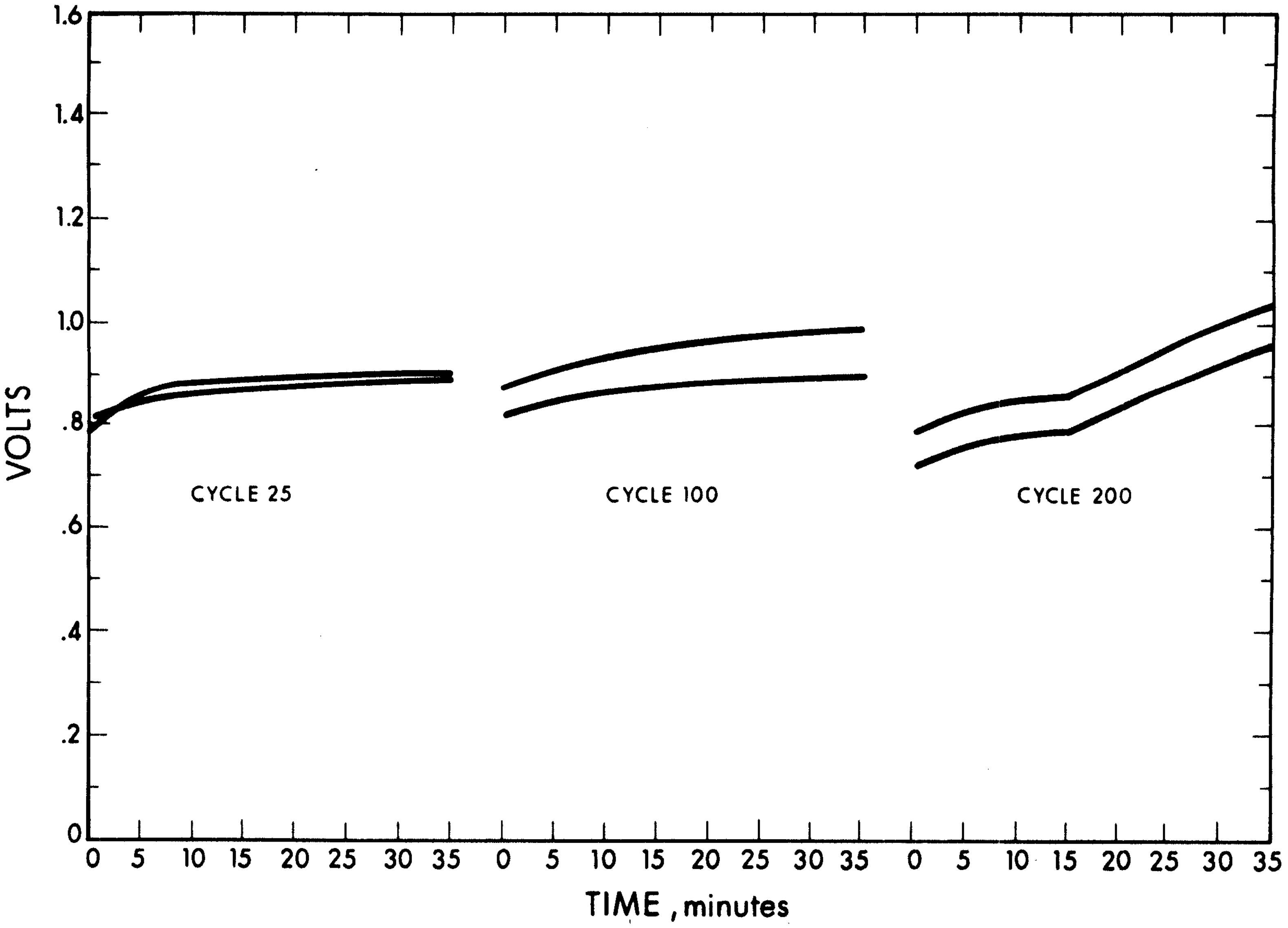

FIG. 9 PERFORMANCE OF O_2 CONCENTRATION CELL 193

volts, and the test was discontinued.

Cell No. 201 employed a platinized gold screen oxygen electrode and platinized gold screen counter electrode and was subjected to test in oxygen concentration cell test mode. The matrix was composed of two layers of teflon cloth that had been impregnated with potassium hydroxide electrolyte. The intent of the test was to use a complete inert matrix and determine if any degradation in performance occurred with these types of electrodes. However, when the cell was started on test, the voltage rose very rapidly to 1.5 volts and therefore concentration cell tests cannot be conducted. The high voltage was due to the high resistance of the teflon cloth matrices.

Table 2 represents a summary of all the concentration cell tests conducted thus far including cells tested in the previous report period. It shows the cell tests employing potassium titanate have performed superior to asbestos type cells. Based on the satisfactory performance of the KT mats and considering the limited test facilities available, it has been decided to do further testing and the perfecting of these matrices utilizing actual fuel cell cycling tests.

3.1.2 Primary Fuel Cell Tests

Cell No. 178 contained gold coated platinized electrodes with a fuel cell grade asbestos mat and was subjected to test in a primary mode in which gaseous oxygen and hydrogen were continuously fed to the cell. This test was conducted to determine if similar degradation would be observed in a primary mode of operation. The oxygen was dead ended and the hydrogen was allowed to vent from the cell to remove product water that was formed during discharge. For this test the standard single cell hardware that has been used in the testing of the regenerative fuel cell was utilized. In the testing of the cell, there was a gradual decrease in voltage with time. This was due to the type of construction in the cell which did not allow for intimate contact of the hydrogen gas flowing through the cell to pick up product water, and therefore the cell flooded out with time. The cell construction consists of a solid Monel back-up plate behind the electrode which contains four

TABLE 2

SUMMARY OF CONCENTRATION CELL RESULTS

Cell No.	Electrolyte	Matrix Type	Time to .9-1.1 V.	Final KOH
	Continuous			
162	Gold Coated and Pt.	F.C. Asbestos	140 hrs.	Reaction
167	" "	" "	130 hrs.	29.8%
170	" "	" "	456 hrs.	26.25%
183	Am. Cy.	Acid Washed Asbestos	140 hrs.	36.3%
184	Am. Cy.	" "	72 hrs.	30.5%
187	Am. Cy.	100 % KT	27 hrs.	35.6% (Old poor electrodes)
188	Am. Cy.	100 % KT	-	(Old poor electrodes)
190	Am. Cy.	100 % KT	700 hrs.	
191	Felt Metal	100 % KT	840 hrs.	
194	Am. Cy.	100 % KT	-	(Old poor electrodes)
	Cycling			
163	Gold coated and pt.	F.C. Asbestos	40 hrs.	-
165	" "	" "	152 hrs.	26.8%
171	Am. Cy.	" "	35 hrs.	28.8%
173	Gold Coated and Pt.	Kt. (3 x .020)	224 hrs.	-
181	Am. Cy.	Kt. (3 x .020)	37 hrs.	32.9%
186	Am. Cy.	75 % Kt. 25 % Asb.	372 hrs.	-
193	Platinized Screen	90 % Kt. 10 % Asb.	222 hrs.	-

1/8 inch diameter holes that allow the gas to feed to the electrode. The hydrogen gas flowing behind this back-up plate apparently did not have sufficient contact with the back side of the electrode to remove product water.

Cell No. 179 was a second attempt at a primary fuel cell using a new set of gold coated platinized electrode and fuel cell grade asbestos. In this cell the back-up plate was removed behind the hydrogen electrode and a nickel screen was put in place to facilitate water removal and intimate contact of the flowing hydrogen gas with the back surface of the hydrogen electrode. This cell, too, exhibited a gradual decrease in voltage with time, indicating improper water transport processes. These cells included 0.060 inch thick asbestos. Possibly this material reduced the transport processes as compared to 0.020 inch thick asbestos material that is normally used in primary fuel cells. Due to the water equilibration, difficulties encountered with the primary cells and a lower priority, this type test has been temporarily discontinued.

3.1.3 Potassium Titanate Cycling Tests

Cell No. 182 contained porous nickel plaques that had been chemically platinized with 20 milligrams of platinum per cm^2 and a mat that consisted of 75 weight percent potassium titanate and 25 percent fuel cell asbestos.

The cell was subjected to a standard test cycle and showed good initial performance. However, during the 12th cycle, an internal short developed in the cell. The cell was disassembled and the short traced to the outer edge of one of the electrodes. Shorting was due to an imperfection in the mat edge. Final KOH concentration of the mat was analyzed to be 35.2%.

Cell 185 consisted of a matrix made from 25 weight percent asbestos fibers and 75 percent potassium titanate pigment. Electrodes employed were porous nickel plaques that were gold coated and platinized by electrodepositing platinum black on the surface of the electrodes. Figure 10 shows the voltage performance at various cycles during the

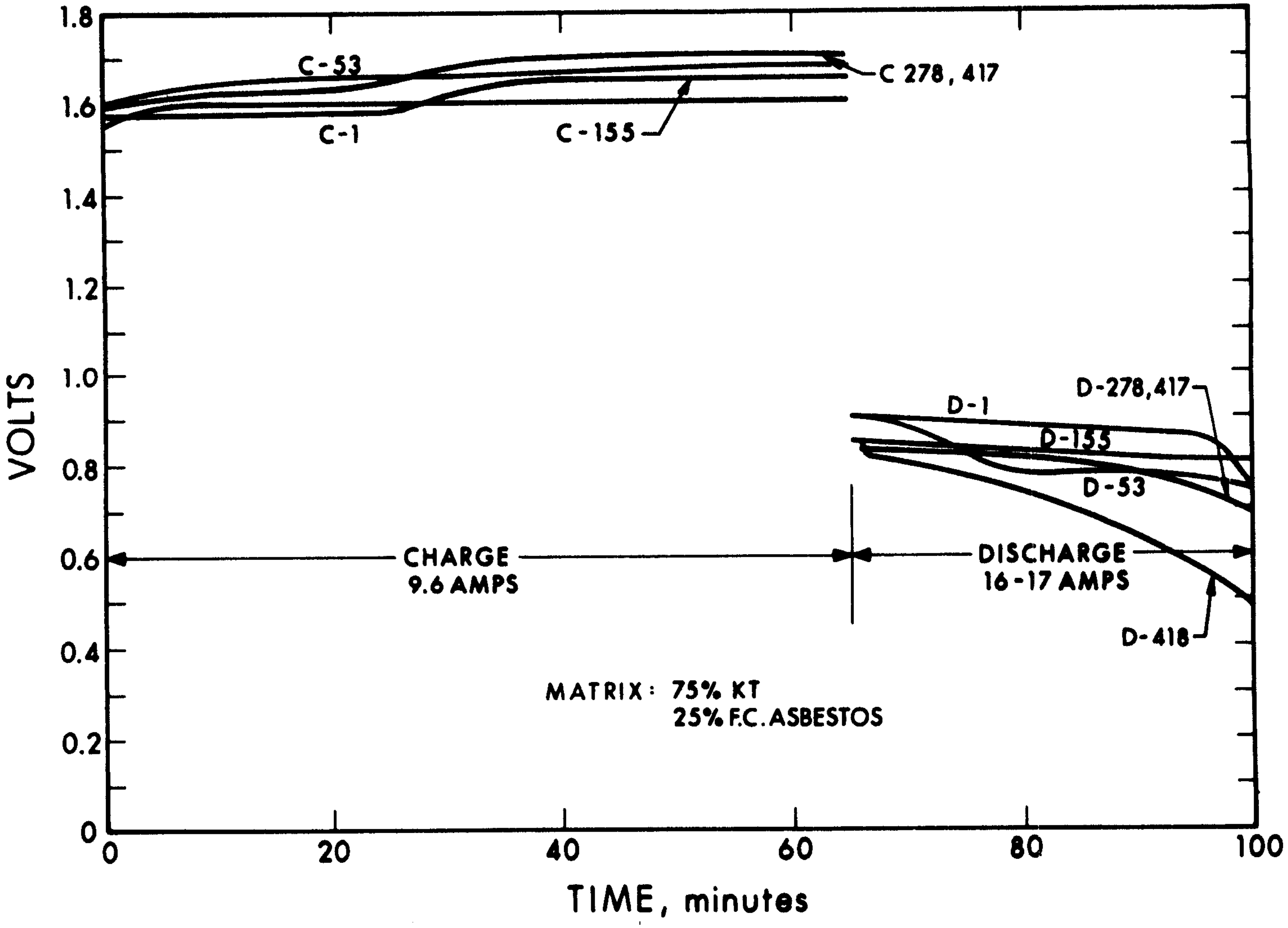

FIG. 10 VOLTAGE PERFORMANCE OF CELL 185

testing. As can be seen, there was a slight degradation in performance throughout the first 200 cycles at which point the voltage performance stabilized and was consistent and reproducible from cycle to cycle up to the 417th cycle. At that point, the discharge voltage exhibited a fall-off in performance towards the end of discharge. This fall-off of discharge voltage was caused by the reduction in operating pressure during the cycle. On subsequent cycles it was observed that on charge it was not possible to build up pressure greater than 200 psi. Beyond the 200 psi point, apparently a slow gas diffusion and recombination was taking place so that the additional charging of the cell did not increase the cell pressure. On the discharge, the performance fell off as the pressure went below 50 psi. The inability to charge the cell beyond the 200 psi level indicated that a structural deterioration had taken place within the mat which enabled gas diffusion and recombination. This cell contained electrodes that had a porous nickel plaque substrate and oxidation of the substrate in cycling had resulted in differential pressure swings as high as 15 psi as it switched from charge to discharge. It is possible that the accumulative effects of this differential pressure pushing on the matrix from side to side resulted in its structural deterioration and enabled the cross gas leakage and diffusion. The cell was cycled an additional 10 cycles beyond the point of rapid fall-off in performance to a total accumulated cycles life of 420 cycles, and the test was stopped. Examination of the internal cell components revealed no obvious visible changes in the matrix structure. A limited amount of platinum black was observed on the matrix adjacent to the electrodes which was apparently due to mechanical adherence, but the depth of the mat was relatively clean. An analysis of final electrolyte concentration in the mat revealed 32.5 percent.

Cell No. 192 consisted of a new type of oxygen electrode that was made by taking a close mesh nickel screen, gold plating it, and then electrodepositing platinum black on the screen to a loading of 20-25 milligrams platinum per cm^2. The platinum black was rolled lightly to press it into the holes of the screen and make a more adherent structure.

This electrode type was an attempt to make a platinum electrode that contained no wet proofing and would not be subject to oxidation of the substrate. The cell was assembled with a hydrogen electrode of the EOS standard type that was fabricated by recycling hot chloroplatinic acid solution through a porous nickel plaque to a loading of 20 milligrams per cm^2. The matrix was fabricated from a 90 percent potassium titanate, 10 percent asbestos mix by weight. The cycling test results are shown on Figure 11. As can be seen, there was a gradual increase in charge voltage with cycling, and a gradual decrease in discharge voltage with cycling. On the 327th cycle, the cell started to recombine during charge, and the cell pressure only rose to 200 psi. Therefore, the test was discontinued. The results of this test and previously observed results of Cell 185 indicate that the titanate asbestos matrices are possibly too porous, and minute structural changes that occur due to differential pressures could be causing cross leakage. Attempts to densify and increase the controls of these type of matrices will be investigated in the next period.

Cells 195 consisted of American Cyanamid AB6 type oxygen electrodes and hydrogen electrodes that were fabricated by recycling hot chloroplatinic acid through porous nickel plaques. The mats were fabricated from a 90 percent KT, 10 percent asbestos weight mix.

The cell was cycled continuously and showed virtually no degradation of performance to 278 cycle point at which time the discharge voltage dropped to a very low level. The cycling performance is shown in Figure 12. No explanation was apparent for the sharp drop in performance of the cell. This cell deterioration was different than previously observed type deteriorations since over a period of 1 cycle there was a sharp drop in performance. Due to this type of failure, the cell was disassembled and a new matrix consisting of 90 percent potassium titanate, 10 percent asbestos was inserted in the cell utilizing the same electrodes without any washing. This new cell was designated No. 202 and was put back on cycling. The cycling performance of cell 202 is shown in Figure 13. As can be seen, there has been a

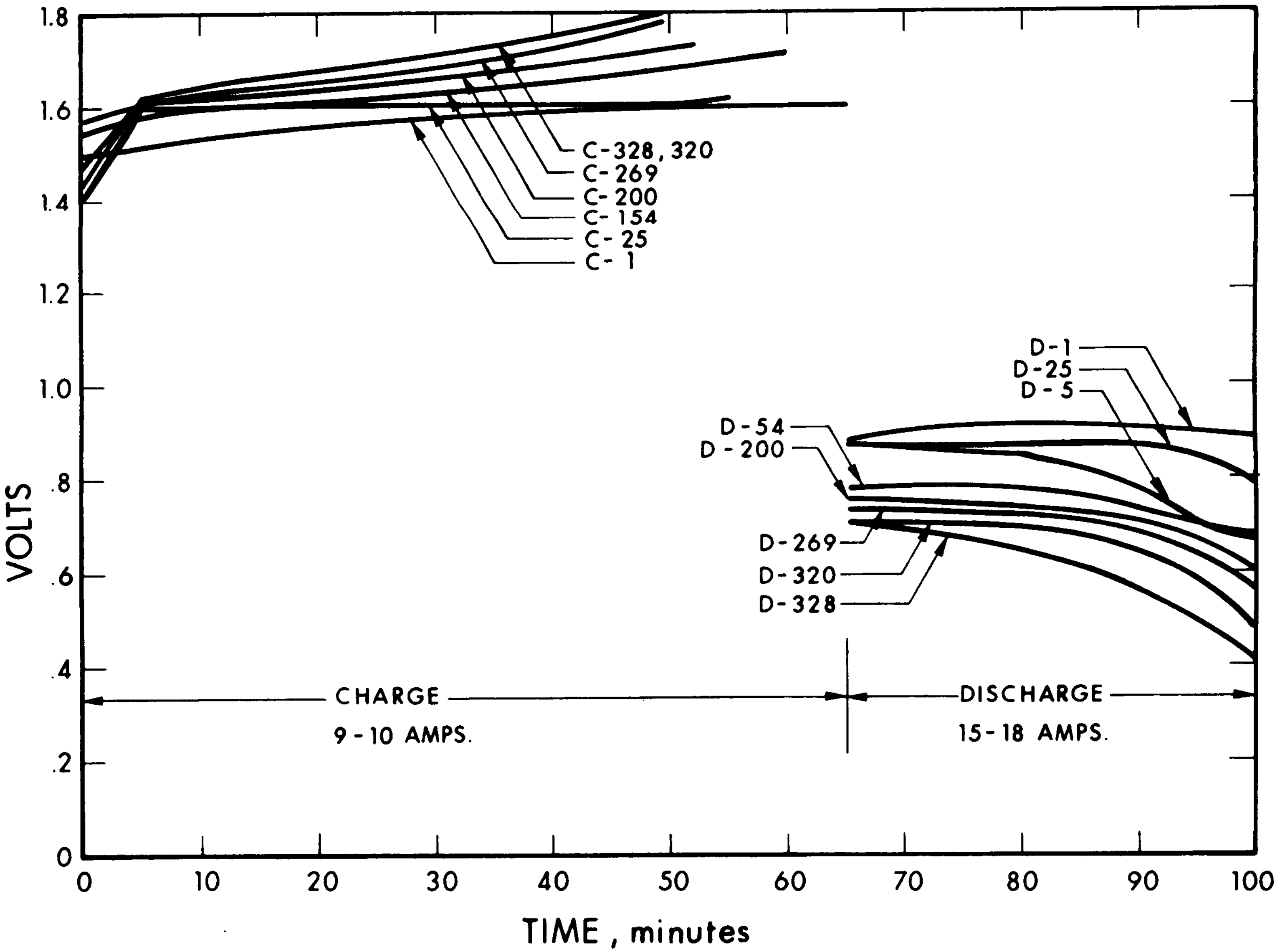

FIG. 11 CYCLING PERFORMANCE OF CELL 192

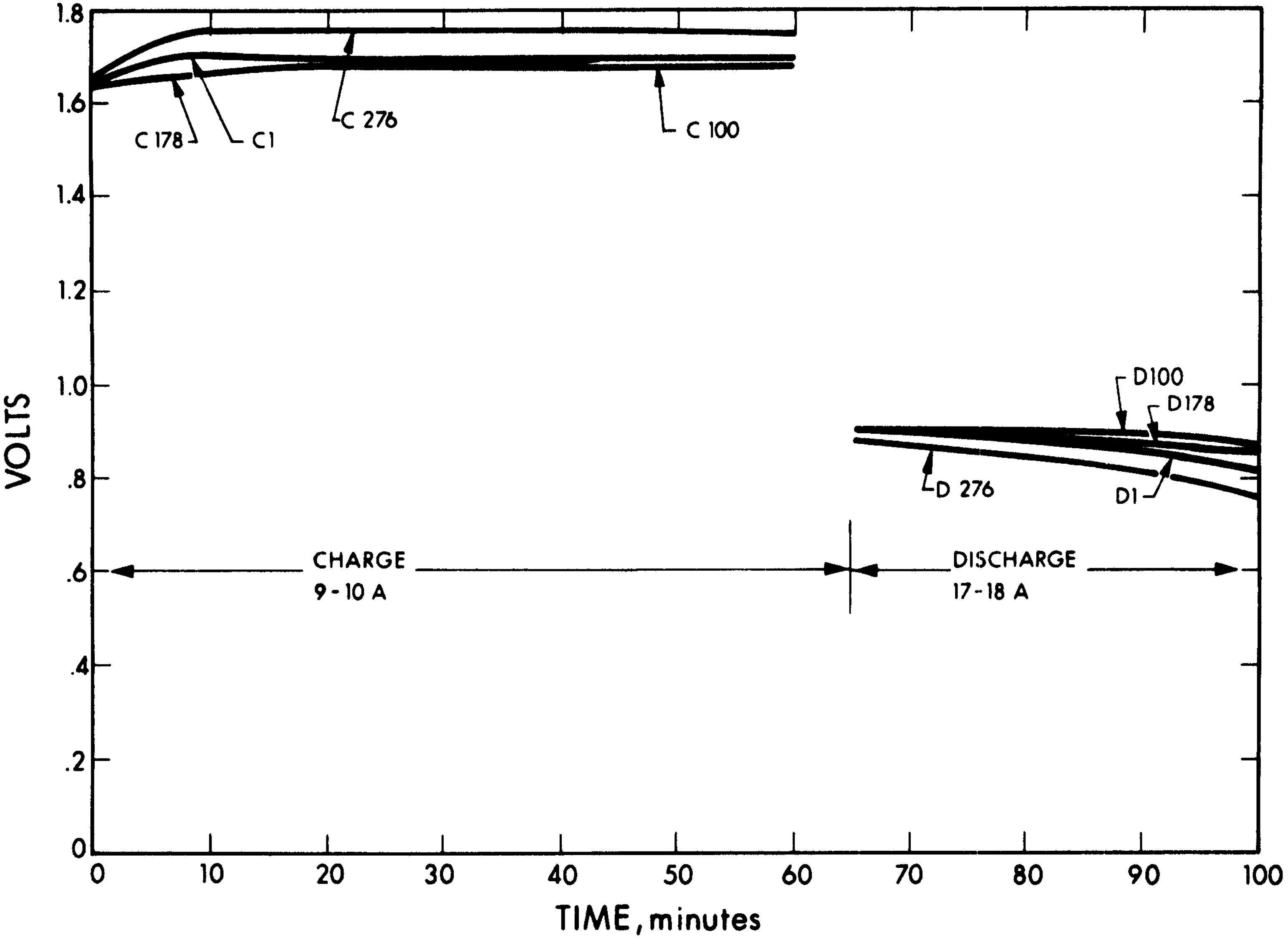

FIG. 12 CYCLING PERFORMANCE OF CELL 195

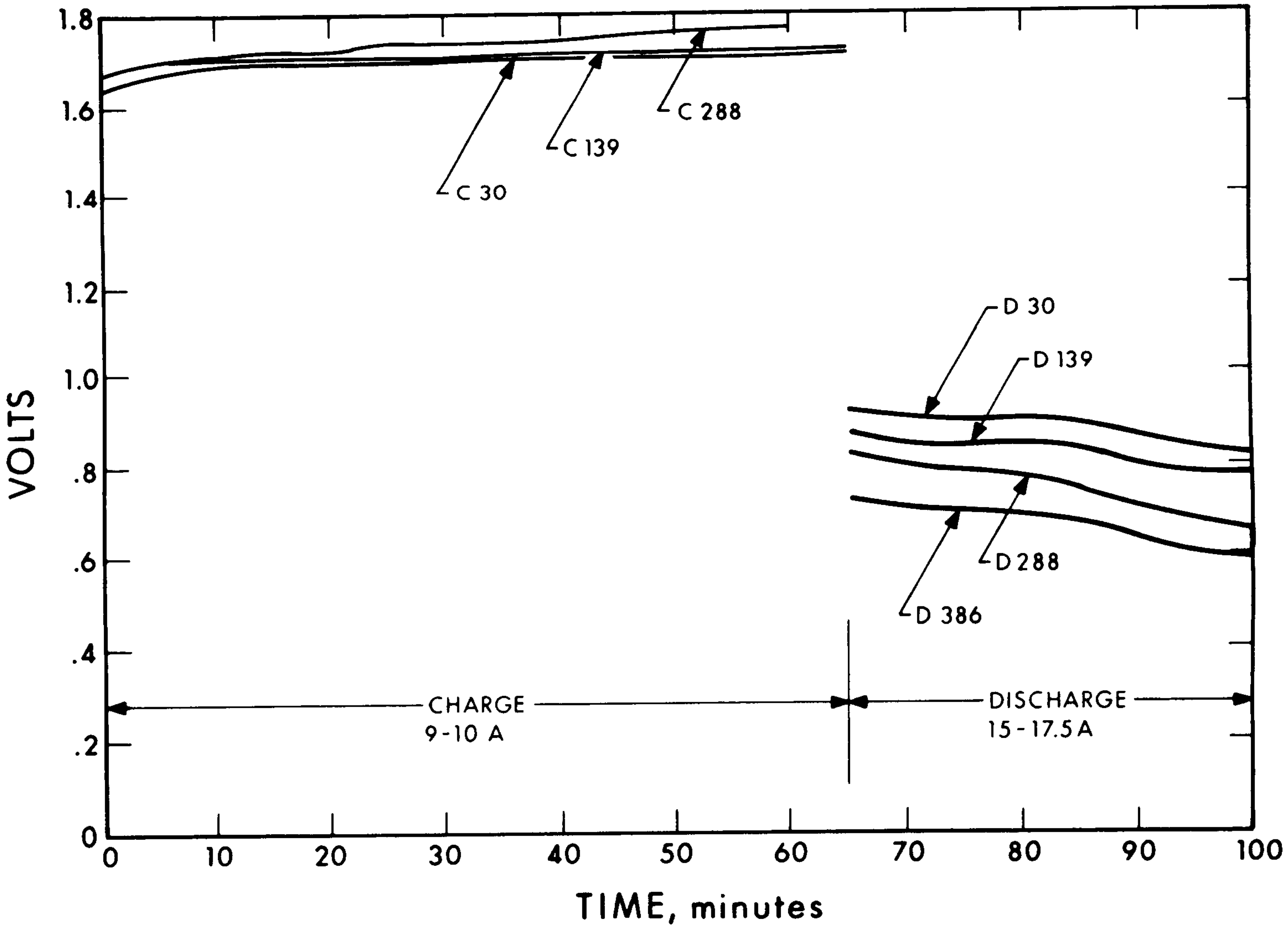

FIG. 13 CYCLING PERFORMANCE OF CELL 202

gradual degradation in performance with cycling, in particular, the discharge voltage. The cell has reached 386 cycling point at this time and is still under test. The accumulated number of cycles on the electrodes adding the cycles obtained with cell No. 195 is 664 cycles. The recovery of the cell with the insertion of the new matrix indicates possibly a high resistance contact had developed within the cell. The final electrolyte concentration in the matrix of cell 195 was found to be 30.03 percent. A one inch square sample of the matrix was analyzed for platinum and found to contain .0078 grams of platinum which is approximately 1.2 milligrams of platinum per cm^2. It was not established whether this platinum in the matrix was material that adhered to the surface of the matrix adjacent to the electrodes during the disassembly, or whether it had actually migrated into the mat. This represents a relatively small quantity of platinum, and it has not been established yet whether any substantial platinum migration occurs with the use of potassium titanate matrices.

Cell 196 showed good performance but on the 60th cycle exhibited an internal slow initial recombination and cross leakage, and the pressure during charge did not rise above 200 psi. The cross leakage difficulty encountered with this and previous cells with fabricated potassium titanate mats points to an area of required improvement in the mat fabrication procedure. This will be studied in the next period.

Cells 197 and 198 consisted of platinized nickel screen oxygen electrodes and platinized porous nickel plaque hydrogen electrodes with matrices 90 percent potassium titanate and 10 percent asbestos. Cell 197 exhibited a volume balance problem and the cell was only subject to a limited number of cycles and the test was discontinued.

Cell No. 198 was cycled continuously and has achieved 516 cycles to this point, and is still under test. Figure 14 shows the performance of the cell at various cycles. As can be seen, the performance was relatively stable during the first 350 cycles at which point there was a drop in performance. The low performance of the 417th cycle could be attributed to a difficulty encountered with the charger power

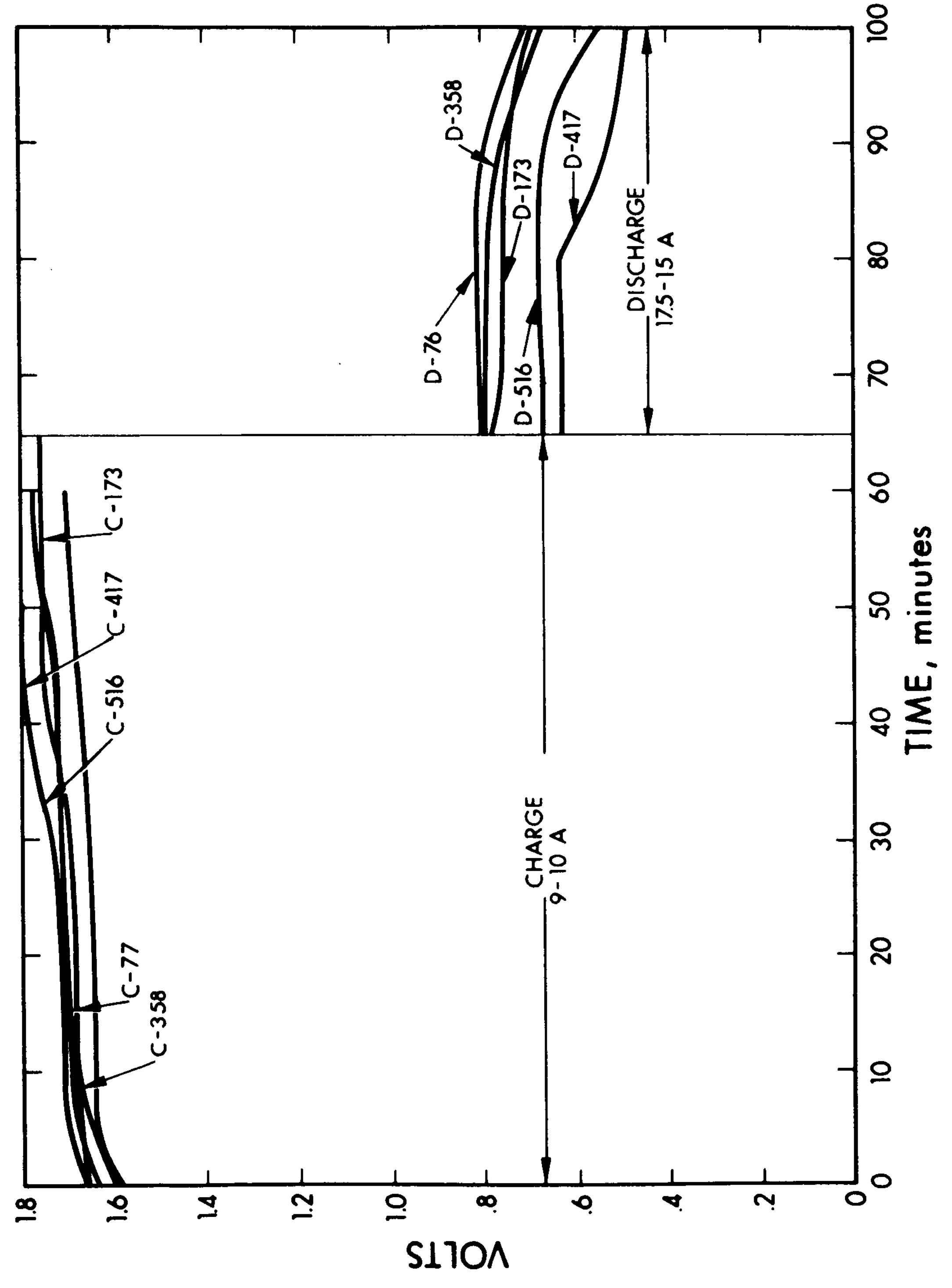

FIG. 14 CYCLING PERFORMANCE OF CELL 198

supply which dropped off in current during that cycling period. The power supply was replaced and the cell cycling continued, and it has improved somewhat as shown by the performance of the 516th cycle. The performance of this single cell which is still under test is superior to life testing of previous cells, and indicates a very promising cell configuration for improved life.

Cells 199 and 200 consisted of American Cyanamid oxygen electrodes platinized porous nickel plaque hydrogen electrodes and matrices made from 90 percent potassium titanate, 10 percent asbestos by weight. In both of these cells the total weight of the matrix was increased from the previously tested 20 grams mats to 24.5 grams. This increase in weight was an attempt at improving the structural characteristics of the matrix since difficulties had been encountered with cross-leakage with some of the prior tests. Cell 199 has been subjected to 400 cycles and is still under test. The performance of this cell is shown in Figure 15. As can be seen, there has been a gradual deterioration in the discharge voltage performance of the cell with cycling. The test will be discontinued, and a new matrix substituted in an attempt to determine the component that is causing the deterioration. Cell 200 was given a limited number of cycles, and the test was discontinued due to gas leakage and cell imbalance problems in the test cell set up.

Cell No. 203 consisted of American Cyanamid oxygen type electrode and a platinized porous nickel plaque hydrogen electrode. The matrix consisted of a 90 percent potassium titanate, 10 percent teflon fiber mix. This matrix was an attempt to substitute teflon fibers for the previously used asbestos fibers as a mechanical strengthener to the matrix. Teflon fibers were obtained by cutting and shredding teflon cloth that contained multi-filament threads. Initially difficulty was had in obtaining a uniform mixture of teflon fibers and the potassium titanate since the non-wetting nature of the fibers resulted in a floating to the top in the mix solution during the processing. The use of potassium hydroxide as a wetting agent enabled obtaining fairly uniform mix and therefore the matrix was made and assembled and subjected to test in this cell. The performance

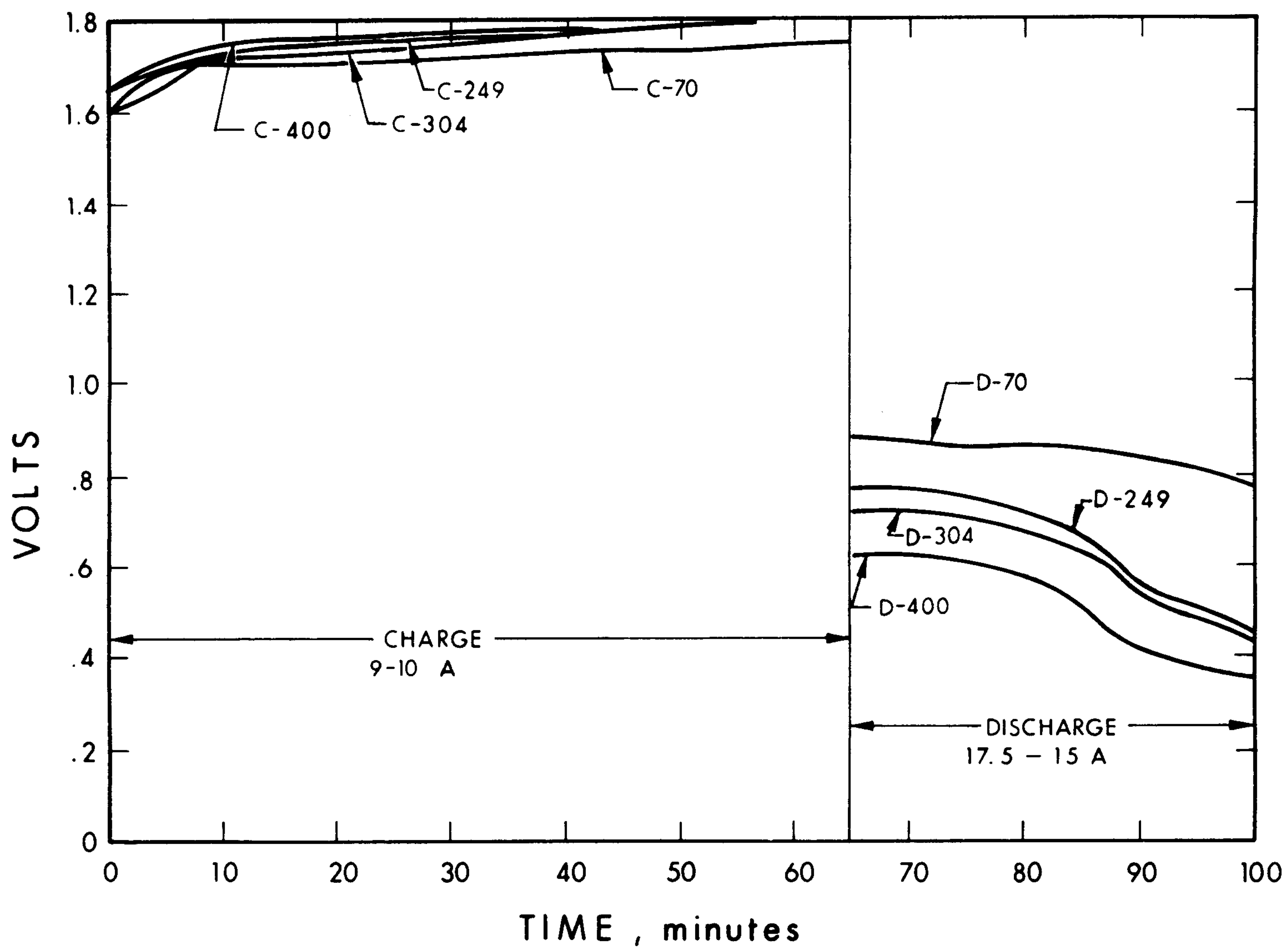

FIG. 15 CYCLING PERFORMANCE OF CELL 199

of this cell is shown in Figure 16. Initial performance was fair, and as can be seen, there was a rapid deterioration in performance. The cause for the deterioration has not been determined yet, but additional mats of this type will be evaluated.

Cell No. 205 consisted of American Cyanamid oxygen electrode and a porous nickel plaque hydrogen electrode. The matrix was 100 percent potassium titanate, and the cell was subjected to standard test cycle. However, during the charge slow recombination occurred and pressures beyond 200 psi were not achieved. Therefore, the test was discontinued.

Table 3 summarizes the results of the potassium titanate matrices cell tests. As can be seen, cells 185, 192 and 196 employing 20 grams mats developed cross leakage, indicating that an improvement in the process procedure is required. The 24.5 gram mats have not been fully evaluated yet.

Gradual deterioration in voltage performance has been observed in the 300 - 500 cycle range with those cells that reach this cycle life. The component, the hydrogen electrode, the oxygen electrode or matrix that is causing the difficulty has not been isolated yet. This will be attempted in the next period.

3.1.4 Additional Single Cell Tests

Cell No. 175 contained an oxygen electrode that was fabricated by pasting black platinum on the surface of a gold plated nickel screen. The hydrogen electrode was a standard type platinized nickel plaque. Fuel cell grade asbestos was employed as the matrix. The oxygen electrode was fabricated in an attempt to make a non-nickel substrate, non wet-proofed type of oxygen electrode. The cell was subjected to test and showed poor performance both on discharge and charge. After 10 cycles, this test was discontinued.

Cell No. 176 contained hydrogen and oxygen electrodes fabricated by the standard technique of recycling hot chloroplatinic acid through porous nickel plaques until 20 milligrams of platinum per cm^2 is deposited on the electrode. The mat consisted of four 0.020 inch layers of ACCO No. 1 asbestos manufactured by the American Cyanamid Co. Four layers of

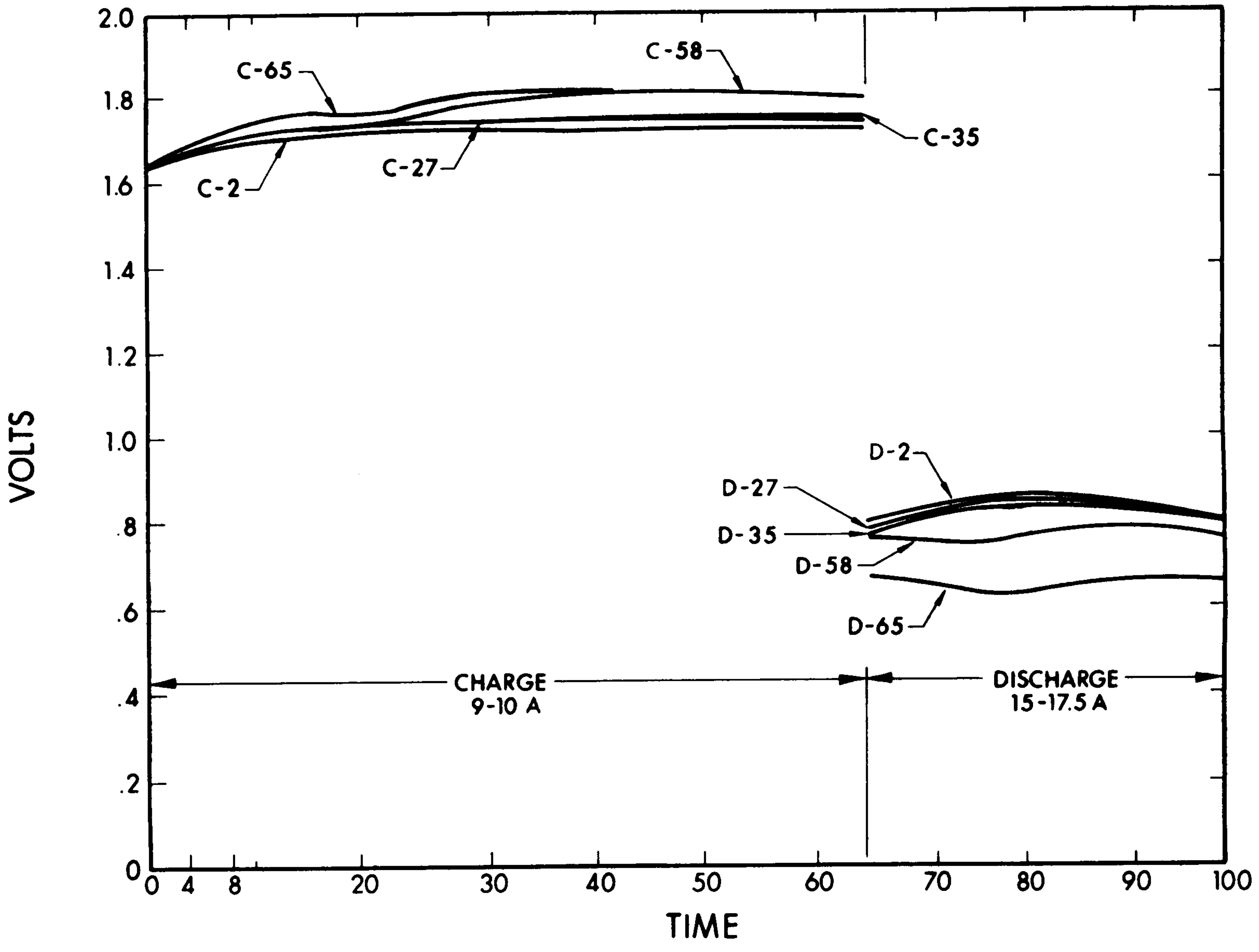

FIG. 16 CYCLING PERFORMANCE OF CELL 203

TABLE 3

Summary of Potassium Titanate Single Cell Tests

Cell No.	O_2 Electrode	H_2 Electrode	Mat Composition	Cycles	Results
182	Plt. porous nickel plaque	Plt. porous nickel plaque	75% KT 25% Asb.	12	Developed internal short
185	" "	" "	" "	417	Developed slow cross leakage at 200 psi
192	Plt. nickel screen	Plt. nickel plaque	90% KT 10% Asb.	327	" "
195	Am. Cy.	" "	" "	278	Developed internal high resistance
202	" "	" "	" "	386 (Still on test)	Same electrodes at 195, gradual deterioration
196	" "	" "	" "	60	Developed slow cross leakage at 200 psi
197	Plt. nickel screen	" "	" "	--	Test stopped due to volume balance
198	" "	" "	" "	516 (Still on test)	Gradual deterioration
199	Am. Cy.	" "	" "	400 (Still on test)	Gradual deterioration
200	" "	" "	" "	--	Test stopped due to gas leakage of cell
203	" "	" "	90% KT 10% Teflon Fiber	20	Rapid deterioration of performance
205	" "	" "	100% KT	--	Had slow cross leakage at 200 psi

material were used to prevent possible cross leakage in the mat, since this asbestos material has a higher porosity and lower bubble through pressure than conventional fuel cell grade asbestos. The cell was subjected to cycle and exhibited low discharge voltage i.e., in a range of 0.6 to 0.7 volts. The charge voltage was good, i.e., 1.5 to 1.6 volts. The test was stopped after four cycles due to the low discharge voltage. Visual examination of the mat material showed a gray discoloration throughout the mat. An analysis of sample of electrolyte squeezed from the mat revealed a final KOH concentration of 27.85 percent, indicating that this type of asbestos also reacts within the cell environment.

Cell No. 177 consisted of previously used American Cyanamid type electrodes that were acid washed in hydrochloric acid, then washed with water, soaked in potassium hydroxide and again washed with distilled water to remove any possible impurities in the electrodes. The cell was assembled with a fuel cell type asbestos mat and subjected to 3 cycles to evaluate the effect of acid washing on the electrodes. The cell exhibited good performance. At the end of three cycles, the test was discontinued to replace the fuel cell mat with an acid washed asbestos mat which was then designated Cell No. 180.

Cell No. 180 contained American Cyanamid type electrodes and a mat made from acid washed fuel cell grade asbestos. The electrodes in this cell had been previously used in Cell No. 177. The cell was cycled on a standard test cycle of 65 minutes charge, 35 minutes discharge for 45 cycles, at which time the test was discontinued. During cycling, the charge and discharge voltage levels were good, with the exception that at the end of discharge, there would be a considerable fall-off in voltage, indicating drowning of the electrodes. Difficulties were had in getting the cell pressure above the 250 psig level, indicating that gas leakage and recombination was occurring as the mat was drying out. Due to this difficulty, the end of discharge pressure was noted to continually decrease. Analysis of a sample of electrolyte squeezed from the mat revealed a final KOH concentration of 32.9 percent.

3.2 Multicell Testing

At the end of the last report period, as described in the 7th Quarterly Report, six cell multicell unit No. 109 was removed from testing after 22 cycles due to poor performance. An examination of the disassembled unit revealed no obvious defects that could have caused the low performance. Analysis of the final electrolyte concentration in the asbestos mats resulted in the following:

Cell No. 1 - 27.9 %
Cell No. 2 - 27.0 %
Cell No. 3 - 23.3 %
Cell No. 4 - 23.0 %
Cell No. 5 - 26.35 %
Cell No. 6 - 28.4 %

The variation in final electrolyte concentration and the relatively low values obtained in cells Nos. 3 and 4 indicate the possible cause for the poor performance. These results indicate once again that the asbestos mat material was reacting with the electrolyte and probably causing the cell's poor performance.

Based on the satisfactory results with the potassium titanate matrices in the single cell test, a new six cell unit was assembled for test in this period with these types of mats. The individual cells were made up of American Cyanamid AB6 type oxygen electrodes, EOS platinized nickel plaque hydrogen electrodes, and 90 percent KT, 10 percent asbestos mats.

The cycling performance of the 6 cell unit is shown in Figure 17. As can be seen, there was a gradual increase in the charging voltage with cycling and decrease in the discharge voltage with cycling. The rate of degradation observed is considerably greater than observed with single cells employing the electrode matrix type that was utilized in this unit. Due to the degradation in performance, the test was stopped at the 175th cycle and the unit was disassembled. An examination of the internal components of the cell revealed that a number of the bipolar plates had areas where severe corrosion had taken place. Apparently, there were

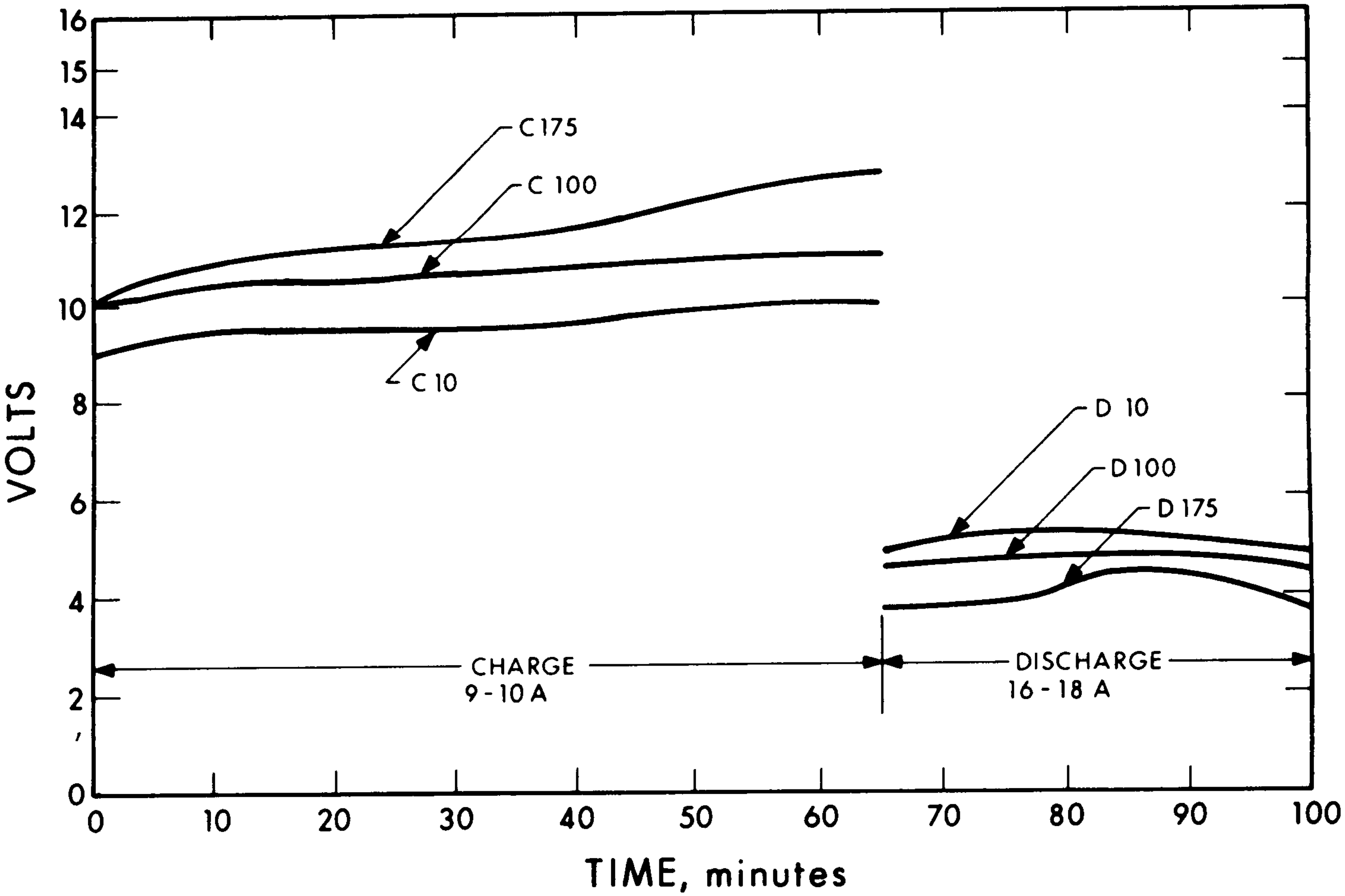

FIG. 17 CYCLING PERFORMANCE OF 6 CELL UNIT 110

imperfections in the electroless nickel and gold plating that is employed over the magnesium plates and potassium hydroxide electrolyte contacted the magnesium and resulted in corrosion and pitting of the plates. There were substantial quantities of muddy type corrosion product throughout the backup screens behind the oxygen electrodes. This muddy sludge could possibly have reduced gas access to the back side of the electrodes. In addition, the corrosion and reaction of the electrolyte with the bipolar plates could consume electrolyte which also would result in poor performance. A chemical analysis of the sludge revealed heavy metal content was primarily nickel, copper and magnesium which confirmed the observations that the sludge resulted from the corrosion of the bipolar plates. In the plating of the magnesium plates, a copper flash is employed prior to the electroless nickel plate. The matrices from the unit were analyzed final KOH concentration and were found to be as follows:

No. 1 - 27 %
No. 2 - 26.35 %
No. 3 - 30.25 %
No. 4 - 32.75 %
No. 5 - 29.35 %
No. 6 - 26.9 %

The variation and also reduction in the electrolyte concentration observed by these tests indicates that the corrosion reaction resulted in some electrolyte consumption. To further evaluate the cause of deterioration, the oxygen American Cyanamid type electrodes were salvaged from the unit and reassembled in a new 6 cell unit with new matrices and hydrogen electrodes. This unit was put on test and on initial charge rose rapidly to 12 volts (2 volts per cell) and on discharge ran at 4 volts at 10 amps. The extremely poor performance of this 6 cell unit indicated that the oxygen electrodes had somehow been poisoned in the prior cell. This poisoning could have been as a result of the corrosion products of the bipolar plate. This poisoning will be further evaluated by removing the electrodes from the unit and testing them in single cells to confirm the results obtained and subjecting them to an acid wash to see if the poisoning can be removed and the electrodes recovered.

3.3 Potassium Titanate Analytical Tests

To determine if any reaction occurs between potassium titanate and potassium hydroxide, tests were conducted as follows:

A 0.8454 gram sample of potassium titanate was mixed with 15 cc of 40.15 % KOH and allowed to sit for a period of 90-1/3 hours at 80°C. The titanate was then filtered from the solution, washed, dried and weighed and a weight loss of 1.67 % was obtained. An analysis of the sample of the potassium hydroxide solution, after the test period, revealed the final potassium hydroxide concentration of 40 %. Both of the changes observed could be considered within experimental error. It therefore appears that under this type of test condition, there is no appreciable reaction taking place. A second sample of 0.9954 grams of potassium titanate was mixed in a 30 grams solution of molten 70 % KOH in a platinum crucible. The solution was maintained molten for a period of 27 hours. (No temperature measurement was made during the molten period for fear of contaminating the solution, but in order for 70 % KOH to be in a molten state, the temperature must exceed 228°C.) After the 27 hour test period, the solution was diluted and the potassium titanate filtered, washed, dried and weighed. The final weight indicated a weight loss of 5.6 % which is considered somewhat high for experimental error.

The above recovered sample was stored again in molten 70 % KOH in a platinum crucible, (greater than 228°C) for a period of 115 hours. After the test period, the solution was diluted and the potassium titanate filtered, washed, dried and weighed. The final weight indicated a weight loss of 11 % which is above what could be considered experimental error. However, this represents only a gradual rate of reaction under a very severe set of test conditions.

An additional test to further determine the corrosion reaction rate of potassium titanate with molten potassium hydroxide was conducted as follows. Three samples approximately 1 gram in weight of potassium titanate that had been water washed, dried and fired, were placed in platinum crucibles containing 30 grams of 70 % KOH in the molten state. The samples were maintained in the molten potassium hydroxide for periods of 115 hours for sample No. 1, 307 hours for sample No. 2, and 547 hours for sample No. 3. At the end of these periods, the samples were removed, washed free of electrolyte and weighed to obtain weight loss. The results of the three samples were 8.25 % loss, 7.5 % loss, and 8 % loss respectively. The relatively constant weight loss over this period, certainly within experimental error, indicate that there is a finite reaction which apparently occurs initially and does not proceed beyond that point. There is potentially a considerable experimental error in this test since in the washing process, it is possible to lose a certain amount of the material. However, these results associated with the results previously obtained and described above are extremely encouraging and indicate that potassium titanate is relatively inert in a potassium hydroxide environment.

Upon inquiry to the E. I. Du Pont Co., the following chemical composition of pigmentary potassium titanate was obtained:

T_1O_2	81 - 83.5 %
K_2O (Net)	14 - 15 %
K Cl	.5 % max.
K_2SO_4	2.0 % max.
Al_2O_3	.2 - .4%
H_2O	2.5 - 4.0 %
Fe	60 - 80 ppm
Pb	10 - 20 ppm
As	10 - 20 ppm

They indicated that the K Cl and K_2SO_4 are surface salts associated with the PKT processing and are removable by washing. Therefore, for further mats, a preliminary washing of the PKT will be included in the process procedure.

4. CONCLUSIONS

The use of potassium titanate matrices as a substitute for asbestos has enabled the improvement of cycle life performance and reduction in the rate of deterioration of the regenerative hydrogen oxygen fuel cell. Potassium titanate enables the achievement thus far of cycle life in the 300 to 500 range relatively reproducibly with slight to moderate rates of deterioration. The improvement in performances obtained by elimination of asbestos confirms that constituents in the abestos were the primary cause of the random irreproducible performance degradations and deteriorations previously observed. Additional modes of deterioration still exist in cells containing potassium titanate matrices. However, it has been necessary thus far to employ a limited amount of asbestos (10 %) in these matrices for structural reasons. Process techniques for potassium titanate asbestos mix matrices have been established. However, additional refinement and perfection of these matrices and the process techniques is still required. The use of American Cyanamid type or electro-deposited platinized nickel screen electrodes developed in this period enable the employment of an oxygen electrode that contains no sub-material that is readily oxidizable. This type of electrodes, therefore, does not result in differential pressure swings, as the cell switches from charge to discharge cycle as previouly obtained with porous nickel plaque type electrodes. The elimination of differential pressure problem improves the cell performance and reduces cross leakage problems. The platinized nickel screen type electrodes have performed relatively well and represent a promising approach for an electrode type to be used in the regenerative fuel cell. It appears that this type electrode and/or combinations of platinized Cyanamid electrodes will enable long cycle life and improve capacity due to less sensitivity to water variations in the matrix during charge and discharge as opposed to a straight water proof Cyanamid type electrode. The development of the potassium titanate matrices, which have demonstrated a relatively low rate of reaction with potassium hydroxide electrolytes, represents what could be a significant contribution that could be utilized in primary fuel cells and water electrolysis units of the alkaline type.

5. PLANS FOR THE NEXT PERIOD

Cycling single cell tests will be continued to evaluate potassium titanate matrices of different total weight, weight-ratio of electrolyte to matrices, thickness, compression ratio, and additions of asbestos, teflon and other fibers that can improve the structure of the matrix. Tests will be continued to determine reaction and corrosion rate of potassium titanate with potassium hydroxide. Analytical tests of matrices removed from cycling cells will be conducted to determine if platinum migrations exist within the matrix and if electrolyte reductions and consumption occur during cell cycling. In the cell tests hydrogen electrodes fabricated by platinizing carbonyl porous nickel plaques will be utilized in all cases. For the oxygen electrode American Cyanamid type modified platinized American Cyanamid type and platinized nickel screen types will be evaluated. Improved process techniques will be studied and instituted in the fabrication of the potassium titanate matrices and the platinized nickel screen type electrodes. Six cell units employing promising electrode matrix configurations will be assembled and subjected to cycle life testing to obtain additional experience and test data of multi cell units. When stable reproducible performance is obtained with six cell units, a cell configuration will be frozen and testing of 34 cell 600 watt unit will be initiated.

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