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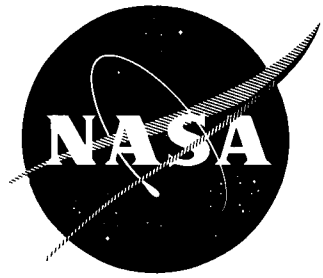

HYDROGEN-OXYGEN ELECTROLYTIC REGENERATIVE FUEL CELLS

by

M. Klein and E. Findl

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS3-2781

EOS ELECTRO-OPTICAL SYSTEMS, INC., PASADENA, CALIFORNIA
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QUARTERLY REPORT

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15 April 1965

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1. INTRODUCTION

This report reviews the progress made on development of a regenerative hydrogen-oxygen fuel cell, under NASA Contract 3-2781, during the period 1 Jan.--31 Mar. 1965. The program objective is the development of an electrically 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 both as an electrolyzer during charge periods, and as a fuel cell during discharge periods. Integral gas storage tanks are used to contain the hydrogen and oxygen gas generated during charge. Such a device offers advantages in the area of watt hours/lb., high ambient temperature operation and greater cycle life that can be obtained from existing secondary batteries. A two phase program is being conducted. Phase I consisted of the design, development and testing of a nominal 75 watt, 44 watt hour, 6 cell unit to demonstrate the feasibility of a multi-cell regenerative device. This phase has been completed. Phase II consists of the design and development of a 500-watt, 600-watt hour, 34 cell unit of minimum weight, for evaluation as a flight prototype.

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2. SUMMARY

During the period covered, major aspects of the design for the 500 watt multi-cell unit were completed and components are being fabricated. Single cell testing is being continued in order to obtain additional information on electrode, electrolyte and asbestos matrix structures. Life testing of single and multi-cell units were initiated. A single cell was cycled continuously for 205 cycles at which point the test was discontinued. A multi-cell, 75-watt unit is on continuous cycle and has exceeded 200 cycles at the writing of this report, with no degradation performance. The instrumentation and control panel for testing of the 500 watt unit has been designed, and fabricated.

3. TECHNICAL DISCUSSION

3.1 Single Cell Test

During this period, single cell tests of the type described in the previous semi-annual report were continued. Table 1 summarizes the single cell tests initiated and describes the cell constructions and results obtained. All of the single cell tests, with the exception of some of the high capacity tests, were conducted using the standard cycle, i.e., 65 minutes charge, 35 minutes of discharge; discharge current in the range of 15-17 amps., charge current in the range of 9-10 amps.

Cell # 44 was set up with the object of running an extended term cycling test to determine effects that might occur over a long period of cycling. Cycling was continued only during the working day, and the cell was allowed to sit at temperature over night at the pressure at which the cycle was stopped. Throughout the entire cycling period, calculations of ampere hour input vs. ampere hour output showed that the cell exhibited poor Faradaic efficiency, i.e., in the order of 75-80 percent. Aside from this deficiency, no other obvious deteriorations were observed throughout this test period.

After 41 cycles, it was decided to disassemble the cell and rebuild it with increased compression on the mat (to reduce Faradaic inefficiencies), and to install a pressure switch for charge cut-off so that the cell could be cycled continuously. An analysis of the mat taken from cell # 44 showed a final electrolyte concentration of 26.7% KOH. The cell had initially been assembled with 40.5% KOH. This was the first single cell subjected to extended cycling with 40% KOH, that did not contain viton rubber edging on the asbestos mat. It was thought that eliminating viton would eliminate the electrolyte deterioration problem, but there were apparently other deteriorating effects.

TABLE 1

SUMMARY OF SINGLE CELL TESTS

Cell #	O_2 Electrode #	O_2 Electrode Catalyst	H_2 Electrode #	H_2 Electrode Catalyst	Mat Thickness and Grade	Mat Dry Wt.	KOH %	KOH Wt.	Comments	Results
44	76	20 Mg Pt/cm^2	77	20 Mg Pt/cm^2	.050 Pure	22	40.5	29		Cell cycled 41 times. Final KOH 26.7% Exhibited poor Faradaic eff. throughout test. No obvious deterioration.
45	32	20 Mg Pt/cm^2	57	20 Mg.Pt/cm^2	.050 Pure	22	40.5	29		Cell used to balance and check out 2nd single cell.
46	72	20 Mg.Pt/cm^2	73	20 Mg.Pt/cm^2	.060 Pure	26.5	40.5	35	No screen behind electrodes Stnl.Steel Cell	Cycled 10 times Stopped test due to large differential pressure swings. Final KOH 34.3%
47	72	20 Mg Pt/cm^2	73	20 Mg.Pt/cm^2	.060 Pure	26.5	40.5	32	Stainless Steel Cell	Cycled 43 times Cell exhibited large differential pressures and fall off in voltage at end of discharge. Final KOH 37.8%.
48	88	20 Mg.Pt/cm^2	91	20 Mg Pt/cm^2	.060 Pure	26.5	40.5	31		Cell cycled 209 times Exhibited gradual degradation in performance between 100th and 200 cycle Cell shorted at 209th cycle.

TABLE 1 (con.)

49	92	10 Mg Pt/ cm^2 10 Mg Pd/ cm^2	96	20 Mg.Pt/ cm^2	.060 F.C.	26.6	40.5	31		Cycled 30 times Developed internal short due to gas mixing caused by leak in ΔP gauge.
50	89	"	100	"	.060 F.C.	26.5	40.5	35	Used no gas distrb. back-up screens	Cell had large differential pressure swings, voltage fell off at end of discharge, cycles two times.
51	103	"	95	"	.060 F.C.	26.4	40.5	31		Cycled 18 times, cell had gas mixing due to leak in ΔP gauge.
52	89	"	100	"	.060 F.C.	26.5	40.5	31	Cell tested at 150°C	Cycled 3 times external plumbing developed leaks due to high temperature.
53	96	20 Mg.Pt/	95	20 Mg.Pt/	.060 + .015	32.6	44.5	38.2	.045 span one back-up screen behind O_2 electrode	Cell dryed out and recombined at end of charge and voltage fell off at end of discharge. Final KOH 38.2%
54	96	"	95	"	"	32.75	40.7	38.4	"	" Final KOH 34%

Cell # 45 was assembled to check and balance a new single cell assembly. Cell # 46 utilized the new cell assembly, and was set up to test increased capacity variables in the mat and electrolyte arrangement. However, the cell exhibited extremely large differential pressure swings, and therefore was disassembled. Cell # 47 also assembled using the new single cell, was cycled 43 times. This cell also exhibited large differential pressure swings, plus a considerable fall-off in voltage at the end of discharge. Disassembly of the cell revealed that the stainless steel back-up plates behind the electrodes had oxidized and corroded considerably. Corrosion of the stainless steel was undoubtedly a contributing factor in the large differential pressure. Therefore, it was decided to have the back-up plates nickel plated to eliminate this problem in the future. The final KOH concentration in the mat after this test was found to have dropped from 40% to 37.8%. The variation between KOH concentrations in cells # 44 to # 47 is difficult to explain.

Cell # 48 was assembled with a pressure switch cut-off and was put on life test at the standard cycle. For the first few days, the cell was cycled only during the working day, and allowed to sit at temperature and pressure overnight. After sufficient confidence was built up in the cell that no leaks existed, the cell was cycled on a continuous 24 hour/day basis. Throughout the first 100 cycles, no deterioration in performance was noted. However, in subsequent cycles, there appears to be a slight deterioration in discharge voltage. Average voltage on discharge initially was 0.86 volts per cell. Discharge voltage gradually dropped to approximately 0.82 volts per cell at the 192th cycle. At the 194th cycle, the cell started to exhibit a drop off in voltage at the end of discharge. This tailing of the discharge voltage became more pronounced through the 209th cycle, at which time a sharp drop in voltage was recorded, indicating an internal short had developed within the cell. The cell voltage at different cycles is shown in Fig. 1. An examination of the internal

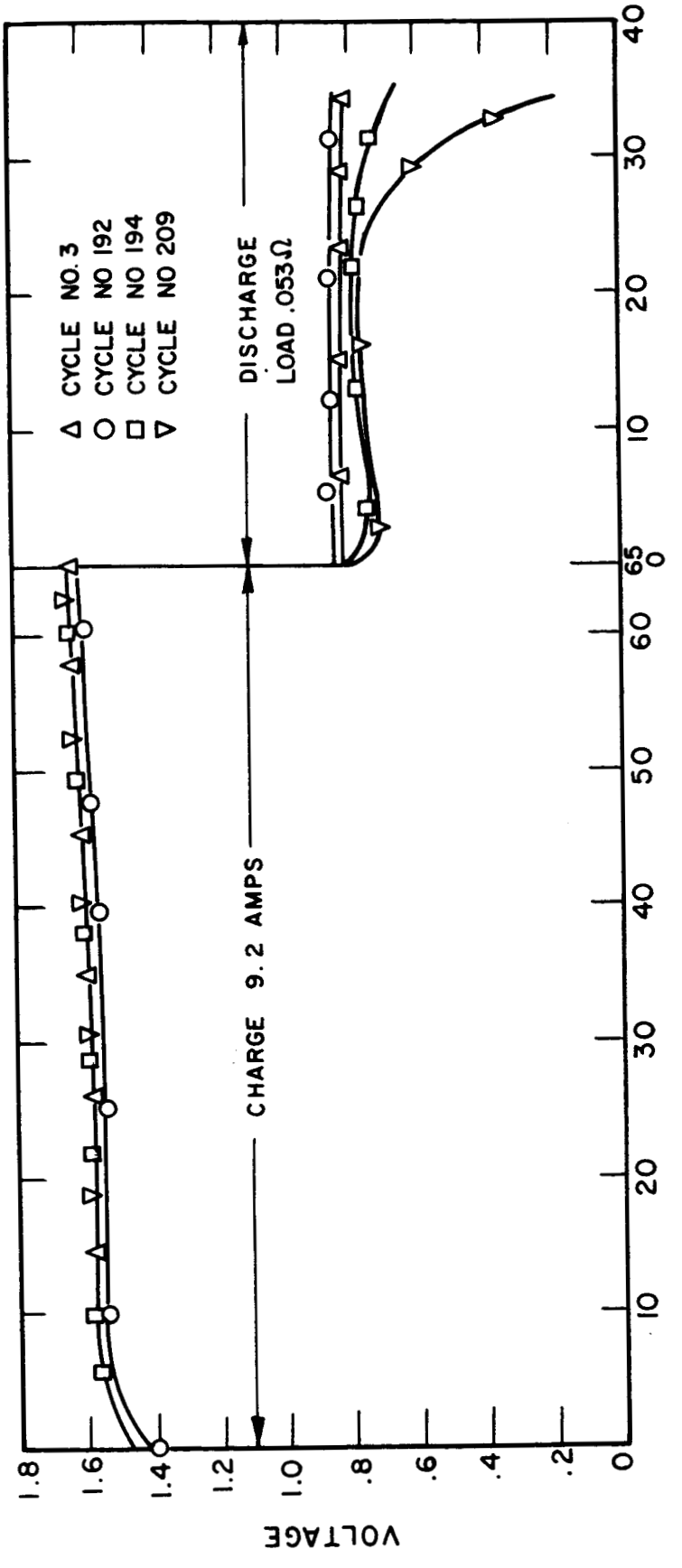


FIG. 1 PERFORMANCE OF HYDROGEN-OXYGEN REGENERATIVE CELL OVER PERIOD OF LONG TERM CYCLING

components of the cell revealed that the cell had probably undergone an internal chemical reaction. As a result of this reaction, the bolt connecting the backup plate to the O_2 tank behind the oxygen electrode was pushed forward against the oxygen electrode resulting in a mechanical short across the mat.

During the dissassembly process, it was noted that an appreciable quantity of water had condensed in the pressure gauge used to monitor H_2 and O_2 tank pressure differentials. The gauge, was kept external to the test oven during the test period. This test arrangement resulted in the condensation of water in the gauge over the long period of cycling. The cause of the chemical reaction can be attributed to gas mixing caused by large differential pressures and drying of the mat due to loss of water into the differential pressure gauge. The fall off in discharge voltage at the end of discharge in the latter cycles could also be attributed to gas mixing. (The large differential pressures obtained in single cells is virtually eliminated in the multicell units since the hydrogen and oxygen compartments are separated by a floating bellows.)

These long term cycle life results are considered very encouraging. The ability to cycle continuously to 200 cycles at complete depths is a considerable achievement, compared to existing battery data. The results indicate no extreme deteriorating effects take place during cycling and the difficulties encountered were caused by the test method.

Cell # 49 was similar in construction to Cell # 48 with the exception that the pressure gauges were assembled inside the oven. However, at the 30th discharge, the voltage fell off rapidly, and the cell apparently developed an internal short. Examination of the internal components did not reveal the location of the short. Water was noted within the oxygen compartment indicating a possibility of a chemical reaction. However, no charring or blackening of the surface was noted. The explanation for the cell difficulty was not initially apparent. Subsequently, on run # 51, it was found that a gas leak

had developed in the differential pressure gauge. This is believed to be the cause of gas mixing.

Cell # 50 was set up as a high capacity test to repeat the performance of Cell # 43 reported in reference 1. However, the cell exhibited very large differential pressures, and a rapid fall-off in voltage towards the end of discharge, so the test was discontinued.

Cell # 51 was a repeat of the construction of cells 48 and 49 for a cycle life test. This test also had the pressure gauges contained within the oven. On the 19th cycle, the voltage exhibited a sharp drop during a discharge portion of the cycle. Subsequently, attempts to recharge this cell were unsuccessful. The pressure within the cell would not build up during charge indicating internal leakage and recombination. The cell was disassembled and examined. It appeared that there had been a chemical reaction within the cell. Examination of the differential pressure gauge revealed a cross leak through the gauge. This resulted in gas mixing between the hydrogen-oxygen compartments and was the cause of the subsequent chemical reaction. Considering the difficulties encountered with the ΔP gauge, it has been decided to use pressure transducers as the differential pressure indication in further long term life cycling tests.

Cell # 52 was assembled to determine the effects of high temperature operation. The cell was cycled at 150°C for the three cycles, but exhibited a drift in differential pressure, indicating gas leakage. It was later found that the teflon insulating sleeving used in a conax fitting to electrically insulate the differential pressure gauge from the cell had relaxed and developed a leak. Tightening of the conax gland eliminated the leak for a short period of time. Aside from the leakage problem, it appeared that the cell performed satisfactorily from a mechanical standpoint, and no other deteriorations were apparent.

Electrical performance was far superior than previous tests with the cell charging at approximately 1.43 volts at 10.5 amps, and discharging at 0.84-0.89 volts at 17-18.5 amps. The data is shown in Figure 2. Due to the leakage in the external accessories, the test was discontinued.

Cell # 53 was an attempt at an increased capacity configuration. It consisted of a mat of one layer of 0.060" and one layer of 0.015" fuel cell asbestos. In addition, one back-up screen behind the hydrogen electrode was removed, and a 0.045" spacer was employed. Figure 3 shows typical performance of the cell. Voltage rose rapidly at the end of charge. The cell also exhibited internal recombination at the end of discharge, as noted by the slow rise in pressure as a function of amp.hr. input. On discharge, the cell exhibited a rapid fall-off in voltage towards the end of the cycle.

Cell # 54 was a repeat of the previous cell with the exception that the electrolyte concentration was changed from 44% to 40%. This cell also exhibited poor performance, i.e., high voltage at the end of charge, and low voltage at the end of discharge.

3.2 Multi-Cell Assembly and Testing

In order to obtain more information on multi-cell units, additional 6-cell, 75 watt units were assembled and subjected to test. Table 2 summarizes the construction variables and results obtained from tests of 75 watt units. For comparison units S/N 101 and 102 which were reported in reference 1 are included in the table. In this period difficulties were encountered in the assembly of the 6-cell unit due to leakage through the seals around the stack bolts. (A stat seal washer is used on the shank of the stack bolt to provide a seal between the hydrogen and oxygen compartments where the bolts go through the stack.) New bolts were used in assembly S/N 103 and were found to have undersized shanks, providing an inadequate seal along the shank. This leakage was determined in the standard check-out of the cell during assembly. Different bolts were used, in assembly S/N 104, and were found to have scratches and imperfections along the

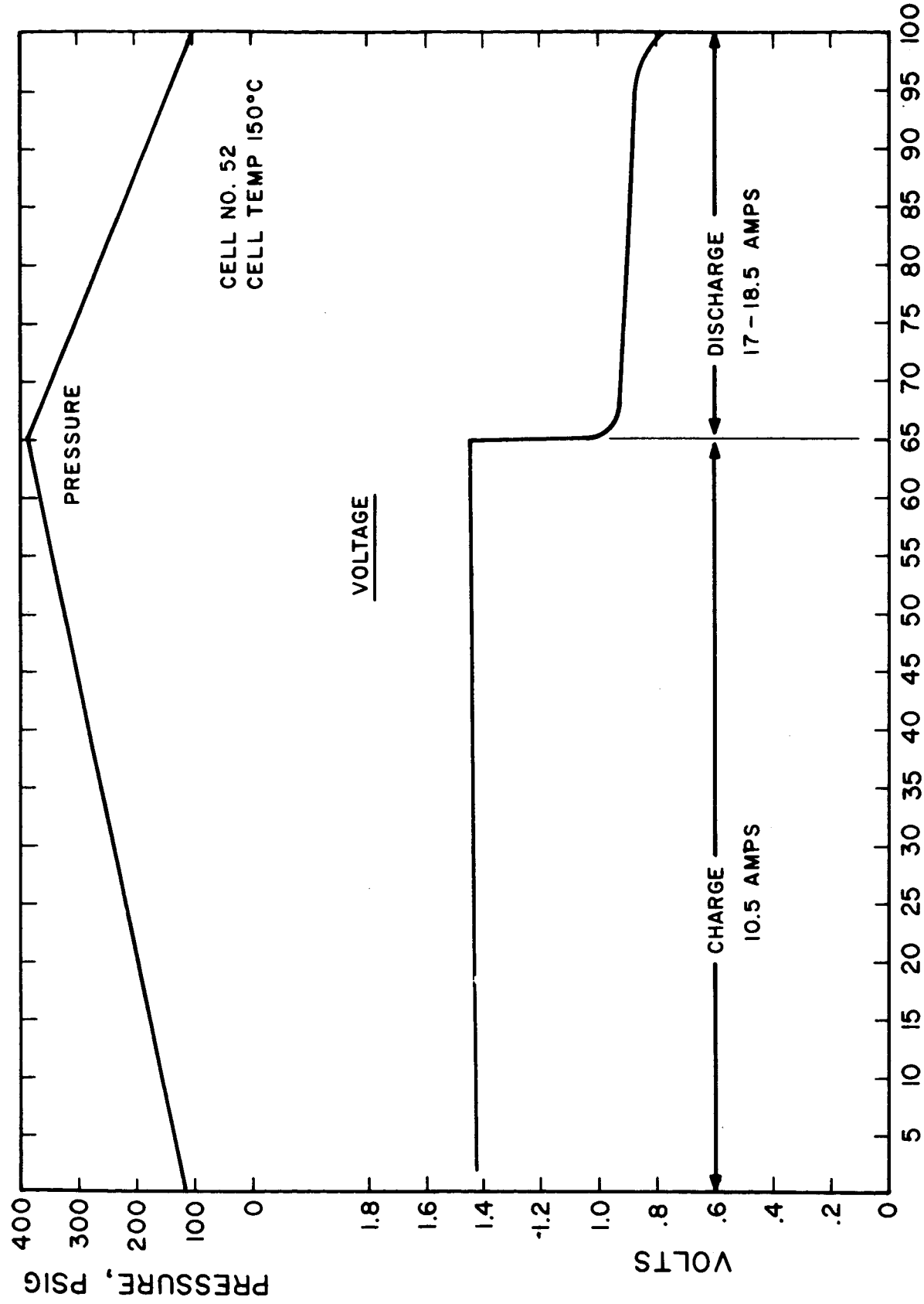


FIG. 2 HIGH TEMPERATURE TEST OF SINGLE CELL REGENERATIVE H_2-O_2 FUEL CELL

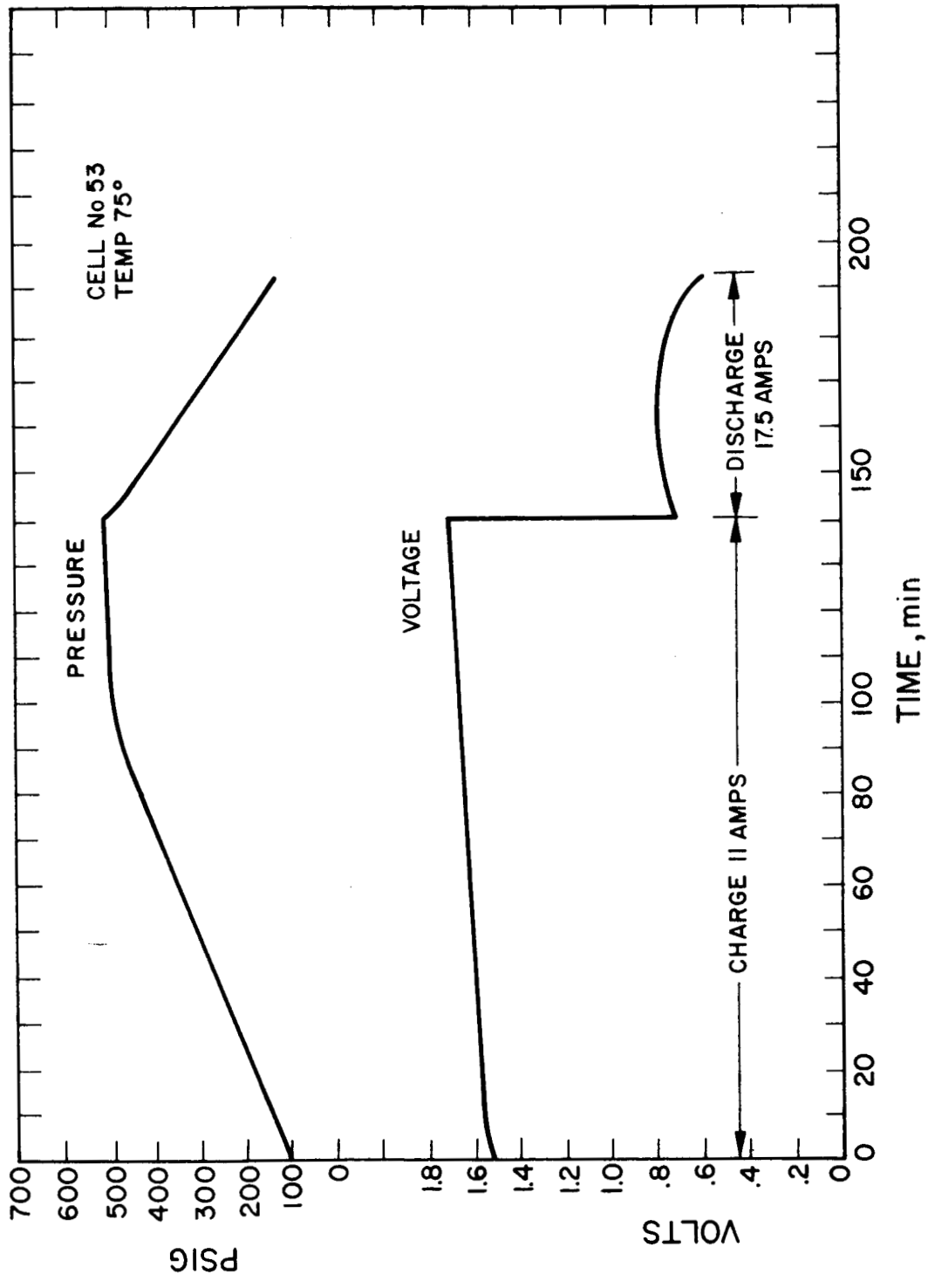


FIG. 3 SINGLE CELL HIGH CAPACITY TEST

TABLE II

SUMMARY OF TESTS ON 6 CELL NOMINAL 75 WATT UNITS

Construction							Results		
			Avg. Mat		Avg. KOH				
S/N	Catalyst O_2 Electrode	Catalyst H_2 Electrode	Dry Wt.	Thickness	Wt.	%	Comments	Total # cycles	Final KOH	
101	20 Mg Pt./ cm^2	20 Mg.Pt./ cm^2	22	.050	29	40.5	Viton rubber edging on Mats	39	13%	Exhibited gradual deterioration in charge and discharge voltage.
102	10 Mg. Pt./ cm^2 + 10 Mg.Pd/ cm^2	"	22	.050	29	40.5	No rubber edging stack bolts insulating washer glass epoxy	16	33-34%	Stack bolts had relaxed and cell could not hold differential pressure.
103	"	"	26.5	.060	31	40.5	Following units had poly.-prop. edging on mats, Bellville Springs on stack bolts	-	-	Found gas leakage through stat seals due to undersides bolt shank
104	"	"	26.5	.060	31	40.5		-	-	Found gas leakage through stat seal due to scratches on bolt shank.
105	"	"	26.5	.060	31	40.5		-	-	Unit developed two internal cell shorts on first charge.
106	"	"	26.5	.060	31	40.5		70	34.2, 34.4%	Developed Oxygen leak through pressure switch during cycling.
107	"	"	26.5	.060	31	40.5	Aluminum gas tanks.	210+		Still under test.

shank which also resulted in leakage through the stack bolts. Finally, a set of specially machined bolts, with ground shanks, had to be fabricated to eliminate this difficulty. After eliminating the leakage problem, the 6 cell unit (SN 105) was assembled, and installed in the test chamber.

About ten minutes after initially starting to charge the unit, a sharp drop in charge voltage of $\approx$ 1.5 volts was noted, followed shortly thereafter by another 1.5 volt drop. This decrease is indicative of cell shorting. The unit was therefore disassembled and inspected. Two cells in the center of the stack were found to be shorted, but the cause of the short could not be determined. An interesting sidelight noted in the disassembly was the fact that the four cells that did not short had asbestos mats that were impregnated with the normal black discoloration. The shorted cells were not discolored. This implies that some electrochemical phenomenon is causing the discoloration.

Cell S/N 106 was reassembled without any further problems, and subjected to test. The cell was cycled during the working day, and was allowed to sit at temperature and pressure over the night. A total of 70 cycles was accumulated. During the cycling period, there was a degradation in performance, as observed by an increase in charging voltage and a decrease in discharge voltage. Initial cell performance had an average discharge voltage of 4.9 volts and a charge voltage of 9.4 volts. At the 70th cycle, the performance had deteriorated to a point where the discharge voltage was 4.2--4.4 volts, and the charging voltage had increased in approximately 9.7 volts. This deterioration in performance is shown in Fig. 4 . In addition, the cell exhibited a sharp dip in voltage at the start and end of discharge. To determine if the fall off in performance was caused by gas mixing, gas samples from the hydrogen and oxygen compartments were taken at the end of the 70th discharge. Analysis for hydrogen in the oxygen sample and oxygen in the hydrogen sample by a gas chromatographic techniques, showed essentially no mixing had taken place.

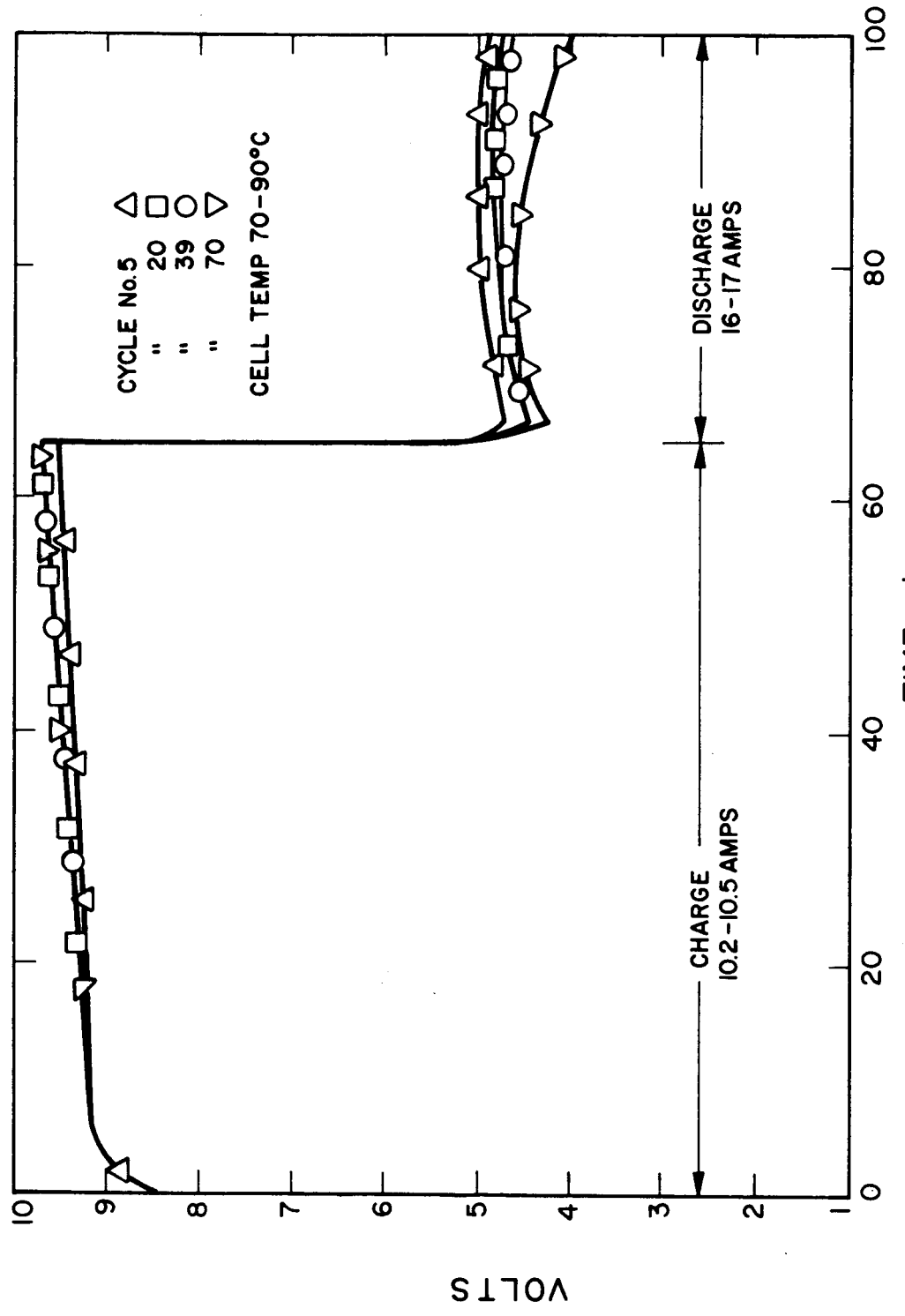


FIG. 4 PERFORMANCE OF S/N 106 6 CELL REGENERATIVE H_2O_2 FUEL CELL

Throughout the cycling, there was a gradual drift in the differential pressure towards an increase in hydrogen pressure, indicating a possible oxygen leak. On the 71st charge, an abrupt shift in the differential pressure was encountered. This was traced to a gross leak that had developed in the diaphragm of the pressure switch (located on the oxygen tank), which is used to shut off the charger when the cell comes up to pressure. Apparently, this diaphragm had had a pinhole which allowed oxygen to escape during the early cycles. The pressure switch contains a brass pressure sensitive diaphragm, which closes a set of contacts when the pressure acting on the diaphragm reaches a pre-set level. A 110 volt source is applied across the contacts of the pressure switch, and is used to activate a relay which shuts down the charger. Most probably, the hot oxygen that leaked into the pressure switch in conjunction with the arcing of the contacts increased deterioration of the diaphragm. The cell was at approximately 100 psi when the oxygen gas vented. However, the bellows and stack held the differential pressure that was created, and no other reactions took place. Considering the deteriorated performance, and the difficulties with the pressure switch, it was decided to discontinue the test and examine the cell stack.

Examination of the internal components of the multi-cell revealed moderate mechanical deterioration of the Pd/Pt oxygen electrodes. Analysis of samples of electrolyte obtained from two cell mats, show KOH concentration of 34.2 and 34.4%. As in the past, the asbestos mats were substantially discolored with a blackish-blue color adjacent to the hydrogen electrode. There was no free liquid in the gas storage tanks, and no discoloration of the tank walls. The deterioration in performance observed could have been the result of excessive handling of this particular cell assembly since it had been assembled, disassembled and reassembled a number of times due to the gas leakage problems around the stack bolts.

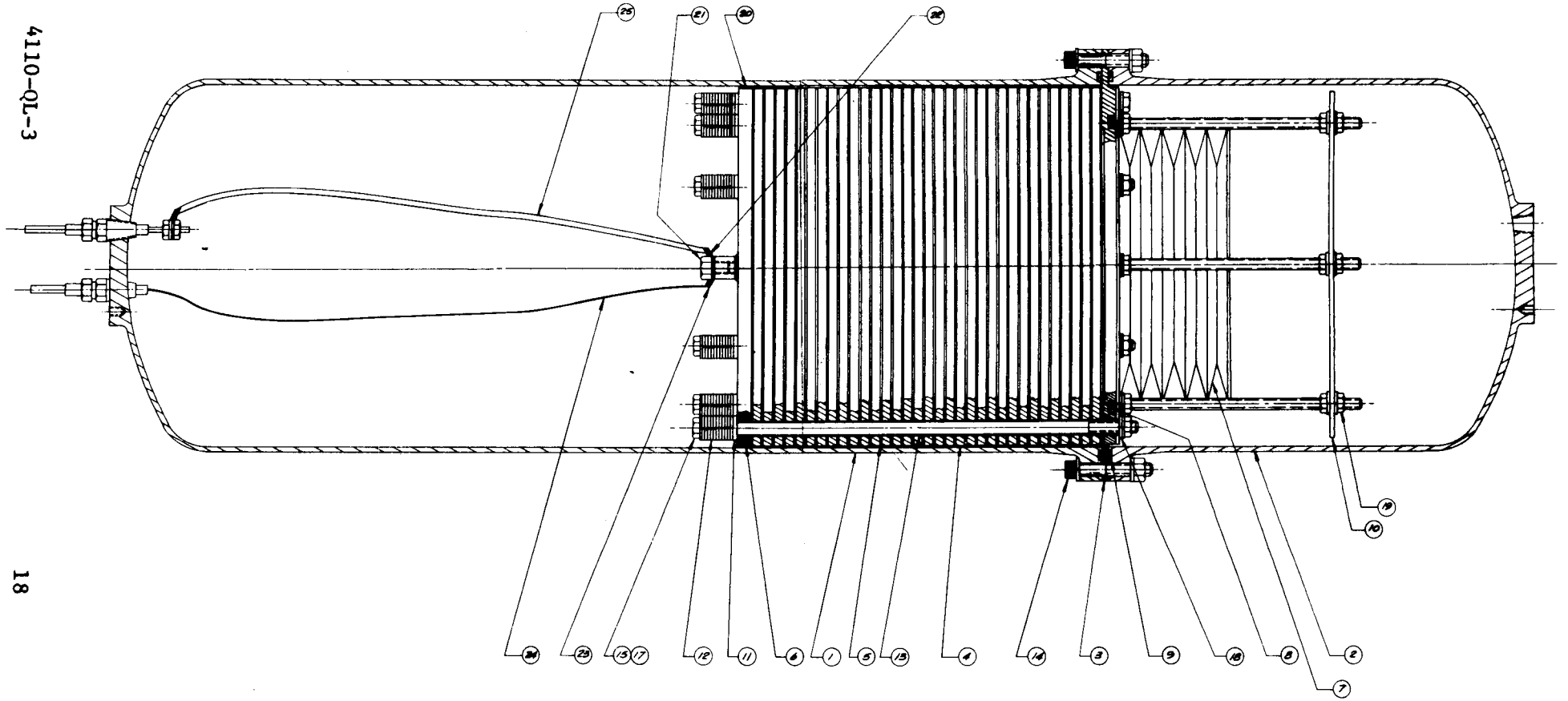
Considering this, an identical cell assembly (SN 107), was constructed and put on test. The only difference between this unit and the prior unit, was this unit was assembled in the nickel plated aluminum gas tanks. Cell # 107 was put on a standard cycling test of 65 minutes charge, 35 minutes discharge. It has at the time of this report been cycled 210 times, and no appreciable deterioration has been observed. The unit is being cycled continuously, day, night and over the week-ends. It will be cycled until cell performance shows signs of degradation.

3.3 Multi-Cell 500 W Design

The first prototype design for the 500 volt, 600 watt hour multi-cell unit has been completed. The design consists of 34 series connected cells employing the bipolar plate stack arrangement developed in Phase I of the program. Figure 5 shows the assembly drawing of the complete unit. The self-contained unit utilizes tanks that will contain the stored hydrogen and oxygen. The tanks are capable of storing sufficient gas to deliver 21.5 amp. hours. The design is based on a pressure cycle of 50-500 psig. Also included within the cell is a pressure compensating bellows to minimize differential pressures between the hydrogen and oxygen compartments. Orders have been placed for all components whose design can be frozen at this time, which include the tanks, bellows, bipolar plates, fasteners, etc. An aspect of the design that remains to be fixed is the mat electrolyte quantity and compression to deliver the optimum capacity. The first prototype tankage will be fabricated from stainless steel. The delivered prototype will incorporate nickel plated aluminum tanks of the same geometry as shown in Figure 5.

3.4 Instrumentation

A control and instrumentation unit was designed and fabricated for the 500 W cell. This unit consists of a constant current power supply (for charging the cell), a load bank for discharge, and cycling timers such that any cycle from 10 minutes to 26 hours can be programmed.



A/R	25		WIRE	#8		
A/R	24		I.C. THERMOCOUPLE WIRE			
1	23	MS25036-54	TERMINAL LUG	1/4		
2	22	MS20659-3	TERMINAL LUG	1/4		
1	21	MS35314-1	SCREW-HEX HD MACH.	1/4-28 X 3/8	ALUM.	
A/R	20		INSULATOR	5 MIL	TEFLON MESH	
30	19		NUT-PLAIN HEX	1/4 - 28	TITANIUM	
12	18		WASHER, INSULATING	.265 I.D.	FIBERGLASS	
74	17	AN960-D4L	WASHER, FLAT	.265 I.D.	ALUMINUM	
3	16	G13651-2	STACK BOLT	1/4-28 X 1 1/2	6061-T6 ALUM. AL	
9	15	G13651-1	STACK BOLT	1/4-28 X 9/16	6061-T6 ALUM. AL	
16	14		SCREW-ALLEN HD. MACH.	1/4-28 X 1-3/4	TITANIUM	
12	13	G13654	BOLT INSULATOR		TEFLON	

130	12	85 1/2-1/4-0.5	BELLEVILLE SPRING			
12	11	PARKER 200-130-1/4	STAT-O-SEAL	.242 I.D.	CRES.	
1	10	G13646	BELLOWS STOP	.101 X .1875 DIA.	6061-T6 ALUM. AL	
2	9	PARKER 2-267	TANK O-RING	8.234 I.D.	COMPOUND 77-565	
1	8	PARKER 2-361	END PLATE O-RING	5.975 I.D.	COMPOUND 77-565	
1	7	G13650	BELLOWS ASSY			
1	6	G13647	H_2 END PLATE		AE 318	
34	5	G13652	SEPARATOR		GLASS FILLED TEFLON	
33	4	G13645	BI-POLAR PLATE		AE 318	
1	3	G13591	O_2 END PLATE			
1	2	G13556-2	O_2 TANK		6061-T6	
1	1	G13556-1	H_2 TANK		6061-T6	

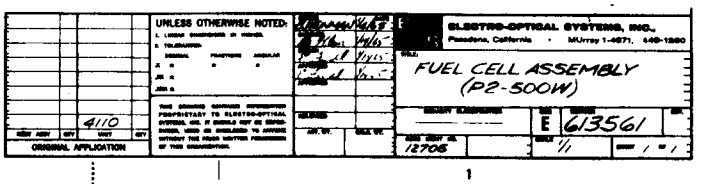


FIG. 5 FUEL CELL ASSEMBLY

In addition, the instrumentation unit contains pneumatic controls for filling and flushing the cell during start-up. For cell operation, an environmental control chamber was also fabricated that will enable heating and/or cooling of the cell as required. The instrumentation unit contains controls for adjusting the environmental chamber to the desired ambient. The unit is pictured in Fig. 6.

3.5 Electrode Preparation

The high porosity electrodes employed in the regenerative cell to date, have been prepared by manual application of the catalyst fluid to the basic electrode plaque. This procedure is both time consuming and subject to variations in quality. Since the 500 watt unit will require 78 electrodes per assembly, this manual process has been deemed unsuitable. Therefore, we are attempting to set up an automated process for electrode preparation. The process consists of a recycling flow system in which the catalyst solution is pulled, via vacuum, through the nickel plaques in controlled pulses.

To date electrodes made by the automated process have not been found to be suitable. The problem has been that the catalyst has been deposited as a loose surface coat that is mechanically unstable. Indications are that this is due to incorrect (too slow) cycle timing. A new cycle timer has been purchases. Preliminary tests, at higher cycle rates, indicate that high performance electrodes can be obtained.

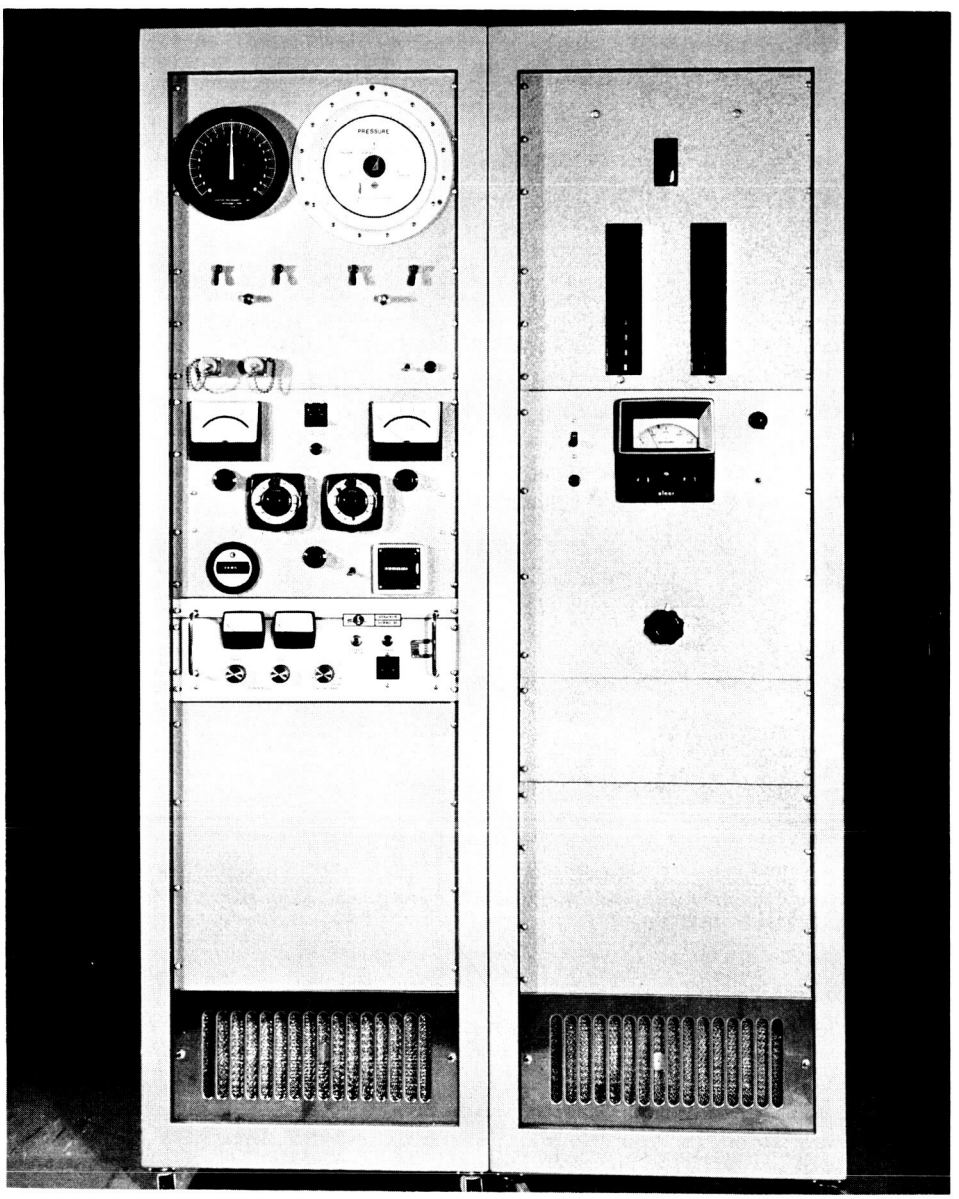


FIG. 6 CONTROL AND LOAD BANK FOR 500 WATT FUEL CELL

4. CONCLUSIONS

Both single and multiple cell tests have demonstrated that >200 full depth of discharge cycles can be attained. This represents a considerable improvement in performance and reliability. Major problems that have been noted in this report period are chiefly due to accessory and instrumentation difficulties. There appears to be no fundamental problem in achieving >500 cycles.

Additional effort will be required to further define the electrolyte and asbestos requirements for the attainment of >20 amp hour capacities.

5. PLANS FOR THE NEXT PERIOD

The six cell unit, SN 107, will be cycled until performance degrades. The cell will then be disassembled and a failure analysis made. Instrumentation for the six cell unit will be removed from the test area during the disassembly and replaced with the 34 cell unit instrumentation. Single cell testing will be continued with the objectives of attaining reliable, high performance operation at >20 amp. hour capacities. In addition, certain additional high temperature sterilization tests ($150^{\circ}C$) on single cells will be made to obtain pertinent operational data.

Fabrication of the 34 cell unit is scheduled for completion during April, 1965. It is anticipated that preliminary assembly and checkout of this unit will be conducted during the next quarter.

Additional tests and evaluations of the automated electrode process will be conducted. Single cell performance tests of randomly selected electrodes will be used to evaluate the electrodes produced.

REFERENCES

1. Klein, M., Findl, E., "Hydrogen-Oxygen Electrolytic Regenerative Fuel Cells" NASA Report CR-54279, Feb. 1965.