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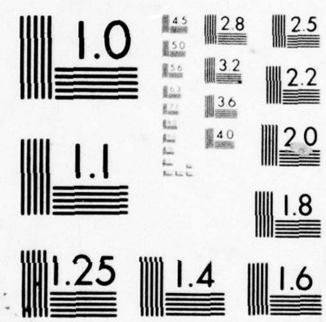

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FUEL CELL STACKS

FOURTH INTERIM TECHNICAL REPORT

FEBRUARY 1977

by

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and

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US ARMY MOBILITY EQUIPMENT
RESEARCH & DEVELOPMENT COMMAND
Fort Belvoir, VA 22060

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Production and testing of phosphoric acid fuel cell stacks is described. Electrodes, matrices, and bipolar gas distribution plates with an active area of 0.4 sq. ft. have been tested. Initial performance of 10- and 35-cell stacks has been obtained. Some stacks have been operated for over 2,000 hours at 300F, and tolerance to CO has been demonstrated. Life testing of stacks has been impeded by lack of complete gastightness and deterioration of gas diffusion plates.

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1. INTRODUCTION

The third interim technical report on this program described construction and testing of the first 15 10-cell stacks. The modifications undertaken in component manufacture, which were necessary because some critical raw materials became unavailable, were also described.

In the evaluation of 10-cell stacks constructed with the modified components, voltage decay and evidence of gas cross leakage were observed. Subsequent inspection of stacks indicated deterioration of the modified bipolar plate and edge seals. Further modifications in plate processing and stack assembly procedures were, therefore, undertaken. Minor modifications were also undertaken in the electrode and matrix processes; these were largely dictated by availability of vendor supplied materials.

The pilot plant capacity has been brought to a level considerably above that required to produce components for this program. However, only limited stockpiling has been undertaken pending determination of reasons for stack performance decay.

The present report describes component manufacturing and stack assembly processes, as well as stack test results obtained to date on this program.

2. COMPONENT MANUFACTURE

As part of the required effort on this program, we have developed a pilot production facility which, assuming minimal scrap, can produce components for the weekly assembly of 100 5 x 15" cells. However, the pilot plant has generally operated at only a fraction of this capacity. The major reasons for this have been

(1) difficulties in pinpointing the exact reasons for poor stack performance, and

(2) tightening of the dimensional tolerances for the bipolar plate resulting in a higher scrap rate.

2.1 Bipolar Plate

Since the Hercules H-resin has become unavailable, our efforts on bipolar plates have centered on the application of phenolformaldehyde (PF) molding resins in this component. This material was selected on the basis of its apparent acid resistance and high temperature stability.

2.1.1 Initial Testing of PF Resins

For initial evaluation, sections of bipolar plates molded with PF resins were immersed in H_3PO_4 at 350F for several weeks without any observable dimensional or chemical instability (the initial testing was conducted with resin concentrations in the range of 22 to 26% by weight). Plates were also molded for small (2 x 2") cell tests (all ERC fuel cell component modifications are initially evaluated in small cells). Several of these plates underwent many thousands of hours of testing without any evidence of deterioration.

Conductivity of plates molded with PF resins was found to be comparable or better than obtained with H-resin. With resin content of 22%, voltage drop thru the plate at 100ASF was measured to be in the order of 0.004V, certainly an acceptable figure in our application. These resins are also considerably less expensive than H-resin.

2.1.2 Evaluation of Graphites

In the early work with PF resins it became apparent that molding conditions developed for H-resin did not yield satisfactory plates with PF resins. The critical parameters for compression molding with graphite have been found to include:

- Mold temperature
- Molding pressure
- Top/bottom temperature differential
- Rate of mold closure
- Dwell time in mold
- Dwell time in mold before application of pressure (preheat)
- Type of graphite or mixture of graphites
- Preform density (precompression)

In addition to these, we have the mechanical requirements of uniform preform density (weight per unit area) and die parallelism in the mold.

In an effort to improve moldability, we turned to modifications in graphite composition. Molding art with these has long included mixing several types of graphite to improve moldability, i.e., defect-free discharge from the mold. In general, a quantity of fine graphite is mixed with a coarser one to provide improved density and strength.

In our work, a number of graphites were tested. A mixture which gives good strength and moldability with PF resin and has, for the present, been adopted as standard, consists of 11 parts of a coarse synthetic graphite (Asbury A-99, average particle size 50μ) and 4 parts of a fine natural graphite (Asbury 850, average particle size 0.6μ). This composition has been found to provide good moldability with resin concentrations over the range of 18 to 37% by weight.

2.1.3 Selection of PF Resins

All of the PF resins tested on this program were of the two-step, Novolac type. Materials included Resinox (Monsanto), Arofene (Ashland Chemical Co.) and Colloid Resin 8440 (Colloid Chemical). These resins are suitable for molding over the same temperature range that was employed for H-resin, and can be postcured in a few hours at 325-375F.

From information supplied by vendors, it appears that no significant chemical differences exist between the various materials employed. The major variables appear to be particle size and the amount of unreacted phenol remaining in the material as supplied.

Most of the work on the PF resins plates has been conducted with Ashland Arofene 890 (now replaced by Ashland with Arofene 889). More recently, Arofene 882, which contains even less unreacted phenol than Arofene 890, and Colloid 8440, which appears to have a finer particle size than the Arofene resins and gives somewhat better molding properties, have been evaluated.

Plates made with all of the above resins have shown similar mechanical, conductivity, and phosphoric acid resistance properties. The choice of the PF resins has, therefore, been mostly on the basis of moldability rather than final plate characteristics.

2.1.4 Evaluation of Composition

The initial work with PF resin plates was conducted with compositions containing 18 to 26% by weight resin. The main reason for experimentation over this range was the need to improve yield. Sections broken from these plates did not show signs of disintegration when immersed in H_3PO_4. However, in stacks built with these plates gradual performance degradation was observed.

Post mortem inspection of these stacks showed softening and swelling of plate corners around the fill hole. We believe this to be caused by lack of complete densification of the plate corners during molding because of the variable thickness of the plate (the mold comes to a stop on the completely densified web area, which is about 0.050" thick compared to 0.170" thickness in the corners). The acid is able to penetrate corners because of their porosity and to produce a reaction over the very large surface area of the powdered resin in the resin-graphite matrix.

The obvious approach for correcting the above condition appeared to be densification to the corners by adding more material in this area to the preform, i.e. to mold plates with a "shaped" preform. This approach was pursued with some success; the plate corners did in fact show good resistance to H_3PO_4 in beaker immersion tests. However, molding plates with consistently acceptable dimensions was found to be difficult.

Another workable approach to producing plates which are impervious to acid is the use of high resin content. Plates containing over 30% by weight of resin appear to form corners which are not penetrated by the acid. The limiting condition for this approach is, of course, the conductivity of the plate. We have measured conductance thru plates having 18 to 37% by weight resin. As shown in Table I, voltage drops over 20mV at 100ASF, are not reached with resin concentrations below 35%. The figures in this table were obtained by clamping a 2 x 2 in. section of the plate between flat electrodes at 80 psi and measuring the voltage drop thru the plate under a direct current of 100 amperes/square foot. Contact to the plate was made with stackpole graphite electrode support paper.

We are currently molding plates containing 33% resin. These plates have been evaluated in stacks without any evidence of deterioration over several hundred hours of operation. The stacks have exhibited somewhat steeper voltage-current slopes than we have seen with lower resin contents. (4.5 vs. 3mV/A). It is not clear at the present time if the somewhat higher resistance of the plate contributes significantly to the higher apparent cell resistance.

2.2 Matrix

Production of Kynol fibers has been discontinued by American Kynol Corporation, a subsidiary of Carborundum Company. A similar material is offered by Nippon Kynol who is now the sole source for the blown phenolic fiber. Consequently, all of our matrices are now produced from the Japanese fibers. To date, we have received two 50-pound shipments of this material with no apparent differences in the quality (the material appears to be from the same lot). The fibers supplied by Nippon Kynol are of good quality with little "shot" (unblown resin). This has obviated the need for hand picking of the material to eliminate lumps, which was required with the material supplied by American Kynol.

The matrix manufacturing process has been continued essentially as described in the Third Interim Report. Minor variations in weight per unit area and final matrix thickness have been made mainly as an effort to improve stack sealing. The matrix density was also shown to have an effect on operating voltage of the cells (see Third Interim Report).

Matrix variations are shown in Table II. We do not see any evidence that there has been a major difference in stack performance between the various matrices.

TABLE I

VOLTAGE DROP THRU PLATE

Contact Pressure: 75 lbs sq/in.
Current: 2.7A DC (100ASF)

PLATE NO.	% RESIN	VOLTAGE DROP, mV
558	18	0.004
1641	22	0.006
2569	27	0.008
2491	33	0.018
2285	37	0.032

TABLE II

MATRIX SPECIFICATION

	CODE	MAT'L WT., G/2.2 SQ.FT. FIBER	RESIN	THICKNESS, in.	% POROSITY (CALCULATED)
A.	2 x 24/17	28	1.2	0.017	55
B.	3 x 24/24	42	1.2	0.024	67
D	3 x 48/17	42	2.4	0.017	52
E.	3 x 48/24	42	2.4	0.024	66
H.	3 x 48/20	42	2.4	0.020	60

2.3 Electrodes

The electrode process as described in the Third Interim Report has been continued. Recently, a support material from Stackpole Carbon Company has replaced the Union Carbide material previously used. The reason for this change was inability to secure a continuous supply of material from Union Carbide with uniform properties. After evaluation in small cell tests, the Stackpole material appeared to provide performance equal to that with the Union Carbide backing. Since the Stackpole material has been available in uniform quality and also produces stronger electrodes for easier handling, it has, for the present, become the electrode support of choice. A comparison of the two electrode support materials is shown in Table III.

3.0 STACK MANUFACTURE

Since the Third Interim Report, 28 stacks have been built and tested on this program. Of these, 5 were 35-cell stacks, and 23 were 10-cell stacks.

Most stacks in this series were built with 0.024 in. thick Kynol matrices, bipolar plates containing 20 - 26% PF resin, and standard electrodes, (see Section 2.3). Minor modifications to the assembly were incorporated in some stacks: these included a 1/4 in. wide, 0.005 in. thick tantalum shim over the anode ribs at the edges of the plate to prevent collapse of the electrode support into the anode grooves and to improve matrix compression over this area. This modification was used in all stacks from Build No.39 on.

Teflon film inserts 0.002 in. thick, were used over the seal areas of the air side of the plate in order to allow easier separation of cells should it become necessary to replace individual cells in a stack because of poor cell performance. This approach has, in fact, found to be workable, and individual cells were replaced successfully in several stacks without losing stack performance or gas tightness. The Teflon inserts were employed in all stacks from Build No. 34 on.

The electrodes, matrices, and plates were assembled in the conventional bipolar stack fashion as shown in Figure 1. Viton cement was applied in about 1/4" width around the periphery of the matrix to facilitate sealing. Component dimensions and stack test hardware remained unchanged since the previous progress reports (see pp. 11 - 15, Second Semi-Annual Report). The stack compression force existed by the tierods on the endplates was 8,000 lbs for both the 10- and the 35- cell stacks.

Two ten-cell stacks (nos. 34 and 37) were also built with 1 in. thick honeycomb end panels. These lightweight panels had phenolic core and skins with a 1/2" deep solid phenolic border for manifold bolt holes. These panels showed bowing during operation of the stacks possibly contributing to gas cross leak condition at the cell edges (there is no measurable bowing with the

TABLE III

SUPPORT MATERIALS

	STACKPOLE	UNION CARBIDE
Thickness, typical (in.)	.018	.017
Thickness range (in.)	.016 - .020	.015 - .019
Weight typical (g/sq.ft.)	11.1	7.63
Weight range (g/sq.ft.)	10.6 - 13.5	5.7 - 7.9
Porosity (%)*	84.6	88.8

*Calculated from graphite density of 1.7 g/cm^3

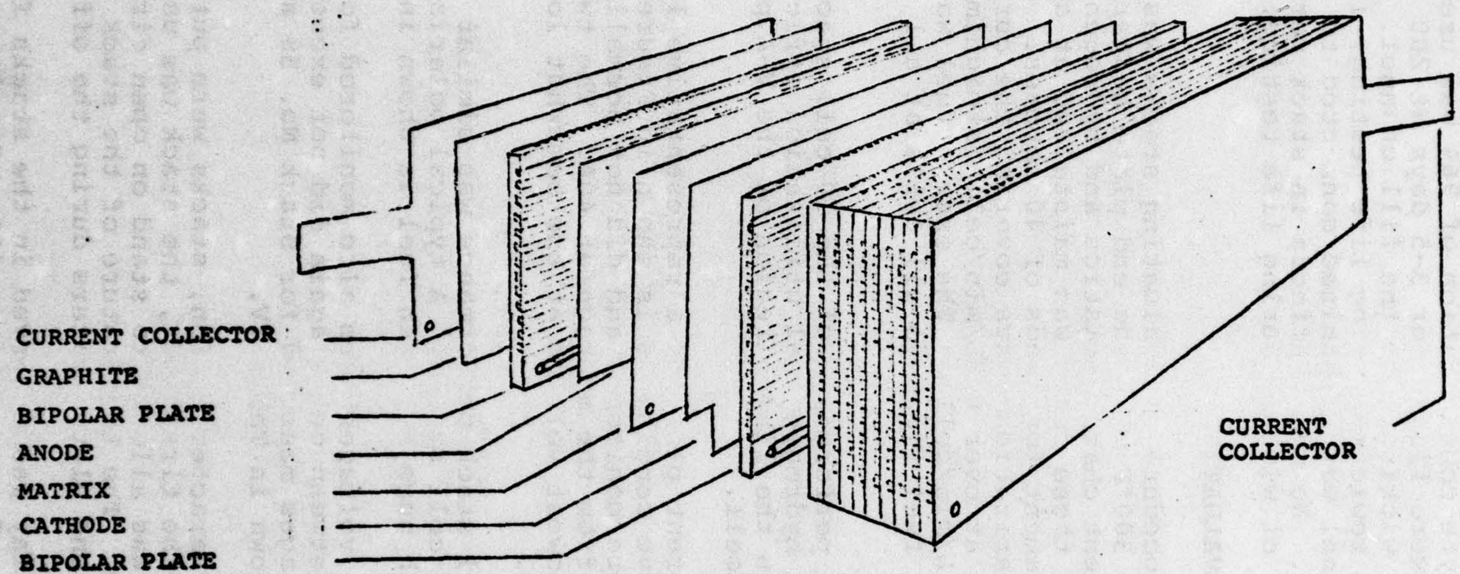

FIGURE 1 STACK ASSEMBLY

3/4 in. solid steel endpanels)

A fill electrolyte concentration of 96% was used for all stacks. The stacks were filled for 3-5 days at 200 to 250°F in the usual fashion by wicking from the fill channel. Occasionally, the stacks were also rewicked during life testing; in this case, the electrolyte channel was maintained connected to a fill bottle containing 96% H_3PO_4. No major effects in stack performance were observed as a result of wicking during life testing.

4.0 STACK EVALUATION

The standard procedure for evaluating stacks was to bring the stack temperature to 300°F with the end plate heaters and to determine voltage-current characteristics and hydrogen utilization at 40A. Air flow in these tests was maintained at about 10 times stoichimetric requirement for loads of 40 amperes. Fuel flow to the stack during polarization tests covering the current range of 5-80A was maintained at over 0.8L/min/cell (stoichimetric requirement at 80A is 0.62 L/min/cell). The excess fuel was used to insure that sufficient fuel flows to all cells at all current densities.

Typical initial performance of the 10 cell stacks at 300°F was 6.0V at 40A with hydrogen fuel utilization efficiency over 90%. Polarization in the linear region of the V-A plot was typically about 3 mV/A/cell.

The voltage current plot for a representative 10 cell stack tested under the above conditions is shown in Figure 2. Individual cell voltages were monitored and did not normally show deviations of over 30 mV from the average at 40A. The two end cells usually showed the lowest voltage at the somewhat lower temperature for these cells.

Thirty-five cell stack performance was similar (on a per cell basis) to that of 10 cell stacks. A typical polarization curve obtained at 300F with pure hydrogen fuel is shown in Figure 3.

Individual cell voltages were also monitored for the 35 cell stack. Variations between cells again did not exceed 30mV at 40A. Individual cell voltages measured for Stack No. 58 at 300°F with a load of 40A are shown in Table IV.

After initial characterization, stacks were put on load at 40 amperes. During the first week, the stack was usually operated for 8 hours/day and was allowed to stand on open circuit for the remainder of the day. The temperature of the stack was maintained at 250-300°F by the end plate heaters during the off-load period.

Load voltage decay was observed in the stacks following the first 10 to 50 hours of operation at 40A. Simultaneously, hydrogen utilization in the stack decreased and some cells began to

FIGURE 2 TEN-CELL STACK PERFORMANCE

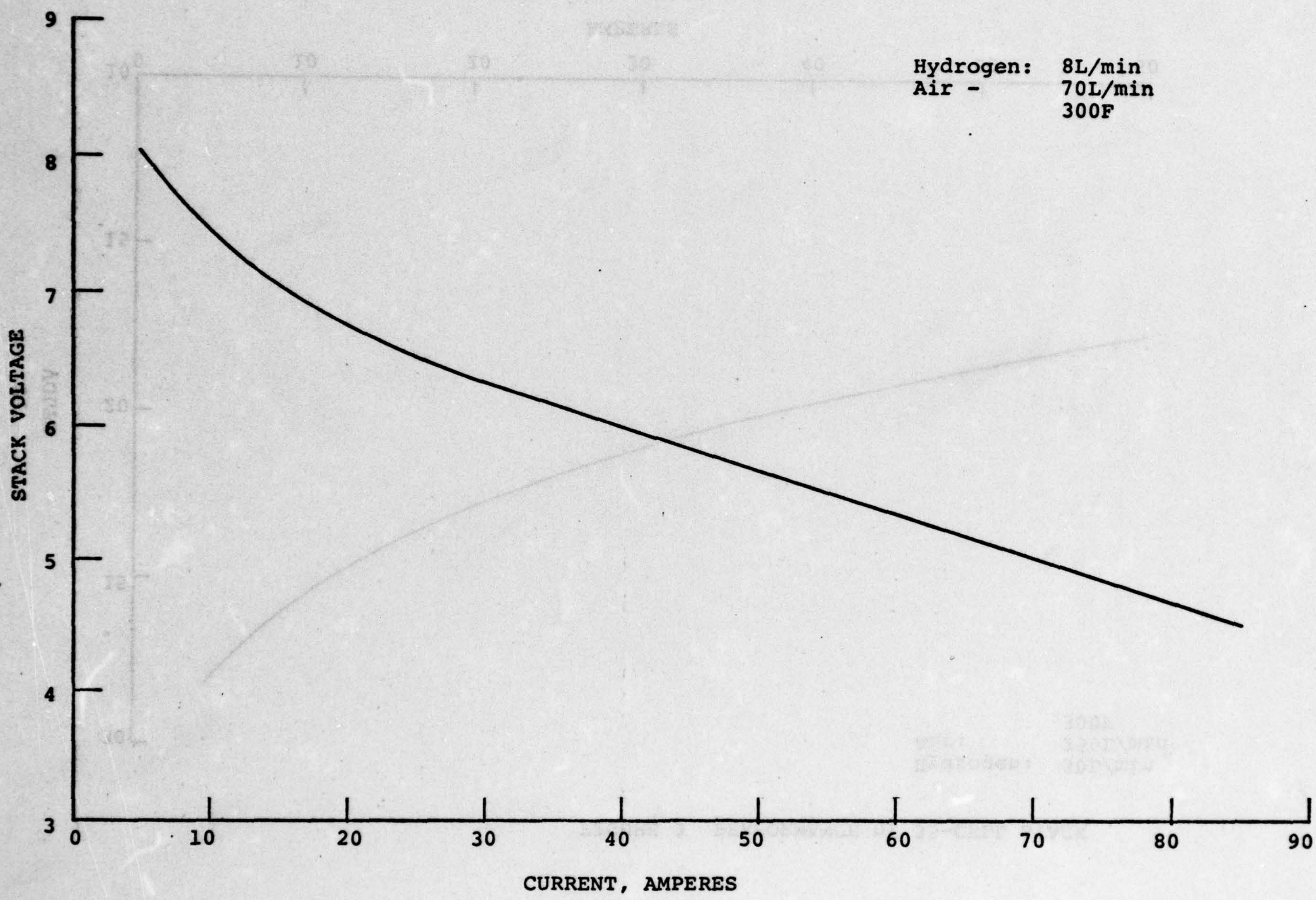

FIGURE 3 PERFORMANCE of 35-CELL STACK

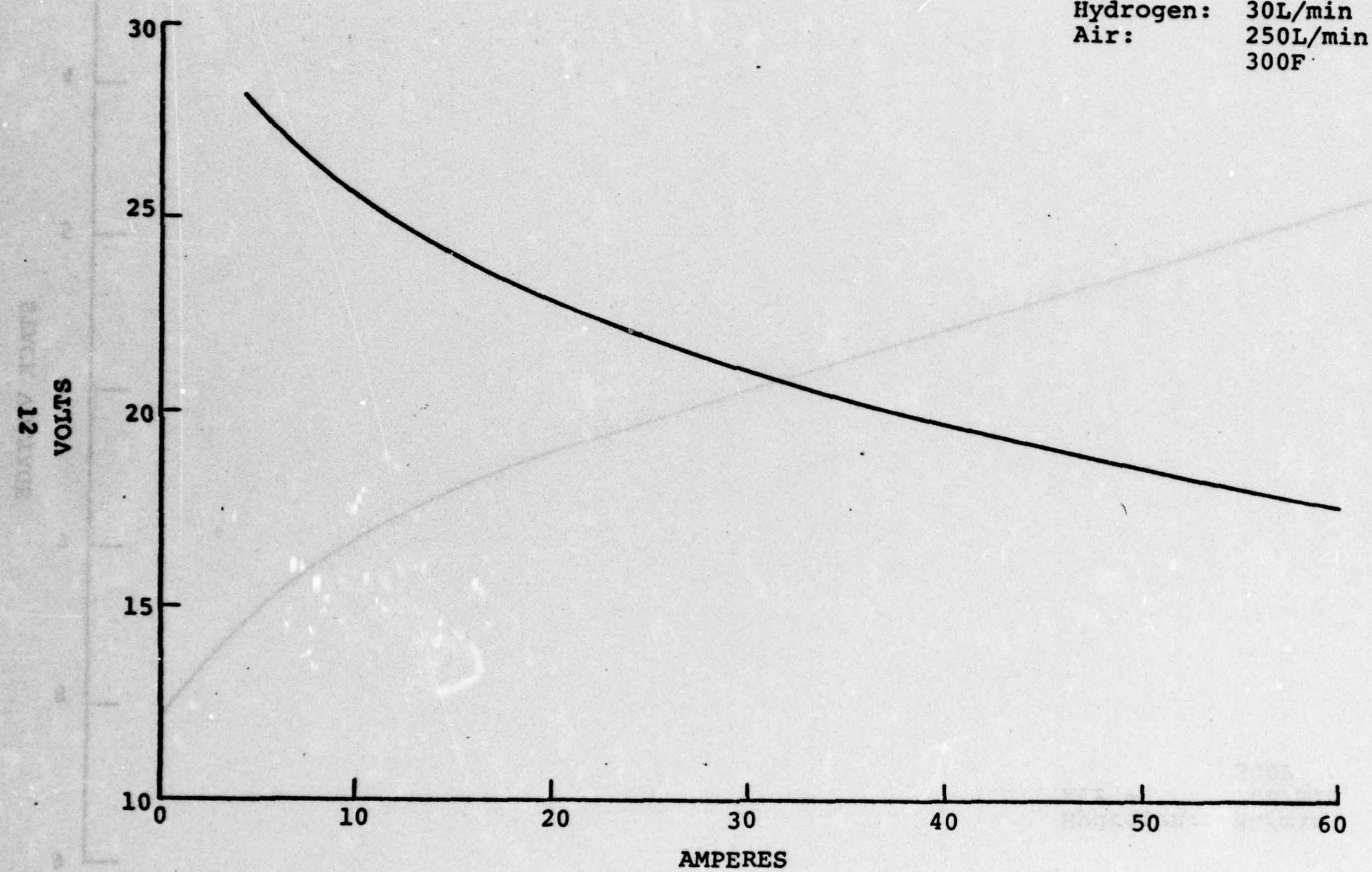

TABLE IV

CELL LOAD POTENTIALS, 35-CELL STACK

LOAD: 40A
TEMPERATURE: 320F
FUEL: Hydrogen

CELL NO.	VOLTAGE	CELL NO.	VOLTAGE
1	0.55	18	0.61
2	0.59	19	0.61
3	0.58	20	0.60
4	0.58		
5	0.59	21	0.61
		22	0.61
6	0.59	23	0.61
7	0.59	24	0.60
8	0.60	25	0.61
9	0.60		
10	0.61	26	0.61
		27	0.61
11	0.61	28	0.62
12	0.60	29	0.59
13	0.60	30	0.61
14	0.62		
15	0.60	31	0.60
		32	0.59
16	0.60	33	0.60
17	0.61	34	0.59
		35	0.57

exhibit variation in load voltage with full flow rate variations. This type of stack behavior is typical of a gas cross leak condition.

Disassembly and inspection of the stacks at the conclusion of testing at 40A revealed the plate corner condition described in Section 2.1.4. This appears to have been the major cause of stack degradation. Other degradation mechanisms, such as edge seal leaks, may also have contributed to performance decay.

In order to gain life data with the present components, one of the 10 cell stacks was continued on 40A load even after it had become evident that some gas cross leakage was occuring in the first four cells.* In order to maintain the performance at over 5.5 volts in this stack, hydrogen flow rates in excess of 150% of stoichimetric were generally employed. This stack had accumulated over 2500 hours by the end of this reporting period, with some decay in load voltage in the four cells in which gas cross-leakage was indicated, but with little decay in the remaining six cells (Figure 4). We believe that this data indicates a basic capability for all component materials employed in this stack to perform adequately over several thousand hours.

Four 10-cell stacks were delivered to MERADCOM as part of the contractual requirement. Voltage current data taken for these stacks before delivery are presented in Figures 5 - 8.

Stack assembly and test data for all 10- and 35-cell stacks built during this period are summarized in Tables V and VI.

5.0 CONCLUSIONS AND RECOMMENDATIONS

It appears at the present time that the difficulties encountered with stack performance are associated with deterioration of the bipolar plates, and with lack of gas tightness of both the intercell and manifolds seals.

Steps are now being taken to correct the plate degradation condition thru composition and process modifications. It does appear that the PF resins now being used will be suitable in this application if plate porosity can be eliminated.

A need to further study the sealing methods is indicated. This will be done by building and testing short (2-3 cells) stacks. Assembly of the remaining 10- and 35-cell stacks will be resumed upon development of a reliable sealing technique.

* A numbering convention has been established at ERC whereby cells are numbered starting at the anode (negative) side of the stack.

FIGURE 4 POLARIZATION TEST, STACK 17 (CELLS 5-10)

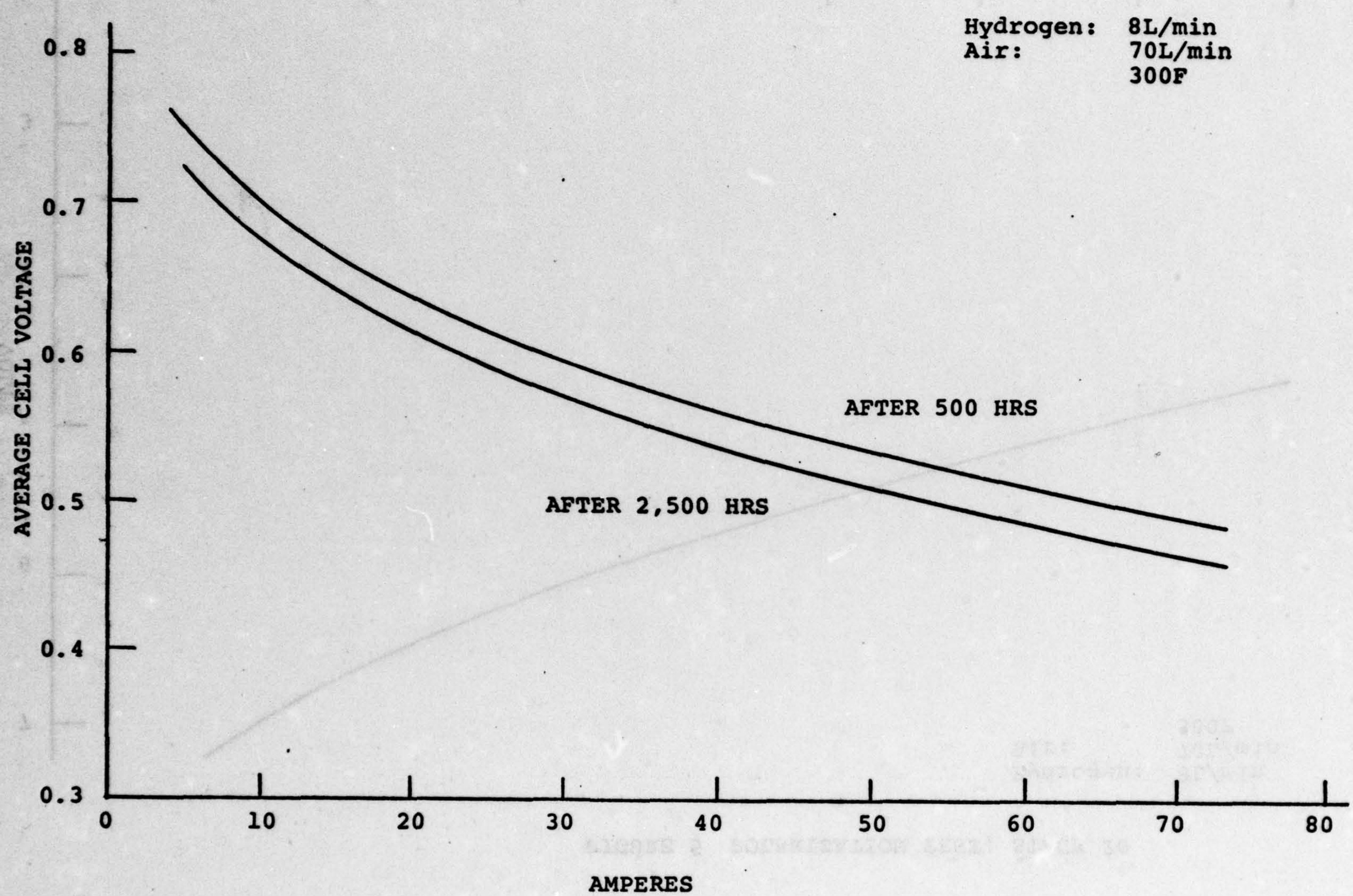

FIGURE 5 POLARIZATION TEST, STACK 20

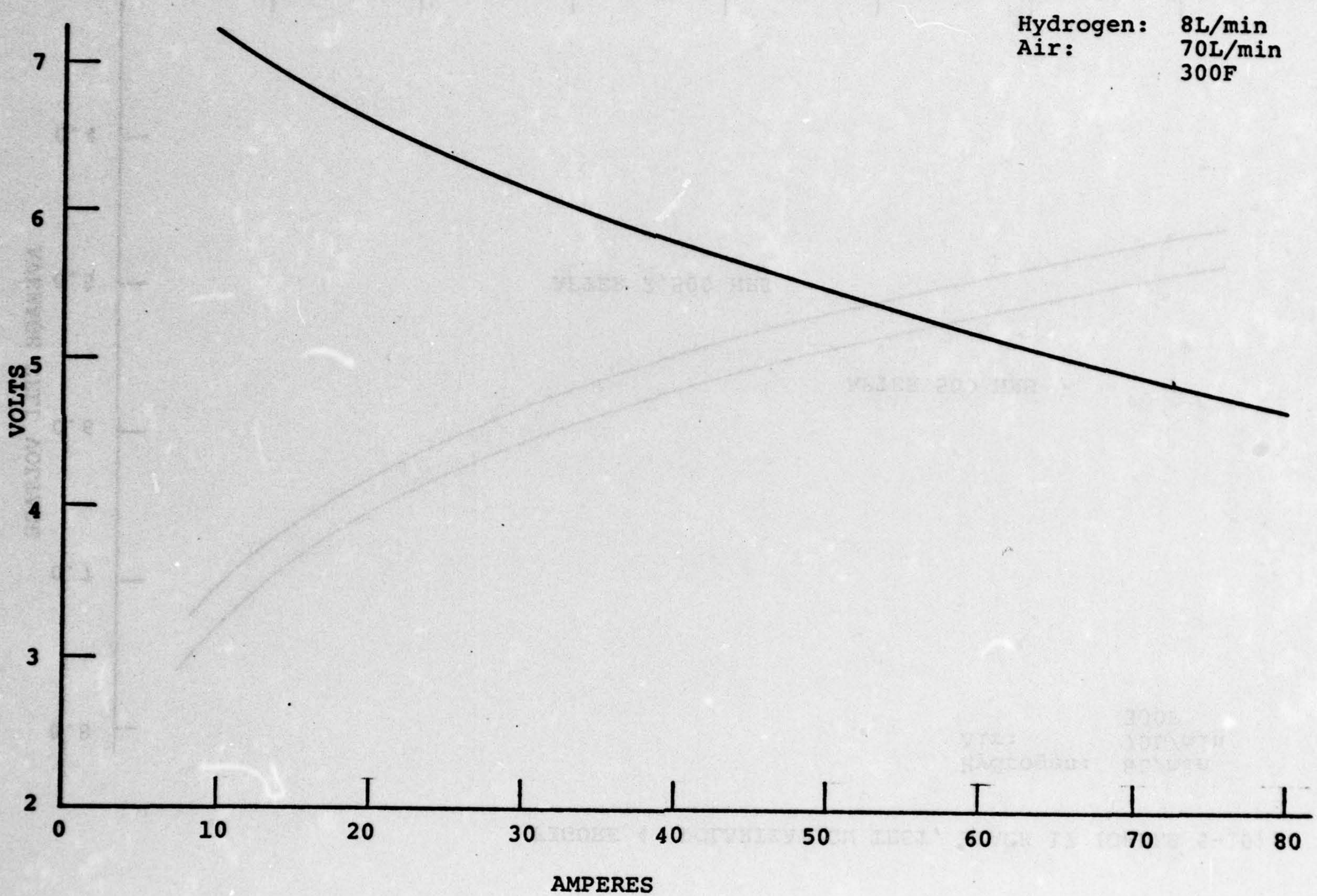

FIGURE 6 POLARIZATION TEST, STACK 25

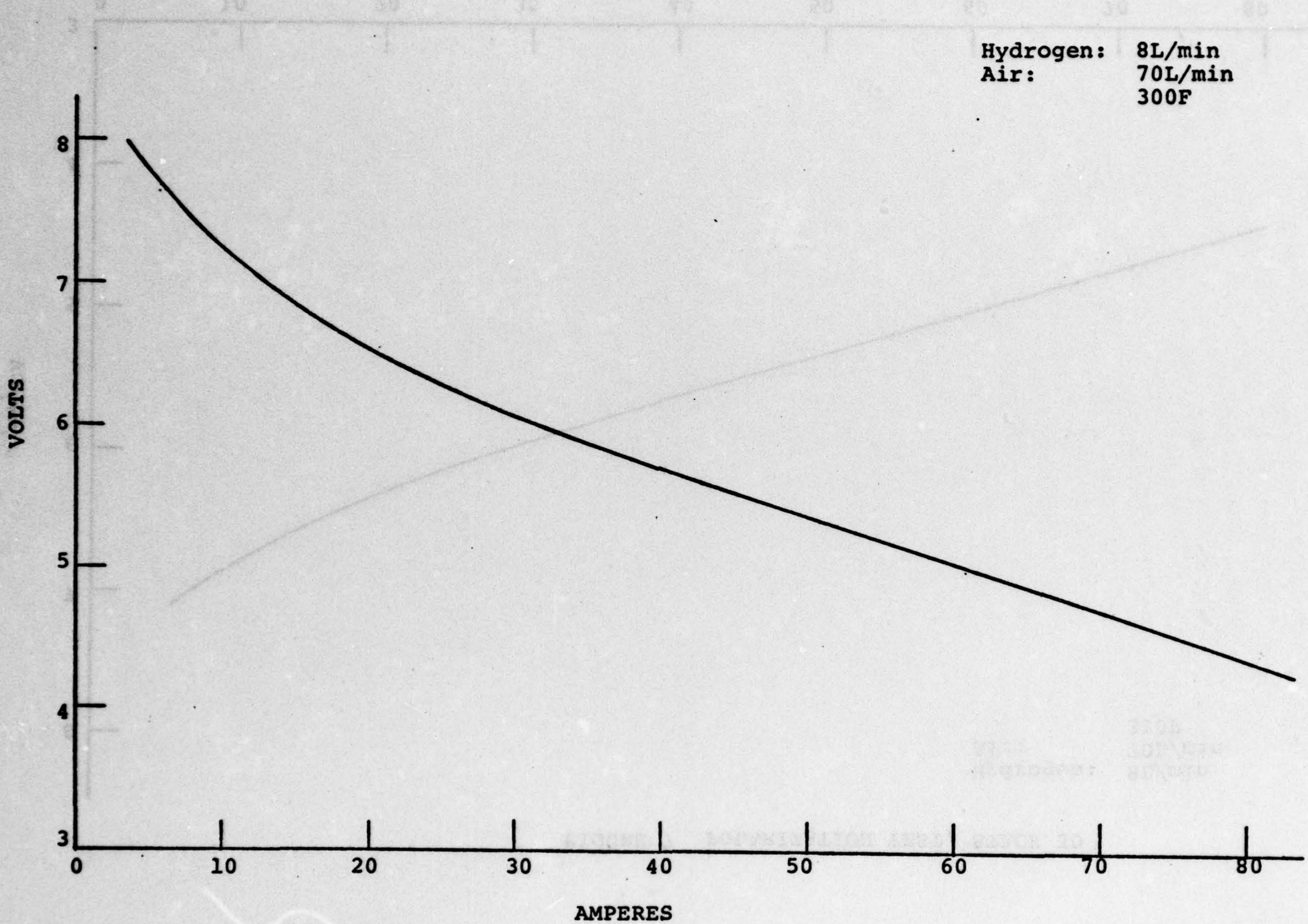

FIGURE 7 POLARIZATION TEST, STACK 30

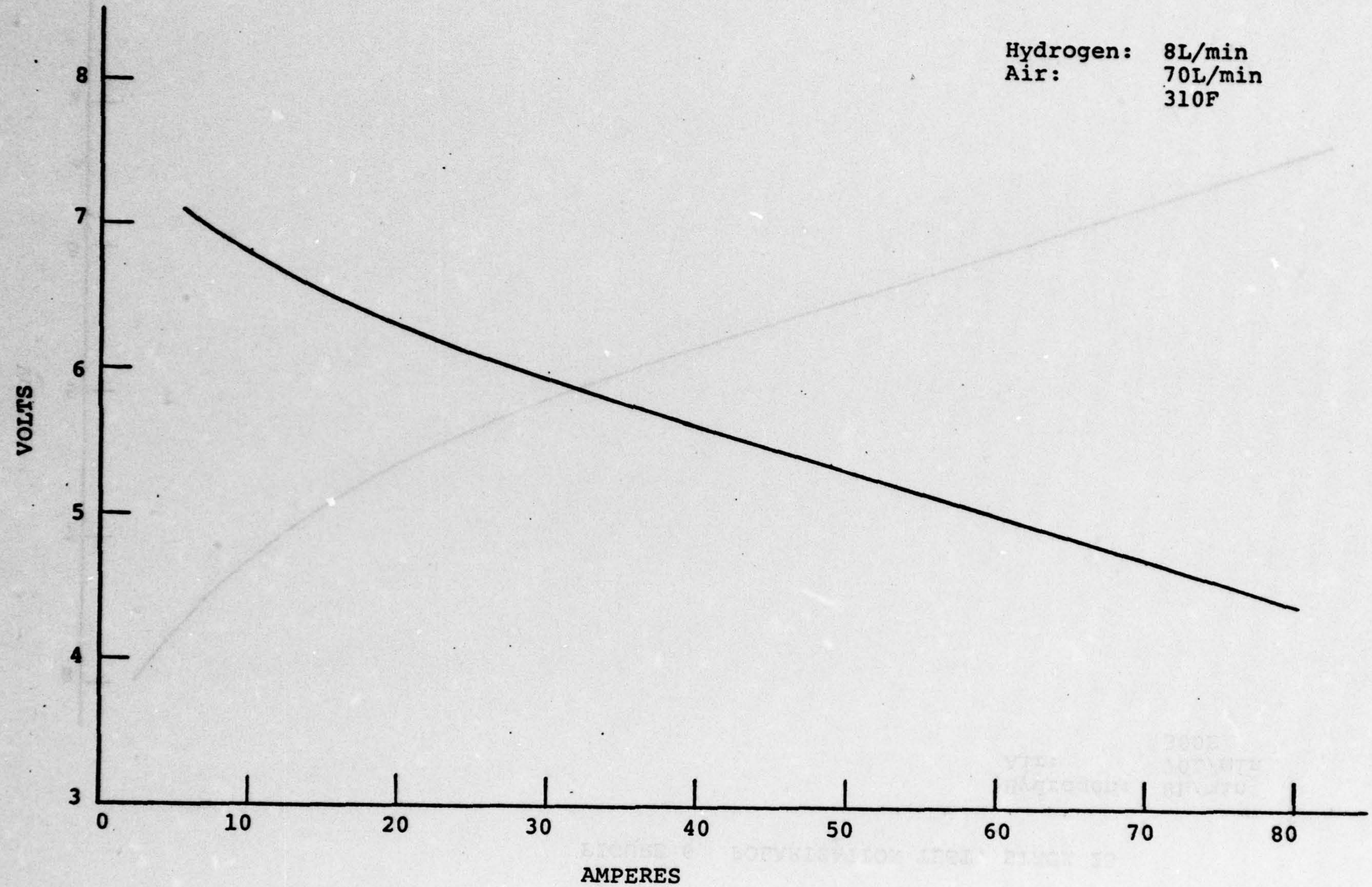

FIGURE 8 POLARIZATION TEST, STACK 35

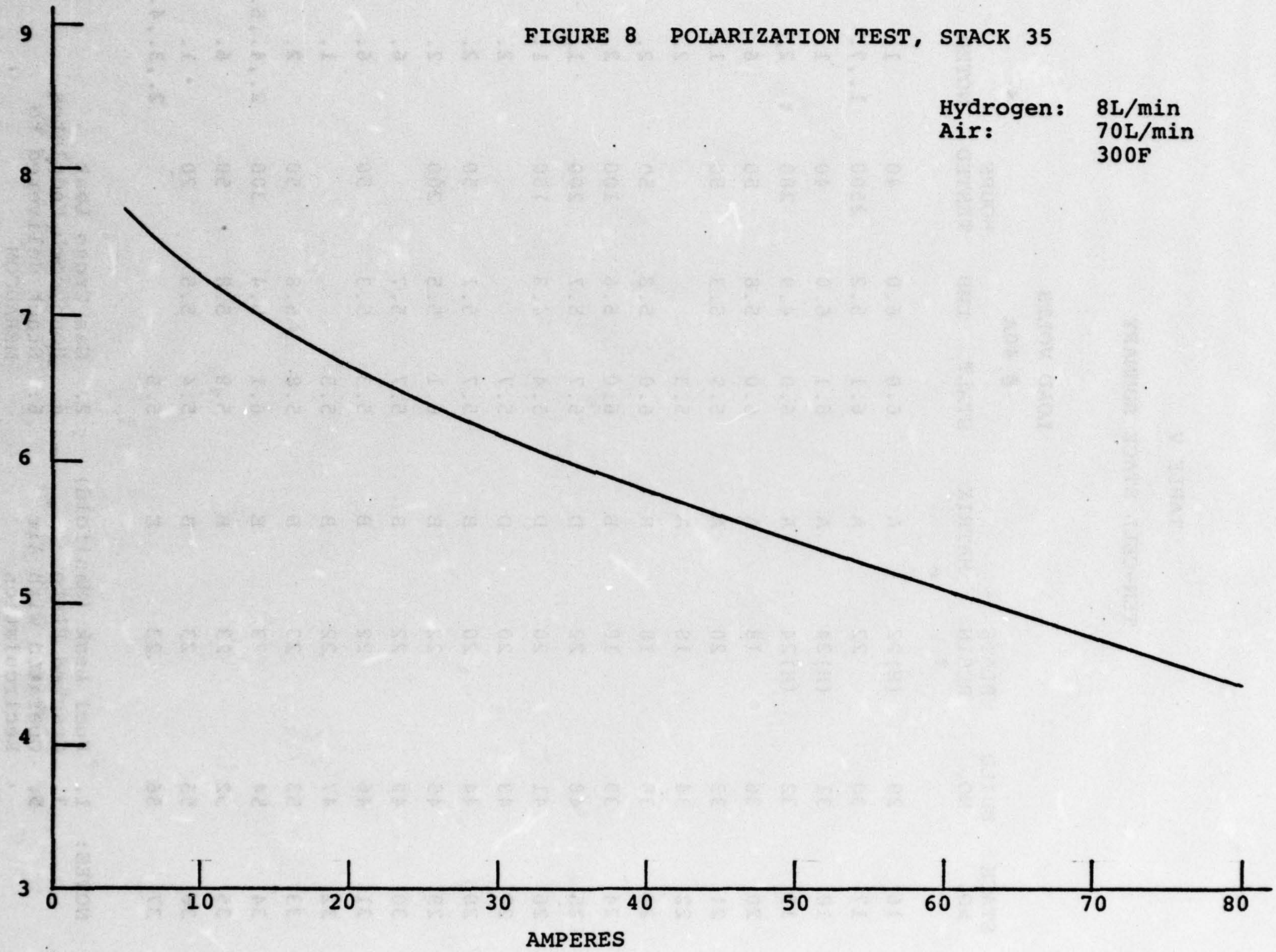

TABLE V

TEN-CELL STACK SUMMARY

STACK NO.	BUILD NO.	PLATE RESIN %	MATRIX	LOAD VOLTS @ 40A START	LOAD VOLTS @ 40A END	HOURS TESTED	NOTES
16	29	(H)22	A	6.0	6.0	40	1.
17	30	22	A	6.1	5.2	2500	1.,7.
18	31	(H)24	A	6.1	6.0	40	1.
19	32	(H)24	A	6.0	4.9	280	2.
20	36	18	A	6.0	5.8	50	6.
21	33	20	A	5.9	5.3	50	1.
22	34	18	A	5.3			2.
23	38	18	B	6.0	5.8	50	2.
24	39	18	B	6.0	5.6	100	2.
25	48	22	D	5.7	5.7	200	1.
26	41	20	D	5.4	4.3	100	1.
27	43	20	D	5.7			2.
28	44	20	B	5.7	5.1	50	2.
29	45	22	B	6.1	5.5	200	2.
30	49	22	B	5.7	5.7		6.
31	46	22	B	5.3	5.3	50	6.
32	47	22	B	5.5			1.
33	53	23	B	5.8	5.8	50	2.
34	54	23	E	6.1	5.4	300	2.,4.,5.
35	52	23	B	5.8	5.8	50	6.
36	55	23	B	5.4	5.5	20	1.
37	56	23	E	5.5			2.,3.,4.

NOTES:
1. Fuel Leak (Manifold)
2. Gas Cross Leak
3. Cracked Plate
4. Honeycomb Endplates
5. Operated with Air Recirculation
6. Stack delivered to MERADCOM
7. On test

(H) = H-Resin

TABLE VI

35-CELL STACK SUMMARY

STACK NO.	BUILD NO.	PLATE RESIN %	MATRIX	LOAD VOLTS @ 40A START	LOAD VOLTS @ 40A END	HOURS TESTED	NOTES
1	50	22	D	21.0	19.1	20	1.,2.
2	51	23	D	19.6		10	2.
3	57	23	D	19.9		10	1.,2.
4	58	24	D	20.5		10	3.
5	60	24	D & H	20.5		10	3.

NOTES: 1. Fuel Leak (Manifold) 2. Gas Cross Leak

3. On test

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